Synthesis Of Schiff Bases By Green Solvent Method: A Green Chemistry Approach

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Abstract

In transmutation reaction Schiff bases play a roles as an intermediate. In antibacterial and antifungal activities heterocyclic schiff bases play a major role. Schiff bases are synthesized by the condensation between primary amines and aldehyde or ketones by different methods. From literature survey it is observed that many researchers have done their work on Schiff bases.But only conventional method used for this work. But very few researchers were synthesized Schiff bases by green approach. Therefore, some Schiff bases were prepared with help of aromatic primary amines and highly substituted aromatic aldehydes in ethanol and in presence of lemon juice as catalyst in present work.

Keywords: -Schiff base, Aldehyde, Amine , Lemon juice .

1. Introduction

Hugo Schiff reported the condensation of primary amines with carbonyl compounds in 1864.[1] Commonly these compounds is the azomethine group with a general formula RHC=N-R1, where R and R1 are alkyl, aryl, cyclo alkyl or heterocyclic groups also known as anils, imines or azomethines. Aromatic amines and aromatic aldedydes are a very important class of organic compounds from which Schiff bases derived. These are applicable in many fields,such as biological[2-10], inorganic[11-18] and analytical chemistry[16-20]. In the progress of science, the chemistry of the carbon nitrogen double bond plays a important role [21]. In the synthetic laboratories green chemistry approach and microwave assisted synthesis is now a day widely practiced.

Microwave reactions gives high yields together with simplicity in processing and handling under solvent-free conditions was studied due to some factor such as low cost, pollution free reaction and shorter reaction time[24-28] · Acceptance and popularity among the synthetic, chemist community assures safe and reproducible experimental procedures and microwave synthesis has gained.

In the present work, some Schiff's bases were synthesized by solvent free technique using microwaves. They were purified and characterized by means of spectral data and elemental analysis. In 1990, at the EPS, UMB's Paul Anastas and John Warner defined Green Chemistry: "The design of chemical products and processes that reduce or eliminate the use and generation of hazardous substances". There are some environmental issues and problems

which constantly facing by society such as soil and water pollution, air pollution, global climate change, ozone depletion, acid rain, the depletion of natural resources. Members of the California green chemistry science advisory panel, Paul T. Anastasand and John C. Warner published their important principles of green chemistry in their book in1998[30]. In place of treating or cleaning waste, prevent it. Incorporate all materials used in the manufacturing process in the final product. Use methods that generate substances with little or no toxicity to people or the environment. Design chemical products with lesst toxicity but effective.when possible Phase-out solvents and auxiliary substances. Use temperature and pressure, to reduce costs and environmental impacts. Renewable raw materials for feedstock and to reduce or eliminate waste.Use blocking agents and chemical intermediates.Use chemicals with low risk for accidents, explosions, and firesand readily break down into innocuous substances in the environment Select catalysts that carry out a single reaction many times

2. Experimental Techniques

1. General procedure for extraction of Lemon juice: Fresh lemon cut by using clean dry knife and then pieces were pressed manually using domestic presser to extract juice. Then juice was filtered with clean cotton cloth and then through filter paper to remove solid material and to get clear juice. Then it was used as catalyst.
2. Melting Point: The melting points were determined in a theilstube apparatus and are notcorrected.
3. I.R Spectrum:- I.R.Spectra was recorded on Shimadzu FTIR (ModelISI118675A) using KBr pallets.
4. Thin Layer Chromatography:Thin layer chromatography is detected on chromatographic paper. The TLC was performed using mobile phase, petroleum ether: ethyl acetate (4:1) and detected in U.V.Chamber.

3. Materials and Methods

Experimental

All required chemicals were used from Merck, Fluka and Acros chemical companies.
Shimadzu FT-IR 8000 spectrophotometer used for IR spectra. 1H NMR and 13C NMR spectra were recorded in CDCl3 using a Bruker Avance DPX instrument (1H NMR 250 MHz, 13C NMR 62.9 MHz). Chemical shifts were reported in ppm (N) downfield from TMS. All of the coupling constants (*J*) are in Hertz. Shimadzu GC-MS QP 1000 EX instrument were recorded mass spectra. Melting points were determined in open capillaries with Buchi 510 melting point apparatus and are not corrected. Thin-layer chromatography was carried out on silica gel 254 analytical sheets obtained from Fluka.

