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Eggshell Waste : An Efficient Solid Catalyst for the Synthesisof 5-Arylidene Barbituric Acids under Solvent-Free Condition

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ABSTRACT

Efficient synthesis of 5-arylidene barbituric acid derivatives in presence of chicken eggshell waste at room temperature with grinding has been reported. Biologically active 5-arylidene barbituric acid derivatives were obtained in high yields (88-96 %) in a short reaction time (2-7 min) under solvent-free condition. This method has advantages such as avoidance of the organic solvents, short reaction times and production of pure products without any by product.

Keywords : Chicken eggshell waste, 5-Arylidene barbituric acid derivatives, Green catalyst, Grinding, Solvent free condition.

I. INTRODUCTION

Derivatives of barbituric acids have been widely used as anaesthetic, sedative, anticonvulsant, hypnotic, antitumor agents and antiosteoporosis[1].The 5-Aryl-pyrano pyrimidine-2, 4-diones obtained from 5-arylidene barbituric acids has biological activities like antibacterial[2], antiviral[3], prostate-protective[4]and antifungal[5]. Arylidene barbituric acids are useful as intermediates in synthesis of benzyl barbituric derivatives[6], unsymmetrical derivatives[7]and loxadiazaflavines[8]. Recently some of them have been used as dyes[9].A 5-arylidene barbituric acids can be considered as models of redox coenzymes such as FAD, NAD and used as oxidants for mild oxidation of thiols[7, 10] and alcohols[11].

Various methods have been reported for the synthesis of 5-arylidene barbituric acid derivatives like amino sulfonic acid[12], microwave irradiation[13],$CoFe_2O_4$ nanoparticles[14], ionic liquid[15],infra-red promoted[16], nickel nanoparticles[17], $LaCl_3.7H_2O$[18] and various acid-base catalyzed condensation reactions[19-22].However, many of these methods suffer from the drawbacks of green chemistry[23]. Therefore the eco-friendly, heterogeneous, clean process and green catalysts which can be recycled are under permanent attention. The solvent-free reaction represents very powerful green chemical procedure from both economical and synthetic point of view.

In continuation to our ongoing research on the synthesis of heterocyclic molecules using nanoparticles as a catalyst[24-29]and to explore the importance of chicken eggshell waste as a catalyst in organic synthesis[30], hereinwe wish to report a simple synthesis of 5-arylidene barbituric acid derivatives using chicken eggshell wasteas reusable catalyst under solvent-free conditions with grinding at room temperature.

II. EXPERIMENTAL SECTION

Preparation of chicken eggshell waste catalyst

The waste chicken eggshell was collected, washed and dried at room temperature. The eggshell was crushed and grind by mortar and pestle. The fine powder was dried at 150°C in heating oven and used as catalyst in reaction.

Synthesis of 5-arylidene barbituric acid derivatives

Aromatic aldehydes **1** (1.0 mmol) and barbituric acid **2** (1.0 mmol) were mixed with chicken eggshell waste as a catalyst (0.040 g) in a mortar at room temperature. The reaction mixture was grounded and reaction was monitored by TLC. The solid residue was dissolved in hot ethanol and filtered off. The catalyst was separated by filtration. The crude product was collected from filtrate after cooling to room temperature and recrystallized from ethanol to give products **3** (**a-i**). The spectral data IR, ^{1}H NMR, ^{13}C NMR and MS of all synthesized compounds are reported.

5-Benzylidene barbituric acid (Table II, entry 3a**)**: m. p. 263°C; IR (KBr): 3459, 3219, 3062, 1747, 1565 cm^{-1};^{1}H NMR (400 MHz, DMSO-d_6): δ 11.22 (s, 1H), 11.10 (s, 1H), 10.58 (s, 1H), 8.33-8.27 (m, 3H), 6.85 (d, 2H, *J*=7.10 Hz);^{13}C NMR (100 MHz, DMSO-d_6): δ 163.6, 162.2, 155.7, 150.4, 133.7, 133.3, 132.4, 128.3, 119.2; M. F: $C_{11}H_8N_2O_3$; M. W: 216; MS (*m/z*): 217 (M+1)$^{+}$.

5-(4-Chlorobenzylidene) barbituric acid (Table II, entry 3b**)**: m. p. 299°C; IR (KBr): 3430, 3214, 3087, 1752, 1675, 1578, 1090 cm^{-1};^{1}H NMR (400 MHz, DMSO-d_6): δ 11.37 (s, 1H), 11.20 (s, 1H), 8.24 (d, 2H, *J*=8.0 Hz), 8.07 (s, 1H), 7.51 (d, 2H, *J*=8.0 Hz);^{13}C NMR (100 MHz, DMSO-d_6): δ 163.6, 162.1, 153.4, 150.1, 137.5, 135.3, 132.1, 128.3, 120.2; M. F: $C_{11}H_7ClN_2O_3$; M. W: 251; MS (*m/z*): 252 (M+1)$^{+}$.

5-(4-Methylbenzylidene) barbituric acid (Table II, entry 3c**)**: m. p. 279°C; IR (KBr): 3490, 3350, 3092, 1729, 1679, 1658, 1574 cm^{-1};^{1}H NMR (400 MHz, DMSO-d_6): δ 11.43 (s, 1H), 11.26 (s, 1H), 8.34 (s, 1H), 8.13-7.38 (m, 4H), 2.15 (s, 3H);^{13}C NMR (100 MHz, DMSO-d_6): δ 163.8, 162.1, 155.7, 150.6, 143.8, 134.5, 130.2, 129.4, 118.1, 21.6; M. F: $C_{12}H_{10}N_2O_3$; M. W: 230; MS (*m/z*): 231 (M+1)$^{+}$.

5-(4-Bromobenzylidene) barbituric acid (Table II, entry 3d**)**: m. p. 293°C; IR (KBr): 3495, 3360, 3090, 1737, 1672, 1561 cm^{-1};^{1}H NMR (400 MHz, DMSO-d_6): δ 11.41 (s, 1H), 11.24 (s, 1H), 8.24 (s, 1H), 8.01-7.77 (m, 4H);^{13}C NMR (100 MHz, DMSO-d_6): δ 165.11, 160.2, 153.2, 150.2, 134.5, 131.8, 131.1, 127.6, 119.7; M. F: $C_{11}H_7BrN_2O_3$; M. W: 295; MS (*m/z*): 296 (M+1)$^{+}$.

5-(4-Hydroxybenzylidene) barbituric acid (Table II, entry 3e**)**: m. p. >300°C; IR (KBr): 3542, 3418, 3260, 3080, 1742, 1660, 1581, 1527, 1281 cm^{-1};^{1}H NMR (400 MHz, DMSO-d_6): δ 11.22 (s, 1H), 11.10 (s, 1H), 10.58 (s, 1H), 8.33-8.27 (m, 3H), 6.85 (d, 2H, *J*=8.80 Hz);^{13}C NMR (100 MHz, DMSO-d_6): δ 164.01, 163.16, 162.02, 156.25, 150.09, 138.57, 123.65, 115.37, 113.45; M. F: $C_{11}H_8N_2O_4$; M. W: 232; MS (*m/z*): 233 (M+1)$^{+}$.

5-(3-Nitrobenzylidene) barbituric acid (Table II, entry 3f**)**: m. p. 246°C; IR (KBr): 3442, 3240, 3095, 1780, 1697, 1596, 1537, 1435 cm^{-1};^{1}H NMR (400 MHz, DMSO-d_6): δ 11.45 (s, 1H), 11.30 (s, 1H), 8.90 (s, 1H), 8.34 (s, 1H), 8.31-8.28 (m, 1H), 8.23-8.21 (m, 1H), 7.73 (t, 1H, *J*=8.0 Hz);^{13}C NMR (100 MHz, DMSO-d_6): δ 165.0, 162.4, 152.3, 150.4, 150.1, 145.0, 135.0, 133.4, 132.4, 131.2, 118.1; M. F: $C_{11}H_7N_3O_5$; M. W: 261; MS (*m/z*): 262 (M+1)$^{+}$.

5-(4-Nitrobenzylidene) barbituric acid (Table II, entry 3g**)**: m. p. 294°C; IR (KBr): 3323, 3242, 3095, 1742, 1692, 1596, 1517 cm^{-1};^{1}H NMR (400 MHz, DMSO-d_6): δ 11.46 (s, 1H), 11.30 (s, 1H), 8.31 (s, 1H), 8.23 (d, 2H, *J*=8.70 Hz), 8.01 (d, 2H, *J*=8.70 Hz);^{13}C NMR (100 MHz, DMSO-d_6): δ 165.2, 162.0, 157.3, 150.2, 136.4, 136.2, 135.1, 134.4, 118.1; M. F: $C_{11}H_7N_3O_5$; M. W: 261; MS (*m/z*): 262 (M+1)$^{+}$.

5-(4-Methoxybenzylidene) barbituric acid (Table II, entry 3h**)**: m. p. 277°C; IR (KBr): 3401, 3233, 3094, 1712, 1672, 1546, 1206 cm^{-1};^{1}H NMR (400 MHz, DMSO-d_6): δ 11.32 (s, 1H), 11.17 (s, 1H), 8.30 (s, 1H), 8.12-7.35 (m, 4H), 3.84 (s, 3H);^{13}C NMR (100 MHz, DMSO-d_6): δ 164.4, 163.6, 162.7, 155.2, 150.2, 137.5, 125.4, 115.7, 114.2, 56.2; M. F: $C_{12}H_{10}N_2O_4$; M. W: 246; MS (*m/z*): 247 (M+1)$^{+}$.

5-(2-Chlorobenzylidene) barbituric acid (Table II, entry 3i**)**: m. p. 253°C; IR (KBr): 3360, 3230, 3075, 1732, 1657, 1547 cm^{-1};^{1}H NMR (400 MHz, DMSO-d_6): δ 11.56 (s, 1H), 11.29 (s, 1H), 8.33 (s, 1H), 7.77 (d, 1H, *J*=7.60 Hz), 7.72 (d, 1H, *J*=7.60 Hz), 7.49 (d, 1H, *J*=7.60 Hz), 7.38 (d, 1H, *J*=7.60 Hz); ^{13}C NMR (100 MHz, DMSO-d$_6$): δ 162.7, 161.1, 150.9, 150.1, 133.6, 132.4, 132.1, 130.9, 129.4, 126.7, 122.3; M. F: $C_{11}H_7ClN_2O_3$; M. W: 251; MS (*m/z*): 252 (M+1)$^+$.

III. RESULTS AND DISCUSSION

The reaction between benzaldehyde **1** (1.0 mmol) and barbituric acid **2** (1.0 mmol) was used as a model reaction to optimize the amount of catalyst (**Scheme**). It was found that 0.040 g ofchicken eggshell wastewas the appropriate quantity of the catalyst to offer the reaction (**Table I**). All reactions were performed by grinding method at room temperature. The reactions were completed within 2-7 min with excellent product yields (88-96 %). A blank reaction of benzaldehyde and barbituricacid was performed to confirm the effectiveness of chicken eggshell wasteduring this reaction. In absence of chicken eggshell waste, the reaction was incomplete even after 9 hrs.

After optimizing the reaction conditions, a variety of aromatic aldehydes with barbituric acid were employed under reaction conditions to evaluate the scope of this reaction. A series of 5-arylidene barbituric acid derivatives **3(a-i)**were prepared by using chicken eggshell wasteas a catalyst (**Table II**) with excellent yields (88-96 %) at room temperature. The reactions proceeds smoothly and no undesirable side reactions were observed. The nature of substituents on the aromatic ring does not affect on the condensation reaction. The condensation reactions of aromatic aldehydes carrying electron-donating or electron-drawing groups were also successfully carried out with this method in excellent yields and short reaction times.

In view of eco-friendly procedure, the catalytic efficiency of chicken eggshell wastewas also checked by its recovery and reusability. It was found that catalyst showed good results after four successive runs. It shows the same activity as fresh catalyst without any significant loss in its activity (**Table III**).

Table I. Effect of catalyst

Entry	Amount of catalyst (g)	Time (min)	Yield(%)
1	0.010	5	87
2	0.020	4	89
3	0.030	3	91
4	0.040	2	92
5	0.050	2	92
6	0.060	2	92

Table II. Synthesis of 5-Arylidene barbituric acid derivatives using chicken eggshell waste

Entry	Products (R group)	Time (min)	Yield(%)
3a	H	2	92
3b	4-Cl	3	94
3c	4-CH_3	7	93
3d	4-Br	2	95
3e	4-OH	3	96
3f	3-NO_2	4	88
3g	4-NO_2	2	94
3h	4-OCH_3	3	89
3i	2-Cl	3	90

Table III. Recycling of catalyst

Cycles	Yield(%)
Initial	92
1	92
2	91
3	89
4	88

IV. CONCLUSIONS

In this study chicken eggshell wasteas an efficient solid catalyst for the synthesisof biologically active 5-arylidene barbituric acid derivatives with grinding method under solvent-free condition has been described. The present method offers significant advantages such as simple work up, shorter reaction times, excellent yields, environmentally benign, grinding method, solvent-free reaction condition and reusability of catalyst, this protocol is economic and eco-friendly.

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Bio-plastic Synthesis from Plant Substances

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ABSTRACT

Bio-plastic from acetylated starch of phaseolus vulgaris beans, Hylocereous undatus fruit peels and Euphorbia tirucalli latex offers a new combination of starting material, easy and inexpensive method, effective results for crucial tests. The starch, latex and fruit peel paste were mixed in certain proportion and heated at 1200C. The mixture was spread onto petri dish and dried in oven at 600C. The solvent uptake capability of bioplastic was checked with solvents like NaOH, dilute HCl, Ethanol, Acetone and water. The solvent uptake test was carried out to determine the absorption capability of bioplastic for selected solvents. The results shows that the plastic dipped in dilute HCl and NaOH (2N) get swollen. Less absorption is observed in ethanol, distilled water and acetone. Polyhydroxybutyrate (PHB) is a bio-degradable polymer belonging to the polyhydroxyalkanoates (PHA). The high plasticity generating sources when combined under appropriate conditions help in the synthesis of biodegradable polymer i.e. PHB.[1]

Keywords : Phaseolus vulgaris, Hylocereous undatus and Euphorbia tirucalli Polyhydroxyalkanoate, NaOH, dilute HCl, Ethanol, Acetone

I. INTRODUCTION

The overabundant use of plastic generate enormous environmental as well as health issues. Usually plastic is made up of heterochain polymers obtained from polymerization of raw material such as oil, natural gas, and coal. This raw material is limited and synthetic plastic are resistant to degradation. Consequently, their disposal problem is the driving force for the piercing of ecological alternatives. Since plastic is inseparable in our daily lives numerous substitutes are being launched. Replacement of fossil fuel with biomass for production of new polymers can have both economic and environmental benefits. Starch is the most preferred key ingredient for the synthesis of bio-plastic. The other composites with starch are agricultural byproducts and plasticizers like glycerine, plant seed oil, cellulose molding compounds etc.[2]

Selection of starting material

1. Starch

Starch is a easily available, affordable and bio-degradable polymer. Synthesis of bioplastic from starch of plants like yam, corn, potato, cassava, pumpkin, various beans is already reported. Starch in its native form has limited applications; hence, to process it with desired properties, acetylation of starch was carried out. In the acetylation process, the hydroxyl group of the glucose monomer are converted to the CH_3COO- group[3] The substitution of bulky acetyl groups increases the hydrophobicity and thermo plasticity and decreases the gelatinization temperature of the starch. In this work acetylated starch of *phaseolus vulgaris* is used.

2. Latex

To improve the flexibility, tensile strength and expediency the plasticizers are used the plastic synthesis. Preferring the natural plasticizers the latex of plants can be used. The *Euphorbia tirucalli* is well known and easily available plant in regions of India. The latex of *Euphorbia tirucalli* was used as plasticizer.

3. *Hylocereous undatus* fruit peels

The peels *Hylocereous undatus* i.e dragon fruit is highly fibrous and antioxidant.[4] The peels are the agricultural waste of fruit processing industries. It is cultivated in regions of tropical climate.

II. MATERIAL AND METHODS

Extraction of starch

For the extraction of starch from *phaseolus vulgaris* beans, 500 gm beans were soaked in water for 15 hours. The beans are peeled off and pestle in grinder. The fine paste was mixed with 2 litre distilled water and strained.The filtrate was allowed to set for 8 hours. The starch settled at bottom was collected. The wet starch deposition was dried in oven at 80^0C for one hour. The dried starch weighed 55gm.

Acetylation of starch

The acetylation of starch in aqueous medium was carried out by the process mentioned by[3] Rosana Colussi, Shanise Lisie Mello El Halal, Vania Zanella Pinto, Josiane Bartz, Louz Carlos Gutkoski, elessandra da Rosa Zavareze, Alvaro Renato Guerra Dias / LWT- Food science and Technology 62 (2015) 1076-108. 25 gm of starch was dissolved in 56 ml distilled water with stirring on magnetic stirrer 900 rpm at room temperature. The P^H was adjusted to 8 by addition of 3% NaOH solution. The acetic unhydride was added portion wise 2.5 ml, 5ml and 10 ml. After the complete addition of acetic unhydride the reaction was continued for 15 min. The P^H was adjusted to 4.5 with 0.5M HCl solution. The suspension was centrifuged for 5 min and washed with 95% ethanol. The acetylated starch was dried in oven at 60^0C.

Collection of latex

The latex was collected from *Euphorbia tirucalli plant* near Rajapur village, Sangamner taluka of Ahmednagar district, Maharashtra, India. The branches of plant were given slight cut to get latex.

Preparation of bio-plastic

The *Hylocereous undatus* fruit peel was sterilized and cut into small pieces. The pieces were grinded in grinder to fine paste. The fruit peel paste and acetic acid in 2:1 proportion was mixed on placing it on magnetic stirrer for 10 minutes. Then drop by drop addition of the 2ml latex followed by 1gm of acetylated starch was carried out. The blend was stirred for 10-15 minutes, then heated to 120^0C and immediately spread on Petri dish. The set layer was dried in oven at 60^0C for 10 minutes. After complete removal of moisture from bio-plastic, the product was used to analyze by FTIR spectrum and the solvent uptake test.

Solvent uptake test

This test is based on method performed by M Hasan, et al. 2018 IOP Conf. Ser.Mater.Sci.Eng.333 012087) The bio-plastic was cut into 1x1 cm pieces and weighed. The 5ml of each solvents like H2O, ethanol, acetone, dilute HCl, sodium hydroxide are taken into a beaker. The weighed pieces are dipped into the solvents for 5 minutes. After 5minutes the pieces are removed and wiped with tissue paper. The pieces are again weighed and the % absorption is determined by using the formula

$$\text{Solvent uptake} = \frac{W - W_0}{W_0} \times 100\%$$

Where W_0 = weight of dry sample, W = weight of sample after immersion in the solvent.

III. RESULT AND DISCUSSION

The bio-plastic obtained by this method is flexible at room temperature. The color of the product is off white to yellow. The well dried film is uneasy to powdered for FTIR-spectrum.

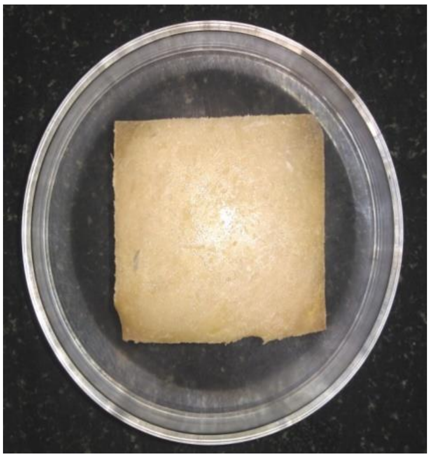

Fig.1. Bio-plastic film

Solvent uptake test

The solvent absorption test is carried out to check the lyophobicity of the synthesized plastic. The results are tabulated below. The plastic can be said hydrophobic as % absorption of water is less. The negative percent is due to the slight solubility of plastic in acetone and ethanol. The lyophobicity is less in case of HCl and NaOH.

Table 1. % Absorption of solvent

Solvent used	**% Absorption**
Distilled water	10.20%
Ethanol	-7.69%
Dil.HCl	22.55%
NaOH	31.09%
Acetone	-0.72%

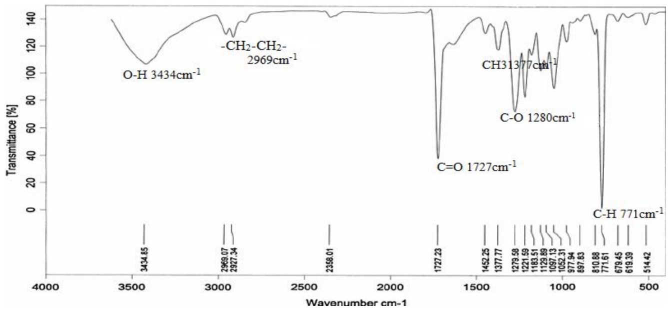

Fig 2. FTIR-spectrum of polyhydroxybutyrate – PHB

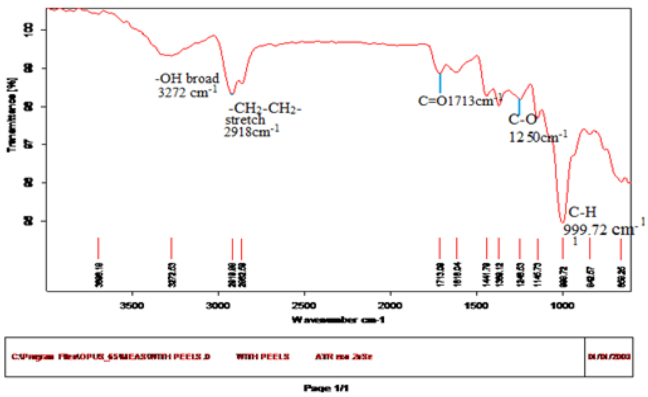

Fig 3. FTIR-spectrum of bioplastic

Table 2. Comparision of IR frequencies of bio-plastic and standard PHB FTIR spectra

No.	C=O cm^{-1}	C-O cm^{-1}	O-H cm^{-1}	CH2-CH2 cm^{-1}	C-H cm^{-1}
Standard PHB	1727.23	1280.58	3434.15	2969.07	771.63
Bio-plastic	1713.09	1250.53	3272.63	2918.88	999.72

The IR frequencies of bio-plastic nearly matches with the standard PHB frequencies, denoting the presence of polyhydroxybutyrate.

IV. CONCLUSION

The combination of phaseolus vulgaris beans, Hylocereous undatus fruit peels and Euphorbia tirucalli latex indicates effective results when compared with standard PHB IR frequencies. The solvent absorption test will help for further modification of process.

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Study of Fluid Flow Using Matrix Analysis

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ABSTRACT

Fluids are either gases or liquids. Mathematical methods in Chemistry are one of the core subjects in applied mathematics. Fluid dynamics is one of important branch of Mathematical Chemistry deals with study of motion of fluids i.e. liquid, gases and vapors. Fluid flows as a continuum. It is considered as a single entity whiles it in motion. Electricity, heat transfer, light prorogate are various forms of energy are some of the important examples of fluid flow. The all equations of motions of fluid are very important to study and it is important to note that all these equations are expressed in mathematical tools such as differential equations, nonlinear equations partial differential equations. In present paper, we are converting the Nevier-Stokes equations into matrix form. The motion of fluid is studied using properties of matrix such as determinant, rank, eigen value properties are used to study path of the motion of fluid.

Keywords : Fluid Flow, Chemistry, Navier-Stokes Equations, Matrix Analysis

I. INTRODUCTION

Most of these fluid flow equations are nonlinear in nature. They are called Navier-Stokes equations as they are firstly derived by French mathematician Claude-Louis Navier (1822) and then by Englishman G. G. Stokes (1845) independently.

II. BASIC TERMINOLOGIES

In general fluid motion takes place in 3-d space. Also it involves more than one independent variable. It is important to study multivariable calculus in order to understand the equations. First consider the space R^3. It is set of all ordered 3- tuples.

i.e. $R^3 = \{(x, y, z) / x, y, z \in R\}$. It's dimention is 3. It is real vector space under component wise addition and scalar multiplication. In general $R^n = \{(x_1, x_2 ,\dots x_n) / x_1, x_2 \dots x_n \in R\}$. is n-dimentional real vector space under component wise addition and scalar multiplication. R^n is a metric space under Euclidean metric. As study is on motion of fluid in R^3, let us define some of the important terminologies in fluid dynamics in order to formulate the Navier-Stokes equations.

Fluid[1]**:** A fluid is a liquid, gas or vapor. A liquid has no shape. It will fill the container which contains it. **Ideal and rear flow**[1]**:** when there is no friction between adjacent moving particles, the flow is called an ideal flow, internal forces at section are always normal to the section. **Velocity**[1]**:** Velocity of a particle is a time derivative of displacement of the particle in a certain direction. **Density:** Mass of particle divided by it's volume is density and is denoted by ρ. In the CGS system the dimensions of ρ

are g/cm^3. C**ompressible fluid:** A fluid whose pressure changes it's volume is called compressible fluid. **Acceleration**[2] : The time rate of change of velocity is called acceleration. The velocity involves both directions and magnitude. So change in either magnitude or direction of the velocity produces acceleration. **Viscosity**[3]**:** The viscosity of a fluid is a measure of it's resistance to shear or angular deformation. Consider two parallel plates large enough that edge conditions may be neglected and placed a small distance apart and assume the space between is filled with the fluid shown in

Absolute Viscosity: In the metric system, the units of absolute viscosity is the poise (1 poise = 1 dyne-sec / cm^2). **Kinematic Viscosity**[2]**:** kinematic viscosity is the ratio of viscosity to the density. That is kinematic viscosity $\nu = \frac{\mu}{\rho}$. To derive equations of motion of liquid, let us first understand some of the basic physical laws. By Newton's 2nd law[2] of motion $\sum f_B + \sum f_S = m.\ a$. where f_B and f_S are body force and surface force as total external force while right hand side represents the inertial force.

Derivation of Navier-Stokes equations:

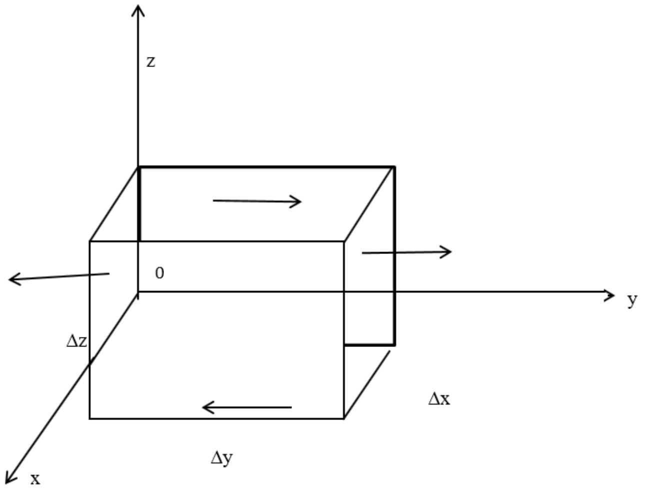

Fig 1. Dynamic of fluid element

Consider the motion of infinitesimal fluid element as shown in Fig. No. 1.3. No by Newtons laws of motion, the net resultant force on fluid element in y-directions with control volume[3] $\Delta v = \Delta x.\Delta y.\Delta z$ is equal to mass into acceleration[4] in y direction. So mathematically,

$$\sum F_{By} + \sum F_{Sy} = m.\ a_y \ldots \text{ is the momentum equation} \qquad \ldots\ldots\ldots\ldots\ldots(1)$$

Body force along y direction $F_{By} = \rho.B_y.\Delta x.\Delta y.\Delta z$,

$$a_y = \frac{Dv}{Dt} = \frac{\partial v}{\partial t} + u.\frac{\partial v}{\partial x} + v.\frac{\partial v}{\partial y} + w.\frac{\partial v}{\partial z},$$

$$\begin{aligned} \text{Also } \sum F_{Sy} &= \left(\sigma_{yy} + \frac{\partial\sigma_{yy}}{\partial y}.\frac{\Delta y}{2}\right)\Delta x.\Delta z - \left(\sigma_{yy} - \frac{\partial\sigma_{yy}}{\partial y}.\frac{\Delta y}{2}\right)\Delta x.\Delta z \\ &\quad + \left(\tau_{xy} + \frac{\partial\tau_{xy}}{\partial x}.\frac{\Delta x}{2}\right)\Delta y.\Delta z - \left(\tau_{xy} - \frac{\partial\tau_{xy}}{\partial x}.\frac{\Delta x}{2}\right)\Delta y.\Delta z \\ &\quad + \left(\tau_{zy} + \frac{\partial\tau_{zy}}{\partial z}.\frac{\Delta z}{2}\right)\Delta x.\Delta y - \left(\tau_{xy} - \frac{\partial\tau_{zy}}{\partial z}.\frac{\Delta z}{2}\right)\Delta x.\Delta y \\ &= 2.\ \frac{\partial\sigma_{yy}}{\partial y}.\frac{\Delta y}{2}.\Delta x.\Delta z + 2.\ \frac{\partial\tau_{xy}}{\partial y}.\frac{\Delta x}{2}.\Delta y.\Delta z + 2.\ \frac{\partial\tau_{zy}}{\partial z}.\frac{\Delta z}{2}.\Delta x.\Delta y \\ &= \left(\frac{\partial\sigma_{yy}}{\partial y} + \frac{\partial\tau_{xy}}{\partial x} + \frac{\partial\tau_{zy}}{\partial z}\right).\Delta x.\Delta y.\Delta z. \end{aligned}$$

Aslo mass of controlled volume so chosen as $\rho.\Delta V = \rho.\Delta x.\Delta y.\Delta z$.

So equation (1) becomes

$$\left(\frac{\partial\sigma_{yy}}{\partial y} + \frac{\partial\tau_{xy}}{\partial x} + \frac{\partial\tau_{zy}}{\partial z}\right).\Delta x.\Delta y.\Delta z + \rho.B_y.\Delta x.\Delta y.\Delta z = \rho.\Delta x.\Delta y.\Delta z\left(\frac{\partial v}{\partial t} + u.\frac{\partial v}{\partial x} + v.\frac{\partial v}{\partial y} + w.\frac{\partial v}{\partial z}\right)$$

We devide both sides by factor Δx.Δy.Δz we get,

$$\left(\frac{\partial \sigma_{yy}}{\partial y} + \frac{\partial \tau_{xy}}{\partial x} + \frac{\partial \tau_{zy}}{\partial z}\right) + \rho.B_y = \rho.\left(\frac{\partial v}{\partial t} + u.\frac{\partial v}{\partial x} + v.\frac{\partial v}{\partial y} + w.\frac{\partial v}{\partial z}\right) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(1)$$

On the left hand side first 3 terms represent surface force per unit volume while the fourth term represents body force of fluid per unit volume[4]. On the right hand side the terms is inertial force per unit volume. Similarly we can derive the equation of motion along x and z direction. So we get total 3 sets of equations are called Navier-Stokes equations.

By conservation of mass, momentum and energy and using Newton's II law we can derive Navier-Stokes Equations in R^3 as

$$\frac{\partial(\rho u_x)}{\partial t} + \frac{\partial(\rho u_x^2)}{\partial x} + \frac{\partial(\rho u_x u_y)}{\partial y} + \frac{\partial(\rho u_x u_z)}{\partial z} = -\frac{\partial p}{\partial x} + \frac{1}{Re_r}\left[\frac{\partial \tau_{xx}}{\partial x} + \frac{\partial \tau_{xy}}{\partial y} + \frac{\partial \tau_{xz}}{\partial z}\right] \quad \text{(along x-axis)}$$

$$\frac{\partial(\rho u_y)}{\partial t} + \frac{\partial(\rho u_x u_y))}{\partial x} + \frac{\partial(\rho u_y^2)}{\partial y} + \frac{\partial(\rho u_y u_z)}{\partial z} = -\frac{\partial p}{\partial y} + \frac{1}{Re_r}\left[\frac{\partial \tau_{xy}}{\partial x} + \frac{\partial \tau_{yy}}{\partial y} + \frac{\partial \tau_{yz}}{\partial z}\right] \quad \text{(along y-axis)}$$

$$\frac{\partial(\rho u_z)}{\partial t} + \frac{\partial(\rho u_x u_z)}{\partial x} + \frac{\partial(\rho u_y u_z)}{\partial y} + \frac{\partial(\rho u_z^2)}{\partial z} = -\frac{\partial p}{\partial z} + \frac{1}{Re_r}\left[\frac{\partial \tau_{xz}}{\partial x} + \frac{\partial \tau_{yz}}{\partial y} + \frac{\partial \tau_{zz}}{\partial z}\right] \quad \text{(along z-axis).}$$

These are three equations and unknown are more so for unique and smooth solution, add two more constraints 1) continuity of conservation of momentum and 2) conservation of energy equation given as below

$$\frac{\partial(\rho)}{\partial t} + \frac{\partial(\rho u_x)}{\partial x} + + \frac{\partial(\rho u_y)}{\partial y} + \frac{\partial(\rho u_z)}{\partial z} = 0 \text{ i.e. } \nabla.(\rho.U) = 0. \quad \text{(Continuity equation)}$$

And $$\frac{\partial(E_t)}{\partial t} + \frac{\partial(\rho u_x E_t)}{\partial x} + + \frac{\partial(\rho u_y E_t)}{\partial y} + \frac{\partial(\rho u_z E_t)}{\partial z} = -\frac{\partial(u_x p)}{\partial x} - \frac{\partial(u_y p)}{\partial y} - \frac{\partial(u_z p)}{\partial z} - \frac{1}{Re_r Pr_r}\left[\frac{\partial q_x}{\partial x} + \frac{\partial q_y}{\partial y} + \frac{\partial q_z}{\partial z}\right] + \frac{1}{Re_r}\left[\frac{\partial(u_x\tau_{xx} + u_y\tau_{xy} + u_z\tau_{xz})}{\partial x} + \frac{\partial(u_x\tau_{xy} + u_y\tau_{yy} + u_z\tau_{yz})}{\partial y} + \frac{\partial(u_x\tau_{xz} + u_y\tau_{yz} + u_z\tau_{zz})}{\partial z}\right] \quad \text{(Energy Equation)}$$

The Naiver-Stokes equations are second ordered non-linear partial differential equations. Let U =(u_x, u_y, u_z) represents the velocity of particle S and p denotes pressure exerted by fluid on S due to it's motion. Here E_t denotes total energy, Re is Reynolds number, ρ denote the density of fluid, P_r is Prandtl number, and τ denote amount of flux transport through unit area having nine components along all three directions. Here x, y, z and t are free variables and six unknown quantities as P = pressure, ρ = density, T= temperature, u_x = velocity component along x direction, u_y = velocity component along y direction, u_z = velocity component along z direction.

Let us write the system of first four of these equations into matrix form as

$$R_{e_r}\left(\left[\frac{\partial}{\partial t} \quad \frac{\partial}{\partial x} \quad \frac{\partial}{\partial y} \quad \frac{\partial}{\partial z}\right]\begin{bmatrix} \rho u_x & \rho u_y & \rho u_z & \rho \\ \rho u_x^2 + p & \rho u_x u_y & \rho u_x u_z & \rho u_x \\ \rho u_x u_y & \rho u_y^2 + p & \rho u_y u_z & \rho u_y \\ \rho u_x u_z & \rho u_y u_z & \rho u_z^2 + p & \rho u_z \end{bmatrix}\right) = \left(\left[\frac{\partial}{\partial t} \quad \frac{\partial}{\partial x} \quad \frac{\partial}{\partial y} \quad \frac{\partial}{\partial z}\right]\begin{bmatrix} 0 & 0 & 0 & 0 \\ \tau_{xx} & \tau_{xy} & \tau_{xz} & 0 \\ \tau_{xy} & \tau_{yy} & \tau_{yz} & 0 \\ \tau_{xz} & \tau_{yz} & \tau_{zz} & 0 \end{bmatrix}\right.$$

So the momentum matrix of the above equation is

$$\begin{bmatrix} \rho u_x & \rho u_y & \rho u_z & \rho \\ \rho u_x^2+p & \rho u_x u_y & \rho u_x u_z & \rho u_x \\ \rho u_x u_y & \rho u_y^2+p & \rho u_y u_z & \rho u_y \\ \rho u_x u_z & \rho u_y u_z & \rho u_z^2+p & \rho u_z \end{bmatrix} = \rho . \begin{bmatrix} u_x & u_y & u_z & 1 \\ u_x^2 & u_x u_y & u_x u_z & u_x \\ u_x u_y & u_y^2 & u_y u_z & u_y \\ u_x u_z & u_y u_z & u_z^2 & u_z \end{bmatrix} + \begin{bmatrix} 0 & 0 & 0 & 0 \\ p & 0 & 0 & 0 \\ 0 & p & 0 & 0 \\ 0 & 0 & p & 0 \end{bmatrix}$$

So determinant of velocity matrix is = $\det \left(\rho . \begin{bmatrix} u_x & u_y & u_z & 1 \\ u_x^2 & u_x u_y & u_x u_z & u_x \\ u_x u_y & u_y^2 & u_y u_z & u_y \\ u_x u_z & u_y u_z & u_z^2 & u_z \end{bmatrix}\right) =$

$$\rho^4 . u_x . u_y . u_z \ .\det\left(\begin{bmatrix} u_x & u_y & u_z & 1 \\ u_x & u_y & u_z & 1 \\ u_x & u_y & u_z & 1 \\ u_x & u_y & u_z & 1 \end{bmatrix}\right) = 0$$

Rank of velocity matrix is 1. So the range of motion of incompressible fluid is 1 dimensional. Hence we conclude that the motion of fluid is 1 dimensional curve in R^3. If R_{e_r} is constant throughout the motion then we have

$$\left[\frac{\partial}{\partial t} \quad \frac{\partial}{\partial x} \quad \frac{\partial}{\partial y} \quad \frac{\partial}{\partial z}\right] . R_{e_r} . \begin{bmatrix} \rho u_x & \rho u_y & \rho u_z & \rho \\ \rho u_x^2+p & \rho u_x u_y & \rho u_x u_z & \rho u_x \\ \rho u_x u_y & \rho u_y^2+p & \rho u_y u_z & \rho u_y \\ \rho u_x u_z & \rho u_y u_z & \rho u_z^2+p & \rho u_z \end{bmatrix} = (\left[\frac{\partial}{\partial t} \quad \frac{\partial}{\partial x} \quad \frac{\partial}{\partial y} \quad \frac{\partial}{\partial z}\right] \begin{bmatrix} 0 & 0 & 0 & 0 \\ \tau_{xx} & \tau_{xy} & \tau_{xz} & 0 \\ \tau_{xy} & \tau_{yy} & \tau_{yz} & 0 \\ \tau_{xz} & \tau_{yz} & \tau_{zz} & 0 \end{bmatrix}$$

$$\rightarrow \left[\frac{\partial}{\partial t} \quad \frac{\partial}{\partial x} \quad \frac{\partial}{\partial y} \quad \frac{\partial}{\partial z}\right] \left(R_{e_r} . \begin{bmatrix} \rho u_x & \rho u_y & \rho u_z & \rho \\ \rho u_x^2+p & \rho u_x u_y & \rho u_x u_z & \rho u_x \\ \rho u_x u_y & \rho u_y^2+p & \rho u_y u_z & \rho u_y \\ \rho u_x u_z & \rho u_y u_z & \rho u_z^2+p & \rho u_z \end{bmatrix} - \begin{bmatrix} 0 & 0 & 0 & 0 \\ \tau_{xx} & \tau_{xy} & \tau_{xz} & 0 \\ \tau_{xy} & \tau_{yy} & \tau_{yz} & 0 \\ \tau_{xz} & \tau_{yz} & \tau_{zz} & 0 \end{bmatrix} \right) = 0$$

$\rightarrow \nabla A = 0$ where

$$A = \left(R_{e_r} . \begin{bmatrix} \rho u_x & \rho u_y & \rho u_z & \rho \\ \rho u_x^2+p & \rho u_x u_y & \rho u_x u_z & \rho u_x \\ \rho u_x u_y & \rho u_y^2+p & \rho u_y u_z & \rho u_y \\ \rho u_x u_z & \rho u_y u_z & \rho u_z^2+p & \rho u_z \end{bmatrix} - \begin{bmatrix} 0 & 0 & 0 & 0 \\ \tau_{xx} & \tau_{xy} & \tau_{xz} & 0 \\ \tau_{xy} & \tau_{yy} & \tau_{yz} & 0 \\ \tau_{xz} & \tau_{yz} & \tau_{zz} & 0 \end{bmatrix} \right)$$

Here we also find that all the points where fluid motion takes placed are critical points at constant temperature. So the turbulence in motion can occur even if very small external force is applied. Also the divergence matrix is applied on the velocity matrix to get acceleration. So there is scope to study entire description motion of fluid i. e. extreme values, change of nature of path using Eigen values and trace of the acceleration matrix. Hassian matrix can be used to find extreme values of motion.

III. CONCLUSION

The Nevier-Stokes equations are converted into matrix form. Also all points of the motion through which the fluid is passing are critical points. So the turbulence for motion can occur even if very small force is applied. Velocity matrix has rank 1. So the motion of fluid is 1 dimensional curve in 3-space.

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Appraisal of Available Groundwater in Rural Area of Sinnar Tehsil (Nashik District, Maharashtra, India) for Drinking Purpose

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ABSTRACT

In the present investigation, the suitability of available groundwater for drinking application is assessed using parameters like pH, total dissolved solids (TDS), Fluoride, Iron and Nitrate. Fifteen samples collected from different villages in Sinnar tehsil area are analysed using standard water analysis protocol. Iron concentration oscillated from 0.09 to 1.35 mg/L and Nitrate concentration ranged from 01 to 124 mg/L, and the average is 51.86 mg/L in the study area. The Fluoride concentration in the study area is averaged to 0.18 mg/L. Only five of fifteen samples are found potable.

Keywords : Groundwater Quality, Iron, Nitrate

I. INTRODUCTION

Due to the growing of modernization, mechanization and agricultural activities the groundwater sources are becomes highly polluted. Drinking water is very essential for all kind of livestock but very few of groundwater sources are found to be potable. The quality of drinking water is disturb day by day in rural as well as in urban areas. In the present investigation, the suitability of available groundwater for drinking application was assessed using parameters like pH, total dissolved solids (TDS), Fluoride, Iron and Nitrate. Fifteen samples collected from different villages in Sinnar tehsil area are analysed using standard water analysis protocol.

II. METHODS AND MATERIAL

1. Sampling

Total fifteen samples (S_1 to S_{15}) are collected for analysis in cleaned polythene bottles. Out of fifteen samples, 01 is from Dug well, 10 are from Hand pumps, and 04 are from power pumps.

2. Experimental Methods

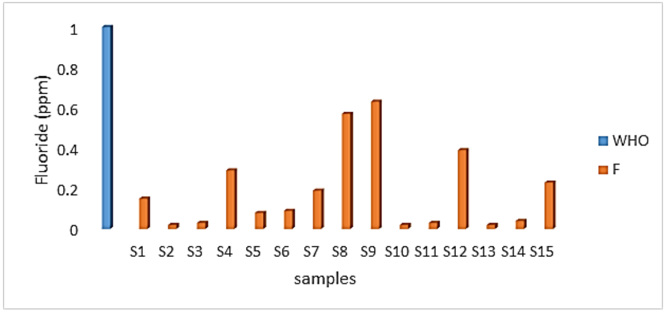
The standard methods of APHA are used for the complete analysis. Fluoride is analyzed by SPANDS method at 570 nm. Iron is estimated by UV Visible spectrophotometer at 480 nm (Chemito UV 2100). The nitrate concentration is measured by phenol disulphonic acid method at 410 nm using UV Visible spectrophotometer (Chemito UV 2100).

III. RESULTS AND DISCUSSION

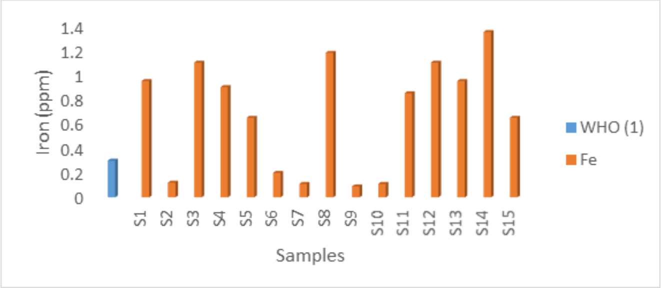
The investigated results are depicted in Table 1 along with permissible range as declared by WHO and BIS. The variation of parameters like Fluoride, Iron and Nitrate is shown graphically through Graph 1, Graph 2 and Graph 3 respectively.

Parameters of drinking water	WHO (1993)	BIS		Analyzed samples range
		Acceptable limit	Maximum Permissible Limit	
pH	Not mentioned	6.5-8.5	No relaxation	7.28-7.93
TDS (ppm)	No guidelines	500	2000	132-804
Fluoride(ppm)	1.5	1.0	1.5	0.02-0.63
Iron (ppm)	0.3	0.3	1.0	0.09-1.35
Nitrate(ppm)	45	45	45	1-124

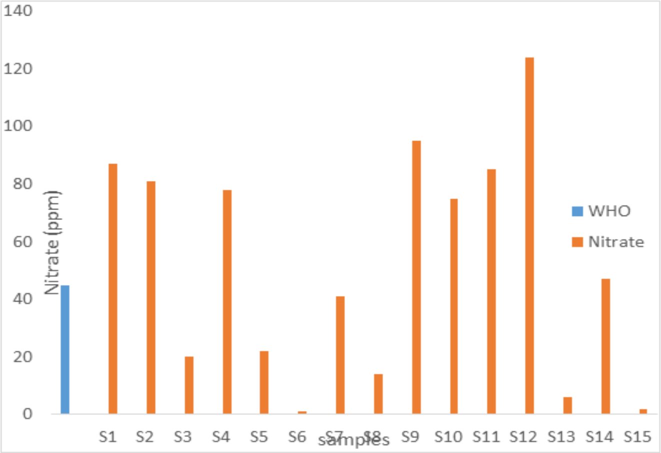
Graphical Representation



Graph 1. Variation of Fluoride in water samples



Graph 2. Variation of Iron in water samples



Graph 3. Variation of Nitrate in water samples

pH is an ecological factor of major importance in controlling the activities and distribution of aquatic Flora and Fauna[1, 2] and it is clear that all fifteen samples are within the pH range given in WHO recommendations for drinking water.

Generally the TDS contains the inorganic salts, with the small amount of organic matter. TDS in drinking water originates from natural sources, sewage, urban runoff and industrial wastewater[3]. The analyzed samples exhibited the TDS values from 132-804 ppm. Out of fifteen, 09 are within the permissible limit given by WHO.

Fluoride ions have dual significance in water supplies. High concentration of Fluoride ion causes dental fluorosis at the same time a less concentration results in dental caries[4,5]. The concentration of fluoride in analyzed samples are ranging from 0.02-0.63 ppm. All have fluoride content within the permissible limit declared by WHO.

The easily water soluble Iron (III) plays a very important role for human and organism in transportation of oxygen through the bloodstream. Less amount of Iron is essential for human nutrition and high concentration causes the hemochromatosis in tissues[4]. The Iron concentration oscillated from

0.09 to 1.35 mg/L in the studied area. Five samples lie within the permissible limit of Iron in drinking water.

The Nitrate ion is involved in Nitrogen Cycle. The sources of Nitrate in surface and groundwater is originated from agricultural activity, wastewater disposal and oxidation of nitrogenous waste products in human and animal excreta[3]. Nitrate concentration ranged from 1 to 124 mg/L. Seven samples qualify for the permissible limit given by WHO.

IV. CONCLUSION

Parameter	S_2	S_5	**S_6**	**S_7**	S_8	S_{13}	S_{15}
pH	√	√	**√**	**√**	√	√	√
TDS (ppm)	×	√	**√**	**√**	√	×	√
Fluoride(ppm)	√	√	**√**	**√**	×	√	√
Iron (ppm)	√	×	**√**	**√**	×	×	×
Nitrate(ppm)	×	√	**√**	**√**	√	√	√

From the above findings, it is concluded that only two water sources (S_6 & S_7) qualify to be potable water sources. For rest of the above sources, one has to make compromise for one or two parameters.

V. Acknowledgment

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Green Synthesis of Novel substituted 2-(1, 3-diphenyl-1*H*-pyrazol-3-yl) benzo [*d*] thiazole and using Ionic Liquid under Ultrasound and Microwave Condition

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ABSTRACT

A green protocol for the synthesis of series of novel substituted 2-(1, 3-diphenyl-1*H*-pyrazol-3-yl) benzo [*d*] thiazole is developed through the condensation of substituted 1, 3-diaryl pyrazole aldehyde and *o*-amino thiophenol in the presence of 1-butyl,3-methyl,1-imidazolium tetrafloroborate [BMIM][BF_4] as a catalyst under the conventional reflux condition, ultrasound and Microwave irradiation. The use of [BMIM][BF_4] under USI and MWI conditions, and with easier isolation of the products in good yields makes this protocol eco-friendly and versatile compared to the conventional reflux conditions. The structures of the products were confirmed by IR, Mass, ^{1}H-NMR, ^{13}C- NMR spectral data.

Keywords: 1, 3-diaryl pyrazole aldehyde, Microwave, Ultrasound, Thiazole, [BMIM][BF_4].

I. INTRODUCTION

Ionic liquids (ILs) offer an excellent and environmentally benign technique for the synthetic chemistry. The application of ionic liquids as reaction media and catalyst can offer a solution to solvent emission and catalyst recycle problems. Ionic liquids possess the important properties like negligible vapor pressure, thermal stability, recyclability, and higher solubility. Nowadays, ionic liquids have been successfully employed as solvents as well as catalyst for a variety of reactions [1-4]. ILs has been successfully used for various organic reactions like diels-alder reaction, electrochemical reaction, esterification, friedal-Craft reaction, hydrogenation, multicomponent reaction, coupling reaction [5-9].

The benzothiazole and their derivatives are an important class of heterocyclic compounds in medicinal, industrial, agricultural and synthetic organic chemistry. They are widely found in bioorganic and medicinal chemistry with applications in drug discovery such as antitumor, anticonvulsant, and antiviral applications [10–16]. They also found applications in industry as antioxidants, vulcanization accelerators, and as a dopant in a light-emitting organic electroluminescent device [17, 18]. Also the benzothiazole is an important nucleus in some rigid-rod polymer possessing high tensile strength, thermal stability and modulus [19, 20].

