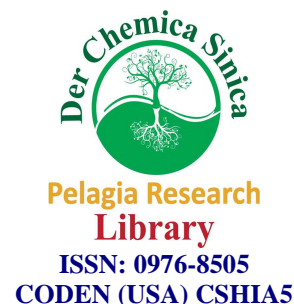




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Synthesis and characterization of Schiff Base ligands derived from 2-amino pyridine and 3-aminophenol by using green approach

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ABSTRACT

Schiff bases (**1a-1f**) were synthesized by the environmentally eco friendly green approach. The synthesis was carried out at room temperature in ethanol solvent. The reaction takes place efficiently with excellent yield and high purity of Schiff bases without any harsh and hazardous condition. The synthesized Schiff bases were confirmed on the basis of Elemental analysis, ¹H NMR and IR spectroscopic analysis.

Keywords: Green solvent, 2-amino pyridine, Schiff base, 2-aminophenol, Vanillin, 3-methoxy benzaldehyde, furfuraldehyde.

INTRODUCTION

Schiff bases are well-known to have biological activates such as antibacterial [1-3], antifungal [4-5], antitumor [6-7], antiviral [8-10], anti bacterial [7], antifungal [7], anti-HIV-1[11], herbicidal [12] and anti influenza A virus [13] activities. Perhaps the most common method for preparing Schiff bases is the reaction of aldehydes and ketones with primary amines [14]. The reaction is generally carried out by refluxing the carbonyl compounds and amines in organic solvents. Recent years have witnessed a major drive to increase the efficiency of organic transformations while lowering the amount of waste materials Furthermore; The green methods in the organic synthesis which have numerous advantages: reduced pollution, low costs, and simplicity in process and handling [15-16]. Many organic solvents are hazardous, volatile, flammable and delicious to human health. In this paper we have modified the synthesis method of Schiff bases by using Green solvent. Reaction was carried out at room temperature.

MATERIALS AND METHODS

All the chemicals used were analytical reagent grade and purchased from Spectrochem or Loba. The solvents used were of high purity and distilled in laboratory before use. Thin layer chromatography was carried out on silica gel 60/UV254. Melting points of products were recorded in open capillaries on digital melting point apparatus (optics technology) and were uncorrected.

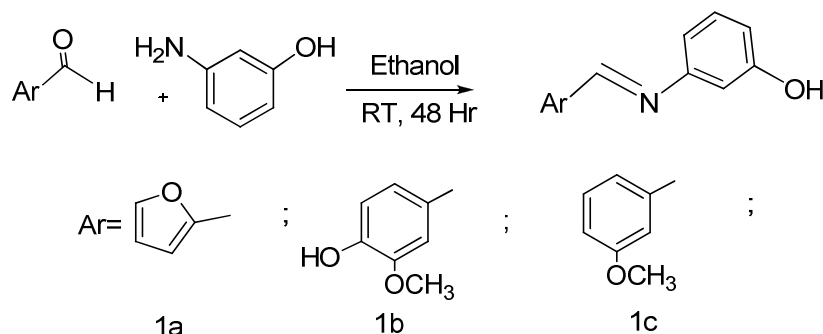
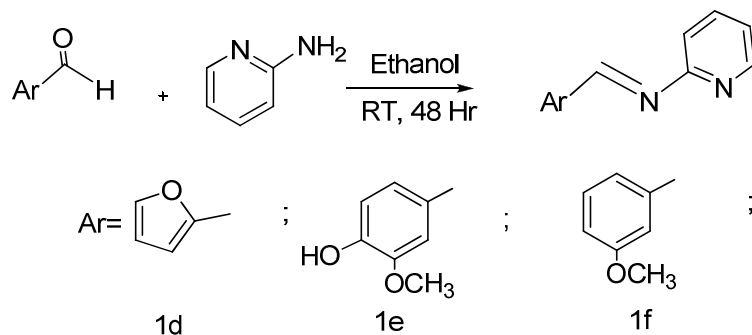
IR spectra were recorded on Perkin-Elmer FTIR Spectrophotometer in range 4000-500 cm⁻¹ using ATR Instrument. Samples were kept directly without KBr pallets. ¹H NMR spectra were obtained on a Perkin-Elmer 300 MHz spectrophotometer using TMS as internal standard in DMSO-d₆ as the solvent.

