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Synthesis, Characterization and corrosion Inhibition Studies on Mn (II) and Co (II) Complexes Derived from 1-{(Z)-[(2-hydroxyphenyl) imino]methyl}naphthalen-2-ol in 1M HCl Solution

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ABSTRACT

Schiff base derived from the reaction of 2-amino phenol and 2-hydroxy-1-naphthaldehyde and its Co (II), and Mn (II) complexes have been synthesized and characterized by solubility test, melting point/ decomposition temperatures, molar conductance, IR and magnetic susceptibility. The number of ligands coordinated to the metal ion was determined using Job's method of continuous variation. Their molar conductance values indicate that all the complexes are non-electrolytes. Magnetic moment values of the complexes showed that both Mn (II) and Co (II) are paramagnetic. The spectroscopic data of metal complexes indicated that the metal ions are complexed with azomethine nitrogen and deprotonated oxygen atom. Corrosion inhibition of the schiff base and Mn (II) and Co (II) complexes were evaluated using the weight loss method in a 0.1MHCl solution for copper metal. The inhibition efficiency increased with increasing inhibitors concentration. The negative values of Gibb's free energy of adsorption (ΔGads) confirmed the spontaneity and physical adsorption of the inhibition process which is inconsistent with Langmuir adsorption isotherm.

1. Introduction

Deterioration of the material due to its environmental reaction is called corrosion which occurs in the presence of oxygen and humidity on the metal surfaces. Corrosion is a redox system in which anode oxidation and cathode reduction take place (Charles *et al.,* 2020). The use of corrosion inhibitors is one of the most practical and cost-effective methods of corrosion safety available (Gupta *et al.,* 2016). This is because of their cost-effectiveness and ease of implementation (Pandey *et al.,* 2017). By adsorbing on the surface, they inhibit the metallic surface, creating a protective barrier between metal and electrolyte (Kannam *et al.,* 2016).

Several factors, such as the molecular size of the inhibitor, the type of the substituent, the nature of the metal and the electrolyte, influence the rate of adsorption of these inhibitors to the surface (Verma *et al.,* 2016).

In different acidic media at room temperatures and elevated temperatures, the different ligands of the metal complexes Co (II), Ni (II), Zn (II) and Sn (II) have shown good corrosion inhibition properties (Mishra *et al.,* 2015; Singh *et al.,* 2011). Transition metal complexes derived from Schiff's base are more effective inhibitors due to their greater size and compactness, and metal-organic blends also have synergistic action (Amar *et al.,* 2007).

The aim of this research was to synthesize and characterized Co (II) and Mn (II) complexes of 1-{(Z)-[(2-hydroxyphenyl)imino]methyl}naphthalen-2-ol. The ability of this Schiff base and its metal (II) complexes to inhibit the corrosion of copper in 1M HCl solution using the weight loss method was then investigated.

2. Literature Review

Singh *et al.,* (2015) reported the synthesis of Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) complexes with 2-amino-benzoic acid (phenyl-pyridin-2-yl-methylene)- hydrazide. The complexes were characterized by different physicochemical and spectral studies viz. molar conductance, magnetic susceptibility measurements, electronic, IR and NMR spectra. The molecular structures of the ligand Habph and its Mn(II), Ni(II), Cu(II), Zn(II) complexes were further confirmed by single-crystal X-ray diffraction technique. The results of electrochemical impedance spectroscopy (EIS) and adsorption behavior of the ligand and metal complexes showed appreciable corrosion inhibition efficiency for mild steel in a 1 M HCl medium. The metal complexes show a better inhibition effect than the ligand.

Anticorrosion properties in oilfield produced water of the *N*′-phenylbenzohydrazide and its metal complex derivatives by experimental studies and quantum chemical calculations as well as investigation of the relationship between experimental and theoretical study results through a correlation study was reported by Mahross *et al.,* 2018. The molecular structures of *N*′-phenylbenzohydrazide and its metal complexes (Cu, Mn, Co) were characterized using UV–Vis and FT–IR techniques. The electrochemical polarization measurements, electrochemical impedance spectroscopy (EIS) and scanning electron microscopy techniques were used experimentally in the discovery of corrosion inhibition properties of the metal complexes in oilfield produced water. The results showed that the metal complexes acted as anodic-type inhibitors. The experimental inhibition activities of studied metal complexes were found to be highly correlated with calculated some parameters, ET, E_{LUMO}, E_{HOMO} and E_{Gap}.

