Research Journal of Chemical Sciences ISSN 2231-606X
Vol. 3(10), 97-99, October (2013) *Res. J. Chem. Sci.*

Short Communication

Rapid and Economic Synthesis of Schiff Base of Salicylaldehyde by Microwave Irradiation

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Available online at: www.isca.in, www.isca.me
Received 4th September 2013, revised 20th September 2013, accepted 15th October 2013

Abstract

Schiff base synthesis is usually acid-catalyzed and usually require refluxing the mixture of aldehydes (or ketone) and amine in organic medium. However, assistance of microwave irradiation for sysnthesis of schiff base is introduced now a day. In the present study microwave promoted condensation reaction of salicylaldehyde and p-amino phenol are displayed. The method was also compared with conventional method for determination of production efficiency and production economic. Characterization of these Schiff base were done by TLC and IR spectra. From the study, it was concluded that the microwave irradiation method is very rapid, reliable and economic method for production of schiff base.

Keywords: Schiff base, salicylaldehyde**,** microwave irradiation, metal complexes.

Introduction

A Schiff base, named after Hugo Schiff, is a compound with a functional group which contains a carbon-nitrogen double bond with the nitrogen atom connected to an aryl or alkyl group, not hydrogen and in general it can be represent as R1R2C=NR3, where R is an organic side chain. Some restrict the term to the secondary aldimines having general formula RCH=NR' (azomethines where the carbon is connected to a hydrogen atom)[1]. The chemistry of the carbon-nitrogen double bond plays a significant role in the progresses of chemical science. Schiff-base compounds are widely applicable as fine chemicals and in medical substrates. Recently in a study, multi-dentate complexes of iron and nickel showed high activities of ethylene oligomerization and polymerization[2-5]. In our efforts for ligands of polymerization catalysts, synthesis of Schiff-base through classical condensation and microwave assisted of aldehydes (or ketones) and amines were pursued, and the yields of products were found high. Driven by industrial application of polymerization catalysts with Schiff-base ligands, the aim of this research project is to screen simple and economic methods for preparation of Schiff-bases.

In classical organic synthesis of Schiff bases, it commonly meets the problem of removing solvents from the reaction mixture or liquid extraction especially in the case of aprotic dipolar solvent with high boiling point, or product isolation through liquid-liquid extraction. Microwave-assisted reactions have been intensively investigated as mentioned in previous study[6-8]. Microwave-assisted technique has been popularly used in organic synthesis. The organic synthesis mediated by microwave irradiation performs several advantages such as higher atom economy, environmental friendship, reducing the hazard, etc[6-9].

Reaction Mechanism: Schiff bases are synthesized from an aromatic amine and a carbonyl compound by nucleophilic addition forming a hemiaminal, followed by a dehydration to generate imines. The mechanism is explained as shown in the figure.

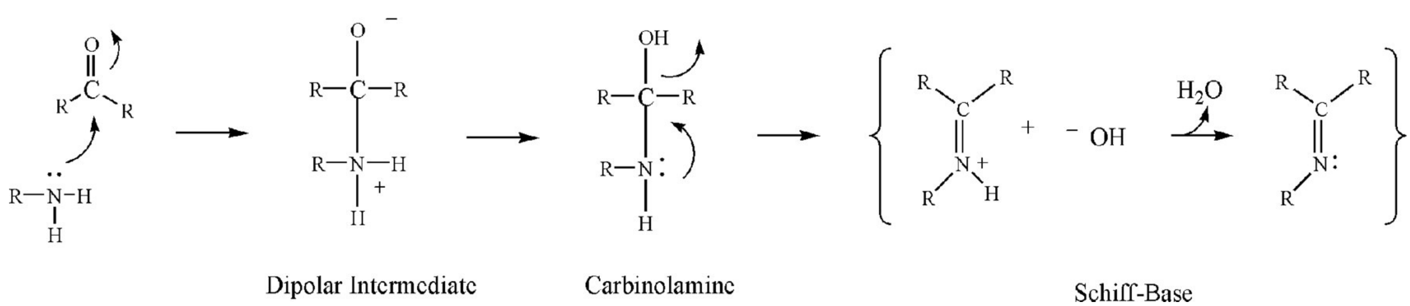

Figure-1
General reaction mechanism of schiff base

Schiff bases derived from aromatic amines and aromatic aldehydes have a wide variety of applications in varieties of fields, e.g. biological, inorganic and analytical chemistry. Applications of many new analytical devices require the presence of organic reagents as essential compounds of the measuring system.

