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Synthesis of Schiff Base Complexes of Lanthanides in Microwave Assisted via Eco Friendly Method

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Abstract: *Some new Schiff base complexes of dysprosium derived from alpha napthyl amine with different benzaldehyde. Reactions were carried out under microwave conditions. The ligands and their dysprosium complexes show a good activity against the bacteria S.aureus E.coli and fungi Penicillium Crysogenum, Aspergillus niger. The Schiff bases and their dysprosium complexes show variable activity of inhibition on the growth of bacteria.*

Keywords: Microwave synthesis, Schiff's base, antibacterial activity, eco-friendly

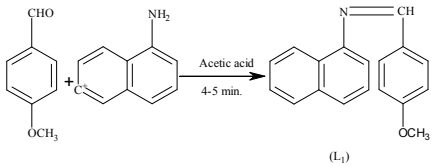
I. INTRODUCTION

Microwave irradiated reactions under solvent free or less solvent conditions are attractive offering reduced pollution, low cost and offer high yields together with simplicity in processing and handling. Recently, Microwave heating has emerged as a powerful technique to promote a variety of chemical reactions [1-2].

Schiff base ligands have been widely studied in the field of coordination chemistry mainly due to their facile syntheses, easily availability, electronic properties and good solubility in common solvents and they easily form stable complexes with most transition metal ions [3-5]. Schiff base metal complexes have played a key role in the development of coordination chemistry, resulting in an enormous number of publications, ranging from pure synthetic work to modern physicochemical and biochemically relevant studies of metal complexes[6] Lanthanide Schiff base complexes have various applications in agriculture and medicine [7-8]. The Schiff base complexes having a wide range of applications in analytical Chemistry [9]. The Schiff base complexes are highly remarkable class of compounds having a wide range of applications in antibacterial [10], antifungal [11], antitumor [12] and anti-inflammatory [13] activities and they possess considerable physiological activities [13] as solvents. Mass Spectra were recorded on Q-TOF MICROMASS spectrometer.

1.1 Microwave method for the Synthesis of Schiff Bases

The equimolar (1:1) ratio of alpha napthylamine different aldehydes viz.4-methoxy benzaldehyde, 4-hydroxy benzaldehyde



(L_1)

Scheme 1

$$Dy(NO_3)_3.XH_2O + \text{Ligand MW 7-10 min } [\ Dy(L)NO3\ (H_2O)]NO_3$$

Scheme 2

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II. MATERIAL AND METHODS

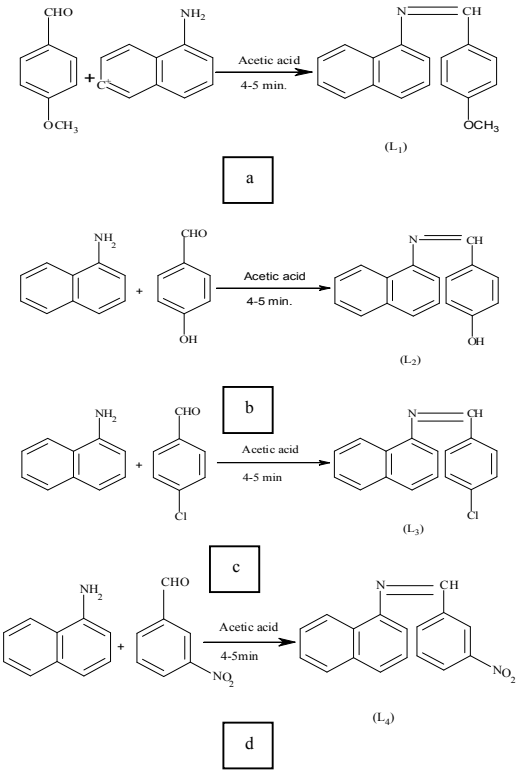
Under the work of Green Chemistry we have developed an environmentally benign method for synthesizing some Schiff bases and their metal complexes. All the chemicals and solvents used were of A.R. grade. All chemicals used were of Merck and S.D. fine Ltd. The IR spectra were recorded on a PERKIN ELMER spectrophotometer in the frequency range 4000-400 cm-1 in Nujol mull and as KBr pellets. 1HNMR spectra were recorded on 4-chloro benzaldehyde, 3-nitro benzaldehyde were mixed thoroughly in a grinder. The reaction mixture was then irradiated by the microwave oven by taking 3-4 ml acetic acid. The reaction was completed in a short time (4-5 min) with higher yields. The resulting product was then recrystallized with ethanol and finally dried under reduced pressure over anhydrous CaCl2 in a desiccators. The progress of the reaction and purity of the product was monitored by TLC using silica gel.

The ligand and the metal salts were mixed in 1:2 (metal: ligand) ratio in a grinder. The reaction mixture was then irradiated by the microwave oven by taking 3-5 ml acetic acid. The reaction was completed in a short time (7-10 min) with higher yields. The resulting product was then recrystallized with ethanol and ether and finally dried under reduced pressure over anhydrous CaCl2 in a desiccator. The progress of the reaction and purity of the product was monitored by TLC using silica gel.

The following Schiff base ligands obtained and characterized.