General Procedure for Schiff Base Synthesis:

Schiff base (**1a -1f**) of aldehyde was synthesized by adding amine (0.01 mole of each) in 20 ml of ethanol. The reaction mixture was heated under reflux for three hours. The reaction mixture was allowed to cool, filter, recrystallized from ethanol and then dried under vacuum to give crystal yield of product.

Modified Green Procedure for Schiff Base Synthesis:

Schiff bases (1a-1f) were synthesized by reaction between aldehyde and amine (0.01 mole of each) in 20 ml of ethanol. Kept at room temperature in round bottom flask for 48 hours. The completion of reaction monitored by TLC then reaction mixture was poured on ice with constant stirring yellow or brown coloured product precipitated then that was filter, recrystallized from ethanol and then dried under vacuum. The yield of product and M.P. was recorded.

Scheme: 1**Scheme:2****RESULTS AND DISCUSSION****3.1 Spectral, analytical and physical data:****1a) 3-(furan-2-ylmethyleneamino)phenol :**

Anal.Cal..For $\text{C}_{11}\text{H}_9\text{NO}_2$ Molecular Weight: 187.19 Elemental Analysis: C, 70.58; H, 4.85; N, 7.48; O, 17.09 IR: 3350, 2940, 1650, 1550, 1350, 1240, 740 cm^{-1} NMR δ ppm: (300 MHz, DMSO- d_6 , 297K) δ =6.52 to 7.57 (m, 7H), 5.35 (Ar-OH, s, 1H), 8.36 (N=C-H, s, 1H).
M.P. =280 $^{\circ}$ C Colour = Brown Yield=65%

1b) 4-((3-hydroxyphenylimino) methyl)-2-methoxyphenol:

Anal.Cal..For $\text{C}_{14}\text{H}_{13}\text{NO}_3$ Molecular Weight: 243.26 Elemental Analysis: C, 69.12; H, 5.39; N, 5.76; O, 19.73 IR: 3370, 2930, 2840, 1640, 1560, 1340, 1240, 740 cm^{-1} NMR δ ppm: (300 MHz, DMSO- d_6 , 297K) δ =6.54 to 7.52 (m, 7H), 5.32 (Ar-OH, s, 2H), 8.66 (N=C-H, s, 1H), 3.83 (CH_3 , s, 3H).
M.P. =136 $^{\circ}$ C Colour = Yellow Yield=70%

1c) 3-(3-methoxybenzylideneamino) phenol:

Anal.Cal..For $\text{C}_{14}\text{H}_{13}\text{NO}_2$ Molecular Weight: 227.26 ;Elemental Analysis: C, 73.99; H, 5.77; N, 6.16; O, 14.08 IR: 3350, 2945, 2830, 1650, 1550, 1350, 1250, 741 cm^{-1} NMR δ ppm: (300 MHz, DMSO- d_6 , 297K) δ =6.54 to 7.50 (m, 7H), 5.35 (Ar-OH, s, 1H), 8.68 (N=C-H, s, 1H), 3.84 (CH_3 , s, 3H).
M.P. =160 $^{\circ}$ C Colour = Brown Yield=72%

1d) N-(furan-2-ylmethylene) pyridin-2-amine:

Anal.Cal..For $\text{C}_{10}\text{H}_8\text{N}_2\text{O}$ Molecular Weight: 172.18;Elemental Analysis: C, 69.76; H, 4.68; N, 16.27; O, 9.29 IR: 2950, 1670, 1600, 1550, 1340, 1280, 743 cm^{-1} NMR δ ppm: (300 MHz, DMSO- d_6 , 297K) δ =6.52 to 7.66 (m, 7H), 8.36 (N=C-H, s, 1H).
M.P. =180 $^{\circ}$ C Colour = Brown Yield=74%

