

Montmorillonite K 10 clay catalyzed Microwave Synthesis of some Mannich bases and their Characterisation

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ABSTRACT: The synthesis of Mannich bases N-[1-(piperidinobenzyl) acetamide (**PBA**), N-[1-(morpholinobenzyl) acetamide (**MBA**), N-[1-(piperidinobenzyl) benzamide (**PBB**), N-[1-(morpholinobenzyl) benzamide (**MBB**) have been reported under microwave radiation catalysed by Montmorillonite K 10 clay. The product is obtained in good yield.

Key words: PBA, MBA, PBB, MBB, Microwave.

INTRODUCTION

It is well known from the literature¹⁻³ that the compounds containing amide moiety exhibit a wide range of biological activities. In the last few years **microwave-induced Organic Reaction Enhancement (MORE)** Chemistry has gained popularity as a non-conventional technique for rapid organic synthesis and many researchers have described accelerated organic reactions, and a large number of papers has appeared forming the synthetic utility of **MORE** chemistry in reactive organic synthesis. It can be termed as '**e-chemistry**' because it is easy effective, economical and eco-friendly and is believed to be a step towards green chemistry.

In continuation of our research programme⁴⁻⁷, we herein report the microwave assisted synthesis of some Mannich bases like, **PBA**, **MBA**, **PBB** and **MBB** catalysed by Montmorillonite K 10 clay. The synthesized compounds have been characterized by

TLC, Elemental analysis, IR and ¹H-NMR Spectroscopy.

Recently use of inorganic solid supports⁸⁻¹¹ as catalysts has been developed for solvent-free reactions resulting in milder conditions and easy experimental procedures. Clay catalyzed organic reactions are gaining importance owing to their inexpensive nature and special catalytic attributes in heterogeneous reactions.