Many routes have been reported in the literature for the synthesis of benzothiazoles derivatives. However the most commonly used method involves the reaction of o-amino-thiophenols with substituted benzaldehydes in the presence of a catalyst such as animal bone meal, $ZnBr_2$/ABM, $ZnCl_2$/ABM, $CuBr_2$/ABM [21], Pt/Al_2O_3 [22], SiO_2 [23], vanadium

(IV)-salen complexes [24], $Fe_2(SO_4)_3$/TEMPO [25], *o*-benzene disulfonimide [26], sodium dodecylsulfate [27], H_2O_2/CAN [28], cetyl trimethyl ammonium bromide (CTAB) [29], molecular Iodine [30], *p*-TSA [31], diethyl bromo-phosphonate, tert-butyl hypochlorite [32], methanesulfonic acid/SiO_2 [33], $NaHSO_4$-SiO_2 [34], H_2O_2/$Fe(NO_3)_3$ [35], silica, montmorillonite K-10 [36], PPA [37], etc.

The precursor substituted 1,3-diphenyl pyrazole aldehydes were synthesized by the Vielsmeier Haack formylation reaction. Firstly the phenyl hydrazone derivative was prepared from the reaction of differently substituted acetophenone with phenyl hydrazine in the presence of glacial AcOH in ethanol and then formylation of hydrazone in presence of N,N-dimethyl formamide and Phosphorous Oxychloride yield substituted 1,3-diphenyl pyrazole aldehyde [38]. These pyrazole aldehyde analogues have acknowledged significant attention because of their broad range of pharmacological and biological activities [39-41]. Thus the pyrazole aldehydes are used to gain more powerful biologically active heterocyclic systems.

II. METHODS AND MATERIAL

GENERAL PROCEDURE FOR THE SYNTHESIS OF BENZO[*D*]THIAZOLE DERIVATIVES

A) UNDER REFLUX CONDITION

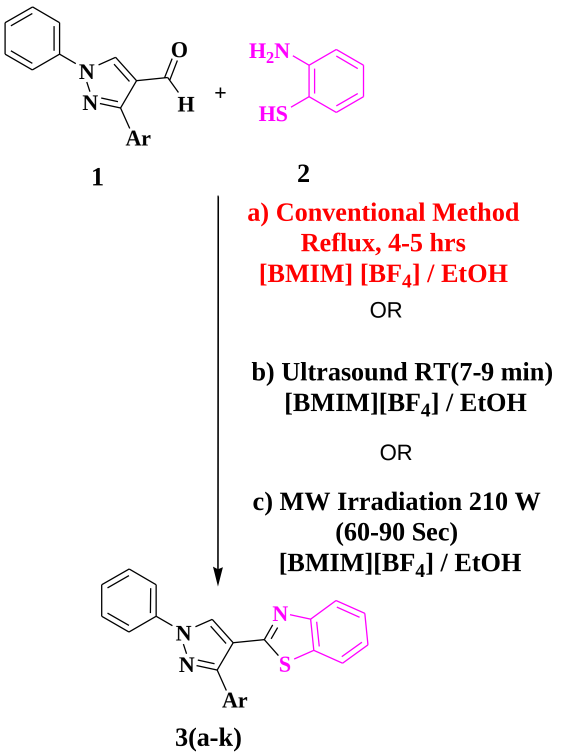
A mixture of 3-aryl-1-phenyl-1*H*-pyrazole-4-carboxaldehyde 1 (1 mmol), *o*-amino-thiophenol 2 (1 mmol) and a catalytic amount of [BMIM][BF_4] (10 mmol %) was taken in a round bottom flask containing 10 mL of ethanol. The reaction mixture was refluxed for completion of the reaction. The course of the reaction was monitored by thin layer chromatography. After completion of the reaction the mixture was poured into crushed ice. Solid product thus obtained was separated by filtration, dried well, and recrystallised by ethanol. The physical data of synthesized compounds are given in Table 2.

B) UNDER US IRRADIATION

A mixture of 3-aryl-1-phenyl-1*H*-pyrazole-4-carboxaldehyde 1 (1 mmol), *o*-aminothiophenol 2 (1 mmol) and catalytic amount of [BMIM][BF_4] (10 mmol %) was taken in a round bottom flask containing 10 mL of ethanol. The round bottom flask was placed in an US bath for 7-9 min at room temperature. The course of the reaction was monitored by thin layer chromatography. After completion of the reaction the mixture was poured into crushed ice. Solid product derivative thus obtained was separated by filtration, dried well, and recrystallised by ethanol. The same procedure was applied for the remaining substituents for confirming the consistency of the method. The physical data of synthesized compounds are given in Table 2.

C) UNDER MW IRRADIATION

A 10 mL round bottom flask was charged with 3-aryl-1-phenyl-1*H*-pyrazole-4-carboxaldehyde 1 (1 mmol), *o*-aminothiophenol 2 (1 mmol), catalytic amount of [BMIM][BF_4] (10 mmol %), and placed under MW irradiation at 210 W for 3-5min. The course of the reaction was monitored by thin layer chromatography. After completion of the reaction the mixture was poured into crushed ice, the solid product thus obtained was separated by filtration, dried well, and recrystallised by ethanol. The same procedure was applied for the remaining substituents for confirming the consistency of the method. The physical data of synthesized compounds are given in Table 2.



Scheme I : Synthesis of substituted 2-(1, 3-diphenyl-1*H*-pyrazol-3-yl)benzo[*d*]thiazole

III. RESULTS AND DISCUSSION

To achieve an optimum condition, o-aminothiophenol 2 (1 mmol) were treated with 1-phenyl-3-*p*-tolyl-1*H*-pyrazole-4-carbaldehyde 1a (1 mmol) in ethanol with catalyst [BMIM][BF_4] as a model reaction. It was observed that, the reaction did not proceed in the absence of [BMIM][BF_4] and the good results were obtained with 10 mmol % [BMIM][BF_4] under reflux condition; thus the catalyst is essential for the synthesis of benzthiazoles (Table 1, Entry 6).

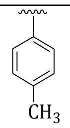
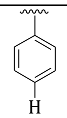
Table 1 : Optimization of reaction condition to synthesize under Reflux condition, USI and MWI

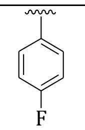
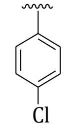
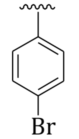
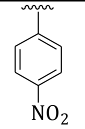
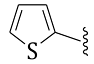
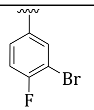
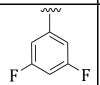
Entry	Catalyst / Solvent	Condition	Time	Yield
1	No Catalyst /EtOH	Stirring	5 h	NR
2	No Catalyst /EtOH	USI	2 h	NR
3	No Catalyst /SF	MWI	30 min	NR
4	10mmol% [BMIM][BF_4] /EtOH	Stirring at RT	2 h	NR
5	5mmol% [BMIM][BF_4] /EtOH	Reflux	6 h	35
6	10mmol% [BMIM][BF_4] /EtOH	Reflux	4 h	80
7	10mmol% [BMIM][BF_4] /EtOH	US	9 min	86
8	10mmol% [BMIM][BF_4] /SF	MWI at 140 W	20 min	Trace
9	10mmol% [BMIM][BF_4] /SF	MWI at 210 W	4 min	90

Reaction Condition- benzil 1 (1eq), substituted 1, 3-diaryl pyrazole aldehyde 2 (1eq), ammonium acetate 3 (2eq) and Molar ratio [BMIM] [BF_4]/substrate

When the same reaction was carried out under ultrasound irradiation at room temperature, the desired product was obtained with high yield in few minutes (Table 1, Entry 7). Also under microwave irradiation, the model reaction at 140 W did not work, but it proceeds with the best results at 210 W (Table 1, Entry 9).

B) Table 2 : Synthesis of 3(a-i) using [BMIM] [BF_4]

Entry	Ar – Group	Reaction Time in Min.			Yield in %			MP in (°C)
		Reflux	US	MW	Reflux	US	MW	
3a		240	9	4	80	86	90	144
3b		240	7	4	82	92	92	126

3c		240	9	5	85	86	84	134
3d		270	9	5	84	92	94	170
3e		270	9	5	80	84	86	190
3f		300	9	5	75	80	82	194
3g		240	9	3	84	90	92	92
3h		270	9	5	80	86	90	186
3i		270	9	5	82	90	92	160

Reaction Condition : 1, 3-diaryl pyrazole aldehyde 1 (1eq), *o*-amino thiophenol 2 (1eq) and 10 mmol% [BMIM] [BF_4]

a) Reflux in ethanol (10 mL)
b) Ultrasound Irradiation, in ethanol (10 mL)
c) Microwave Irradiation, Solvent free.

To evaluate the scope and limitations of this work, our attempts focused on the synthesis of the 2-substituted benzothiazoles using differently substituted heterocyclic aldehyde and o-aminothiophenol (Table 2). Aldehyde containing electron-donating as well as electron-withdrawing groups gave products in good yield by conventional as well as non-conventional methods.

C) Spectral Data of Synthesized Compounds

3a: 2-(1-phenyl-3-*p*-tolyl-1*H*-pyrazol-4-yl)benzo[*d*] thiazole: Yellow Solid; M.P. 144°C; FT-IR (KBr) ν: 2917, 1593, 1554, 1505, 1407, 1217, 1044, 749; ^{1}H NMR (DMSO-d_6, 400 MHz) δ: 2.38 (s, 3H, Ar-CH3), 7.295 (d, 2H, Ar-H), 7.37-7.42 (m, 2H, Ar-H), 7.48-7.57 (m, 3H, Ar-H), 7.63 (d, 2H, Ar-H), 7.96 (d, 1H, J=7.6 Hz, Ar-H), 8.04 (t, 3H, J=7.6 Hz, Ar-H), 9.25 (s, 1H, Pyrazole ring-H); ^{13}C NMR (DMSO- d_6, 100 MHz) δ: 20.93, 116.40, 118.82, 122.01, 122.18, 125.02, 126.35, 127.11, 128.88, 128.95, 129.11, 129.61, 129.90, 134.45, 138.44, 138.84, 150.97, 152.84, 159.56; MS: m/z= 368.32 [M+1]+.

3b:2-(1,3-diphenyl-1*H*-pyrazol-4-yl)benzo[*d*]thiazole: Faint Yellow Solid; M.P. 126°C; FT-IR (KBr) ν:1594, 1557, 1504, 1406, 1218, 1046, 959, 749; 1H NMR (DMSO-d_6, 400 MHz) δ: 7.33-7.45 (m, 2H, Ar-H), 7.47-7.54 (m, 4H, Ar-H), 7.52 (t, 2H, Ar-H), 7.76-7.85 (m, 2H, Ar-H), 7.98 (d, 1H, Ar-H), 8.05-8.09 (m, 3H, Ar-H), 8.42 (s, 1H, Pyrazole ring-H); MS: m/z=354.28 [M+1]+.

3c: 2-3-(4-fluorophenyl)-1-phenyl-1*H*-pyrazol-4-yl) benzo[*d*]thiazole: Faint Yellow Solid; M.P. 134°C; FT-IR (KBr) ν: 1595, 1556, 1472, 1312, 1218, 1158, 1046, 751; ^{1}H NMR (DMSO-d_6, 400 MHz) δ: 7.33-7.35 (m, 2H, Ar-H), 7.37-7.45 (m, 2H, Ar-H), 7.51-7.54 (m, 1H, Ar-H), 7.56-7.60 (m, 2H, Ar-H), 7.84-7.87 (m, 2H, Ar-H), 7.98 (d, 1H, Ar-H), 8.05 (dd, 2H, Ar-H), 8.09 (d, 1H, Ar-H), 9.31 (s, 1H, Pyrazole ring-H).

3d:2-(3-(4-chlorophenyl)-1-phenyl-1*H*-pyrazol-4-yl) benzo[*d*]thiazole: Yellow Solid; M.P. 170°C; FT-IR (KBr) ν: 1598, 1559, 1505, 1447, 1394, 1209, 1061, 930, 755; ^{1}H NMR (DMSO-d_6, 400 MHz) δ: 7.44 (t, 2H, Ar-H), 7.53 (t, 1H, Ar-H), 7.58 (m, 4H, Ar-H), 7.85 (d, J=6.4 Hz, 2H, Ar-H), 7.98 (d, J=6.4 Hz, 1H, Ar-H), 8.05 (d, J=6.4 Hz, 2H, Ar-H), 8.10 (d, J=6.4 Hz, 1H, Ar-H), 9.32 (s, 1H, Pyrazole ring-H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ: 116.87, 119.44, 122.58, 122.83, 125.68, 126.93, 127.81, 128.92, 130.15, 130.98, 131.22, 131.48, 13.24, 134.94, 139.25, 150.06, 153.43, 159.77; MS: m/z=388.26 [M+1]+

3e: 2-(3-(4-bromophenyl)-1-phenyl-1*H*-pyrazol-4-yl) benzo[*d*]thiazole: White Solid; M.P. 190°C; FT-IR (KBr) ν: 1597, 1546, 1502, 1478, 1218, 1033, 1010, 751; ^{1}H NMR (DMSO-d_6, 400 MHz) δ: 7.43 (m, 2H, Ar-H), 7.49 (m, J=7.6 Hz, 1H, Ar-H), 7.58 (t, 2H, Ar-H), 7.71 (m, 2H, Ar-H), 7.77 (m, 2H, Ar-H), 7.98 (d, 1H, Ar-H), 8.05 (d, J=6.0 Hz, 2H, Ar-H), 8.10 (d, J=6.0 Hz, 1H, Ar-H), 9.31 (s, 1H, Pyrazole ring-H).

3f: 2-(3-(4-nitrophenyl)-1-phenyl-1*H*-pyrazol-4-yl) benzo[*d*]thiazole: Brown Solid; M.P. 194˚C; FT-IR (KBr) ν: 1597, 1563, 1506, 1394, 1340, 1314, 1033, 933, 852, 753; ^{1}H NMR (DMSO-d$_6$, 400 MHz) δ: 7.10 (dd, 2H, Ar-H), 7.41-7.60 (m, 5H, Ar-H), 7.99-8.19 (m, 5H, Ar-H), 8.35 (d, J=5.6 Hz, 1H, Ar-H), 9.39 (s, 1H, Pyrazole ring-H).

3g: 2-(1-phenyl-3-(thiophen-2-yl)-1*H*-pyrazol-4-yl) benzo[*d*]thiazole: Yellow Solid; M.P. 92˚C; FT-IR (KBr) ν: 1595, 1561, 1501, 1473, 1310, 1224, 1033, 751; ^{1}H NMR (DMSO-d$_6$, 400 MHz) δ: 6.44 (t, 1H, Ar-H), 6.75 (dd, J=5.2 Hz, 1H, Ar-H), 7.02 (dd, 1H, Ar-H), 7.09 (t, 1H, Ar-H), 7.58 (m, 2H, Ar-H), 7.69 (dd, 1H, Ar-H), 8.02 (m, 2H, Ar-H), 8.08 (d, J=6.4 Hz, 1H, Ar-H), 8.14 (d, J=6.4 Hz, 1H, Ar-H), 8.19 (m, 1H, Ar-H), 9.31 (s, 1H, Pyrazole ring-H); ^{13}C NMR (DMSO-d$_6$, 100 MHz) δ: 116.27, 119.38, 122.94, 125.85, 126.98, 127.83, 128.19, 129.29, 13016, 131.61, 133.98, 134.91, 135.04, 139.04, 150.17, 153.54, 159.73; MS: m/z=360.26 [M+1]+.

3h: 2-(3-(3-bromo-4-fluorophenyl)-1-phenyl-1*H*-pyrazol-4-yl)benzo[*d*]thiazole: Yellow Solid; M.P. 186˚C; FT-IR (KBr) ν: 1598, 1558, 1506, 1447, 1405, 1234, 1048, 937, 747; ^{1}H NMR (DMSO-d$_6$, 400 MHz) δ: 6.47 (m, 1H, Ar-H), 6.78 (dd, J=5.2 Hz, 1H, Ar-H), 7.07 (m, 1H, Ar-H), 7.19 (m, 1H, Ar-H), 7.31 (t, 1H, Ar-H), 7.49 (m, 3H, Ar-H), 7.83 (d, 2H, Ar-H), 7.90 (m, 1H, Ar-H), 8.05 (s, 1H, Pyrazole ring-H), 8.20 (m, 1H, Ar-H).

3i: 2-(3-(3,5-difluorophenyl)-1-phenyl-1*H*-pyrazol-4-yl)benzo[*d*]thiazole: Faint Yellow Solid; M.P. 160˚C; FT-IR (KBr) ν: 1628, 1594, 1562, 1432, 1396, 1202, 1114, 1033, 983, 748; ^{1}H NMR (DMSO-d$_6$, 400 MHz) δ: 6.51 (m, 1H, Ar-H), 6.74 (dd, 1H, Ar-H), 7.10 (dd, 1H, Ar-H), 7.21 (m, 1H, Ar-H), 7.26-7.32 (m, 2H, Ar-H), 7.52 (t, 2H, Ar-H), 7.69 (d, 2H, Ar-H), 7.86 (d, 2H, Ar-H), 8.11 (s, 1H, Pyrazole ring-H).

IV. ABBREVIATIONS

MWI-Microwave Irradiation, USI- Ultrasound Irradiation, MCRs-Multicomponent Reactions, SF-Solvent Free, ILs-Ionic liquids, NR- No Reaction, RT-Room Temperature, [BMIM][BF$_4$] -1-butyl, 3-methyl, 1-imidazolium tetra floroborate.

V. CONCLUSION

In summary, we have described a novel simple, fast and environmentally benign protocol for the synthesis of 2-(3-aryl-1-phenyl-1*H*-pyrazol-4-yl) benzo [*d*] thiazole from 3-aryl-1-phenyl-1*H*-pyrazole-4-carbaldehyde and *o*-aminothiophenol in the presence of [BMIM][BF$_4$] under conventional reflux condition, US and MW irradiation. The present protocol avoids the use of less hazardous solvent, toxic catalysts, and long reaction times.

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Phytochemistry of Nerium Oleander L. Root

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ABSTRACT

The present study revealed that, In *Nerium Oleander L.* plant roots are present bioactive phytoconstituents. In Apocyanace family of plant majority contains alkaloids and terepenoids. Extraction of phytoconstituents done by hot extraction method using soxhlet apparatus. The methanol extract have 18.20 % extractive value after successive extraction in n-hexane, chloroform, ethyl acetate extractive value 4.01 %, 6.12% and 9.35% respectively. The Brown colored root contains other minor phytoconstituents are stereoid, saponins, polar glycosides, carbohydrates and phenolic compounds. The phytochemical tests are performed for to finding bioactive secondary metabolic compounds. Plant have been reported many pharmaceutical activities such as antibacterial, antifungal, cytotoxic, antitumor activity.

Keywords : *Nerium Oleander L.*, alkaloids, trerepenoid.

I. INTRODUCTION

Current day's herbal medicines are best remedies for various indigenous diseases. Traditional drugs are a big part of pharmaceutical science. India is a native place of ayurveda. According to WHO 80 -85% of the word population use traditional medicine for various types of disease. In ayurveda *Nerium Oleander L.* plant is mentioned for it's therapeutically uses. The height of plant is two to six meter, linear stem, firstly stem have a glaucous bloom after mature stem have a grayish black. The green colored leaves have in pairs, thick and narrow lanceolae, 5 to 2o cm length, 1 to 3.4 broad. The inflorescence grows in cluster at the end of every branch with white, pink and red colored with 2 to 5 cm diameter. The fruit is a long narrow pair of follicles 5 to 23 cm long, which splits open at maturity to spread numbers of downy seeds.

Nerium oleander L. is native to a from Mauritania, Morocco and Portugal. It occurs in river valley, tolerate long seasons of droughts and inundation from winter rains. It is cultivated in many tropical and subtropical region. In India plants are planted in midterm region of national highway for pollution control. *Oleander* roots are poisonous. It contains toxins hence it is can not feed to caterpillars but in low dose it is non toxic. In Mediterranean climate oleander can be florescence from April to October.

The reported phytochemicals are below:

Table 1. Reported phytochemicals in *Nerium Oleander L.* Plant[1]

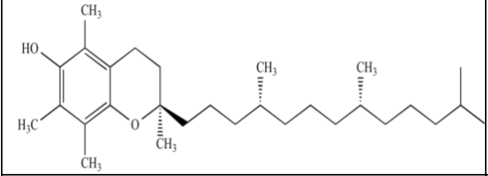
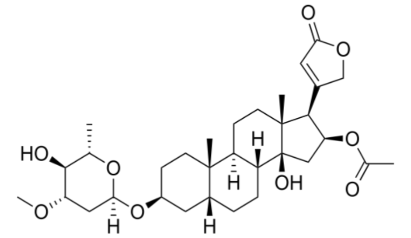
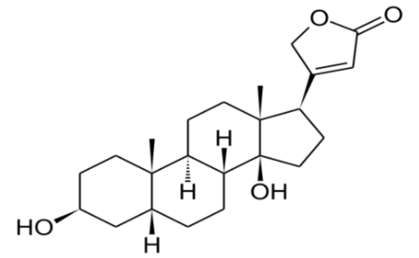
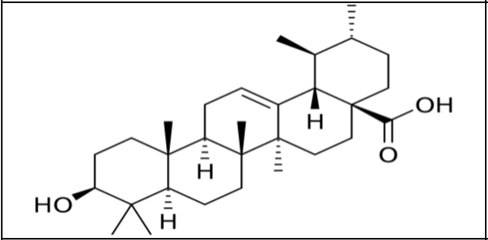
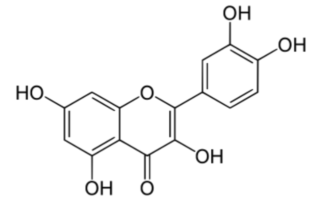
	**α-tocopherol**
	**Oleandrin**
	**Digitoxigenin**
	**Ursolic Acid**
	**Quercetin**

Figure 1. *Nerium Oleander L.* Plant

II. METHODS AND MATERIAL

1.1: Collection of Plant material:

The Nerium oleander L. roots were collected from western region of sahydri ghat, Maharashtra. The Coordinates of sahydri ghat is 10°10'N 77°04'E.

1.2: Preparation of herbarium and its authentication:

The plant leaf, inflorescence was authenticated by **Dr. M.S. Khyade of department of botany, S. N. Arts, D.J.M.Commerce and B.N.S.Science college, Sangamner. Taxonomic nomenclature done of plant is Nerium Oleander L.**

1.3: Drying and grinding of material:

Nerium Oleander L. roots were washing with tap water and then distilled water. The material was chopped into small cuts then grinding to small size powder and store in cool condition for further use.

1.4: Extraction:

Powder material was extracted by two methods.

1.4.1: Maceration:

Weigh 10 gm sample using calibrating balance with least count 0.001mg. The root powder was soaked in ethanol for 8 days with occasional shaking. After 8 days mixture was filtered by whatmann no.41 filter paper. Naturally solvent evaporated and extract stored in deep freezer for further study.

1.4.2: Hot extraction:

Weigh 100 gm plant root powder fill in the thimble of soxhlet. Soxhlet was run in ethanol solvent for 72 hrs. Thereafter root suspension was filtered and solvent evaporated using rotavapour with chiller. Extract obtained stored in cooler for further successive fractionation and preliminary phytochemicals study.

1.4.3: Successive extraction:

The extract was fractionating with non polar solvent to polar solvent. The n-hexane, chloroform, ethyl acetate extractive value calculated as NOH, NOC, NOE 1.25%, 1.83% and 2.66% respectively (w/w %).

1.5: Preliminary Phytochemicals screening test[6]

1.5.1: Test for alkaloids

a) Wagner's test: Take 4mg of extract add HCl and few drops of Wagner's reagent. Observed yellow colour.

b) Mayer's test: Take 6mg of extract add the Mayer's reagent. Observed yellow ppt.

1.5.2: Test for Carbohydrates

a) Fehling's test: Take 3mg of extract was shaken with 10ml of water, filtered and the filtrate was concentrated. Add 2 ml mixture of equal parts of Fehling's solution A and B were added and boiled for few minutes. Formation of red or brick red colored precipitate indicates the presence of reducing sugar.

b) Molisch's test: Take 2mg of extract was shaken with 5ml of water, filtered and the filtrate was concentrated. To this 4 drops of freshly prepared 15% alcoholic solution of α- naphthol was added. 4 ml of conc. H_2SO_4 was added so as to form a layer below the mixture. Red violet ring appear, indicating the presence of carbohydrates which disappear on the addition of excess of alkali.

1.5.3: Test for Flavonoids

Shinoda's test: Take 5 mg of extract was dissolved in 4ml of ethanol and to this 8drops of dilute Hcl followed by a small piece of mg metal were added. Formation of pink, reddish or brown colour indicates the presence of Flavonoids.

1.5.4: Test for Phenols

Nitric acid test: Take 2 mg extract, add conc. HNO_3 shake well, red colour indicates phenol present.

1.5.5: Test for Triterpenoids

Liebermann - Burchard's test: 5mg of dry extract was dissolved in acetic anhydride, heated to boiling, cooled and then 2 ml of concentrated H_2SO_4 was added along the sides of the test tube. Formation of a pink colour indicates the presence of trerepenoid.

1.5.6: Test for stereoids

Salkowski reaction: 4 mg of dry extract was shaken with chloroform, to the chloroform layer sulphuric acid was added slowly by the sides of test tube. Formation of red colour indicated the presence of steroids.

III. RESULTS AND DISCUSSION

The detailed report of the phytoconstituents tests carried out of root parts of Nerium Oleander L. are presented in Table 1. In this study the phytochemicals test reveals that the presence of the triterpenoids and steroids in n-hexane fraction, flavonoids in ethyl acetate extract. The alkaloids, carbohydrates, glycosides, are present in the ethanol extract.

Table 1. Phytochemical screening of root parts of *Nerium Oleander L.*

Phytoconstituents	*NOH*	*NOC*	*NOE$_A$*	*NOE*
Alkaloids	-	-	++	+
	-	-	++	+
Carbohydrates	-	-	-	+
	-	-	-	+
Flavonoids	-	-	+	-
Triterpenoids	+	-	-	-
stereoids	+	-	-	-

NOH- hexane fraction, NOC- chloroform fraction, NOEA - ethyl acetate fraction, NOE- ethanol extract. (+ = present - = absent)

IV. CONCLUSION

The present study revealed that plant contains bioactive phytoconstituents as a class of alkaloids, triterepenoids, carbohydrates, phenolic compounds and stereoids. Due to this secondary metabolic compound plant have therapeutic and pharmaceuticals properties.

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Efficacy of Some Medicinal Plants on MDR Staphylococcus saprophyticus Isolated from Urine sample

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ABSTRACT

Staphylococcus saprophyticus is Gram positive spherical shaped bacteria. It is opportunistic pathogen. It is a normal flora of skin and periurethral area. It can cause UTI particularly in sexually active young women.UTI is second most common type of infection in community practice. UTI refers to the presence of microorganism in the urinary tract including urinary bladder, prostate, collecting system or kidney. The syndrome ranges from asymptomatic bacteriuria to perinephrid abscess with sepsis. Due to indiscriminate use of antibiotics there is an increase in resistance among microorganisms. Bacteria become resistant to antibiotic by different ways. Patent suffering from antibiotic resistant strain fail to respond antibiotic treatment. So there is a continuous demand of new drug in the world. This problem of drug resistance among microorganism could overcome by drugs of plant origin. Due to this reason now a day the demand of herbal products as therapeutic agents is increasing all over the world. In the present study antibacterial activity of *Aloe vera, Azadirachta indica* & *Citrus limon* was studied against MDR *Staphylococcus saprophyticus* isolated from urine sample collected from civil hospital. Antibiotic susceptibility test of the clinical isolates of *Staphylococcus saprophyticus* was done by using modified Kirby-Bauer disc diffusion method in accordance with the guidelines of the clinical & laboratory standards institute. Interpretation of resistance was based on the NCCLS criteria. The most common pattern of multiple drug resistance of isolates of *Staphylococcus saprophyticus* observed was erythromycin-nalidixic acid-penicillin-sulfamethoxazole-chloramphenicol. The antibacterial activity of solvent extract of plant was tested by agar well diffusion method. *Citrus limon* and *Azadirachta indica* showed moderate antibacterial activity while *Aloe vera* showed least antibacterial activity.

Keywords : Antibacterial activity, Disc diffusion method, MDR, Medicinal plant extracts, *Staphylococcus saprophyticus.*

I. INTRODUCTION

Staphylococcus saprophyticus is Gram positive, facultative anaerobic spherical shaped bacteria arranged in grape like clusters belongs to family Micrococcaceae. It is opportunistic pathogen. Young women are very susceptible to colonize this organism in the urinary tract and it is spread through sexual intercourse [2, 15, and 21]. It is a normal flora of skin and periurethral area. It can cause UTI particularly in sexually active young women. UTI is second most common infection in community practice. UTI refers to the presence of microorganism in the urinary tract including urinary bladder, prostate, collecting system or kidney. The syndrome ranges from asymptomatic bacteriuria to perinephrid abscess with sepsis [13]. All over the world *Escherichia coli* account for 75 to 90% of UTI isolates and *Staphylococcus* *saprophyticus* accounts for 5 to 15 % of cases of uncomplicated cystitis [23]. Several studies showed that rectal, vaginal and urethral colonization of *Staphylococcus saprophyticus* was associated with UTI caused by this bacterium [16]. Indiscriminate use of antibiotics has led to an increase in antibiotic resistance in the microorganisms [3, 17]. Bacteria become resistant to antibiotic by different ways. R-plasmid often contains genes for resistance to different antibiotics [7]. Plasmid can be transferred between closely related bacterial populations [17].

The application of plants is as old as 4000 to 5000 B.C. In India earliest references of curative properties of medicinal plants appear in Rig-Veda which is said to be written between 3500 to 1600 B.C. The rural population in different parts of the world is more disposed to traditional treatment [12, 14]. It is estimated that about 80 % of the rural population in developing Asian nation depend on home care and traditional medicine for major therapies [11]. Plants produce different types of bioactive compound, making them rich sources of different types of medicine [19]. These compounds are secondary metabolites such as alkaloids, flavonoids , resins , steroids, , fatty acids, tannins ,phenol compounds etc .The problem of drug resistance could overcome by drugs of plant origin. Due to this reason now a day the demand of herbal products as therapeutic agents is increasing throughout the world. Keeping this view in mind in the present study an attempt was made to isolate multi drug resistant *Staphylococcus saprophyticus* from human urine sample & to study the effect of medicinal plant extract on it.

II. METHODS AND MATERIAL

2.1. Isolation of MDR *Staphylococcus saprophyticus* from urine sample

Staphylococcus saprophyticus was isolated from urine sample collected from civil hospital in Latur. Midstream urine sample were collected in sterile small bottles. Urine samples were streaked specific media. Mannitol salt agar (MSA), *Staphylococcus* isolation agar & Blood agar were used for isolation. Isolates of *Staphylococcus saprophyticus* were identified by using different morphological, cultural characters & biochemical tests [2, 6 and 8]. Antibiotic susceptibility testing was carried out by Kirby-Bauer's disk diffusion method [4] for drug susceptibility according to National Committee for Clinical Laboratory standards [20]. The Muller Hinton agar plates were spread with isolates of *Staphylococcus saprophyticus.* Then antibiotic discs were kept by using sterile forceps & then gently pressed down onto the agar. Plates were kept at low temperature in the freeze for 30 minutes for proper diffusion and then incubated at 37 ºC for 16- 48 hrs. Antibiotics used in this study were Amoxicillin (20mcg), Chloramphenicol (30mcg), Ciprofloxacin (5mcg), Erythromycin (5mcg), Gentamycin (10mcg), Nalidixic acid (30mcg), Penicillin (10 Units), Rifampin (5mcg), Sulfamethoxazole (50mcg), Vancomycin (30mcg) supplied by Hi-Media Laboratories, Mumbai.

2.2. Preparation of Herbal extract

Fresh leaves of *Azadirachta indica*, *Aloe vera* and peels of *Citrus limon* were collected and cleaned to remove soil and dirt. Plant materials were completely dried, powdered and mixed with methanol. Methanol extract of above plant material (5% w/v) were prepared separately. The obtained liquid extract was subjected to rotary evaporator and subsequently concentrated and stored in refrigerator at 4 ºC & tested by using agar well diffusion method [18].

2.3. Antibacterial testing of herbal extract

A 0.2ml of suspension of isolates of *Staphylococcus saprophyticus* was thoroughly mixed with sterile molten nutrient agar and poured into sterile Petri plates under aseptic conditions. After solidification, plates were used for making of well by using flamed, cooled cork borer. 0.5 ml of single plant extract was added in each well. Plates were kept at low temperature for diffusion then incubated at 37 ºC for 1 to 2 days. After incubation, zones of inhibition were measured & noted [8].

III. RESULTS AND DISCUSSION

Isolates of *Staphylococcus saprophyticus* were isolated from urine sample. 10 isolate *Staphylococcus saprophyticus* showed antibiotic resistance to one or more antibiotics. Isolates of *Staphylococcus saprophyticus* were Gram-positive, non motile, non spore forming cocci, arranged in grape-like clusters. It forms white, raised colonies about 1-2 mm in diameter. It is coagulase -ve, DNAse -ve ,golden pigmentation -ve, hemolysis on blood agar -ve, growth on mannitol salt agar +ve, catalase +ve, urease +ve, DNase –ve, nitrate reduction test +ve, MR +ve, VP +ve, Indole –ve, novobiocin resistance +ve.

Table 1 : Percent resistance of *Staphylococcus saprophyticus* isolates against antibiotic.

Sr No	Antibiotics	No of isolate showing resistance	Percent resistance
1.	Amoxicillin (20mcg)	00	00
2.	Chloramphenicol (30mcg)	01	10
3.	Ciprofloxacin (5mcg)	00	00
4.	Erythromycin (5mcg)	06	60
5.	Gentamycin (10mcg)	00	00
6.	Nalidixic acid (30mcg)	04	40
7.	Penicillin (10Units)	03	30
8.	Rifampin (5mcg)	00	00
9.	Sulfamethoxazole(50mcg)	03	30
10	Vancomycin (30mcg)	00	00

The most common pattern of multiple drug resistance patterns of isolates of *Staphylococcus saprophyticus* observed was erythromycin-nalidixic acid-penicillin-sulfamethoxazole-chloramphenicol.

The MAR index of each isolate was calculated by using Eq. (1). The MAR index of each isolate was calculated by using following formula:

$$\text{MAR Index} = \frac{\text{No. of Antibiotics to which the isolate was resistant}}{\text{Total no. of antibiotics tested}} \quad (1)$$

The antibacterial activity of plant extract on isolates of *Staphylococcus saprophyticus* was studied, zone of diameter were measured & noted in the Table 2.

Table 2 : Antibacterial activity of herbal extract on isolates of *Staphylococcus saprophyticus*

Isolate of *Staphylococcus saprophyticus*	**Zone Diameter in mm**		
	Aloe vera	***Azadirachta indica***	***Citrus limon***
ISS01	---	---	++
ISS02	---	++	----
ISS03	+	+	++
ISS04	---	++	---
ISS05	+	---	++
ISS06	---	++	++
ISS07	--	---	+
ISS08	---	+	---
ISS09	--	--	--
ISS10	+	--	+

(--: no antibacterial activity, +: antibacterial activity)

Citrus limon and Azadirachta indica showed moderate antibacterial activity while *Aloe vera* showed least antibacterial activity. *Aloe vera* has long been used in Ayurveda for its anti-burn effect and also as a tonic, alternative, antiseptic, antibiotic, anti-diarrheal, anti-fungal, anti-viral and good hair conditioner [10]. *Aloe vera* gel that contains 97-99% water and 1-2% active compounds like aloesin, aloin, aloe-emodin, aloe-mannan, flavonoids, saponin, sterols, amino acids and vitamins. Free anthroquinones and their derivatives like barbaloin-IO-aloe emodin-9 anthrone, isobarbaloin and chromones in *Aloe vera* leaves exert a strong purgative effect and are potent anti-microbial agents [22]. Antimicrobial properties of *Azadirachta indica* can be attributed to several bioactive compounds found in it, which are categorized into two major classes, isoprenoids and non- isoprenoids . Azadirachtin is mainly responsible for antibacterial activity of neem [5]. More than 130 compounds have been isolated from different parts of neem, which have tremendous biological activity as antiarthritic, antipyretic, hypoglycemic, spermicidal, antifungal, antibacterial, diuretic, antimalarial, antitumour, antiinflammatory, immunomodulatory etc [9]. *Citrus limon* fruits have a broad spectrum of biological activity including antibacterial, antifungal, antidiabetic, anticancer and antiviral activities due to alkaloids [1].The indiscriminate use of antibiotics has led to an increase in antibiotic resistance among microorganisms. The problem of drug resistance could overcome by drugs of plant origin. Hence plants can be used for treatment of serious infections caused by multi drug resistant bacteria.

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Influence of Slow Release Nitrogen Fertilizer on Soil Enzymes, Yield and Nutrient Uptake of Maize Grown on Inceptisol

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ABSTRACT

The field experiment entitled, “Influence of slow release nitrogenous fertilizers on soil enzymes, yield and nutrient uptake of maize on Inceptisol” was conducted during 2015-16 and 2016-17 in randomized block design. The treatment consist of control, GRDF, 100% recommended dose of nitrogen through Crotonylidene diurea (CDU), neem coated urea (NCU) and sulphur coated urea (SCU), 50% recommended dose of nitrogen through urea plus 50% RDN through NCU, CDU and SCU. These treatments are replicated thrice. The urease enzyme activity in both the year and pooled mean at 30 DAS were increased and decreased at 90 DAS. The treatment of RDN-CDU, NCU and SCU and their combinations 50% +50% RDN were recorded the less values of urease enzyme activity. The application of nitrogen to maize crop through neem coated urea was recorded significantly higher soil available nitrogen at 30 days after sowing. The nitrogen application to maize crop through NCU, SCU and CDU were equally beneficial for higher grain and straw yield. The total uptake of nitrogen, phosphorus and potassium by maize was significantly higher in recommended dose of nitrogen through neem coated urea. The soil pH and electrical conductivity (EC) was significantly influenced by slow release nitrogen fertilizer. The soil organic carbon and calcium carbonate content did not influenced by the slow release nitrogenous fertilizers. The use of neem coated urea (NCU), Crotonylidene diurea (CDU) and sulphur coated (SCU) alone or in combination with urea performed better than urea alone, by increasing nitrogen use efficiency through controlled urea hydrolysis. Treating urea with neem and sulphur has the potential to increase nitrogen use efficiency, grain and straw yield of maize grown on Inceptisol.

Keywords : Yield, NCU, CDU, SCU, Enzymes, Nutrient uptake)

I. INTRODUCTION

The role of nitro in increasing the crop production has been now well established. Nitrogen in soils is present mainly in three form *viz.*, organic ($R\text{-}NH_2$), ammonical (NH_4-N) and nitrate (NO_3^-). Urea is one of the most popular nitrogenous fertilizer used in India. Nitrogen use efficiency (NUE) is usually less for annually cultivated crops and cereals. The large amount of nitrogen is lost through denitrification, runoff, volatilization, immobilization and leaching. There are many management practices that can increase fertilizer use efficiency among which the use of enhanced efficiency nitrogenous fertilizers (EENF) is one. Different types of coated urea fertilizers are coming into the market apart from commonly used prilled urea. The difference in release of N from these fertilizers are of major concern to improve the NUE. The release pattern of nitrogen from coated urea fertilizers help in understanding the nitrogen availability over a period of time. The NUE can be increased if the release of nitrogen from these coated urea fertilizers coincide with nitrogen demand of the crop. The advantages of coated urea fertilizers are minimization of leaching and volatilization losses of nitrogen, delay in nitrification and prolonged N supply. Keeping in view the studies pertaining to effect of slow release nitrogenous fertilizers on yield, nutrient uptake of maize grown on Inceptisol are studied.

II. METHODS AND MATERIAL

Field experiments on maize was conducted on same site during 2015-16 and 2016-17 at Research Farm of Mahatma Phule Krishi Vidyapeeth, Rahuri. The soils of experimental was clay texture with low available nitrogen (158.3 kg ha^{-1}), medium in available phosphorus (15.0 kg ha^{-1}) and high in available potassium (430 kg ha^{-1}). The soil reaction was alkaline in reaction (pH 8.0). The experiments consist of Absolute control, GRDF (120:60:40 kg N:P_2O_5:K_2O ha^{-1} + 10 t ha^{-1} FYM), RDN-crotonylidene diurea (CDU), RDN-Neem coated urea (NCU), RDN-Sulphur coated urea (SCU), 50% NDN-urea + 50% N NCU, 50% RDN-urea + 50% N-CDU, 50% RDN-urea + 50% N SCU, 50% RDN-NCU + 50% N-CDU, 50% RDN-NCU + 50% N-SCU and 50% N-CDU + 50% N-SCU. These treatments were replicated thrice and statistical method used was randomized block design.

III. RESULTS AND DISCUSSION

The nitrogen requirements of all the field crops are comparatively higher than other nutrients. However, the use efficiency of applied nitrogen through fertilizer are less because of hydrolysis of nitrogen fertilizer and their transformation by means of soil chemical reactions in soil or by microbial activity. This might be mismatched with nitrogen requirement of crop as per their crop growth stage and influenced the yield of field crops.

Yield

The grain yield of maize was significantly influenced by slow release nitrogenous fertilizers during 2015-16, 2016-17 and pooled mean (Table 1). The application of recommended dose of nitrogenous to maize through neem coated urea was recorded significantly higher grain yield (72.44 q ha^{-1}) and statistically on par with recommended dose of nitrogen through sulphur coated urea (71.84 q ha^{-1}) and crotonylidene diurea (69.01 q ha^{-1}) respectively during 2015-16. Similar trend was observed during 2016-17 and pooled mean. The higher grain yield of maize in neem, sulphur and crotonylidene diurea might be associated with the slow release of nitrogen in soil, it was absorbed by the maize crop through out its critical grow stages (Blaylock *et al.*, 2005). The stover yield of maize was showed the similar trend to that of grain yield.

Nutrient uptake

The application of nitrogen to maize crop as RDN-NCU recorded the significantly higher nitrogen uptake during 2015-16, 2016-17 and pooled mean (122.75, 131.45 and 127.10 kg ha^{-1} respectively) followed by RDN-CDU (113.10, 102.90 and 108.0 kg ha^{-1} respectively). The higher uptake of nitrogen by maize might be because of coating reduce the contact with soil particles, restrict the activity of micro organisms and also urease enzyme activity phosphorus uptake of maize was found similar to that of nitrogen uptake. The higher phosphorus uptake might be because of slow release nitrogen enhanced the vegetative growth and biomass, which increased biomass promote phosphorus uptake. The recommended dose of nitrogen through NCU recorded significantly higher potassium uptake during both the year of experimentation and pooled mean (88.80, 87.91 and 87.99 kg ha^{-1} respectively) and on par with RDN-SCU (83.80, 83.38 and 83.59 kg ha^{-1} respectively). The potassium uptake was increased due to increased nitrogen uptake and has synergistic effect between nitrogen and potassium.

Soil enzyme

Urease enzyme is unique among soil enzymes and greatly influenced the fate and performance of important fertilizer like urea. The urease enzyme activity during both the year and pooled mean at 30 DAS were 37.98, 30.34 and 34.16 µg NH_4-N g^{-1} soil h^{-1} and decreased at 90 DAS (Table 3). The treatment of RDN-CDU, NCU and SCU and their combinations as 50% + 50% RDN were recorded the less values of urease enzyme activity in soil during both the year. This might be because of coating of urea fertilizer. The coating material inhibiting the activity of urease enzyme by avoiding the direct contact with urea (Xiaoguang *et al.*, 2004).

Dehydrogenazes are generally present in upper layer of soils, plays the major role in the energy production of organisms. The soil dehydrogenase enzyme activity was found statistically on par with each other in treatment 50% RDN-urea + 50% NCU, 50% RDN-urea + 50% CDU and 50% RDN-urea + 50% SCU at 90 DAS during 2015-16 (1.46, 1.42 and 1.43 µg TPF g^{-1} soil hr^{-1} respectively), 2016-17 (1.51, 1.49 and 1.49 µg TPF g^{-1} soil hr^{-1} respectively) and pooled mean (1.48, 1.45 and 1.46 µg TPF g^{-1} soil h^{-1} respectively) (Quireshi *et al.*, 1995).

Soil available nutrients

The soil available nitrogen was increased at 30 DAS with the application of nitrogen through neem coated urea was found significant to recorded the higher content of soil available nitrogen at 30 DAS during 2015-16, 2016-17 and pooled mean (232.06, 223.70 and 227.88 kg ha^{-1} respectively) and decreased at 90 DAS (196.33, 203.84 and 200.09 kg ha^{-1} respectively). Similar treatment showed soil available phosphorus content higher at 30 DAS (27.08, 34.42 and 30.75 kg ha^{-1} respectively.

IV. CONCLUSION

The application of nitrogen through neem coated, sulphur coated and crotonylidene diurea to maize are equally beneficial for higher grain and stover yield. The total uptake of nitrogen, phosphorus and potassium by maize was significantly influenced by RDN-SCU. The treatments of RDN-CDU, NCU and SCU and their combinations as 50% N-CDU/ NCU/ SCU and 50% RDN were reduce the urease enzyme activity.

Table 1. Effect of slow release nitrogenous fertilizer on yield and total nutrient uptake by kharif maize grown on Inceptisol

(Pooled mean)

Sr. No	Treatment	Yield (q ha^{-1})		Nutrient uptake (kg ha^{-1})		
		Grain	Stover	N	P	K
1.	Control	37.89	83.33	51.40	28.90	48.72
2.	GRDF	70.50	105.0	101.77	48.08	70.80
3.	RDN-CDU	68.83	106.0	108.0	52.57	78.77
4.	RDN-NCU	73.02	111.5	127.10	57.92	87.99
5.	RDN-SCU	72.59	107.40	120.49	56.52	83.59
6.	50% RDN-Urea + 50% NCU	65.65	104.1	117.78	50.46	77.15
7.	50% RDN-Urea + 50% CDU	62.70	100.0	105.54	45.28	69.81
8.	50% RDN-Urea + 50% SCU	64.81	100.9	107.69	46.98	70.72
9.	50% RDN-NCU + 50% CDU	58.82	95.37	96.42	39.63	62.35
10.	50% RDN-NCU + 50% SCU	61.75	100.0	99.84	44.69	67.08
11.	50% RDN + 50% SCU	51.10	87.50	82.83	36.83	56.05
	SE ±	1.98	4.03	3.49	1.35	2.55
	CD 5%	5.67	11.52	9.98	3.86	7.28

Table 2. Effect of slow release nitrogenous fertilizer on soil urease and dehydrogenase enzyme activity at different growth stages of maize grown on Inceptisol

(Pooled mean)

Sr. No.	**Treatment**	**Urease (µg NH_4-N g^{-1} soil h^{-1})**			**Dehydrogenase (µg TPF g^{-1} soil h^{-1})**		
		30 DAS	**90 DAS**	**At harvest**	**30 DAS**	**90 DAS**	**At harvest**
1.	Control	18.21	16.48	6.61	0.95	1.27	1.18
2.	GRDF	34.16	34.02	13.21	1.20	1.51	1.44
3.	RDN-CDU	21.43	17.72	7.10	0.98	1.32	1.21
4.	RDN-NCU	26.76	24.51	9.28	1.10	1.43	1.35
5.	RDN-SCU	25.25	20.72	8.31	1.06	1.41	1.29
6.	50% RDN-Urea + 50% NCU	32.11	31.29	11.30	1.18	1.48	1.40
7.	50% RDN-Urea + 50% CDU	28.77	28.40	9.99	1.12	1.45	1.36
8.	50% RDN-Urea + 50% SCU	30.19	28.61	10.47	1.14	1.46	1.39
9.	50% RDN-NCU + 50% CDU	24.68	18.79	8.23	1.03	1.37	1.26
10.	50% RDN-NCU + 50% SCU	22.18	21.17	8.45	1.08	1.42	1.32
11.	50% RDN + 50% SCU	26.05	17.91	7.93	1.00	1.32	1.24
	SE ±	0.60	0.71	0.31	0.01	0.02	0.01
	CD 5%	1.71	2.02	0.89	0.04	0.05	0.04

Table 3. Effect of soil available nitrogen, phosphorus and potassium in maize grown on Inceptisol

(Pooled mean)

Sr. No.	**Treatment**	**Soil av. Nitrogen (kg ha^{-1})**		**Soil av. Phosphorus (kg ha^{-1})**		**Soil av. Potassium (kg ha^{-1})**	
		30 DAS	**At harvest**	**30 DAS**	**At harvest**	**30 DAS**	**At harvest**
1.	Control	162.03	150.28	15.93	13.71	383	334
2.	GRDF	179.94	157.60	16.73	14.65	427	360
3.	RDN-CDU	216.38	177.38	27.09	20.25	519	459
4.	RDN-NCU	227.88	204.69	30.75	21.94	549	498
5.	RDN-SCU	222.66	184.94	28.41	20.27	532	468
6.	50% RDN-Urea + 50% NCU	202.27	176.35	25.28	18.80	472	440
7.	50% RDN-Urea + 50% CDU	189.73	171.62	22.98	17.21	513	416
8.	50% RDN-Urea + 50% SCU	194.43	174.16	23.44	17.91	595	435
9.	50% RDN-NCU + 50% CDU	180.32	163.86	18.38	15.80	461	396
10.	50% RDN-NCU + 50% SCU	185.02	167.57	21.01	16.60	472	413
11.	50% RDN + 50% SCU	176.14	162.17	18.67	15.48	461	388
	SE ±	3.24	4.39	6.89	0.40	19.68	7.56
	CD 5%	9.25	12.55	2.53	1.15	56.25	21.59

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Effect of Chlorpyriphos 20% EC on Respiratory Physiology of Mosquito Fish, Gambusia Affinis

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ABSTRACT

The enormous use of pesticides in the agricultural system, leads to deposition of large amount of toxicant which produces adverse effects on human as well as fresh water organisms. The respiration is an important physiological activity in all living organism These pesticides decreases the level of dissolved oxygen in the water bodies and leads to changes in the respiratory physiology of the aquatic organisms. The pesticides used in the fields get collected in the water bodies enter into the body of aquatic organisms like fish and may cause adverse effects on it. The oxygen consumption tendency is decreased when the time of exposure to toxicant is increased. The reduced O_2 consumption could be due to gill damage. The total O_2 consumption is one of the indicators of the general wellbeing of the fish. The study reveals the effect of pesticide Chlorpyriphos 20 % EC on the respiratory physiology of mosquito fish *Gambusia affinis*. The O_2 consumption level of fish *Gambusia affinis* was observed in the different concentration of (0.02, 0.04, 0.06, 0.08 ppm) at different time of exposure periods (24, 48, 72 & 96 hrs). The oxygen consumption exhibited decreasing trend in treated group up to 96hrs as compared with control group. The present study has clearly showed that commonly used pesticide in agriculture, chlorpyriphos 20% EC affects the oxygen consumption of mosquito fish, *Gambusia affinis* under all exposed concentration. The oxygen consumption level of the exposed fish at different times of exposure (24, 48, 72 and 96 hrs.) was found to be declined gradually till the end of exposure period (96hrs), the gradual decline was attributed to the interference of pesticide with oxidative metabolism. This pesticide caused respiratory distress, decreased level of oxygen consumption in fish *Gambusia affinis*, which suggested that Chlorpyriphos 20% EC is highly toxic to fish. Possibility of water pollution by this pesticide can be monitored through adapting proper assessment mechanism.

