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THE RAPID SYNTHESIS OF NOVEL SCHIFF-BASE WITHOUT SOLVENT UNDER MICROWAVE IRRADIATION

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ABSTRACT

A microwave-assisted preparation of a series of Schiff-base via efficient condensation of 2-Amino 4,6-dimethoxy pyrimidine and arayl aldehyde without solvent is described in high yield as well as environmental friendship reaction in organic synthesis.

KEYWORDS: Microwave-assisted reaction, Schiff-base, 2-Amino 4, 6 dimethoxy pyrimidine, Arayl aldehyde

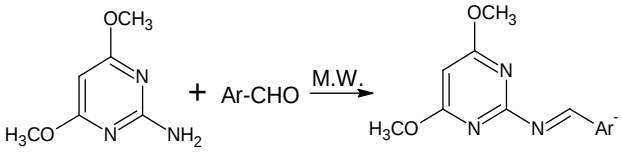
INTRODUCTION

The chemistry of the carbon-nitrogen double bond plays a vital role in the progresses of chemistry science.[1] Schiff-base compound have been used as fine chemicals and medical substrates. The aim of this research is to screen simple and economic methods for preparation of Schiff-bases. Synthesis of Schiff base is often carried out with acid-catalyzed and generally by refluxing the mixture of aldehyde (or ketone) and amine in organic medium.[2] Here in the microwave (M.W.) promoted condensation reaction of 2-Amino 4,6 dimethoxy pyrimidine and aryl aldehyde displayed the convient practicing way for forming a series of aldimine (Scheme-1). 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. The absence of solvent reduces the risk of hazardous explosions when the reaction take place in a closed vessel in a microwave oven.[3] Microwave-assisted reaction have been intensively investigated since the earliest publication of Gedye and Majetich in 1986.[4,5] Microwave-assisted techniques has been popularly used in organic synthesis.[6] The solvent free organic

synthesis mediated by microwave irradiation performs several advantages such as higher atom economy, environmental friendship, reducing the hazard etc.

MATERIALS AND METHODS

Melting point were recorded in open capillaries and on Veergo melting point apparatus. The ^{1}H NMR Spectra were recorded on a Bruker 300 MHz using TMS as an internal standard. The IR spectra were recorded on a Perkin Elmer Spectrum 100 FTIR Spectrophotometer and the Mass Spectra on a Waters Micromass Q-fit instrument. The chemical used are of A.R. grade. The microwave-assisted condensation carried out in a "Q-Pro-M Microwave synthesis system" manufactured by questron Technologies corporation, Ontario L4Z 2E9, Canada.



1 2 3

Scheme-1

Procedure for the synthesis of compound

However, with the assistance of microwave irradiation, it was found that the condensation reaction of 2-amino 4,6 dimethoxy pyrimidine and various aryl aldehyde could proceed fast and efficiently without solvent (Table1).

3-a: The microwave-assisted condensation of 0.01 mol 2-amino 4,6 dimethoxy pyrimidine with equal mole benzaldehyde were mixed together at ambient temperature in an Erlenmeyer flask (25 ml). The mixture was subjected to microwave for an optimized time on the "M-high" setting, the crude product were re-crystallized with methanol.

3-b: The microwave-assisted condensation of 0.01 mol 2-amino 4,6 dimethoxy pyrimidine with equal mole pera hydroxy benzaldehyde were mixed together at ambient temperature in an Erlenmeyer flask (25 ml). The mixture was subjected to microwave for an optimized time on the "M-high" setting, the crude product were re-crystallized with methanol.

3c: The microwave-assisted condensation of 0.01 mol 2-amino 4,6 dimethoxy pyrimidine with equal mole pera chloro benzaldehyde were mixed together at ambient temperature in an Erlenmeyer flask (25 ml). The mixture was subjected to microwave for an optimized time on the "M-high" setting, the crude product were re-crystallized with methanol.

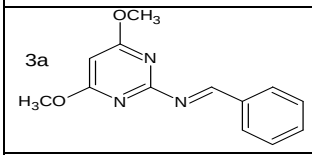
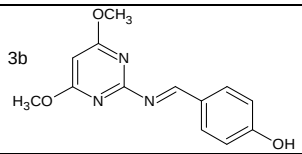
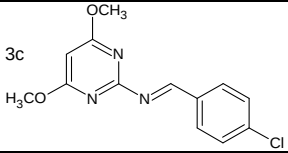
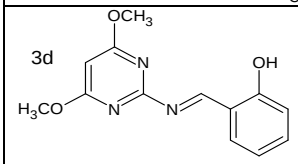
3-d: The microwave-assisted condensation of 0.01 mol 2-amino 4,6 dimethoxy pyrimidine with equal mole ortho Hydroxy benzaldehyde were mixed together at ambient temperature in an Erlenmeyer flask (25 ml). The mixture was subjected to microwave for an optimized time on the "M-high" setting, the crude product were re-crystallized with methanol.