Devika *et al.,* (2019) reported the corrosion behavior of three metal [Co(II), Ni(II), and Fe(III) complexes of antipyrine based azo dye ligand {5-[(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-yl)diazenyl]-6-hydroxy-1,4-dimethyl-2-oxo-1,2-dihydropyradine-3-carbonitrile} for soft-cast steel in 1M HCl acid solution using experimental and theoretical methods. The study revealed that the ligand and their metal complexes show good inhibition efficiency, with Ni complex showing significant inhibition efficiency at an optimum concentration of 25mg/L.

El-tabash et al. (2020) reported the effect of Bis-phenanthroline chloro copper (II) chloride di-para-aminobenzoic acid tetrahydrate complex $[Cu(Phen)_2Cl]Cl\ (pABz)_2{\cdot}4H_2O$, CuPAB, on the corrosion behavior of carbon steel in 0.5 M sulfuric acid solutions by electrochemical impedance spectroscopy (EIS), and potentiodynamic polarization techniques. Theoretical fitting of different adsorption isotherms such as Langmuir, Flory–Huggins, and the kinetic–thermodynamic models has been tested. The adsorption isotherm parameters indicate that the adsorption behavior of complex on the metal surface is not ideal. The high binding constant of CuPAB showed a stronger interaction between the metal surface and the complex. The obtained data revealed that CuPAB complex has remarkable inhibiting effects on the corrosion of steel in 0.5 M H_2SO_4.

3. Methodology

3.1 Materials

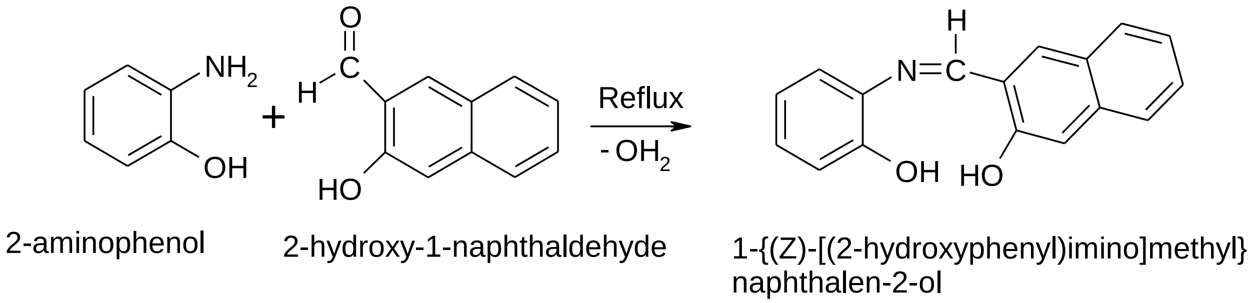
2-hydroxy-1-napthaldehyde was of analytical grade and procured from Sigma-Aldrich. Other chemicals and reagents were of analytical grade and used without further purification. The metal salts: cobalt (II) and manganese (II) chlorides were obtained from a chemical store, Bayero University, Kano.

3.2 Instruments

All weighing was carried out on electrical balance model H3OAR, melting point and decomposition temperatures were recorded using Gallenkamp melting point apparatus. Conductivity measurements were carried out using Jenway conductivity meter model 4010 in DMSO solvent. The infrared spectral analyses were recorded using SHIMADZU FTIR 8400S model. Magnetic susceptibility measurements were conducted and recorded using mk1 Sherwood magnetic susceptibility meter.

3.3 Preparation of Schiff base

0.01mol 2-aminophenol in 25cm^3 ethanol was slowly added to a solution of 0.01mole of 2 – hydroxy –1– naphthaldehyde in 25cm^{3}ethanol. The mixture was refluxed for two hours. The Schiff base that formed was filtered and washed with ethanol and dried over phosphorus pentoxide (Aliyu and Sani 2011).



Scheme1: Preparation of Schiff Base

3.4 Preparation of Metal (II) Complexes

A general method has been used for the preparation of complexes using the reaction of metal salts and the corresponding Schiff-base in a molar ratio (1:2). An ethanolic solution of ligand (0.04 mol) and metal (II) chlorides (0.02 mol) both were mixed gently and refluxed for 4 h. The volume of the resulting solution was concentrated by evaporating the solvent. The reaction mixture was cooled to room temperature which solidified on cooling. The solid thus obtained was filtered, washed thoroughly with ethanol to apparent dryness and dried over phosphorus pentoxide (Aliyu and Sani, 2011).

