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Original Article

Novel one pot tandem reductive Schiff base synthesis and characterization

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ABSTRACT

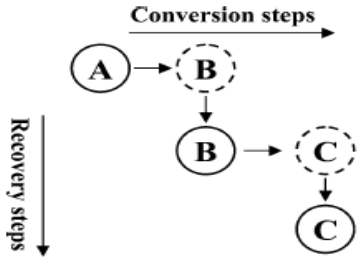
In the present study an intermolecular reductive Schiff base formation from 3-nitro benzoic acid 4- dimethyl amino benzaldehyde carried out in the presence of iron powder and ammonium chloride. Schiff base was synthesized namely 3-(4-(dimethyl amino) benzylidene amino) benzoic acid and identified by microanalysis (CHN), infrared, 1H-NMR, 13C-NMR and GC-Mass spectroscopy. Advantage of this efficient method is excellent yield of products in crystalline form, short reaction time, simplicity of work up procedure and no use of any type of hazardous solvents.

Key words: Cascade/Tandem reaction, intermolecular reductive Schiff base, green chemistry.

INTRODUCTION

The development of cascade conversions /reactions without intermediate recovery steps is considered as one of the important future directions for carrying out sustainable organic syntheses with inherently safer designs. It will drastically reduce operating time and costs as well as consumption of auxiliary chemicals and use of energy.

However, organic synthesis, i.e., chemistry by mankind, still uses often a simple step-by-step approach to convert a starting material **A** into a final product **C**, in which intermediate products B is isolated and purified for next conversion step.



One of the primary motivating goals of this research is the development of cleaner, more efficient transformations to shorten syntheses and save money on chemicals. The strategy of using reactions in tandem (multistep one-pot reactions) is also aimed at shortening syntheses.

Tandem reactions have several advantages over a series of individual reactions. First, they allow construction of complex structures in as few steps as possible. In theory, they also eliminate the need for a purification step (or steps). Since the intermediates are not isolated it becomes easier to work with sensitive or unstable intermediates.

The chemistry of biological science has produced a number of compounds that are now employed as antibacterial agents. Such type of compounds revealed great promise in this area is the Schiff bases.

Schiff bases are important intermediates for the synthesis of various bioactive compounds. Furthermore, they are reported to show a variety of biological activities including antibacterial, antifungal, anti-cancer and herbicidal activities. The chemistry of the carbon-nitrogen double bond plays a vital role in the progress of chemical science. Schiff-base compounds are used as fine chemicals and medical substrates [1-13].

Traditional formation of Schiff bases from nitroarene starting materials requires a two-step process in which the nitroarene is first reduced to the aniline, then isolated, and subsequently condensed with the desired carbonyl [14, 15]. Over the past few decades, significant research has been directed towards the development of new technologies for environmentally benign processes

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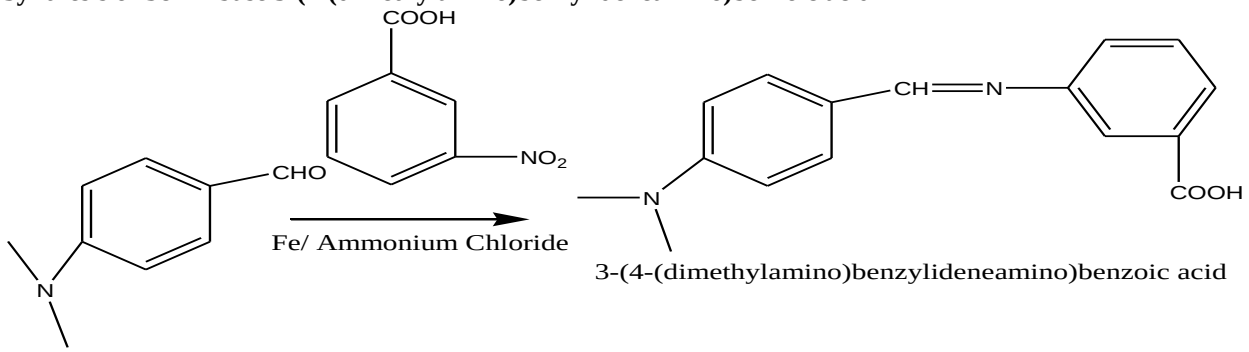
(green chemistry) [16]. In conclusion, we describe a green and efficient method for the synthesis of Schiff bases with easy workup; facile conditions, fast reaction rates, good yields, and selectivity of the reaction make the present methodology attractive.