4. Method Of Synthesis of Schiff Bases Derivatives

General procedure for the synthesis of Schiff bases: for this synthesis we use finely powdered of Solid starting materials. A mixture of aldehyde (0.5 mole) and amino benzoic acid (0.5 mole) was grinded in a small amount of lemon juice (2ml) at room temperature for the mentioned time. After completion of the reaction as indicated by TLC. All solvent-free

reactions were performed by grinding the mixture. Then pour the mixture in crushed ice stirr it for 5 min. The crystalline powder formed was collected by filtration, washed with cold water and dried in an infrared light. Then recrystalize from ethanol.

1. 4-[(4-methoxybenzylidene) amino] benzoic acid: - L1

2. 4- {[(*Z*) (3hydroxyphenyl)methylidene]amino}benzoic acid :- L2

3. 4 - (Methylideneamino) benzoic acid: - L3

SYNTHESIS OF SCHIFF BASE

1. 4-[(4-methoxybenzylidene) amino] benzoic acid: - L1

4- Amino benzoic acid {0.5 mole} first taken in a mortal pestal and grind for a while then add Anisaldehyde {0.5 mole} then add lemon juice and grind for a while then add in to crushed ice. Wash with cold water then filter it and dry it recrystalize from ethanol.

IR :(ν max) cm-1: - (Metasusti Ph ring bend)= 700.16 cm^{-1} ,(Ar C-H str) = 2941.4 cm^{-1}

(C=C str) = 1421.5 cm^{-1}, (C= N str) = 1591.2 cm^{-1} ,(C=O str) = 1674.2 cm^{-1}

(C-O str) = 1161.1 cm^{-1}

2. 4- { [(*Z*)(3hydroxyphenyl)methylidene]amino}benzoic acid :- L2

IR:(ν max) cm-1: - Ar (C-H str)= 2883.3 cm^{-1},Ar (C=O Str) = 1681.9 cm^{-1}

(C-O str) = 1278.8cm^{-1}, (O-H Str) = 3371.5 cm^{-1}, (C=N Str) = 1566.2 cm^{-1}

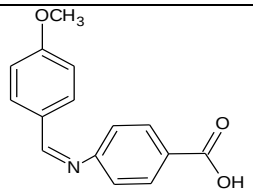
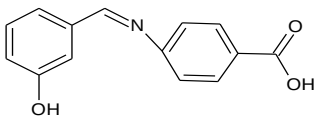
(C=C str) = 1516.2 cm^{-1}

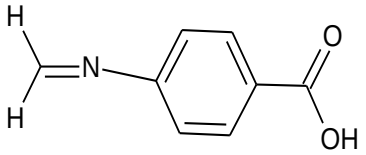
3. 4 - (Methylideneamino) benzoic acid:- L3