Keywords : Pesticides, Respiratory Physiology, Chlorpyriphos, Gambusia Affinis, Oxygen Consumption.

I. INTRODUCTION

Abundant use of fertilizers and pesticide became essential for better agricultural practices in most of the developing countries including India. Environmental pollution caused by pesticides, especially in aquatic ecosystem, has become a serious problem. These pesticides even when applied in restricted areas are washed and carried away by rains and floods to large water bodies like ponds and rivers and there by alter the physiochemical properties of water (Neelam Sharma *et.al.*, 2016), this proved to be highly toxic, not only to fishes but also to aquatic life forms and their environment (Sudha Summarvar et, al, 2016). Aquatic animals have to pass large quantities of water over their respiratory surfaces and are subjected to relatively greater risk of exposure to toxic substances. Oxygen consumption has been reported as a valuable index of the overall physiological activity of animals and is generally used as an indicator under stress conditions either due to pathological state of the animal or adverse ambient environmental conditions Fish are largely being used for assessment of the quality of aquatic environment and as such can serve as bio indicators of environment pollution. Pesticides became one of the leading polluting agents of aquatic ecosystem. The organic pollutants decrease the level of dissolve oxygen in the water bodies. It leads to many changes in organisms physiology (Rajkumar *et.al.*, 1998). Excessive use of such chemicals resulting in environmental pollution and toxicity risks to non-target organism. Toxicants in the environment mainly enter into fish by means of their respiratory distress (Tovell, 1975). These pesticides have various physiological effects such as enzyme inhibition, inhibitory effects on growth, metabolism and general development of animal (Bare, 2014). Amongst the pollutants found in agriculture waste, insecticides are most hazardous since they have an ability to immobilize or kill the aquatic organisms at extremely low concentration (Cope, 1965 & Eisler, 1969). Insecticides cause serious toxicology problem mainly due to their persistence and high toxicity. Pesticides affects the ecosystem, reproduction and behavior by causing, pathological & physiological changes (Holden, 1973). Pesticides are not highly selective but are generally toxic to many non-target organisms such as fish (Mohammed A. AI Kahtani 2011). The poisoning by pesticides from agricultural fields is a serious water pollution problem and its environmental long term effect may results in the incidence of poisoning of fish and other aquatic life (Jyothi and Narayan, 1996).

Respiration in fish

Respiration is an important physiological activity in all living organism. The oxygen is necessary to provide energy to carry out metabolic activities .Respiratory activity of a fish is often the first physiological response to be affected by the presence of toxic pollutants including pesticide in the aquatic media. Any change in the aquatic medium affects respiratory potential of the fishes. Some pollutants could be depressants and some stimulants. It appears logical that internal poisoning could damage the respiration rate, (Sprague, 1971. A change in respiratory rate is one of the common physiological response to toxicant including pesticide and easily detectable through change in metabolic under environmental deterioration (Magor, Shaikh, 2012). The respiratory rate of an animal are the important physiological parameters to assess the toxicity stress (Prosser, 1977). The metabolic rate in relation to respiration of fish could be increased under chemical stress (Chebbi and David, 2010). Changes in oxygen consumption, is indices of energy expenditure, are a useful tool to assess the physiological stress on aquatic organism (Lee,1969). Total oxygen consumption of fish reflects it's basal metabolic status and is one of the indicators of the general health and wellbeing of the fish. The differential oxygen consumption can be used as bio indicator to evaluate the basic damage caused on the animal which could either increase or decrease the oxygen uptake (Venice, 2014). Variation in respiration rate is an indicator of stress and frequently to evaluate the changes in metabolism (Chebbi and David, 2016). Oxygen consumption is a valuable indication of sub lethal stress. The decrease in oxygen consumption appears to be a protective measure to ensure that there is low intake of the toxic substance. Gills, are the most important organs of the respiration, continuously exposed to water to absorb vital oxygen, they become the first target to come in contact with external toxicant environment. (Pawar. Neelam, et..al 2016). They are the major respiratory organs were subjected to damage due to pesticide toxicity and causing chain of destructive events, which ultimately lead to respiratory distress. (Magar and Patil 2000). Gills perform various functions like respiration, osmoregulation and excretion of nitrogenous waste. So all metabolic pathways depend upon the efficiency of the gills and they reflect the metabolic state of fish through respiratory activities. Hence gills are important biomarkers of the water pollution and good indicator of the water quality. It has been tried to assess (Chlorpyriphos EC 20%) altered respiratory responses in mosquito fish, *Gambusia affinis* which can be used as a bioindicator for assessing pesticide toxicity to fish. Hence, the present study was undertaken to evaluate the effect of the pesticide, Choropyriphos 20% EC on oxygen consumption of the mosquito fish *Gambusia affinis*.

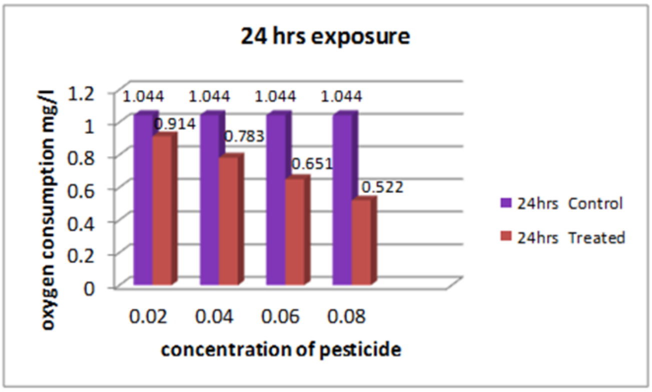
II. METHODS AND MATERIAL

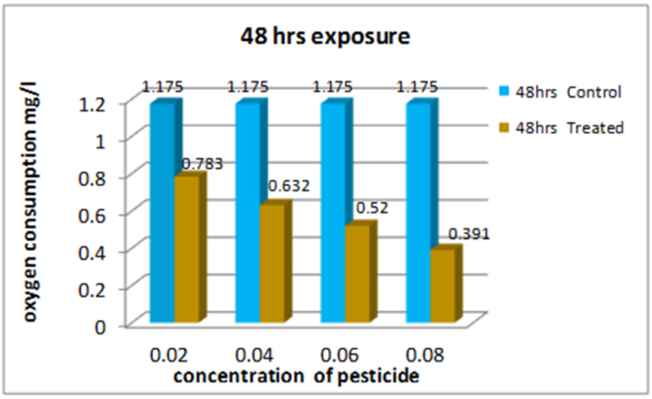
In the present investigation almost equal sized mosquito fish *Gambusia affinis* were collected from the local pond of the Sangamner College, Sangamner. Dist, Ahemadnagar India. The fish were cleaned by using 0.1% $KMNO_4$, to avoid dermal infection. The fish were acclimatized for two weeks in the laboratory condition. During the period of acclimatization the water was changed for every 24hrs. The fishes were fed with pond water containing zooplanktons. The most widely used organic insecticide in and around Sangamner for agriculture Chlorpyriphos 20% EC was selected as the toxicant for the present study. Feeding of the experimental animal was stopped prior to experiment. After acclimatization, fish were kept in up to 3 liters capacity jars. Fish were exposed to sub lethal concentration of this pesticide (Chlorpyriphos) for 24hrs to 96 hrs respectively. The oxygen consumption was carried out in a respiratory chamber (stopper bottle or BOD bottle). Two sets were carried out for control and treated fish. One set kept as control consisting of fishes, which were placed in de chlorinated water without any pesticidal residue. In treated, fishes were exposed to different concentration (0.02, 0.04,0.06,0.08) of chlorpyriphos EC 20%. The consumed oxygen was measured from 24hrs to 96hrs with an interval of 24hrs. 6 fishes were taken for each experiment as well as control. The amount of oxygen consumption in this sample was determined by using Wrinkle's method. The same procedure was repeated for 48, 72, 96hrs and control set also. The difference in the oxygen concentration of the initial and final sample is taken as the amount of oxygen consumed by the fish during the period of experiment. The oxygen consumed by control and exposed fish were determined accordingly, the values for oxygen consumption were expressed as mg/l/24hrs.

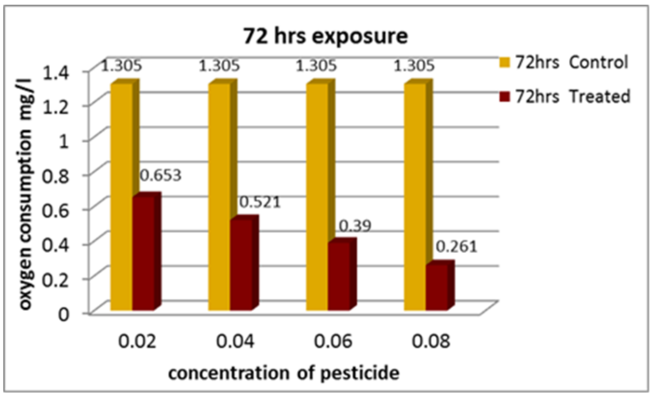
III. RESULTS AND DISCUSSION

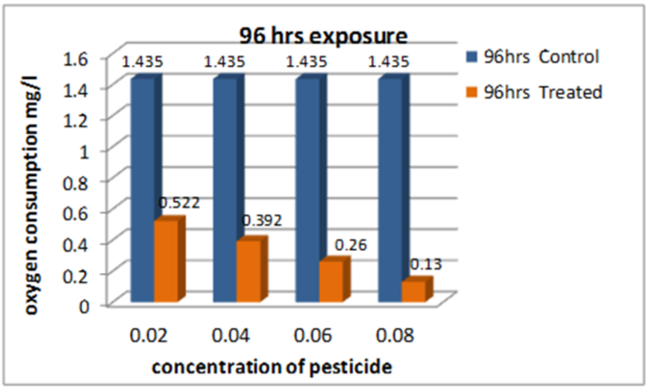
In the present investigation, the oxygen consumption level was observed in the different concentration (0.02, 0.04,0.06,0.08) of chlorpyriphos pesticide at different time of exposure periods (24, 48, 72, and 96 hrs). The oxygen consumption was seen gradually decreasing with increasing exposures periods. Oxygen consumption exhibited decreasing trend in treated group up to 96 hrs as compared with control groups. The result of the experiments and control values are graphically represented in fig -1 by taking different concentration of pesticide (chlorpyriphos) on X-axis and oxygen consumption mg/L on Y-axis.

Fig.1: Oxygen consumption level (mg/L) by *Gambusia affinis* at different concentration of Chlorpyriphos 20%EC, at different time of exposures (hrs)









The rate of oxygen consumption of fish *Gambusia affinis*, exposed to different concentration of pesticide for 24, 48, 72, and 96 hours of exposure are shown in above figure. It was noted that the oxygen consumption of control fish were 1.044, 1.175, 1.305, 1.435 mg/L at 24, 48, 72 and 96 hours respectively. The oxygen consumption of fish exposed to at 24hrs to the Chlorpyriphos 20%EC concentration of 0.02ppm, 0.04ppm, 0.06ppm, 0.08ppm; oxygen consumption were obtained to be 0.914, 0.783, 0.651, 0.522 mg/L respectively. The maximum oxygen consumption was observed at 0.02, whereas the minimum content was recorded at 0.08ppm. The fish exposed at 48hrs of treatment showed the different level of oxygen consumption. The mean of three reading were taken. The mean values were found to be 0.783, 0.632, 0.520, and 0.391mg/L in 20 % concentration of 0.02, 0.04, 0.06 and 0.08 ppm respectively. The maximum consumption was observed at 0.02 whereas minimum was at 0.08 ppm. At 72hrs of exposure, the values showed decline the level of oxygen consumption. The values were found to be 0.653, 0.521, 0.390, and 0.261mg/L in different concentration of 0.02, 0.04, 0.06, and 0.08ppm respectively. The oxygen consumption was higher in 0.02ppm than the remaining concentration. At the 96hrs of exposure, concentration of 0.02, 0.04, 0.06,and 0.08ppm depicted a reduction In oxygen consumption of 0.522, 0.392, 0.260 and. 130 mg/L respectively.

In the present study, the oxygen consumption was gradually decreasing with increasing exposure periods. Manjula Sree Veni S (2014) reported that the oxygen consumption of the exposed fish *Cirrhinus mrigala* exposed to Cypermethrin found to be declined gradually till the end of exposure period (24), due to the interference in oxidative metabolism and also due to the histopathalogical changes that occurred in the gill anatomy. During the study, the rate of oxygen consumption increased in lower concentration and decreased in the higher concentration as reported by several scientists in the toxicant exposed fishes several authors (Rao, 2005, Siva kumar and David, 2004, Veeneth kumar and David 2008) reported that the disturbance in oxidative metabolism leads alteration in whole oxygen consumption in different species of fish exposed to pesticide. It has been well documented that the toxicity of these pesticide to fish may decreased the level of oxygen consumption at different times of exposures under all exposed concentration. Oxygen consumption is decreased when the time of exposure to toxicant is increased. It has been found that, in mosquito fish *Gambusia affinis*, long or short term exposure of pesticide

Chlorpyriphos 20%EC, decrease the amount of oxygen consumption to a significant level as compared to control. The decreased oxygen consumption in Chlorpyriphos 20%EC, exposed fish is likely due to the absorbance of a greater amount of pesticide, which directly effect on the respiratory physiology of fish (O_2 consumption). Thus, it is clear from this study that, the commonly used pesticide chlorpyriphos 20%EC affect the oxygen consumption of mosquito fish, *Gambusia affinis* under all exposed concentration.

IV. CONCLUSION

The present study has clearly showed that commonly used pesticide in agriculture, Chlorpyriphos 20%EC affect the oxygen consumption of mosquito fish, *Gambusia affinis* under all exposed concentration. The oxygen consumption level of the exposed fish at different times of exposure (24, 48, 72 and 96 hrs) was found to be declined gradually till the end of exposure period (96hrs), the gradual decline was attributed to the interference of pesticide with oxidative metabolism. This pesticide caused respiratory distress, decreased level of oxygen consumption in fish *Gambusia affinis*, which suggested that Chlorpyriphos 20% EC is highly toxic to fish. Possibility of water pollution by this pesticide can be monitored through adapting proper assessment mechanism.

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Potability of Groundwater from Several Villages in Sinnar Area, Nashik District, Maharashtra, India

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ABSTRACT

The water and environment has become a poignant concern with the societies and approach makers. The contamination of water and atmosphere due to anthropogenic actions of human beings is a serious problem. The primary objective of this paper is to study the various groundwater sources in the surrounding of several villages from Sinnar Area, Nashik District (MS), India. The Iron concentration varied from 0.04 to 1.48 mg/L, and average concentrations of Iron was reached to 0.46 mg/L. Nitrate concentration oscillated from 02 to 162 mg/L, and average value of Nitrate is 53.13 mg/L in the study area. The average concentration of Fluoride in the study area is 0.57 mg/L. Among the fifteen samples collected seven are found to be potable.

Keywords : Groundwater Superiority, Anthropogenic Actions

I. INTRODUCTION

Water is valuable gift given by the nature for all types of live stocks. Availability of clean water is the first priority for human civilization but now a days due to increasing globalization, the groundwater sources have become highly polluted. The primary objective of this paper is to study the various groundwater sources in the surrounding of several villages from Sinnar tehsil, District Nashik (MS), India.

II. METHODS AND MATERIAL

1. Sampling

Total fifteen samples (S_1 to S_{15}) are collected for analysis in cleaned polythene bottles. Out of fifteen samples, 04 was from Dug well, 07 are from Hand pumps, and 04 are from power pumps.

2. Experimental Methods

The standard methods of APHA are used for the complete analysis. Fluoride is analyzed by SPANDS method at 570 nm. Iron is estimated by UV Visible spectrophotometer at 480 nm (Chemito UV 2100). The Nitrate concentration is measured by phenol disulphonic acid method at 410 nm using UV Visible spectrophotometer (Chemito UV 2100).

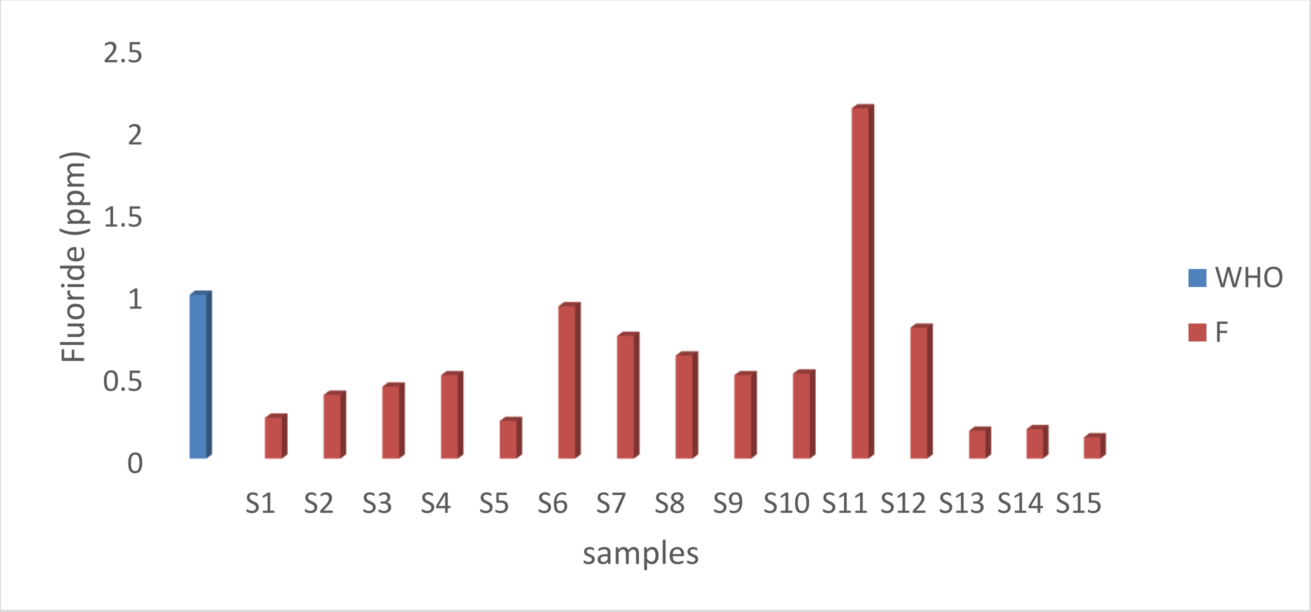
III. RESULTS AND DISCUSSION

The investigated results are depicted in Table 1 along with permissible range as declared by WHO and BIS. The variation of parameters like Fluoride, Iron and Nitrate is shown graphically through Graph 1, Graph 2 and Graph 3 respectively.

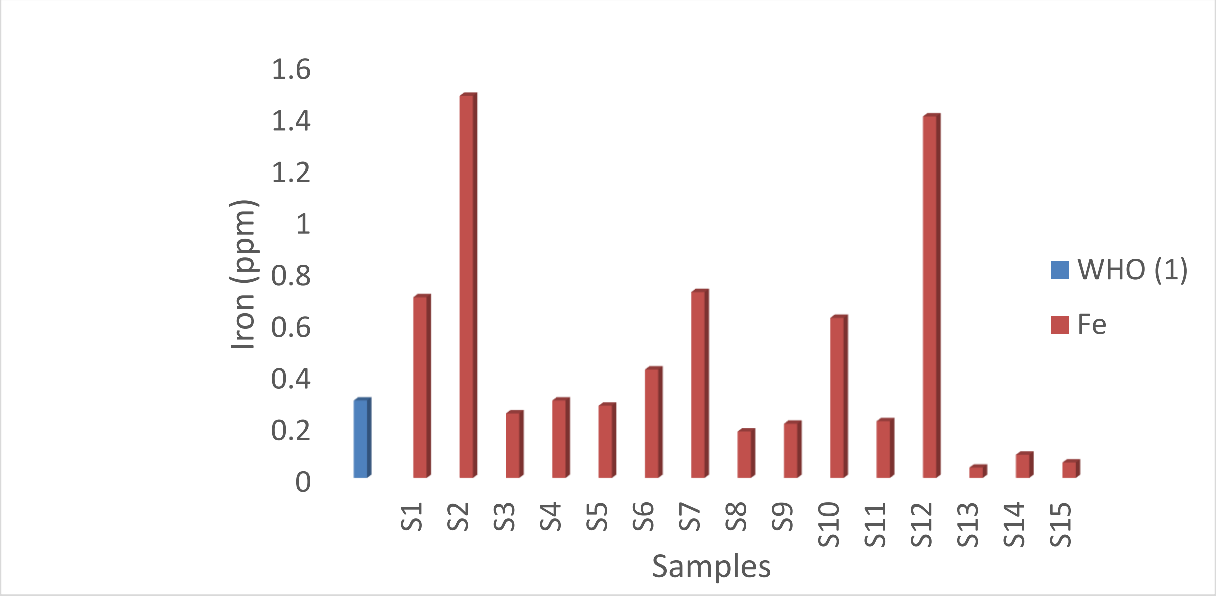
Table 1 : Groundwater quality of collected samples compared with standard data

Parameters of drinking water	WHO(1993)	BIS		Analyzed samples range
		Acceptable limit	Maximum Permissible Limit	
pH	Not mentioned	6.5-8.5	No relaxation	6.96-7.99
TDS (ppm)	No guidelines	500	2000	79-1255
Fluoride(ppm)	1.5	1.0	1.5	0.13-2.13
Iron (ppm)	0.3	0.3	1.0	0.04-1.48
Nitrate(ppm)	45	45	45	2-162

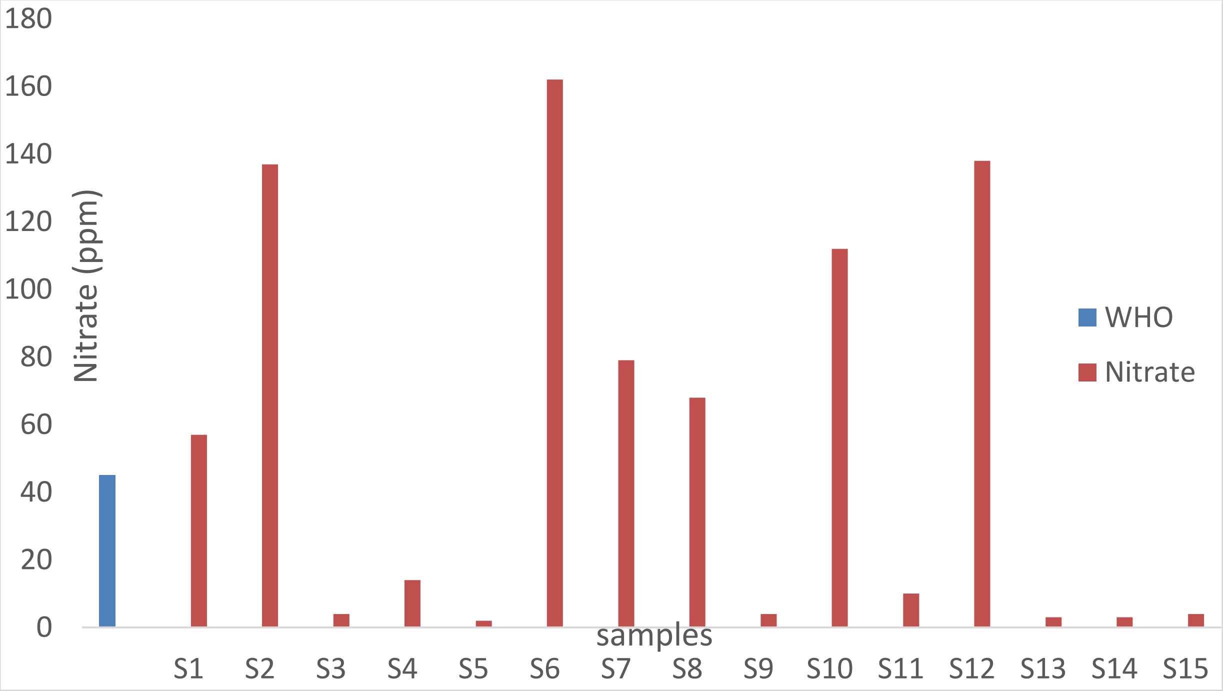
Graphical representation



Graph 1. Variation of Fluoride in water samples



Graph 2. Variation of Iron in water samples



Graph 3. Variation of Nitrate in water samples

The analysed samples show pH in the range 6.96-7.99. It is clear that all fifteen samples are having the values within the range given by the WHO[1]. The analyzed samples shown the TDS values ranging from 79-1255 ppm. Out of fifteen, 08 are within the permissible limit.

High concentration of Fluoride ion causes dental fluorosis as well as skeleton fluorosis[2]. The concentration of fluoride in 15 analyzed samples are ranging from 0.13-2.13. Out of fifteen samples, 14 samples are in permissible limit given by WHO.

The high level of Iron may cause several health problems such as liver cancer, diabetes, cirrhosis of liver, diseases related to heart and central nervous system, infertility etc. The overload of Iron leads to adverse changes in colour, odour and taste of water and it also stains clothes[3]. The Iron concentration oscillated from 0.04 to 1.48 mg/L in the studied area. The maximum permissible limit of Iron in drinking water is 1.0 ppm. Out of fifteen, 08 are within the permissible limit.

Infants and pregnant or nursing women are especially susceptible to health problems from drinking water with Nitrate levels above 10 ppm. The nitrate contaminant affect the human health[4]. Nitrate concentration ranged from 2 to 162 mg/L in studied area. Out of fifteen, 08 are within the permissible limit.

IV. CONCLUSION

Parameter	S1	S3	S4	S5	S8	S9	S11	S13	S14	S15
pH	√	√	√	√	√	√	√	√	√	√
TDS (ppm)	√	×	√	√	×	√	√	√	√	√
Fluoride(ppm)	√	√	√	√	√	√	×	√	√	√
Iron (ppm)	×	√	×	√	√	√	√	√	√	√
Nitrate(ppm)	×	√	√	√	×	√	√	√	√	√

From the above findings, it is concluded that only five water sources (S_5, S_9, S_{13}, S_{14} and S_{15}) qualify to be potable water sources. For rest of the above sources, one has to make compromise for one or two parameters.

Acknowledgment

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Molecular Interaction Studied of Binary Liquid Solutions of isoamyl alcohol and isobutyl alcohol in benzyl amine

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ABSTRACT

Viscosity(η), Density(ρ) and Ultrasonic velocities(U) are reported for binary mixtures of of isoamyl alcohol and isobutyl alcohol with benzyl amine over entered range of mole fractions at 303.15 and 313.15 K and atmospheric pressure. The viscosity deviation (Δη) Excess molar volume (VE) and isentropic compressibility (Ks) have been calculated. These values were fitted with Redlich-Kister type polynomial equation.

Keywords : Viscosity, Density, Ultrasonic velocities, Redlich-Kister.

I. INTRODUCTION

The Viscosity (η), Density (ρ) and Ultrasonic velocities (U) measurements find wide applications in characterizing the physico-chemical behavior of liquid mixtures and in the study of molecular interactions[1-3]. The measurements of Ultrasonic velocity of a liquid and mixtures allows the calculations of compressibility and hence enables to obtain structural information [4], In turn the data of sound velocity can be subjected to scrutiny by applying Jacobins free length and schaaffs collision factor theory. The deviations observed in free length and other parameters have been attributed to dipole-dipole, dipole-induced dipole and other dispersive force interactions[5-7]. It is well known that aqueous solutions of hydrocarbons, alcohols, amines, ethers are characterized by H-bonding and hydrophobic interactions. The investigations regarding the molecular association in organic binary mixtures having alkanol group as one of the component is of particular interest, since alkanol group is highly polar and can associate with any other group having some degree of polar attractions. benzylamine is protic, strongly associated due to highly polar -NH_2 group in the molecule and has large dipole moment [14].

In view of the importance mentioned, an attempt has been made to elucidate the molecular interactions in the mixtures of benzylamine with branched alkanols (C_5 & C_4) at all compositions and four temperatures. In this article only the data of binary mixtures of benzyl amine with isoamyl alcohol and isobutyl alcohol respectively at *303.15 and 313.15 K* is reported.

II. METHODS AND MATERIAL

A. Experimental:

All the chemicals used in the present research work are analytical reagent (AR) and spectroscopic reagent (SR) grades of minimum assay of 99.9% obtained from E-Merck, Germany and Sd Fine chemicals, India, which are used as such without further purification. The purities of the above chemicals were checked by density determination at *303.15 and 313.15 K* the uncertainty is less than ± 1×10^{-4} gcm^{-3}. The binary liquid mixtures of different known compositions were prepared in stopper measuring flasks. The density, viscosity and velocity were measured as a function of composition of the binary liquid mixture of benzyl amine with isoamyl alcohol and isobutyl alcohol respectively at *303.15 and 313.15 K*. The density was determined using a Bi-capillary pyknometer. The weight of the sample was measured using electronic digital balance with an accuracy of ± 0.1 mg (Model: Shimadzu AX-200). An Ubbelohde viscometer (20ml) was used for the viscosity measurement and efflux time was determined using a digital clock to within ±0.01s. An ultrasonic interferometer having the frequency of 2 MHz (Mittal Enterprises, New Delhi, Model: F-81) with an overall accuracy of ± 0.1% has been used for velocity measurement. An electronically digital operated constant temperature bath (RAAGA Industries) has been used to circulate water through the double walled measuring cell made up of steel containing the experimental solution at the desired temperature with an accuracy of ± 0.01 K.

B. Theory and Calculations

Excess volumes of the mixtures have been calculated using density and mole fraction data given by equation:

$$V^E = (M_1X_1+M_2X_2)/\rho_{12} -(M_1X_1) /\rho_1 - (M_2X_2) / \rho_2 \qquad \text{-- (1)}$$

Viscosity of Binary Mixtures is calculated by:

$$\ln\eta_m = X_1\ln\eta_1 + X_2\ln\eta_2 \qquad \text{--(2)}$$

The measured viscosities of the mixtures have been used to obtain deviation in Viscosity parameters on the basis of linearity in following way,

Deviation in Viscosity of Binary Mixtures is calculated by :

$$\Delta\eta_m = \eta_{12} - X_1\eta_1 - X_2\eta_2 \qquad \text{--(3)}$$

Deviation in isentropic compressibility have been evaluated by using the equation

$$\Delta k_S = k_S - (\Phi_1 k_{S1} + \Phi_2 k_{S2}) \qquad \text{--(4)}$$

where k_{S1}, k_{S2} and K_S are isentropic compressibility of liquid mixtures and Φ is volume fraction of pure i^th component in the mixture and is defined as

$$\phi = (x_i V_i) / (\Sigma x_i V_i)$$

where x_1 and V_i are mole fraction and molar volume of ith component in the mixture

The excess properties y^E are fitted by the method of non linear least squares to a Redlich kister type polynomial (5)

$$y^E = X_1X_2\sum A_i(X_1 - X_2)^i) \qquad \text{--(5)}$$

In each case the optimum number of coefficients Ai was determined from an examination of the variation of standard deviation as calculated by :

$$\sigma y^E = [\sum(y^E_{obs} - y^E_{cal}) / (n-m)]^{1/2} \qquad \text{--(6)}$$

where n represents the number of experimental points and m represents the number of coefficients in fitting the data.

$$Z^E = Z_{mix} - x_1Z_1 - x_2 Z_2 \qquad \text{--(7)}$$

$$L_f^E = L_{fmix} - x_1L_{f1} - x_2L_{f2} \qquad \text{--(8)}$$

Various physical and thermo dynamical parameters are calculated from the measured data such as

AdiabaticCompressibility

$$\beta s = 1/(U^2\rho) \qquad \text{--(9)}$$

Intermolecularfreelength

$$L_f = K\sqrt{\beta} \qquad \text{-- (10)}$$

where K is a temperature dependent constant.

Freevolume

$$V_f = (M_{ef}U/K\eta)^{3/2} \qquad \text{--(11)}$$

where M_{ef} is the effective molecular weight (=mM M x, in which m and x are the molecular weight and the mole fraction of the individual constituents respectively). K is a temperature for all liquids.

Availablevolume

$$V_a = VT(1-U/ U) \qquad \text{-- (12)}$$

where U is the limiting velocity and is taken as 1600 ms and V is the molar volume at TK.

Excess values of the above parameters can be determined using

$$A^E = A_{exp} - A_{id} \qquad \text{--(13)}$$

where $A_{id} = \sum Ai\ Xi$, Ai is any acoustical parameters and Xi the mole fraction of the liquid component.

In this content, the excess properties like excess volume, excess partial volume, excess free energies and entropies have been found useful in characterizing the molecular interactions. Alcohols are linear H-bonded liquids, have been studied in detail, they show aggregation in non polar solvents like CCl_4[5]. In this we thought on the problem that the self-association of alcohol molecules can be detected in polar solvents like benzyl amine. It has been examined by studying the excess volume, excess viscosity and other parameters. The results and other details are given bellow.

III. RESULTS AND DISCUSSION

The experimental values of density (ρ) viscosity (η) ultrasonic velocity (U), Excess volume (V^E), viscosity deviations (Δη) and deviation on isentropic compressibility (Δk_S) for the two binary liquid systems at 303.15 and 313.15 K, are given in Tables 1 and 2. The variation of excess parameters with the mole fraction of alkanols (x_2) at 303.15 and 313.15 K are plotted in Fig.1-8.

The measured parameters viz. density (ρ) viscosity (η) ultrasonic velocity (U) and calculated parameters such as adiabatic compressibility (K_s), intermolecular free length (L_f) and voiume fractions(Φ)for the System 1 : isoamyl alcohol (1) + benzyl amine (2) at 298.15 K are given in Table 1 and 2, for the System 2 : isobutyl alcohol (1) + benzyl amine (2) at 303.15 and 313.15 K are given in Table 1 and 2.

It is evident from the Table 1 and Table 3, that both the systems (I) and (II), ultrasonic velocity increases with increase in concentration of benzyl amine (2). This indicates that, strong interaction observed at lower concentration of alcohols. The density values also have the same trends with velocity in the systems (I) and (II). Density increases due to the increased electrostriction in that solution. This more electrostriction decreases the volume and hence increases the density.Viscosity decreases in systems I and II with increase in benzyl amine, suggesting there by more association between solute and solvent molecules.

The adiabatic compressibility (Ks) increases with increase in the concentrations of isoamyl alcohol / isobutyl alcohol in benzyl amine (2). This is due to increase in structural disorder of benzyl amine resulting in less cohesion, and leads to a increase in K_s. The increase in K_s results in an decrease in the value of U.

The parameter, the free length (L_f) is calculated from the ultrasonic velocity (U) and adiabatic compressibility (K_s). It is observed that L_f, increases with the concentration of isoamyl alcohol / isobutyl alcohol in benzyl amine (2). Increase in intermolecular free length leads to negative deviation in sound velocity and positive deviation in compressibility. This indicates that the molecules are away from each other in the both system.

Values of G^{*E} are more negative at lower temperature provides additional evidence for the existence of interactions of weak magnitude like dipole-induced dipole type between components of liquid mixtures[12]. The magnitudes of G^{*E} for isoamyl alcohol are slight positive than that of isobutyl alcohol at both temperatures.

In their pure state, the self association of alkanols decreases with increasing chain length, when alkanols are mixed with benzyl amine then there is interaction between their individual functional groups (-OH and $-NH_2$). The presence of electron withdrawing group on benzene ring decreases its electron densitie[8,9]

The curves for V^E, $\Delta\eta$, Δk_S and G^{*E} values are plotted against mole fractions of alkanols, these curves are negative over the most mole fraction of the alkanols at 303.15 and 313.15 K (Fig.1-8). These curves are U-shaped with minima around x_1 = 0.4-0.5 mole fraction of the alkanol.These excess parameters at a particular mole fraction of the alkanols becomes less negative with increase of temperature. The negative values may be attributed to the existence of dispersion and dipolar forces between unlike molecules and related to the difference in size and shape of the molecules[10,11] The V^E values are negative at lower temp. as decrease in temperature disturb hereto and homo-association of the molecules which causes decrease in fluidity of the liquid. The V^E values are positive at 313.15K. The magnitude of V^E, $\Delta\eta$, Δk_S, and G^{*E} the sign and the extent of deviation of these properties from ideality depend on the strength of interaction between unlike molecules. The excess viscosity gives the strength of the molecular interaction between the interacting molecules. For systems where dispersion, induction and dipolar forces which are operated by the values of above excess parameters are found to be negative, the large negative values of excess viscosity for all the systems can be attributed to the presence of the dispersion, induction, and dipolar forces between the components. The magnitude of V^E, $\Delta\eta$ and Δk_S are more at higher temperature provides inverse relation with G^{*E}

IV. CONCLUSION

The present investigation shows that greater molecular interaction exists in isobutyl alcohol mixtures which may be due to hydrogen bond formation and weak molecular interaction that exists in the isoamyl alcohol mixtures and which may be due to the dominance of dispersion forces and dipolar interaction between the unlike molecules[12]. The V^E values are positive at 313.15K as the interaction tends to be weaker with rise in temperature which may due to weak intermolecular forces and thermal dispersion forces[13,14]. The existence of molecular interaction in the mixture is in the order: isobutyl alcohol > isoamyl alcohol

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Identification of anti-inflammatory constituents from pet ether extract Justicia Gendarussa Burm. f. with GC-MS

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ABSTRACT

Justicia gendarussa Burm.f.is a medicinal plant in Western ghats of Maharashtra in India. The study aims to determine active fractions of *Justicia Gendarussa* for its anti-inflammatory activity and identify their chemical constituents. Physicochemical parameter 5.57 % ± 0.430 ash value indicate quality and purity of crude drug. It also indicates the presence of inorganic radicals like phosphates, carbonates and silicates of sodium, potassium, magnesium, calcium etc. Sometimes inorganic variables like silica, calcium carbonate, calcium oxalate content of the crude drug can affects total ash value 5.97% ± 0.212 moisture content WHO guidelines for quality control recommended for less moisture content in crude drug during storage to avoid the bacterial and fungal growth.14.81 % ± 0.519 alcohol soluble extractive and 30.91 % ± 2.220 water soluble extractive. Phytochemical tests showed the presence of alkaloids, steroids and terpenoids, tannins, glycosides, saponins, flavonoids, phenolic compounds and carbohydrates. Elemental analysis by atomic absorption spectroscopy results 61.04 % Fe, 37.14 % Mg, 21.8 % Zn, 10.28% Cu, 2.33 % B in *Justicia gendarussa* Burm.f. Fluorescence analysis gives qualitative assessment some crude drugs with standardization crude drug of *Justicia gendarussa* Burm.f. act differently at different light wavelength. Various constituents of extract showed fluorescence in U.V. range, visible range or in both. The identification of phytoconstituents by GCMS are Caryophyllene, Epiglobulol, 1-Penten-3-one, Hexadecanoic acid, 9, 12, 15-Octadecatrienoic acid, Hexadecanoic acid etc in pet ether extract. As the NIST data Caryophyllene, Hexadecanoic acid and Beta sitosterol showed strong Anti-inflammatory activities.

Keywords : Anti-Inflammatory, Physicochemical Parameter, WHO, GCMS, NIST

I. INTRODUCTION

Herbal medicines have affective treatments on kind of physical and mental health problems, including rheumatism. Contemporary herbal drugs have changed notably from its traditional methods to commercial merchandise being extensively to be had to the general public as over the counter supplements (Zhang Y et.al.). According to reports of worldwide health agency (WHO), Out of overall world's population and about 80 % individual relies on traditional remedy to conform their primary healthcare requirements. From early Nineties, the use of herbal medicines has steadily extended in Western nations as trading and used extensively by medicine practitioners. Posadzkiet al. in 2013 reported that lifetime effect in Western nations were 31% in UK. As per survey of Thompson et al. in 2013 37% in Australia has lifetime effect. Out of 121 medicines come from herbal resources in America, ninety isolated drugs are plant based. Top 25 bestselling drugs in world are originated from the herbal origin. The nonsteroidal anti inflammatory drugs like naproxen and diclofenac, and immunosuppressant like cyclosporine are derived from the herbal resources.

Kali Tilwan belongs to family Acanthaceae with *gendarussa* species. The whole plant has been used in clinical for years as a traditional Indian medicine (TIM). The whole plant is used to treat cold, acute gastricabsces and arthritis by ethanaopharmacological practitioner. The previous pharmacological studies demonstrated that the extracts of *Justicia gendarussa* burm f. reported antioxidant and rheumatic properties. Although more than fifty compounds including phenols, triterpenes, long chain hydrocarbons and so on. Only very few chemical components were recognized to be responsible for certain effects. Roots of Justicia *gendarussa* burm f. showed protective effect of *Justicia gendarussa Burm.f.* on carrageenan-induced inflammation (Kavitha S K et al). Anti-arthritic potential of the plant Justicia gendarussa Burm f. on stem (Paval Jaijesh et al.). Anti-inflammatory potential of an ethyl acetate fraction isolated from *Justicia gendarussa Burm f.* roots through inhibition of iNOS and COX-2 expression via NF-kB pathway (Kumar et al.).

II. METHODS AND MATERIAL

Collection of plant material carried out from Western Ghats of Sahyadri ranges i.e. Kalsubai and Harishchandragad region. Authentification carried out at BSI, Pune (voucher number: BSI/WRE/Tech/2013/1154). Extraction of dried leaves carried out by Soxlet hot extraction method followed by preliminary phytochemical screening of crude extracts. The leaves of *Justicia gendarussa* Burm.f. were shade dried (3.0 Kg), grinded to powder and then defattation carried out by with pet-ether for 48 h by soxhlet extraction. Pet-ether extract dried under reduced pressure using rotary evaporator (Heidolph Labrota 4000 Efficient, Germany). The colour of extract obtained was dark brown and the percentage yield was 4.50% (w/w). Physicochemical parameter analyzed as like moisture content, water / alcohol soluble extractive values, Total ash value, Acid-insoluble ash value, Alcohol-soluble extractive value etc. Fluorescence analysis carried out as per the method of Chase and Pratt (1949), the fluorescence analysis was carried out qualitatively. Powdered drug monitored in day light and ultra-violet light (254 nm and 365 nm) with different chemicals. Elemental analysis carried out by AAS. The preliminary phytochemical tests of various extracts of Justicia *gendarussa Burm.*f. leaves was carried out. Test for carbohydrates carried out by Molisch test, Fehling's test. Test for glycosides.

Carried out by Keller-Killani test, Borntrager test. Test for proteins carried out by Biuret test, Millon's test. Test for steroids carried out by Salkowski test, Liebermann-Burchard test, Liebermann's test. Test for triterpenes are carried out by Vanillin-sulphuric acid test. Saponins are estimated by foam formation test. Test for alkaloids Dragendorff's test, Mayer's test, Hager's test, Wagner's test. Test for tannins and phenolic compounds are estimated by Ferric chloride test, Dilute nitric acid test etc. The GC-MS analyses were performed by using the TRACE™ 1300 gas chromatography instrument coupled to the mass spectrometer make Thermo Scientific (Model :TSQ 8000).Column (make TG 5MS) 30 m × 0.25 mm dimensions which coated with 0.25 μm film. Carrier gas: Helium gas, with a constant flow rate of 1 ml/min. Hydrogen gas used as fuel. Oven temperature: 60 to 280°C. Thermal Conductivity Detector has temperature: 250°C.

The electron impact mass spectra were measured at acceleration energy of 70 eV. The manual injection of 1.0 μl of the solution of isolated compound was performed in the split mode at a 20:1 split ratio. The interpretation of GC-MS was conducted using the database of National Institute Standard and Technology (NIST). The mass spectrums of unknown compounds were compared with the spectrum of the known components stored in the NIST library and published data. Identification of Phytoconstituent are carried out by GCMS and compared the data with NIST library. Fingerprinting of phytoconstituents on basis of retention time.

III. RESULTS AND DISCUSSION

A. Physicochemical parameters:

Powdered leaves of *Justicia gendarussa* Burm.f used for physicochemical standardization for estimation of impurities present in crude drug. The different parameters with their results are presented in standardization of the crude drugs the extractive values play a very significant role. With different solvents extraction carried out gives assurance to find different contamination and fatigued constituents e.g. presence of the adulterants water are indicated by alcohol soluble extractive values, substandard processing and poor value of the drug. Ash value gives quality and purity of crude drug. It also indicates the presence of inorganic radicals like phosphates, carbonates and silicates of sodium, potassium, magnesium, calcium etc. Sometimes inorganic variables like silica, calcium carbonate, calcium oxalate content of the crude drug can affects total ash value. WHO guidelines for quality control recommended for less moisture content in crude drug during storage to avoid the bacterial and fungal growth.

Table 1 Physicochemical standardization of the leaves of *Justicia gendarussa* Burm.f.

Sr.No.	Parameter	Values (% w/w)
01	Water soluble extractives	30.91 % ± 2.220
02	Alcohol soluble extractives	14.81 % ± 0.519
03	Total ash value	5.57 % ± 0.430
04	Acid insoluble ash value	2.56 % ±0.121
05	Water soluble ash value	5.7 % ± 0.231
06	Moisture content (Loss on drying)	5.97 % ± 0.212

B. Fluorescence study

The fluorescence phenomenon shows by plant powder or its extracts. Crude drug may be act differently at different light wavelength. Various constituents of extract showed fluorescence in U.V. range, visible range or in both. Some substances do not show fluorescence phenomenon directly. After decomposition with various reagents to form products showed fluorescence phenomenon.

Table 2. Fluorescence analysis of *Justicia gendarussa* Burm.f. leaf powder.

C. Elemental Detection

Treatment	Visible	UV at 254nm	UV at 365 nm
Powder as such	Dark green	Green	Green
Powder + water	--	Greenish	--
Powder + ethanol	Dark green	Green	Red
Powder + 1N NaOH (aq.)	Yellow	Green	--
Powder + 1N NaOH (alc.)	Light green	Green	--
Powder + 1N HCl	Light green	Green	--
Powder + Conc. HCl	Dark green	Green	--`
Powder + 50% HNO3	--	Green	--
Powder + Conc. H2SO4	Brown	Dark green	Greenish

Sr.No.	Element analyzed	Content in *Justicia Gendarussa* Burm.f.

01	Fe	61.04 %
02	Mg	37.14 %
03	Zn	21.8 %
04	Cu	10.28%
05	B	2.33 %

Table 3 Elemental analysis of *Justicia gendarussa* Burm.f.

D. Preliminary phytochemical screening of extracts

The preliminary phytochemical screening of various extracts from the leaves of *Justicia gendarussa* Burm.f. as shown in **Table 4.**

Phytoconstituents	**Test**	**Pet-Ether extract**
Alkaloids	Dragendorff's test, Mayer's test, Hager's test, Wagner's test	+
Steroids	Liebermann-Burchard reaction Liebermann reaction Salkowski reaction	+
Triterpenes	Vanillin-Sulphuric acid	–
Tannin and Phenolics	5% $FeCl_3$ solution Dilute nitric acid	–
Glycosides	Keller-Killani Borntrager	–
Flavonoids	Shinoda Lead Acetate test NaOH test Zn/HCl test	–
Saponins	Foam test	–
Carbohydrates	Molisch's test Fehling's test Benedict's test	

E. GCMS analysis

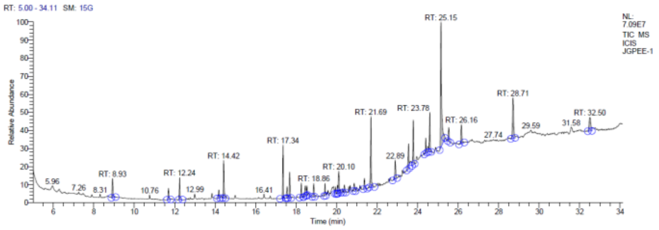

Fig 1. Chromatogram of Pet-ether extract

Table 5. Identified phytoconstituents from extract JGPEE I extract from the leaves of *Justicia gendarussa* Burm.f.

Retention time (Min)	**Components**	**Peak area (%)**
8.93	3-Dodecene-E	2.51
12.24	**Caryophyllene**	2.13
14.42	Epiglobulol	4.41
17.35	1-Penten-3-one	5.18
18.45	**Hexadecanoic acid**	2.08
20.10	9,12,15-Octadecatrienoic acid	3.28
21.69	2- Heptanone	8.79
23.54	Spiro [4,5] decan-7-one	3.31
23.78	2-Pentanoic acid	5.12
24.59	2-Pentanoic acid	4.60
25.15	**Hexadecanoic acid**	21.86
26.16	Tetraacontane	2.80
32.50	**Beta sitosterol**	3.76

IV. CONCLUSION

Physicochemical parameter 5.57 % ± 0.430 ash value indicate quality and purity of crude drug. It also indicates the presence of inorganic radicals like phosphates, carbonates and silicates of sodium, potassium, magnesium, calcium etc. Sometimes inorganic variables like silica, calcium carbonate, calcium oxalate content of the crude drug can affects total ash value 5.97%±0.212 moisture content WHO guidelines for quality control recommended for less moisture content in crude drug during storage to avoid the bacterial and fungal growth.14.81 % ± 0.519 alcohol soluble extractive and 30.91 % ± 2.220 water soluble extractive.

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Fluorescence analysis gives qualitative assessment some crude drugs with standardization crude drug of *Justicia gendarussa* Burm.f. act differently at different light wavelength. Various constituents of extract showed fluorescence in U.V. range, visible range or in both. The identification of phytoconstituents by GCMS are Caryophyllene, Epiglobulol, 1-Penten-3-one, Hexadecanoic acid, 9, 12, 15-Octadecatrienoic acid, Hexadecanoic acid etc in pet ether extract. As the NIST data Caryophyllene, Hexadecanoic acid and Beta sitosterol showed strong Anti-inflammatory activities.

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Preparation of 2,4 Dinitro-phenyl hydrazone (2,4-DNP) derivatives of aldehydes R-CHO / ketones R-Co-R

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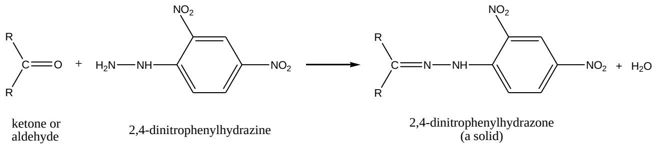
ABSTRACT

An alternative variation of the most common preparation of 2,4-dinitrophenylhydrazine reagent is presented. This method is suitable for scaling up for quantities required for preparing stock reagent for classes. Since the action of 2, 4-dinitrophenylhydrazine (Brady's reagent) on aldehydes and ketones was first investigated by Purgotti in 1894, its usefulness as an analytical reagent for carbonyl containing compounds has been thoroughly exploited. The 2, 4 -DNHP reagent is an orange crystalline solid melting at 1990C. Its usefulness as a reagent stems from the fact that it forms crystalline derivatives which are easily purified and have melting points supposedly characteristic of the aldehyde or ketone used in preparing the derivative. In principle, each aldehyde and ketone should form a single 2,4-dinitrophenylhydrazone (DNPH) with a characteristic melting point. In practice, however, a number of the aldehydes and ketones do not follow this general rule but form multiple derivatives, differing either in melting point, crystal structure, or both. Even the colors of pure DNPH's of a particular aldehyde or ketone are sometimes different.

