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Synthesis, Spectral, Magnetic Susceptibility and Antifungal Studies On Symmetrically Substituted Metal (II) Octafuryl methanimine Phthalocyanine

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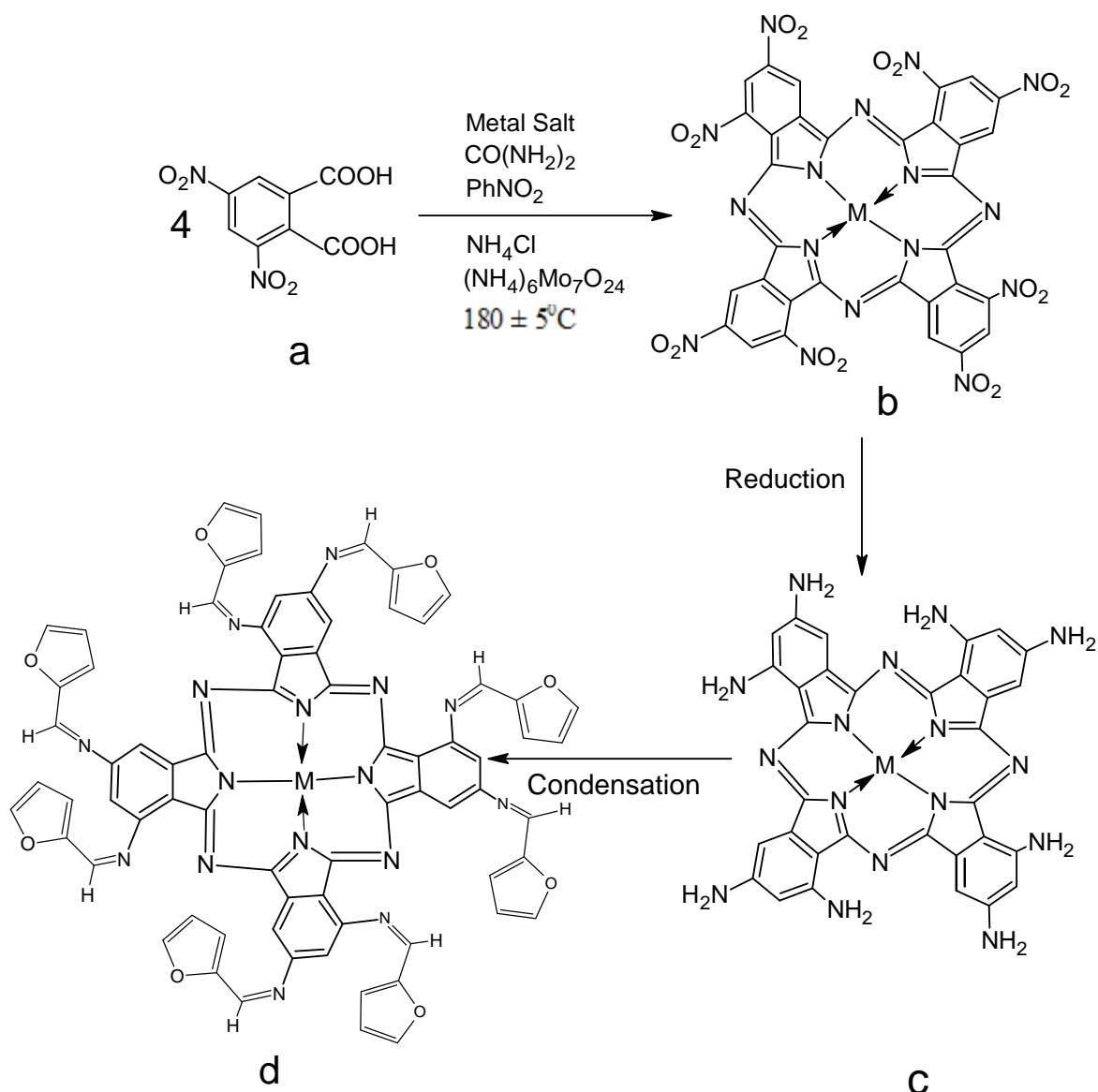
ABSTRACT

Novel phthalocyanines with eight octa-1-(3-furyl) methanimine phthalocyanine, (Cu-furylImPcO) groups. Present paper discuss the synthesis and characterization of metal (II) 1, 3, 8, 10, 15, 17, 22, 24- octa-1-(3-furyl) methanimine phthalocyanines (M-furylImPcO) (M = Cu, Co, Ni, Zn) by an efficient simple method. Octaamino metal (II) phthalocyanines were synthesized by the reduction of the corresponding nitro phthalocyanines. The dark green octa-1-(3-furyl)methanimine phthalocyanine derivatives were characterized by elemental analysis, magnetic susceptibility, electronic, IR and powder X-ray diffraction studies to check the purity, structural integrity and crystalline properties of the complexes. Magnetic susceptibility studies on Co (II) and Cu (II) octa-1-(3-furyl) methanimine phthalocyanine complexes exhibit a variation of the magnetic moments as a function of field strength indicating the presence of inter molecular co-operative effect. The complexes were screened for antifungal activity.