3-e: The microwave-assisted condensation of 0.01 mol 2-amino 4,6 dimethoxy pyrimidine with equal mole pera Nitro benzaldehyde were mixed together at ambient temperature in an Erlenmeyer flask (25 ml). The mixture was subjected to microwave for an optimized time on the "M-high" setting, the crude product were re-crystallized with methanol.

3-f: The microwave-assisted condensation of 0.01 mol 2-amino 4,6 dimethoxy pyrimidine with equal mole 2,6 dimethyl benzaldehyde were mixed together at ambient temperature in an Erlenmeyer flask (25 ml). The mixture was subjected to microwave for an optimized time on the "M-high" setting, the crude product were re-crystallized with methanol.

3-g: The microwave-assisted condensation of 0.01 mol 2-amino 4,6 dimethoxy pyrimidine with equal mole 3,4,5 trimethoxy benzaldehyde were mixed together at ambient temperature in an Erlenmeyer flask (25 ml). The mixture was subjected to microwave for an optimized time on the "M-high" setting, the crude product were re-crystallized with methanol.

Table-1: Preparation of compound 3(a-g)

Compound	Yield(%)	Time
	93	2 mins
	92	4 mins
	94	30 Sec
	90	4 mins

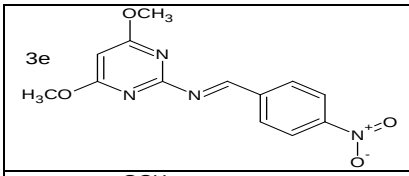
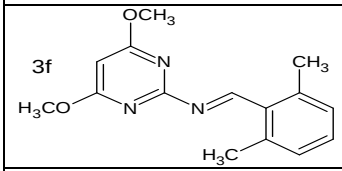
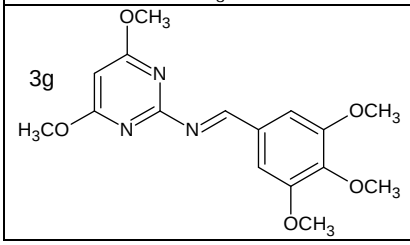
	95	1 mins
	98	3 mins
	89	3 mins

Table-2: Characteristics of Synthetic Compound

Comp. Id.	Molecular Formula	M.W.	M.P. (°C)
3a	$C_{13}H_{13}N_3O_2$	243.26	140
3b	$C_{13}H_{13}N_3O_3$	259.26	100
3c	$C_{13}H_{12}ClN_3O_2$	277.70	170
3d	$C_{13}H_{13}N_3O_3$	259.26	290
3e	$C_{13}H_{12}N_4O_4$	288.25	120
3f	$C_{15}H_{17}N_3O_2$	271.31	145
3g	$C_{16}H_{19}N_3O_5$	333.33	100

Table-2 show the molecular formula, molecular weight and melting point.

RESULT AND DISCUSSION

Characterization of compound 3a-3g

3a:Elemental Analysis : Found(Calcd.) C, 64.20 (64.19), H, 5.38(5.39), N,7.25(7.27) :IR (K Br. cm^{-1}) :1623 cm^{-1}(C=N), 3060 cm^{-1}(C-H, str), 1247 cm^{-1} (C-O-C,str), 1430 cm^{-1} (C=N, Ar)1H NMR(ppm) ($CDCl_3$):8.32(s, 1H, N=CH), 6.81-7.82(m, 6H), 2.65(s, 6H), MS:243[M.].

3b:Elemental Analysis : Found(Calcd.) C, 60.23 (60.22), H, 5.04(5.05), N,16.23(16.21):IR(KBr. Cm^{-1}) 1625 cm^{-1}(C=N), 3050 cm^{-1}(C-H, str), 1240 cm^{-1} (C-O-C,str), 1430 cm^{-1} (C=N, Ar), 3775 cm^{-1} (-OH Asym. Streach.) 1H NMR(ppm) ($CDCl_3$): 8.33(s, 1H, N=CH), 6.90-7.85(m, 5H), 2.68(s, 6H),11.61(s,1H), 3.32 (s, OH) MS:259[M.].

