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SOLVENT FREE MICROWAVE ASSISTED SYNTHESIS OF A NOVEL BIOLOGICAL AGENT

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ABSTRACT

Novel compound was synthesized with N and O in its structure. The system was assisted by microwave technique in a solvent free environment. Satisfactory microbial activity was observed.

Key words: Solvent free, Microwave assisted synthesis, Biological agent.

INTRODUCTION

The interest in azomethines relates to their function as primary reagents for cycloaddition, cyclizations and as important building blocks in enantioselective oxidations. They have been used as, for the synthesis of fine chemicals, which have wider applications as agricultural chemicals, dyes and medicines[1,2].

Their potentiality as antifungal, hypotensive, hypothermic reagent[3], antibacterial, antituberculosis, anticancer[4] and antiinflammetry[5] reagents is well established. During the last decade, there has been a growing interest in new reaction condensations and activation, e.g dry conditions (reactions without solvent), reactions under extreme or non-conventional conditions (high pressure, ultrasound or microwave activation). The effects usually expected are rate enhancement, yield or selectivity improvement, easier work up and less polluting processes[6,7]. Microwave heating makes convenient to perform reactions efficiently in absence of any organic solvents. Thus the "green chemistry" concept comes into play[8-10].

EXPERIMENTAL

The identity of the synthesized compounds were confirmed by IR, ^{1}H NMR and UV methods. Melting point was determined by usually capillary method and is uncorrected. The chemical shifts are expressed in δ (ppm). IR spectra was recorded on Brucker α-E model. UV spectra scanned on double beam UV-visible spectrophotometer CE 7400 and ^{1}H NMR spectra

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on Hitachi Perkin-Elmer spectrometer in deuteriochloroform solution gave the δ values.

Conventional procedure

To a mixture of 5-amino-2-methyl phenol 0.12 g and 3-phenoxy benzaldehyde 0.20 g (15 mmol) was added a solution of chloroform (20 mL) and piperidine (0.05 mL) as catalyst. This mixture was heated by conventional thermal method for 6 hrs. The reaction mixture was evaporated and solid crystalline mass was extracted in ethanol.

Microwave procedure

Same procedure was followed but was heated by microwave for 2 to 10 min. The "environmentally friendly", SFMW (solvent free microwave assisted) synthesis method is a clean method, which avoids large quantity of solvent. Satisfactory elemental parameters were obtained.

RESULTS AND DISCUSSION

Table 1: SFMW method / Thermal method elemental parameters

Ligand PBPMP m.p. (152°C)	Time s/hr.	Temp. (°C)	Product (w)	Yield %	N% Found (calculated)	O% Found (calculated)
	8 sec.	140°C	0.59 g	65%	7.49	4.28
	6 hrs.	250°C	0.31 g	52%	(7.58)	(4.33)

All synthesized compounds were subjected to IR, ^{1}H NMR and UV test and the results confirm the purity of the compounds.

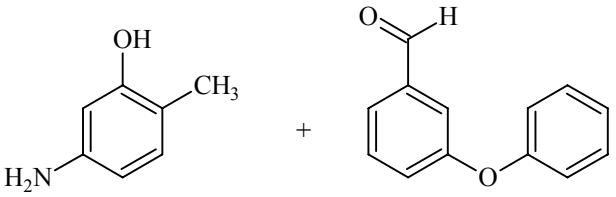
IR spectra provided the valuable information regarding the nature of functional group attached. Besides the bands at 1640 cm^{-1} (C=N), 3500 cm^{-1} (OH), 1175 cm^{-1} (asymmetric str) and 1090 cm^{-1} (symmetric str) (C-O-C), and an additional band for (C-N ring) was observed at 1121 cm^{-1}. The UV absorption spectra resulted in two bands (i) 277 nm (π-π* at high intensity) (ii) 289 nm (n-π* at low intensity). Characteristic signals in the ^{1}H NMR spectra are summarized in Table 2.

Table 2

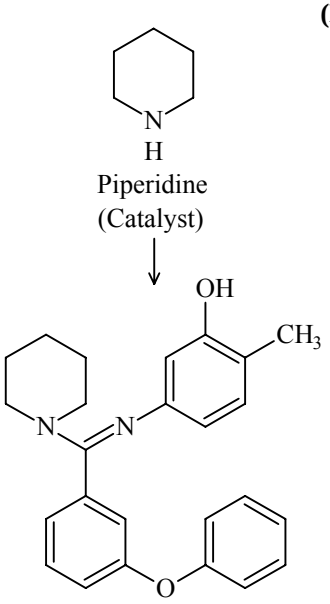
S. No.	Type of proton	δ (ppm)
1	Phenolic (Ar-OH)	5.2
2	Benzylic (Ar-CH_3)	2.5
3	Aromatic ring (=CH)	7.2
4	Piperidine ring (-CH_2)	1.3

Antibacterial activity

Ligand PBPMP was screened for its antibacterial activity using disc diffusion method suggested by Maruzella and Percival.[11]



5-Amino-2-methyl phenol **(A)** + 3-Phenoxybenzaldehyde **(B)**



3-Phenoxybenzaldimino-piperidine-6-methyl phenol

(PBPMP)

The bacterial strains used are *Vibro cholerae*, *Salmonella typhii*, *Shigella flexneri*, *Proteus vulgaries*, and *Escherichia coli*, *Staphylococous aureus*, *Klebsiella pneummonia, Streptococcus aureus and Pseudomonas aeruginosa*. Results are formulated (Table 3) as an average of three determinations.

Anti fungal activity

The inhibitory effect of the ligand against certain selected fungi by disc diffusion method are studied in detail. The fungal stains used in the study are *Aspergillus flavus*,

Aspergillus niger, *Aspergillus fumigatus*, *Mucor*, *Microsporum gypseum*, *Candida albicans* and *Rhizopus*. Result values is an average of three determinations (Table 3).

Table 3: *In vitro* inhibition profile of ligand against test bacteria and fungi

Bacteria	ppm			ppm			Fungi
	100	200	500	100	200	500	
V. Cholerae	8	12	17	10	14	23	***A. flavus***
S. typii	16	19	24	23	25	28	***A. niger***
S. flexneri	12	16	21	13	15	25	***A. fumigatus***
P. vulgaries	17	21	25	17	23	26	***Mucor***
E. coli	12	18	24	16	20	22	***M. gypseum***
S. aureus	17	21	24	15	19	25	***C. albicans***
K. pneummonia	12	16	21	14	18	21	***Rhizopus***
S. aureus	22	24	26				
P. aeruginosa	20	22	23				

All values are in millimeter, representing the diameter of the zone of inhibition.

CONCLUSION

Satisfactory microbiological parameters reveal that the ligand can act as antibacterial and antifungal agent, though the values are weak.

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