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Microwave Promoted Synthesis of Some Schiff Bases

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Abstract

Schiff bases have a wide range of applications such as corrosion inhibitor, intermediates in various reactions, in perfumery etc. Some are known to be used in many potential drugs and are known to possess broad spectrum of biological activities such as antiviral, antifungal, antiparasitic, antibacterial, anti-inflammatory. It can be synthesized by Microwave irradiation. Reaction of 6-methoxy-1, 3-benzothiazol-2-amine with substituted aldehydes under microwave irradiation (M.W.) gave the corresponding imines (Schiff bases). The structure of all the synthesized compounds was elucidated on the basis of IR, ^1H NMR and mass spectral data.

Keywords: Schiff bases, 6-methoxy-1, 3-benzothiazol-2-amine, Microwave irradiation

INTRODUCTION

A big challenge facing academia and industry is the relationship of modern societies to the environment that requires reinventing the manufacture and use of materials. Synthetic methodologies now a day should be designed to use and generate substances that possess little or no toxicity to human health and the environment. Schiff bases belong to a widely used group of organic intermediates important for production of specialty chemicals, e.g. pharmaceuticals, or rubber additives [1] and as amino protective groups in organic synthesis [2-3]. They also have uses as liquid crystals, [4] and in analytical, [5-7] medicinal [8] and polymer chemistry [9]. Conventionally Schiff bases have been prepared by refluxing mixtures of the amine and the carbonyl compound in an organic solvent, for example, ethanol or methanol [10], but variations are known, such as treatment of the same mixture at room temperature, refluxing the mixture in heptane in the presence of acetic acid [11], or azeotroping the mixture with benzene in a Dean-Stark apparatus in the presence of acid [12]. In general, ketones react more slowly than aldehydes and higher temperatures and longer reaction times are often required as a result. In addition, the equilibrium must often be shifted, usually by removal of the water, either azeotropically by distillation or with suitable drying agents [13]. In recent years, environmentally benign synthetic methods have received considerable attention and some solvent-free protocols have been developed [14]. Grinding together solid anilines and solid benzaldehydes yielded

various kinds of benzylideneanilines [15]. The synthesis of primary imines by condensation of 2-hydroxylaryl ketones with ammonium iodide and piperidine under solvent free conditions [16]. Based on these facts, we decided to synthesize some Schiff bases of 6-methoxy-1, 3-benzothiazol-2-amine in Microwave irradiation.

MATERIALS AND METHODS

Experimental

General

NMR spectra were recorded on BRUKER spectrometer (400 MHz) by CDCl_3 or DMSO as solvent with TMS as internal reference. Results are expressed in ppm. IR spectra were recorded on SHIMADZU-FTIR-8400 spectrometer over a frequency range $4000\text{-}400\text{ cm}^{-1}$ (DRS system). MS spectra were measured on a SHIMADZU-QP2010 mass spectrometer. TLC was conducted on standard aluminium sheets precoated with a 0.2 mm layer of silica gel (Merck). Microwave-assisted procedures were carried out in a microwave synthesizer Qpro-M operating at 1000 W.

General microwave procedure for the synthesis of Schiff bases (AK₁-AK₅).

To a mixture of 6-methoxy-1, 3-benzothiazol-2-amine (0.1 mol.) and substituted aromatic aldehyde (0.1 mol.) in methanol, catalytic amount of glacial acetic acid added then the contents were subjected to microwave irradiation at an interval of 1min at 400 W for about 8-10 min; progress of the reaction was monitored by TLC. After the completion of the reaction, the obtained product was poured into water stirred well; solid obtained was recrystallized from suitable solvent.

Spectral Data:-

6-methoxy-N-[(E)-(2-nitrophenyl)methylidene]-1,3-benzothiazol-2-amine (AK₁):

Yellow crystals; (recrystallized); ¹H-NMR (CDCl_3): $\delta = 3.86$ (s, 3H, OCH_3), 8.88 (s, 1H, N=CH), 6.91-7.88 (m, 7H, Ar-H), 10.28 (s, 1H, OH); MS: m/z 284; IR (KBr): -OH (3038 cm^{-1}), N=CH str. (Schiff base, 1583 cm^{-1}).

6-methoxy-N-[(1Z,2Z)-3-phenylprop-2-en-1-ylidene]-1,3-benzothiazol-2-amine (AK₂):

Yellow crystals; (recrystallized); ¹H-NMR (CDCl_3): $\delta = 3.85$ (s, 3H, OCH_3), 9.13 (s, 1H, N=CH), 6.94-7.46 (m, 7H, Ar-H), 1 s, 12.12H, OH); MS: m/z 284; IR (KBr): -OH (3039 cm^{-1}), HC=N str. (Schiff base, 1599 cm^{-1}).