3.6 Determination of Number of Coordinated Ligand

The ligand to the metal ratio in the complexes was determined using the continuous variations method (Job's method) (Agelici, 1971). 0.001M of the ligand and the metal (II) Chlorides in DMSO were prepared. The following ligand to metal salt ratio (ml); 1:15, 3:13, 5:11, 7:9, 9:7, 11:5, 13:3 and 15:1 were taken from ligand solution and each of the metal (II) chloride respectively. A total volume of 16ml was maintained (in the order) through the process and the mole fraction of the ligand was calculated in each mixture. The solution of the metal (II) chlorides were scan (as blank) to find the wavelength λ_{max} for the particular metal ion before taking the absorbance values. The absorbance values were extrapolated against the mole fraction of the ligand and the number of coordinated ligands was determined using the relation below

$$n = \frac{Xi}{1-Xi}$$

Where n is the number of coordinating ligand and Xi is the mole fraction at maximum absorbance.

3.7 Preparation of Coupons

The sample (copper), purchased from yan-gwangwan along K/marusa road, Katsina, was mechanically pressed and cut. The specimen's size was $1.0 \times 1.0 \times cm$ and is used in all experiments with pre-treatments such as cleaning, polishing, and degreasing. Surface cleaning is done by immersing the specimen in ethanol; polishing is done by using various grades of emery paper and then finally degreasing by acetone. The specimen was stored in dessicator (Obot *et al.,* 2011).

3.8 Preparation of Solution

Acidic solution containing 1.0 moldm^{-3} HCl was prepared by dissolving 8.60 cm^3 of concentrated HCl in distilled water and diluting the resulting solution to volume in 100 cm^3 volumetric flasks (Maibulangu *et al.,* 2017). The concentration of the inhibitors (Schiff base, Mn (II) complex and Co (II) complex) used for the study were 0.02, 0.04, 0.06, 0.08 and 0.10 gL^{-1}.

3.9 Gravimetric Experiment

The weight loss experiments were performed using standard methods described by Gupta *et al.,* 2016. The corrosion rate was calculated using the equation:

$$C_R = \frac{W}{At}$$

Where W is the mean value of weight loss of three parallel metal coupons, A is the total area of a coupon, and t is the immersion time (2hr). The percentage of inhibition efficiency (η%) and surface coverage (θ) was evaluated corrosion rates using the following relations:

$$\eta\% = \frac{C_R - C_{R(i)}}{C_R} \times 100$$

$$\theta = \frac{C_R - C_{R(i)}}{C_R}$$

Where C_R and $C_{R(i)}$ are the corrosion rate (mgcm^{-2}h^{-1}) of copper coupons in the absent and presence of the inhibitors, respectively.

4. Results and Discussion

4.1 Results

Table 1: Physical Properties of the Ligand and its Corresponding Metal (II) Complexes

Ligand/complexes	**Colour**	**Yield (%)**	**Melting point (^{0}C)**	**Decomposition temperature (^{0}C)**
Ligand	Orange	82	186	_
[MnL_2]	Cream	56		240
[CoL_2]	Brown	75		280

L=Ligand

Table 2: Solubility of the Ligand and its Metal (II) Complexes in some common solvents

Ligand/complex	**Distilled water**	**Methanol**	**Ethanol**	**DMSO**	**DMF**	**$CHCl_3$**	**n-hexane**	**CCl_4**
Ligand	IS	S	S	S	S	S	SS	IS
[MnL_2]	IS	S	S	S	S	SS	SS	IS
[CoL_2]	IS	S	S	S	S	SS	SS	IS

Key: S=Soluble, SS=Slightly Soluble, IS=Insoluble, L=Ligand

Table 3: The Infrared Frequencies of the Ligand and its Metal (II) Complexes.

Ligand/complex	**ν (OH) cm^{-1}**	**ν (H_2O) cm^{-1}**	**ν (C=N) cm^{-1}**	**ν (C-O) cm^{-1}**	**ν (M-O) cm^{-1}**	**ν (M-N) cm^{-1}**
Ligand	3119		1618	1356	_	_
[MnL_2]		3440	1627	1428	528	393
[CoL_2]		3446	1628	1438	538	406

L=Ligand

Table 4: Molar Conductance of the Complexes in 10^{-3}M DMSO solution

Complex	**Concentration in moldm^{-3}**	**Specific conductance Ohm^{-1} cm^{-1}**	**Molar conductance Ohm^{-1} cm^{2}mol^{-1}**
[MnL_2]	1×10^{-3}	8.40×10^{-6}	8.40
[CoL_2]	1×10^{-3}	7.07×10^{-6}	7.07

L=Ligand

Table 5: Magnetic Susceptibility Measurement

Compound	**gram susceptibility, χ_g (erg•G^{-2} g^{-1})**	**molar magnetic susceptibility , χ_M (erg•G^{-2} mol^{-1})**	**Effective magnetic moment in Bohr Magnetons (μ_{eef})**
[MnL]	3.26×10^{-6}	1.88×10^{-3}	2.11
[CoL_2]	7.5×10^{-6}	4.37×10^{-3}	3.21

L=Ligand

Table 6: Corrosion Parameters of Inhibitors for Copper in 1M HCl by Weight Loss Measurements.

