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Synthesis of 3-Phenyl coumarin by Using Phase Transper as a Green Catalysts

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ABSTRACT

Coumarins play an important role in biological activities and perfumery industries, their derivatives are widely used. In this method 3- Phenyl Coumarins prepared by using Salicylaldehyde, Phenyl acetyl chloride, Toluene, PTC, aqueous NaOH solutions .The reaction is simple and one pot. The aqueous layer and solvents recycled for next reactions .The kinetic study also done. Thus due recyclability of catalyst, solvent and raw materials, the process adheres principles of green chemistry.

Keywords: Salicylaldehyde, Phenyl Acetyl Chloride, Toluene, Sodium Hydroxide, Different Phase Transfer Catalysts, Automatic Hot Plat With Stirring Setup.

I. INTRODUCTION

Coumarins and their derivatives has biological activities such Anti HIV, Antidiabetics, Antifertility, Anticoagulents, Antimicrobial, optical brightening agents etc. [1-14] .

A new method for the synthesis of 3- phenyl coumarin by the reaction of salicylaldehyde in aqueous sodium hydroxide with phenyl acetyl chloride in toluene in presence of ionic liquid tetra phenyl phosphonium bromide as biphasic catalyst has been developed. The method is simple, one pot reaction and satisfies the rules of Green Chemistry. The phase transfer catalysts, ionic liquids and their applications including the use of biphasic green catalysts and the objectives of the present investigation.

The chemical reagents used, various instruments used and the details of the procedures for the preparation of phenyl acetyl chloride and 3- phenyl coumarin have been described.

Green Chemistry addresses not only safety but also development of technology to ensure that the chemical reaction produce less waste and pollution. Increased local and global concern for environmental pollution offers incentive to explore new green materials for safeguarding the environment. Green materials have been applied in the design. A phase transfer catalyst or PTC in chemistry is a catalyst which facilitates the migration of a reactant in a heterogeneous system from one phase into another phase where reactions can take place. Ionic reactants are often soluble in an aqueous phase but insoluble in an organic phase unless the phase transfer catalyst is present. Phase transfer catalyst refers to the acceleration of the reaction by the Phase transfer catalyst.

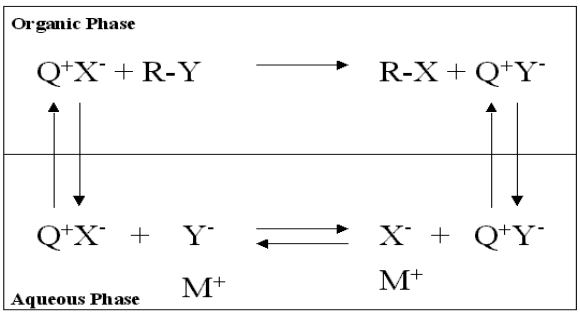
In many methods there is use of $POCl_3$, Phosphotungstic acids, $ZnCl_2$, $AlCl_3$, H_2SO_4 ,PPh_3 ,NaH, anhydrous condition , Sodium Carbonate, Solid Amberlyst 15 ,Ultrasonic irradiation, microwave oven . Almost all methods suffer drawbacks such as use of active compounds, corrosion problems, high temperatures, expensive catalysts, corrosive condensing agents, environmental problems.

Among the various technique Scientists use greener methods that is ultrasonic irradiation and microwave

Techniques that gives good yields and fast reaction but that technique unable to scale up for commercial production. In our method simple stirring a mixture of Salicylaldehyde in aqueous NaOH , PTC and phenyl acetyl chloride at 40 ^{0}C for 3 hours. The reaction was monitored by TLC .The reaction mass was extracted in Toluene and aqueous layer was recycled for next batch. Coumarins are prepared by various Phase transper catalysts (PTC) in chemistry.