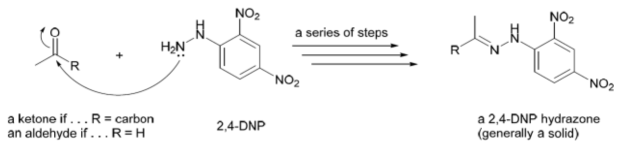
Keywords : 2,4 DNP, Microwave, Aryl Aldehyde.

I. INTRODUCTION

2,4-Dinitrophenylhydrazine reacts with ketones and aldehydes, and is used to distinguish these functional groups from other carbonyl compounds, such as esters, which do not react. The formation of a red or orange precipitate (a 2,4-dinitrophenylhydrazone) constitutes a positive test.



Aldehydes and ketones react with 2,4-dinitrophenylhydrazine reagent to form yellow, orange, or reddish-orange precipitates, whereas alcohols do not react. Formation of a precipitate therefore indicates the presence of an aldehyde or ketone. The precipitate from this test also serves as a solid derivative. A discussion on derivatives will be given later in this handout. The mechanism of this reaction is that of imine formation and can be found in any organic lecture text.

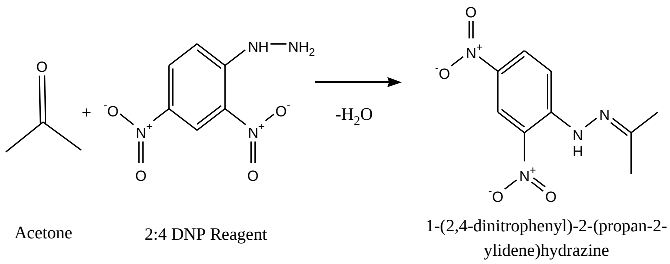


Crystals of the title compound, $C_{13}H_{10}N_4O_4$, were obtained from a condensation reaction of benzaldehyde and 2,4-dinitrophenylhydrazine. The molecule assumes an approximately planar E configuration. Within the dinitrophenyl moiety, the average distance for the aromatic C-C bonds close to the imino group [1.417 (3) Å] is appreciably longer than the average distance for the other aromatic C-C bonds in the same phenyl ring [1.373 (3) Å]. This increased distance may be a result of the overlap of the non-bonding orbital of the imino N atom with the π orbitals of the arene. It is likely that π-π stacking exists in the crystal structure.

Principle :- 2,4 Dinitro-phenyl hydrazine reagent is much more reactive than phenyl hydrazine towards aldehydes and ketones. Both aldehydes and ketones contain a carbonyl group (-co-) of low reactivity often readily react with 2:4 DNP reagent to give a 2,4- dinitro-phenyl hydrazone derivative

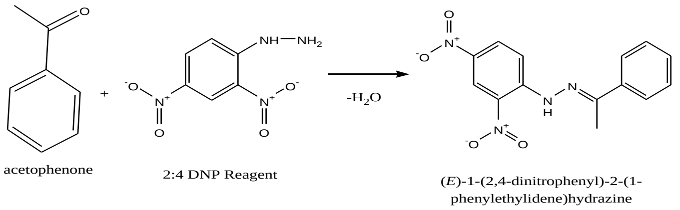
1. Synthesis of 1-(2,4-dinitrophenyl)-2-(propan-2-ylidene)hydrazine.

Take in a clean hard glass test tube 1 gm /ml of the acetone containing aldehyde/ ketonic group. Dissolve it in minimum amount of ethyl alcohol or methyl alcohol (if required heat on water bath).Cool the test tube and add 10 ml of 2:4 DNP reagent. Stir well with the help of glass rod and cool in ice bath.2,4 dinitro-phenyl hydrazones usually separate in well-formed crystals. Filter it on Buchner funnel. Dry the crystal well and record the yield. Recrystallize 2,4 dinitro-phenyl hydrazone derivatives from ethyl alcohol. Dry the crystals well and find its melting point.



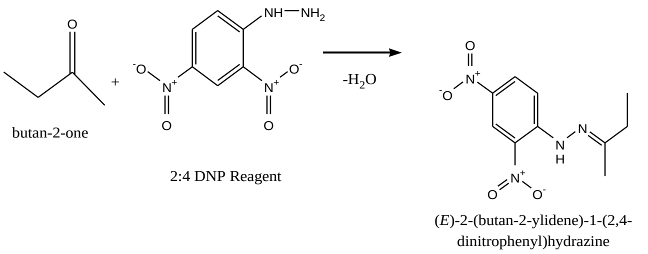
2 . Synthesis of (E)-1-(2,4-dinitrophenyl)-2-(1-phenylethylidene)hydrazine

Take in a clean hard glass test tube 1 gm /ml of the acetophenone containing aldehyde/ ketonic group. Dissolve it in minimum amount of ethyl alcohol or methyl alcohol (if required heat on water bath). Cool the test tube and add 10 ml of 2:4 DNP reagent. Stir well with the help of glass rod and cool in ice bath. 2,4 dinitro-phenyl hydrazones usually separate in well-formed crystals. Filter it on Buchner funnel. Dry the crystal well and record the yield. Recrystallize 2,4 dinitro-phenyl hydrazone derivatives from ethyl alcohol. Dry the crystals well and find its melting point.



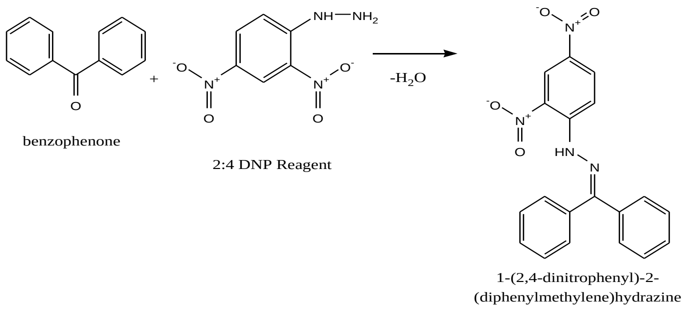
3. Synthesis of (E)-2-(butan-2-ylidene)-1-(2,4-dinitrophenyl)hydrazine.

Take in a clean hard glass test tube 1 gm /ml of the ethyl methyl ketone containing aldehyde/ ketonic group. Dissolve it in minimum amount of ethyl alcohol or methyl alcohol (if required heat on water bath). Cool the test tube and add 10 ml of 2:4 DNP reagent. Stir well with the help of glass rod and cool in ice bath. 2,4 dinitro-phenyl hydrazones usually separate in well-formed crystals. Filter it on Buchner funnel. Dry the crystal well and record the yield. Recrystallize 2,4 dinitro-phenyl hydrazone derivatives from ethyl alcohol. Dry the crystals well and find its melting point.



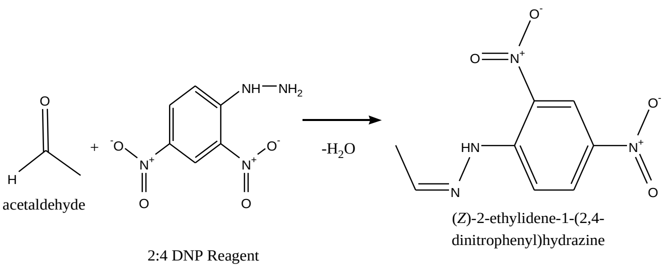
4. Synthesis of 1-(2,4-dinitrophenyl)-2-(diphenylmethylene) hydrazine.

Take in a clean hard glass test tube 1 gm /ml of the benzophenone containing aldehyde/ ketonic group. Dissolve it in minimum amount of ethyl alcohol or methyl alcohol (if required heat on water bath). Cool the test tube and add 10 ml of 2:4 DNP reagent. Stir well with the help of glass rod and cool in ice bath. 2,4 dinitro-phenyl hydrazones usually separate in well-formed crystals. Filter it on Buchner funnel. Dry the crystal well and record the yield. Recrystallize 2,4 dinitro-phenyl hydrazone derivatives from ethyl alcohol. Dry the crystals well and find its melting point.



5. Synthesis of (Z)-2-ethylidene-1-(2,4-dinitrophenyl)hydrazine

Take in a clean hard glass test tube 1 gm /ml of the acetaldehydecontaining aldehyde/ ketonic group. Dissolve it in minimum amount of ethyl alcohol or methyl alcohol (if required heat on water bath). Cool the test tube and add 10 ml of 2:4 DNP reagent. Stir well with the help of glass rod and cool in ice bath. 2,4 dinitro-phenyl hydrazones usually separate in well-formed crystals. Filter it on Buchner funnel. Dry the crystal well and record the yield. Recrystallize 2,4 dinitro-phenyl hydrazone derivatives from ethyl alcohol. Dry the crystals well and find its melting point.



II. RESULTS AND DISCUSSION

Sr. No.	Compound Name	Molecular formula	Molecular Weight	M.P. ^{0}C	Yield %
01	1-(2,4-dinitrophenyl)-2-(propan-2-ylidene)hydrazine.	$C_9H_{10}N_4O_4$	238	126^{0}C	82%
02	(E)-1-(2,4-dinitrophenyl)-2-(1-phenylethylidene)hydrazine.	$C_{14}H_{12}N_4O_4$	300	146^{0}C	86%
03	(E)-2-(butan-2-ylidene)-1-(2,4-dinitrophenyl)hydrazine.	$C_{10}H_{12}N_4O_4$	252	119^{0}C	80%
04	1-(2,4-dinitrophenyl)-2 (diphenylmethylene)hydrazine.	$C_{19}H_{14}N_4O_4$	362	238^{0}C	76%
05	(Z)-2-ethylidene-1-(2,4-dinitrophenyl)hydrazine	$C_8H_8N_4O_4$	224	165^{0}C	69%

- **All the compound synthesized were adequately characterized by their spectral IR, ^{1}H-NMR & Mass Spectra.**

***Spectral Study of 1-(2,4-dinitrophenyl)-2-(propan-2-ylidene)hydrazine:-**

IR cm^{-1} = 3305.64 (-NH), 1492.9(C=C), 1424.26(-NO$_2$)

^{1}H NMR:(δ,ppm) (400MHz, DMSO) 2.1(d,3H), 2.0(dd,3H), 7.8(s, 1H), 8.3(s,1H), 8.9(s,1H), 10.8(1H,(exch.))(NH).

***Spectral Study of (E)-1-(2,4-dinitrophenyl)-2-(1-phenylethylidene)hydrazine**

- ^{1}H NMR:(δ,ppm) (400MHz, DMSO) 3.1(s,3H), 8.5(s,1H), 8.1(s,1H), 8.90(s,1H), 7.9(1H, (exch)(NH), 8.42(d,2H), 7.82(dd,2H), 7.5(d,1H).
- IR cm^{-1} = 3305.64 (-NH), 1498.70 (C=C), 1367.17(-NO$_2$)

***Spectral Study of (E)-2-(butan-2-ylidene)-1-(2,4-dinitrophenyl)hydrazine**

- ^{1}H NMR:(δ,ppm) (400MHz, DMSO) 2.1(t,3H), 2.5(q,2H), 3.3(s,3H), 10.0(1H(exch).(NH), 7.8(s, 1H), 8.39(s,1H), 8.84(s,1H).
- IR cm^{-1} = 3384.95 (-NH),1605.69(C=C), 1490.60(-NO$_2$),1272.83(-C-C-)

***Spectral Study of 1-(2,4-dinitrophenyl)-2-(diphenylmethylene)hydrazine**

- IR cm^{-1} = 3384.95 (-NH),1605.69(C=C), 1490.60(-NO$_2$),1272.83(-C-C-)

***Spectral Study of (Z)-2-ethylidene-1-(2,4-dinitrophenyl)hydrazine**

- IR cm^{-1} = 3384.95 (-NH),1605.69(C=C), 1490.60(-NO$_2$),1272.83(-C-C-)

III. CONCLUSION

1. 2,4 Dinitrophenyl hydrazine can be used to qualitatively detect the carbonyl functionality of a ketone or aldehyde fuctional group.
2 If the carbonyl compound aromatic then the precipitate will be red.
3. If aliphalitic, then the precipitate will have a more yellow colour.
4. The “DNP” test is positive for both aldehydes or ketones, But not for alkenes or ester / acids / amids.
5. This is representative of how H$_2$N-Z reagents react with aldehydes or ketones to eliminate water and make “imines” with a C=N-Z bond. In the chemical test, the DNP reagent is soluble , if a derivative forms, it precipitates from solution.
6. The DNP-derivatives to tend to be highly crystalline.
7. The colour of the precipitate is often informative, saturated carbonyl compounds tend to give yellow derivatives, while unsaturated aldehydes or ketones tend to give red or orange derivatives

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Proximate Analysis of Centella asiatica

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ABSTRACT

Natural products have traditionally provided most of the traditional drugs in use. Despite the achievement of synthetic organic chemistry and advances towards rational drug design system, natural products continue to be the most essential in providing medicinal compounds and as starting points for the development of synthetic analogous. With the increasing power of screening programme and increasing interests in the reservoir of untested natural products, many future drug developments will be based on natural products. The aim of the present work is to carry out proximate analysis of leaves of *Centella asiatica.* They shows presence of **0.84%** foreign organic matter, **9.03%** ethanol soluble extractives, **17.20%** water soluble extractives, **10.50%** total ash, **5.30%** Acid insoluble ash, **10.45**% loss on drying and **6.10%** moisture content.

Keywords: Phytochemicals, Proximate Analysis, *Centella Asiatica* Leaves

I. INTRODUCTION

Most of the crude drugs (Plant materials) are usually put in quarantine store and they remain there for long time. During storage proper ventilation, humidity controls, suitable temperature and light conditions should be ensured to maintain their original pharmacological action. However, it is observed that, crude plant materials, before being taken for processing, are not analyzed which can lead to changes in original characteristics. To avoid this, the crude drugs should be tested for the following tests as per the USP and Indian Herbal Pharmacopoeia (IHP). The Study includes foreign organic matter, Ethanol soluble extractives, Water soluble extractives, Total ash contents, Acid insoluble ash, Water soluble ash, Loss on drying and Percentage moisture content.

Medicinal plant materials should be entirely free from visible signs of contamination, i.e. moulds, insects and other animal contamination, including animal excreta, fungus and dust. It is seldom possible to obtain marketed plant materials that are entirely free from some form of innocuous foreign matter. However, no poisonous, dangerous or otherwise harmful foreign matter or residue should be allowed. Any soil, stone, sand, dust and other foreign organic matter must be removed before medicinal plant materials are cut or ground for testing. Macroscopic examination can conveniently be employed for determination of foreign matter in whole or specific plant material.

II. METHODS AND MATERIAL

Sampling

Centella asiatica leaves were collected from various places in Avsari Forest, Ambegaon Taluka in bulk, cleaned to remove the dust particles on the surface of the plant material. Leaves were allowed to dry by spreading them on filter papers in shade.

Extractable Matter

This method determines the amount of phytoconstituents extracted with solvents from a given amount of medicinal plant material in the form of powder. Here according to Indian Herbal Pharmacopoeia ethanol and water were used as common solvents to determine the extractable matter.

Procedure

Accurately weighed five grams of leaves powder was placed in glass-stoppered conical flask. To it 100 cm^3 of water was added. The flask was shaken frequently for six hours, and then allowed to stand for eighteen hours. The contents were filtered rapidly to avoid loss of solvent. The filtrate was transferred to a previously weighed clean beaker and evaporated to dryness on a water-bath. After evaporation the extract was dried at 105°C for six hours and kept in desiccators for cooling. The beaker was weighed and percent extractable matter in water was calculated. The above procedure was repeated thrice for determination of water-soluble extractable matter.

Ethanol soluble extractable matter was determined by following the above procedure except ethanol was used instead of water, as extracting solvent. The experiment was repeated for three times.

Ash Content

The ash remaining following ignition of medicinal plant materials is determined by three different methods, which measures

The Total Ash method is designed to measure the total amount of material remaining after ignition. This includes both 'physiological ash', which is derived from the plant tissue itself, and 'non-physiological ash', which is the residue of the extraneous matter (e g sand and soil) adhering to the plant surface.

Acid-Insoluble Ash is the residue obtained after boiling the total ash with dilute hydrochloric acid and igniting the remaining insoluble matter. This measures the amount of silica present as sand and siliceous earth.

Water-Soluble Ash is the difference in weight between the total ash and the residue after treatment of the total ash with water.

Total Ash

The total ash was obtained by taking Accurately weighed 2 g of the dried plant material was taken in a tarred Silica dish and was ignited with a flame of Bunsen burner for about one hour. The ignition was completed by keeping it in a muffle furnace at 550°C ± 20°C till grey ash was formed. It was then cooled in desiccators and weighed. The process was repeated (ignition, cooling and weighing) till the difference in the weight between two successive weighing was less than 1 mg.

Acid Insoluble Ash

Acid Insoluble Ash was obtained by following method.

Procedure

Accurately weighed 2gm of the dried plant material was taken in a porcelain/silica dish and was ignited with a bunsen burner for about one hour. The porcelain dish was kept in a muffle furnace at 550°C ± 20°C till grey ash was obtained. The ash was moistened with concentrated HCI and evaporated to dryness after which it was kept in an electric air oven maintained at 135°C ± 2°C for 3 hr. After cooling, 25 cc. of dilute HCI was added, and was kept covered with watch glass and heated on a water bath for 10 minutes. It was then allowed to cool, and was filtered through Whatmann filter paper No. 41. The residue was then washed with hot water till washings were free from chloride (as tested with $AgNO_3$ solution). The filter paper and the residue were put in a dish and ignited in a muffle furnace at 550°C ± 20°C for one hour. The process of cooling in a desiccators and weighing was repeated till the difference between two successive weights was found to be less than one mg.

Water-Soluble Ash

Water soluble ash was obtained by following method.

Procedure

Twenty-five cm3 of distilled water was added in a silica dish containing the total ash and boiled for ten minutes. The insoluble matter was collected on an ash-less filter paper. The residue was washed with hot water and ignited in a crucible for fifteen minutes at a temperature not exceeding 450°C. The weight of this residue was subtracted from the weight of the total ash and the water-soluble ash was calculated.

Loss on Drying

The percentage of loss on drying was obtained by following method.

Procedure

Five grams of powdered plant sample was weighed in wide mouthed stoppered weighing bottle. The bottle was then placed with lid open in an air oven maintained at 100°C ± 2°C. The sample was kept in an oven for 2 hours. The bottle was then removed, covered and placed in desiccators. The bottle was weighed after cooling to room temperature and weighed. The bottle was again kept in the oven for 2 hrs. and the above procedure was repeated (heating. cooling and weighing) till the difference in the weight between two successive weighing was less than 1 mg. Three readings for each sample were recorded.

Moisture Content

The moisture of plant powder was obtained by Karl-Fischer Titrimetric Method

Procedure

Reaction vessel was rinsed thoroughly with methanol magnetic stirring rotor was inserted in the vessel and placed in proper position. The large rubber cork was removed and some K/F grade methanol was added using funnel, to the reaction vessel just enough to submerge the metal wires of sensors in the reaction vessel. The cork was replaced immediately. The K/F reagent and methanol bottles were placed in position. Then the instrument was turned on and the speed of magnetic stirrer was adjusted. Methanol was neutralized and the titer factor was determined by calibrating the K/F reagent. This was done by adding 10 µl of distilled water with the help of a µL syringe in the reaction vessel and completing the titration. The calibration of the reagent was done in triplicate. The readings were noted and the titer factor was calculated. The data for determination of titer factor is given in following table QC 8 and it was calculated using the following formula.

III. RESULTS AND DISCUSSION

The results of proximate analysis of leaves powder of *Centella asiatica* plant were tested for proximate analysis and it shows presence **0.84%** foreign organic matter, **9.03%** ethanol soluble extractives, **17.20%** water soluble extractives **10.45%** total ash, **15.30%** Acid insoluble ash, **10.45**% loss on drying and **6.10%** moisture content.

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Comparative Study of some Synthetic Reactions with Green Aspects

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ABSTRACT

On the basis of green chemistry, minimum utilization of energy that reduces or eliminates the use or generation of hazardous substances in the design, manufacture and application of chemical products. Different green approach of synthetic reactions like Vilsmeier Haack formylation, Benzoylation, Salicylic acid to Aspirin, Acetanilide to p-Acetamide sulphonyl chloride carried out by sonication. Evidence of product and purity confirmed by thin layer chromatography and melting points.

Keywords : Vilsmeier Haack formylation, green chemistry, Sonication

I. INTRODUCTION

Green Chemistry provides a unique forum for the publication of innovative research on the development of alternative sustainable technologies. The scope of Green Chemistry is based on, but not limited. On the basis of green chemistry definition minimum utilization of energy that reduces or eliminates the use or generation of hazardous substances in the design, manufacture and application of chemical products. Green Chemistry is at the frontiers of this interdisciplinary science and publishes research that attempts to reduce the environmental impact of the chemical enterprise by developing a technology base that is inherently non-toxic to living things and the environment. Submissions on all aspects of research relating to the endeavor are welcome. The journal publishes original and significant progressive research that is likely to be of wide general appeal. To be published, work must present a significant advance in green chemistry. Exposure includes the following aspects as the application of innovative technology to establish industrial procedures. The development of environmentally improved routes, synthetic methods and processes to important products. The design of new, greener and safer chemicals and materials. The use of sustainable resources, the use of biotechnology, alternatives to chemistry-based solutions, methodologies and tools for measuring environmental impact and application to chemical aspects of renewable energy.

II. METHODS AND MATERIAL

Procedure:

Routine Procedures of some reactions:

1.Vilsmeier Haack formylation:

In round bottom flask, take 1.2 ml DMF. Cool it in ice bath, then add into it 1.2 ml $POCl_3$ with constant stirring. Viscous liquid formed then add 1.0 gm 2-methoxy Naphthalene in one lot. Attach air condenser with guard tube. Heat reaction mixture using water bath about 2 hrs. pour reaction mixture in ice cold solution of sodium acetate for decoloration and maintained stirring with glass rod for 15 minutes. Solid white coloured compound is formed. Recrystallize it with ethanol and monitored by M.P and TLC.

Green approach: This reaction carried out at sonicator for 15 mins under sonication. Time effective and fuel saving with less hazardous.

2. Benzoylation:

Dissolve 1gm glycine in 10 ml 10% NaOH containing in conical flask. Add 2ml Benzyol chloride in one portion to the above solution. Put cotton plug on conical flask and shake vigorously for 10 mins. Add crushed ice to this reaction mixture and acidify with conc. HCl. Check with Congo red litmus paper. Filter crude product and wash with cold water.

Green approach: Instead of NaOH preferred the use of LiOH which is less corrosive and hazardous and in sonication reaction time reduced upto 5 mins.

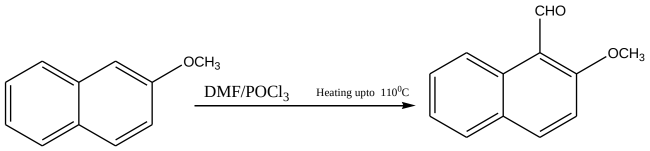
3. Salicylic acid to Aspirin:

Place 3.0 gm of Salicylic acid in 100 ml Erlenmeyer flask. Add 6 ml of acetic anhydride and 5 drops of conc.H_2SO_4. Mix the above mixture and heat the flask in a water bath which is warm up to 80-90⁰C for 10 mins. Remove the conical flask and allow cooling to room temperature. Add 40 ml water and keep it in ice water bath. Wash the product with cold water.
Green approach: Fuel saving and instead of Acetic anhydride we used Maleic anhydride.

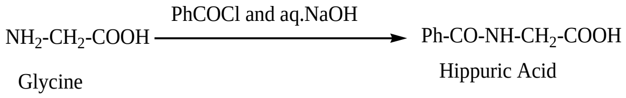
4. Acetanilide to p-Acetamide Sulphonyl Chloride: Take 2 gm of acetanilide in round bottom flask fitted with water condenser and guard tube. Add 5 ml chlorosulphonic acid, then heat the reaction mixture in water bath for 60 mins. After cooling the reaction mixture, pour it in crushed ice. White granular solids are formed. Wash with cold water.

Green approach: Time period of reaction reduced from 60 to 15 mins.

Reaction 1st :

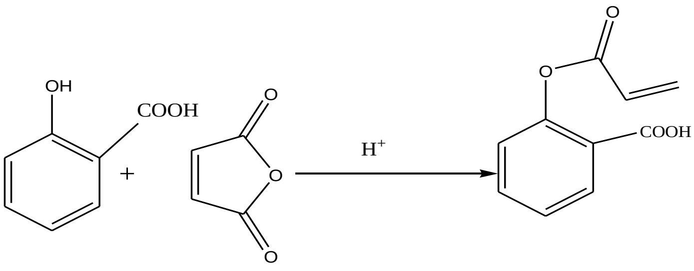


Reaction 2nd :

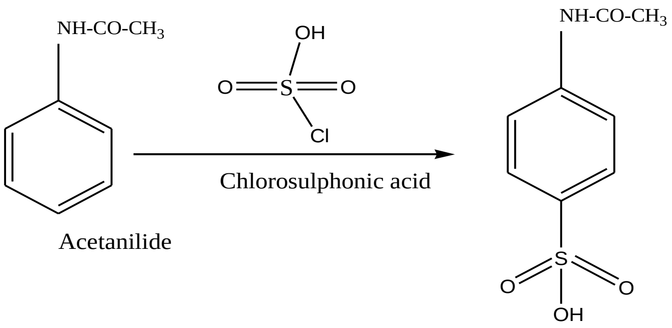


$$\underset{\text{Glycine}}{NH_2\text{-}CH_2\text{-}COOH} \xrightarrow{\text{PhCOCl and aq.NaOH}} \underset{\text{Hippuric Acid}}{Ph\text{-}CO\text{-}NH\text{-}CH_2\text{-}COOH}$$

Reaction 3rd :



Reaction 4th :



III. RESULTS

Sr. No.	**Name of Reaction**	**R_f value of Reactant**	**R_f value of Product**	**Physical Constant (^{0}C)**	**Practical yield of Product (%)**
01	Vilsmeier Haack formylation	0.69	0.51	78-82	92
02	Benzoylation	0.60	0.55	186-188	88
03	Salicylic acid to Aspirin	0.52	0.67	136-138	89
04	Acetanilide to p-Acetamide Sulphonyl Chloride	0.56	0.72	140-142	87

Compound	1	2	3	4
Name of Product	1-formyl-2-methoxy naphthalene	Hippuric acid	Aspirin	p-acetamide sulphonyl chloride
Physical Constant	78-82 ^{0}C	186-188 ^{0}C	136-138 ^{0}C	140-142 ^{0}C
TLC				
Solvent used for TLC	MobilePhase: n-Hexane	MobilePhase: n-Hexane	MobilePhase: n-Hexane + Ethyl acetate (7:3)	MobilePhase: n-Hexane + Ethyl acetate (8:2)

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Hydrochemical Study of Agricultural Ponds Near Sangamner in Ahmednagar District of Maharashtra State India

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ABSTRACT

Hydrochemical study is very necessary to know the water quality of any water resource. Agricultural ponds are very useful for agricultural practices and livestock rearing. Sangamner and nearby area is having less average rain fall. Several farmers are preparing agricultural ponds with government help for agricultural and other agro based practices. These ponds are also useful for the Pisciculture activities. Therefore the present work was undertaken. The water samples were collected from the agricultural ponds from various villages near by Sangamner. Temperature was recorded by using thermometer; pH and TDS were measured using digital pH and TDS meters, dissolved oxygen wad estimated by suing Winkler's method. Acidity, Alkalinity, dissolved Carbon dioxide, electrical conductivity and hardness was measured by using standard methods. The acidity was found in the range of 25 mg/L to 153 mg/L. The alkalinity was found in the range of 140 mg/L to 546 mg/L, dissolved CO_2 was in the range of 10.12 mg/L to 27.72mg/L, dissolved oxygen was in the range of 4.2 to 7.24 mg/L, pH was in the range7.2 to 9.1., TDS was in the range of 130 to 560, Electrical conductivity was in the range of0 .22 to 1.37mS, Hardness was in the range of 82.6 to 99.6mg/L.

Keywords: pH, TDS, Agricultural Ponds

I. INTRODUCTION

Agricultural ponds are very useful as source of water to the farmers. These are guaranteed source of water throughout the year to carry out agricultural practices along with livestock rearing and aquaculture practices.. In Ahmednager district of Maharashtra state several farmers have agricultural ponds with the help of government subsidies. Water is siphoned out from near by river or canals during rotation and sometimes by using water tankers also. This water stored was useful during scarcity of water or whenever it is needed. It is observed that much literature is not available regarding hydrochemistry of such ponds therefore it was need to do the present work. Some researchers have done work on physicochemical parameters in India and in some other countries. Among them Elina et al (2014) has carried out work on pond water of fish seed farm in Bangladesh. Anyanwa and Soloman (2015) have done the work on physicochemical parameters of fish pond. Physicochemical characteristics of Bhamka pond in Rewa district of Madhya Pradesh was assessed by Mishra et al (2013) Evaluation of physicochemical parameters of fish culture pond water at Hoshangabad was done by Sheikh et al (2017)Assessment of seasonal water quality and fish pond conservation was studied by Dinesh Kumar et al (2017). In Nigeria physicochemical parameters of water from artificial concrete fish ponds in Abraka was done by Agbaire et al (2015). Kiran (2010) has also done work on physicochemical characteristics.

II. METHODS AND MATERIAL

Water samples were collected from agricultural pons in Sangamner and Akole talukas of Ahmednagar district. Temperature, pH and TDS were recorded on the spot by using thermometer, digital pH meter and TDS meter respectively. Acidity, alkalinity, hardness, dissolved carbon dioxide were estimated by using methods given by Maiti 2011.Dissol;ved oxygen was fixed in DO bottles and it was estimated by using Winkler's method.

III. RESULTS AND DISCUSSION

Table1

Sr. No.		Rajapur	Ganore	Maldad	Ghulewaqdi	Vadgaonpan
1	Acidity	76.5	72	153	28	25
2	Alkalinity	433	420	140	513	546
3	Dissolved carbon oxide	27.72	14.52	10.12	24.64	22
4	Dissolved oxygen	5.16	4.2	5.11	7.24	6.84
5	pH	7.2	7.5	7.5	8.0	9.1
6	TDS	130	140	200	560	210
7	Electrical conductivity	0.22	1.37	0.33	0.81	0.43
8	Hardness	82.6	104.6	996	322.6	183.2

Acidity : The acidity was found in the range of 25 mg/L to 153 mg/L. At Rajapur it was found to be 76.5 mg/L at Ganore 72 mg/l at Maldad 153 mg/L and at Ghulewadi 28 mg/L and at Vadgaonpan 25 mg/L.

Alkalinity : The alkalinity was found in the range of 140 mg/l to 546 mg/L. It was 433 mg/L at Rajapur , at Ganore 420 mg/L, at Maldad 140 mg/L and at Ghulewadi 513 mg/L and 546 mg/L at Vadgaonpan.

Dissolved CO_2 : It was in thr range of 10.12 mg/L to 27.72mg/l at Rajapur 27.72 mg/L, at Ganorei 14.52 mg/l, at Maldad 10.12 mg/L and at Ghulewadi 24.64 mg/L and 22 mg/L at Vadgaonpan.

Dissolved Oxygen : It was in the range of 4.2 to 7.24 mg/L at Rajapur 5.16 mg/L, at Ganore 4.2 mg/L, at Maldad 5.11 mg/L and at Ghulewadi 7.24 mg/L and 6.84 mg/L at Vadgaonpan.

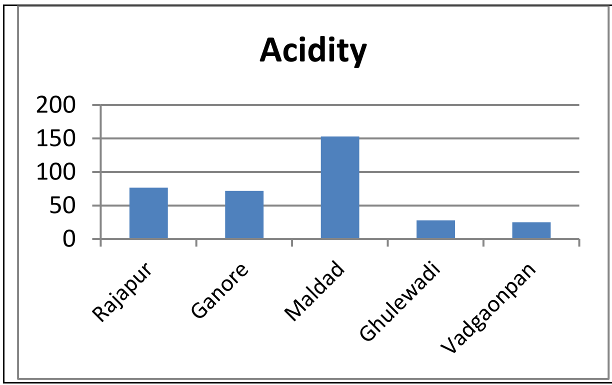
pH: It was in the range7.2 to 9.1.At Rajapur 7.2 , at Ganore 7.5, at Maldad 7.5 and at Ghulewadi8.0 and 9.1 at Vadgaonpan.

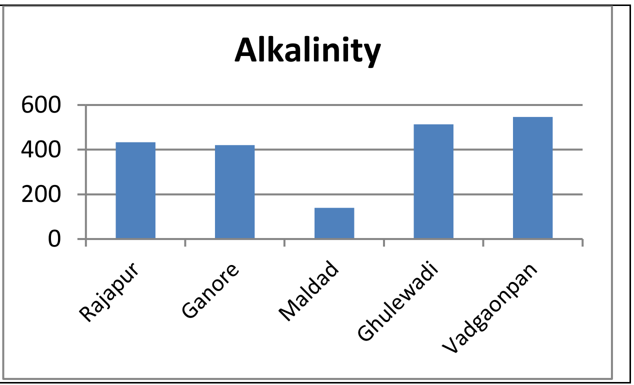
TDS: It was in the range of 130 to 560 ppm.It was 130 at Rajapur , at Ganore 140 ppm, at Maldad 200 ppm and at Ghulewadi 560 and 210 ppm at Vadgaonpan.

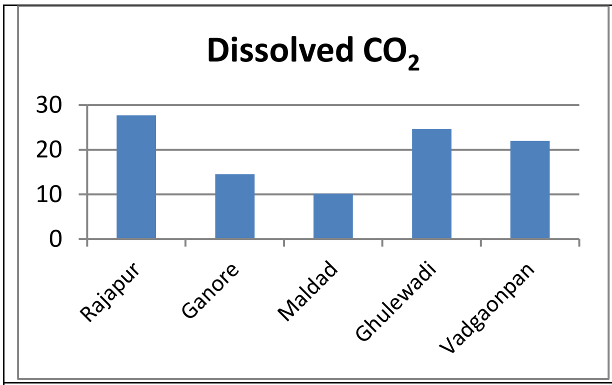
Electrical conductivity: It was in the range of 0.22mS to 1.37mS. It was 0 .22 mS Rajapur, at Ganore1.37mS, at Maldad 0 .33mS and at Ghulewadi.81mS and 0 .43mS at Vadgaonpan.

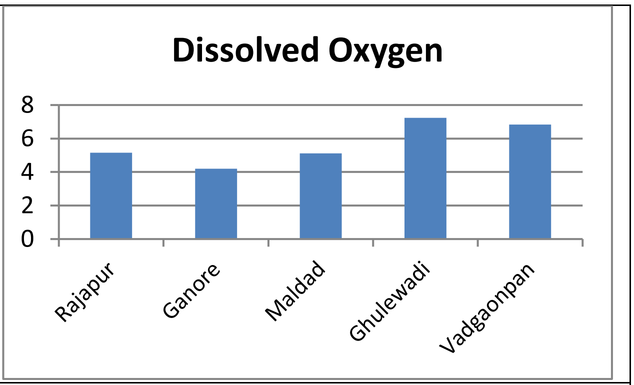
Hardness : It was in the range of 82.6 to 996. It was 82.6_ at Rajapur , at Ganore104.2, at Maldad 99.6 and at Ghulewadi 322.6 and 183.2 at Vadgaonpan.

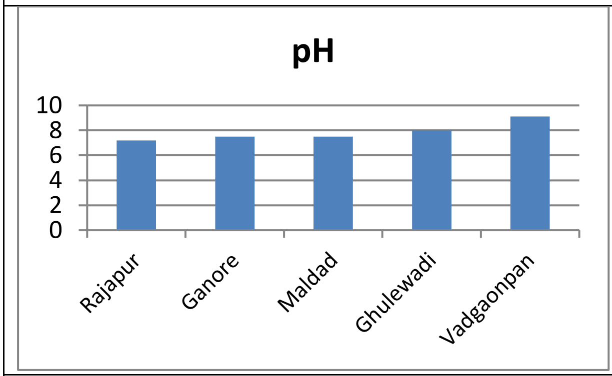
Acidity was minimum at Vadgaonpan and maximum at Maldad. Alkalinity was minimum at Maldad and maximum at Vadgaonpan. Dissolved Carbon di oxide was minimum at Maldad and maximum at Ghulewadi. Dissolved Oxygen was minimum at Ganore and maximum at Ghulewadi. pH was minimum at Rajapur and maximum at Vadgaonpan. TDS was minimum at rajapur and maximum at Ghulewadi. Electrical conductivity was minimum at Rahapur and maximum at Ganore. Hardness was minimum at Rajapur and maximum at Maldad. It is observed that there is variation in the values of Hydrochemical parameters of different agricultural ponds. It is may due to the original source from where water was broght are different.

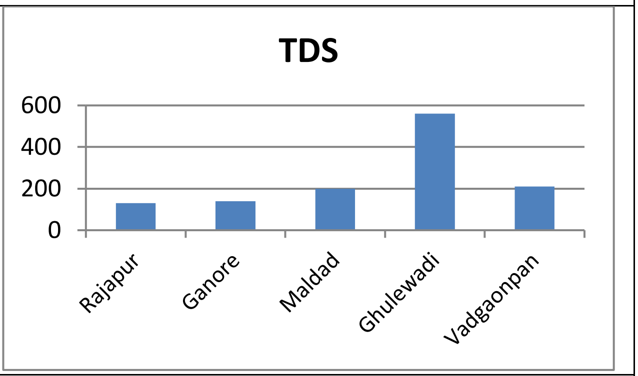


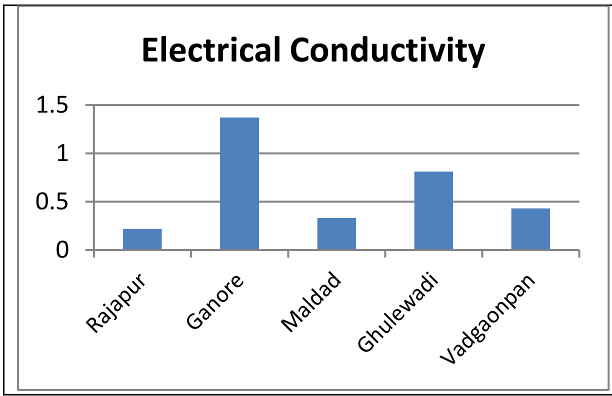


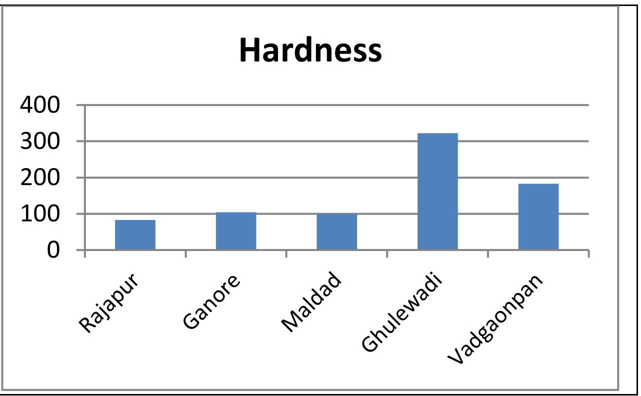












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An Empirical Study on Predicting the Wind Speed after Landfall of Tropical Cyclones Over Bay of Bengal

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ABSTRACT

The post landfall wind speed forecast of tropical cyclones (TC) over Bay of Bengal (BoB) are explained by using an empirical study. The study parameters are obtained from the database of 19 tropical cyclone of 1988-2017. The study is based upon the assumption of tropical cyclone wind speed decay after landfall. A method for correcting the forecast during subsequent observation hours is also presented. The method for predicting the cyclone wind speed is tribute from the most recent study over the Indian and USA coast. Results show that without the correction factor the absolute mean error (MAE) ranges from 10.1 To 5.7 Kt, root mean square error (Rmse) ranges from 12.5 to 7.7 Kt and mean absolute percent error (MAPE) ranges from 30.0 to 10.1 Kt with these parameters decreasing over time for wind speed. A significant improvement in the forecast skill is observed with including the correction procedure. For the operational forecaster, this model will be helpful and important.

Keywords: Empirical Study, Prediction, TC, Wind Speed, BoB

I. INTRODUCTION

Acute rainfall, drastic winds, overland flooding and storm surges due to tropical cyclones cause heavy losses of things and the tropical cyclones are known as the most annihilating hazards on earth [1]. The origins and landfalls of the cyclones are responsible for the effect and destruction [2]. Bangladesh, India and Myanmar face more than 75% of global casualties while only 5% of global tropical cyclones originate in Bay of Bengal [3-5]. Only 1% of all annual tropical cyclones are affected in Bangladesh and that responsible for 50% of all global casualties [6]. Tropical cyclones associated with high winds and huge water levels make drastic property damage and loss of life all over the world, but particularly in this densely populated coastal region of Bay of Bengal [7,8]. Several significant tropical cyclones that happened in 2007 and 2009 provide recent examples of devastating storm-surge and wind speed in Bangladesh as well as killed 4234 and 190 respectively [9,10]. Ineffective detection of cyclones landfall and inaccuracy of wind intensity are responsible for these high casualties [11]. These recent great cyclones have illustrated the need for the accurate prediction of inland effects of tropical cyclones.

Some important factors such as water availability, soil moisture and shape of the coastline have impact on the post-landfall intensity of tropical cyclones [12-15]. The decay of tropical cyclone wind speed is critically linked to the low moisture and heat fluxes from the surface [12,16] and lack of moisture and heat fluxes make the wind speed weaken rapidly than after landfall [16,17]. Considering all of the factors, it is important to correctly determine the wind speed and decay rate of tropical cyclone after landfall. From the last few decades, the prediction of cyclone track has improved very much and very widely in the field of forecast modelling and data gathering research but the prediction of tropical cyclone wind speed has not developed much [18].

The numerical or empirical models has significant socio-economic importance which can accurately predict the wind speed after landfall [19]. Only numerical and empirical model can give the most accurate and easy used because it is not possible to use statistical analysis of the highest annual wind speeds at a particular site in order to determine the extreme wind speeds in cyclone-prone regions [20]. Therefore, measuring the post-landfall wind speed decay of tropical cyclone using observed data can be helpful to develop the wind speed decay model to enhance the skill of intensity prediction. There have some continuous attempts to develop and improve the models for prediction of post-landfall intensity over the US coast [13,21]. Besides, Bhowmik et al. [22] had worked about the intensity of wind speed decay over the Indian Ocean and Bay of Bengal. However, there are a few attempts taken to analyze the nature of post-landfall cyclone intensity over the Indian subcontinent [22] but these regions are the most densely populated and also vulnerable to tropical cyclone. Real time operational applications, this model can be useful for stochastic simulation of cyclone related risk in coastal areas, which helps in designing building and structures that can withstand the impact of land falling cyclones [23].

The Kaplan and DeMaria [17] wind speed decay model has been chosen and used to complete the empirical modelling. This study is also conferred on Bhowmik et al. [22], where they made an empirical model of tropical cyclone wind speed decay after landfall from 1981 to 2000. The current study is a little bit different from them, because the cyclones' data are selected from 1988 to 2017. The main objective of this study is to apply the empirical model for forecasting of post-landfall wind speed according to the forecast hypothesis of Kaplan and DeMaria [17], Vickery and Twisdale [24], Bhowmik et al. [22] which can be helpful for reducing the death tolls or property damage due to cyclone.

II. METHODS AND MATERIAL

Data Sources and Sampling

For this present study, tropical cyclones to be those formed over the Bay of Bengal (BoB) during the period of 1988–2017 were considered. The data period includes 31 cases of landfalling tropical cyclones, but most of them lost their intensity of depression (wind speeds less than 17 Kt and sea level pressure less than 200 hPa) immediately after landfall (within 1–2 hrs.). Tropical cyclones best track records were obtained from the Joint Typhoon Warning Center (JTWC), NASA Earth Observatory and Bhowmik et al. [22].

In order to develop the empirical technique, we used only those 19 tropical cyclones (Table 1) that maintained the minimum intensity of depression (wind speeds of more than 17 Kt) and sustained more than 6 hours after landfall. The tracks of these tropical cyclones are depicted in Figure 1. In Bangladesh, surface wind observations are based upon 3-min-averaged winds. In this study, the landfall intensity was considered as the Maximum Sustained Surface Wind (MSSW) associated with a cyclone at the time of tropical cyclone crosses the coastline. As per the convention of the Bangladesh Meteorological Department, the classification of tropical disturbances is as follows: low: wind speeds less than 17 Kt; depression: wind speeds of 17–33 Kt; cyclonic storm: wind speeds of 34–47 Kt; severe cyclonic storm: wind speeds of 48–63 Kt; very severe cyclonic storm: wind speeds of 64–119 Kt; and super cyclone: wind speeds above 119 Kt. The data period (1988–2017) includes three cases of a super cyclone and 10 cases of very severe cyclonic storms. It is observed that all 19 cases of intense tropical cyclones (wind speed more than 64 Kt) maintained the minimum intensity of depression for more than 6 h after landfall.

Table 1 : Database of 19 tropical cyclones from 1988 to 2017.

Sl. No.	Cyclone Name/Year	Wind Speed (Kt)					
		0 h	6 h	12 h	18 h	24 h	30 h
1	NARGIS	115	85	70	50	40	30
2	SIDR	130	100	60	50	35	25
3	MALA	105	90	65	55	40	32
4	1997	115	100	90	65	50	30
5	1995	90	85	45	30	30	30
6	1994	125	110	80	50	30	20
7	1991	135	110	85	60	40	25
8	1988	115	65	35	30	16	15
9	1988	110	90	60	45	31	20
10	MORA	60	35	28	25	15	15
11	ROANU	60	55	55	40	27	27
12	MAHASHEN	45	40	35	30	20	20
13	BIJLI	50	45	45	40	40	25
14	AILA	65	55	55	40	30	15
15	2004	65	60	55	30	30	30
16	2000	35	30	27	25	25	25
17	1990	45	35	35	30	20	20
18	1998	70	60	40	25	25	25
19	1997	65	55	35	25	25	25

Figure 1: Map of the 19 tropical cyclones over Bay of Bengal *(Source: Google Earth, 2018 and JTWC, 2018)*

In order to illustrate the effect of landfall intensity on the decay rate of winds and the increase rate of sea level pressure, the 19 tropical cyclones considered in this study are placed into one of two stratifications, namely, MSSW > 70 Kt as "Category A" and MSSW ≤ 70 Kt as "Category B". This stratification is used to divide the 19 tropical cyclones into two groups and that groups used for wind decay prediction. It may be seen from Table 2 that for the major cyclones (Category A) the wind decay rate, during early hours after landfall, is significantly higher compared to weak cyclones (Category B).

Table 2: Six-hourly decay rate of MSSW (Kt) for mean wind speed.

Hours	0 - 6	6 - 12	12 - 18	18 - 24	24 - 30
A (Kt)	23	27	17	14	9
B (Kt)	9	6	10	5	3

(Note: Decay Rate: Difference between the mean value of two-time variation for wind speed. e.g. Difference between 0 hour's average wind speed and 6 hours' average wind speed).

Empirical Formula for Tropical Cyclone Wind Speed

The assumption that tropical cyclones wind speed decay at a rate that is proportional to their landfall intensity is the basic of the empirical inland wind decay model (IWDM) and can be expressed by the following differential equation:

$dV/dt = -\alpha t$……………………………..………..(1)

Where V (Kt) is the MSSW, α is the decay constant (h^{-1}), and t (h) is the time after landfall. The differential solution to equation (1) is given by,

$V(t) = V_0 e^{-\alpha t}$ ………………..……………………....(2)

Where V_0 is the MSSW at t = 0.

As shown in Table 2, the MSSW decays to some background wind speed V_b. This effect can be included by adding an extra term to equation (1) to give,

$dV/dt = -\alpha (V - V_b)$……………………..………….…(3)

Which has a solution given by Bhowmik *et al.* [22]; Kaplan and DeMaria [17],
$V_t = V_b + (V_0 - V_b)\, e^{-\alpha t}$……………………..…..…..…(4)

The observational results of Myers [25], Schwerdt *et al.* [26], Powell [27,28], Ho *et al.* [29] and Fung *et al.* [30] indicate that hurricane winds decrease abruptly as the landfalling storm crosses the coastline. Fung *et al.* [30] noted that this rapid decrease in wind speed occurs within a few kilometers of the coastline as onshore winds quickly adjust to the increased roughness of the underlying land surface. Tuleya [12] noted that the primary mechanism responsible for the rapid decay of TCs after landfall is the largely reduced latent heat and sensible fluxes. They introduced a Reduction Factor (R) for model validation. Bhowmik *et al.* [22] determined decay constant from 0.095 to 0.354 and the reduction factors (R) from 0.079 to 0.620 where, this study is introducing a reduction factor (R) for the actual forecast results.
Consequently, V_0 in equation (4) is multiplied by a reduction factor (R). The optimal value of R was 0.9 [17,31].

From equation (4), the decay constant "α" can be written as
$\alpha = \{\ln [(V_0 - V_b) / (V_t - V_b)]\} / t$…………………....(5)

From equation (5), the decay constant "α_1" for the first 6 h after landfall (t = 0 – 6 h) are written as,
$\alpha_1 = \{\ln [(V_0 - V_b) / (V_6 - V_b)]\} / 6$...……..…..…..…....(6)

The decay constant "α_2" for the remaining 12 h (for t = 6 – 18 h) is taken as,
$\alpha_2 = \{\ln [(V_6 - V_b) / (V_{18} - V_b)]\} / 12$...…… …......(7)

It is presumed that for the first 6 h the decay constant is α_1 and, thereafter it remains as α_2. The corresponding 6-hourly "current reduction factors" are defined as

$R_1 = e^{-\alpha_1 * 6.0}$……………………….…………..……..…...(8)
$R_2 = e^{-\alpha_2 * 6.0}$…………………………………..……..…….(9)

Kaplan and DeMaria [17] introduced a correction factor as a function of inland distance to take into account the effect of the tropical cyclone's proximity to water on the rate of decay after landfall. This effect was first discussed by Malkin [32] and was confirmed in the study of Kaplan and DeMaria [17]. Kaplan and DeMaria [17] investigated a reduction factor (R) in their forecast model which have a great similarity to this study.

