International e-Conference on New Horizons And Multidisciplinary Applications In Science And Technology
In Association withInternational Journal of Scientific Research in Science and Technology
Volume 9 | Issue 6 | Print ISSN: 2395-6011 | Online ISSN: 2395-602X (www.ijsrst.com)

Synthesis of Cobalt Oxide Nanoparticles and Characterization through Various Spectroscopic Techniques

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ABSTRACT
The cobalt oxide nanoparticles were synthesized by chemical reduction approach. The synthesized cobalt oxide nanoparticles were characterized through various spectroscopic techniques which includes Fourier transform infra-red spectroscopy (FT-IR), x-ray diffraction spectroscopy (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM). FT-IR spectrum of Co3O4 nanoparticles showed significant absorption peaks at 588.74 and 664.69 cm-1. From the X-ray diffraction pattern of chemically synthesized Co3O4 nanoparticles. The size of the nanoparticles was calculated through the Scherer's formula, $D=0.94\lambda/\beta \cos\theta$, the size and morphology of the nanoparticles was studied using scanning electron microscopy (SEM). The nanocrystalline nature of the chemically synthesized Co3O4 sample was further confirmed by Transmission electron microscope.

Keywords : - cobalt oxide (Co3O4), FT-IR, XRD, SEM, TEM.

I. INTRODUCTION

Nanotechnology
The first ever concept was presented in 1959 by famous Professor of physics Dr. Richard Feynman and term nanotechnology was coined by an eminent scientist professor Norio Taniguchi in 1974. "Nanotechnology" is a technology of applied science which works in the field of theoretical as well as experimental changes at molecular level of compound [1,2]. The word nano derived from Greek word which means extremely small at nano level the prefix nano a size of 10^{-9}. It allows the arrangement of small structure with accuracy, in intelligibility and in expensive Drexler is known as the father of nano technology is the man who explains the nano technology in depth and the popularized the subject [3], he is an American engineer known for increasing the value of molecular nano technology. Nano technology has a break through as a multidisciplinary scientific field and is undergoing uncontrollable development as the products of nanotechnology are nano particle, nanotubes, and nano rods, nanospheres that have size below 100 nm and have high surface area to volume ratio [4]. The minimization of size brings about marked changes in their morphological properties with respect to thus properties observed in massive materials. The products of nano technology can be of metallic nature,

having mineral and polymer-based materials [5]. Nano technology is big tree of research having branches in every dimension and touches in different fields like cosmetics, electronics, packaging, biosensor medicine, paints, automobiles, bioengineering, and catalysts in Canada there are 80 companies that make 150 products that use 88 different nano materials. Nanomaterials are used in various applications, such as drug delivery [6] bio-separation, magnetic refrigeration systems, gas sensing, catalytic application, absorbent materials [7], ultra-high-density recording, magnetic resonance imaging, data processing devices, microwave and radio frequency devices, multilayer chip inductor and electromagnetic interference (EMI) suppression [8]. These applications of nanomaterial depend on its structural, magnetic and electrical properties.

Metal oxide nanoparticles:

Bi_2O_3, CeO_2, CuO, ZnO and Co_3O_4 are some metal oxide nanoparticles These particles have wide range of properties like magnetic, electronic, and optical properties which are used in different fields like agriculture, medicine and electronics these particles pusses a large surface area in which it acts like a substrate for the electron transfer reaction just before the electron transfer reaction both reactants are adsorbed upon the surface of metal consequently there will be gain of an electron by reactants and then reduced thus through the electron transfer process cobalt oxide nanoparticles will acts as an efficient catalyst [6-11] .