MATERIALS AND METHODS

Melting points were determined in open capillary tubes and are uncorrected. IR spectra were recorded in KBr on a Perkin Elmer Spectrum RX-1 FTIR spectrophotometer. 1H-NMR spectra were measured on Jeol JNM-ECX400P at 400 MHz. All chemicals used were of analytical grade.

Ammonium chloride (4.5 mmol) was added to a mixture of 3- nitro benzoic acid (0.72 mmol), 4-dimethyl amino benzaldehyde (0.72 mmol), and iron powder (7.32 mmol) in 26 mL of MeOH–H2O (2:1 v/v) solution. The reaction was heated to 65°C for 8 h before being filtered while hot. The filtrate was extracted using chloroform (100 ml) after which the organic layers were combined, dried over MgSO4, filtered, and concentrated in vacuo to yield light orange crystalline powder. The product was collected by filtration, washed several times with cold ethanol and recrystallized from hot ethanol. Orange crystals (75%), Melting Point: 190-192 ^{0}C.

Synthesis of Schiff base 3-(4-(dimethylamino)benzylideneamino)benzoic acid



3-(4-(dimethylamino)benzylideneamino)benzoic acid

RESULT AND DISCUSSION

Table I Microanalysis data for prepared Schiff base

compound	Mol weight	carbon	hydrogen	Nitrogen
		Calculated /found	Calculated /found	Calculated /found
3-(4-(dimethyl amino) Benzylidene amino) benzoic acid	268	71.71 /71.63	5.98 /5.93	10.48/10.40

Figure 1: **IR spectra (cm-1) for prepared Schiff base 3-(4-(dimethyl amino) Benzylidene amino) benzoic acid**

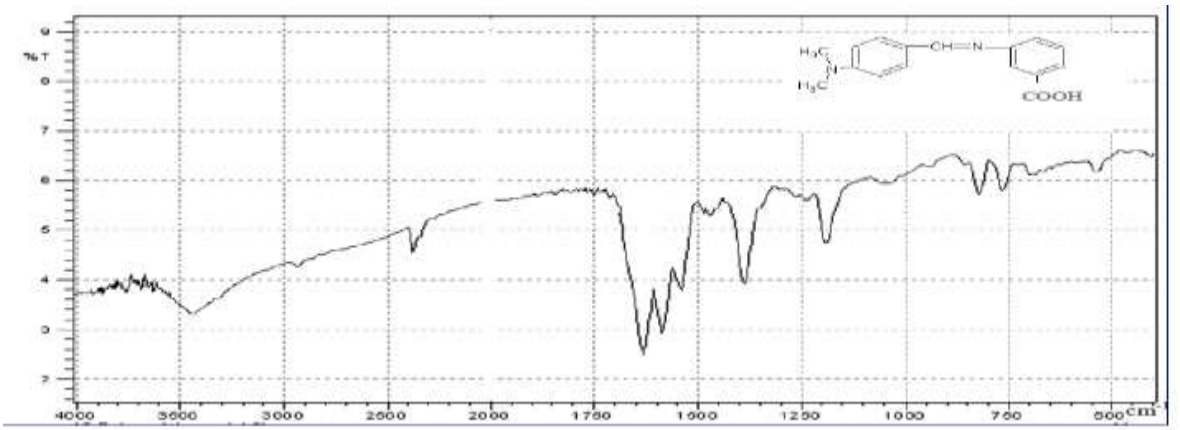


Table 2

Schiff base	v(C-OH)	VCh ar	VCh ali	Δv(C=O)	v(C=N)	v(C-C)	v(C-N)	v(C-O)
3-(4-(dimethyl amino) benzylidene amino) benzoic acid	3422 br	3081 w	2942 w	1663 m	1592 m	1541 m	1384 m	1180 m