Now, from Equation (4), the decay equation for 6-hourly forecasts (Appendix-IV and V) is written as,

$V_{t+6} = V_b + (V_t - V_b) R_1$ for t = 0
$= V_b + (V_t - V_b) R_2$ for t = 6, 12, 18 & 24...(10)

The decay coefficients (α_1, α_2, R_1, R_2, and V_b) computed from the decay rate of wind speed (Table 2) using equation (6) – (9), taking the mean intensity as a function of time after landfall, are shown in Table 3. Once V_0, the landfall intensity is known, 6-hourly forecasts valid up to 30 h can be made using parameters (R_1, R_2, V_b) from Table 4 in Equation (10).

Table 3: Wind speed decay parameters from the decay rate of table 2.

MSSW (Kt)	α_1 (h^{-1})	R_1 $(6\ h)^{-1}$	α_2 (h^{-1})	R_2 $(6\ h)^{-1}$	V_b (Kt)
A	0.049	0.747	0.090	0.582	25
B	0.053	0.727	0.092	0.577	23

III. RESULTS AND DISCUSSION

Mean Wind Speed Decay Curve

The mean wind speed rate for the all 19 cyclones are decreasing within a time interval and at the last time the wind speed dissipated (Figure 2). From the landfall of a cyclone the wind speed is constantly decreased by the influence of various parameters like temperature, sea level pressure and wind flux. From the historical cyclone event it's easy to seem that the wind speed decreasing phenomena is actual and constant.

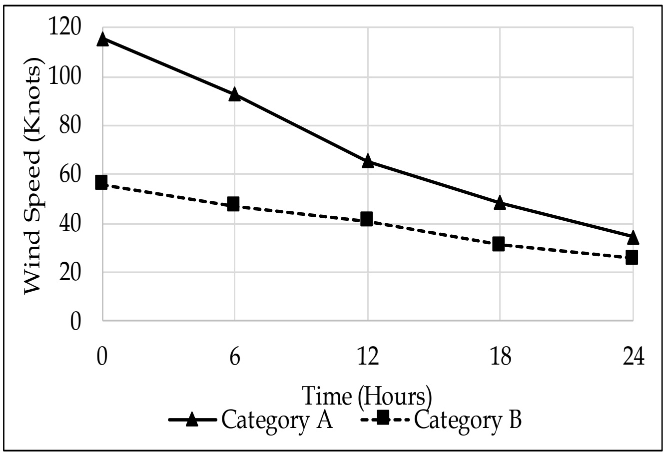

Figure 2: The mean wind speed decay curves for category A (dotted line) and for category B (solid line) cyclones.

The wind speed of Category A cyclones has a strong and significant trend for decreasing. Bhowmik *et al.* [22] proved that the wind speed >65 knots cyclones had a strong trend to dissipate sooner than the ≤ 65 knots wind speeds cyclone. Besides, Kaplan and DeMaria [17] also agreed to this decrease trend where this study shown the actual decreasing system with time interval. Actual decay system of wind speed for tropical cyclone had a great difficulty to be explained. Therefore, some analytical methods were followed and also inputting some important factors for describing the actual wind speed decay.

Individual Cyclone Wind Decay Parameters

The present study of 19 cyclones post landfall wind decay rate had no significant regional variation. In order to examine how the decay constant and current reduction factors change from cyclone to cyclone, decay constants (α_1, α_2) and current reduction factors (R_1, R_2) computed for each of the 19 cyclones are presented in Table 4. These coefficients for the individual cyclone are computed, taking V_b as the lowest intensity reached by each of them by using the equation 6 and 7.

Table 4: Wind decay parameters for individual 19 cyclones over Bay of Bengal.

Sl. No.	α_1 (h^{-1})	α_2 (h^{-1})	R_1 $(6h)^{-1}$	R_2 $(6h)^{-1}$
1	0.068	0.073	0.667	0.645
2	0.056	0.092	0.714	0.577
3	0.035	0.064	0.813	0.679
4	0.030	0.052	0.833	0.730
5	0.013	0.207	0.923	0.289
6	0.027	0.102	0.850	0.542
7	0.043	0.074	0.773	0.642
8	0.135	0.173	0.444	0.354
9	0.045	0.098	0.765	0.555
10	0.188	0.149	0.324	0.408
11	0.024	0.053	0.865	0.729
12	0.043	0.074	0.773	0.642
13	0.034	0.021	0.815	0.879
14	0.045	0.053	0.762	0.729
15	0.021	0.139	0.881	0.435
16	0.090	0.104	0.583	0.535
17	0.101	0.045	0.545	0.764
18	0.040	0.243	0.787	0.232
19	0.045	0.231	0.762	0.250

Figure 3 shows a scattered diagram that explains a regression equation relating R_1 and R_2, as given as $R_2 = 0.74R_1$.

In Bangladesh, in the case of a cyclone situation, warnings/ forecasts issued by the Bangladesh Meteorological Department are updated at 3–6-h intervals, based on the latest available synoptic observations. In such a case, additional synoptic observations are taken at hourly intervals for the likely affected coastal stations, until the cyclone weakens into a low-pressure area. Thus, in the Bangladesh scenario, the first forecast (valid up to 24–30 h) issued at the time of landfall can be corrected and updated during the subsequent observation hours, taking into account the trend of the decay rate. Because a dense population resides at or near the Bay of Bengal coasts, this update forecast has direct relevance to daily activities over a coastal zone (such as transportation, tourism, fishing, sports, etc.) apart from disaster management.

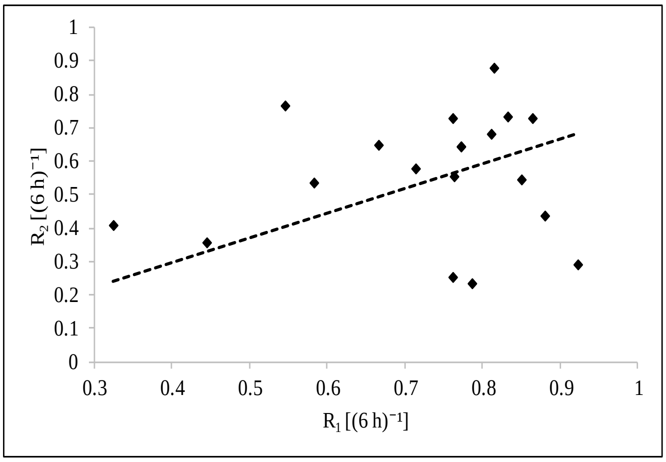

Figure 3 : The scatter diagram relating R_1 and R_2 [Current reduction factors at t = 6 h and for t = 12 h, respectively; Units: $(6\ h)^{-1}$].

Six Hourly Wind Speed Forecast

In order to verify the method, we apply the technique for the development database of 19 cyclones. Results of 6-hourly forecasts after landfall for the 19 cyclones, both with and without the correction procedure based on subsequent observations, are shown in Table 5. The table shows that there is generally good agreement between the predicted and observed values when the correction procedure is included. Without correction procedures there have no agreement among the results. From 6 hours to 30 hours, almost the cyclones have agreed to their observed and predicted values.

Table 5: Comparison between observed, without correction and with correction procedures of six-hourly wind speed (Kt).

Sl. No.	6 h	12 h	18 h	24 h	30 h
1	85	70	50	40	30[a]
	92	64	48	38	33[b]
	–	70	56	47	41[c]
2	100	60	50	35	25[a]
	103	71	52	40	34[b]
	–	70	51	40	34[c]
3	90	65	55	40	32[a]
	85	60	45	37	32[b]
	–	68	56	49	43[c]
4	100	90	65	50	30[a]
	92	64	48	38	33[b]
	–	75	63	54	48[c]
5	85	45	30	30	30[a]
	74	53	41	35	31[b]
	–	43	34	31	30[c]
6	110	80	50	30	20[a]
	100	68	50	40	34[b]
	–	63	43	33	27[c]
7	110	85	60	40	25[a]
	107	73	53	41	34[b]
	–	78	59	47	39[c]
8	65	35	30	16	15[a]
	92	64	48	38	33[b]
	–	42	25	18	16[c]
9	90	60	45	31	20[a]
	89	62	46	37	32[b]
	–	58	41	32	26[c]
10	35	28	25	15	15[a]
	50	39	32	28	26[b]
	–	29	21	17	16[c]
11	55	55	40	27	27[a]
	50	39	32	28	26[b]
	–	44	39	36	33[c]
12	40	35	30	20	20[a]
	39	32	28	26	25[b]
	–	32	28	25	23[c]
13	45	45	40	40	25[a]
	43	34	30	27	25[b]
	–	41	39	37	36[c]
14	55	55	40	30	15[a]
	54	41	33	29	26[b]
	–	43	35	30	26[c]
15	60	55	30	30	30[a]
	54	41	33	29	26[b]
	–	40	34	32	31[c]
16	30	27	25	25	25[a]
	32	28	26	25	24[b]
	–	29	27	26	26[c]
17	35	35	30	20	20[a]

Sl. No.	6 h	12 h	18 h	24 h	30 h
	39	32	28	26	25[b]
	-	35	31	28	26[c]
18	60	40	25	25	25[a]
	57	43	34	30	27[b]
	-	32	27	25	25[c]
19	55	35	25	25	25[a]
	54	41	33	29	26[b]
	-	32	27	25	25[c]

Note: a = Observed Wind Speed; b = Wind Speed without Correction Procedures; c = Wind Speed with Correction Procedures.

Comparing the forecast data with Kaplan and DeMaria [17], have made an agreement that can be effective to this study. The observed data set and prediction of this study is strongly similar to the hypothesis of Kaplan and DeMaria [17]. From the methods, model implementation and results of that hypothesis is quietly agreed to this study. They worked out a huge dataset and this study is implemented a limited dataset but the prediction rate is more than 80% accurate on depending the methods of Kaplan and DeMaria [17].

Bhowmik *et al.* [22] implemented a study about the wind decay model after landfall and the predicted results of that study is quite consistent to this present forecast of wind speed. From the above table observation, a decision can be made that the prediction of wind speed is not void or invalid. The comparison between observed wind speed data with the corrective and non-corrective data have shown a significand variation. The comparison is made for understanding the variation among these three-dimensional observation and analysis. Without correction procedure the data show that huge similarity to the observed data where with correction procedure have a different magnitude. Mainly, the purpose of this comparison is to determine the actual and better forecast data than the observational wind speed data.

Skill Score for Wind Speed

Table 6 shows the error statistics for the model with and without the use of the correction procedure. For the case without the use of the correction procedure, Mean Absolute Error (MAE) ranges from 10.1 to 5.7 Kt, the Root-Mean Square Error (Rmse) from 12.5 to 7.7 Kt and Mean Absolute Percent Error (MAPE) from 30.0 to 10.1 Kt, all of the errors decreasing with time. When the correction procedure to the forecast from the use of the current observations is applied, MAE becomes 6.4 to 2.8 Kt, Rmse is from 8.2 to 3.3 Kt and MAPE is from 26.1 to 8.1 Kt. Kaplan and DeMaria [17,21] obtained MAE of 6.5 and 8.8 Kt and Rmse of 8.8 and 11.4 Kt, respectively, for the southern (south of 37°E latitude) and northern latitudes over the United States. With the incorporation of the correction procedure, a significant improvement in the wind forecast skill is noticed for the case in which it is tested using the dependent sample in the present study.

Table 6: Skill scores (MAE, Rmse and MAPE; Kt) of 6-hourly wind forecast made for the 19 cyclones.

Skill	6 h	12 h	18 h	24 h	30 h
MAE (without correction)	6.2	10.1	6.6	6.1	5.7
MAE (with correction)	-	6.4	2.8	3.6	6.1
Rmse (without correction)	8.7	12.5	8.4	8.2	7.7
Rmse (with correction)	-	8.2	3.3	4.6	8.0
MAPE (without correction)	10.1	20.1	18.5	25.8	30.0
MAPE (with correction)	-	11.7	8.1	12.9	26.1

Procedures for Application of the Methods

In order to apply this method in operational forecasting and correct the forecast at 6-h intervals, the following steps are suggested:

1. At the time of landfall (at t = 0), employ the observed landfall intensity V_0 and climatological values of R_1, R_2, and V_b, which are obtained based upon the sample average decay rates (Table 3), to make 6-hourly predictions of V_t using Equation (10).
2. Six hours after landfall (at t = 6), use the observed V_0, V_6, and climatological V_b to compute the actual R_1 from Equations (6) and (8). Then, get the new R_2 from Equation (9) and use Equation (10) to revise the forecast for 12 h after landfall and later.
3. Twelve hours after landfall (at t = 12), employ the observed V_{12} to make a 6-hourly prediction using Equation (10).
4. Eighteen hours after landfall, employ the observed values of V_0 and V_{18} to calculate the actual R_2 from Equations (7) and (9) and revise the forecast for 24 h and beyond using Equation (10).
5. Twenty-four hours after landfall, use the observed V_{24} to make a final forecast for V_{30}.
6. The climatological background wind speed (V_b) from Table 3 is considered for these computations.

IV. CONCLUSION

The burning topic of the present era is considered as the inland wind speed of any cyclone to the disaster or hurricane community. This present study explains a formulation for predicting six-hourly maximum sustained wind speed that is valid up to 30 hours after landfalling tropical cyclone by using the decay equation of Kaplan and DeMaria [17] and also followed the hypothesis of Bhowmik et al. [22] for supplementary documents, data etc. A modified correction procedure (current reduction factors) is introduced to update the first forecast (valid up to 24–30 h, issued at the time of landfall) at 6- hours intervals taking into account the trend of the decay constant and increase constant as well as the use of current observations. With applying the correction procedure, a significant improvement in the forecast skill is noticed. Results of skill score are well comparable with the results obtained by Kaplan and DeMaria [17,31] and Bhowmik et al. [22]. The method appears to be very important and promising for operational application in Bangladesh scenario in which a dense population lives in most coastal areas. For reducing the damage of properties and deaths, this model would help to forecast accurate wind speed and sea level pressure. A few limitations were raised when preparing this model and that's why some important features such as, cyclone trend plotting, dynamic observation and actual live prediction was avoided. But a further study is required to implement these kinds of features. Applying similar technique, another model will be developed for the coast of Bay of Bengal with a huge database and other features in my Ph.D. or other research.

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Role of Judiciary in Protection of Environment in India

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ABSTRACT

Protection and preservation of the environment has become integral to the cultural and religious ethos of most of the human communities. Nature has been venerated by ancient Hindus, Greeks, native Americans and other religions around the world. They worship all forms of nature believing that it has the spirit of God. According to Hinduism Earth is our mother and we are all her children. So the concept of environment protection and preservation is not modern. It can be seen from the period of Vedas. It is the duty of every individual in our society to protect nature and the term nature includes land, water, trees and animals which are of great significance to us.

Keywords: Spirit of God, Hinduism Earth, Industrialization, Environment Protection.

I. INTRODUCTION

Industrial development is considered to be essential for economic growth and development. It is a common practice that the process of industrialization is based on maximum use of natural resources. However, the process of industrialization exploits the natural resources and violates the ecological equilibrium which results in issues like global warming, climate change, acid rain etc. Indian legislature has made a number of legislation which proves failure and disappointment and passing new bills consistently is just old wine in new bottle. In recent years' higher judiciary plays an important role in monitoring the implementation of measures for pollution control, conservation of forest and wildlife protection.

Meaning of Environment

According to the Webster Dictionary Environment is, 'Aggregate of all external condition and influences affecting the life and development of an organism.'

According to Justice P.N Bhagwati, the term Environment refers to all condition within and around an organism which affect the behavior, growth and development or life processes directly or indirectly. It includes the conditions with which the organisms interact.

The Environment (protection)Act 1986 Section 2(a) environment includes water, air and land and the inter relationship which exists among and between water, air and land and human beings, other living creature, plants, microorganisms and property

Constitutional Aspect on Environmental Law

Initially Constitution of India has no direct provision for environmental protection. global consciousness for the protection of environment in the Seventies, Stockholm Conference and increasing awareness of the environmental crisis prompted the Indian government to enact 42nd amendment to the Constitution in 1946. The 42nd amendment added Article 48(A)to the Directive Principles of State Policies and Art.51(A)(g) to the fundamental duties.

Article 48-A of the constitution says that "the State shall endeavor to protect and improve the environment and to safeguard the forest and wildlife of the country. Article 51-A(g), says that "it shall be duty of every citizen of India to protect and improve the natural environment including forests, lakes, rivers and wildlife and to have compassion for living creature."

In Sachidanand Pandey vs State of West Bengal, the Supreme Court held that "Whenever a problem of ecology is brought before the court, the court is bound to bear in mind Article 48(a) and Article 51-(A)(g)

Remarkable Principles and Doctrines Propounded by Indian Judiciary

In the case of **Subash Kumar vs State of Bihar**, the Supreme Court of India recognized water and air are inalienable part of life under Article-21 of the constitution of India. This was the first time of the constitutional interpretation for protection of healthy environment for life

1. Doctrine of Absolute Liability

In **M.C Mehta vs Union of India** the principle of absolute liability was developed by the supreme court," Where an industrial unit is engaged in a hazardous or naturally unsafe activities and harm results to anyone on account of an accident in the operation of such hazardous or naturally unsafe activities resulting. for example, in escape of toxic gas the industrial unit is strictly an absolutely liable to compensate to all those who are affected by the accident.

2. Polluter Pays Principle

The principle of polluter pay was propounded by the Supreme Court of India in the following cases

a. **Indian Council for Enviro-legal Action Vs Union of India** the Court held that, 'once the activity carried on is hazardous or inherently dangerous, the person carrying on such activity is liable to make good the laws caused to any other person by his activity irrespective of the fact whether he took reasonable care while carrying on his activity.

b. **Vellore Citizen's Welfare Forum Vs Union of India**, the Court interpreted the meaning of polluter pay principle as the absolute liability for harm to the environment extends not only to compensate the victims of the pollution but also the cost resorting the environmental degradation.

c. **M.C Mehta vs Union of India (the oleum gas leak case)** the court held that the enterprise engaged in hazardous or dangerous or dangerous industry which poses a potential threat to health and safety of person working in the factory and those residing in the surrounding areas is absolutely liable to compensate for such harm and irrespective of all reasonable care taken on his account

3. Precautionary Principle

In **A.P Control Board Vs M.P Nayudu and Others**, the Court relied upon precautionary principle and explained that the environmental harm and taking measures to avoid it or to choose least environmentally harmful activity

4. Public Trust Doctrine

a. In **M.C Mehta vs Kamal Nath**, an attempt was made to divert flow of a river for augmentation facilities at a motel. It was held that State and it instrumentalities as trustees have a duty to protect and preserve natural resources

b. **M. I. Builders Pvt. Ltd vs Radhey Shyam Sahu** the Court asks a city development authority to dismantle an underground market build beneath garden of historical importance

5. Public nuisance

a. In **Ratlam Municipal Corporation Vs Vardhichand** the Supreme Court made the use of dormant provisions of Code of Criminal Procedure 1973 as an instrument for resolving pollution problems and for the enforcement of statutory duty

6. **Doctrine of Sustainable Development**

a. **In Vellore Citizens Welfare Forum,** the Supreme Court observe that sustainable development has come to be accepted as a viable concept to eradicate poverty and improve the quality of human life while living within the carrying capacity of supporting eco-system.

b. **Rural Litigation and Entitlement Kendra VS State of UP**, the court held that environment is permanent asset of mankind and not intended to be exhausted in one generations.

II. CONCLUSION

The Indian judiciary has made several attempts to resolve the conflict between the development and environment .The enviromental juriprudance In India develop through public intrest litigation .The Supreme Court has not only played a living role in the implementation of environmental laws but also interpreted right to life under Article-21 to include right to healty and pollution free environment as fundamental right .Indian judiciary plays remarkble role of protecting and preservating the environment through its various ladmark judgement .Environment is the gift of nature and it is duty of every person to conserve the beauty of environment .So environment protection is really a need of hours It is needed to understand the message of Rabindra Nath Tagore's words

> **"GIVE US BACK THE FOREST CULTURE AND TAKE BACK THE MODERN CIVILIZATION"**

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Role of Law In Protection and Promotion of Environment in India : A Preliminary Study

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ABSTRACT

All human beings are entitled for healthy and pollution free environment. every nation have take efforts through various legislations for recognition of this right. The first step towards protection and promotion of environment started with the Stockholm Conference on Human Environment in 1972.This conference consider as Magnacarta for all Environmental legislation in India. The Constitution of India also includes provisions for protection of environment. It cast Duties on both individual and state to protect environment and also to take efforts for promotion of environment. The Indian judiciary also played important role in environmental protection through its various judgments. The new principles were laid down by the judiciary while interpreting the provisions of the constitution, such as Polluter pay principle, Precautionary Principle, Environmental Impact Assessment and so on. With the growth of Industrial development this principle plays vital role in protection of Environment. In India various legislations was made to protect and to promote environment This paper deals with study of various environmental protection legislations and various policies such as environmental laws related with protection of environment, role of Indian judiciary in protection and promotion of environmental laws.

Keywords: Magnacarta, Environmental legislation, Environment Protection

I. INTRODUCTION

LEGAL CONCEPT OF ENVIRONMENT:-

The study of environmental legislation starts from the important question that is what is Environment .The concept of environment is very wide concept and difficult to define. In its general meaning it relates to the surrounding. Under the Environment Protection Act 1986 the concept of environment was defined. This definition is inclusive in nature. Section 2(a) of the act .According to this act Environment" includes water, air and land and the interrelationship that exists among and between water, air and human beings, other living creatures, plants, micro-organism and property".

Generally environment laws deals with the environment their protection, promotion, and inter relationship with human being with other. This legislation cast duty on human being to maintain this relationship with environment at proper level by balancing both Economic and Ecological development. All this refers to the principal of sustainable development and key element in protection and promotion of environment.

LEGISLATIONS IN INDIA

1) The Water(prevention and control of pollution)Act 1974
2) The Air(prevention and control of pollution)Act 1981

3) The Environmental protection act1986
4) The Disaster Management Act 2005
5) The Green Tribunal Act 2010
6) The Wild Life Conservation Act 1972

The Forest Conservation Act 1980

1) The Water(prevention and control of pollution)Act 1974

This act came into force in the year 1974 for the purpose of prevention and control of pollution of water and maintain or restoring the wholesomeness of water. The rapid growth of Industrialization and Urbanization is the main cause of water pollution. To control this water act came into force. The Water act cast obligations on the part of industries and local bodies to obtain the prior consent to establish and operate Industry for new discharge of sewage and trade effluent. This act also established various boards to controlled water pollution such as,

a) Central pollution Control Board
b) State Pollution Control Board

2) The Air (prevention and control of pollution) Act 1981

This Act was enacted on the same lines of Water act 1974.The unique features of the act is that in include Noise within the definition of Air pollution and also include the provisions for Air Pollution Control Zone. The Central and State boards constituted under the Water act which perform the same function under the Air Act1981

3) The Environment Protection Act 1986

The Environment Protection Act 1986 is an act of Parliament of India. The Government of India enacted this act to implement the decisions of the United Nations Conference on the Human Environment held at Stockholm in 1972.The Act is a general legislation on protection and improvement of human environment. Under this act the Central Government may constitute authorities such as Central and State authorities as like Central Pollution Control Board and State Pollution Control Board for implementing legislation relating to prevention and control of environmental pollution

4) The Disaster Management Act,2005

The Disaster Management Act2005 was passed by the Parliament of India in the year 2005.The Act provides for “the effective management of disaster and for matters connected there with or incidental thereto”. This act establishes various authorities for the effective management natural calamities like disaster. The authorities such as

a) National Authority (section 3)
b) National Executive Committee (section 8)
c) State Disaster Management Authority (section 14)
d) District Disaster Management Authority (section 25)
e) National Disaster Response Force (NDRF) (section 44-45)

5) The National Green Tribunals Act 2010

The National Green Tribunal Act (NGT) was enacted by the parliament in the year 2010 to establish the special tribunals to resolve the speedy disposal of Environment cases or various issues pertaining to protection and promotion of environment .This act provides jurisdiction to Green Tribunal to deals with all matters related to environment protection and also cast obligations to provide speedy justice in environmental matters. The establishment of this Green Tribunals will reduce the burden of cases of the superior courts, such as Supreme Courts .This Tribunals follow flexible procedure as compare to other courts such as they are not abide to follow strict procedure of CPC and Evidence Act, but they should follow the principle of Natural Justice .

6) The Wild Life Conservation Act 1972

The Wild Life Protection Act 1972 is an act of Parliament of India enacted for the protection of plants and animal species. This act prohibits capturing, killing, poisoning, or trapping of wild animal. This act also prohibits hunting of wild animal. According to this act Hunting includes. (Section 2) “capturing, killing, poisoning, snaring ,or trapping any wild animal, and every attempt to do so, driving any wild animal for any of the purpose specified in sub clause, injuring, destroying or taking any body part or any such animal ,or in the case of wild birds, or reptiles, disturbing or damaging the eggs or nests of such birds or reptiles.”

The implementation of the act carried out with the help of authorities like

a) The Police
b) The Central Bureau of Investigation
c) The Forest Department
d) The Custom

7) The Forest (Conservation)Act 1980

The Forest Conservation Act 1980 an Act of the Parliament of India enacted with the intention to provide the conservation of forest and to control deforestation of forest areas in India. This act impose restriction upon the state government for making any order to change the Forest land into Non- forest land.(section 2).according to this act the central government having power to constitute an advisory committee.

The Role of Judiciary in Protection of Environment

In India the judiciary plays a vital role in conservation of environment through its Judicial Decisions. In various environmental cases, the Supreme Court of India gives new dimensions and interpretation to fundamental rights under Article 21 of the constitution. In the case of *Subhash Kumar V. State of Bihar (1991)1SCC598* ,the Supreme Court of India held that right to live is a fundamental right under Article 21 of the constitution and it includes the right to enjoy pollution free environment for healthy life. The Supreme Court of India in its judgment *Vellore Citizens' Welfare Forum V. Union of India (1995)5SCC 647* reiterated the need for creating the Environmental Courts in India.

In various Public Interest Litigations by way of Judicial Activism the new principles was evolved by the judiciary which cast duty not only on the individual but also on State for protection and promotion of environment .In the case of Indian Council For *Enviro -Legal Action V. Union (1996)3SCC212* Of India the Supreme Court Of India evolved the " precautionary principle and polluter pay principle" In the case of *Vellore Citizens' Welfare Forum V. Union Of India (1995)5SCC 647* The court express that "precautionary principle " and the "polluter pay principle" are essential principles of sustainable development and that have to be accepted as part of law of the land and consider as a part of the environmental law of the country.

II. CONCLUSION

The Environmental Protection legislations plays vital role in protection and promotion of environment, but it is not the ultimate solution to save the environment unless the individual consider saving the environment as his own responsibility. We all have to come together to save the environment for survival of present and future generation.

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Study of Physicochemical Parameters of Lentic Ecosystems in Ahmednagar District of Maharashtra State India

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ABSTRACT

Study of physicochemical parameters is very necessary to know its quality. As quality of water is very important for use of water for drinking for human beings and the animals. Lentic ecosystems are important as lakes and pons are one of the useful surface water resources..In the present study water samples were collected from the lakes in Sangamner and Akole Taluka of Ahmednagar district of Maharashtra state India. The temperature, TDS and pH were recorded on the spot by using thermometer, digital pH and TDS meters were used to measure pH and TDS. The acidity was found in the range of 13mg/l to 48mg/L. The alkalinity was found in the range of 166 mg/L to 506 mg/L . Dissolved Carbon di oxide was found in the range of 11.44 mg/L to 22 mg/L. The dissolved Oxygen was found in the range of 2.03 mg/L to7.64 mg/L. The pH was found in the range of 7 to 9.5. The electrical conductivity was found in the range of 0.17 to 0.61mS. The TDS was found in the range of 100 to 310 ppm.

Keywords : Physicochemical Parameters, Ecosystem.

I. INTRODUCTION

Water is extremely necessary for the survival of life. Apart from drinking it is very necessary for agriculture, industries, washing of cloths, vehicles, animals and also for dairy industry. Indian economy is mainly agro based. Majority of the Indian population is directly or indirectly depends on the agriculture and agro based industry for their survival. Maharashtra state in India and especially Ahmednagar district is having several milk producing animals. The lakes are one of the major sources of surface water. Review of literature reveals that not musch work is done on the physicochemical parameters of lentic ecosystems in Sangamner and Akole talukas of Ahmednagar district of Maharashtra state in India. Therefore the present work was undertaken. .Physicochemical parameters of Gulabsagar water body in Jodhpur was studied by Chouhan and Vyas 2017. Nayaka and Shridhara 2018 has carried out assessment of physicochemical parameters of Gundalamma lake in Tumkur in Karnataka. Nama and Dhanraj 2018 has assessed the water quality of Palasani pond in Jodhpur by studying physicochemical parameters. Bhagde et al 2016 has studied hydrobiology of Devtale lake in Sangamner Taluka of Ahmednagar district of Maharashtra state. Water quality index of some lakes around Kolhapur city of Maharashtra was determined by Anekar and Dongre 2016. Gautam and Shrivastva 2017 studied the physicochemical parameters of pond in Bhopal by analyzing the water sample. Nakhate and Kale 2018 studied the physicochemical parameters in Kankaleshwar lake in Beed district. Joseph Ndjama et al 2017 has done the work to study physicochemical and biological characteristics of Nkilobisson artificial lake in Yaounde Cameroon.

II. METHODS AND MATERIAL

Water samples were collected from the lakes in Sangamner and Akole Taluka of Ahmednagar district in Maharashtra state, India. Temperature ,pH and TDS were recorded on the spot. Thermometer was used to record the temperature. Digital pH and TDS meters are used to record pH and TDS respectively. Acidity, dissolved carbon di oxide ,Alkalinity and Hardness was estimated by the methods given by Maiti 2011.Oxygen was fixed using DO bottles and Winkeler's solution. Dissolved oxygen was estimated by using Winkler's method.

III. RESULTS AND DISCUSSION

Sr. No	Parameters	Kombhalne	Chandgirwadi	Khirvire 1	Khirvire 2	Dhamangaon Awari
1	Acidity	48	13	13	35	43
2	Alkalinity	166	273	343	410	506
3	Carbon di oxide	22	22	11.44	14.52	10.12
4	Dissolved oxygen	2.024	4.29	3.23	7.64	4.11
5	pH	8.7	8.8	9.5	7.0	7.0
6	TDS	130	100	180	310	300
7	Electrical conductivity	0.26	0.17	0.31	0.61	0.38
8	Hardness	104.6	124.6	116.6	210.4	120

Acidity : The acidity was found in the range of 13mg/l to 48mg/L ,at Kombhalne it was found to be 48mg/L, at Chandgirwadi 13mg/L, at Khirvire 1mg/L, it was 13mg/L, 35mg/L at Khirvire 2mg/L and at Dhamangaon Awari 43 mg/L.

Alkalinity : The alkalinity was found in the range of 166 mg/l to 506 mg/l . It was 166 mg/l at Kombhalne , at Chandgirwadi 273 mg/l, at Khirvire 1 343 mg/l and at Khirvire 2mg/L it was 410 mg/L and at Dhamangaon Awari 506 mg/L.

Dissolved Carbon di oxide : Dissolve Carbon di oxide was found in the range of . 11.44 mg/L to 22 mg/L. At Kombhalne 22mg/L at Chandgirwadi 22mg/l, at Khirvire 1 it was 11.44mg/L, at Khirvire 2mg/L, 14.52 mg/L and Dhamangaon Awari 10.12 mg/L.

Dissolved Oxygen : The dissolved Oxygen was found in the range of 2.03 mg/lLto7.64 mg/L. It was 2.024 mg/L at Kombhalne , at Chandgirwadi 4.29 mg/L, at Khirvire1 it was 3.23 mg/L and 7.64 mg/L at Khirvire 2mg/L and at Dhamangaon Awari 4.11 mg/L.

pH : The pH was found in the range of 7 to 9.5. It was 8.7 at Kombhalne , at Chandgirwadi 8.8, at Khirvire 1 it was 9.5 and at Khirvire 2 it was 7 and at Dhamangaon Awari 7.

TDS : The TDS was found in the range of 100 to 310. It was 130 at Kombhalne , at Chandgirwadi 100, at Khirvire 1 it was180 , at Khirvire 2 it was 310 and at Dhamangaon Awari 300.

Electrical Conductivity: The electrical conductivity was found in the range of 0.17 to 0.61mS.It was 0.26mS at Kombhalne , at Chandgirwadi 0.17mS, at Khirvire 1 it was 0.31mS, 0.61mS at Khirvire 2mS and at Dhamangaon Awari 0.38mS.

Hardness : The hardness was found in the range of 104 to 210.It was 104.6 at Kombhalne , at

Chandgirwadi 124.6, at Khirvire 1 it was 116.6 and at Khirvire 2 it was 210.4 Dhamangaon Awari 120.

Acidity was minimum at Chandgirwadi and Kombhalne. Alkalinity was maximum at Dhamangaon Awari. And minimum at Kombhalne. Dissolved Carbon dioxide was maximum at Kombhalne and Chandgirwadi and minimum at Dhamangaon Awari. dissolved oxygen was maximum at Khirviri 2 and minimum at Kombhalne .pH was maximum at Khirvire 1 and minimum at Khirvire 2 and Dhamangaon Awari. TDS was maximum at Khirvire 2 and minimum at Chandgirwadi. Electrical conductivity was maximum at Chandgirwadi and minimum at Khirvire 2. Hardness was minimum at Kombhalne and maximum at Khirvire 2.At all the spots acidity and alkalinity were found inversely proportional. Similarly dissolved Carbon di oxide and dissolved Oxygen were also inversely proportional.

IV. CONCLUSION

From the results obtained it is observed that the physicochemical parameters vary at different locations. It is might be due to variation in topography geographical conditions.

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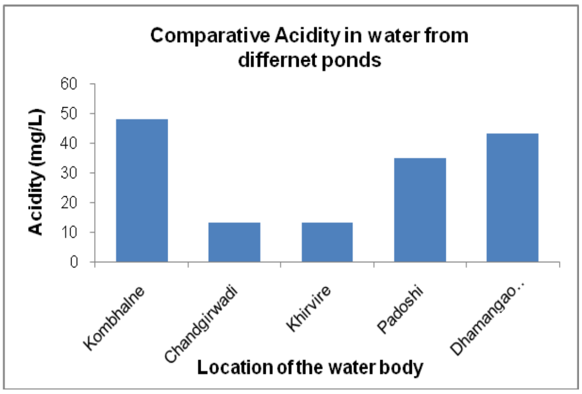

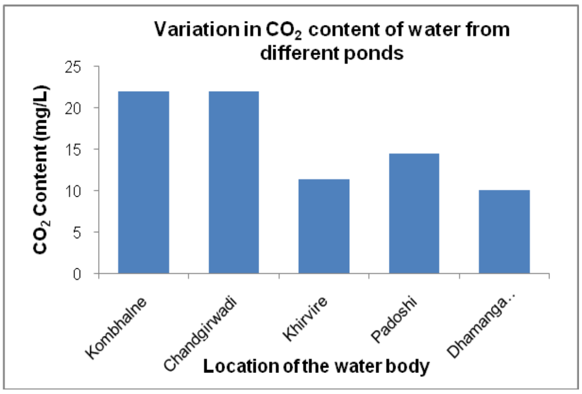

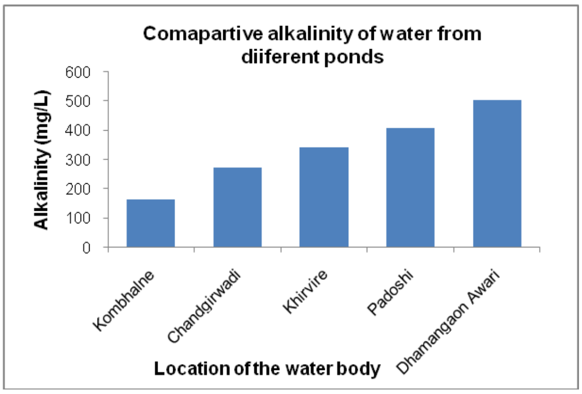

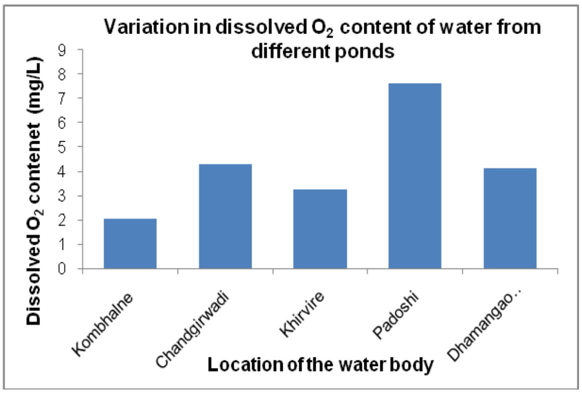

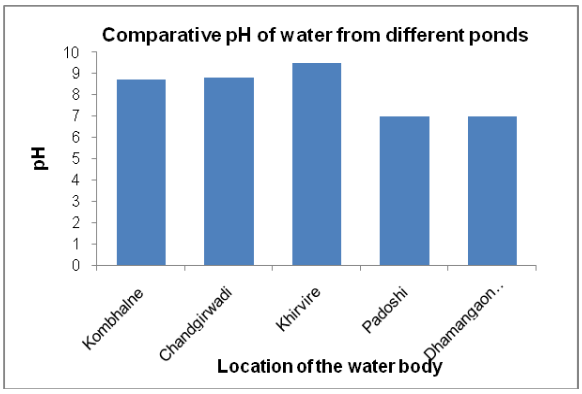

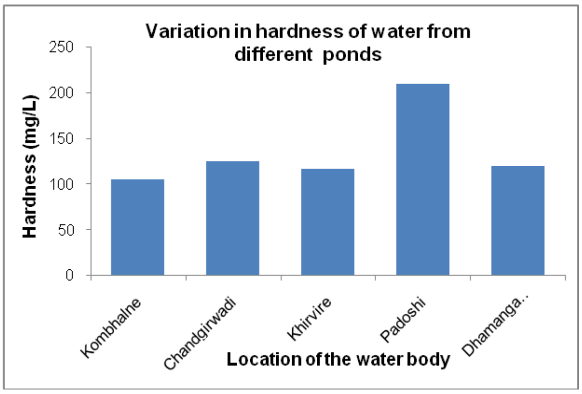

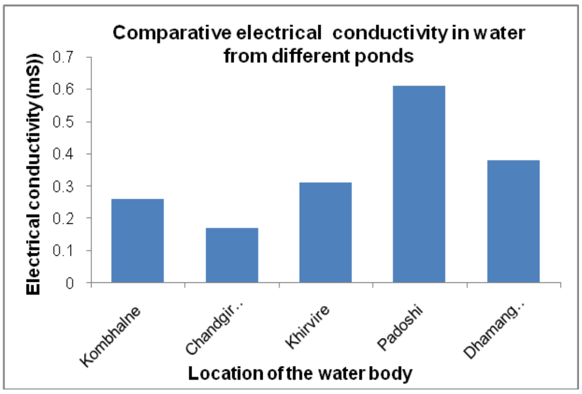

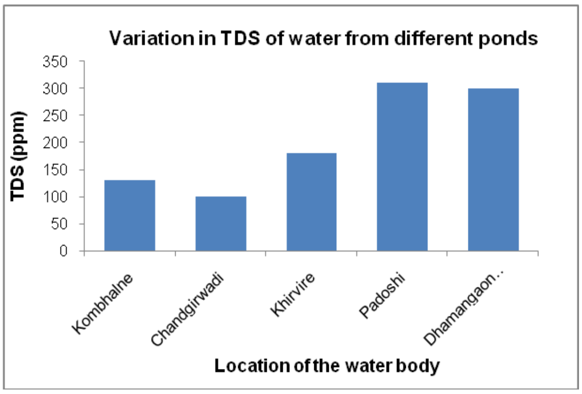

Kombalne

Changdirwadi

Khirvire 1

Khirvire 2

Dhamangaon Awari

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New Approach of Indole Synthesis - A Boom

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ABSTRACT

Indoles, both naturally occurring and synthetic, exhibit wide-ranging biological activity. Unusual and complex molecular architectures occur among their natural derivatives. As a result, this important ring system continues to attract attention from the international chemical community, and new methodologies for the construction of this ever relevant heteroaromatic ring continue to be developed. Unfortunately, many methods frequently start from ortho-substituted anilines, thereby greatly restricting the availability of starting materials. A more general approach would start from a mono-functionalized arene such as an aniline or halobenzene, followed by cyclization with C–C or C–N bond formation to an inactivated C–H bond. Such methods are the subject of this perspective. Indole efficiently synthesized by using different Lewis acids like HCl, H_2SO_4, $ZnCl_2$, PCL_5.

Keywords : Hetero Aromatic, Mono-Functionalized Arene

I. INTRODUCTION

Heterocyclic chemistry is one of the most valuable sources of novel compounds with diverse biological activity, mainly because of the unique ability of the resulting compounds to mimic the structure of peptides and to bind reversibly to proteins. Indole is an aromatic heterocyclic organic compound with formula C8H7N. It has a bicyclic structure, consisting of a six-membered benzene ring fused to a five-membered pyrrole ring. Indole is widely distributed in the natural environment and can be produced by a variety of bacteria. As an intercellular signal molecule, indole regulates various aspects of bacterial physiology, including spore formation, plasmid stability, resistance to drugs, biofilm formation, and virulence. The amino acid tryptophan is an indole derivative and the precursor of the neurotransmitter serotonin. Macrocyclic heterocyclic chemistry is one of the upcoming research areas in the chemical science. Macrocyclic compounds continue to attract significant attention due to their numerous possible applications particularly in the areas like medicine, catalysis and industry. This article summarizes the synthetic developments in Indole based heterocyclic compounds. To medicinal chemists, the true utility of heterocyclic structures is the ability to synthesized by different ways which are studied in this present research work which play an important part in new ways of synthesis.

II. METHODS AND MATERIAL

The routine procedure has been used for study of Fisher Indole synthesis and modification carried out in different aspects.

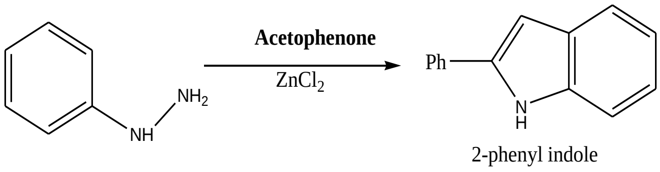
1. Routine method of Indole synthesis:

Prepare 1gm acetophenone and 0.9 gm phenyl hydrazine by warming a mixture in 3 ml of alcohol and few drops of glacial acetic acid. Filter the reaction mixture. Wash with dilute HCl followed by rectified spirit. Take 1 gm of Phenyl hydrazones in a beaker containing 6.42 gm of PPA. Heat on boiling water bath.Stirr with maintained temperature at 100-

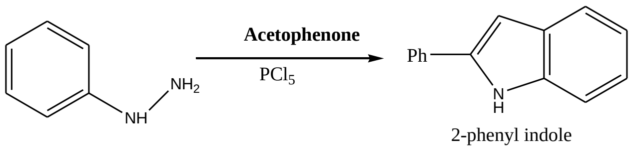
120^0C for 10 mins. Add cold water stir well to complete solution of PPA .Filter the product.

2. Use of $ZnCl_2$ as Lewis Acid:

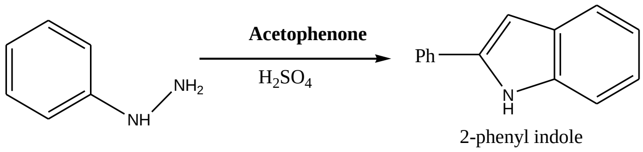
The routine method is modified by use of $ZnCl_2$ as Lewis acid as follows



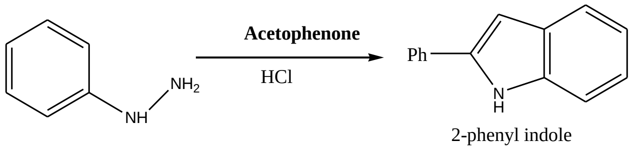
3. PCl_5 as Lewis acid:



4. Use of H_2SO_4:



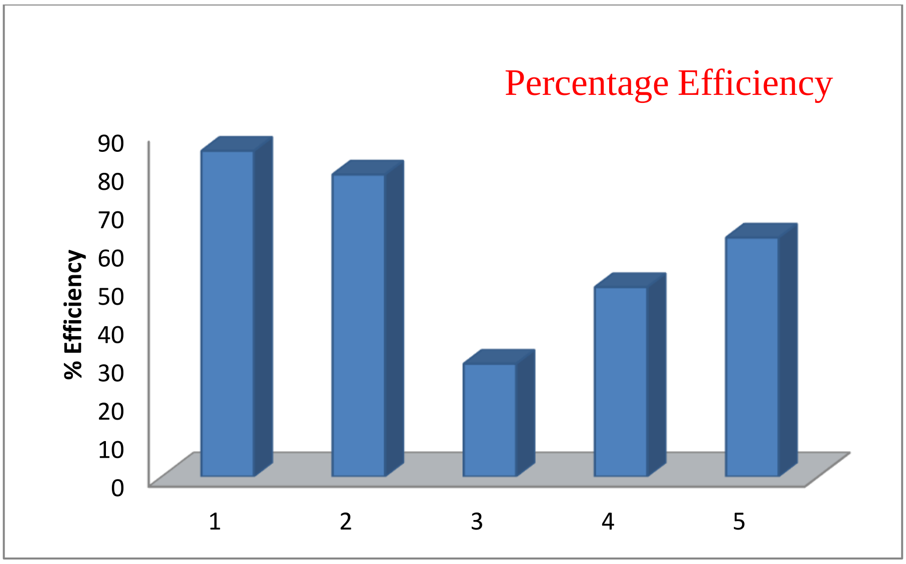
5. Use of HCl:



III. RESULTS AND DISCUSSION

Table 1

S.N	Name of Reaction	Dist. travelled by reactant in cm	Dist. travelled by product in cm	Solvent run in cm	Yield
01	Fisher Indole Synthesis	2.9	3.4	4.0	85%
02	Indole synthesis by $ZnCl_2$	3.8	4.3	5.0	67%
03	Indole synthesis by PCl_5	2.5	2.9	3.6	25%
04	Indole synthesis by H_2SO_4	2.5	3.0	3.4	42%
05	Indole synthesis by HCl	3.2	3.7	4.2	53%



IV. CONCLUSION

1. Routine synthesis of indole is as per method given as above. The synthesis can efficiently carried out by ZnCl2, H2SO4, HCl etc.
2. These reactions are cost effective and less polluted.

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Ultrasonic and Thermodynamic Studies in Ternary Liquid Systeam of Choline Chloride Urea+ L-Glutamine + Water at 298.15 And 308.15k

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ABSTRACT

The density(ρ), viscosity(η) and ultrasonic velocity(U) were measured for ternary system [choline chloride urea(1) + L-Glutamine(2) + water(3)] at 298.15 and 308.15K .Using Experimental values, the various acoustical parameters such as the adiabatic compressibility (ß),Intermolecular free length (L_f), Free volume (V_f), Internal pressure (Π_i), Acoustic impedance (Z) have been studied and have been estimated using the standard relations. The results have been analyses on the basis of variations of thermodynamic parameters. The presence of strong interactions is noticed in the ternary system.

Keywords : Ultrasonic Velocity, Adiabatic Compressibility, Intermolecular Free Length, Free Volume, Internal Pressure.

I. INTRODUCTION

Knowledge of thermodynamic and acoustical properties is of great significance in studying the physico-chemical behaviour and molecular interactions in a variety of liquid mixtures[1-3]. Considerable scientific and practical interest has been stimulated by the investigation of organic liquids using ultrasonic measurements[4, 5]. Such a type of study has been found to yield information regarding the intermolecular process and the structure of the liquid state[6]. The compositional dependence of thermodynamic properties has proved to be a very useful tool in understanding the nature and extent of pattern of molecular aggregation resulting from intermolecular interaction between components Acoustic and thermodynamic parameters have been used to understand different kinds of association, the molecular packing, molecular motion, and various types of intermolecular interactions and their strengths, influenced by the size in pure components and in the mixtures[7]. Theoretical evaluation of ultrasonic velocity in ternary liquid mixtures and its comparison with the experimental values reflects the molecular interactions in liquid mixtures. Ultrasonic velocities evaluated using Nomoto's relation[8] (NR), Impedance dependence relation[9] (IDR) and Collision factor theory[10] (CFT) have been compared with those obtained from the experimental values.

II. EXPERIMENTAL DETAILS

Chemicals used in this study (> 99.5% of purity) were supplied S.D. fine chem. Industries, Mumbai (purity 99%) used without further purification. The mixtures of the desired composition were prepared by weighting on a HR-120 (A & D Japan) electronic balance with a precision of ± 0.0001 g. all mixed solvents were prepared by molality. The prepared solution was used within 12hrs. The densities of pure liquids and binary mixtures were measured by using 15cm^3 double arm pycnometer as describe earlier [11]. The Pycnometer was calibrated by using conductivity water with 0.9970 g/cm^3 as its density at 298.15 K. The pycnometer filled with air bubble free experimental liquid was kept in transparent walled water bath for 10-15 min. to attain thermal equilibrium. The position of liquid levels in the two arms was recorded.

The dynamic viscosities were measured using an Ubbelohde suspended level viscometer , calibrated with conductivity water. An electronic digital stopwatch with readability of ± 0.01 sec. was used for the flow time measurement, at least three repetitions of each data reproducible to ± 0.05 sec. were obtained and the result was averaged. Since all flow times were greater than 200 sec. and capillary radius (0.5 mm) was far less than its length (50 to 60mm.). The kinetic energy and corrections respectively were found to be negligible. The uncertainties in dynamic viscosities are of the order ± 0.003 m Pa s. The speed of sound waves was obtained by using ultrasonic interferometer, (Mittal Enterprises, New Delhi) at a fixed frequency of 2 MHz with an accuracy of ±2 ms^{1}. An electronically digital operated constant temperature bath has been used to circulate water through the double walled measuring cell made up of steel containing the experimental solution at the desired temperature. The accuracy in the temperature measurement was ±0.1 K.