1e) 2-methoxy-4-((pyridin-2-ylimino) methyl) phenol:

Anal.Cal..For $C_{13}H_{12}N_2O_2$ Molecular Weight: 228.25 Elemental Analysis: C, 68.41; H, 5.30; N, 12.27; O, 14.02 IR: 3380, 2940, 2850, 1640, 1610, 1560, 1340, 1230, 748 cm^{-1} NMR δ ppm: (300 MHz, DMSO- d_6 , 297K) δ =6.91 to 8.52 (m, 7H), 5.35 (Ar-OH, s, 1H), 8.59 (N=C-H, s, 1H). 3.83 (CH₃, s, 3H).
M.P. =140⁰ C Colour = Yellow Yield=60%

1f) N-(3-methoxybenzylidene) pyridin-2-amine:

Anal.Cal..For $C_{13}H_{12}N_2O$ Molecular Weight: 212.25 Elemental Analysis: C, 73.56; H, 5.70; N, 13.20; O, 7.54 IR: 2940, 2850, 1645, 1615, 1545, 1342, 1238, 735 cm^{-1} NMR δ ppm: (300 MHz, DMSO- d_6 , 297K) δ =7.00 to 8.52 (m, 7H), 8.65 (N=C-H, s, 1H). 3.85 (CH₃, s, 3H).
M.P. =165⁰ C Colour = Brown Yield=

2.2 ¹H NMR Spectra :

The signal observed in ¹H NMR Spectra of the Schiff base under study are collected in Table 1. The spectra exhibit a multiplet at 6.52-8.36 δ ppm for hydrogen of aromatic ring. The azomethine hydrogen (-CH=N) leads to a singlet of integration intensity equivalent to one hydrogen at 8.36-8.68 δ ppm. The spectra of 1a, 1b, 1c and 1e shows singlet signal at 5.35 δ ppm for the hydrogen of hydroxyl group. The spectra of 1b, 1c, 1e and 1f shows singlet signal at 3.83-3.85 δ ppm for the hydrogen of the OCH₃.

Table 1: Data from ¹H NMR spectra of Schiff base 1a-1f

Compound no.						Assignment (δ ppm)
1a	1b	1c	1d	1e	1f	
5.35	5.32	5.35	-	5.35	-	O-H proton
6.52-7.57	6.54-7.52	6.54-7.50	6.52-7.66	6.91-8.52	7.00-8.52	Ar C-H proton
-	3.83	3.84	-	3.83	3.85	Satu. C-H proton
8.36	8.66	8.68	8.36	8.59	8.65	-CH=N Azomethine proton

2.3 IR Spectra:

The IR spectra of Schiff bases under study are recorded in solid state selected bands of diagnostic importance are collected in Table 2. IR spectra of 1a, 1b, 1c and 1e shows broad band for OH group. All Schiff bases show C=N at 1640-1670 cm^{-1} . Aromatic ring C=C obtained at 1540-1560 cm^{-1} . All spectral data has good agreement with structure.

Table 2: Selected bands of diagnostic importance from the IR spectra for 1a-1f

Compound no.						IR Strech
1a	1b	1c	1d	1e	1f	
3350	3370	3350	-	3380	-	ν O-H bonded
2940	2930	2945	2950	2940	2940	ν ArC-H
-	2840	2830	-	2850	2850	ν Satu. C-H
1650	1640	1650	1670	1640	1645	ν C=N Schiff
-	-	-	1600	1610	1615	ν C=N ring
1550	1560	1550	1550	1560	1545	ν C=C bond
1350	1340	1350	1340	1340	1342	ν C-O Bond
1250	1240	1250	1280	1230	1238	ν C-N bond
740	740	741	743	748	735	Ar-CH bending

CONCLUSION

In Conclusion, we have developed convent and Environmentally Eco friendly Green method for synthesizing Schiff bases (1a-1f). Reaction does not require heating and catalyst. The reaction is carried at room temperature and yield of product were excellent.

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