CHARECTERISATION AND ANTIMICROBIAL STUDY OF SCHIFF BASES - SYNTHESIS VIA ULTRASOUND METHOD BEST ALTERNATIVE TO THERMAL METHOD

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Abstract

In the present research work the Schiff bases have been synthesized via sonochemical and thermal method. The various researchers Synthesized Schiff bases and reported there work, but the Synthesis using ultrasound pathway became the novel as well as green approach for the Synthesis, it was observed that used of ultrasound not only increased the rate of reaction but also increased the yield of reaction product. In the present research work the number of different Schiff bases has been synthesized using ethylene diamine, phenyl hydrazine, orthophenyl diamine, hydrazine with substituted carbonyl compounds. The synthesized Schiff base ligands are bi, tri and tetra dented. It is found that the ultra sound method takes about few minutes rather than few hours by non-ultrasound method .The yield and quality of the product was found more superior over non ultra sound method. The Synthesized compounds were confirmed by physical properties and spectroscopic method like FT-IR; NMR spectra. The synthesized compounds are shown better Biological activity.

Key words - Ultrasound Pathway, schiff bases, characterization, antimicrobial activity, novel method.

INTRODUCTION

Sonochemical energy delivery has been used as an excellent alternative to thermal energy in promoting organic reactions [2]. The used of ultra sound has also been known to improve the rates of reaction and yield [3-4] thereby saving tremendous amount of energy required for synthesis. The product material was synthesized by using the ultrasound pathway (US) and thermal method (NUS) [1]. To save the time and energy is very important in synthesis because Schiff base appear to be important intermediate in a number of enzymatic reaction and also very useful to form metal –complex with transition metal and some those metals who have vacant "d" orbital. Schiff bases can be prepared by condensing carbonyl compounds and amines in different condition with the elimination of water molecules [5-7].The common structural feature of these compounds is the azomethine group with general formula RHC=N-R'[12-15]. Imines and azomethines group are presents in various natural, natural derived or non-natural compound [6-9].

Schiff bases have been playing an important part in the development Co-ordination chemistry [13].Schiff base are widely used for industrial purposes and also exhibits a broad range of biological activities .Schiff bases were also used as pigments, dyes and polymer stabilizers[6-10]. The Schiff base ligands were used to prepare metal complexes [12-14] . The Schiff base ligands metal complexes have

been studied extensively because of their attractive chemical and physical properties and their wide range of applications in numerous scientific areas [19]. These types of complexes have been vigorously explored in recent years and such studies have been the subject of many papers and reviews. Many of them are centered on the catalytic activity of Schiff base complexes in a large number of homogeneous and heterogeneous reactions [16]. It is also shows antimicrobial activity therefore the importance of the synthesis is increased [9]. In the present work to understand the importance of Schiff bases ligand try to used US method which is widely used now days in synthesis [6].

Experimental work: The different chemical reactions were prformed by using the amines which contains one or two amino groups by using different methods-

Material-

In this work All the solvents and different chemical like amines and carbonyl compounds (Substituted aldehyde and ketones) were used analytical grade and used without purifiation.The reaction were monitored by TLC by using 0.25mm E-Merck Silica gel 60 precoated plates,which were visualized with UV light. The solvents like ethanol,methanol,DMSO,DMF were used for recrystallisation and Preparation of sample. The physical constant was taken by melting point apparatus and by open capillary method.

Method -(Ultrasound set-up) -

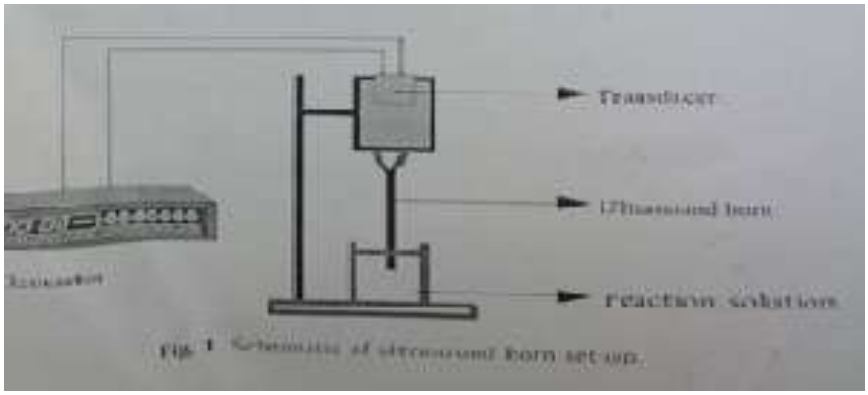
Ultrasound for sonochemical Synthesis is generated with the help of ultrasonic instrument set-up (horn type/probe type) the specification and details used during the experiments[4]-

Make:ACE,USA

Operating Frequency:22LKHz

Rated output Power: 750W

Diameter of stainless steel tip probe:1.3 x 10^{-2} m



Synthesis of Schiff bases by thermal and ultrasound method-

The several reactions were studied by thermal and ultrasound pathways and study their physical properties like color, time, melting point, nature.