Theory

The various acoustical parameters such as adiabatic ompressibility (ß), free length (*L*f), free volume (*V*f), acoustic impedance (Z),Internal pressure (Πi) and their excess values were determined using the following equations:

$$\text{ß} = \frac{1}{U^2\rho} \qquad \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

$$L_f = K_T\, \text{ß}^{1/2} \qquad \ldots\ldots\ldots\ldots\ldots (2)$$

$$V_f = \frac{Meff}{\eta K} \qquad \ldots\ldots\ldots\ldots\ldots (3)$$

$$\pi_i = bRT\left(\frac{K\eta}{U}\right)\left(\frac{\rho^{2/3}}{M_{eff}{}^{7/6}}\right) \qquad \ldots\ldots\ldots\ldots (4)$$

$$Z = u\rho \qquad \ldots\ldots\ldots\ldots (5)$$

where K_T is the temperature dependent constant, M_{eff} the effective molecular weight of the solution, *K* the temperature independent constant (K= 4.28×10^9), *b* a constant which is 2 for cubic packing, *R* the gas constant and *T* is the temperature in K.

III. RESULTS AND DISCUSSION

The experimentally measured values of Density (ρ), Ultrasonic velocity (U) and thermodynamic parameters adiabatic compressibility (ß), Intermolecular free length (L_f), Free volume (V_f), Acoustic impedance (Z) and Internal pressure (π_i) of ternary liquid system at temperature 298.15 K & 308.15 at frequency 2MHz are presented in table-1 and 2 respectively. Table-1 and 2 shows that, ultrasonic velocity, density, adiabatic compressibility (ß), Intermolecular free length (L_f), Free volume (V_f), Internal pressure (π_i), Acoustic impedance (Z) acoustic impedance and relative association increases while adiabatic compressibility and intermolecular free length decreases with concentration of ionic liquid with amino acid at temperature 298.15 and 303.15K.

The ultrasonic velocity (U) for ionic liquid and aqueous solution of L-Glutamine at 2MHz frequency and at 298.15 and 308.15 K temperature have been determined. The variations in ultrasonic velocity in liquid mixtures depend on concentrations (x) of solutes and temperature. Ultrasonic velocity (U) is related to intermolecular free length. As the free length decreases due to the increase in concentrations of solutes, the ultrasonic velocity has to increase. Ultrasonic velocity increases with increase in concentrations of solutes.

The increase in density with molal concentration suggests a solute solvent interaction exist between the electrolyte and water. In other words the increase in density may be interpreted to the structure makes of the solvent due to H-bonding.

The viscosity is an important parameter in understanding the structure as well as molecular interaction, occurring in the solutions. From above table, it is observed that viscosity of the solutions shows a non-linear behavior in the system. The increase in viscosity with concentration in all the system suggests that the extent of complexation increase with increase in concentration.

The adiabatic compressibility is calculated using equation (1). The calculated values of (ß) have been presented in table-1 and 2. From these tables it is clear that the compressibility of solvent is higher than that of a solution and it decreases with increase in concentrations.

The values of intermolecular free length for given system have been calculated using the equation (2). Increase in concentrations leads to decrease in gap between two species which is referred by intermolecular free length (L_f). With the increase in concentrations of solutes, intermolecular free length (L_f) has to decrease, intermolecular free length (L_f) is predominant factor in determining the variations of ultrasonic velocity in liquid mixtures. From table-1 and 2, it has been observed that, in the present investigation on increasing concentration. The decrease in Lf with increase of concentration in solution between solute and solvent suggesting the structure, promoting behavior of solutes, The value of free volume calculated using the equation (3).The calculated values of free volume (V_f) decreases whereas, the internal pressure (Π_i) increases due to the various degree of dispersive interaction and the columbic interaction existing between the component molecules. Internal pressure (Π_i) calculated using the equation (4). Acoustic impendence (z) calculated using equation (5), from table 1 and 2 it is observed that acoustic impendence increases with increase in concentrations. For a given concentration the values of acoustic impendence (z) increases with increase in concentration in liquid system .The increase in (z) with the increase in concentration of solutes can be explained in terms of inter and intra molecular interactions between the molecules of liquid mixtures.

IV. CONCLUSION

The observed trends and variations of thermo dynamical parameters with molar concentration of Ch Cl urea in aqueous amino acids provide useful information about the nature of intermolecular forces existing in the mixture. The existence of ion-solvent (or) solvent-solvent interaction resulting in attractive forces promote the structure-making tendency, An appreciable existence of solute-solute interactions present in the system with varying degrees hence it is evident that the ultrasonic velocity measurement in the given medium serves as a powerful probe in characterizing the physio-chemical properties of the media. The almost linear variation of acoustical parameters with temperature shows that there exist less intermolecular forces between the components of the ternary liquid mixtures.

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Table 1 : Density (ρ), viscosity (η) velocity (U), adiabatic compressibility (β), Intermolecular free length (L_f), Free volume (V_f), Internal pressure (π_i), Acoustic impedance (Z) of Ch-Cl-urea (1) + L-Glutamine (2) + Water (3) at 298.15 K

X_1	X_2	ρx10^3 (kg/m^{-3})	ηx10^3 (Nsm^{-2})	U (msec^{-1})	βx10^{-10} (Pa^{-1})	L_fx10^{-10} (m)	V_fx10^{-6} (m^3 mol^{-1})	π_i x10^{-6} (Pa^{-1})	Zx10^6 (Nm^{-2})
0.0001	0.0016	1.0100	2.5995	1445	4.756	0.431	2.413	0.4569	1.455
0.0002	0.0015	1.0068	2.5922	1447	4.743	0.430	2.410	0.4571	1.457
0.0003	0.0013	1.0069	2.5883	1449	4.727	0.430	2.409	0.4575	1.460
0.0004	0.0011	1.0074	2.5817	1450	4.717	0.429	2.403	0.4584	1.462
0.0005	0.0007	1.0083	2.5776	1455	4.684	0.428	2.407	0.4583	1.467
0.0005	0.0009	1.0085	2.5711	1458	4.663	0.427	2.405	0.4587	1.471
0.0006	0.0005	1.0089	2.5672	1461	4.642	0.426	2.402	0.4595	1.474
0.0007	0.0004	1.0092	2.5608	1463	4.626	0.425	2.400	0.4598	1.478
0.0008	0.0002	1.0122	2.5562	1467	4.590	0.423	2.402	0.4604	1.485

Table 2 : Density (ρ), viscosity (η) velocity (U), adiabatic compressibility (β), Intermolecular free length (L_f), Free volume (V_f), Internal pressure (π_i), Acoustic impedance (Z) of Ch-Cl-urea (1) + L-Glutamine (2) + Water (3) at 308.15 K

X_1	X_2	ρx10^3 (kg/m^{-3})	ηx10^3 (Nsm^{-2})	U (msec^{-1})	βx10^{-10} (Pa^{-1})	L_fx10^{-10} (m)	V_fx10^{-6} (m^3 mol^{-1})	π_i x10^{-6} (Pa^{-1})	Zx10^6 (Nm^{-2})
0.0001	0.0016	1.0048	2.3362	1408	5.020	0.450	2.566	0.4573	1.415
0.0002	0.0015	1.0052	2.3405	1413	4.982	0.448	2.571	0.4569	1.420
0.0003	0.0013	1.0057	2.3456	1417	4.952	0.447	2.572	0.4571	1.425
0.0004	0.0011	1.0065	2.3509	1422	4.913	0.445	2.574	0.4573	1.431
0.0005	0.0007	1.0067	2.3573	1425	4.892	0.444	2.571	0.4577	1.435
0.0005	0.0009	1.0072	2.3602	1426	4.882	0.444	2.569	0.4582	1.436
0.0006	0.0005	1.0076	2.3682	1428	4.867	0.443	2.560	0.4596	1.439
0.0007	0.0004	1.0083	2.3732	1431	4.843	0.442	2.561	0.4596	1.443
0.0008	0.0002	1.0103	2.3764	1433	4.820	0.441	2.560	0.4604	1.448

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Use of Ultrasonic Interferometer to Study the Interaction Between Solvent-Solvent Molecules in a Tertiary Mixture at Different Temperatures

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ABSTRACT

The basic parameters like viscosity (η), density(ρ) and velocity (U) can be measured by ultrasonic Interferometer. From these three parameters various thermodynamical and acoustical parameters such as specific acoustic impedance (Z), Intermolecular free length (L_f), adiabatic compressibility 's (β) etc can be estimated using standard relations from measured values of Ultrasonic viscosities, densities and velocities in the wide range of concentrations at 35^0 C, 40^0C and 45^0C temperatures for Acetone + Propanol-2 +Chloroform tertiary system. The solvent-solvent interactions are studied on the basis of increase or decrease in ultrasonic velocity, density, viscosity and other derived acoustical parameters in terms of structure making and structure breaking tendencies of various solvent molecules.

Keywords: Density, Ultrasonic Interferometer, Ultrasonic Velocity, Viscosity, Water Bath

I. INTRODUCTION

The study of molecular interactions in the liquid mixtures is of considerable importance in the elucidation of the structural properties of the molecules. Lagemann and Dunbar [4] were the first to point out the sound velocity approach for qualitative determination of the degree of association in liquids. Recent developments have made it possible to use ultrasonic energy in medicine, engineering, agriculture and other industrial applications.[5,6] .Ozawa and Minamisawa [7] have observed concentration of ultrasonic velocity invariant with respect to temperature in alcohol-water mixtures. Hanel[8] has measured sound velocity and thickness of thin samples by time – resolved acoustic microscopy. Bae and Yun [9] have studied the ultrasonic velocity in binary solutions of silicon dioxide and water. Knowledge of thermodynamic and acoustical properties is of great importance in studying the physio-chemical behavior and molecular interactions in a variety of liquid mixtures(1,3). The compositional dependence of thermodynamic properties has proved to be a very useful tool in understanding the nature and extent of pattern of molecular aggregation resulting from intermolecular interaction between components.

II. EXPERIMENTALDETAILS

Ultrasonic velocity for the mixture was measured using the ultrasonic interferometer (Model M 81) supplied by Mittal Enterprises, New Delhi, that has a reproducibility of ± 0.4 m/s at 25^0 C with a fixed frequency of 3 MHz. The temperature was maintained constant by circulating water from a thermodynamically controlled water bath (accuracy ±0.1 ^{0} C). The temperature of the cell is measured using a thermocouple was found to accurate to ± 0.25 ^{0} C. The density of the mixtures has been measured using a sensitive pycnometer with an accuracy of 0.5 kg/m^3. Chemicals used in this study are ultra pure ,supplied by Sigma-Aldrich Ltd and used without purification. Tertiary system is studied at different temperatures, 35^0 C, 40^{0}C and 45^{0}C with different concentrations of the system .Especially for this system ultrasonic velocities, densities and viscosities of the mixtures have been measured at different temperatures.

III. THEORY

Other acoustical parameters such as adiabatic compressibility ((β),Intermolecular free length (L_f) ,Molar Sound velocity(R), Specific acoustic impedance (Z) etc can also be determined.:

Intermolecular free length (L_f) =$K\beta^{1/2}$ (1)

Adiabatic compressibility (β)= $\frac{1}{U^2\rho}$ (2)

Where k values for different temperatures were taken from the work of Jacobson[29]; at 35,40 and 45^o C the K values are 637, 642 ,647 respectively.:

Molar sound velocity (R) =$U^{1/3}V$ (3)

Molar compressibility(B) = $\left(\frac{M}{\rho}\right)\beta^{-1/7}$ (4)

where V and M are the molar volume and molecular weight of the mixtures, respectively.:

Specific acoustic impedance (Z) =ρU (5)

The excess adiabatic compressibility (β^E) and excess intermolecular free length (L_f^E) are evaluated by the following expressions:

$B^E = \beta_{exp} - \beta_{ideal}$ (6)

$(L_f^E) = L_{f.exp} - L_{f.ideal}$ (7)

For β_{ideal} and $L_{f.ideal}$, the densities and the ultrasonic velocities of various components in pure state at the three given temperatures have been measured. Further, the velocities of both the systems at different concentrations and temperatures have been evaluated theoretically using volume additive rule[21] as :

$U_{ideal} = U_1\emptyset_1 + U_2\emptyset_2 + U_3\emptyset_3$ (8)

Where U_1,U_2, and U_3 are the velocities of the three components of the ternary liquid mixture in pure state and $\emptyset_1$,$\emptyset_2$ and $\emptyset_3$ are their volume fractions .

Simillarly ideal density is evaluated using :

$P_{ideal} = \rho_1\,\emptyset_1 + \rho_2\,\emptyset_2 + \rho_3\,\emptyset_3$ (9)

Finally β_{ideal} and $L_{f.ideal}$ are evaluated using following equations :

$\beta_{ideal} = \frac{1}{U^2_{ideal.}\,\rho_{ideal}}$ (10)

and

$L_{f.ideal} = K\beta^{1/2}{}_{ideal}$ (11)

IV. RESULTS AND DISCUSSION

Table 1. Conversion of CGS units to SI units.

No	Parameter	CGS units	SI units
1	Ultrasonic velocity (U)	1 cms^{-1}	$10^{-2}ms^{-1}$
2	Density (ρ)	1 g cm^{-3}	10^3 Kg m^{-3}
3	Adiabatic compressibility (β)	1dyn^{-1} cm^2	10 $N^{-1}m^2$
4	Intermolecular free length(L_f)	1A^o	10^{-10} m
5	Molar sound velocity (R)	1 cm^3 mol^{-1} $(cm\ s^{-1})^{1/3}$	$10^{-20/3}$ m^3 mol^{-1} $(ms^{-1})^{1/3}$
7	Molar compressibility (B)	1 cm^3 mol^{-1} $(dyn^{-1}\ cm^2)^{-1/7}$	10 $^{-43/7}$ m^3 mol^{-1} $(N^{-1}m^2)^{-1/7}$
8	Wave number (λ)	1 cm^{-1}	10 m^{-1}

Table 2. Ultrasonic velocity, Density and viscosity of Tertiary mixture at different temperatures

Temp	**Mole Fraction**			**Ultrasonic velocity**	Density(ρ)	Viscosity (η)
	(Acetone) X_1	**(Propanol) X_2**	**(Chloroform) X_3**	**m/sec**	gm/cm^3	centipoise
35 ^{0} C	**0.2432**	**0.06832**	**0.7892**	**845**	**1.2700**	**0.4982**
	0.2431	**0.06834**	**0.7894**	**847**	**1.2692**	**0.4979**
	0.2429	**0.06836**	**0.7895**	**849**	**1.2689**	**0.4971**
	0.2427	**0.06839**	**0.7896**	**849**	**1.2688**	**0.4969**
	0.2425	**0.06842**	**0.7898**	**852**	**1.2682**	**0.4965**
	0.2421	**0.06845**	**0.7900**	**854**	**1.2580**	**0.4964**
	0.2418	**0.06847**	**0.7904**	**851**	**1.2580**	**0.4962**
	0.2416	**0.06847**	**0.7913**	**850**	**1.2578**	**0.4961**
	0.2415	**0.06850**	**0.7915**	**849**	**1.2575**	**0.4961**
	0.2413	**0.06852**	**0.7918**	**849**	**1.2574**	**0.4955**
40^0 C	**0.2432**	**0.06832**	**0.7892**	**849**	**1.2575**	**0.4954**
	0.2431	**0.06834**	**0.7894**	**851**	**1.2571**	**0.4953**
	0.2429	**0.06836**	**0.7895**	**853**	**1.2571**	**0.4951**
	0.2427	**0.06839**	**0.7896**	**856**	**1.2569**	**0.4947**
	0.2425	**0.06842**	**0.7898**	**858**	**1.2569**	**0.4946**
	0.2421	**0.06845**	**0.7900**	**865**	**1.2567**	**0.4943**
	0.2418	**0.06847**	**0.7904**	**861**	**1.2565**	**0.4943**
	0.2416	**0.06847**	**0.7913**	**860**	**1.2564**	**0.4940**
	0.2415	**0.06850**	**0.7915**	**858**	**1.2564**	**0.4938**
	0.2413	**0.06852**	**0.7918**	**857**	**1.2563**	**0.4930**
45^0 C	**0.2432**	**0.06832**	**0.7892**	**857**	**1.2564**	**0.4931**
	0.2431	**0.06834**	**0.7894**	**860**	**1.2561**	**0.4928**
	0.2429	**0.06836**	**0.7895**	**864**	**1.2559**	**0.4927**
	0.2427	**0.06839**	**0.7896**	**865**	**1.2550**	**0.4926**
	0.2425	**0.06842**	**0.7898**	**867**	**1.2549**	**0.4924**
	0.2421	**0.06845**	**0.7900**	**870**	**1.2540**	**0.4921**
	0.2418	**0.06847**	**0.7904**	**865**	**1.2538**	**0.4919**
	0.2416	**0.06847**	**0.7913**	**864**	**1.2535**	**0.4919**
	0.2415	**0.06850**	**0.7915**	**862**	**1.2533**	**0.4910**
	0.2413	**0.06852**	**0.7918**	**860**	**1.2532**	**0.4908**

Ultrasonic velocity, density and viscosity for the acetone-propanol-2 and chloroform have been listed in table 2.The appropriate conversion of CGS units to SI units have been provided in Table 1.

It is seen from that at 350 C ultrasonic velocity (U) increases with increasing concentration of the solution which attains a maximum value at 0.7900 mole fractions. The non- linear variation of ultrasonic velocity with concentration indicates occurrence of complex formation between unlike molecules. The molecular association becomes maximum at those concentrations where velocity maxima occurs. This may be interpreted due to the formation of strong hydrogen bonding resulting into complex formation producing displacement of electrons and nuclei. The chemical interaction may involve the association due to hydrogen bonding, due to dipole –dipole interaction or due to the formation of charge transfer complexes. All these processes may lead to strong interaction of forces.(fort and Moore,1965). The density and viscosity of the tertiary solution decreases with decrease in concentration of the solution. The study shows that the mixture can be used in agriculture, industry and hospitals as a medicine at different temperatures to the some extent .Major drawback is that the solution mixture can not be used at higher temperature because of the less interaction between the solvent solvent molecules.

V. CONCLUSION

It is observed that when the reaction mixture is heated at 35,40 & 450 C the Ultrasonic Velocity increases up to certain concentration (0.7900 Mole Fraction)but the density and viscosity decreases as the concentration decreases. This indicates that the complex formation is takes place between unlike molecules. Here the strong hydrogen bonding results into complex formation. Due to dipole-dipole interaction association of water molecules takes place easily.

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Organic Synthesis By natural Catyalyst : An Eco-friendly

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ABSTRACT

In present study the natural catyalyst is the main component in the development of eco-friendly compound and eco-friendly reaction nature. We have to study the natural catyalyst is used for synthesis of organic compound because content of main catyalyst (citric acid) is present in natural catyalyst, that's why natural catyalyst (Citrus Limon) is used in reaction of Amidoalkyl Naphthol. Citrus Limon catalyzed the reaction efficiently without using any other harmful organic reagents or solvents moreover this method have the advantages of shorter reaction time solvent-free condition easy workup and cost effective. The demand of eco-friendly benign procedure promoted us to develop a safe alternative method for the synthesis of amidoalkyl naphthol.

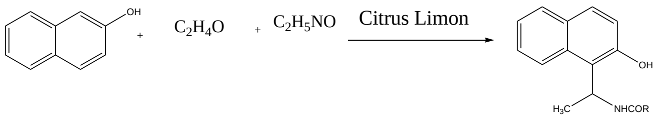
Keywords : Citrus Limon, amidoalkyl naphthol, One-Vat synthesis, Environmentaly-free condition.

I. INTRODUCTION

One vat multi-component reaction have attracted a considerable attention in organic synthesis as they can produce the target products in a single operation without isolating the intermediates and thus reducing the reaction time and energy Multi component contribute to the requirement of an ecofriendly process by reducing number of synthetic step, energy consumption, less amount of solvent or no. of. solvent and waste material production. One such example is the synthesis of 2-amidoalkyl 2-methyl naphthol. Compound bearing 1, 3 amino oxygenated functional group are ubiquitous to a variety of biologically important natural product and potent drug including a no of nucleosides antibiotic and HIV protease inhibitors such as ritonavir and lipinavir Amino alkyl naphthol have attracted strong interest to their useful biological and pharmacological properties such as adrenoceptor blocking, antihypertensive, and say Ca2^{+} channel blocking activities. Amidoalkyl methyl naphthol are also important synthetic building block and are used as precursor for the synthesis of 2-aminomethyl-2-naphthol derivatives, which exhibit important cardiovascular activity. The hypotensive and bradycardiac affects of these compounds have been evaluated.

Therefore, to overcome these limitations the discovery of new eco-friendly and easily available catalyst with high catalytic activity and short reaction time for the preparation of amidoalkyl naphthol is still desirable. The demand of eco-friendly benign procedure promoted us to develop a safe alternative method for the synthesis of amidoalkyl naphthol

Reaction



II. METHODS AND MATERIAL

Experimental

General procedure for the synthesis of amido alkyl napthol-

To a mixture of 2-naphthol 0.5mmol, aldehyde 0.5mmol, amide 0.5mmol and citric acid 5mol %was added the mixture was stirred at 120^{0}c in an oil bath. After completion of reaction the crude product was cooled to room temperature and wash with ice-cold water and stirred well. The catalyst is soluble in water and was remove from the reaction mixture. The pure product was obtained by recrystallization using ethyl alcohol.

III. RESULTS AND DISCUSSION

For our initial study reaction of acetaldehyde, acetamide, 2-naphthol and Citrus Limon as a catalyst was consider as a standard model reaction. Model reaction in the absence of catalyst did not lead to product formation. It means intervention of a catalyst was must for initiation of the reaction. So the catalytic activity of Citrus Limon as an Organopromotor was investigated on the model reaction under environment free condition. To evaluate the temperature effect on reaction rate, the model reaction was performed at different temperature 118^0 C, 120^0 C, 82^0 C, 85^0 C temperature of 120^{0}c found to carry out the reaction efficiently in 90 % yield. Any further increase in temperature failed to enhance the reaction rate substantially, while lowering the temperature below 120^{0}c slow down the reaction rate. To know the exact requirement of catalyst for the transformation , we investigated the model reaction using different concentrations of citric acids such as 0.5% and 5% of catalyst was found to be optimum. Decreasing the amount further did not improve the yield.

IV. CONCLUSION

In summary, we have developed an efficient, mild and clean synthetic method for the synthesis of amidoalkyl naphthols. In this method, attempt has been made to exploit catalytic activity of Citrus Limon in organic synthesis. Citrus Limon catalyzed the reaction efficiently without using any other harmful organic reagents or solvents moreover this method have the advantages of shorter reaction time solvent-free condition easy workup and cost effective.

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Chemical Analysis of Faunal Remains in Middle Bhima Basin, Maharashtra

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ABSTRACT

Bhima River is major left side tributary of Krishana Basin. It's originated from Bhima Shankar in Sayyhadri Hills of Western Ghats at Ambegaon Tehsil of Pune district. Upper reaches area is parts of Pune and Satara district, Middle reaches area is parts of Solapur, Satara, Ahmednagar, Osmanabad, Sangali district of Maharashtra and tributary's of Bhima river, Sina, Man, Bori and Bor river. Lower reaches part is Bijapur and Kalubargi (Gulbarga), Bidar district of Karnataka. During the Geo-archaeological exploration of Middle reaches of Bhima Basin faunal remains Animal fossils, Teeth recovered from river bed, bank and dug-well. Florin, Phosphorous ratio absolute dating method are used for date of faunal remains. To understand the details of the paleoenvironment, apart from the other type of studies, Fluorine dating method is applied to understand the period of deposition. The result of this technique indicates Pleistocene Holocene climate in which the fossil is preserved in the archaeological sediment section. The significant conclusions of the bones from archaeological sites can be dated to the Late Holocene when ratio is less than 1. 2. The ratio increases to 2 or 3 in bones from the Early Holocene to terminal Pleistocene period. 3. The ratio varies from 3 to 6 in bones of the Upper Pleistocene period. 4. In the case of bones from late Middle Pleistocene and earlier periods, this ratio varies from 6.5 to 9.0. Fluorine saturation occurs beyond the late Middle Pleistocene; the theoretical saturation value of this ratio is 8.92. The present paper is deals with the multidimensional studies of the archaeo-sediment which required for the reconstruction of the paleoenvironment of the area (Kshirsagar A.A. and B.C. Deotare, 2001).

Keywords : Chemical Analysis-Faunal-Bhima Basin

I. INTRODUCTION

During the course of field work undertaken in 2015, about a dozen Quaternary sites were brought to light in the middle reaches of the Bhima basin of Maharashtra. It may be mentioned that parts of the Bhima Valley (especially the upper reaches) have received lot of attention during the last three decades or so after the initial work in the upper reaches of the Bhima Valley at Inamgaon on the Ghod, Morgaon (Karha river), Bori (Kale, 1989, Korisettar, 1989, Venkatesan, 1988)(Kuakdi river) etc. The present work represents the first detailed study in the middle reaches of the Bhima which has brought to light a lot of faunal and cultural material, geological and geomorphological data and other aspects of the Quaternary geology. As mentioned earlier, the faunal and cultural material have confirmed the presence of man during the Palaeolithic and Mesolithic times in the area (Kajale *et al.,* 1976; Kshirsagar, 2015;

Kshirsagar, *et al.;* 2015; Kshirsagar, *et al.*; 2016; Kshirsagar, 2016; Sabale, 2015)

Study Area

The main river in the district is the Bhima, with the Nira and Man being the right-bank and the Sina and Bori the left-bank feeders. The Bhima and the Sina flow with a roughly south-easterly trend while the Nira runs east and the Man north-easterly. During the summer season, all these rivers generally remain dry because their source areas fall under severe chronic draught prone region viz. Balaghat for Sina on the left corridor and Mahadeva from Nira and Man along the right corridor of Bhima river middle reaches. However, during the peak of south-west monsoon season, not only the main streams but also the seasonal feeder streams are flooded, though for a short span of time. These bring huge volumes of coarse material inclusive of gravels and cobbles from the barren uplands and cover the shallow beds of the streams quite extensively.

Aims and Objective

To reconstract cultural sequence middle Bhima basin

To understand subsistant pattern of study area

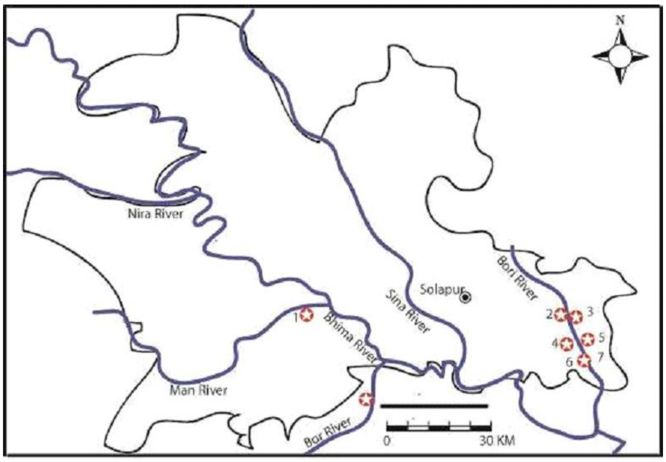
To date the biologicl cultural material

Research Methodology

Survery of river bed, bank and dug-wells of Bhima river and its tributory

To collect faunal material from the study area

To use releative dating method of Flourin/Phosphouous for animal bone and fossil



1. Mudhavi 2. Gaudgaon locality -A 3. Gaudgaon locality -B 3. Umbargi locality -A 4. Umbargi locality- A 5. Umbargi locality - B 6. Hallali locality - A 7. Hallali locality- B

Fig. 1 Location map of site

I- The Bori River

The following are the main sites on the river.

a. Halalli Locality A (N° 17° 25’13” E 76° 54’19”), 11 km South of Akkalkot: yielding cleavers, flakes, scrapers, from dug-well section; along right bank of Bori river
b. Halalli Locality B (N 17° 26’ 01” E 76° 17’05”), 13 km SE of Akkalkot: yielding flake, scrapers, fossils from dug-well section; along left bank of Bri river section
c. Umbargi Locality A (N 17° 29’49” E 76° 14’26”), 5 km SE of Akkalkot: yielding flakes, from dug-well section; along left bank of Bori river
d. Umbargi Locality B (N 17° 56’58” E 76° 14’39”), 6 km SE of Akkalkot: yielding flakes, from dug-well section; along left bank of Bori river

The Sites:

Fig. 1: Fossil sites of the study area (1. Mudhavi

MDV 22	**N 17° 35’ 17”**	**Part of limb bone**	***Bos. namadicus***	**Dug-well**
MDV 11	**E 75° 28’ 20’’**	**Molar**	**Bos sp**	**River bed**
MDV 12		**Last molar**	**Bos sp**	**River bed**
MDV 14		**A shaft of limb bone**		**Dug-well**

2. Gaudgaon Locality-A, 3. Goudgaon Locality-B, 4. Umbergi Locality- A, 5. Umbargi Locality- B, 6. Hallali Locality-A, 7. Hallali Locality- B)

The new sites are Mudhavi on the left bank of Man River in Mangalwedha Tehsil, district Solapur, Umbargi Locality- A, Umbargi Locality- B, Hallali Locality- A and Hallali Locality- B on Left bank of Bori river in Akkalkot Tehsil, district Solapur (Fig.1).

Umbrage Locality-B: Location of the dug well, 5 km south east from the Akkalkot- Manindargi road near the Bori River Bridge and Survey of India toposheet no. 56 C/ 3, 4, 6 and 7 on 430 MSL. Lower Palaeolithic stone tools (Cleavers) are recovered from this gravel deposit.

UBR 11: This is a well preserved upper or second molar of *Equus namadicus*. The crown is well developed and displays all the equine features. Anterior and posterior fossetts on the crown are rectangular in shape with crenulated enamel. Protocone is typically compressed and saucer shaped. All the styles, metastyle, mesostyle and parastyle are prominent with deep interstylar faces.The tooth is hypsodont, however part of the crown is broken (Fig. 2)

UBR 11A: This is a fragment of the limb bone with an effort to be made in to a tool by chipping of a portion which has been sharpened to make it into a sort of scraper (Fig. 2).

Halalli Locality- A: The site is located on the left bank of Bori river, 16 km south from the Akkalkot tehsil in Survey of India toposheet no. 56 C/7 on 420 MSL. Lower Palaeolithic tools, scrapers, flakes and cleavers made on basal rock have been discovered from the Dug-well gravel.

Stratigtraphy of the Dug-well is 5-6 m. cement construction of sediment and 6- 15 m. thick deposit of compact silty sand mixed with calcium carbonate bands. 15-17 m. thick deposit of river conglomerate, pebbly-boulder gravel rests on the basal bed rock. Palaeolithic tools are recovered from gravel which is spread around the dug well (Fig.).

Halalli Locality- B: This site is located on the Bori river bed, 4 km north from present Halalli village and 12 km south east from Akkalkot tehsil in Survey of India toposheet no. 56 C/7 on 430 MSL. Dug-well material of brown sandy-pebbly gravel is spread around the dug well area. Palaeolithic cultural materials of flake tools are recovered from this gravel. The dug well deposits are present in the second order small stream of Bori River. Lower Palaeolithic cleaver tools (Plate-3.5 a b) are recovered from this gravel. Microlithic tools are recovered from the river bed sandy pebbly gravel. Altitude is 420 AMSL and SOI toposheet number 56C/7.

Hallali Locality- A: A Dug-well was observed south of Halalli on the opposite bank of river Bori. Palaeolithic cultural material, stone tools such as scrapers, cleavers, flakes etc. were found at the bottommost layer of river conglomerate of unsorted round shaped material. The depth of tool bearing bed from the surface is approximately 20-22 m. This clearly indicates the high flood episodes in the upper reaches of Bori river.

HLI 101 and 102: These two fragments belong to a tusk portion of an elephant, *Elephas* . The incomplete nature of the specimens does not enable us to specify the species. However, the associated material with the fossils are cleavers, handaxes, flakes and cores of the Lower Palaeolithic culture and hence we may consider the elephant fragments as belonging to *E. hysudricus* from a comparative study of similar artefacts found in the neighboring areas.

The layering of the dentine in the specimens HLI 102 is straight indicating that it might belong to upper part of the tusk, whereas in HLI 101 the lineation is circular, as generally is the case towards the tip (Fig. 3).

Specimen HLI-104 is a heavily mineralised part of the calcaneum of a mammal. The facet for the attachment of astragalus in eroded. The specimen has a rounded shape on account of erosion. The specimen seems to have been transported for some distance hence the shape. Presence of superficial linear cracks on the surface indicates exposure to sunlight and also effect of water current (Fig. 4).

Relative Dating Of Bones Found In Bhima, Man and Bori River Basin

Providing chronological framework to archaeological findings has one of the primary goals of interdisciplinary science of archaeological chemistry. Bones is an excellent material for dating by using a method of fluorine dating, which has become a standard procedure in archaeological chemistry today

(Joshi 2006-07). The examination of fluorine content in bones was initially undertaken by Morichine over two centuries ago in England. Towards the end of 19th century Carnot in France also examined the method of fluorine dating. (Middleton 1844) In India, collective efforts of the (Late) R.V. Joshi, V.d. Gogte, and A.A. Kshirasagar have given a new dimension to the subject of relative dating. The hypothesis is that there is a marked difference in the fluorine contents of bones from prehistoric to historical period, implying that the amount of fluorine in bones goes on increasing with antiquity. During the exploration around the Bhima, Man, Bori, Bor and Sina River Dr. Shivaji Dadaso Kshirsagar has found fossilized and semi fossilized bones. It was not possible to securely date of these sites in the absence of datable material and statigraphy. Bones from these sites were analyzed to put them into proper chronological framework by using relative dating technique by fluorine method.

II. CONCLUSION

Fluorine and Phosphate analysis of these samples give the ratio (100F/P2O5) in between the range of 01.31 to 15.03 (Table 1). UBR-11, Ratio ranges is 01.31 which gives the dates at Early Holocene period. MDV-14, Ratio ranges is 05.06 which gives the dates at Upper Pleistocene period. HLI-102, HLI-104, MDV-22 Ratio ranges is 7, 7.03, 7.96 which gives the dates at Late Middle Pleistocene and earlier periods. Ratio ranges is MDV-11, 11.31, MDV-12, 14.49, HLI-101, 15.03 Pleistocene and earlier period. Dr. Anupama Kshirsagar has proposed a chart correlating the fluorine values in bones with dates obtained by other dating method; it becomes clear that the bones in this study fall in the relative time scale covering a small to long time span of late to Mid Holocene period to Late Middle Pleistocene and earlier periods. Analysis indicates that these bones are semi fossilized to well fossilize. Amount of fluorine accumulation in the bone is very less to high.

This note summaries the discovery of about a dozen Quaternary sites in the Bhima basin of Maharashtra. The work represents a first detailed study of the various aspects of geology and archaeology in the region and throws important light on various areas enriching our knowledge of Quaternary aspects of the region as a whole. Several new sites discovered in this survey have added to our understanding of the prehistoric cultures in this part of India.

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Table 1. Fluorine and Phosphate analysis

Sr. No.	Site Name	% Phosphate	% P2o5	% Fluorine	Ratio 100x F/p2o5	Age
1	HALLALI =HLI-104	9.11	20.4	1.6	7.84	Late Middle Pleistocene and earlier periods
2	MUDHAVI =MDV-12	8.5	19.04	2.76	14.49	Pleistocene and earlier periods
3	MUDHAVI =MDV-11	8.24	18.21	2.06	11.31	Pleistocene and earlier periods
4	HALLALI =HLI-101	7.75	17.21	2.62	15.03	Pleistocene and earlier periods
5	MUDHAVI =MDV-22	12.5	27.62	2.2	7.96	Late Middle Pleistocene and earlier periods
6	MUDHAVI =MDV-14	10.1	22.32	1.13	5.06	Upper Pleistocene period
7	HALLALI =HLI-104	12.1	26.74	1.88	7.03	Late Middle Pleistocene and earlier periods
8	HALLALI =HLI-102	11.5	25.41	1.78	7	Late Middle Pleistocene and earlier periods
9	UMBARGI =UBR-11	10.3	22.76	0.3	1.31	Early Holocene

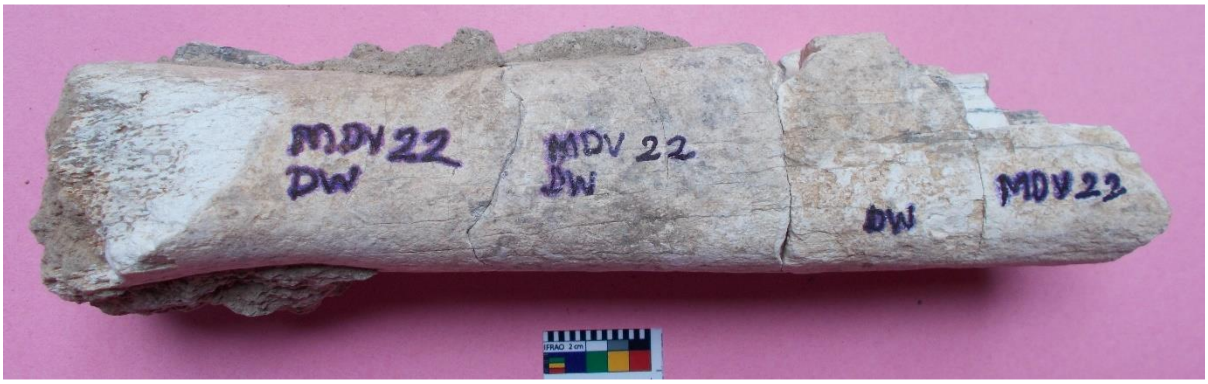

Fig. 1a MDV-22 Part of Limb Bone of Bos. Namadicus

Fig. 1b MDV -12 Last Molar Bos. sp., MDV-11 Molar Bos. sp.

Fig. 1c MDV-14,

Fig. 2 UBR-11 Upper or Second Molar of *Equus namadicus,* UBR-11A Fragment of the Limb Bone,

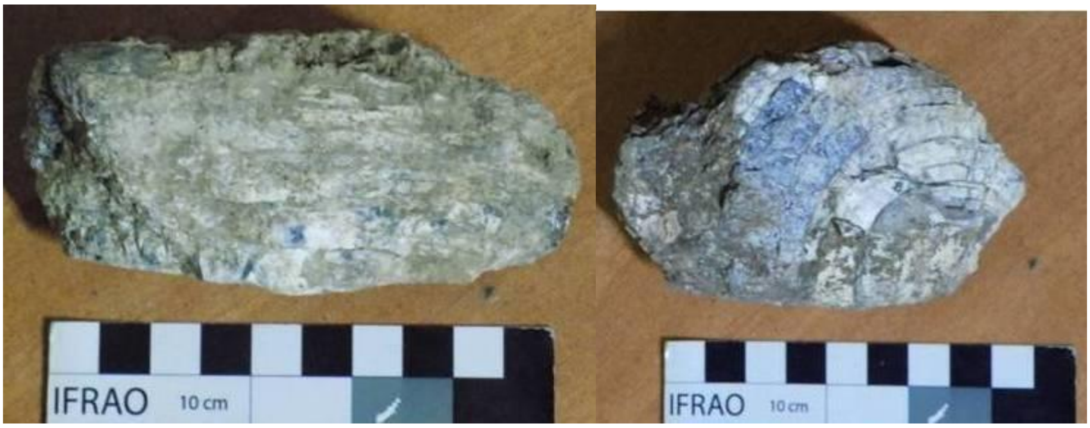

Fig. 3 HLI 101 and 102: These two fragments belong to a tusk portion of an elephant, *Elephas*

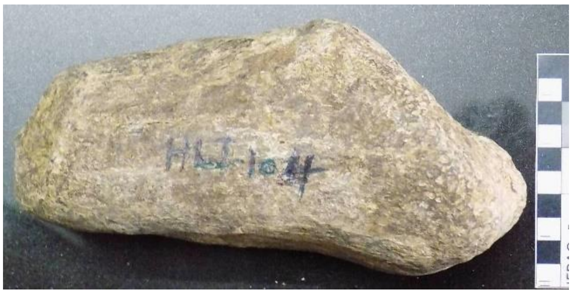

Fig. 4 HLI-104 is a heavily mineralised part of the calcaneum of a mammal.

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Analytical study of ground water from Sangamner Taluka of Ahmednagar District Maharashtra, India

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ABSTRACT

Water is very important for the entire life on earth. Like surface water, ground water is a source of water in many parts of the world. It's very necessary to assess the ground water quality for its use. In order to diagnose the water quality for consumption of earthling the ground water samples from Sangamner Tehsil region were analyzed. Total nine samples were examined with parameters such as p^H, EC and T.D.S. The Ca, Mg, HCO_3, CO_3 were determined titrametrically and Na by flame photometer. The result obtained were compared with the Indian standard drinking water – specification IS 10500-1991 (Reaffirmed-2009) and also the standards declared by WHO (2005). The selected region is agro-developed area, that being the case the chemical fertilizers oozes through soil and contaminate the ground water increasing the salinity, TDS, and other physicochemical properties of soil. The consequences of such contamination are acute along with chronic diseases. The result showed that the P^H in the range of 7.61 to 8.48, Electrical conductivity from 370 to 2680, TDS 317 to 2460, Sodium 42 to more than 100, Calcium was in the range of 84.4 to 295.6, Magnesium 19.4 to 269.4, HCO_3^- 42 to 530, carbonate 80 to 640. Total hardness, Alkalinity, Nitrite, Sulphate, Silica, Chloride and Fluoride were also determined by using standard methods. Total Hardness was found from 400 to 1950, Alkalinity 130 to 400, Nitrite 15 to 25, sulphate 50 to 110, Silica 75 to 125, Chlorides 50 to 800

(N.B.: All concentrations are reported in mg/l except p^H and EC (μS/cm) and Na in ppm.)

Keywords: Physicochemical analysis, MIDC, TDS

I. INTRODUCTION

It is important to have reliable source of water for agriculture, drinking and domestic purpose. Surface water and ground water are the major sources of water on earth. Use of ground water is depends on its quality. Considering the importance of water, it is necessary to focus on protection and management of ground water quality. Review of literature reveals that Several researchers has carried out work on ground water in India and other countries. Behailu et al 2017 has carried out analy6sis of physical and chemical parameters in ground water in Ethiopia. By using groundwater quality index and pollution index study of characteristics of ground water in Basara province were studied by Dawood et al 2018. Kotagi and Sadhya Kiran 2018 analysied the ground water. Study on physicochemical characteristics of ground water samples at ganga region in Allahabad was done by Sebastein et al 2018. .Madhusudan et al 2017 has done physicochemical analysis of ground water in the selected area of Vikhakhapatnam in Andhra Pradesh.

Physicochemical analysis of ground water of selected areas in Raipur city was done by Jena and Sinha 2017.Ashiyani et al 2015 has analysied the physicochemical properties of ground water from Matar Taluka of kheda district. Physicochemical analysis of ground water of village Jalalabad was carried out by Bhatti et al 2016.Patel et al 2016 has done physicochemical analysis of ground water quality of Dhrol. Moayedi et al 2011 has carried out ground water quality assessment of Labuan island. Assessment of ground water suitability for drinking purpose from narangal was done by Patil et al 2018.Bansal and Dwivedi 2018 assesed the ground water quality by using quality index and physicochemical parameters.

II. METHODS AND MATERIAL

We randomly collected Nine ground water sample from wells, from some villages of Sangamner tehsils in Nov. 2018. Samples are collected in clean polythene bottles. The parameter such as pH, EC and T.D.S. were analyzed immediately. The Ca, Mg, HCO_3, CO_3 determined titrametrically and Na by flame photometer.

Observation Table:

Table 1. Table Showing Collection Spots

Sampling Code	Sampling Station	Sampling Code	Sampling Station
S-1	Ghulewadi	S-6	Jawale Kadlag
S-2	Sangamner MIDC	S-7	Ganore
S-3	Velhale	S-8	Vadgaon Landga
S-4	Gunjalwadi	S-9	Pimpalgaon Konjire
S-5	Rajapur		

Charts:

Table 2. Table Showing Physicochemical Parameters

No.	pH	EC	TDS	Na	Ca	Mg	HCO_3	CO_3	Total Hardness	Alkalinity	Nitrite	Sulphate	Silica	Cl^-	F^-
S-1	8.28	2680	2460	>100	295.6	269.4	530	162	1950	130	15	80	125	800	16
S-2	8.14	280	240	>100	84.4	19.4	308	220	400	160	25	60	75	50	9
S-3	8.03	1540	1440	70	221.1	121.8	120	80	1300	180	15	100	75	350	11
S-4	7.98	560	469	58	100.3	60.94	42	400	450	300	15	50	75	100	5
S-5	7.61	1290	1090	89	158.4	96.22	240	320	950	220	15	110	75	230	4
S-6	8.39	1170	984	53	110.8	80.19	320	640	850	400	25	80	75	300	5
S-7	7.95	1080	892	89	105.6	168.9	85	260	750	300	20	60	75	300	8
S-8	8.48	1050	870	24	121.4	150.8	45	90	800	300	15	90	100	250	7
S-9	8.40	370	317	42	105.6	105.8	260	560	450	300	25	70	75	200	8

Table 3. Table showing standard range of the minerals

Parameter	Indian standard drinking water – specification IS 10500-1991 (Reaffirmed-2009)		Sample exceeding permissible limit	Range of sample	
	Desirable limit	Permissible limit	%	Min	Max
TDS mg/lit	500	2000	9	100	2600
Ca mg/lit	75	200	54.54	64	529.6
Mg mg/lit	30	100	100	108.8	690
TH (as$CaCO_3$ mg/lit)	300	600	27.27	80	720

Table 4. Table showing standard range of Physicochemical parameters

Water quality parameters	Units	WHO(2005)		No. Of sample exceeding allowable limit	Percentage of sample exceeding limits	Undesirable effects
		Desirable Limit	Maximum Limits			
pH	-	7-8.5	9.2	-	-	Taste
EC	µS/cm	1000	2000	2	18	Gastrointestinal irritation
TH	mg/L	300	500	3	27	Scale Deposition
Cl^-	mg/L	200	600	2	18	Salty Taste
Ca^{2+}	mg/L	75	200	6	54.54	Scale formation
Mg^{2+}	mg/L	50	150	11	100	Encrustation in water supply structure
Na^+	mg/L	-	200	5	45	Salinity
TDS	mg/L	500	1500	4	36	Gastrointestinal irritation

III. RESULTS AND DISCUSSION

The result from analysis is predicted in above charts. We can see S-1 and S-3 has more percentage at Ca^{2+} and Mg^{2+}. This causes intestinal problems, Kidney stone. In additional, several epidemiological investigation have demonstrated. The relation between risk of cardiovascular diseases, growth retardation, reproductive failure and other health problems. Cl^- were common constituent at ground water but which is exceeding in Sl cause discharged into septic system

IV. CONCLUSION

From above analysis, we see that, there is lot of contamination in ground water. This contamination is due to exceeding use of chemical fertilizer in agriculture sector. This chemical residue are percolated through rain water and contaminate with ground water. In our analysis we see there is some villages where ground water is not safely use for drinking and agriculture. So we have to focus on its management.

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Biosynthesis of CuO nanoparticles using aqueous extract of Ziziphus mauritiana L. leaves and their Catalytic performance for the 5-aryl-1,2,4-triazolidine-3-thione derivatives synthesis

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ABSTRACT

Copper oxide (CuO) nanoparticles (NPs) were synthesized by biological method using aqueous extract of *Ziziphus mauritiana* L. leaves as a reducing and stabilizing agent. The merits of this procedure are an easy operation, use of cheap, nontoxic and environmentally benign precursors. The structural and morphological properties of the as-prepared nanocatalyst were done by using XRD, SEM, EDX, TEM and BET-surface area. Furthermore, these NPs are used as an effective nanocatalyst for simple and one-pot synthesis of 5-aryl-1,2,4-triazolidine-3-thione derivatives. The reaction steps including imine formation, cyclization, condensation and aromatization occurs, without use of any oxidizing or reducing reagents. The present methodology offers several remarkable merits such as shorter reaction time, mild reaction conditions, excellent yield, simplicity, safer reaction pathway, easy workup and recyclable catalyst without any significant loss in catalytic activity and can be used for large scale synthesis. Hence, this study describing the synthesis of CuO NPs by effective biogenic method followed by the investigation of potent catalytic activities may be useful for nanochemistry research opening new horizons in this field.

Keywords : Nanotechnology, *Ziziphus mauritiana* L., CuONPs, Recyclable catalyst.

I. INTRODUCTION

Nowadays metal oxide NPs has got great attention in catalysis because of their redox properties, coordination environment of surface atom and oxidation state at surface layer. Extensive surface area of nanoparticles and number of active sites than the bulk material, makes NPs more practicable catalyst for organic synthesis.[1–5] High atom efficiency, moderate reaction conditions, simple separation of product and recyclability of the catalysts are the merits of NPs catalyzed reactions.

Prominent applications of CuO NPs generated a great deal of interest in biomedical[6], catalysis[7], lubricant[8] and gas sensing[9]. CuO NPs were synthesized using microwave irradiation, ionic liquid assisted, sol-gel technique, sonochemical, electrochemical and thermal treatment[10–15] are reported recently. The existing methods have some drawbacks due to use of some drastic conditions such as use of toxic chemicals, external additives required during the reaction, high temperature etc. Therefore, there is massive scope for the development of green protocol for the synthesis of CuO NPs. Nowadays, nanomaterial synthesis using plant extract is receiving lot of attention with some advantages over chemical methods.[16–18] The inspection of the literature exposed some plant extract mediated synthesis of CuO NPs by, *Camellia japonica*[19] *Rauvolfia serpentina*[20], *Leucaena* *leucocephala*[21], *Calotropis gigantean*[22], *Aloe barbadensis*[23], *Ficus religiosa*[24], *Albizia lebbeck*[25] and *Acanthospermum hispidum*[26] have been reported.

The synthesis of 5-aryl-1,2,4-triazolidine-3-thione derivatives was carried out by using synthesized CuO NPs. Literature survey reveals that the sulfur containing 1, 2, 4-triazole have been reported, as antidepressant[27,28], anti-tubercular[29], antibacterial[30,31], anti-HIV[32], antimicrobial activity[33] and antifungal activity[33]. The biological applications of, 5-aryl-1,2,4-triazolidine-3-thione derivatives have fascinated attention of many researchers. Analysis of literature exposes that the 5-aryl-1,2,4-triazolidine-3-thiones involved the use of [C_{16}MPy]$AlCl_3Br$[34], [2-HMPyBSA]HSO_4[35], PEG-400[36], glycine nitrate[37], Sm_2O_3/FAp[38], Fe-FAp[39], sulfamic acid[40], DMAP[41], [$(Py)_2SO$][HSO_4]$_2$[42] for the synthesis.

The present study we report CuO NPs catalyzed one pot methodology that comprises both, condensation and cyclization of aldehyde with thiosemicarbazide. CuO NPs assisted synthesis improves the rate of reaction towards 5-aryl-1,2,4-triazolidine-3-thione.