N-[(E)-(4-fluorophenyl)methylidene]-6-methoxy-1,3-benzothiazol-2-amine (AK₃):

Greenish yellow crystals; (recrystallized); ¹H-NMR (CDCl_3): $\delta = 3.87$ (s, 3H, OCH_3), 9.08 (s, 1H, N=CH), 6.96-7.78 (m, 7H, Ar-H); MS: m/z 302; IR (KBr): HC=N str. (Schiff base, 1592 cm^{-1}).

6-methoxy-N-[(Z)-naphthalen-1-ylmethylidene]-1,3-benzothiazol-2-amine (AK₄):

Light yellow crystals; (recrystallized); ¹H-NMR (CDCl_3): $\delta = 3.86$ (s, 3H, OCH_3), 9.10 (s, 1H, N=CH), 6.91-7.68 (m, 7H, Ar-H); MS: m/z 302; IR (KBr): HC=N str. (Schiff base, 1603 cm^{-1}).

N-[(E)-anthracen-9-ylmethylidene]-6-methoxy-1,3-benzothiazol-2-amine (AK₅):

Yellow crystals; (recrystallized); ¹H-NMR (CDCl_3): $\delta = 3.87$ (s, 3H, OCH_3), 9.21 (s, 1H, N=CH), 7.08-7.93 (m, 7H, Ar-H); MS: m/z 313; IR (KBr): HC=N str. (Schiff base, 1608 cm^{-1}).

RESULTS AND DISCUSSION

In the given **Scheme-1** the starting compounds **1** were prepared according to the literature method. [17] The final compounds **3** were synthesized by the reaction between the substituted aromatic aldehyde **2** in the presence of acetic acid as catalyst under microwave irradiation. In presence of acetic acid catalyst, the reaction takes place more efficiently, milder conditions, with greater selectivity and it gives better yields within minutes. Moreover, the work up and purification procedures are simple as the catalyst is water soluble. The results of this study are summarized in **Table 1**.

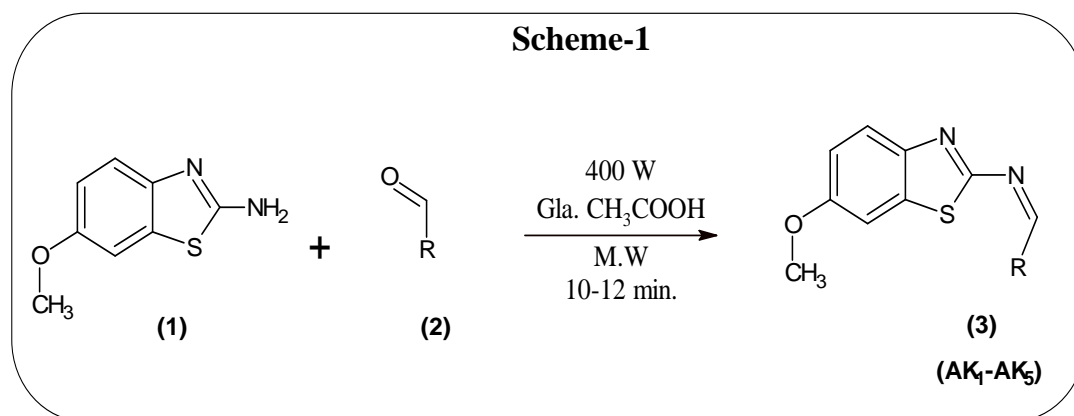


Table-1 Synthesis of Schiff bases by Microwave irradiation method

Compounds	R	Yield %	Time Taken (min)	Molecular Formula	Molecular Weight (gm/m)	M. P. °C	R _f * Value
AK ₁	2-NO ₂ -C ₆ H ₄	95	8	C ₁₅ H ₁₁ N ₃ O ₃ S	313.33	156	0.50
AK ₂	-CH=CH-C ₆ H ₅	92	11	C ₁₇ H ₁₄ N ₂ OS	294.37	102	0.58
AK ₃	4-F-C ₆ H ₄	94	12	C ₁₅ H ₁₁ N ₂ OSF	286.32	172	0.46
AK ₄	1-Naphthalene	91	10	C ₁₉ H ₁₄ N ₂ OS	318.39	164	0.54
AK ₅	9-Anthracene	89	8	C ₂₃ H ₁₆ N ₂ OS	368.45	140	0.59

CONCLUSION

In conclusion, a simple efficient and fast method has been developed for the synthesis of novel Schiff bases under microwave irradiation conditions in presence of acetic acid as catalyst.

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