Key words: Phthalocyanines: Octa-1-(3-furyl)) methanimine, Electronic, IR, Magnetic, XRD, Thermal.

INTRODUCTION

Phthalocyanines are man's analogues of nature's pigments of life, the porphyrins, such as chlorophyll and hemoglobin. Since their discovery in 1928, phthalocyanines have become second only to the ubiquitous azo colorants in terms of commercial importance. Phthalocyanine ligand has a heteroaromatic π system and readily forms complexes with many groups and transition metals. The azo-nitrogen and peripheral fixed benzene rings imparts chemical and thermal stability to the ligand [2].



Scheme.1: Synthesis of metal (II) 1, 3, 8, 10, 15, 17, 22,24- octa-1-(3-furyl) methanimine phthalocyanine. a. 3, 5-dinitrophthalic acid, b. M-PcON, c. M-PcOA and d. M-furylImPcO

The earlier interest in metal phthalocyanines was mainly because of their importance as dyes and pigments [3]. Phthalocyanines are well known commercial blue green pigments. The colour is due to intense absorption in the visible region of the spectrum, the Q-band [4-5]. Currently intensive research work is aiming at the production of useful phthalocyanines as sensitizers in PDT, catalysts in fuel cells[6], sensors, display devices, information storage systems, semiconductors [7] and toners in colour copiers and laser printers and hexadeca fluocopper phthalocyanines as the leading electron transport materials for organic semiconductors[8-9].

In the present paper an attempt has been made to have a suitable substituent at the periphery of the molecule. The imine group was introduced by the reaction of amino derivatives of the

phthalocyanine with substituted aldehydes. Even though, the literature survey revealed about the reports on synthesis and structural investigation of metal (II) 1, 3, 8, 10, 15, 17, 22, 24-octaamino phthalocyanines, no documentary evidences were available on synthesis and structural studies on metal (II) 1, 3, 8, 10, 15, 17, 22, 24- octa-1-(3-furyl) methanimine phthalocyanine complexes.

The procedure available in the literature is suitably modified and the synthetic route adopted for the synthesis of octa-1-(3-furyl) methanimine substituted metal (II) phthalocyanines is given in the scheme-1

MATERIALS AND METHODS

Experimental

The 3,5-dinitrophthalic acid was synthesized as reported elsewhere [10] and all other chemicals used in the experiment were of analytical grade. The analytical data of the synthesized compounds were in good agreement with reported ones and are used in the synthesis of octa-1-(3-furyl) methanimine substituted metal phthalocyanines.

Preparation of Cobalt (II) 1, 3, 8, 10, 15, 17, 22, 24- octa-1-(3-furyl) methanimine phthalocyanine complex.

The procedure adopted for the synthesis of cobalt (II), 1, 3, 8, 10, 15, 17, 22, 24-octanitrophthalocyanine complex is reported elsewhere [11]. The nitro derivative of the aforesaid complex is converted into amino derivative quantitatively by reduction using sodium sulphide nonahydrate ($\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$) in aqueous medium [12]. The finely grounded metal (II) 1, 3, 8, 10, 15, 17, 22, 24-octaaminophthalocyanine (6.30g, / 0.1mole) was dissolved in stiochiometric quantity of 15 M sulphuric acid, to this (12.9g, / 0.1mole) furfuraldehyde is dissolved in ethyl alcohol and catalytic amount of concentrated sulphuric acid is added, and the contents were refluxed with stirring for about 5 hrs [13].

The settled green colored condensed octa-1-(3-furyl) methanimine phthalocyanine complex is washed with alcohol several times until it is free from aldehyde. Finally it is washed with distilled water and dried over anhydrous phosphorous pentaoxide in vacuum desiccator.

The pigment form of the above complex is obtained by the acid pasting process, in which 1 part of the powdered sample was dissolved in 6-10 parts of concentrated sulphuric acid. The mixture is allowed to stand for 1-2 hour and then poured on to 45-50 parts of crushed ice and stirred thoroughly. The pigment thus obtained is filtered off and washed with hot water. Finally it is washed with distilled water and dried in vacuum over phosphorous pentaoxide.

Metal (II) 1, 3, 8, 10, 15, 17, 22, 24- octa-1-(3-furyl) methanimine phthalocyanines of copper (II), nickel (II), and zinc (II) were prepared by the above procedure using respective amino metal phthalocyanines.

