

ULTRASONIC ASSISTED AND CONVENTIONAL SYNTHESIS OF SCHIFF BASE AND METAL COMPLEXES: A COMPARATIVE STUDY

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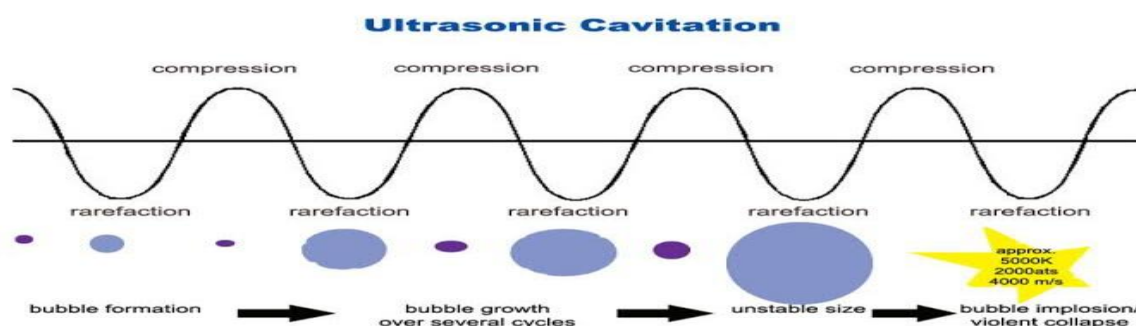
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ABSTRACT

Green chemistry is the latest and most researched topic now-a-days. Ultrasonication is technique in which ultrasonic (> 20 KHz) waves are irradiated into a liquid sample resulting in agitation . This method is now-a-days a well considered technology in green chemistry being advantageous over the conventional thermal methods as increased reaction rates, formation of purer products, improved yield, increased selectivities, easier experimental procedures and use of milder conditions both in case of heterogenous and homogenous reactions. Here, we were synthesized 2-(salicylideneamino) benzoic acid and its metal complexes by conventional and ultrasonic irradiation methods. Synthesized schiff base acts as chelating agent in the preparation of metal complex.

I. INTRODUCTION

Compounds containing azomethine group (-C=N-) are formed by condensation of primary amines with carbonyl compounds, such compounds are known as schiff bases. Schiff bases and their metal complexes exhibit various biological applications including antibacterial^[1-6] , antifungal^[2,6] , antioxidant^[7,8] , anti-inflammatory^[9] , antihypertensive^[10] , antitumor activity^[11,12] , anticonvulsant^[13] anti-HIV^[14] , antifilarial^[15] , herbicidal, insecticidal, schistosomicidal and antihelmintic^[16] activities. Schiff bases also act as chelating ligand. These are also used as protective agents in natural rubber^[17]. Impact of ultrasound waves on chemical reactivity is explained by using term 'Sonochemistry' . Ultrasound irradiation method for organic synthesis as a green synthetic technique is a powerful method that is being increasingly used to accelerate organic reactions^[18-22]. Ultrasound waves have frequencies higher than the ones to which the human ear can respond (> 20 KHz) (Hz = Hertz = cycles per second). The ultrasonic frequencies of interest for chemical reactions are about 20-100 KHz. Sound waves propagating through a series of compression (high pressure) and rarefaction (low pressure) cycles cause molecules to vibrate around their mean position as they pass through a liquid medium. During the compression cycle (high pressure), the average distance between the molecules is decreased and during rarefaction (low pressure), the average distance between the molecules is increased. In the rarefaction cycle, bubbles can be formed by overcoming the attractive forces under appropriate conditions. If the internal forces are sufficiently large, the collapse of these bubbles can generate very high local temperatures (approximately 5000 °C) and pressures (over 1000 bar). It is very high temperatures and pressures that cause chemical reactions. Ultrasonic cavitation is the formation and collapse of cavities or bubbles in a liquid medium when low pressure is suddenly applied.



In the laboratory, it is usually employed by utilizing an ultrasonic bath or probe, this instrument identified as a sonicator [23]. Compared with traditional methods, this technique is more convenient and easily controllable. A large number of organic reactions can be performed under milder conditions with shorter reaction times, thus providing higher yields with ultrasonic irradiation [24]. This can be considered as a processing aid in terms of waste minimization and energy conservation compared to conventional heating.