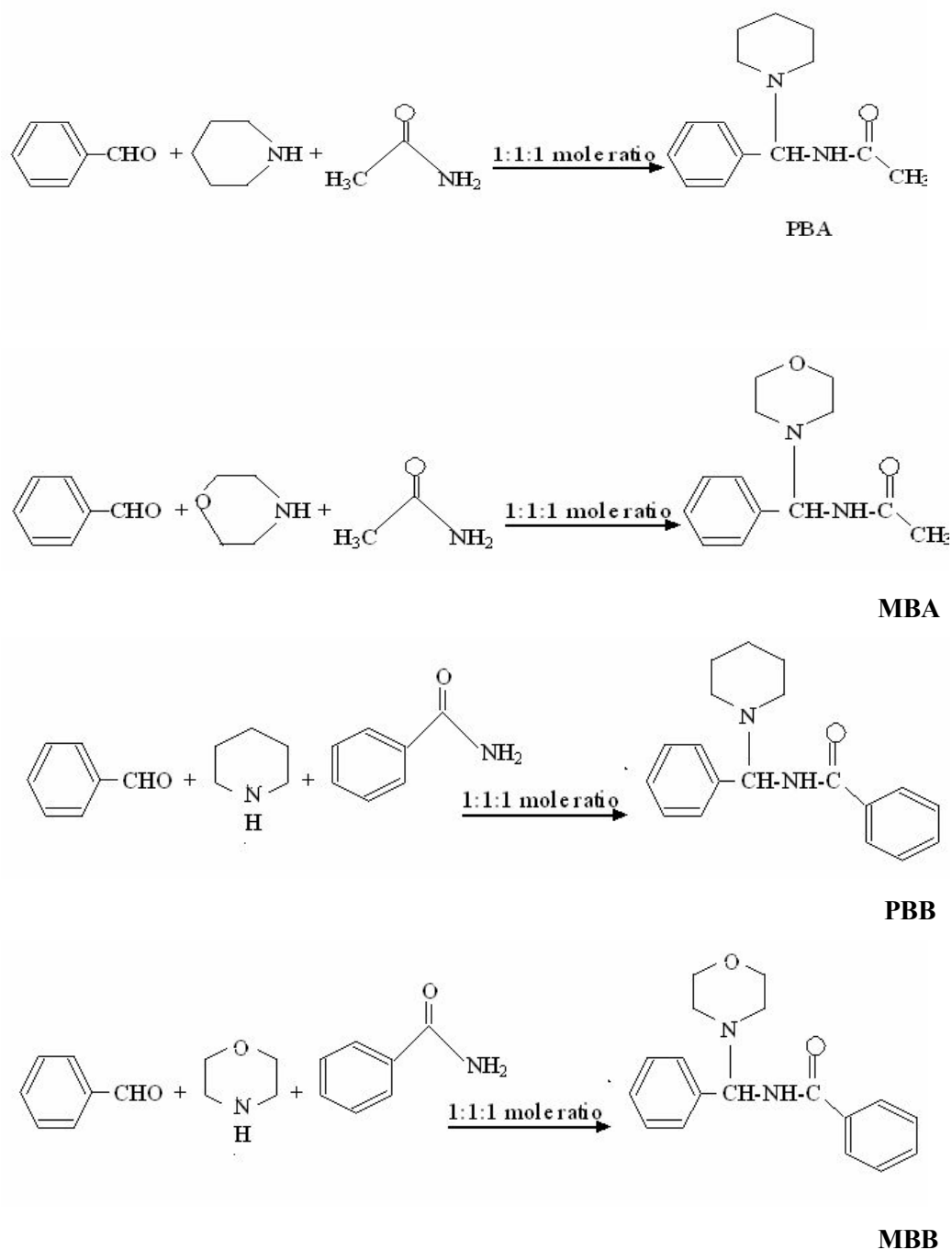
EXPERIMENTAL

Melting points were determined in an open capillary tube with a Buchi melting point apparatus and are uncorrected. Elemental analyses were carried out using Perkin-Elmer 240C CHN-analyzer. IR spectra were recorded on a Perkin Elmer IR spectrophotometer. ¹H-NMR spectra was run in (CDCl₃) solvent at 200 MHz on a NMR spectrophotometer (chemical shifts in δ ppm).

General procedure for the Synthesis of Mannich base PBA/MBA/ PBB and MBB

Benzaldehyde, piperidine/ Morpholine and acetamide/ Benzamide were taken in 1:1:1 mole ratio. Piperidine / Morpholine 0.9 mL (10 mM), acetamide 0.6 g (10mM)/ Benzamide (0.9 g) and then 1 mL of benzaldehyde (10 mM) was added and kept under microwave radiation at 130°C for 2 min. Then the

reaction mixture was acidified with 5 mL of concentrated HCl and the lower layer was separated, washing the water layer with dichloromethane (DCM). The organic layer was dried and, after vacuum distillation, provided the desired Mannich base in reasonable yields, **Table 1** and **Scheme 1**. Thin layer chromatography was used to check the purity of the compound.

Scheme 1

RESULTS AND DISCUSSION

The elemental analyses value consistent with the stoichiometry for all the Mannich bases and the elemental analyses value of Mannich bases are given in the Table 1.

Table 1. Analytical data of the Mannich bases

Complex	Found (calculated)			m.p.	Yield (%)
	C %	H %	N %		
PBA	71.65 (72.36)	8.13 (8.68)	11.87 (12.06)	167 ⁰ C	81
MBA	65.86 (66.64)	7.12 (7.74)	11.23 (11.96)	169 ⁰ C	79
PBB	76.8 (77.52)	7.1 (7.53)	8.7 (9.52)	171 ⁰ C	80
MBB	71.86 (72.7)	6.91 (7.12)	9.08 (9.42)	177 ⁰ C	74

The IR-spectral data of the Mannich bases are given in the Table 2.

Table 2. IR spectral data of the Mannich bases $\nu(\text{cm}^{-1})$

Compound	$\nu(\text{NH}_2)$	$\nu(\text{C=O})$	$\nu(\text{C-N-C})$
PBA	3312	1646	1160
MBA	3325	1640	1160
PBB	3366	1636	1121
MBB	3367	1634	1140

The ¹H NMR spectral data of the Mannich bases are given in the table 3.

Table 3. ¹H NMR spectral data of the Mannich bases

Compound	$\delta(\text{H-Aro})$	$\delta(\text{H-NH})$	$\delta\nu(\text{Mor N-CH}_2)$	$\delta\nu(\text{Pip N-CH}_2)$	$\delta(\text{Methyl})$	$\delta\nu(\text{Mor-o-CH}_2)$
PBA	7.2-7.7	5.56	-	2.5	2.2	
MBA	7.2-7.7	5.5	2.5	-	-	3.6
PBB	7.2-7.7	5.9	-	2.6	2.4	
MBB	7.2-7.7	5.0	2.7	-	-	3.6

In conclusion this condensation method offers an alternative route for the novel synthesis of Mannich base in reasonable yields making the process more economic than other conventional methods.

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