These phase transper catalysts are catalysts which are used in pollution prevention. PTC facilitates the migration of reactants from one phase into other phase where reaction can take place. In developing green technology Scientist mainly concentrate, how the methods becomes greener, decrease steps, increase quality and yield. In this we use various PTCs and study the reaction with optimum yields. In recent there has been much interest in synthesis by using PTCs. In our study, the aqueous layer was recycled in next batches because PTCs are highly soluble in water.

The Extraction Mechanism of PTC



II. METHODS AND MATERIAL

All raw materials were purchased from S. D. Fine Chem , Mumbai and used as such without further purifications .

TPPBr –Tetraphenyl phosphonium Bromide,TBAB – Tetrabutyl ammonium Bromide,BTEAC – Benzyl Triethyl ammonium chloride,TMAOH – Tetramethyl ammonium Hydroxide.

Phenyl Acetyl Chloride was prepared in lab by known methods [5-6] . In this method ,Phenyl acetic acid add thionyl chloride and one drop of Dimethyl Formamide with reflux mode setup .The reaction mass was gradually heat under stirring to reflux for 2 hours on water bath in fuming cupboard. After digestion the reaction setup changed to distillation mode with vacuum arregements, excess thionyl chloride was distilled under reduced pressure and main product distilled under full vacuum to clear ,colorless liquid and stored in freeze.

In 50 Mls reaction flask with heating, stirring arrangement as shown in Figure. In the reaction flask taken aqueous solution of sodium hydroxide [6 MMol] under stirring add salicylaldehyde [1 MMol].The temperature of reaction mass goes to 40 ^{0} C when it comes down Phenyl acetyl chloride [2.2 MMol] added due to exothermic temperature reaction temperature increase to 40 ^{0}C .The reaction mass heat to 40 ^{0}C and every half hours reaction product monitored by TLC. When reaction completes. In the reaction mass add Toluene and stir well to extracts product in Toluene layer ,separate Toluene layer and dry on sodium sulphate and distill out Toluene .The bottom contains product was crystallise in Ethanol . Dry in Oven.

The same method was followed for other catalysts. The different parameters are studied to get optimum product. These parameters are as. Mole ratio of reactants, Temperature, Solvents Catalyst loading and Time.

III. RESULTS AND DISCUSSION

Different phase transfer catalysts are used and their recyclability was studied. In differents catalysts Tetabutyl ammonium bromide and Tetaphenyl phosphonium bromide gives higher yields .Kinetic study shows which solvents,temperature,mole ratios,time gives higher yields and same parameters

are apply to this methods. Duo to reuse of solvents and recycle of aqueous layer methods adheres to green chemistry. The product was characterized by MP, GC, IR, and Mass Spectroscopy.

Tables1. Kinetic Study

Sr.No.	Parameters	Moles taken	% Yields
1	Sodium Hydroxide	6 MMol	98
2	Salicylaldehyde	1 MMol	98
3	Time	180 Minutes	98
4	Solvent	Toluene	98

Table 2. Phase Transper Catalysts

Sr No.	Name of PTC	Time (Minutes)	Yield %	M.P. ^{0}C
1	TPPBr	180	98	140
2	TBAB	180	98	140
3	Aliquat	180	39	140
4	BTEAC	180	10	141
5	18 Crown Ether	180	15	141
6	TMAOH	180	5	140
7	Choline chloride	180	10	140

IV. CONCLUSION

The method developed for the synthesis of 3- phenyl coumarin reported in thesis is simple, one pot reaction and adheres to the rules of Green Chemistry of EPA.

The yield of 3- phenyl coumarin prepared by this method is 98.37% which is fairly good compound to the previously reported methods. The product can be easily purified by crystallistion alone without the use of any costly separation techniques. The catalyst quantity required is very less [$1x10^{-4}$ M] as compared to the reactants.

The method is simple and one pot. The product isolated was characterized by GC, IR, MP and Mass Spectrometry. Author is thanks to authority of Kirti College, Dadar for all supports of this works at all times.

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Author is thanks to authority of Kirti College for all supports of this works at all times of works.

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