The aim of this study was to make of Schiff bases using salicylaldehyde using different methods and comparison of the production methods

Material and Methods

Conventional Method: Take 0.01 M (1.09 g) p-amino phenol in a RBF. Add 8 ml of methanol as a solvent in flask. Dissolve p-amino phenol completely in solvent. Add porcelain pieces to avoid bumping of solvents. Take 0.01 M (1.047 ml) salicylaldehyde into the RBF. Reflux it for 4 hours under observation such that the solvent does not evaporate completely. Allow it to cool down. After completion of reaction cool the product on crushed ice. Orange yellow colored crystals will appear. Add 12 ml methanol and dissolve the product and re-crystallize the product

Microwave Irradiation Method: Take 0.0is1 M (1.09 g) p-amino phenol in a glass beaker. Add 12 ml of methanol as a solvent in beaker. Dissolve p-amino phenol completely in solvent. Now add 0.01 M (1.047 ml) salicylaldehyde to it. Keep it under microwave irradiation for 3 ½ minutes at medium low temperature. After completing the reaction immediately put it in cold water bath having temperature less than 15^{o}C. Within short time orange yellow colored crystals will appear. Add 10 ml methanol and dissolved the product in it and re-crystallize the product.

TLC Analysis: Commercially available silica plate by Merck was used as stationary phase, while toluene:methanol in the ratio of 7:3 was used as mobile phase. Plates were developed by charring of carbon using strong acids.

IR analysis: IR spectra of the products were taken on FTIR affinity I (Shimadzu, Japan) from 4000 to 400 nm. DRS 8000A assembly was used for direct determination of IR spectra.

Results and Discussion

Net production: With the conventional method 78.0% yield was obtained while in case of microwave assisted reaction 94.3% yield was obtained in significantly less period of time. This indicates microwave irradiation increases the production by 20% than the conventional method. In most of the precious study similar observation was made, where yield of microwave assisted method has increased upto 10.0 to 30.0% depends of the method.[6–8] Similarly they have also noted that production takes very less time as compare to conventional methods. Not only have these, microwave methods avoided reflux of mixture and hence saving the energy input to make the production viable[6–9].

TLC Analysis: TLC analysis shows all the p-amino phenol were utilized completely for the production of schiff base resulted in to no band of p-amino phenol in the lane B and C as present in lane A. In the TLC respective Rf values of 0.45 and 0.62 for p-amino phenol and salicylaldehyde schiff base were obtained.

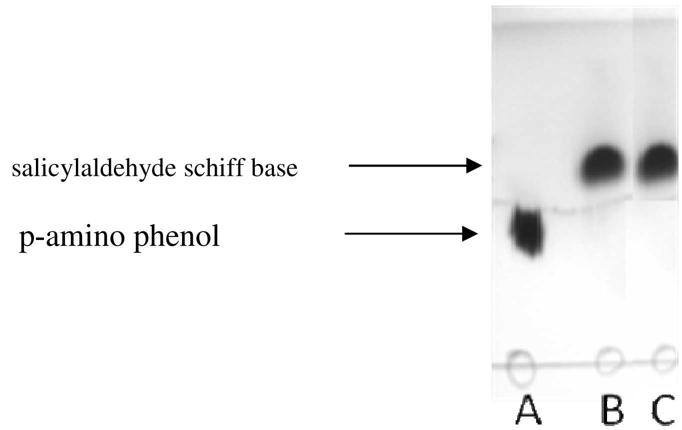

Figure-2
TLC of schiff base, A: p-amino phenol, B: product of conventional method and C: Product of microwave method

IR Analysis: IR spectra of the schiff base showed the absence of bands at ~1735 and 3315 cm^{-1} due to the carbonyl (C = O) and (NH2) stretching vibrations and a strong new band appeared at ~163^{0} cm^{-1} assigned to azomethine (HC=N) linkage, showing that amino and aldehydes moieties of the starting material are absent and have been converted into the ligand, i.e. p-aminophenol n-salicylidene. A broad band appearing at 3415 cm^{-1} assigned to the (OH) in the ligand was no longer found in the spectra of the metal complexes but instead a new band appeared at ~1380 cm^{-1}, indicating deprotonation and coordination of hydroxyl oxygen with the metal ion.

Conclusion

From the experiment it was concluded that both the methods could be used for production of schiff bases but microwave irradiation has several advantages over conventional one. It gives higher production in significantly lesser period of time and less utilization of energy input resulted into more economic production.

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