

Synthesis of Transition Metal Complexes of Schiff Base by Ultrasound Irradiation Method and Conventional Method –A Comparative Study

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Abstract — Ultrasound irradiation has allowed preparation of the synthesized product along good yield in extremely short reaction period. The quality of the product was determined by SEM- TEM, HNMR, CMR etc. The high intensity ultrasound is regarded as an easy environmentally-friendly and flexible synthetic instrument compounds of co-ordination. The ultrasonication supply the synthesized molecule in excessive yield and clarity in shorter reaction time differentiated along the conventional method. Ultrasound irradiation extensively utilized in the preparation of nano particles and polymer supported material. The reaction are selected that requires additional stirring for accomplishment and fulfilled the ordinary temperature through the conventional method correlated along the ultrasound irradiation technique. Ultrasound technique is an supportive also in photosynthesis of nano complex can be extremely interesting for manufacturing function. In actual fact chemical changes in the period ultrasonic irradiation can also be helpful certain areas of chemical synthesis. Time for the reaction was very much less through ultrasound irradiation and the amount of product of reaction was too dominant as differentiated to the conventional method using reflux condition. The incorporate Schiff base and our metal complexes remain distinguish by the spectroscopic technique like UV-Visible, IR, and ¹H NMR. The prepared Schiff base and there metal complexes showed satisfactory antimicrobial activity.

Keywords: Ultrasonication, Metal Complexes, Time, Yield, Salicylaldehyde, Anthranilic Acid, Purity, Organic Reaction

I. INTRODUCTION

The range of ultrasound frequency used commonly in sonochemistry is 20 KHz to 2 MHz but laboratory apparatus utilize range uniting 20 KHz to 40 KHz.[1] High powered range technique in chemical process is ultrasonic irradiation. Synthesis of ligand and metal complexes are much organic

synthesis where as it can be applied to numerous chemical reaction along outstanding result.[2-4]The clean and useful protocol in organic synthesis considered as ultrasound irradiation by last three decades. [5] Ultrasound irradiation has been utilize progressively in organic synthesis.[6-7]The effect of ultrasound irradiation besides the rate of chemical reaction was firstly introduced by Loomis and Richards in 1917. [8] Morad et al. also prepared Schiff base from salicylaldehyde and anthranilic acid. They synthesized Schiff base complex of the technique also studied the antibacterial activity of the above Schiff base and its metal complexes.[9]Under ultrasound irradiation, organic transformation occur in high yield, short reaction times or milder condition. [10-11]Comparing along conventional method ultrasonic irradiation is acceptable and clearly manage and it studied as a green powerful manufactured approach in chemical process. [12]Ultra sonication can bring various benefits such as environmental friendliness, power saving (no toxic chemicals are used or produced) cost-efficiency, compact in nature, solvent free. [13]Ultrasonic assisted synthesis is utilize in those reactions where very expensive reagents, high temperature and prolonged reactions condition are required. This technique is also used to increase the yield of the reaction. [14-16]Schiff bases are important class of compound in therapeutic and pharmaceuticals field. They show biological significance including antibacterial, antioxidant and anti-inflammatory activity. [17]Antibacterial activity of the prepared Schiff bases and there metal complexes remain approved byagar well diffusion technique oppose to gram positive (*S. Aureus*) and gram negative (*E. coli*) bacterial strain.[18]Anti-oxidant activity of the prepared Schiff base and there metal complexes remain estimated by DDPH technique.[19]Importance of ultrasonic irradiation in organic synthesis are characterized specially in case where traditional method appropriate drastic situation.[20-25]

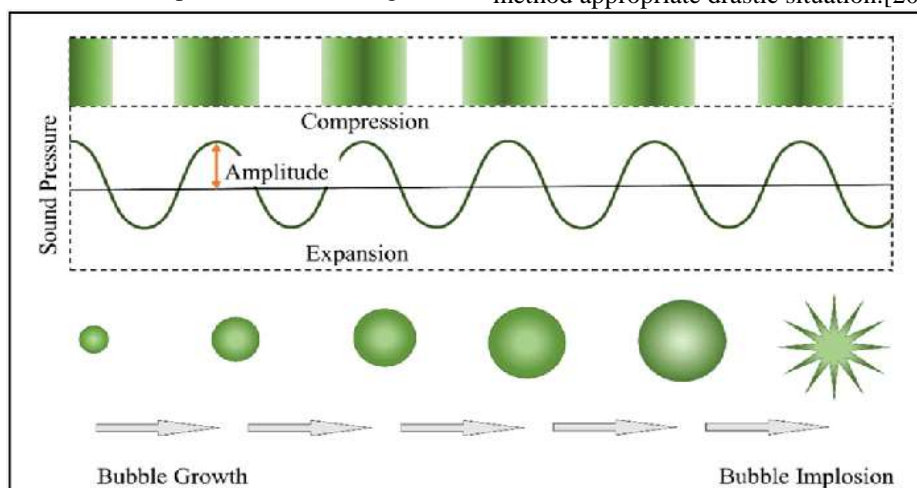


Fig. 1: Ultrasonication Mechanism

II. METHOD AND MATERIALS:

Preparation of 2 salicylidine amino benzoic acid:

A. By Ultrasound Irradiation Method:-

The Schiff based formed from salicylaldehyde and 2 amino benzoic acid was prepared by adding 25 cm³ of salicylaldehyde ethanolic solution (1.22g, 0.01mol) to the same volume of ethanolic solution of 2 amino benzoic acid (1.37g, 0.01 mol). The reaction mixture was stirred in ultrasound for 5-6 hours. The progress of reaction was monitored by TLC and then decided the reaction is complete. And then complete the reaction the reaction mixture was filtered and washed several time with ethanol then collect the orange coloured solid product. Orange coloured product was dried in dissicator. Obtained product was recrystallized from ethanol.