Thermal method (NUS)-

In this method the proper ratio were [4-5]takes as per the carbonyl group presents in the compounds and that mixture are taken in the round bottom flask and keep that for reflux and reaction were Monitored by TLC. The amines like orthoamino benzene (1,2diamino benzene),ethylene diamine,hydrazine and phenyl hydrazine were used with substituted and heterocyclic carbonyl compounds like salicylaldehyde,2-hydroxyacetophenone,4-chloro acetophenone and 4-acetyl pyridine. The following reactions were carried out by traditional method as per reported [13-17]. The stirring with reflux method were used in this synthesis. Ethanol was used as a solvent.

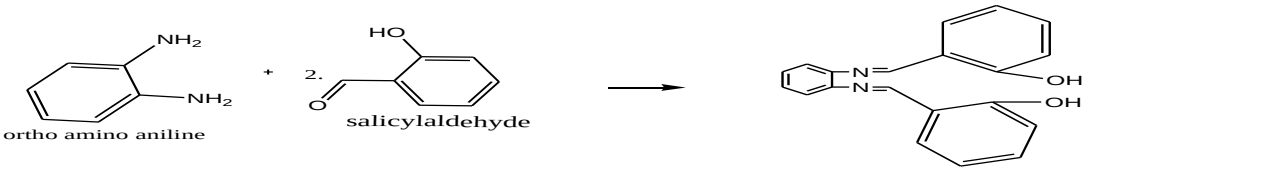
Ultrasound method (US)-

The same reaction mixture which were predicted in following reaction were taken in 50 ml. beaker and placed under the sonicator probe[2-5], the probe inserted in the beaker and time set for ultrasound was 3s per interval. Reaction were Monitored by TLC. The observations are noted in table no.1.

Reaction Scheme –

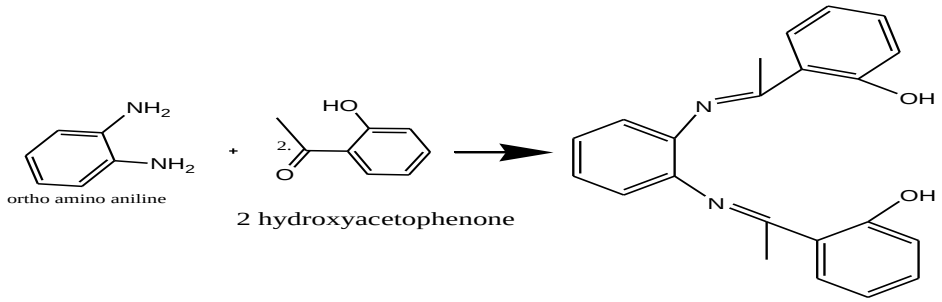
ICD chemdraw ultra software were used for write the reaction and labeled the Schiff bases, labeling of Schiff bases based on structure geometry not confirmed yet[10-16]**,**the 1:2 ratio regarding to the present of amino group in amines and carbonyl compounds[13].both the method same volume were used of reactant.

A



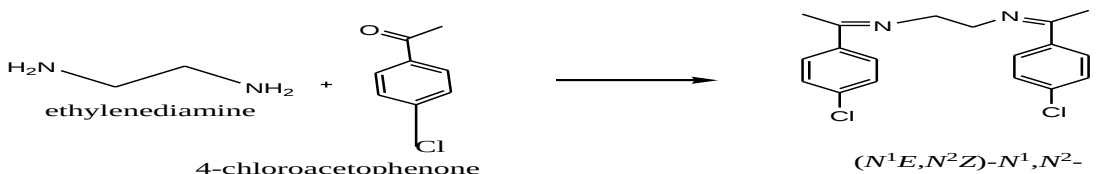
2,2'-(1,2-phenylenebis(azan-1-yl-1-ylidene))bis(methan-1-yl-1-ylidene)diphenol

B.