Aspergillus niger was used for its growth and sporulation color characteristics in the presence of the selected metal phthalocyanine complexes. *Aspergillus niger* was procured from the Department of Biotechnology, Sahyadri science college, Shimoga. A 100 ppm solution of different M-furylImPcO complexes were prepared by dissolving required amount of complexes in 2 ml of DMSO and 98 ml of PDA medium and sealed with aluminum foil and sterilized in an

utoclave at a temperature 120 °C and 15 psi pressure for 15 min. The hot sterilized medium was poured into sterile Petri plates in an aseptic chamber and cooled. *Aspergillus niger* was inoculated on the solidified medium as a point at the center of the plate. The inoculated plates were incubated at $23 \pm 1^\circ\text{C}$ for one week, and the observations were made on 3rd, 5th and 7th day.

RESULT AND DISCUSSION

C, H and N analysis were performed at Cochin University, Sophisticated Test & Instrumentation Center, Kochi, Kerala, India. The metal content was determined by incinerating them to the oxides. Magnetic susceptibility studies were carried out at room temperature ($301\text{ }^\circ\text{K}$) using a Gouy balance consisting of NP-53 type electromagnets with a DC power supply unit and a semi microbalance. Pascal's constants were used to calculate the diamagnetic corrections [14]. A mercury tetra thiocyanato cobalt (II) ($\text{Hg}[\text{Co}(\text{SCN})_4]$) complex is used as a calibrant and doubly distilled water used in the experiments. Shimadzu UV-Visible recording spectrophotometer, UV-160A with 1 cm width cells is used for electronic absorption spectral studies. IR spectra were recorded using Nicolet MX-FT IR spectrometer with KBr pellets in the range $4000\text{-}500\text{cm}^{-1}$. Phillip Analytical PW1710 X-ray diffractometer is used to study the diffraction pattern. The spectra were recorded using Cu $\text{K}\alpha$ at the voltage of 40 KV, a current of 20mA, a time constant of 4, a channel width of 7mm and chart speed of 10mm/min. Perkin-Elmer Thermal analyzer is used for simultaneous record of TGA and DTA at the rate of $10\text{ }^\circ\text{C}/\text{min}$ both in air, and nitrogen atmosphere.

The procedure used for the synthesis of M-furylImPcO's results in pure complexes and have dark green color for Cu (II), Co (II), Ni (II) and green with brown ting for Zn (II). The metal phthalocyanine complexes, which are thermally stable and resistance towards concentrated sulfuric acid was attributed to the atomic radii of the metal in the close range of 1.35 \AA . These complexes give clear solution in DMSO, and DMF, but insoluble in water and most of the organic solvents, like alcohol, ether, carbon tetrachloride and benzene. The results of elemental analysis for carbon, hydrogen nitrogen and metal are in good agreement with the calculated and are summarized in Table. 1. The above results are consistent with the suggested structure in fig-1.

Electronic Spectra

The electronic spectra of octa-1-(3-furyl) methanimine metal phthalocyanines are recorded in the range of $1.0\text{-}1.5 \times 10^{-4}$ in 18M sulfuric acid and the results are tabulated in Table 2. For all the complexes, a peak in the range of 601-615 nm [15] was observed and assigned to Q-band, attributed to $a_{1u} \longrightarrow e_g$ transition. Peaks appeared in the range 266-276 nm [16] are attributed to C-band. The M-furylImPcO's showed a peak in the range of 374-390 nm is assigned to B-band and is attributed to $a_{2u} \longrightarrow e_g$. The peaks in the longer wavelength region 727-748nm. A weak L-band was observed in the range of 207-216. For all the complexes showed a bathochromic shift with increasing intensity with respect to the corresponding parent phthalocyanines [17].

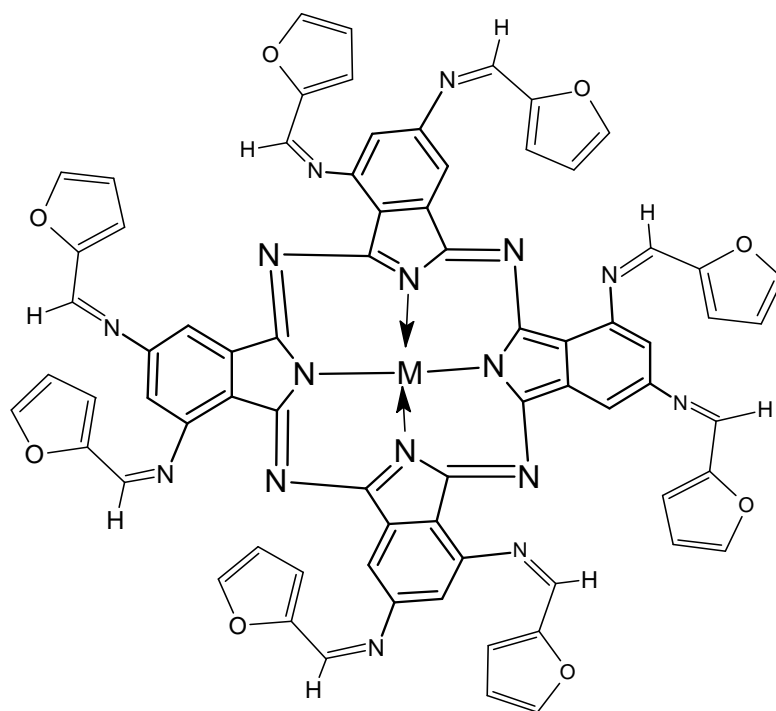


Fig.1: Suggested structure of symmetrically substituted octa-1-(3-furyl) methanimine phthalocyanines, where M= Co, Cu, Ni, and Zn.