In recent years, there has been an increasing interest in the synthesis of nanosized crystalline cobalt oxide (Co_3O_4) nanoparticles because of their large surface area, unusual adsorptive property, surface defect and fast diffusivity. Co_3O_4 is an important transition metal oxide because of its application in various field of research and industry include pigments, gas sensor, magnetic materials, catalyst, anode materials for rechargeable Li batteries, electrochromic devices, electrochemical systems and high-temperature solar selective absorbers Co_3O_4 is a very important material extensively used in catalysis, gas sensors, electrochromic films, battery cathodes, heterogeneous catalytic materials and magnetic materials [12-15]. Due to their small size, nanoparticles exhibit novel material properties that are significantly different from those of their bulk counterparts [16-21]. Cobalt oxide is a well-known p-type semiconducting material of its electronic and electro optic properties. Co_3O_4 is an important ceramic oxide used for electro chromic and, magnetic and catalytic applications [22]. Electrochromism is characterized as a reversible change of the optical properties of the material by the application of a voltage stimulus and Co_3O_4 represents a promising anodic electrochromic material [23]. Its optical properties have attracted considerable attention. Co_3O_4 is a very important material extensively used in catalysis, gas sensors, electrochromic films, battery cathodes, heterogeneous catalytic materials and magnetic materials. Cobalt oxide (Co_3O_4) is a promising material for use as a gas sensor and catalyst in hydrocracking processes of crude fuels, pigment for glasses and ceramic [24-27]. The cobalt oxide nanoparticles play an important role as a catalyst in water electrolysis process [28].

Materials required: -

Cobalt Chloride and Sodium Succinate was obtained from Sigma- Aldrich chemicals, polyvinyl pyrrolidone (PVP) was procured from Merck, India. 99% hydrazine hydrate was taken from Loba chemise. Malachite green (MG), Eriochrome Black-T (EBT), Bromophenol blue (BPB), Methyl Red (MR) were taken from Loba chemise. All the chemicals used as received.

Preparation of cobalt oxide nanoparticles

In a 250 ml conical flask 1g cobalt chloride was dissolved in 10ml of distilled water with help of magnetic stirrer. In another beaker 3.6g sodium succinate was dissolved in 50 ml distilled water. Sodium succinate solution was added into cobalt chloride solution at 70^0 c with continuous
stirring for 10 min. 0.05g PVP was weighed and dissolved in 6ml distilled water. Transfer this PVP solution in cobalt chloride solution and heated for another 5 min. Now added approximately 1.5ml hydrazine hydrate into the above solution drop wise with constant heating. Color changes from pink to grayish. Filtered solution through whatmann filter paper, washed with absolute alcohol and acetone, dried in oven at 60^oc. After drying this dried powder was calcined in muffle furnace at 450^oc for 3hrs. Black colored powder was obtained which is further characterized by various spectroscopic techniques.