IR:(ν max) cm-1: - Ar (C-H str)= 2970.3 cm^{-1}, Ar (C=O Str) = 1683.8 cm^{-1}

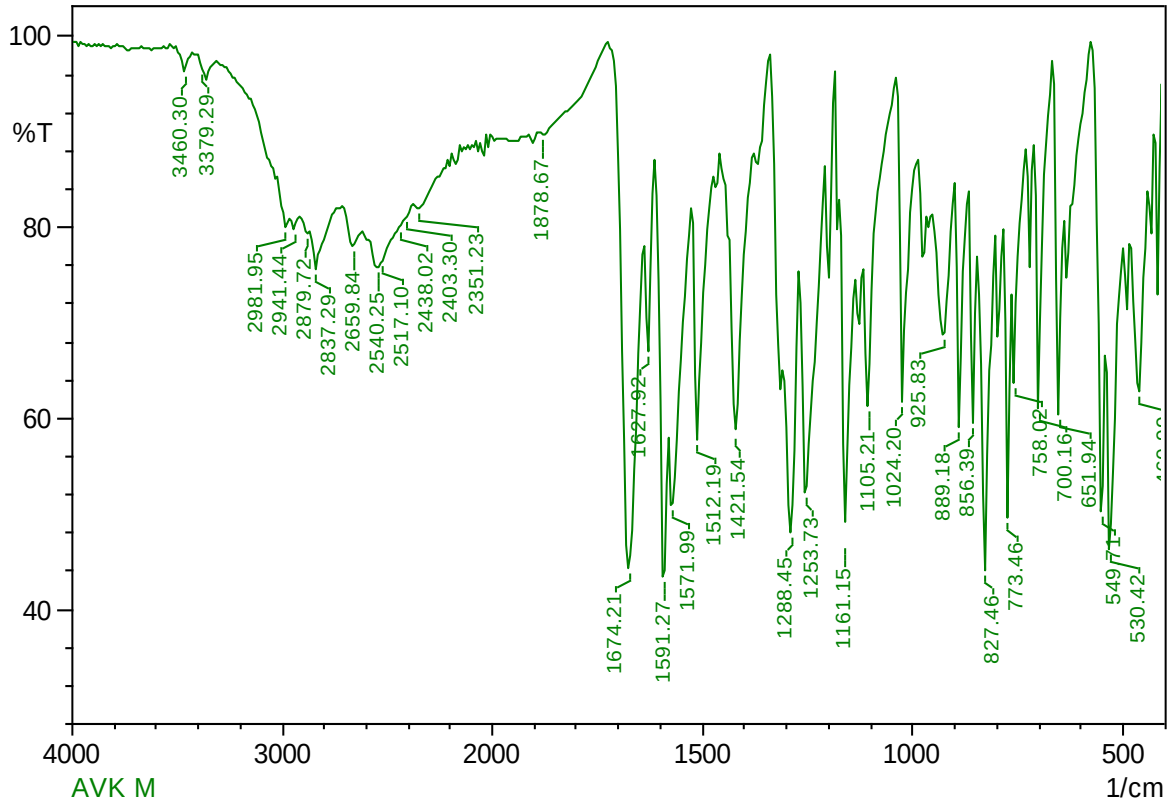
(C-O str) = 1282.6 cm^{-1}, (C=N Str) = 1571.9 cm^{-1}

Chart 1:- New Synthesized Schiff Base

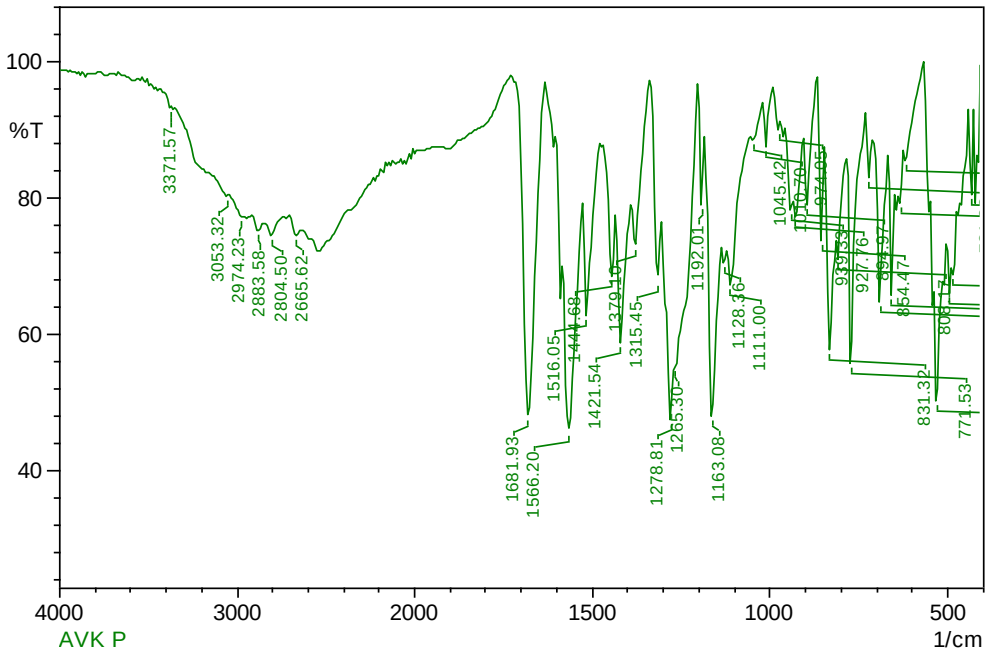
Sr no	Compound	Colour	Reaction Time	% yield	Melting Point
1	 4-{[(*Z*)-(4-methoxyphenyl)methylidene]amino}benzoic acid	Cremish	3 hours 45 minutes	85.93%	203 ºc
2	 4-{[(*Z*)-(3-hydroxyphenyl)methylidene]amino}benzoic acid	Reddish yellow	3 hour's	98.43%	198 ºc