Infrared Spectra

IR spectral data of all the metal (II) octa-1-(3-furyl) methanimine phthalocyanine pigments were recorded in KBr pellets and the results are tabulated in Table.2. (Fig.2.) A peak was observed for all M-furyllmPcO's in the range $3400-3465\text{cm}^{-1}$ may be accounted for the presence of hydrogen bonding between nitrogen group and hydrogen of moisture absorbed on KBr pellets [18]. A very weak signal observed in the range $2309-2325\text{cm}^{-1}$ is due to C-H stretching vibration on the periphery of the phthalocyanine moiety. The sharp peak at $1616-1621\text{cm}^{-1}$ is assigned to C=N of imine group and the peaks in the $1365-1388\text{cm}^{-1}$ is due to C-N aromatic stretching frequency. The peaks at $1315 - 1327\text{cm}^{-1}$ are due to C - H symmetric bending. All other remaining peaks appeared around $848 - 881\text{cm}^{-1}$, 1077 , 1088 , 1125cm^{-1} are attributed to the various skeletal vibrations of phthalocyanine ring [19]

Magnetic Susceptibility

The magnetic susceptibility measurements for octa-1-(3-furyl) methanimine phthalocyanines of copper, cobalt, are consistent with paramagnetic nature and nickel, zinc an diamagnetic nature. A summary of the magnetic properties over the range of applied magnetic field strength 2.20-4.45 KGauss are reported in Table. I. The values reported in the table are the average of the three independent determinations. Magnetic susceptibility measurements on Cu-furyllmPcO and Co-furyllmPcO are in agreement with paramagnetic nature of the complexes and that of Ni-furyllmPcO and Zn-furyllmPcO are diamagnetic. The observed magnetic moment values for Cu-furyllmPcO and Co-furyllmPcO are higher than expected for $S = \frac{1}{2}$ spin state but less than their corresponding unsubstituted counterparts. Also, the μ_{eff} values of the complexes are found to

vary with the strength of the applied magnetic field and this is due to intermolecular cooperative effect [20].

The orbital contributions is found to be higher at lower magnetic field than that of higher one evidenced by higher μ_{eff} values at lower field strength and it may be accounted for the contribution of magnetic anisotropy of the strong phthalocyanines π electronic current as reported in the literature [21].

The observed higher values of magnetic moment, at lower magnetic field strength may be due to the intermolecular magnetic interaction coupled with strong π -electron current of phthalocyanine molecule. Crystallographic studies [22] on phthalocyanines revealed that, during the process of stacking, each phthalocyanine molecule is stacked to the neighbouring molecule along the b-axis contribute either a -N-atom at a distance of 3.38\AA one above and one below about the central metal atom as shown in Fig (2a) or may be placed one above the other with the separation of two molecules by a distance of 3.38\AA as represented in Fig.2(b). This minimum interplanar distance seems to provide a suitable pathway for spin-spin coupling interaction between adjacent metal atoms either directly or super exchange through the N atoms of the molecule. The extent of super exchange interaction between the molecules depends primarily on angle of inclination of the plane of the molecules, type of the metal atom, nature of the substituent and metal-metal distance. It seems, the exchange interaction coupled with orbital contribution is the cause for the observed higher magnetic moments for Cu-furylImPcO and Co-furylImPcO at the lower magnetic field strength. But at higher magnetic fields, it seems molecules suffer tumbling, resulting in decrease in intermolecular distance which in turn favour an effective intermolecular interaction.

The crystallographic studies revealed that the metal phthalocyanines of Cu, Co, Ni and Zn has square planar structure with D_{4h} symmetry and are isomorphous [19]. The molecular plane is approximately normal to a-b plane and molecules are stacked along the short b-axis. The molecular planes are inclined to the a-c axis at an angle of 45° . Thus the complexes stacked in column with N-atoms above and below on every metal atom (Fig.3) and hence the nearest neighbouring molecule along the b-axis contributes N-atom at the interplanar distance 3.4\AA [16].