B. By Conventional Method:-

The Schiff based formed from salicylaldehyde and 2 -amino benzoic acid was prepared by adding 25 cm³ of salicylaldehyde ethanolic solution (1.22g, 0.01mol) to the same volume of ethanolic solution of 2 amino benzoic acid (1.37g, 0.01mol). Then the reaction mixture was stirred in ultrasound for 8-9 hours. The progress of reaction was monitored by TLC and then decided the reaction is complete. And then complete the reaction the reaction mixture was filtered and washed several time with ethanol then collect the orange coloured solid product. Orange coloured product was dried in dissicator. Obtained product was recrystallized from ethanol.

C. Preparation of Schiff Base Metal Complex:

1) Preparation of Cr (III) metal complexes:-

a) By Ultrasound Irradiation Method:-

The metal complexes were prepared by adding 25 cm³ of ethanolic solution of metal chromium (2.66gm,0.01mol) with ethanolic solution of the prepared Schiff base (2.41g, 0.01mol) followed by the drop wise addition of aqueous ammonia. The reaction mixture was irradiated for 4 hours under ultrasound. After 4 hours the metal complex formed. The metal complex compounds that precipitated out were filtered and then washed repeatedly with hot ethanol. The product was air dried in disicator. And to gives greyish colored crystal of Cr (III) metal.

b) By Conventional Method:-

The metal complexes were prepared by adding 25 cm³ of ethanolic solution of metal chromium (2.66gm,0.01mol) with ethanolic solution of the prepared Schiff base (2.41g, 0.01mol) followed by the drop wise addition of aqueous ammonia. The reaction mixture was irradiated for 6 hours under ultrasound. After 6 hours the metal complex formed. The metal complex compounds that precipitated out were filtered and then washed repeatedly with hot ethanol. The

product was air dried in dissicator. And to gives grayish colored crystal of Cr (III) metal.

2) Preparation of Mn (II) metal complexes:-

a) By Ultrasound Irradiation Method:-

The metal complexes were prepared by adding 25 cm³ of ethanolic solution of metal manganous chloride (1.97gm,0.01mol) with ethanolic solution of the prepared Schiff base (2.41g, 0.01mol) followed by the drop wise addition of aqueous ammonia. The reaction mixture was irradiated for 4 hours under ultrasound. After 4 hours the metal complex formed. The metal complex compounds that precipitated out were filtered and then washed repeatedly with hot ethanol. The product was air dried in dissicator. And to gives pistachio green colored crystal of Mn (II) metal.

b) By conventional method:-

The metal complexes were prepared by adding 25 cm³ of ethanolic solution of metal manganous chloride (1.97gm,0.01mol) with ethanolic solution of the prepared Schiff base (2.41g, 0.01mol) followed by the drop wise addition of aqueous ammonia. The reaction mixture was irradiated for 6 hours under ultrasound. After 6 hours the metal complex formed. The metal complex compounds that precipitated out were filtered and then washed repeatedly with hot ethanol. The product was air dried in dissicator. And to gives pistachio green colored crystal of Cr (III) metal.

3) Preparation of Ni (II) metal complexes:-

a) By ultrasound irradiation method:-

The metal complexes were prepared by adding 25 cm³ of ethanolic solution of metal nickel chloride (2.37gm,0.01) with ethanolic solution of the prepared Schiff base (2.41g, 0.01mol) followed by the drop wise addition of aqueous ammonia. The reaction mixture was irradiated for 4 hours under ultrasound. After 4 hours the metal complex formed. The metal complex compounds that precipitated out were filtered and then washed repeatedly with hot ethanol. The product was air dried in dissicator. And to gives light blue colored crystal of Ni (II) metal.

b) By conventional method:

The metal complexes were prepared by adding 25 cm³ of ethanolic solution of metal nickel chloride (2.37gm, 0.01mol) with ethanolic solution of the prepared Schiff base (2.41g, 0.01mol) followed by the drop wise addition of aqueous ammonia. The reaction mixture was irradiated for 6 hours under ultrasound. After 6 hours the metal complex formed. The metal complex compounds that precipitated out were filtered and then washed repeatedly with hot ethanol. The product was air dried in dissicator. And to gives light blue colored crystal of Cr (III) metal.

III. RESULT AND DISCUSSION:-

The various product synthesized by using ultrasonicator, where compared with the conventional method are as follows:

Sr. no	Name of compounds	Conventional method		Ultrasound irradiation		Colour	Melting point/ Transition temp
		% yield	Time	% Yield	Time		
1	2 Salicylidine amino benzoic acid	65.76	8-9hrs	89	5-6hrs	Orange	180 ⁰ C
2	Cr(III) complex	70.5	6hrs	85	4hrs	Gray	250 ⁰ C
3	Mn(II) complex	55.15	6hrs	80	4hrs	Pistachio green	280 ⁰ C
4	Ni(II) complex	52.11	6hrs	83	4hrs	Light blue	220 ⁰ C

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

IV. CONCLUSION

Ultrasound has come out as a unique technique in green chemistry. In the present work we have been synthesized the ligand and its metal complexes by conventional and ultrasonic irradiation method to compare the reaction time and yield of the product. The target product were prepared using conventional heating and ultrasonic irradiation. The synthesized compound were screened for their antibacterial activity where very good result were obtained. The complexes are shows moderate to good antibacterial and antifungal activity compared to its ligand. The wide acceptance has been gained at empirical/ practical level. The use of sonochemistry continues to expand in the area of biotechnology, electrochemistry and photochemistry. The synthesized compound were characterized by various analytical technique. In comparison to the order conventional methods the time of reaction was greatly reduced and yields of the product were high as compare to the other method used for similar kind reaction.

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