3	 4-(methylideneamino)benzoic acid	*Cremish*	*2 hour's 35 minutes*	*95.08%*	17 1°c

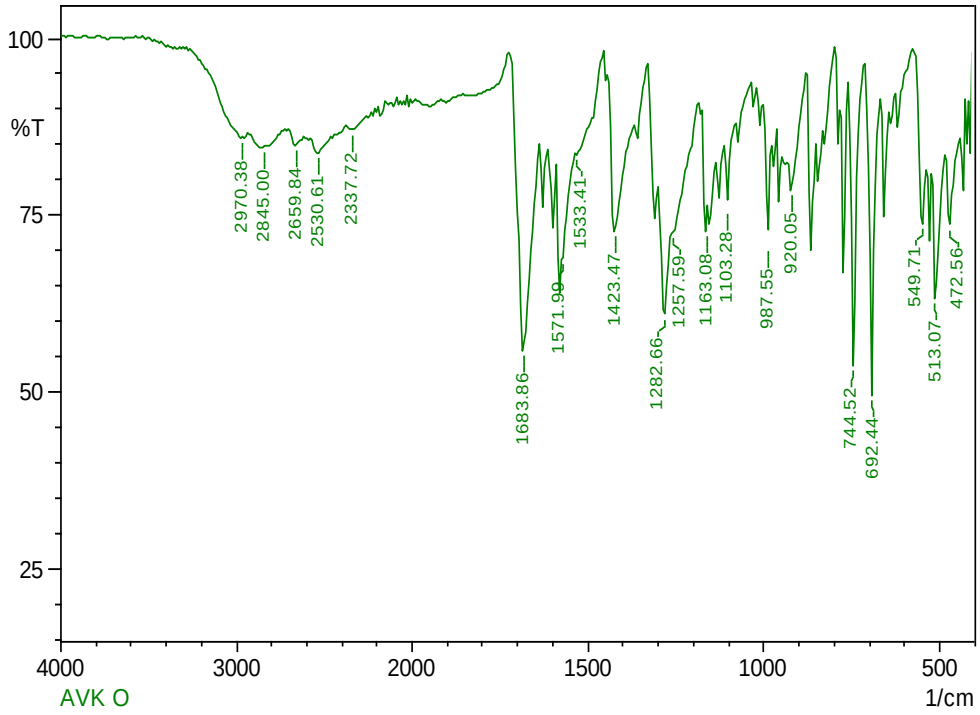
5. Results and Discussion



1. 4-[(4-methoxybenzylidene)amino] benzoic acid :- L1



2. 4- { [(*Z*)(3hydroxyphenyl)methylidene]amino}benzoic acid :- L2



3. 4 - (Methylideneamino) benzoic acid:- L3

The newly synthesized shiff base stable at room temperature. The schiff bases are soluble in common organic solvents, such as ethanol, methanol, and chloroform but partially soluble in hexane. The schiff base compounds were relatively well soluble in DMF and DMSO. The

synthesized compounds were characterized by elemental analysis, spectra data. The biological properties of the compounds are in progress.

6. Conclusion

We have developed a simple, efficient and more eco-friendly method for synthesis of substituted Schiff bases by using greener technique. The main advantages of this procedure are simple and convenient, and time consuming. In addition to this, comprised to traditional method , this new method is cleaner, safer and more eco-friendly involving mild reaction condition such as reaction time use of hazardous solvents con be reduce by maintaining good yet of product. The yields were excellent and reactions were fast.

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