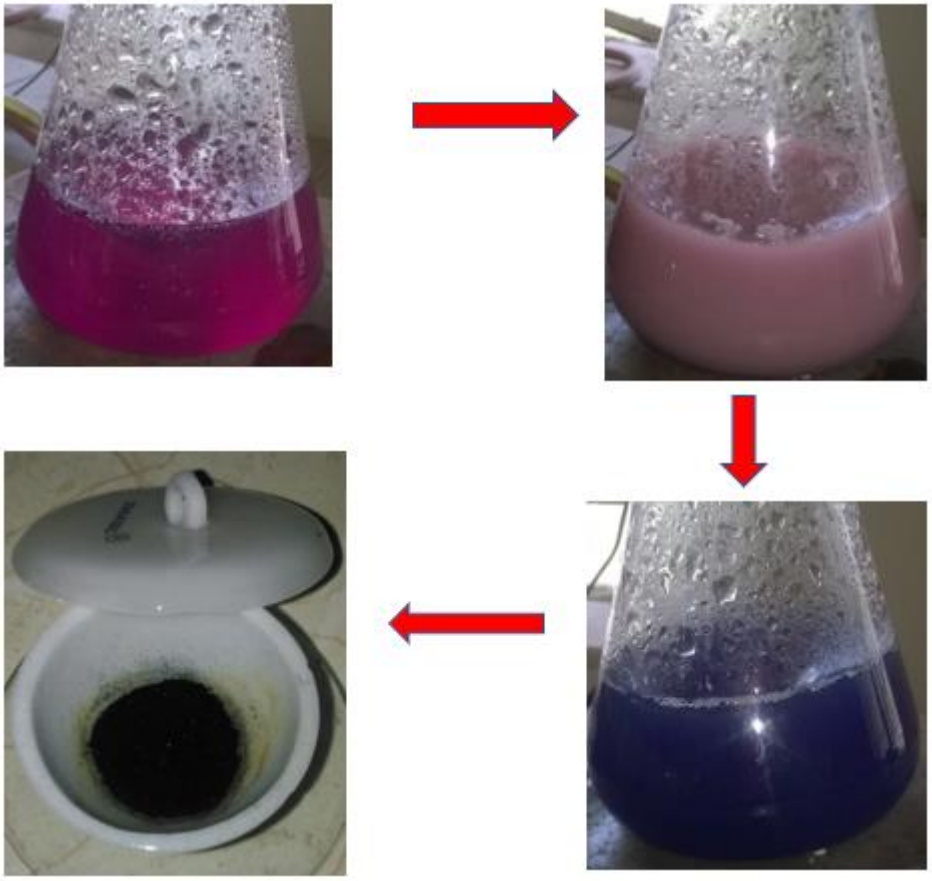


Fig 1: Process of synthesis of cobalt oxide nanoparticles by Chemical reduction method

Results and Discussion: -

1. FT-IR

Figure 2. shows FTIR spectra of Co_3O_4 nanoparticles synthesized by chemical reduction method. FTIR spectroscopy was carried out in order to ascertain the purity and nature of metal or metal oxide nanoparticles. FT-IR spectrum of Co_3O_4 nanoparticles showed significant absorption peaks at 588.74 and 664.69 cm-1. The absorption band at 588.74 cm-1 was assigned to Co-O stretching vibration mode and 664.69 cm-1 was assigned to the bridging vibration of O-Co-O bond. The absorption peak at 1049.86 cm-1 may be due to $–CH_3$ stretching

vibrations. The absorption peaks at 1627.37, 3422.02 cm-1 may be due to –CH_2 stretching, and –CH stretching vibrations.

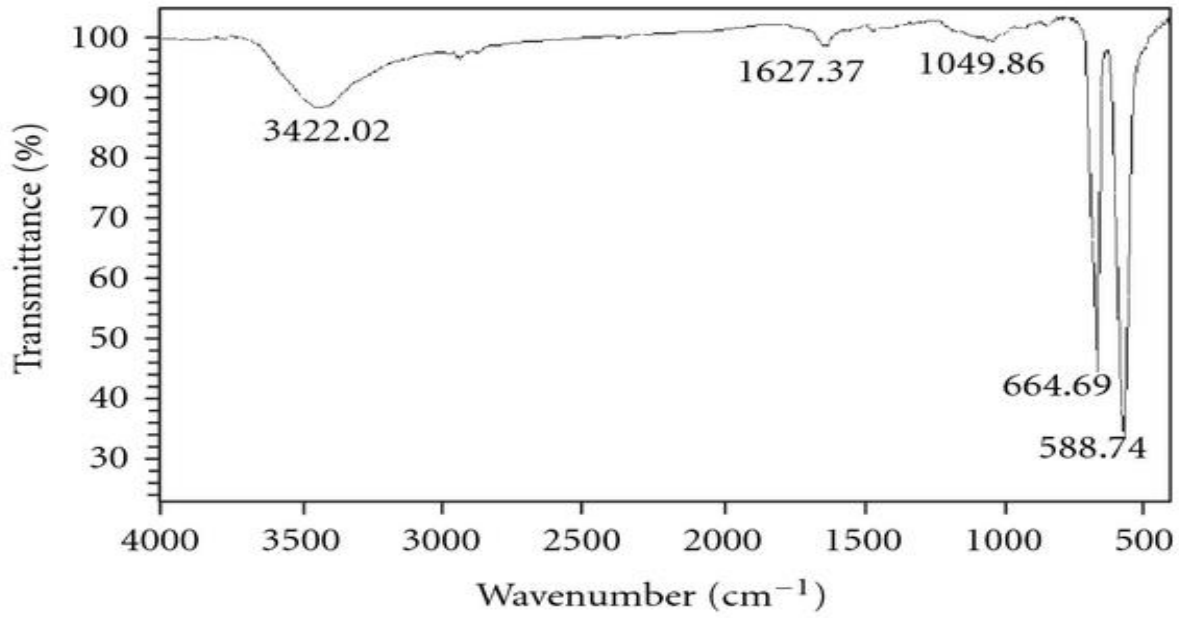


Figure 2 : FT-IR spectrum was recorded for cobalt oxide nanoparticles after calcination at 450^0 C for 3 Hours.