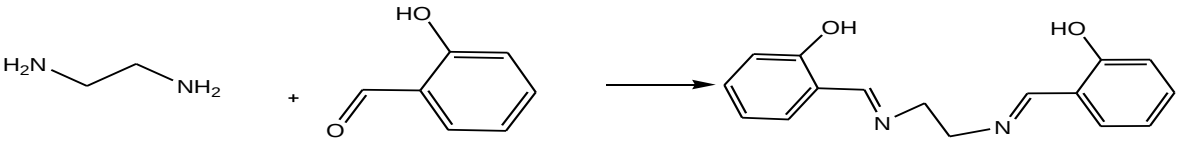
2,2'-(1Z,1'Z)-1,1'-(1,2-phenylenebis(azan-1-yl-1-ylidene))bis(ethan-1-yl-1-ylidene)diphenol

C.



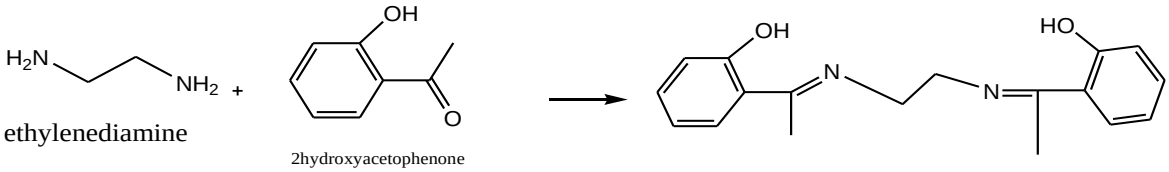
(N^1E,N^2Z)-N^1,N^2-bis(1-(4-chlorophenyl)ethylidene)ethane-1,2-diamine

D.



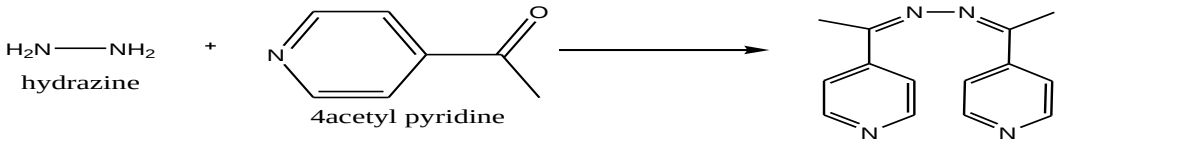
ethylenediamine + salicylaldehyde → 2,2'-(1*E*,1'*E*)-(ethane-1,2-diylbis(azan-1-yl-1-ylidene))bis(methan-1-yl-1-ylidene)diphenol

E.



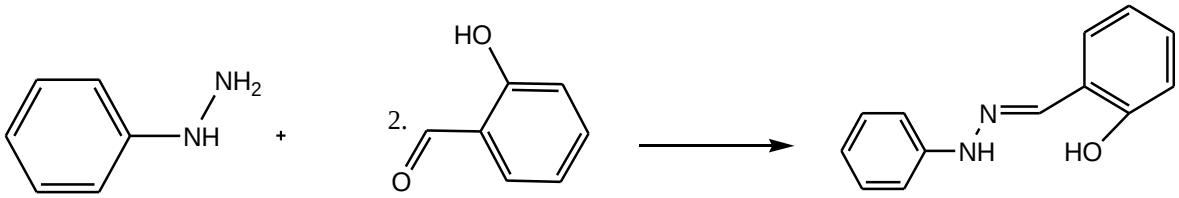
ethylenediamine + 2hydroxyacetophenone → 2,2'-(1*E*,1'*Z*)-1,1'-(ethane-1,2-diylbis(azan-1-yl-1-ylidene))bis(ethan-1-yl-1-ylidene)diphenol

F.



hydrazine + 4acetyl pyridine → (1*Z*,2*Z*)-1,2-bis(1-(pyridin-4-yl)ethylidene)hydrazine

G



phenylhydrazine + salicylaldehyde → (*E*)-2-((2-phenylhydrazono)methyl)phenol

RESULTS AND DISCUSSION

The physical properties of the prepared Schiff bases-

To understand the process and reaction output by ultrasound method it is necessary to work on the above reaction by both the method. In NUS method the quantity of solvents was to much higher than the US method. The time regarding to both the method were too much considerable. In US method time is very less as compared to the NUS method[1-4] The results regarding to physical properties are predicted in table no.1.in the given table no.1 the color ,M.P,and Yield were predicted the time required for the NUS method is from 30 min to 4 hrs. [12,13-18].