Powder XRD

The X-ray diffraction spectra of M-furylImPcO's are taken through a range of 2θ angles $6-70^\circ$ showed are not identical peaks. Table.2. Two peaks were observed with one sharp at lower angle with maximum intensity and the other at higher angle with higher intensity. The inter planar spacings on these angle gave the following values. Co-furylImPcO $3.45, 37.86\text{\AA}$; Cu-furylImPcO $3.48, 39.82\text{\AA}$; Ni-furylImPcO $3.46, 38.21\text{\AA}$ and Zn-furylImPcO $3.21, 45.27\text{\AA}$ clearly indicating the crystalline nature of the complex. The observed patterns are very much similar to unsubstituted parent phthalocyanines except the broadening of the peaks in the case of title complexes with diffused intensity. The broadening may be due to the presence of substituents and which seems to play an important role in the stacking of the metal phthalocyanine derivatives. X-ray diffraction patterns are used only to explain crystallinity qualitatively.

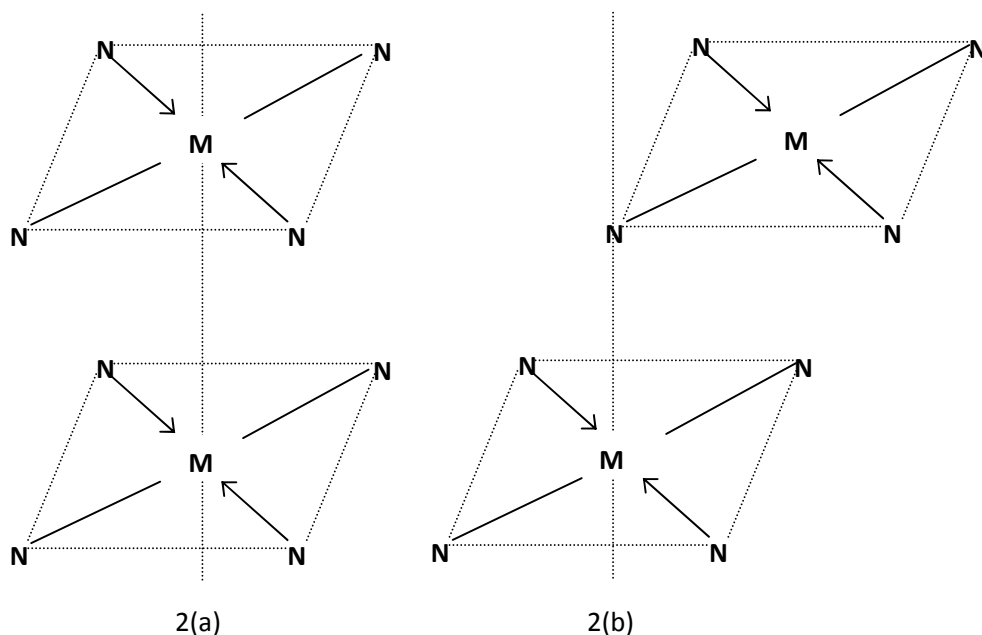


Figure 3. Probable molecular stacking of metal phthalocyanines M = Cu, Co, Ni and Zn, N = central nitrogen atoms of the phthalocyanine structure.

Thermogravimetric and Kinetic Studies.

Thermogravimetric analytical data of octa-1-(3-furyl) methanimine phthalocyanine complexes both in air and nitrogen atmosphere are summarized in the Table 3 and 4. It is observed that the decomposition of the above complexes occurs generally in two steps, revealed that the initial weight loss of 2-3% corresponding to moisture. The first step degradation in air, which takes place in the temperature range of 250-350°C, may be accounted for the loss of eight substituted imine groups. The major weight loss is observed for all the complexes in the second step in the temperature ranges of 350-600°C corresponds to the oxidative degradation of remaining phthalocyanine moiety. Analysis of the final charred residue corresponds to the corresponding metal oxides [23]. The thermal decomposition of imino substituted metal phthalocyanine complexes in the nitrogen atmosphere appears to be very slow. For Co-furylImPcO, 69% of the complex was found to be decomposed at 700°C. For Cu-furylImPcO, Ni-furylImPcO about 54%, 51% and 40% loss of the mass was observed for Zn-furylImPcO. Above trend confirms the relative stability of these complexes in the order Co-furylImPcO > Cu-furylImPcO > Ni-furylImPcO > Zn-furylImPcO. DTA results revealed that all degradation steps are exothermic in nature. Kinetic and thermodynamic parameters of the title complexes have been evaluated by Boride's method [24].