2. X- ray diffraction studies: -

Fig.3 shows the X-ray diffraction pattern of chemically synthesized Co_3O_4 nanoparticles. Several dominant 2θ peaks were observed. According to JCPDS data (74-2120), the distinct diffraction peaks at 2θ = 18.972^o, 31.383^0, 36.81^0, 44.708^0, 59.375^0, 65.232^o can be well indexed to the cubic phase and corresponds to (1.1.1),(2.2.0),(3.1.1),(4.0.0),(5.1.1),(4.4.0), plane respectively of crystalline Co_3O_4 . Any other characteristics peaks are not found, indicating that the as- prepared Co_3O_4 is pure. The strong and sharp diffraction peaks implied that the as- prepared Co_3O_4 nanoparticles possess good crystalline nature. The size of the nanoparticles was calculated through the Scherer's formula, $D=0.94\lambda/\beta \cos\theta$, Where D is the average crystal size, β is the half-height width of the diffraction peak, θ is the diffraction angle, and λ is the X-ray wavelength (0.1541 nm). The average particle size of the bio synthesized $Co_3O_4$was 49.27nm.

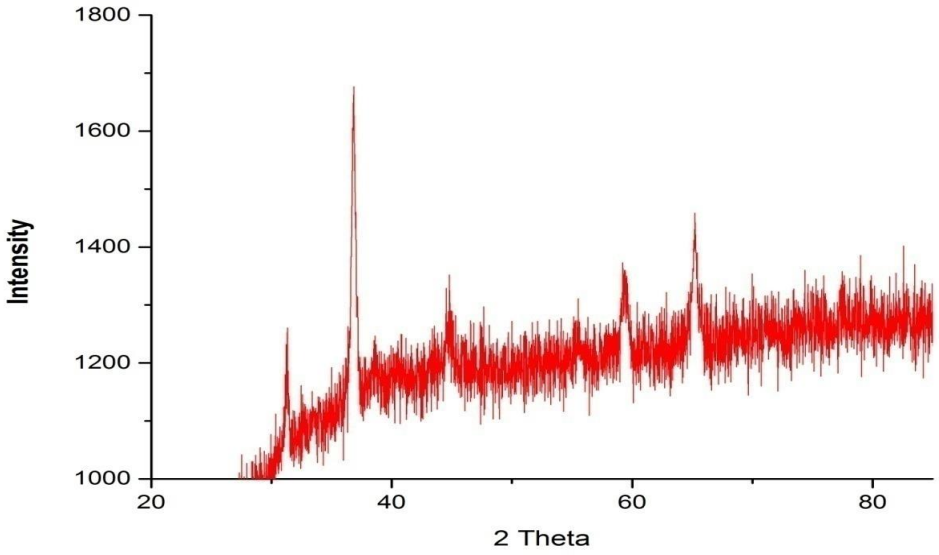
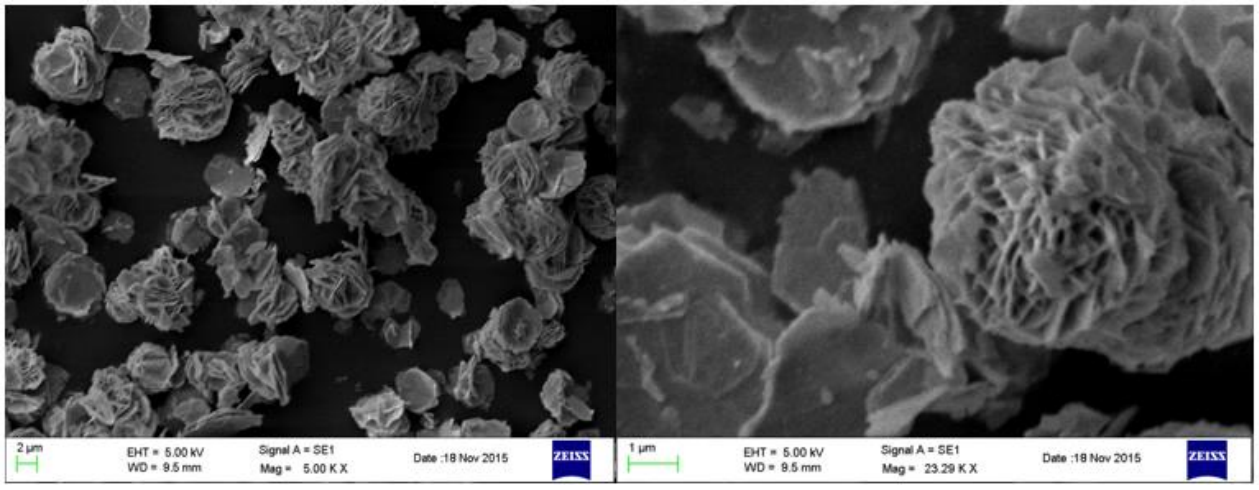