3c:Elemental Analysis : Found(Calcd.) C, 56.24 (56.22), H, 4.35(4.36), N,15.15(15.13):IR(KBr. Cm^{-1}) 1622 cm^{-1}(C=N), 3055 cm^{-1}(C-H, str), 1242 cm^{-1} (C-O-C,str), 1432 cm^{-1} (C=N, Ar), 788 cm^{-1} (Ar-Cl)

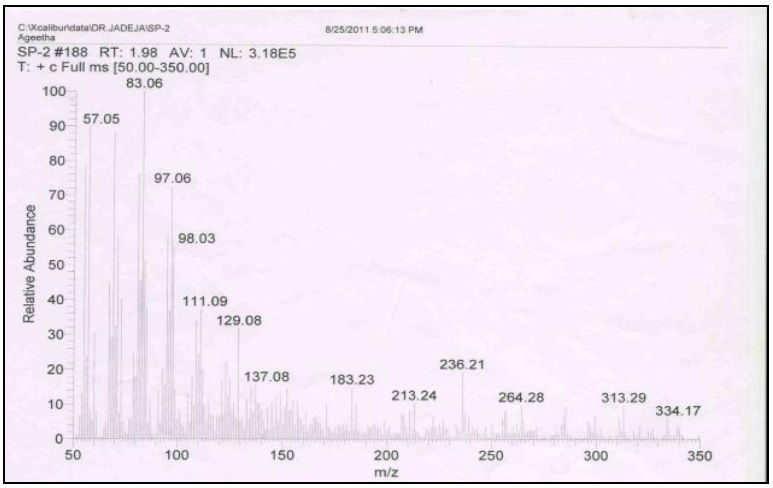
1H NMR(ppm) ($CDCl_3$): 8.38(s, 1H, N=CH), 6.87-7.90(m, 5H), 2.78(s, 6H), MS:277[M.].

3d:Elemental Analysis : Found(Calcd.) C, 60.20 (60.22), H, 5.03(5.05), N,16.22(16.21):IR(KBr. Cm^{-1}) 1627 cm^{-1}(C=N), 3059 cm^{-1}(C-H, str), 1248 cm^{-1} (C-O-C,str), 1439 cm^{-1} (C=N, Ar), 3709 cm^{-1} (-OH Asym. Streach.)
1H NMR(ppm) ($CDCl_3$): 8.34(s, 1H, N=CH) , 6.75-7.95(m, 5H), 2.72(s, 6H), 11.63(s, 1H), 3.40 (s, OH) MS:259[M.].

3e:Elemental Analysis : Found(Calcd.) C, 54.18 (54.17), H, 4.21(4.20), N,19.45(19.44):IR(KBr. Cm^{-1}) 1630 cm^{-1}(C=N), 3062 cm^{-1}(C-H, str), 1249 cm^{-1} (C-O-C,str), 1437 cm^{-1} (C=N, Ar), 1538 cm^{-1} & 1298 cm^{-1} (-NO2, two band)
1H NMR(ppm) ($CDCl_3$): 8.39(s, 1H, N=CH) , 6.72-7.81(m, 5H), 2.69(s, 6H), MS:288[M.].

3f:Elemental Analysis : Found(Calcd.) C, 66.41 (66.40), H, 6.33(6.32), N,15.48(15.49):IR(KBr. Cm^{-1}) 1629 cm^{-1}(C=N), 3064 cm^{-1}(C-H, str), 1242 cm^{-1} (C-O-C,str), 1439 cm^{-1} (C=N, Ar)
1H NMR(ppm) ($CDCl_3$): 8.31(s, 1H, N=CH) , 6.99-7.75(m, 4H), 2.56(s, 6H), 2.67(s,6H), MS:271[M.].

3g:Elemental Analysis : Found(Calcd.) C, 57.64 (57.65), H, 5.76(5.75), N,12.63(12.61):IR(KBr. Cm^{-1}) 1632 cm^{-1}(C=N), 3069 cm^{-1}(C-H, str), 1249 cm^{-1} (C-O-C,str), 1440 cm^{-1} (C=N, Ar)
1H NMR(ppm) ($CDCl_3$): 8.32(s, 1H, N=CH) , 6.95-7.88(m, 3H), 2.78(s, 9H), 2.56(s, 6H)
MS:334[M+1].Mass spectra of 4,6-dimethoxy-*N*-[(*E*)-(3,4,5-trimethoxyphenyl) methylidene] pyrimidin-2-amine (**3g**)



CONCLUSION

The method herein, showed is the most convenient way to form the aldimines, in which microwave irradiation plays an important role for promoting condensation reaction of aldehyde and amine.

REFERENCES

1. S. Patai. The Chemistry of the carbon-nitrogen double bond, John Wiley & Sons Ltd., London, 1970
2. M. M. Sprung. Chem. Rev., 1940; 26: 297.
3. S. A.-E. Ayoubi, F. Texier-Boullet, J. Hamelin., Synthesis, 1994; 3: 258.
4. R. Gedye, F. Smith, K. Westaway, H. Ali, L. Baldisera, L. Laberge, J. Roussel, Tetrahedron Lett., 1986; 27: 1729.
5. R.J. Giguere, T.L. Bray, S.M. Duncan, G. Majetich, Tetrahedron Lett., 1986; 27: 4945.
6. Y.W. Sha, Y. Wang, J. Ge, X. Wang, Chin. J. Org. Chem., 2001; 21: 102.