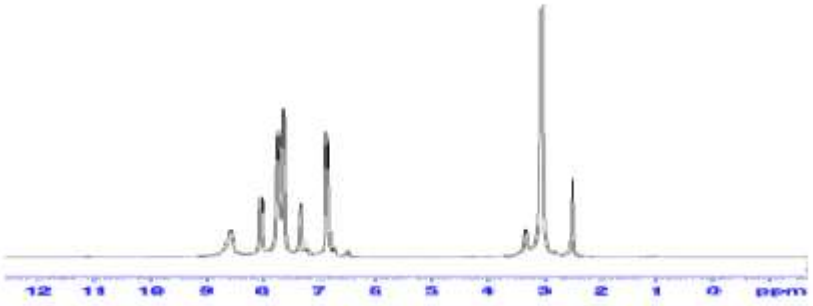


Figure 3 Chemical shifts of 1H NMR for prepared Schiff bases in d DMSO
3.12 (S , 6H , CH3) , 6.57-8.04 (m,8H, Ar-H) 8.58 (S , 1H , HC=N) 15.62 (S , 1H , OH)

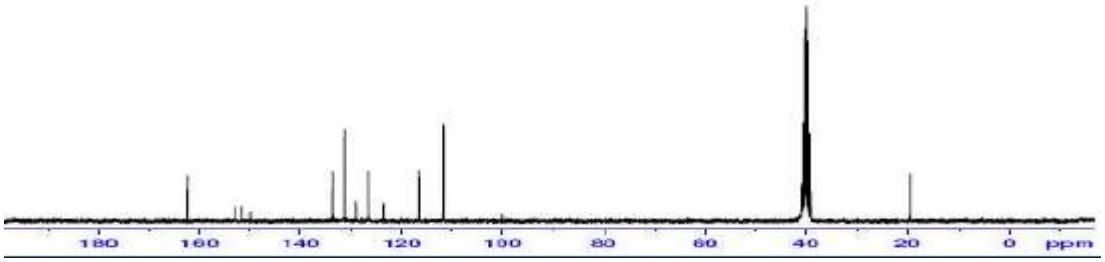


Figure 4: **13C NMR for prepared Schiff base in d DMSO**
Chemical shift (δ ppm) 40 (2C , CH3) ,111.88 – 151.54 (12C , Ar-C) 162.26 (1C , HC=N)

Important fragments appeared in mass spectra for prepared Schiff base

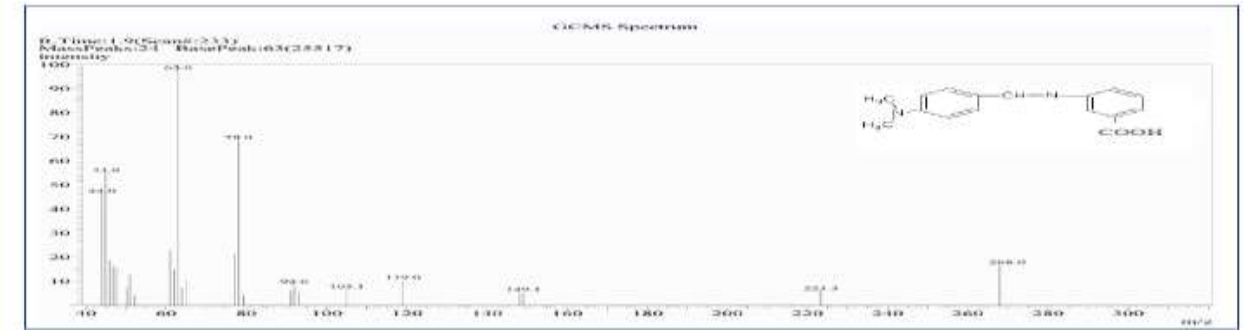


Figure 5: mass spectra

compound	**m/z**	**intensity**	**fragment**
3-(4-(dimethyl amino) Benzylidene amino) benzoic acid	268, 223.3,149 78,63	18,7,5,69,100	[M]° + [C 14H10NO2]° + [C 8H6NO2]° + [C6H6]° + [C5H3]° +

DISCUSSION

Green chemistry for chemical synthesis addresses our future challenges in working with chemical processes and products by inventing novel reactions that can maximize the desired products and minimize by-products, designing new synthetic schemes that can simplify operations in chemical productions, and seeking greener solvents that are inherently environmentally and ecologically benign. This simple technique affords various Schiff base derivatives with short reaction times, excellent yields and without formation of undesirable side products, operation simplicity, cleaner reaction and easy work-up. All spectroscopic analysis confirmed the proposed structures for these compounds.

CONCLUSION

Our present work brings forth a novel method for the synthesis of Schiff bases which offers significant improvements over existing conventional procedures. The synthesized compounds therefore, present a new scaffold that can be used to yield potent antimicrobial compounds. It can be concluded that these compounds certainly holds great promise towards good active leads in medicinal chemistry.

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