II. METHODS AND MATERIAL

2.1 Characterization techniques

The morphology and elemental composition of the fabricated CuO NPs were examined by field emission scanning electron microscopy (FESEM, FEI, Nova Nano SEM 450), FESEM coupled energy-dispersive X-ray spectroscopy (EDX, Bruker, XFlash 6I30). Find the exact morphological structures and size of the CuO NPs using transmission electron microscopic (TEM) analysis is done by using a PHILIPS-CM200. The crystallinity and crystal phases were characterized by X-ray diffraction (XRD, Brukar, D8-Advanced Diffractometer) pattern measured with Cu- Kα Radiation (λ= 1.5406 Å) in the range of 5–90°. These all characterization results have been reported in our previous research work[43].

2.2 General procedure for the synthesis of 5-phenyl 1,2,4-triazolidine 3-thione derivative.

Equimolar mixture (10 mmol) of aldehyde and thiosemicarbazide were mixed with CuO NPs (5 mg) in ethanol (5 mL) taken in a round bottom flask. The reaction mixture was then heated to 70 °C for appropriate time. After completion of the reaction, the reaction mixture was diluted with the ethyl acetate and the catalyst was separated by centrifugation. The crude product was then purified by recrystallization to get pure product.

2.3 Spectral data of synthesized compounds

5-Phenyl -1,2,4-t riazol idine-3-thione (3a):
White solid, Mp 152-154 ˚C; 1H-NMR (500 MHz, DMSO-d6): δ 11.49 (s, 1H, NH), 8.22 (s, 1H, NH), 8.04 (s, 1H, CH), 8.0 (s, 1H, NH), 7.80 (d, 2H, ArH, J = 6.8 Hz), 7.39 (t ,3H, ArH, J = 6.8 Hz); LC-MS- 179.24

5-(4-Nitrophenyl)-1,2,4-triazolidine-3-thione (3b):
Yellow solid, Mp. 226-228 ˚C; 1H-NMR (500 MHz, DMSO-d6): δ 8.09 (s, 1H, CH), 8.11-8.13 (d, 2H, ArH, J = 10 Hz), 8.22-8.23 (d, 2H, ArH, J = 5Hz), 8.26 (s, 1H, NH), 8.41 (s, 1H, NH), 11.71 (s, 1H, NH); LC-MS-223.1

5-(3-Nitrophenyl)-1,2,4-triazolidine-3-thione (3c):
Yellow solid, Mp. 226-228 ˚C; 1H-NMR (500 MHz, DMSO-d6): δ 7.67-7.70 (t, 1H, ArH), 8.15 (s, 1H, CH), 8.20-8.22 (dd, 1H, ArH, J = 10 Hz), 8.24-8.25 (d, 1H, ArH, J = 5 Hz), 8.31 (s, 1H, ArH), 8.33 (s, 1H, NH), 8.66 (s, 1H, NH)11.62 (s, 1H, NH); LCMS- 223.1

5-(4-Chlorophenyl)-1,2,4-triazolidine-3-thione (3d):
White crystal, Mp. 206-208 ˚C;1H-NMR (400 MHz, DMSO-d6): δ 7.46 (d, 2H, ArH, J = 8.4 Hz), 7.84 (d, 2H, ArH, J = 8.4 Hz), 8.02 (s, 1H, CH), 8.08 (s, 1H, NH), 8.24 (s, 1H, NH), 11.49 (s, 1H, NH) ; LCMS-214.1

5-(4-Bromophenyl)-1,2,4-triazolidine-3-thione (3e):
White crystal, Mp. 202-204 ˚C; 1HNMR (400 MHz, DMSO-d6): δ 7.57-7.605 (d, 2H, ArH, J = 8.7 Hz),

7.75-7.78 (d, 2H, ArH, J = 8.4 Hz), 8.00 (s,1H, CH), 8.08 (s, 1H, NH), 8.24 (s, 1H, NH), 11.49 (s, 1H, NH); LCMS-258.1

5-(4-Fluorophenyl)-1,2,4-triazolidine-3-thione (3f):
White crystal, Mp. 172-174 ˚C; 1H-NMR (400 MHz, DMSO-d6): δ 7.23 (d, 2H, ArH, J = 8.4 Hz), 7.85 (d, 2H, ArH, J = 8.4 Hz), 7.88 (s, 1H, CH), 8.03 (s, 1H, NH), 8.20 (s, 1H, NH), 11.43 (s,1H, NH); LCMS-196.1

5-(o-Tolyl)-1,2,4-triazolidine-3-thione (3g):
White crystal, Mp. 174-176 ˚C; 1H-NMR (400 MHz,DMSO-d6): δ 2.37 (s, 3H, CH3), 7.18-7.22 (d, 2H, ArH, J = 8 Hz), 7.25-7.27 (d, 1H, ArH, J = 8 Hz), 7.89 (s,1H, ArH), 8.02-8.05 (t, 1H, ArH, J = 8 Hz), 8.202 (s, 1H, NH), 8.393 (s, 1H, NH), 11.34 (s, 1H, NH); LCMS-192.2

5-(4-Methoxyphenyl)-1,2,4-triazolidine-3-thione (3h):
White crystal, Mp. 156-158 ˚C; 1H-NMR (400 MHz, DMSO-d6): δ 3.78 (s, 3H, OCH3), 6.94-6.96 (d, 2H, ArH, J = 8.8 Hz), 7.71-7.74 (d, 2H, ArH, J = 7.6Hz), 7.92 (s, 1H, CH), 7.98 (s, 1H, NH), 8.11 (s, 1H, NH), 11.32 (s, 1H, NH); LCMS-210.1

5-(3,4-dichlorophenyl)-1,2,4-triazolidine-3-thione (3i):
White crystal, Mp. 174-176 ˚C; 1H-NMR (400 MHz, DMSO-d6): 7.629-7.681 (d, 2H, ArH, J = 8 Hz), 7.703-7.729 (d, 1H, ArH, J = 8 Hz), 7.89 (s,1H, ArH), 8.02-8.05 (t, 1H, ArH, J = 8 Hz), 8.202 (s, 1H, NH), 8.393 (s, 1H, NH), 11.34 (s, 1H, NH); HRMS-247.918

5-(2-pyridinyl)-1,2,4-triazolidine-3-thione (3j):
Yellow solid, Mp. 174-176 ˚C; 1H-NMR (400 MHz, DMSO-d6): 7.36-7.39 (d, 1H, ArH, J = 8 Hz), 7.80-7.84 (t, 1H, ArH, J = 8 Hz), 8.09 (s,1H, CH), 8.18 (s, 1H, NH), 8.27-8.29 (d, 1H, ArH, J=8Hz), 8.36 (s, 1H, NH), 8.55-8.57(d, 1H, ArH J =8Hz) 11.49 (s, 1H, NH); LCMS-179.1

5-(4-pyridinyl)-1,2,4-triazolidine-3-thione (3k):
Yellow solid, Mp. 174-176 ˚C; 1H-NMR (500 MHz, DMSO-d6): δ 7.78-7.79 (d, 2H, ArH, J = 5 Hz), 8.59-8.60 (d, 1H, ArH, J = 5 Hz), 8.01 (s,1H, CH), 8.22 (s, 1H, NH), 8.40 (s, 1H, NH), 11.69 (s,1H, NH); LCMS-179.1

5-(2-thienyl)-1, 2, 4-triazolidine-3-thione (3l):
Yellow solid, Mp. 174-176 ˚C; 1H-NMR (400 MHz, DMSO-d6): 7.098-7.119 (d, 1H, ArH, J = 12.6 Hz), 7.440-7.451 (d, 1H, ArH, J = 6.6 Hz), 7.557 (s,1H, CH), 7.644-7.656 (d, 1H, ArH J = 7.2Hz), 8.213 (s, 1H, NH), 8.234 (s, 1H, NH), 11.49 (s, 1H, NH); LCMS-184.1

III. RESULTS AND DISCUSSION

3.1 Catalytic activity of CuO NPs.

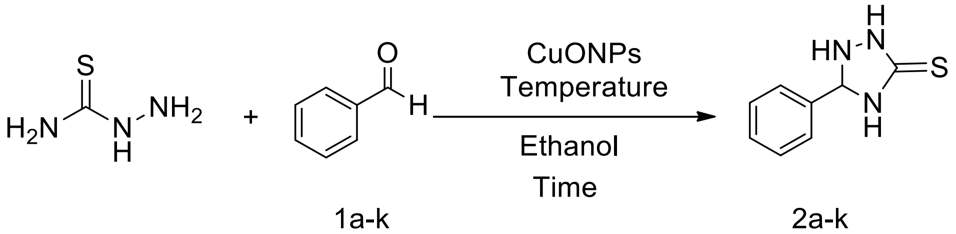
To optimize the reaction condition, the synthesis of 5-phenyl-1,2,4-triazolidine-3-thione (2a) from thiosemicarbazide and benzaldehyde was chosen as a model reaction in ethanol (Table 3.1).

Table 3.1 Effect of CuO NPs loading towards the synthesis of 5-phenyl-1,2,4 triazolidine-3-thione$^{(a)}$

Entry	Amount of CuO NPs (in mg)	Time (in Min.)	Yield$^{(b)}$ (in %)
1	1	10	78
2	2	10	87
3	3	10	89
4	4	10	92
5	**5**	**10**	**95**
6	10	10	97

^{a} Reaction conditions: 10 mmol Thiosemicarbazide, 10 mmol aromatic aldehyde, 5 mg of **CuO NPs**, 5 ml ethanol at 70 ^{0}C. ^{b} Isolated yield.

Table 3.2 CuO NPs catalyzed synthesis of 5-aryl-1,2,4-triazolidine-3-thione[(a)]



Entry	R	Time (Min.)	Product	Yield[(b)]
1	C_6H_5 2a	10	3a	95
2	4-(NO_2) C_6H_4 2b	6	3b	94
3	3-(NO_2) C_6H_4 2c	10	3c	91
4	4-(Cl) C_6H_4 2d	12	3d	94
5	4-(Br) C_6H_4 2e	14	3e	90
6	4-(F) C_6H_4 2f	20	3f	89
7	2-(Me) C_6H_4 2g	10	3g	94
8	4-(OCH_3) C_6H_4 2h	12	3h	95
9	3,4 (Cl) C_6H_3 2i	14	3i	90
10	2-Pyridine 2j	15	3j	93
11	4-Pyridine 2k	18	3k	95
12	2-Thiophene 2l	16	3l	94

[a] Reaction conditions: 10 mmol Thiosemicarbazide, 10 mmol aromatic aldehyde, 5mg of **CuO NPs**, 5 ml ethanol at 70 °C. [b] Isolated yield.

At first 1 mg of CuONPs were used that lead to 78 % of 2a at 70 °C within 10 mints (table 3.1 entry 1). On varying quantity of CuONPs (table 3.1 entries 2-5) yield of 2a were improved suggestively up to 95 % presenting 5 mg of CuO NPs is enough for the transformation to 2a, further increment in amount of CuO NPs doesn't improve the yield of 2a (table 3.2 entry 6). After screening different solvents, the room temperature synthesis of 5-phenyl-1,2,4-triazolidine-3-thione improved yield were obtained with ethanol (Table 3.3)

Table 3.3 CuO NPs catalyzed synthesis of 5-aryl-1,2,4-triazolidine-3-thione[(a)]

Entry	Solvent	Amount of catalyst (mg)	Temperature (^0C)	Time (Min.)	Yield
1	Isopropyl alcohol	5	70	10	88
2	Ethanol	5	70	10	97
3	Methanol	5	70	10	92
4	Water-ethanol	5	70	10	75

Thus, it may be claimed that aldehyde and thiosemicarbazide leading to imine intermediate at the same time undergoes cyclisation due to CuO NPs.

To check the practicability and generality of the CuO NPs, we have studied several substituted aldehydes (1a-l) proficient conversion into the corresponding 5-phenyl 1,2,4-triazolidine 3-thione (2a-l) in brilliant yield were obtained (table 3.2)

Electron donating or withdrawing group bearing aldehydes and five or six membered heterocyclic aldehydes containing one heteroatom were practically converted into the corresponding triazolidine-3-thiones with outstanding yields. Above results, state that there is no substituent effect[36]. The structures of synthesized compound were confirmed by DEPT, ^{1}HNMR, ^{13}CNMR and Mass analysis.

3.2 Reusability Studies

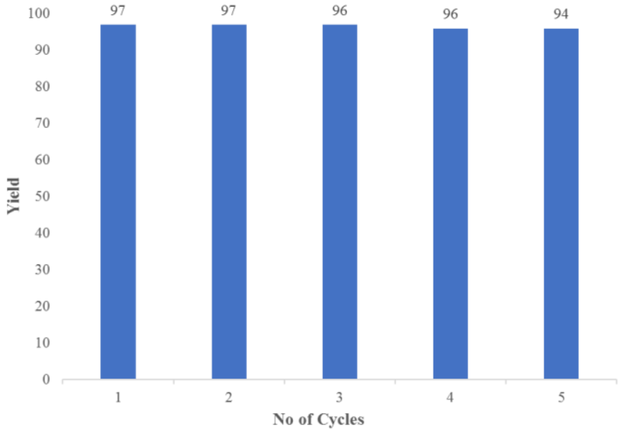


Figure 1. Reusability studies of CuO NPs for the synthesis of 5-phenyl-1,2,4-triazolidine-3-thione (2a)

The synthesis of 5-phenyl-1,2,4-triazolidine-3-thione from benzaldehyde and thiosemicarbazide using CuO NPs as catalyst under optimize reaction condition were adopted as model reaction for reusability study. As the reaction was completed ethyl acetate was added to the reaction mass and the catalyst was separated by centrifugation. Separated catalyst was washed with ethanol and acetone repeatedly, dried under vacuum and then reused for consecutive cycles. The efficiency of catalyst was studied up to five cycles without any significant loss. Figure. 5.8 presents reusability studies of CuO NPs.

IV. CONCLUSION

In present work, the synthesis of CuO NPs using aqueous extract of *Ziziphus mauritiana* L. leaves is developed. The catalytical study of CuO NPs towards the synthesis of 5-phenyl 1,2,4-triazolidine 3-thione derivative gives excellent yield, simple separation of catalyst by just centrifugation. The effectiveness of the catalyst was tested and the loss was negligible up to five cycles.

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Molecular Docking and Virtual Screening of Cefazolin as a Antibiotic Drug

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ABSTRACT

In this research paper Schrodinger software based pharmacophore features of cefazolin mapping and distance involved between 1,3,4- thiadiazole ring and other pharmacophore features of the compound is elaborated. The structure of Cefazolin is observed on Zincpharmer online server to reveal the various intermolecular features for molecular recognition, investigation of binding site (ligand-receptor complexes), characterization of unknown binding sites etc., Computer Aided Drug Design is successfully identified the flexibility of binding sites of target molecule with small ligand based on lock and key model. *In Silico* evaluation based on SBDD and MD is very useful tool to identify the pharmacofeatures and pharmacological significance of molecules before they synthesized. In the published research papers revealed on synthesized derivatives of 1, 3,4-thidiazole and their biological evaluation *in vitro* against NCIM provided Gram positive bacterial strains *Staphallococcus aureus (Sa- 2178) Bacillus subtilis (Bs-2239) Gram negative bacterial strains Eschereschia coli (Ec-25744) Klibesiella aerogenus (Ka-2249).*

Keywords: Molecular Docking, Pharmacophore, Cefazolin, Computer Aided Drug Design

I. INTRODUCTION

A key challenge in the synthesis of bimolecular containing heterocyclic component targets continues to be the development of new pathways and improvement of existing pathways [1]. Docking studies by computer graphics is easiest route rather than mechanical model construction. Macromolecular modeling by docking studies provides most possible view of drug receptor interaction and has created a new rational approach to drug design where the structure of drug is designed based on its fit to three dimensional structures of receptor site rather than by analogy to other active structure or random leads. [2] It is also important to consider that structure based drug design (SBDD) directs the discovery of a drug lead, which is not a drug product but, specifically, a compound with at least micro molar affinity for a drug target [3,4].

1, 3, 4- thiadiazole core containing drugs are currently in the market: acetazolamide®) and methazolamide® are diuretics, acting through inhibition of carbonic anhydrase; their derivatives display additional activities, including anticonvulsant and selective cerebral vasodilation, as well as the anticipated inhibition of carbonic anhydrase, cefazolin sodium® (CFZL; 3) and cefazedon® (CFZD; 4)—first-generation cephalosporins and megazol® an antiparasitic drug. The structure of cefazolin also includes a heterocyclic thiol, 2-methyl-1, 3, 4-thiadiazole-5-thiol (MTD), and this compound can also inhibit the gamma-carboxylation of glutamate. [6-11].

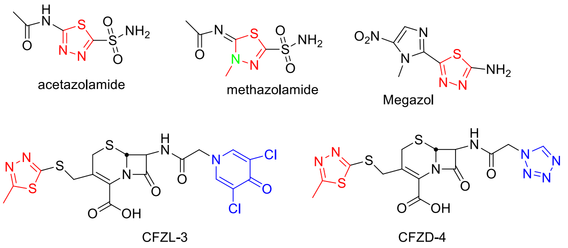

Figure 1. Structures of 1, 3, 4- thiadiazole core containing drugs available in the market

II. METHODS AND MATERIAL

Manual pharmacophore hypothesis generation module of Schrondinger maestro v9.6 was used for pharmacophore features mapping of the compounds along with location and calculation of distance between the pharmacophore features [12]. Computational docking methods, with empirical scoring functions are used to predict binding affinities and ligand orientations inside the binding sites of proteins. While the docking methods give the binding geometries; potential functions follow rules based on the binding affinity statistics. These rules are used to calculate the computer generated ligand orientations "scores" making use of the "pseudo-potentials" [13]. The macromolecule protein and ligand structures as rigid files are imported in the 3D space of the autodock software. Then, the energy scoring grid box was centered with 0.375 angstroms grid points spacing and size of the box was set to 126, 126 and 126 Å (x, y, and z) assigned with default atomic salvation parameters. The grid box was designed such that the whole macromolecule was surrounded by the three dimensional grid box centered. After the grid box fixation, all other required default parameters for grid are assigned and then the file output is saved as grid parameter file (.gpf)

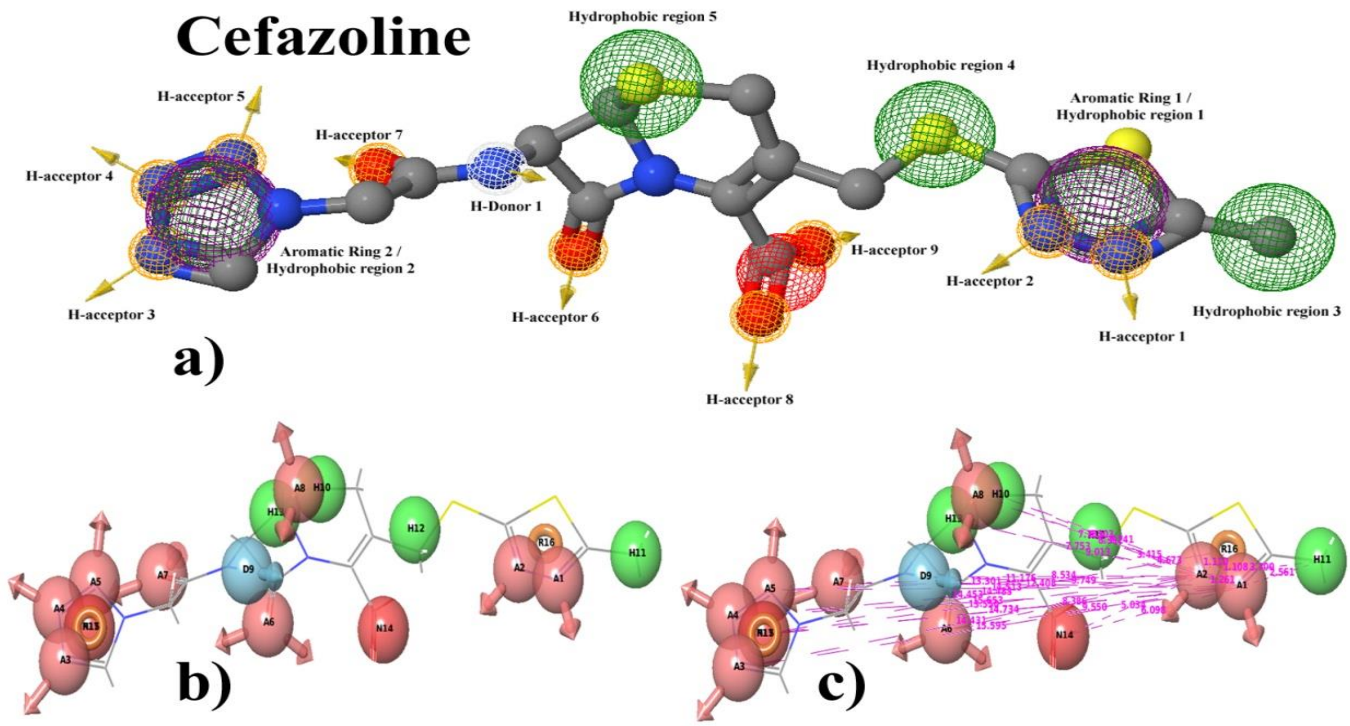

Figure 2 : Pharmacophore features of Cefazoline explained in 3D pharmacophore modeling shown in following images:

a) ZINCPharmer online server based pharmacophore features mapping showing location & direction of two aromatics rings; five hydrophobic regions; one hydrogen donor & nine hydrogen acceptors.

b) Schrodinger software based pharmacophore features mapping and

c) Distance involved between 1,3,4- thiadiazole ring and other pharmacophore features of the compound.

III. RESULTS AND DISCUSSION

Table 1 : XYZ co-ordinates and radius of each pharmacophore feature of Cefazoline

S.No	Site 1	Site 2	Distance in angstroms	Site 1	Site 2	Distance in angstroms
1.	A1	A2	1.261	A5	R16	14.187
2.	A1	A3	15.595	A5	R17	1.103
3.	A1	A4	15.653	A6	A7	3.986
4.	A1	A5	14.513	A6	A8	4.677
5.	A1	A6	9.55	A6	D9	3.77
6.	A1	A7	12.408	A6	H10	4.832
7.	A1	A8	8.392	A6	H11	11.844
8.	A1	D9	9.749	A6	H12	5.54
9.	A1	H10	7.741	A6	H13	3.364
10.	A1	H11	2.561	A6	N14	3.63
11.	A1	H12	4.673	A6	N15	6.847
12.	A1	H13	9.013	A6	R16	9.143
13.	A1	N14	6.098	A6	R17	6.847
14.	A1	N15	14.734	A7	A8	4.94
15.	A1	R16	1.108	A7	D9	3.086
16.	A1	R17	14.734	A7	H10	5.525
17.	A2	A3	14.431	A7	H11	14.826
18.	A2	A4	14.453	A7	H12	7.982
19.	A2	A5	13.301	A7	H13	3.835
20.	A2	A6	8.386	A7	N14	7.202
21.	A2	A7	11.176	A7	N15	3.285
22.	A2	A8	7.152	A7	R16	12.001
23.	A2	D9	8.534	A7	R17	3.285
24.	A2	H10	6.503	A8	D9	2.517
25.	A2	H11	3.7	A8	H10	0.678
26.	A2	H12	3.415	A8	H11	10.697
27.	A2	H13	7.753	A8	H12	3.919
28.	A2	N14	5.034	A8	H13	1.507
29.	A2	N15	13.55	A8	N14	5.158
30.	A2	R16	1.11	A8	N15	7.345
31.	A2	R17	13.55	A8	R16	7.876

32.	A3	A4	1.288	A8	R17	7.345
33.	A3	A5	2.102	D9	H10	3.006
34.	A3	A6	7.61	D9	H11	12.223
35.	A3	A7	4.236	D9	H12	5.498
36.	A3	A8	8.407	D9	H13	2.113
37.	A3	D9	6.118	D9	N14	5.472
38.	A3	H10	9.009	D9	N15	5.127
39.	A3	H11	18.125	D9	R16	9.424
40.	A3	H12	11.541	D9	R17	5.127
41.	A3	H13	7.698	H10	H11	10.028
42.	A3	N14	10.665	H10	H12	3.288
43.	A3	N15	1.112	H10	H13	1.904
44.	A3	R16	15.385	H10	N14	4.82
45.	A3	R17	1.112	H10	N15	7.961
46.	A4	A5	1.287	H10	R16	7.211
47.	A4	A6	7.524	H10	R17	7.961
48.	A4	A7	3.771	H11	H12	6.937
49.	A4	A8	8.04	H11	H13	11.328
50.	A4	D9	5.966	H11	N14	8.32
51.	A4	H10	8.679	H11	N15	17.25
52.	A4	H11	18.15	H11	R16	2.853
53.	A4	H12	11.4	H11	R17	17.25
54.	A4	H13	7.307	H12	H13	4.391
55.	A4	N14	10.697	H12	N14	3.128
56.	A4	N15	1.093	H12	N15	10.585
57.	A4	R16	15.357	H12	R16	4.084
58.	A4	R17	1.093	H12	R17	10.585
59.	A5	A6	6.621	H13	N14	4.704
60.	A5	A7	2.75	H13	N15	6.657
61.	A5	A8	6.77	H13	R16	8.475
62.	A5	D9	4.777	H13	R17	6.657
63.	A5	H10	7.415	N14	N15	9.869
64.	A5	H11	16.993	N14	R16	5.759
65.	A5	H12	10.205	N14	R17	9.869
66.	A5	H13	6.078	N15	R16	14.483
67.	A5	N14	9.681	N15	R17	0
68.	A5	N15	1.103	R16	R17	14.483

The data obtained during virtual screening of cefazoline compound is useful to identify hydrophobic surface regions of a protein structure as possible sites for intra- and intermolecular recognition, e.g. for the association of peptide fragments during protein folding for ligand (substrate, effectors, drug) binding and for protein aggregation[14-18]. The contribution of the hydrophobic effect to globular protein stability has been estimated empirically both by measuring the thermodynamics of transfer of model compounds (e.g. blocked amino acids, cyclic peptides...) from organic solvents to water and by site directed mutagenesis studies on proteins. The number arrived at is usually given as a function of the change in the solvent accessible non-polar surface area upon going from the unfolded to the folded state [19-22]. In silico study of synthesized derivatives 1, 3,4-thidiazole sucessfully docked inside the active site of Topoisomerase IV (PDB ID: 3FV5) domain for antibacterial activity with a binding energy 4.40 to -6.84 Kcal/mole[23].

IV. CONCLUSION

The molecular docking studies help to study the detailed molecular basis of interactions and to estimate the binding affinity of the present studied FDA approved drug Cefazoline.Its important for the analysis of chemical features of designed molecule and its bioisostericity related with target molecule. Computer aided drug designing, in silco activity of planned molecule and virtual screening of molecules help to solve all future problems of synthetic chemist.

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Effect of Temperature on Total Phenolic and Flavonoid Contents of Coriandrum sativum

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ABSTRACT

Coriandrum sativum is a promising functional food which not only provides nutrition, but also has medicinal benefits. It is a widely grown herb and most commonly used spice in India. Total phenolic and flavonoid content of ethanolic extracts of coriander leaves at different temperatures were evaluated to determine the effect of thermal processing on potential health benefits of coriander. The leaves were subjected to boiling (100°C) as well as storage at refrigerated temperature (4°C). A qualitative phytochemical screening was performed for the presence of phytochemicals. The ethanolic extracts were analyzed for total phenolic content using Folin-Ciocalteau assay and Flavanoid content by Aluminium chloride method. The extracts of fresh leaves showed the highest total phenolic and flavonoid content, which reduced significantly after treatment 100°C.Refrigeration also results in reduction of total phenolic and flavanoide content. This indicates that certain bioactive compounds such as polyphenols and phenolic acids are degraded during processing and thereby decreasing the medicinal value of herb. The study thus, suggests the consumption of fresh coriander leaves to obtain the maximum benefit.

Keywords: Coriandrum Sativum L., Total Phenolics, Antioxidant Potential, Phytochemical Screening.

I. INTRODUCTION

Coriander is a common food adjunct, which has been used for flavouring and seasoning throughout the world for thousands of years. Coriander (Coriandrum sativum L.) is an annual, herbaceous plant belonging to family Apiaceae. It is a flavouring substance used since ancient times and has been enjoyed by many cultures for its culinary and medicinal values (Hill and Sharma, 1998). The plant is widely grown for seed, leaf and essential oil. Seeds are widely used in curry powders, sausages and seasonings. Green leaves have a specific flavour and are used to garnish curries, in chutneys and soups. Leaves spoil quickly when removed from the plant, and lose their aroma when dried or frozen (Brechbill, 2012). Since heating also results in diminished flavour and hence, fresh leaves are preferred. Coriander contains many bioactive components - terpinene, cymene along withγ-pinene, αsuch as linalool, various non—linalool alcohols and esters. Other constituents include flavonoids, coumarines, isocoumarines, phthalides and phenolic acids (Verma et al., 2011). Coriander is well known for its antioxidant properties (Wangensteen et al., 2004, Diederichsen, 1996) and recent research has indicated that it is a rich source of flavonoids such as quercetin, kaempferol, and acacetin (Nambiar et al,. 2010). The polyphenols constitute a wide and complex array of phytochemicals that exhibit antioxidant action and consequently a beneficial physiological effect (Al-Juhaimi and Ghafoor, 2011). They can protect human body from free radicals and could retard the process of many chronic diseases such as cancer, cardio-vascular disease and diabetes; they can also reduce lipid oxidative rancidity in foods (Regnault-Roger et al., 2004; Arts and Hollman, 2005; Williamson and Manach, 2005). Polyphenolic compounds have high antioxidant activity due to the reactivity of the phenol ring and are categorized into different classes depending upon the number of phenol rings. The main groups are flavonoids, phenolic acids, phenolic alcohols, stilbenes and lignans. The main reason for this interest is the recognition of the antioxidant properties of polyphenols, their great abundance in our diet and their probable role in the prevention of various diseases associated with oxidative stress, such as cancer, cardio-vascular and neurodegenerative diseases (Nambiar et al., 2010; Chawla and Thakur, 2013). The quantity of phenolic and flavanoide compounds present in plants is influenced by storage method and the environmental conditions. Thus, it is important to determine the level of these compounds present in the plant after different thermal treatments. The present paper highlights the effect of refrigeration and conventional cooking methods such as boiling on the total phenolic and flavanode of coriander leaves.The plant extracts were also screened for qualitative phytochemical screening.

II. METHODS AND MATERIAL

2.1 *Chemicals :*

Ethanol, methanol, sodium carbonate, gallic acid, Folin Ciocalteu reagent ,Aluminium Chloride, K acetate and rutin were used. All the chemicals and reagents used were of analytical grade.

2.2. Collection of plant material and sample preparation:

Fresh leaves of *Coriandrum sativum* were purchased from a local market of Sangamner and washed with tap water. 150 grams of leaves were taken and divided into 3 equal parts (50 grams each). One portion was retained fresh; others were given different thermal treatments, as given below.

Boiling : Leaves (50 g) were boiled for 15 min, drained off and cooled rapidly.

Refrigeration : Coriander leaves were kept at 4°C in refrigerator for 5 days.

2.3. Preparation of extracts :

Leaves of *Coriandrum sativum* were extracted with ethanol at room temperature. Coriander leaves were soaked in 500 ml of 99.9 percent ethanol for 2-3 days separately. The soaked material was filtered and the extracts were collected. The filtrates obtained were concentrated under vacuum on a rotary evaporator (Buchi Rotary Evaporator, Model R-124) and stored at 4°C for further use (Song et al., 2010).

2.4. Phytochemical screening :

Ethanolic extracts of fresh coriander leaves were used for qualitative screening of phytochemicals as per standard biochemical procedures. The tests were performed to confirm the presence of alkaloids, carbohydrates, proteins and amino acids, glycosides, flavonoids, tannins, phenolics, terpenoids and steroids (Tiwari et al., 2011).

2.5. Determination of total phenolic content

The total content of phenol in different fractions of CA was determined by Folin- Ciocalteu reagent using reported method with slight modification (Khatoon et al., 2013). TPC of various fractions was determined from a gallic acid calibration curve. Calibration curve was prepared by mixing 0.5 ml aliquots of 10, 20, 30, 40, 50, 60, 70,80, 90, 100 µg/ml methanolic gallic acid solution with 2.5 ml Folin- Ciocalteu reagent and 2.5 ml (7.5 g/100ml)sodium carbonate. All mixtures were kept for incubation at RT for 30 min and absorbance was measured at 765 nm using spectrophotometer (Systronic UV-Visible -1203). Methanol was used as blank and gallic acid as a standard. A similar procedure was conducted for all fractions. All determinations were carried out in triplicates. TPC was determined from linear equation of standard calibration curve produced with GA and was expressed as gallic acid equivalent per milligrams (µg GAE/mg) of extracts.

2.6. Determination of total flavonoid content

The total flavonoid content of different fractions was determined by using aluminium chloride colorimetric method described by Maddanet al., 2011 and Saeed et al., 201. TFC of various extracts was determined from standard-rutin calibration curve. The solution of rutin of 100 µg/ml concentration was prepared in 80% methanol and further diluted to 10, 20, 30, 40, 50, 60, 70, 80, 90, 100 µg/ml. 0.5 ml aliquots of above concentrations were separately mixed with 1.5 ml of 95% methanol, 0.1 ml of 10% aluminium chloride. 0.1 ml of 1M potassium acetate and 2.8 ml distilled water. All mixtures were kept for incubation at RT for 30 min. Then absorbance of light pink coloured reaction mixture was measured at 415 nm versus reagent blank containing water instead of the sample. Rutin was used as a standard compound for the quantification of total flavonoids and the standard curve was drawn.A similar procedure was conducted for all extracts. All determinations were carried out in triplicates. Total flavonoid content was expressed interms of rutin equivalent per milligrams (µg RE/mg) of extracts.

III. RESULTS AND DISCUSSION

3.1. Phytochemical screening :

The results of qualitative phytochemical analysis of ethanolic extracts of fresh coriander leaves showed the presence of proteins and amino acids, carbohydrates, glycosides, phenolics, tannins, saponins, terpenoids, sterols, and flavonoids in table no.1

Sr. No.	Photochemical	Coriander
1	Alkaloids	- +
2	Protein and Amino acids	+
3	Carbohydrate	+
4	Phenol	+
5	Terpenoide	+
6	Steroids	+
7	Saponin	-
8	Glycoside	+
9	Flavonoide	+
10	Tannin	+

3.2. Total phenolics content

The total Phenolic Content (TPC) was determined by using Folin- Ciocalteu reagent & it reported as microgram per milligram (µg/mg) of Gallic acid equivalent (GAE) by reference to Gallic acid standard Curve ($Y=0.0101X \pm 0.0165$ & $r^2= 0.9902$). All the Fractions contained a considerable amount of phenolic content it was found that Fresh leaves Fraction had highest TPC (80.11 ± 0.55) µg of GAE/mg followed by refrigerated leaves (65.19± 0.00) & leaves after boiling (25.16±0.00)µg/mg of gallic acid equivalent respectively.

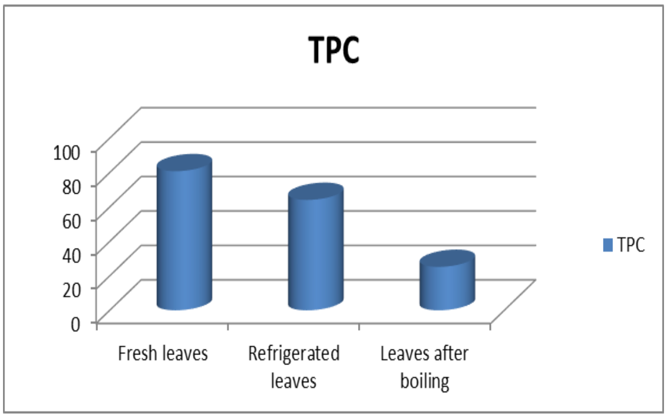

Fig. 1. Total phenolic content of various extract of *Coriandrum sativum*

3.3. Total flavonoids content-

Total Flavanoide Contents – The total Flavonoid Content (TFC) is expressed as microgram per milligram (µg/mg) of rutin equivalent (RE) by reference to rutin standard Curve.It was found that fresh leaves Fraction had the highest TFC (81.38± 3.125) µg of RE/mg followed by refrigerated leaves (39.16± 1.804), & boiling leaves (14.48± 1.005)µg/mg of rutin equivalent respectively.

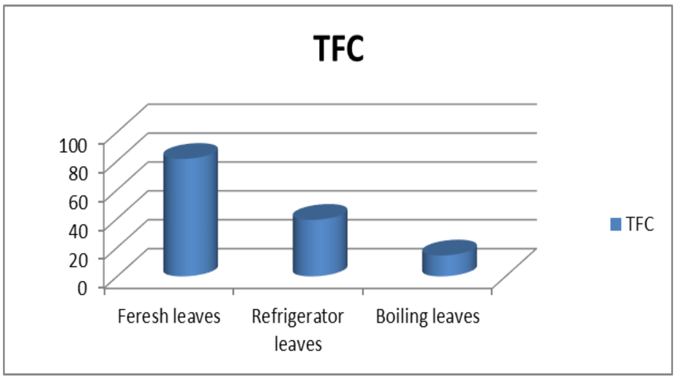

Fig. 2. Total Favonoide content of various extract of *Coriandrum sativum*

IV. CONCLUSION

Phytochemical screening of fresh leaf extracts revealed the presence of phenolic and flavanoide compounds, which are responsible for potential health benefits of coriander such as antioxidant and antimicrobial activity. Due to the changes in the lifestyle, the consumption of fresh leaves has been minimized. The above results indicate that thermal processes like boiling as well as storage under refrigerated conditions for longer periods leads to destruction of phenolic and Flavanoide compounds and other phytochemicals and hence, reduces the antioxidant potential of coriander.The study suggests the use of fresh coriander leaves in order to obtain the maximum potential.

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Green Synthesis of Organic Derivatives

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ABSTRACT

Recent advances in catalysis have been led by the development of new synthetic methods that provide control over size, reaction condition etc. The utilization of high intensity ultrasound offers a facile, versatile synthetic tool for synthesis that are often unavailable by conventional methods. The primary physical phenomena associated with ultrasound that are relevant to materials synthesis are cavitation and nebulization. In this type phase catalysed by auto micro reactors for sonochemistry. It is a hot gas inside bubbles isolated from one another in a liquid and hot droplets isolated from one another in a gas. Some synthetic reaction carried out by sonication with time and energy saving.

Keywords : Cavitation, Nebulization, Ultrasound

I. INTRODUCTION

Chemistry deals with the interaction between energy and matter, and chemical reactions require some form of energy (e.g., heat, light, radiation, electric potential, etc.) to proceed. Precise control over chemical reactions is a key to the success of materials synthesis, but currently such control is limited to the manipulations of various reaction parameters including time, energy input, and pressure. These parameters, however, are adjustable only within a certain boundaries defined by the energy source employed in reactions. Each type of energy has its own realm of reaction conditions determined by its inherent reaction parameters.Compared to traditional energy sources, ultrasonic irradiation provides rather unusual reaction conditions (a short duration of extremely high temperatures and pressures in liquids) that cannot be realized by other methods. Interestingly, such extraordinary conditions are not derived directly from ultrasound itself wavelengths are much larger than molecular dimensions. Thus, molecular level interaction between ultrasound and the chemical species takes place. Instead, acoustic driven by high intensity ultrasound accounts for the chemical effects of ultrasound. When liquids are irradiated with ultrasound, the alternating expansive and compressive acoustic waves creates bubbles (i.e., cavities).The oscillating bubbles can accumulate ultrasonic energy effectively while growing to a certain size. Under the right conditions, a bubble can overgrow and subsequently collapse, releasing the concentrated energy stored in the bubble within a very short time. This cavitational implosion is very localized and transient with a temperature of 5000 K and a pressure of 1000 bar. These extreme conditions created during acoustic cavitation can give rise to light emission. This intriguing physical phenomenon, known as sonoluminescence, was first observed during the ultrasonic irradiation of water in 1934 by Frenzel and Schultes. A variety of sonochemical apparatuses are commercially available with several designs as ultrasonic cleaning baths, direct-immersion ultrasonic horns, and flow reactors.

Cleaning baths have insufficient intensity for most applications, but are useful for liquid-solid reactions with easily passivated but reactive solids (e.g., Li, Mg). A typical laboratory-scale sonochemical apparatus which consists of a high-intensity ultrasonic titanium horn driven by a piezoelectric transducer which is directly introduced into a thermostated glass reactor having gas inlets and outlets. Cavitation occurs over a very wide range of frequencies. The chemical effects of ultrasound were explored for many years nearly always in water. Ultrasonic irradiation of aqueous liquids generates free radicals, and the formation of free radicals by sonolysis of water has been particularly well-studied. Major advancements in sonochemistry have been achieved by moving to less volatile organic liquids, so that the bubble contents are dominated by the intended reactants rather than the solvent.

II. METHODS AND MATERIAL

Procedure:

1.Pthalic anhydride to Pthalamide:

a. Routine Procedure:

In round bottom flask 2 gm of pthalic anhydride and 1 gm of urea. Heat the flask in oil bath at 130-135^{0}C for 10-20 mins till the content melt. Effervescence is observed the volume increases and temperature of mixture is also increases. Then stop heating and allow the reaction mixture to cool. Add 10 ml distilled water white ppt. of pthalamide is separate out. Filter it at suction pump. Wash with cold water and dry well.

Green approach: This reaction carried out at sonicator for 05 mins under sonication. Time effective and fuel saving with less hazardous.

2. Pthalamide synthesis by using Thiourea:

a. Routine Procedure:

In round bottom flask 2 gm of pthalic anhydride and 1 gm of urea. Heat the flask in oil bath at 130-135^{0}C for 10-20 mins till the content melt. Effervescence is observed the volume increases and temperature of mixture is also increases. Then stop heating and allow the reaction mixture to cool. Add 10 ml distilled water white ppt. of pthalamide is separate out. Filter it at suction pump. Wash with cold water and dry well.

Green approach: This reaction carried out at sonicator for 05 mins under sonication. Time effective and fuel saving with less hazardous. In sonication reaction time reduced up to 5 mins.

3. Anthracene to 9, 10 dihydro anthracene:

Take 1.6 gm of anthracene and 0.80 gm of Maleic anhydride. Transfer the reagent into 50 ml round bottom flask containing a stir bar. Add 20 ml of xylene in R.B. Start refluxing the round bottom flask. Heat by using heating mantle up to 180^{0}C for 30 mins. After 30 min. let the flask warm to room temperature. Wait for 15 mins. Place the round bottom flask in crushed ice. Then crystal formation is occure. Wash crystal with 5ml of cold xylene.filter the product dry it well and record yield.

Green approach:

Fual saving and instead of Acetic anhydride we used Maleic

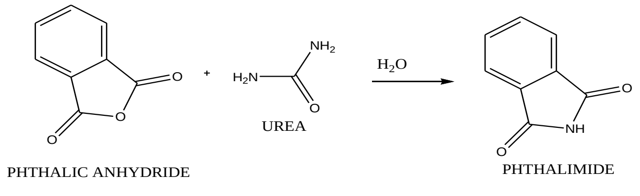
4. Pechmann condensation:

Take 1gm of Resorcinol and 1.3ml of ethyl acetoacetate mix them then 5ml of conc. H2SO4 and put into beaker with cold water for cool condition then add it into the above solution drop by drop and kept that mix side to full overnight. Then pour it into the ice cold water filter the separated solid wash with aqueous NaOH filter the solid dry it and recrystallised take its M.P. and TLC

Green approach:

Time period of reaction reduced from 60 to 15 mins.

Reaction 1st:



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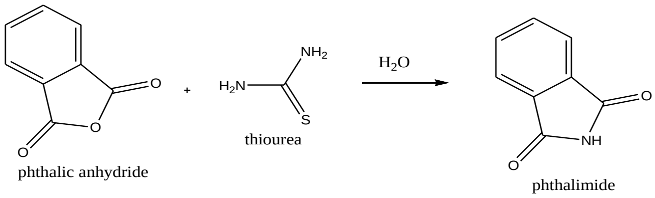

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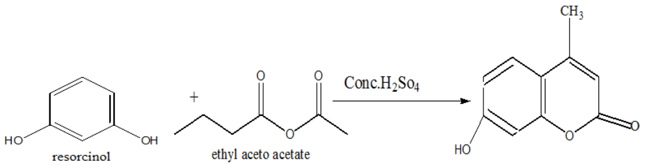

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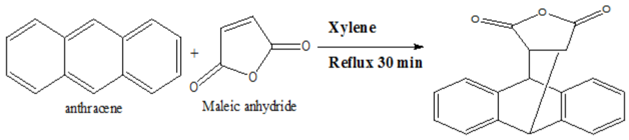

III. RESULTS AND DISCUSSION

Sr.No	Name of Reaction	Dist. travelled by reactant in cm	Dist. travelled by product in cm	Solvent run in cm	Physical Constant in ^{0}C
01	Pthalic anhydride to Pthalamide	2.5	3.1	5.1	238^{0}C
02	Thiourea to Pthalamide	4.1	3.0	5.3	180^{0}C
03	Anthracene to 9,10 dihydro anthracene	2.9	5.1	6.5	278^{0}C
04	Resorcinol to 4-methyl,7-hydroxy Coumarin	3.1	4.3	5.8	180^{0}C

Compound 01	Compound 02	Compound 03	Compound 04
Pthalic anhydride to Pthalamide	Thiourea to Pthalamide	Anthracene to 9,10 dihydro anthracene	Resorcinol to 4-methyl,7-hydroxy Coumarin
Physical Constant: 238^{0}C	180^{0}C	278^{0}C	180^{0}C
			
MobilePhase: n-Hexane	MobilePhase: Toluene + E.A (8:2)	MobilePhase: n-Hexane +Chloroform (8:2)	MobilePhase: Hexane

IV. CONCLUSION

A diverse set of applications of ultrasound have been explored in the synthesis of different compounds. Ultrasonic irradiation provides unique reaction conditions via acoustic cavitation. Bubbles generated during sonication can effectively accumulate the diffuse energy of ultrasound, and upon collapse, an enormous concentration of energy is released to heat the contents of the bubble. These transient, localized hot spots with extremely high temperatures and pressures are primarily responsible for chemical effects of ultrasound. The usefulness of synthesis as a synthetic tool resides in its versatility. The sonochemical method has been even further extended to the preparation of carbons, polymers, and biomaterials. The major challenges that face a wider application of sonochemistry, however, include issues of scale-up and energy efficiency.

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Sonochemical Method for the Synthesis and Characterization of Cadmium Compound Nanoparticles in Aqueous Medium

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ABSTRACT

In Sonochemical methods the researchers have intention on the study of the effects of sound waves and sound properties on chemical reactions or systems. The purpose of this research was to synthesize fast and facile method for the synthesis of CdX nanoparticles. For the present investigation we used Cadmium Chloride and different metal halide or sulphide compounds in aqueous medium. The resultant product is investigated by using X-ray diffraction technique (XRD), UV spectroscopy and Scanning electron microscopy (SEM). During the Course of reaction the increase in the temperature was occurred because of absorption of ultrasound waves was controlled conveniently by ice bath.

Keywords: Son-chemistry, CdX nanoparticles, U.V., XRD, SEM.

I. INTRODUCTION

Now a days nanocrystals of the transition metals and the leading group elements have attracted much interest due to their unique thermoelectric, semiconducting and optical properties. Semiconductor of sulfidesand selenides have al-ready found applications as sensors or laser materials, optical filters, solar cells and in many other devices [1]. Among these materials, CdS is particularly interesting due to its high photosensitivity [2], which makes it an excellent n-type window material in hetero-junction solar cells [3]. Using n-type CdS and P-type CdTe, solar cells of different efficiencies were fabricated [4]. However, a further improvement in the efficiency should be achievable by employing nanocrystalline CdS. It is well known that crystals with dimensions in the nanometer range show characteristics that are substantially different from the characteristics of bulk materials. In nanocrystalline CdS, an effective increase in the band-gap is ob-served due to quantum-confinement effects.

Many approaches have been suggested for the synthesis of group II–VI semiconductors, including the well-known solid-state reaction [5], gas-phase reactions between the elements or its compounds and gaseous H2S/H2Se [6], or the pyrolysis of single-source precursors [7]. All these methods require high temperatures which make the size control difficult) and the use of toxic precursors. A direct combination of elements in amine has been re-ported [8]; however, the obtained products were amorphous and had to be crystallized at elevated temperatures. The group of Gedanken et al. used microwave-assisted methods for the preparation of metal selenides [9,10]. Recent reports include the bacterial biosynthesis of CdS nanocrystals. Chemical reactions such as oxidation, reduction, dissolution and decomposition, which have then been exploited to prepare nanoparticles [11,12]. Ultrasonic waves have, as a result, been extensively used for producing novel materials with unusual properties. They can induce the formation of particles with a remarkably smaller size, which usually show novel physical properties [13]. The interest results from the special properties ofmaterials in the nanoscale rule, such as a large surface-to-volume ratio and increased surface activity, as compared to that of the bulk material. In our laboratory, the sonochemical method has been successfully used in the synthesis of nanoparticles of CdS.

The first synthesis of CdS nanoparticles by ultrasonic irradiation was reported by Wang et al. [2,14,15]. The method used the reaction of a mixed solution of cadmium chloride and sodium thiosulfate in isopropyl alcohol in an Argon atmosphere. A sonochemical approach to the synthesis of CdS nanoparticles using Cadmium acetate and elemental S in dimethyl sulfoxide under a H2/ Ar atmosphere was also reported [1]. Recently, Behboudnia and Khanbabaee reported on the synthesis of CdS nanoparticles by the sonication of an ethylenediamine solution of 1-decanthiol, cad-mium acetate and elemental S [16] and the sonochemical preparation of CdSe nanoparticles from cadmium acetate, tartaric acid, freshly prepared sodium selenosulfate and tiophenol [17]. How-ever, to the best of our knowledge, the sonochemical synthesis of CdS from aqueous solutions has not been reported so far. A direct, simple and general method, avoiding the necessity of using inert atmospheres and non-aqueous systems, seems to be required. In this paper we present a new, simple method to prepare CdS nanoparticles from aqueous solutions in the ambient atmosphere.

II. METHODS AND MATERIAL

2.1 Materials

$CdCl_2$, CdO_2, Na_2S/Selemental sulphur and SeO_2 were obtained from SRL and Sigma Aldrich. All the chemicals were used as received without further purifications. All solutions were prepared in mill moles.

2.2 Synthesis

For the preparation of CdS nanoparticles, acationic precursor solution was prepared by dissolving 2.58 gm (2 mmol) of cadmium oxide in 30mL of water at room temperature.Then we prepared the anionic precursor solution by dissolving 1.56 g (2 mmol) of Na_2S in 30mL of water. The two solutions were mixed in a flat-bottomed beaker and irradiated with high-intensity. After 40 min of sonication (1sec on and 2 sec off) themixture is allowed to stirred for 3 hours. The product thus obtained is filteredand washed twice with distilled water and once with absolute ethanol. Finally, the brown colored product was dried for 2 h at 150 LC followed by calcination at 450 LC in muffle furnace for 4 to 5 hours.