Table No.1:

S.B. Reaction.	NUS (reflux)					US				
	Solvent (20 ml)	Color	M.P ^{o}C	Yield %	Time	Solvent	Color	M.P	Yield %	Time ^{o}C
A	Ethanol	yellow	165-167	70	3 hrs	Alco .5.ml	Yellow	165 - 167	85	15 min
B	Ethanol	yellow	174-176	65	2 hrs	Alco	Yello	174	80	10 min

						.5.ml	w	- 176		
C	Ethanol	White(keep overnight)	175-178	75	3 hrs	Alco .5.ml	White (keep overnight)	174	88	30 min
D	Ethanol	yellow	130-132	75	45 min	Alco .5.ml	yellow	129 - 131	90	5 min
E	Ethanol	yellow	190-194	70	40min	Alco .5.ml	yellow	198 - 200	80	10 min
F	Ethanol	Shiny yellow (keep overnight)	110-112	58	4 hrs	Alco .5.ml	Shiny yellow	110 - 112	65	10 min (keep overnight)
G	Ethanol	yellow	120-122	80	35 min	Alco .5.ml	yellow	119 - 122	90	5 min.

Characterization and spectral data analysis:

Table no.2

Compound	Functional group	C=N cm^{-1}	**-OH** cm^{-1}	**-C-N** cm^{-1}	C=C cm^{-1}	C-CH_3 cm^{-1}	-C-Cl cm^{-1}
A	Frequencies	1616	3406	1454	1366	-	-
B		1631	3290	1454	1442	1361	-
C		1681	-	1477	1585	1377	-752
D		1620	3251	1454	1555	-	-
E		1604	3051	1442	1506	1292	-
F		1597	-	1411	1586	1361	-
G		1635	3286	1433	1492	-	-

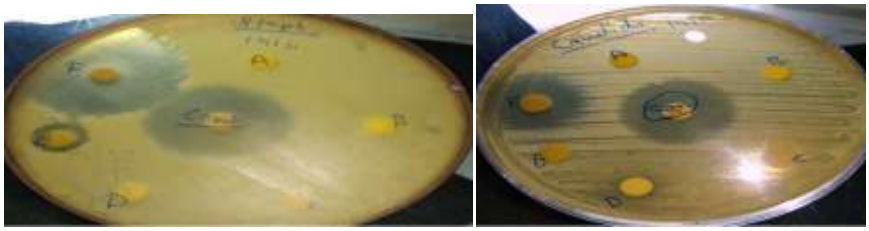
After completion of reaction it is necessary to understand whether the reaction was completed or not therefore the synthesized schiff bases were characterized by FT-IR spectroscopy (Instrumentation lab. Of KTHM College), after getting the satisfactory frequencies in IR spectra we were selected 3 compounds (DMSO used as a solvent) which from the seven reaction product for proton NMR (instrumentation lab.of Savitrybai Phule Pune University) spectroscopy were useful to confirm whether the reaction is completed or not by using the ultrasound method [1-4,27] The characteristics FT-IR frequencies of Synthesized Schiff base predicted in table no.2. H^1NMR analysis of compounds E,F,G and their signals were Predicted in tabel.3.

Table No.3:

Sample code	Ar- singnals	δ chemical Shift	δ chemical Shift	Dissolved water in DMSO 3.35
E	3	-CH2 from EDA 3.94	-CH_3 0.9	
F	2	Pyridine- 8.70	Pyridine -7.86	
G	7	-OH 10.27	N-H-10.53	

Antimicrobial activity of Schiff bases Synthesized by using US method-

Antimicrobial activities of the Schiff Bases were tested by disc diffusion method[9]. For this test the various bacterial culture and fungus culture were used for testing [6]. DMSO had no effect on the microorganism in the concentrations studied [11].2mg/1ml solutionin DMSO of Schiff bases were used for testing. Gentamycine were used as antimicrobial standard. The microorganism were *E.Coli,Pseudomonas aeruginosa,Staphylococcus aureus* and Nystain was used for antifungal for *Candida sp*. The above all compounds A,B,C,D,E,F,G were tested against the E.Coli,Pseudomonas aeruginosa,Staphylococcus aureus and Candila sp.among these the E and F Schiff bases were active against the *Staphylococcus and candeda*[12].



CONCLUSION

Above discussion is clearly indicated that the US method is most economic because it is time saving, less requirement of solvent, better yield and work also less laborious as compared to NUS. The comparative study really important in synthesis of schiff bases because schiff bases have lots of applications in various fields therefore this method (US) is applicable over the NUS it's also providing the green chemistry approached in synthesis of Schiff bases. Antimicrobial activity of some Schiff Bases is active against *Staphylococcus* and *Candila* therefore it's applicable for further study.

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