Fig 3: X-RD spectrum was recorded for cobalt oxide nanoparticles after calcination at 450°C for 3 hours

3. Scanning electron microscopy analysis

The size and morphology of the nanoparticles was studied using scanning electron microscopy (SEM). SEM analysis of chemically synthesized Co_3O_4 nanoparticles was shown in Fig. 4, which depicts the particles were agglomerated and flower shaped like structure. It might be beneficial for its photocatalytic activity improvement because large surface area can enhance more photon absorption.



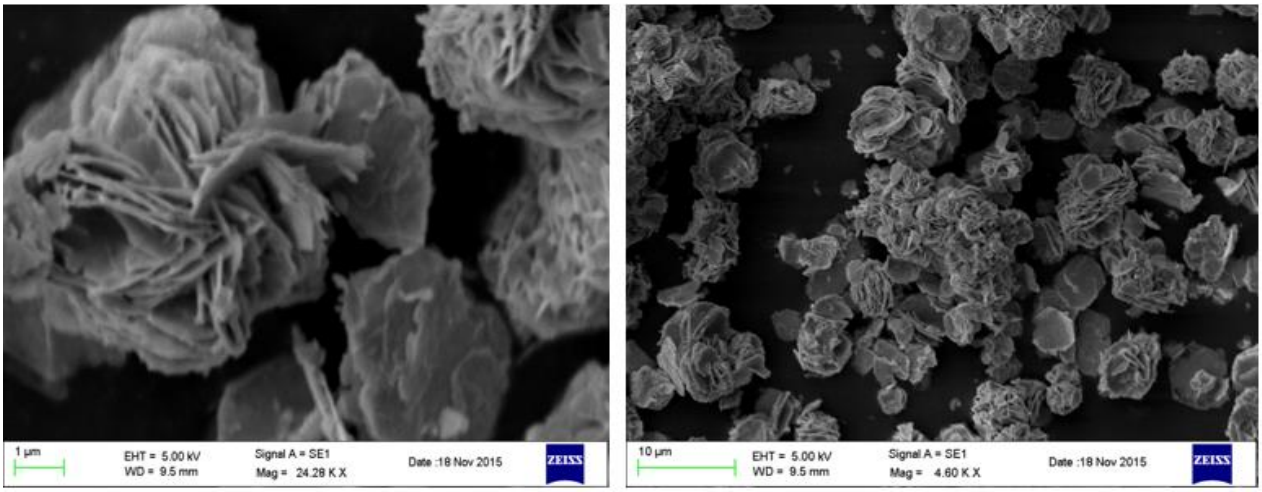


Fig 4 : SEM images were recorded for cobalt oxide nanoparticles after calcination at 450°C for 3 hours.

4. TRANSMISSION ELECTRON MICROSCOPE ANALYSIS (TEM)

The nanocrystalline nature of the chemically synthesized Co_3O_4 sample was further confirmed by Transmission electron microscope image. Figure 5 shows, the bright field TEM micrograph of the prepared cobalt oxide sample. This image represents the crystalline morphology with mostly irregular particle shape and particles are in nano range. The particles seems to be closely joined together to form fine nano spheres.