II. MATERIALS AND METHODS

2.1. Synthesis of Schiff base: (compound 1)

a) By conventional method : The schiff base was prepared by adding 25 cm³ 2-hydroxy benzaldehyde (1.22g, 0.01mol) ethanol solution to an equal volume 2- amino benzoic acid (1.37g, 0.01mol) ethanol solution . The resulting reaction mixture was refluxed for 8-9 hours. The reaction progress was monitored by TLC. After completion of reaction, the formed product was collected by filtration, washed with ethanol and recrystallized from hot ethanol. The orange colour product is then dried in vaccum desiccator.

b) By ultrasound irradiation method: The schiff base was prepared by adding 25 cm³ 2-hydroxy benzaldehyde (1.22g, 0.01mol) ethanol solution to an equal volume 2- amino benzoic acid (1.37g, 0.01mol) ethanol solution . The resulting reaction mixture was then sonicated for 6 hours. The reaction progress was monitored by TLC. After completion of reaction, the formed product was collected by filtration, washed with ethanol and recrystallized from hot ethanol. The orange colour product is then dried in vaccum desiccator.

2.2. Synthesis of Fe (III) complex: (compound 2)

a) By conventional method : The Fe (III) complex was prepared by adding 25 cm³ of ethanolic solution of ferric chloride (1.62g , 0.01 mol) to the same volume of ethanolic solution of schiff base (2.41g, 0.01 mol) followed by addition of aqueous ammonia. The resulting reaction mixture was refluxed for 6 hours. When Fe (III) complex precipitated, filter it, washed several times with hot ethanol. The pale brown colour product was then air dried in vaccum desiccator.

b) By ultrasound irradiation method: The Fe (III) complex was prepared by adding 25 cm³ of ethanolic solution of ferric chloride (1.62g , 0.01 mol) to the same volume of ethanolic solution of schiff base (2.41g, 0.01 mol) followed by addition of aqueous ammonia. The resulting reaction mixture was stirred in sonicator for 4 hours. When Fe (III) complex precipitated, filter it, washed several times with hot ethanol. The pale brown colour product was then air dried in vaccum desiccator.

2.3. Synthesis of CO (II) complex: (compound 3)

a) By conventional method : The cobalt chloride hexahydrate (2.37g, 0.01 mol) in ethanol was mixed separately with ethanolic solution of ligand (2.41g, 0.01 mol) . The resulting reaction mixture was refluxed for 6 hours. The precipitation occurred, the solid metal complex was separated by filtration, washed with boiling ethanol. The off white colour product was then air dried in vaccum desiccator.

b) By ultrasound irradiation method: The cobalt chloride hexahydrate (2.37g, 0.01 mol) in ethanol was mixed separately with ethanolic solution of ligand (2.41g, 0.01 mol) . The reaction mixture was sonicated for 4 hours in sonicator. The precipitation occurred, the solid metal complex was separated by filtration, washed with boiling ethanol. The off white colour product was then air dried in vaccum desiccator.

2.4. Synthesis of Cd (II) complex: (compound 4)

a) By conventional method : The cadmium carbonate(1.72g, 0.01 mol) in ethanol was mixed separately with ethanolic solution of ligand (2.41g, 0.01 mol) . The reaction mixture was refluxed for 6 hours. The precipitation occurred, the solid metal complex was separated by filtration, washed with hot ethanol. The white colour product was then air dried in vaccum desiccator.

b) By ultrasound irradiation method: The cadmium carbonate(1.72g, 0.01 mol) in ethanol was mixed separately with ethanolic solution of ligand (2.41g, 0.01 mol) . The reaction mixture was sonicated for 4 hours in sonicator. The precipitation occurred, the solid metal complex was separated by filtration, washed with hot ethanol. The white colour product was then air dried in vaccum desiccator.

III. RESULTS AND DISCUSSIONS

The various products were synthesized by conventional method and ultrasound irradiation method as follows.

Sr.no.	Name of compounds	Conventional method		Ultrasound irradiation method		Colour	Melting point/ Transition temperature. (°C)
		% yield	Time (min.)	% yield	Time (min)		
1.	2-(salicylideneamino) benzoic acid	73	510	88	360	Orange	180°C
2.	Fe (III) complex of 2-(salicylideneamino) benzoic acid	69	360	85	240	Pale brown	235°C
3.	CO (II) complex of 2-(salicylideneamino) benzoic acid	71	360	84	240	Off white	240°C
4.	Cd (II) complex of 2-(salicylideneamino) benzoic acid	74	360	90	240	White	230°C

From the above data, we conclude that ultrasonic irradiation method gives excellent result as compared to the conventional methods for organic synthesis by considering time required to complete reaction and yield.

IV. CONCLUSION

Ultrasonic irradiation method has been proved here as a better method for synthesis of schiff bases and its metal complexes over conventional method. Ultrasonic irradiation method gives higher yield in shorter reaction times.

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