1. 1-(4-methoxy benzylidene) napthyl amine (MBNA)
2. 1-(4-hydroxy benzylidene) napthyl amine (HBNA)
3. 1-(4-chloro benzylidene) napthyl amine (CBNA)
4. 1-(3-nitro benzylidene) napthyl amine (NBNA)

Microwave method for the Synthesis of metal complexes:



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III. Results and Discussion

3.1 IR Spectral Studies

The IR spectra of the complexes were compared with those of the free ligand in order to determine the coordination sites that may be involved in coordination. Upon comparison it was determined that the ν(C=N) stretching vibration is found in the Schiff base at 1570-163 cm1. This band shifted to lower wave numbers in the complexes indicating the participation of nitrogen in coordination.

Table 1: Physical properties of the Schiff bases and Dysprosium (III) complexes.

Compound	Molecular Formula	Color
MBNA	$C_{18}H_{15}NO$	Grey
$[Dy(L1)(NO_3)(H_2O)]NO_3$	$C_{18}H_{17}N_3O_8Dy$	Pale yellow
HBNA	$C_{17}H_{13}NO$	White
$[Dy(L2)(NO_3)(H_2O)]NO_3$	$C_{17}H_{15}N_3O_8Dy$	Light yellow
CBNA	$C_{17}H_{12}NCl$	Cream
$Dy(L3)(NO_3)(H_2O)]NO_3$	$C_{17}H_{14}N_3O_7ClDy$	Light brown
NBNA	$C_{17}H_{12}N_2O$	Yellow
[Dy(L4)(NO3)(H2O)]NO_3	$C_{17}H_{14}N_4O_9Dy$	Yellow

The new band at ν M-O and M-N stretching vibrations were appeared at 550-440 and 510-560 cm-1in the spectra of metal.

Table 2: IR Spectral data of Schiff base and Dysprosium (III) complexes.

Compound	(Ar-H)	(CH=N))	(-NO_3)	(M-N)	(M-O)
SB_1	1580	3059	-		
$[Dy(L1)(NO_3)(H_2O)]NO_3$	1622	3042	1306	514	440
SB_2	1624	3035	-		
$[Dy(L2)(NO_3)(H_2O)]NO_3$	1622	3055	1398	501	468
SB_3	1615	3050	-		
$Dy (L3)(NO_3)(H_2O)]NO_3$	1615	3032	1355	536	451
SB_4	1616	3055	-		
$Dy (L4)(NO_3)(H_2O)]NO_3$	1613	3053	1350	510	487

^{1}HNMR Spectra:

The ^{1}H NMR spectrum of the Schiff bases complexes and its Dy(III) complexes taken in DMSO exhibits resonance at 8.2-8.8 ppm due to the azomethine proton. The sharp multiplet signals of the phenyl protons are found in the region 7.1-7.8 ppm. The methyl protons of the ligand are observed as a sharp peak at 3.3 ppm.1HNMR spectra of ligands and complexes are presented in above Table 3.

Table 3: ^{1}HNMR Spectral data of Schiff base and Dysprosium(III) complexes

Compound	Aromatic H	C-H azomethine
MBNA	7.28	8.35
$[Dy(L1)(NO_3)(H_2O)]NO_3$	7.4-7.9	8.18
HBNA	7.93	8.49
$[Dy(L2)(NO_3)(H_2O)]NO_3$	7.1-7.8	8.66
CBNA	7.66	8.60
$[Dy(L3)(NO_3)(H_2O)]NO_3$	7.5	8.94
NBNA	7.69	8.45
$[Dy(L4)(NO_3)(H_2O)]NO_3$	7.3-7.9	8.88

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IV. ANTIMICROBIAL ACTIVITY

The ligand and some of their corresponding metal complexes were screened in vitro for their antibacterial and antifungal activity against the fungi Penicillium crysogenum and Aspergillus niger using agar well diffusion method using Streptomycin as standard. The results of their antibacterial and antifungal activity against bacterial strains S.aureus, E.coli and the fungi Penicillium crysogenum and Aspergillus niger using agar well diffusion method using Streptomycin as standard. The results of antibacterial studies are presented in Table 5 comparative study of the ligand and their metal complexes indicates that most of the metal complexes exhibit higher antimicrobial activity than that of the free ligand and the control. Hence complexation increases antimicrobial activity.

Table 4: Antimicrobial activity of Schiff Bases and their Dysprosium(III) complexes.

Compound	Antibacterial		Antifungal	
	E.coli	S.aureus	Aspergillusniger	Penicillium crysogenum
SB_1	18	18	16	22
$[Dy(L1)(NO_3)(H_2O)]NO_3$	22	21	18	16
SB_2	20	18	18	18
$[Dy(L2)(NO_3)(H_2O)]NO_3$	24	24	18	R
SB_3	20	18	15	16
$[Dy(L3)(NO_3)(H_2O)]NO_3$	15	12	14	15
SB_4	20	28	16	R
$[Dy(L4)(NO_3)(H_2O)]NO_3$	22	24	18	16
Streptomycin	22	24	16.5	16

(Diameter of inhibition zone in mm)

In Table 5-

R : Resistant (10.0 mm and below)

S : Sensitive (10.0 mm and above)

Slightly sensitive : (10.0 mm above to 15.0 mm)

Moderately sensitive : (15.0 mm above to 20.0 mm)

Highly sensitive : (20.0 mm above

V. CONCLUSION

In conclusion, we have described here an efficient and environmentally benign synthesis of Schiff base ligands and their corresponding Dy(III) complexes under microwave irradiation using acetic acid as solvent. Further, this method is simple, mild and ecofriendly from green chemistry point of view.

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