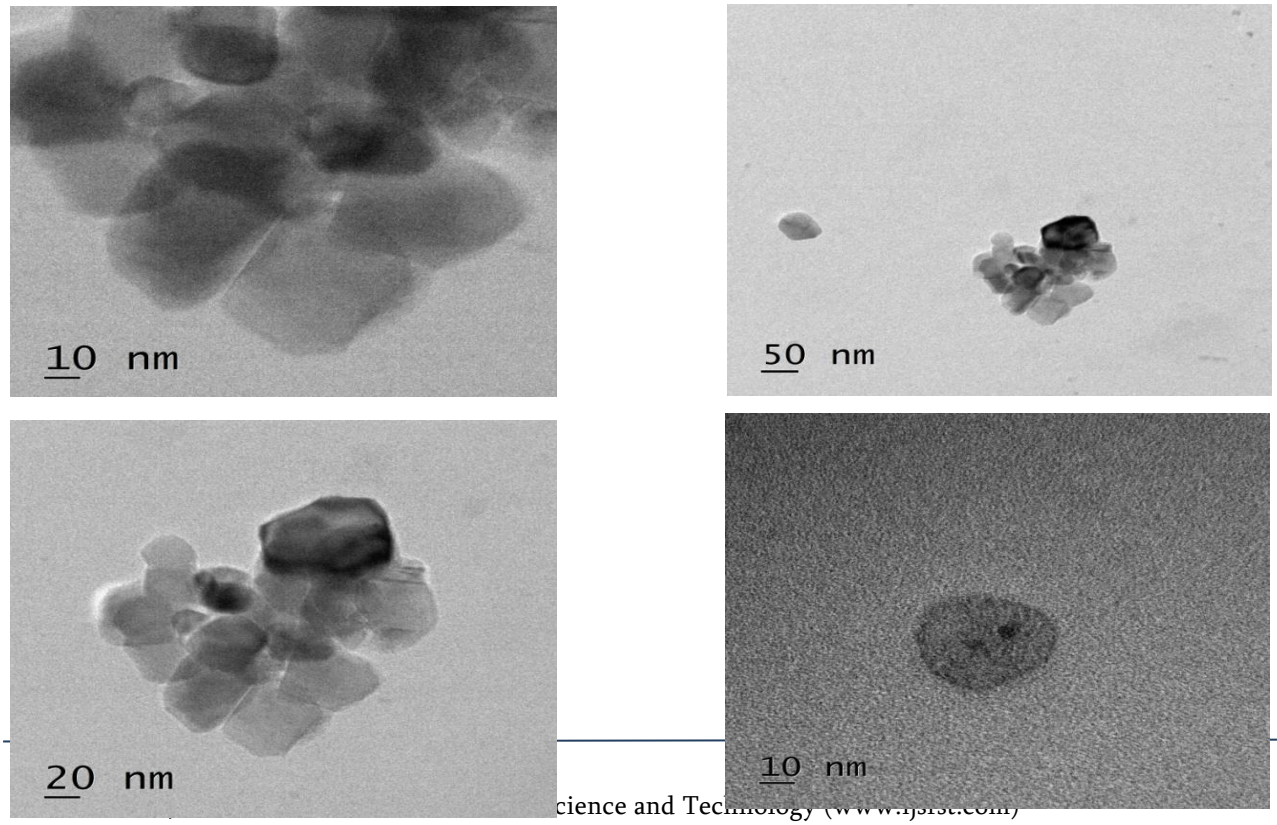


Fig 5: TEM images were recorded for cobalt oxide nanoparticles after calcination at 450°C for 3 hours

Conclusion

1. In FT-IR spectrum of Co_3O_4 nanoparticles synthesized by chemical reduction method has significant absorption peaks at 588.74 and 664.69 cm-1. The absorption band at 588.74 cm-1 was assigned to Co-O stretching vibration mode and 664.69 cm-1 was assigned to the bridging vibration of O-Co-O bond.

2. The average particle size of the bio synthesized $Co_3O_4$was 49.27nm.

3.The particles were agglomerated and flower shaped like structure.

4.Mostly irregular particle shape and particles are in nano range. The particles seem to be closely joined together to form fine nano spheres.

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