The activation energies (E_0) for the loss of periphery furfural imino substituent compound lies in the range of 0.78 to 4.87KJ/mole. It may be expected that as soon as the periphery substituents are expelled, the rupture of the main phthalocyanines ring takes place. Exothermic behavior of the degradation clearly indicated by their negative entropies. The changes in entropies ΔS are negative for little complexes and varies from -169.25 to -173.74 KJ. It is clear

from the values of entropies for the decomposition that the removal of functional groups are associated with more negative entropies

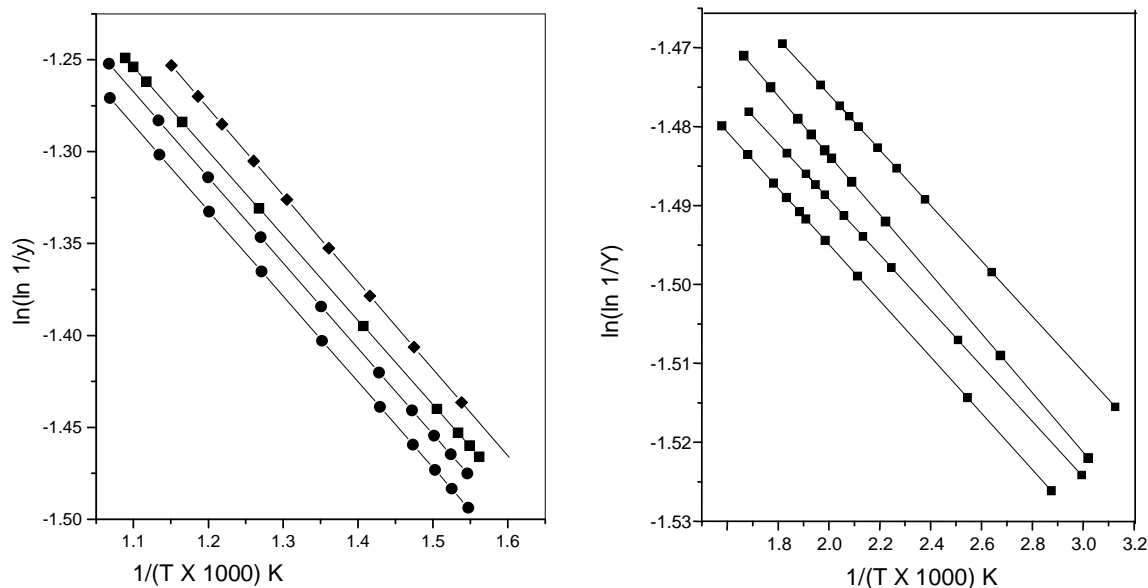


Fig.3. Plots of $\ln(\ln 1/y)$ versus $1/T$ for thermal degradation of (●●●●) CufurylImPcO, (●●●●) CofurylImPcO, (◆◆◆◆) NifurylImPcO and (■ ■ ■ ■) ZnfurylImPcOA in air.

Antifungal activity

It was found that each of the complexes inhibited radial growth of *Aspergillus niger*. After the 3rd day of inoculation, the fungus exhibited minimum growth. The inhibition was greatest for Ni complex compared to other complexes; the rate of inhibition was of the order Table 5. Ni-furylImPcO > Zn-furylImPcO > Co-furylImPcO > Cu-furylImPcO.

An interesting observation made during the investigation is the change in the color of fungus sporulation. *Aspergillus niger* is known for its black color. However in the presence of metal complexes the fungus is pale brown sporulation. It is confirmed by the parallel experiment with and without addition of 2ml DMSO in the medium that the change in colour of the fungus was not due to the presence of DMSO in the medium. The color change of the fungus may be due to the effect of metal complexes on spore pigmentation [15].

CONCLUSION

A simple and convenient method has been optimized for the synthesis of pigments 1, 3, 8, 10, 15, 17, 22, 24-octa-1-(3-furyl) methanimine phthalocyanines of Cu(II), Co(II), Ni(II) and Zn(II). Magnetic susceptibility studies revealed the paramagnetic behavior of Cu(II) and Co(II) octa-1-(3-furyl) methanimine phthalocyanine derivatives and the variation of magnetic moments with magnetic field indicated the presence of intermolecular co-operative effect. Thermogravimetric

analysis of the complexes in an inert atmosphere revealed the stability in the order Cu-furylImPcO > Co-furylImPcO > Ni-furylImPcO > Zn-furylImPcO.