For the preparation of CdSe nanoparticles, a cationic precursor solution was prepared by dissolving 2.58 gm (2mmol) of cadmium oxide in 30mL of water at room temperature. Then we prepared the anionic precursor solution by dissolving 2.22 g (2 mmol) of SeO_2 in 30mL of water. The two solutions were mixed in a flat-bottomed beaker and irradiated with high-intensity. After 40 min of sonication (1sec on and 2 sec off) the mixture is allowed to stirred for 3 hours. The product thus obtained is filtered and washed twice with distilled water and once with absolute ethanol. Finally, the white colored product was dried for 2 h at 150 LC followed by calcination at 450 LC in muffle furnace for 4 to 5 hours.

III. RESULTS AND DISCUSSION

Characterization was performed via X-ray powder diffraction (XRD) and Scanning electron microscopy (SEM). Figs. 1 and 2 show the XRD spectra of typical nanoparticles of CdS and CdSe prepared by the sonochemical method after 40 min of sonication. The peaks can be classified as the 111, 200 and 222 reflections of cubic CdS (JCPDS-411049) and CdSe

(JCPDS-020330) respectively.The broadening of the peaks indicates that the particles are on the nanometer scale. The average size of the nanoparticles was calculated using the Debye–Scherrer formula:

$$D = \frac{0.9\,\lambda}{\beta\cos\emptyset}$$

2 θ	β	θ	θ in rad	β in rad	cosθ	0.9*0.15406 (nm)	Grain size (D) nm
17.75523	0.55312	8.877615	0.154944	0.009654	0.98802	0.138654	14.5368341
17.22608	0.28426	8.61304	0.150326	0.004961	0.988722	0.138654	28.26603838
30.348	0.30536	15.174	0.264836	0.00533	0.965135	0.138654	26.95595082
33.039	0.18177	16.5195	0.28832	0.003172	0.958723	0.138654	45.58685943
38.3501	0.35302	19.17505	0.334668	0.006161	0.944519	0.138654	23.8256524
49.77816	0.89024	24.88908	0.434396	0.015538	0.907124	0.138654	9.837418734
55.33305	0.39894	27.66653	0.482872	0.006963	0.885665	0.138654	22.48422596
58.31929	0.56052	29.15965	0.508932	0.009783	0.873265	0.138654	16.22996683
65.97371	0.44185	32.98686	0.575729	0.007712	0.838795	0.138654	21.43502946
69.31663	0.43788	34.65832	0.604902	0.007642	0.822558	0.138654	22.05633741
						Avg. grain size (D)	**23.12143135**

Here, λ is the wavelength of the X-ray radiation, β is the full width at half maximum (FWHM) of the corresponding peak and θ is the dif-fraction angle. The value obtained for CdS and CdSe is 23-60 nm. The size and morphology of the as-prepared CdS nanoparticles were assessed with Scanning electron microscopy (SEM). The SEM image of the CdS in Fig. 3 shows that the particles are mainly cubic, and Fig. 4 shows the SEM image of the CdSe are hexagonal but strongly agglomerated.

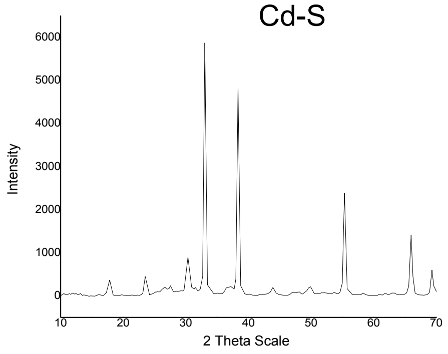

Fig. 1. X-ray diffraction spectra of CdS nanoparticles Synthesized, by ultrasonic irradiation for 40 min.

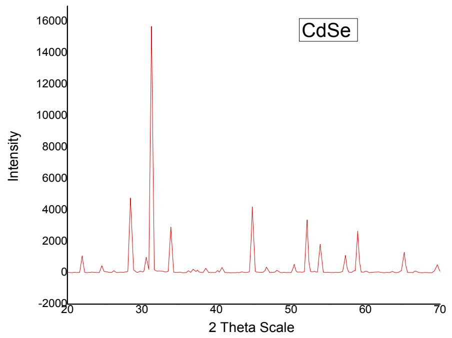

Fig. 2. X-ray diffraction spectra of CdSe nanoparticles Synthesized, by ultrasonic irradiation for 40 min.

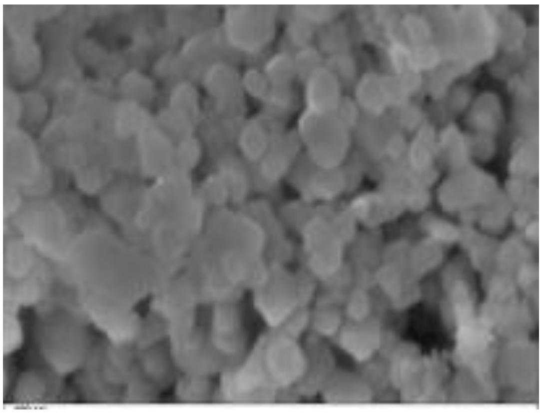

Fig. 3 SEM image of as-prepared CdS nanoparticles

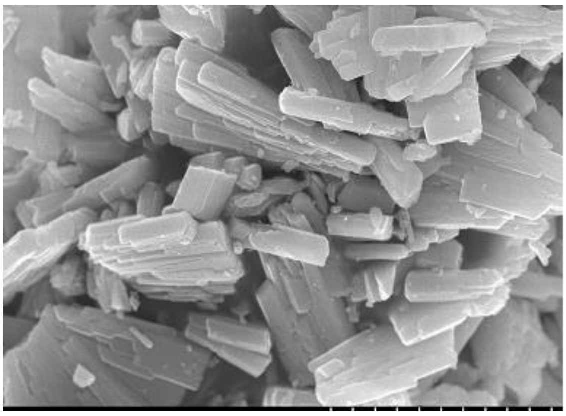

Fig. 4 SEM image of as-prepared CdSe nanoparticles

IV. CONCLUSION

CdS and CdSe nanoparticles have been successfully synthesized via a novel sonochemical route. The preparation method is relatively simple in comparison to other reported methods, avoids the use of an inert atmosphere, and uses less hazardous precursors and environmental friendly aqueous solvents. The average size of nanocrystallites was about 23-60 nm for the CdSand CdSe. The products were characterized with powder diffraction analyses and thermal analyses including characterization of the intermediates. The crystal structure changed from cubic. SEM observations indicated that the particles were agglomerated, but con-firmed their cubic and hexagonal structure and uniform composition. Theobtained nanoparticles are expected to be applicable in modern optoelectronic devices and solar cells. Further investigations may lead to the preparation of a variety of metal chalcogenides using similar preparation methods in aqueous systems.

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Study on Quality of Drinking Water in Non-Irrigated Area of Sinnar Taluka, Dist. : Ahmednagar, Maharashtra

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ABSTRACT

The most of the diseases of human beings are caused by water since it is directly related with human health , so it is very important to study the quality of drinking water. In view of this, we have selected water of some Non-irrigated area of SinnarTaluka to study its suitability for drinking purpose where the ground water is main source for drinking .The samples were collected from Marhal, Nandur, Kankuri, Manori, Wawi, Nirhale, Dodi, Pangri, Bhokani, Chas, Nalwadi which are non-irrigated area. The samples were analyzed for different parameters like p^H, Carbonates, bicarbonates Cl^-, Ca^{2+}, Mg^{2+} by using standard methods. All water samples are best for drinking purpose because all have limited quantity of all above measured parameters. So there is no need of RO machine for purification of drinking water of study area.

Keywords : Drinking Water, Permissible Limits, Water Quality, Water Samples

I. INTRODUCTION

Actually water is basic requirement of human being but use of natural sources in undisciplined way is causing harmful effect. Water pollution is the major problem, which we are facing now a days and is going to be more and more complicated. The contaminated water created large problem for health of human being. The presence of impurities in excess is depends on area, so proper study of such contaminant (impurity) is very necessary

Objective :

To determine the quantity of some parameter like pH, Carbonates, bicarbonates Cl^-, Ca^{2+} and Mg^{2+} in drinking water.

Study Srea:

Marhal, Nandur, Kankuri, Manori, Wawi, Nirhale, Dodi, Pangri, Bhokani, Chas, Nalwadi non-irrigated area of Sinnar Taluka . The drinking water from different area of different sources of Marhal, Nandur, Kankuri, Manori, Wawi, Nirhale, Dodi, Pangri, Bhokani, Chas, Nalwadi were collected for analysis.

II. METHODS AND MATERIAL

The samples from the wells, hand pumps were collected on the basis of its use for drinking purpose only. The samples were collected in polythene bottles of one liter capacity. The pH by using pH meter. The other analysis of water was carried out by using the procedures given by a text book of Practical Agriculture and Dairy Chemistry , Sunny Publication

III. RESULTS AND DISCUSSION

Results:

Table 1.

Parameter Sample	p^H	CO_3^{2-} mg/l	HCO_3^- mg/l	Cl^- in mg/l	Ca^{2+} in mg/l	Mg^{2+} in mg/l
S-1	8.1	A	58	98	20	34
S-2	8.2	A	29	71	87	30
S-3	6.9	A	58	21	24	32
S-4	8.0	A	29	170	43	35
S-5	8.3	A	14	63	21	25
S-6	8.0	A	34	99	10	15
S-7	7.9	A	78	92	05	07
S-8	7.4	A	38	37	20	27
S-9	8.2	A	56	340	27	20
S-10	7.9	A	48	14	43	44
S-11	8.2	A	98	170	10	12
S-12	7.4	A	57	43	21	17

Standard Values:

Table 1. Indian Standard Specifications for Drinking Water IS: 10500 (BIS-2012)

	Parameter	Requirement desirable limit	Remarks
1	pH	**6.5 - 8.5**	**No relaxation**
2	Cl^-	**250 mg/l**	**May be extended up to 1000**
3	Ca^{2+}	**75 mg/l**	**May be extended up to 200**
4	Mg^{2+}	**30 mg/l**	**May be extended up to 100**

Discussion

The pH of all water samples are within the range of desirable limit. The sample S-9 has higher value of chloride but not above the permissible range. The

Sample S-2 has higher value of Calcium but it also not above the permissible range. The samples S-1, S-3 and S-4 has higher values of magnesium but not above the permissible rang. So all water samples from different places are good for drinking purpose.

IV. CONCLUSION

All water samples are best for drinking purpose because all have limited quantity of all above measured parameters. So there is no need of RO machine for purification of drinking water of study area.

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The Effect of Municipal Solid Waste Dump Site on the Various Ground Water Parameter

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ABSTRACT

The environment has become an affecting concern with the societies and approach makers. The contamination of environment due to human activity. The primary objective of this paper is to study the various groundwater sources collected from nearby area of Municipal Solid Waste Dump Site, Sangamner, Ahmednagar District (MS), India. The hardness concentration varied from 488 to 868 mg/L. Chloride concentration oscillated from 19.43-36.94 mg/L. The concentration of Sodium ions in the study area is from 55.6 to 70.25 mg/L. All collected samples are found to be not potable for drinking purposes.

Keywords: Municipal Solid Waste, Human Activity

I. INTRODUCTION

The disposal of municipal solid waste is major concerns as waste generation is increasing day by day. The term municipal waste is an "Applies to those waste generated by house hold and to waste of similar characters derived from shops, offices, and other commercial units."[1] The improper disposal of waste can cause serious environmental or ecological damage. This area is not safe for drinking, outdoor, bathing propagation of aquatic life, industrial cooling and for irrigation. This unscientific disposal gives rise to greenhouse gas emission like methane, carbon dioxide and causes air pollution.

Leachate fomentation and its subsequent accumulation pollutes the ground water and nearby water bodies[2]. The rain water runoff from these dumps contaminates nearby land and water there by spreading diseases. They also the source of air pollution. The municipal solid waste is causes the soil as well as water pollution. The municipal stream may often contain some hazardous waste such as hospital wastes, paints, solvents and batteries careless handling and disposal of municipal wastes may thus pose a threat to human health in many developing countries.

Figure 1 : Municipal Solid Waste Dump Site

II. METHODS AND MATERIAL

Analysis of water samples for parameter etc. pH, Sodium, Potassium, Calcium, Magnesium, Sulphate, Chloride, Bicarbonate, COD, BOD, TDS, TSS and Total Hardness (Table 1).

III. RESULTS AND DISCUSSION

The hardness concentration varied from 488 to 868 mg/L. Chloride concentration oscillated from 19.43-36.94 mg/L. The concentration of Sodium ions in the study area is from 55.6 to 70.25 mg/L (Table 2).

Table 1 : Groundwater quality of collected samples compared with standard data

Parameters of drinking water	WHO(1993)	BIS		Analyzed samples range
		Acceptable limit	Maximum Permissible Limit	
pH	Not mentioned	6.5-8.5	No relaxation	7.57-8.15
Hardness (ppm)	100-125	100.0	125.0	488.0-868.0
COD (ppm)	5.0-50.0	5.0	50.0	16.0-105.00
TDS (ppm)	100-2000	100	2000	240.0-320.0
Sulphate (ppm)	1.0	1.0	-	0.75-2.48
Chlorides (ppm)	10-15	10	15	19.43-36.94
Sodium (ppm)	2-3	2.0	3.0	55.6-70.25
Potassium (ppm)	20.0-100.0	20.0	100.0	2.0-3.0

Table 2: Physicochemical Parameters of Analysed Samples

Parameters of drinking water	S_1	S_2	S_3	S_4
pH	7.57	7.64	7.92	8.15
Hardness (ppm)	488	520	824	868
COD (ppm)	105	57.60	16	38.40
TDS (ppm)	320	240	280	320
Sulphate (ppm)	2.23	0.75	2.48	2.03
Chlorides (ppm)	19.43	36.94	29.19	31.03
Sodium (ppm)	55.6	70.25	60.25	59.01
Potassium (ppm)	3.0	2.0	2.0	2.0

EFFECT OF ANALYSED PARAMETER ON LIVING ORGANISM AND PLANTS

The salinity of water, increase the pH which is affect soil & plant health. Hence the percentage agricultural yield is decreases. The hardness of water increases due to the presence of Ca2+ and Mg2+ ions, which affect the human excretory system. COD increases due to increase organic matter. Total Dissolved Solids effect on turbidance & color of water. The chloride increases the salinity & its effect on water holding capacity of soil. The leaching of sulphate in water by the use of sulphate fertilizers. Higher the percentage of Sulphate, affect the taste of water. High concentration of Sodium in water, peoples affected with certain disease like excretory system & digestive system. The less amount of potassium, affect the growth of plant, decreases the limpness of the seed, effect on formation of carbohydrates & fats in plant.

IV. CONCLUSION

The hardness concentration varied from 488 to 868 mg/L. Chloride concentration oscillated from 19.43-36.94 mg/L. The concentration of Sodium ions in the study area is from 55.6 to 70.25 mg/L. Hence we conclude that all collected samples are found to be not potable for drinking purposes as well as for agricultural uses.

SIGNIFICANCE AND APPLICATION OF THIS STUDY

1. Selection of sites for disposal of municipal solid waste should be done from the area having minimum water sources (Wells).
2. One should avoid digging the wells near to MSW dumping sites.
3. To avoid leaching, the dump sites should be subjected to soling and PCC treatment.
4. The solid waste is separated in cells, first cell contains decomposable materials, second cell contains slowly decomposable materials, & third cell contains non degradable materials. The decomposable material is used for the formation of manure & to avoid the leachate formation.

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Quantitative Analysis of Selected Pesticide Residues in Brinjal Using Gas Chromatography

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ABSTRACT

Pesticide residual analyses of selected pesticides (Carbofuran, Cypermethrin, Mancozeb and Chlorpyrifos) in brinjal fruits are explained by using Gas Chromatographic system. There were two varieties (BARI-8 and UTTARA) of brinjal, cultivated at Department of Environmental Science Field, Bangladesh Agricultural University, Mymensingh and the harvesting time was selected as 0 day, 3rd day and 7th day from the pesticide spray. After harvesting 54 brinjal fruit samples had been dried and packed properly. In GC analysis, calibration curve was drawn for every concentration of standard solution with areas of peak as well as a major eluted peak for one and every pesticides solution was obtained. Results revealed that the contents of pesticide residues were higher in 0-day as compared to 3rd day and 7th day for both of the varieties. In contrast, the incidences of pesticides in fruits of all the samples in 0 day for both varieties exceeded the MRL with the highest significant for all pesticide residues. On the 7 days interval the pesticide residues for all selected pesticide under the MRL of both varieties. UTTARA brinjal fruits had more trend to reduce the pesticide residue within day variation than the BARI-8 variety. The reduction trend (Reduction Factor) ranged from 0.164 ppm to 0.230 ppm for both varieties which had a positive response to the pesticide concentration reduction over day variation. This study also suggested that on considering the consumer safety, brinjal should not be harvested less than 3-5 days as well as it may be consumed at approximately 7 days later from the spray. Public awareness about the pesticides and other related matter should be increased for practicing a pesticide free agriculture as well as gain contaminate free environment.

Keywords : Pesticide, Residues, Brinjal Fruit, GC, Post-Harvest Interval.

I. INTRODUCTION

Agriculture is the principal economic anchor of Bangladesh, which contributes about 33% to the country's gross domestic product (GDP) and 80% of people depend on agriculture for their livings [1,2]. Agriculture plays an important role to protect food scarcity in this densely populated country. Rice, wheat, jute, potato, sugarcane, vegetables and tea are the main crops of this country [3]. Brinjal, tomato, cabbage, bitter gourd, snake gourd, pointed gourd, okra, hyacinth, bean and yard long bean are grown in Bangladesh as vegetables and these are nutritious, valuable and very popular to consumers [4,5]. The importance of brinjal is well known. It helps to avert colon cancer, decreases cholesterol level, helps in the type 2 diabetes management, is very rich in antioxidant, and also helps to control weight [6]. To ensure the food demand, control of insects, pests and diseases plays a key role. Until today, pesticides play a vital role for the control of insects, pests and diseases, but pesticides create several adverse effects on human health, agro-ecosystem and the environment resulting from indiscriminateness, overuse, and misuse of pesticides [7]. Like many other developing countries, pesticides are used extensively in Bangladesh to increase the crop yield per acre [8-10]. As agricultural production is being increased every year to meet the growing demand of the people, uses of pesticides also being rose up. In Bangladesh context, the vegetable growers have been using the pesticides frequently to have the higher yield. The widespread use of pesticides may contaminate the environment as well as foods, which may create health problem [3,11,12]. The most alarming concern is that pesticide use is very indiscriminate in Bangladesh [13]. There are areas where pesticides are used in excessive quantities [14]. Application of different pesticides is increasing day by day in Bangladesh and this trend would be detrimental to mankind as well as environment [15]. Therefore, the presence of these pesticide residues in food commodities has always been a matter of serious concern especially when these commodities are consumed fresh [16-18].

Brinjal is an important vegetable and very much sensitive to pest attack and disease infestation from seedling to fruiting stage [4, 19]. The main pests that attack plants are Brinjal Fruit Borer, Stem borer, Spider Mite, Aphid, Jassid, Whitefly and Roots-cutworm. For the control of these pests, the farmers are used different types of pesticides. The farmer community uses the major pesticides such as Acephate, Chlorpyriphos, Dichlorovos, Carbofuran and Imidacloprid. It has been reported that in brinjal, bitter gourd and beans, the attack of insect pests are severe and farmers' sprayed pesticides quite frequently even every day [4,14]. Farmers believe that for better yield the pesticide application is very important parameter to be conducted during crop production; by following this they tremendously apply the pesticides over crops even on fruiting. This repeated application of pesticides on crops particularly at fruiting stage and no adoption of safe waiting period leads for accumulation of pesticide residues in fruits and vegetables [20].

Most screening procedures in use today are capable of finding only a fraction of the pesticides that are registered around the world. Gas Chromatography (GC) provides the most comprehensive and reliable screening method available for pesticides, metabolites, and suspected endocrine disrupters. It has high sensitivity, a large linear response range, and low noise. It is also robust and easy to use, but it destroys the injected sample [21]. Many scientists [22-24] analyzed pesticide residues in fruit and vegetables in India, Spain, China, Japan and other developed country. But limited pesticide residual analysis conducted by GC for available vegetables in Bangladesh [10,25,26]. Islam et al. [10, 11] were conducted research works on pesticide residues in various vegetables and fruits which from nearby local markets. Most of the scientists e.g. Hossain et al., [15]; Chandra et al. [27]; Fakhruddin et al., [28] worked out to determine pesticide residues in different ways and their maximum work was executed to comparison between verities or depending on various factors. But this study is undertaken to determine the pesticide residues in brinjal from the experimental field. The important thing is that the market product always not enough to determine the exact values but cultivated vegetables can be capable of providing exact values because it maintained by supervision. This study is conducted to determine the residual effect of pesticides over day variation from the day of pesticide application till a week and also shown a comparison between residual effect and Maximum Residue Limit (MRL).

II. METHODS AND MATERIAL

Sampling and Experimentation

Fifty-four brinjal fruits samples (18 samples per sampling day) were collected during 0 day, 3rd day and 7th day after pesticide spraying from 18 plots (9 plots BARI-8 and 9 plots UTTARA variety) of brinjal yields on considering the fruits color, size and freshness. The samples dried and packed separately with properly labeled polythene bags and brought to the Department of Environmental Science Laboratory, Bangladesh Agricultural University, Mymensingh and after few days all samples were transferred to Bangladesh Agricultural Research Institute (BARI), Gazipur for the experiment. The collected samples were extracted (Liquid extraction technique following by Usher and Majors [21] with changing extraction time) properly and cut into small pieces with proper labeling for future identification and kept in deep freezer (-20 °C). About 2 gm of each sample (frozen part) was grinded by hand grinder then allowed in 5 mL Hx (n-Hexane) in test tube for 15 minutes and tightly closed by cork to avoid the evaporation of solvents and volatile chemicals. After 15 minutes, each part was mixed well by shaking. After that it was kept 5 minutes for stabling the mixture. The supernatant solution (Hx with extracted compound) was taken in a vial by small glass pipette carefully and kept for the experiment.

Calibration Curve

Individual stock standard solutions (1 mg/mL) were prepared in ethyl acetate and stored in the dark at −20 °C. Prior to their use, they were kept for 1h at ambient temperature. A mixed stock standard solution of pesticides was prepared in ethyl acetate at 15 μg/mL with respect to each pesticide. Two different types of calibration curve were studied: matrix-matched calibration (MC) and solvent calibration (SC). Matrix-matched calibration solutions were prepared by mixing known volumes of the pesticide working solutions and the volume with extracts of blank samples. Calibration curves at 5 levels of 10, 20, 30, 40 and 50 ppm triplicate were prepared by addition of 10 μL, 20 μL, 30 μL, 40 μL and 50 μL of mixed standard stock solution, respectively, to 15 gm portions of blank brinjal samples in each case. A stock solution of triphenylmethane (TPM) in ethyl acetate at concentration of 1 mg/mL was used as internal standard and an aliquot of 10 μL of TPM solution in ethyl acetate was added to the brinjal sample.

Sample Preparation

An aliquot of 10 μL of internal standard solution (1000 mg/L) was added to 15 g of blended blank brinjal sample in a 50 ml falcon tube and after being left for 1h at ambient temperature in dark, 15 mL acetonitrile (containing acetic acid 1% and sodium acetate tri hydrate (0.45gm) was added. The mixture was mixed at high speed with vortex mixer for 1 min. 4 gm of activated anhydrous MgSO4 was added to the mixture, and mixing was continued for an additional 60 s. The mixture was centrifuged for 5 min at 3000 rpm at -5 °C. The supernatant was transferred to a 15 mL falcon tube containing 2 gm $MgSO_4$, 300 mg PSA and 100 mg florisil. After shaking for 1 min and centrifugation for 5 min at 3000 rpm at -5 °C, 4 mL of supernatant was transferred to a 5 mL vial and evaporated to dryness under a gentle stream of nitrogen gas. The residue was reconstituted by toluene to obtain 1 mL solution, and after shaking for 3 minutes, 2 μL of the solution was injected into gas chromatograph.

Gas Chromatographic (GC) Analysis

The analysis was conducted with Gas Chromatograph (GC) [GC-2014] Shimadzu Corporation, Kyoto, Japan. FID (Flame Ionization Detector) was used in this experiment. The whole GC system is tabulated below (Table 1). Injection of each standard solution was repeated several times (at least 5 times) for the confirmation of the respective retention time. The different concentration of standard solutions was injected in GC system under above mentioned condition and made calibration curve. GC eluted compound was calculated by comparing the retention time with specific peak area of the standard solution and studied samples. After acquisition of the total chromatogram for the mixed stock standard solutions in scan mode, peaks were identified by their retention time. The most abundant compound that showed no evidence of chromatographic interference and had the highest signal-to-noise ratio was selected for quantification purposes.

Table 1: Parameters of gas chromatography (GC).

Parameters	GC/FID	Stability/Reproducibility
Column	RT-Missive 5A, serial number: 1104596. Length: 30 m Inner diameter: 0.53 mm Film thickness: 50.00 um.	Column flow: 6.63 mL/min Maximum temperature: 300°C.
Oven	50°C (3 min), to 200°C at 10°C /min, to 300°C at 15°C/min and hold finally 5 min. (For Mancozeb and Cypermethrin)	Column oven: 50 °C (for Mancozeb); 60 °C (for Chlorpyrifos); 60 °C (for Carbofuran); 50 °C (for Cypermethrin).
	60°C (5 min), to 180°C at 10°C /min, to 280°C at 10°C/min and hold finally 8 min. (For Chlorpyrifos and Carbofuran)	
Carrier	Hydrogen (30 mL/min); Air (142.1 mL/min)	Hydrogen (30 ml/min)
Detection	DFID, 300 °C, Sampling rate: 40 msec	DFID, 300°C
Injection	1μL, Direct injection, 200°C	Split
Linear velocity	60.7 cm/sec	Pressure: 66.8 kPa
Purge Flow	3.0 mL/min	Split ratio: 20

Quantitation

The concentrations of pesticides were determined by interpolation of the relative peak areas for each pesticide to internal standard peak area in the sample on the calibration curve. In order to compensate for losses during sample processing and instrumental analysis, internal standard (TPM) was used.

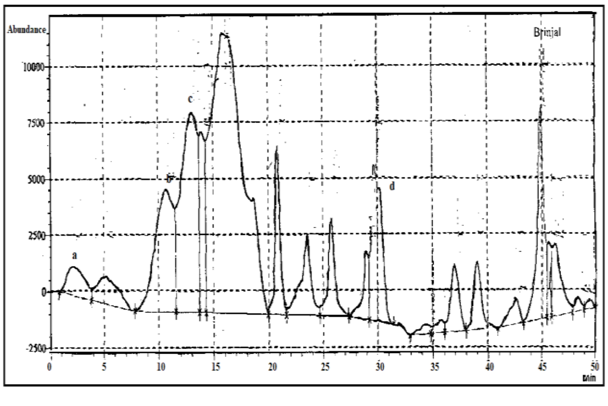

Figure 1: A representative chromatogram obtained for four pesticides and internal standard a) chlorpyrifos; b) mancozeb; c) carbofuran; d) cypermethrin in brinjal samples.

Calibration Curve of Pesticide Solutions

From the figure 2 – 5, the straight-line calibration curves were made by using the peak areas and concentration of standard pesticide solutions where carbofuran (12.3 min), chlorpyrifos (1.5 min), mancozeb (11.68 min) and cypermethrin (33.7 min) solution of different concentration were eluted. These curves were made for measuring the different concentration of pesticide in brinjal fruits where areas of peak for every concentration were calculated.

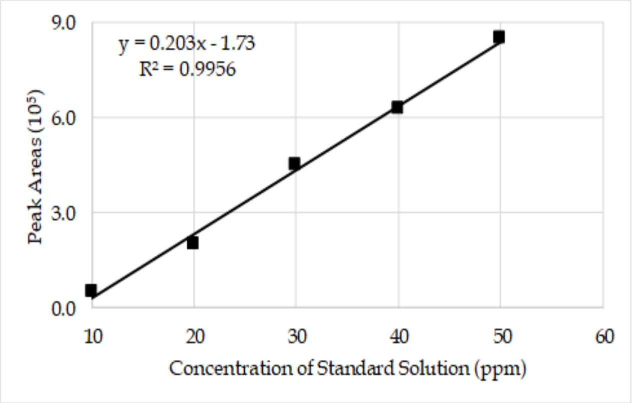

Figure 2: Curve for various concentration of standard carbofuran solution.

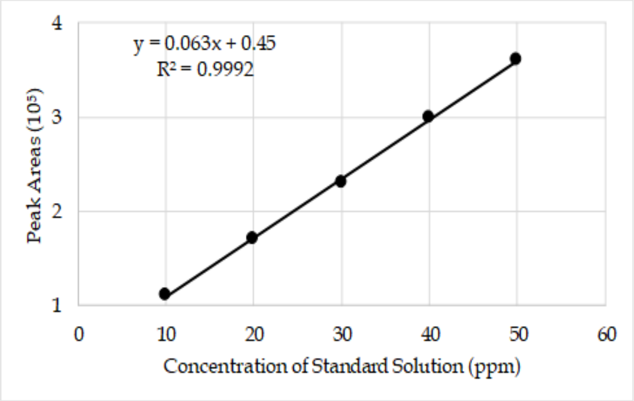

Figure 3: Curve for various concentration of standard chlorpyrifos solution.

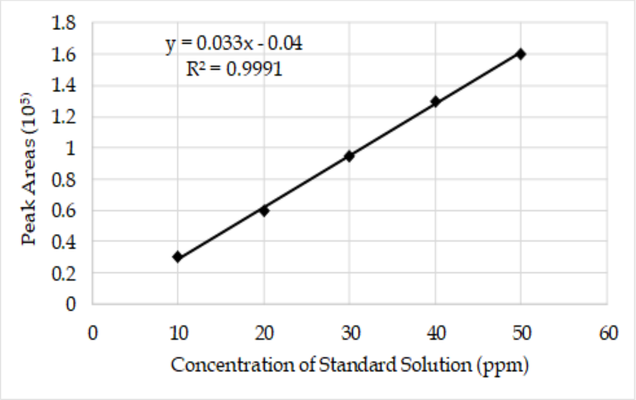

Figure 4: Curve for various concentration of standard mancozeb solution.

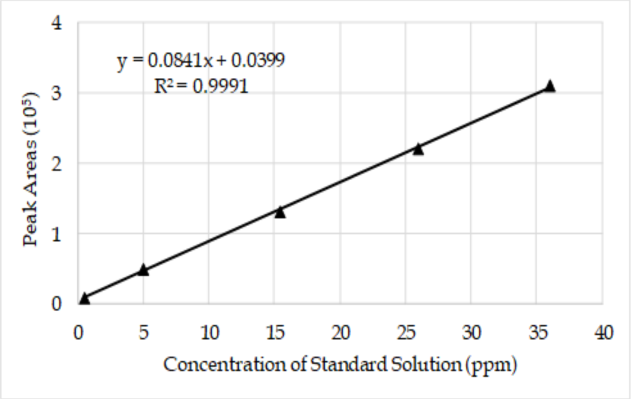

Figure 5: Curve for various concentration of standard cypermethrin solution.

The curve of peak areas and concentration of this present study was agreed to the previous study of Islam *et al.* [10,11] and Afia *et al.* [29] where their drawing curves are quite similar on depending the concentration and peak areas. Islam *et al.* [10,11] showed the GC calibration curve and suggested that peak area of the specific amount of a chemical dependent upon the nature, elution and characteristic of chemicals studied. There was enormous peak area differences found in respect of specific amount of standard chemicals. Few researchers [10,11, 30-32] suggested the similar variation among the different chemicals occurred in their peak areas in GC analysis.

Chromatograms of Pesticide Solution

Figure 6 – 9 are showed that the carbofuran, chlorpyrifos, mancozeb and cypermethrin solution response at 12.3, 1.5, 11.5 and 33.7 minutes in GC system and for confirmation, the system repeated for several times.

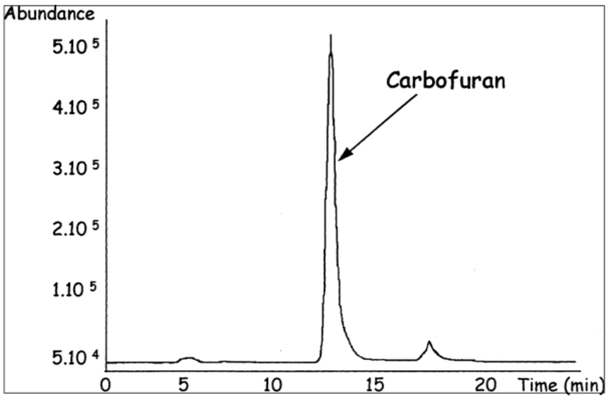

Figure 6: Eluted peak for carbofuran solutions.

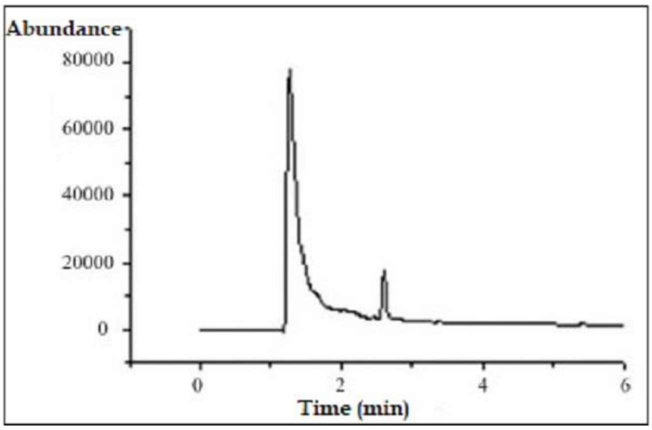

Figure 7: GC Eluted a major peak showed chlorpyrifos solutions.

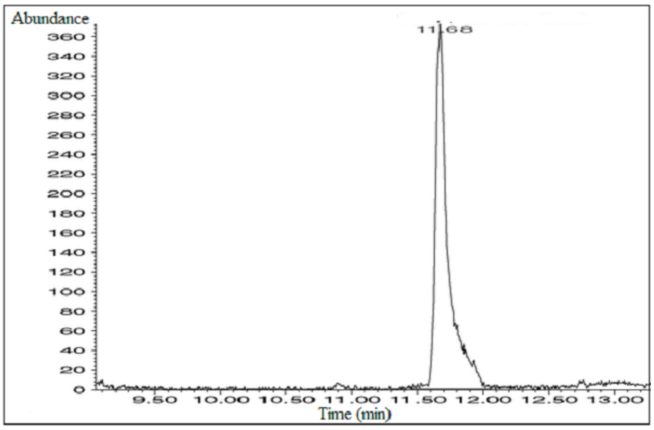

Figure 8: A Major eluted peak for mancozeb solution.

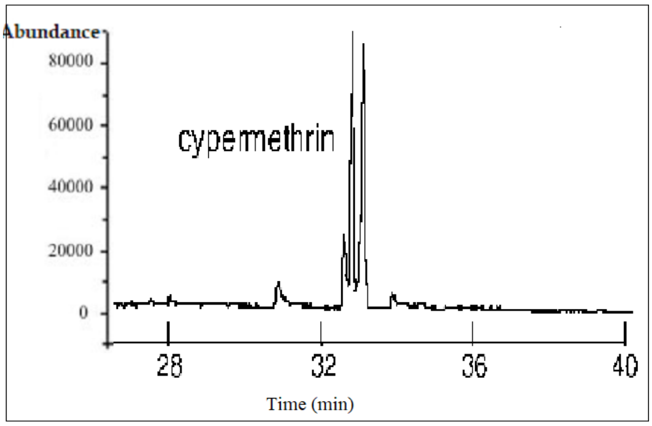

Figure 9: GC eluted peak for cypermethrin solution.

Islam *et al.* [10] found the compound of imidacloprid solution at 19.20 minutes of GC system and Afia *et al.* [29] found the compound of chlorpyrifos at 9.870 minutes that have a good agreement to this study. Islam *et al.* [10] also showed the retention time for mancozeb solution was 14.52 minutes that similar to this present study. The eluted peak and retention time actual and no disagreement.

Residues of Pesticide in Brinjal

Comparison of Pesticide Residue between BARI-8 and UTTARA

At the day of pesticide spray, the concentration was so high and then followed by 3 days and 7 days interval the concentration decreasing randomly for all pesticides (Figure 8 and 9). The pesticide decreasing over time variation was agreed to the previous observation of Li *et al.* [33] where they reported about the cypermethrin, carbofuran and diazinon pesticide reduction trend with passing the time. Hiralal *et al.* [34] indicated that the residues of pesticed had controlled to maintain the 4-7 days interval of spray. So, at a certain stage the concentration of pesticide was reached to the safe consume level or MRL limitation. The brinjal variety of UTTARA was also up taken pesticide concentration and over time variation it's also showed the positive response to the concentration level. UTTARA fruits gave up concentration of pesticide more than the 0 day. At the stage of 7 days interval of harvesting, the fruits showed a lower level of pesticide concentration which was the similar decision of Chowdhury *et al.* [35]. So, it's a clear observation that, after time interval the concentration would be disappeared.

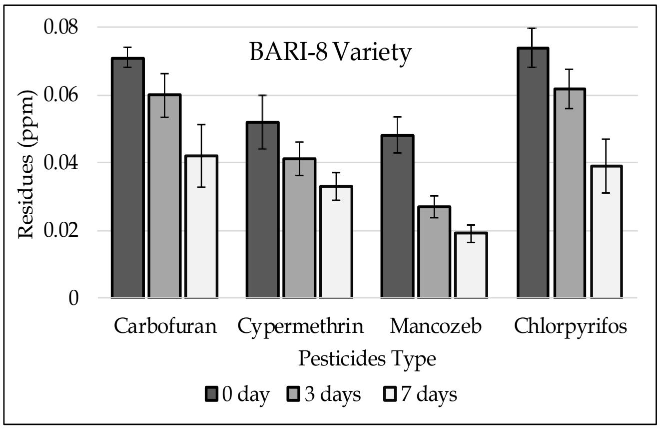

Figure 10: Pesticide residues in BARI-8 with day variation.

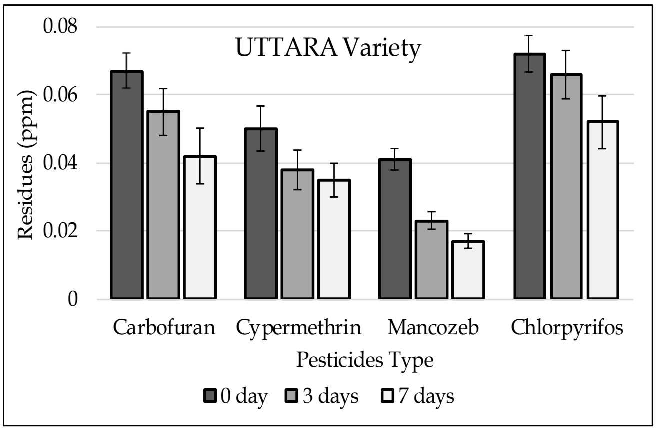

Figure 11: Pesticide Residues in UTTARA with day variation.

Few researcher [14, 36, 37] suggested that by following the recommendation dose and maintained day interval harvesting had a positive response on the residue reduction of pesticide. This study was undertaken for the identification of residue reduction system where a good feedback was obtained as well as that had a similar observation of previous researcher.

Reduction Factors (R_f) of Pesticide Residues

The chromatograph experiment of brinjal fruits for both varieties showed reduction factor (R_f) varying from 0.164 to 0.230 ppm (Table 2). Depending on the day variation, a significant change was observed and which was agreed to the observation of Iqbal *et al.* [38] where, the reduction factor varied from 0.050 to 0.231 ppm and the sample amount was 5 for Carbofuran, Chlorpyriphos, Imidacloprid, Dichlorovos and Acephate pesticides.

Table 2: Reduction factors (R_f) for BARI-8 and UTTARA on depending day variation.

Reduction Factor per 3 days Variation (ppm)				
Variety	**Carbofuran**	**Cypermethrin**	**Mancozeb**	**Chlorpyrifos**
BARI-8	0.195	0.198	0.230	0.210
UTTARA	0.185	0.164	0.225	0.168

The highest reduction rate for BARI-8 was obtained 0.230 ppm in Mancozeb concentration. On the other hand, 0.225 ppm of highest reduction rate was observed in UTTARA fruits for Mancozeb also. Sardana *et al.* [39] conducted a study and indicated about a reduction factor for their results analysis and depending on the residue reduction system. Table 2 revealed that a continuous reduction rate for all pesticides and varieties. From the table, it's a clear observation that the reduction rate of pesticides residues was decreasing with a similar reduction value.

Average Residual Comparison of Both Varieties with Time Variation

Average residues of both brinjal varieties were calculated during the chromatograph. By the day variation, most of the pesticide residues had a trend to reduce the pesticide concentration. Figure 12 revealed that the average value for all day variation of both BARI-8 and UTTARA had a reduction trend to the lower limit of pesticide residue.

The highest average pesticide residue (0.058 ppm for BARI-8 and 0.063 ppm for UTTARA) was observed for both brinjal varieties at 0 day of pesticide spray and lowest concentration (0.031 ppm for BARI-8 and 0.027 ppm for UTTARA) observed in the 7 days intervals which trend was almost similar to the observation of Jyot et al. [40] and Ahuja et al. [41] who observed the trend of reduction within 4 to 7-days variation. From the previous research study, most of the results revealed that the residues of pesticide reduced individually or averagely with time interval. The trend of average residues reduction may be reached a lower limit within more day's variation.

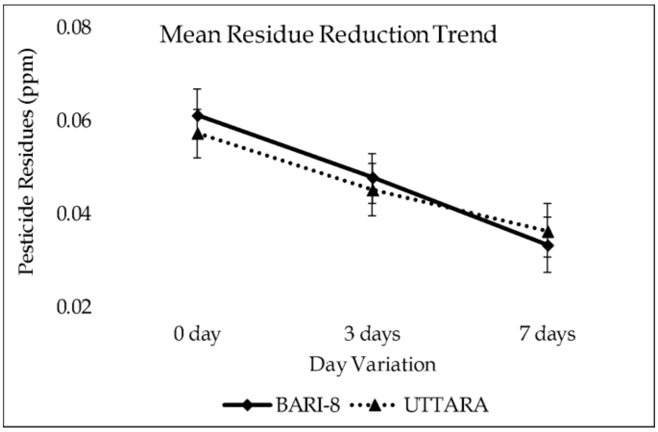

Figure 12 : Average pesticide residue reduction trend with day variation of both BARI-8 and UTTARA.

Comparison of the Residues with MRL for Both Brinjal Varieties

An experimentation of two varieties of brinjal fruits (BARI-8 and UTTARA) had applied four specific pesticides and that responded a significant change in residues on depending the day variation. All of the varieties received the same dose of pesticide where BARI-8 up took pesticide concentration more than the UTTARA variety. Table 3 revealed that on the 0 day of harvesting results for both varieties (0.071, 0.052, 0.048, 0.074 ppm and 0.067, 0.050, 0.041, 0.072 ppm for BARI-8 and UTTARA respectively of four pesticides) exceeded the MRL with a highest significant for all pesticide residues. On the 3 days interval of spray results showed only chlorpyrifos (0.062 ppm) for BARI-8 below the MRL as well as carbofuran (0.055 ppm) and chlorpyrifos (0.066 ppm) for UTTARA within the MRL. Besides, on the 7 days interval the pesticide residues (0.042, 0.033, 0.019, 0.039 ppm and 0.042, 0.035, 0.017, 0.052 ppm for BARI-8 and UTTARA respectively) for all selected pesticide under the MRL of both varieties. UTTARA brinjal fruits had more trend to reduce the pesticide residue within day variation than the BARI-8 variety. Ahuja *et al.* [41] and Kaur *et al.* [42] observed the pesticide residues for lambda – cyhalothrin, cypermethrin, decamethrin pesticide of the aubergine fruits and brinjal within 10 days variation but these pesticide residues and that reached the MRL within 10 days variation from the spray. Another study was conducted by Iqbal *et al.* [38] and they revealed that on the 7 days interval the concentration of Carbofuran, Chlorpyriphos, Imidacloprid, Dichlorovos and Acephate residues of 0.026, 0.035, 0.039, 0.034 and 0.028 ppm respectively in brinjal reached below the MRL but the 0-day residues were 0.084, 0.095, 0.097, 0.070 and 0.071 ppm. The significant results were obtained when comparing to the MRLs. Most of the pesticide concentration violated the MRLs except some pesticides below the MRLs. Although four selected pesticides in brinjal fruits are so dangerous to exposed, 7 days interval from the spray will safe to consume.

Table 3 : Comparison of the residues with MRL for BARI-8 and UTTARA.

Variety	Time Variation	Carbofuran (ppm)	Cypermethrin (ppm)	Mancozeb (ppm)	Chlorpyrifos (ppm)
BARI-8	0 day	0.071	0.052	0.048	0.074
	3 days	0.060	0.041	0.027	0.062
	7 days	0.042	0.033	0.019	0.039
UTTARA	0 day	0.067	0.050	0.041	0.072
	3 days	0.055	0.038	0.023	0.066
	7 days	0.042	0.035	0.017	0.052
MRL		**0.05**	**0.03**	**0.01**	**0.06**

III. CONCLUSIONS

The obtained results from the experiment of both in the brinjal varieties, all of the samples were showed the response to the residue reduction. The experiment was conducted depending on the 0, 3- and 7-days variation and the residues had a trend to reduce. The GC system had the responded for the eluted peak as well as depending on the peak areas and standard solution concentration, straight-line curves were drawn. All of the samples were contained pesticides which detectable. Overall the study, BARI-8 variety had a strong significance to more pesticide absorption than the UTTARA variation. The interaction of pesticides between BARI-8 and UTTARA had a significant response to the time variation. After 7-days interval, all of the residues were minimized and reached below the MRL. Considering the consumer safety in Bangladesh, brinjal as well as other vegetables should not be eaten at least 3-5 days interval from the pesticide spray. The whole study revealed a decision that the reduction rate or factor played an important role for pesticide residue trend for all pesticide and varieties. It's a risk to consume the pesticide contaminated vegetables within the short-day variation of pesticide spray. The cultivators of the vegetables should follow the recommended dose of pesticide and harvest them depending on the recommended day variation for specific vegetables. So, it's easy to say at least that the public awareness and following the recommended dose can be effective to reduce the risk of pesticide related diseases as well as the environmental contamination.

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Eco-friendly Candy Preparation from Paneer Whey

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ABSTRACT

In present study the preparation of candy from paneer whey. The paneer whey obtained from coagulated cow and buffalo milk. In coagulant milk the liquid part is a whey and solid part is a paneer. In paneer whey the 50% solid is prevent their for paneer whey is used in candy preparation.

Keywords: Buffalo Milk, Coagulant Milk, CMC, Fat

I. INTRODUCTION

In 80's and 90's the milk production increases due to dairy institute and farmers. Milk production of country crossed the limit of 130 million tons per annum. Out of 100 % milk the 46 % milk is raw liquid milk consumption and remaining milk is processed under organised and unorganised sector i.e. 54%. Out of 54%, 50% milk is processed and converted into traditional dairy products and remaining 4 % converted into western type of products. Out of traditional dairy products, major quantity of milk is converted into acid coagulated products. Acid coagulated products produces large quantity of Whey products. This Whey is either converted in Whey Powder or disposed as it is. This causes loss of about 50 % of milk solid which is to be prevented.

II. METHODS AND MATERIAL

Material: Milk, Ingredients like sugar, stabilizer, butterscotch, nuts and dry fruits.

Methods:

Collect the fresh whole milk then pre heating of this milk at $35–40^0$c after pre-heating filter the milk then standardization takes place. After standardization condense the 2:1 concentration then addition of whey in condensed milk and sugar simultaneously as per treatment then stir the mixture and add the stabilizer at the rate 0.5% (CMC) then addition of Nuts and dry fruits. After total addition of ingredients freezing below 0^0c for 4 to 6 Hrs. After cooling Kulfi is ready to serve.

Preliminary trials:

Pre-experimental trials have been conducted to decide the levels of addition of whey and sugar in the candy. The samples of paneer whey based candy have been subjected to sensory evaluation. On the basis of results of sensory evaluation, treatments are finalized for experimental trials. Preliminary trails have been conducted in phased manner as follows;

Phase 1: Whey: Milk proportion

W_1 = 25:75:: Whey: Milk
W_2=30:70:: Whey: Milk
W_3=35:65:: Whey: Milk
W_4=40:60:: Whey: Milk
W_5=50:50:: Whey: Milk with 12% constant level of sugar.

Pre-experimental trials were conducted to select the levels of whey: milk, sugar and stabilizer. Cow milk was used in this study contained on an average 3.77, 3.65, 4.39, 0.69 and 12.52 per cent fat, protein, lactose, ash and total solids, respectively. The S.N.F content in cow milk was 8.75 per cent. The milk: whey levels were selected by the judges on basis of organoleptic properties. The milk: whey levels were selected as 75:25, 70:30, 65:35, 60:40 and 50:50 for experimental trials. The sugar level selected for final experimental trials was 12% over and above candy mix. The prime motto to examine sugar level was to nullify the sour taste which is developed by increased content of whey in candy. For final experimental trials, CMC with 0.15% concentration was selected.

Table 1. Final Treatment Table

Sr. No.	Treatment	Ratio	Concentration of sugar
1	T1 (W1 : M1)	(25 : 75)	12%
2	T2 (W2 : M2)	(30 : 70)	12%
3	T3 (W3 : M3)	(35 : 65)	12%
4	T4 (W4 : M4)	(40 : 60)	12%
5	T5 (W5 : M5)	(50 : 50)	12%

Where, T = Treatment, W= Whey, M = Milk.

Whey content: Lactose, Vitamins, Proteins, minerals, and traces of Fat.

Result:

Table 2. Result Shows in Average Value:

Treatment	Colors & Appearance	Flavours	Body & Texture	Overall Acceptability
T1(25:75)	6.2	6.5	9.0	7.1
T2(30:70)	6.4	6.8	8.4	7.2
T3(35:65)	6.5	7.0	8.0	7.4
T4(40:60)	6.7	7.0	8.0	7.8
T5(50:50)	6.8	7.5	7.9	8.0

III. CONCLUSION

It may be concluded that treatment T5 having 50 percent milk and 50 percent whey is most acceptable among whey candy in respect of chemical, organoleptic and microbiological qualities.

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