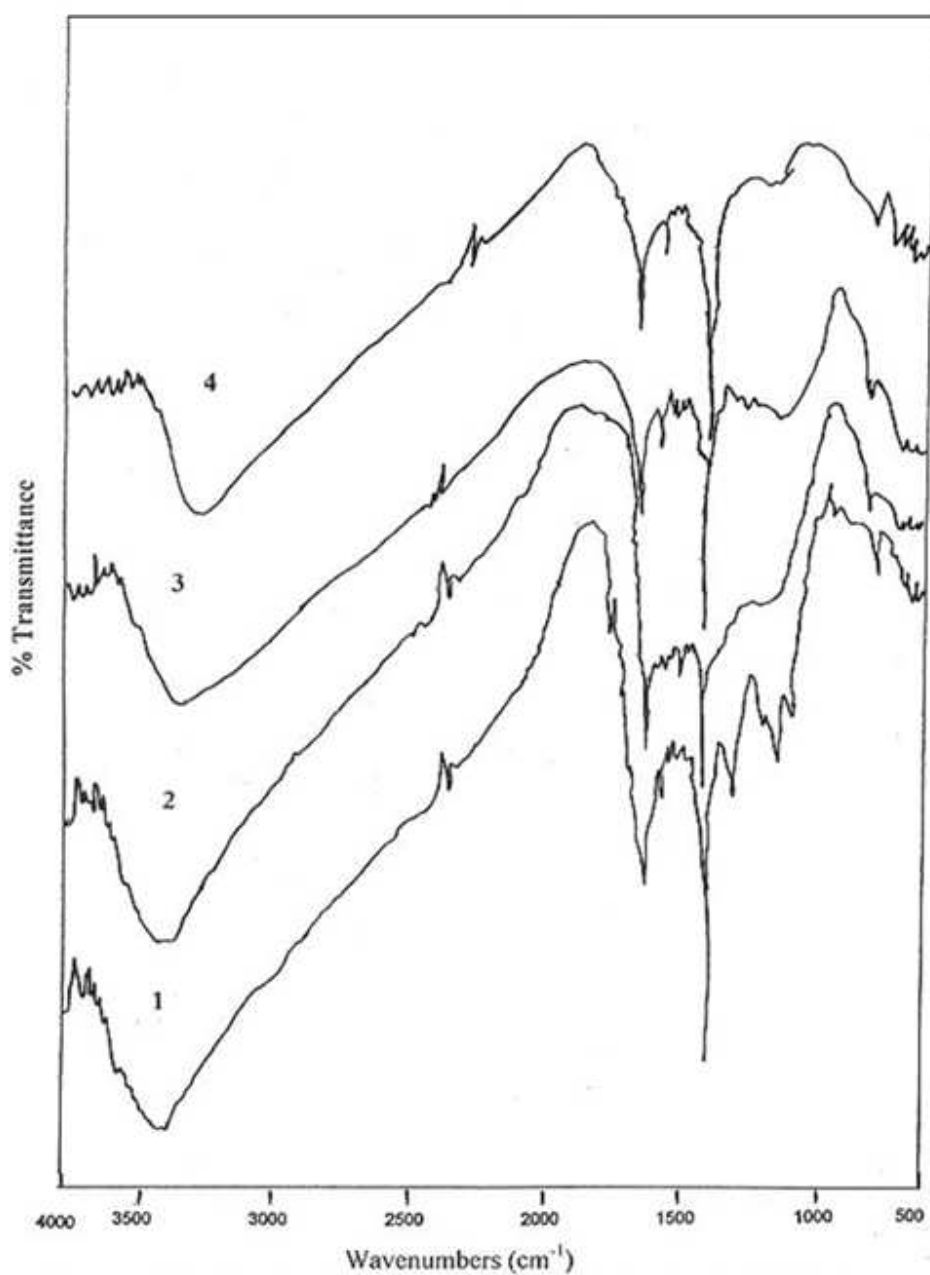


Fig. 2 IR absorption spectra of (1) Cu-FurylImPcO (2) Co-FurylImPcO
(3) Ni-FurylImPcO (4) Zn-FurylImPcO

Table.1: Elemental analysis and magnetic susceptibility data of metal (II) octa-1-(3-furyl) methanimine phthalocyanines.

Complex (Yield) Colour	Empirical formulae. (Formula weight)	Field strength KGauss	Magnetic susceptibility ($\chi_m \times 10^{-6}$ cgs units)	Magnetic moments μ_{eff} (B.M)	Elemental analysis (%) found (calcd)
CofuryllmPcO (80%) Dark green	$C_{72}H_{40}O_8N_{16}Co$ (1316.12)	2.20	+2766.38	2.61	C, 65.70; H, 3.04, N, 17.03; Co, 4.48. C, 65.68; H, 3.01, N, 17.01; Co, 4.48.
		2.66	+2613.56	2.54	
		3.10	+2488.59	2.46	
		3.58	+2319.16	2.39	
		4.01	+2121.04	2.28	
CufuryllmPcO (78%) Dark green	$C_{72}H_{40}O_8N_{16}Cu$ (1320.73)	2.20	+3222.47	2.82	C, 65.47; H, 3.03, N, 16.97; Cu, 4.81. C, 65.45; H, 3.01, N, 16.95; Cu, 4.81
		2.66	+3007.74	2.72	
		3.10	+2842.58	2.64	
		3.58	+2706.28	2.58	
		4.01	+2500.38	2.48	
NifuryllmPcO (75%) Dark green	$C_{72}H_{40}O_8N_{16}Ni$ (1315.88)	2.66	-619.40	---	C, 65.71; H, 3.04, N, 17.03; Ni, 4.48. C, 65.69; H, 3.02, N, 17.01; Ni, 3.89.
ZnfuryllmPcO (70%) Green with brown ting	$C_{72}H_{40}O_8N_{16}Zn$ (1322.58)	2.66	-1059.00	---	C, 65.38; H, 3.02, N, 16.95; Zn, 4.94. C, 65.35; H, 3.00, N, 16.93; Zn, 4.94.

Table.2: Electronic, IR and powder XRD data of metal (II) -octa-1-(3-furyl) methanimine phthalocyanines.

Complex	UV-visible absorption λ nm (log ϵ)	IR-Spectral cm^{-1}	Powder. XRD data 2θ angle ($d \text{ \AA}$)	Relative intensity (%)
Co-furyllmPcO	209 (4.96)	457, 607, 1078, 1124, 1171, 1295, 1395, 1636, 2355, 3426	38.48 (2.97)	100.00
	276 (4.91)		37.86 (3.45)	79.65
	380 (5.54)		26.93 (3.12)	54.72
	610 (5.16)		44.67 (2.95)	36.06
	743 (5.25)			
Cu-furyllmPcO	226 (5.49)	752, 902, 1088, 1347, 1533, 1610, 2360, 2924, 3329	38.87 (2.84)	100.00
	275 (5.50)		25.56 (3.20)	79.15
	379 (5.51)		35.68 (3.36)	48.81
	601 (5.09)		39.82 (3.48)	39.98
	740 (5.26)			
Ni-furyllmPcO	209 (4.89)	674, 1134, 1512, 1548, 1559, 1647, 2360, 3493	33.83 (2.92)	100.00
	267 (4.97)		29.76 (2.45)	76.79
	390 (5.53)		41.84 (3.02)	55.40
	605 (5.22)		38.21 (3.46)	38.54
	740 (5.30)			
Zn-furyllmPcO	207 (4.90)	617, 757, 1124, 1295, 1388, 1631, 1734, 1360	32.45 (2.35)	100.00
	266 (4.91)		20.86 (2.75)	82.16
	374 (5.55)		48.67 (3.12)	57.64
	615 (5.21)		45.27 (3.24)	44.52
	719 (5.21)			

Table-3: TGA data of metal (II) -octa-1-(3-furyl) methanimine phthalocyanines

Complex	Temperature of Decomposition °C	Mass Loss		Probable mode of Fragmentation
		(%) Found	(%) Calcd	
CufurylImPcO	250-350	30.74	31.02	8-Imino groups Pc moiety
	350-600	65.46	65.81	
CofurylImPcO	250-350	30.75	31.41	8-Imino groups Pc moiety
	350-600	65.94	65.04	
NifurylImPcO	250-350	30.72	31.74	8-Imino groups Pc moiety
	350-600	65.94	65.02	
ZnfurylImPcO	250-350	30.64	31.85	8-Imino groups Pc moiety
	350-600	65.61	65.04	

Table-4: Kinetics and Thermodynamic parameters of metal (II) - octa-1-(3-furyl) methanimine phthalocyanines in air and nitrogen atmosphere.

Compounds	Activation energy Eo KJ/mole	Frequency Factor lnA min ⁻¹	ΔH KJ/mole	ΔS J/K	ΔG KJ/mole
Cu-furylImPcO I	4.87 (0.52)	6.18 (2.35)	2.47 (-1.94)	-173.74 (-175.54)	40.47 (42.36)
	3.58 (1.40)	5.58 (6.21)	-0.76 (-2.32)	-167.54 (-173.54)	83.35 (85.24)
Co-furylImPcO I	0.78 (0.46)	2.54 (2.68)	-1.86 (-1.65)	-169.25 (-175.30)	40.87 (42.01)
	3.89 (1.35)	5.65 (4.12)	3.21 (-2.67)	-163.01 (-174.21)	83.36 (84.44)
Ni-furylImPcO I	1.82 (1.45)	4.88 (4.48)	-0.29 (-0.57)	171.82 (-171.82)	42.71 (42.43)
	3.86 (1.59)	5.94 (4.98)	-0.49 (-2.59)	-169.28 (-173.28)	83.64 (84.01)
Zn-furylImPcO I	1.34 (0.45)	4.25 (2.58)	4.25 (-1.71)	-172.79 (-176.58)	42.75 (42.34)
	7.67 (1.43)	7.41 (4.15)	3.25 (-2.79)	-153.78 (-2.79)	78.62 (78.58)

(I and II corresponds to the I and II stage of degradation and the values in the parenthesis are for nitrogen atmosphere)

Table-5: Antifungal data metal(II) - octa-1-(3-furyl) methanimine phthalocyanines.

Samples	Concentration in Ppm	Radial growth of Aspergillus niger (in cm) in respective incubation period		
		3 rd day	5 th day	7 th day
Control (DMSO)	-	1.6	2.4	3.7
Ni-furylImPcO	100	0.9	1.5	2.4
Co-furylImPcO	100	1.1	1.6	2.8
Cu-furylImPcO	100	1.4	1.9	2.9
Zn-furylImPcO	100	1.0	1.4	2.5

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