Ligand/ Metal Complex	Corrosive medium (g/L)	Weight loss	$CR = \frac{\Delta W}{At}$ $(gcm^{-2}hr^{-1})$	$I.E = \frac{W - W_i}{W} \times 100$	$\theta = \frac{W - W_i}{W}$
Blank		0.098	0.049	-	-
Ligand					
	0.02	0.077	0.039	20.4	0.204
	0.04	0.072	0.036	26.5	0.265
	0.06	0.066	0.033	32.6	0.326
	0.08	0.060	0.030	38.8	0.388
	0.10	0.050	0.025	48.9	0.489
Blank		0.103	0.052	-	-
[MnL_2]					
	0.02	0.058	0.029	44.2	0.442
	0.04	0.052	0.026	50.0	0.500
	0.06	0.048	0.024	53.8	0.538
	0.08	0.043	0.022	57.7	0.577
	0.10	0.039	0.019	63.5	0.635
Blank		0.109	0.055	-	-
[CoL_2]					
	0.02	0.055	0.028	49.1	0.491
	0.04	0.050	0.025	54.5	0.545
	0.06	0.045	0.023	58.2	0.582
	0.08	0.040	0.020	63.6	0.636
	0.10	0.036	0.018	67.3	0.673

L=Ligand

Table 7: The Values of Langmuir Adsorption Parameters of the Ligand and its Metal Complexes on the Surface of Copper in IM HCl

Inhibitor	Surface coverage (Θ) 0.02	0.04	0.06	0.08	0.10	Slope	Log Kad	R^2	ΔGad ($Kjmol^{-1}$)
Ligand	0.204	0.265	0.326	0.388	0.489	0.478	-0.174	0.958	-9.421
Mn (II)	0.442	0.500	0.538	0.577	0.635	0.789	0.001	0.997	-10.445
Co (II)	0.491	0.545	0.582	0.636	0.673	0.802	-0.018	0.998	-10.379

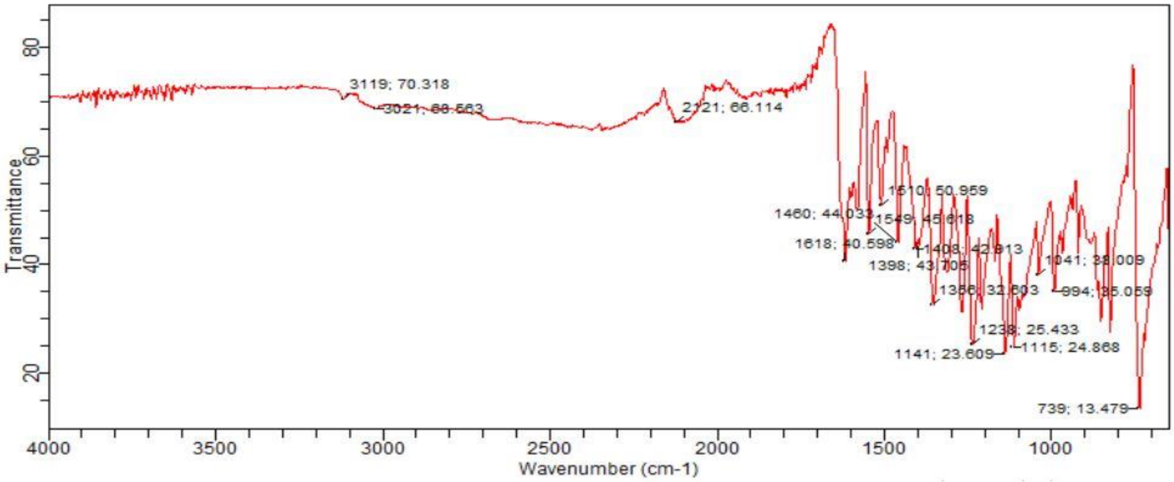


Figure 1: IR Spectrum of Ligand

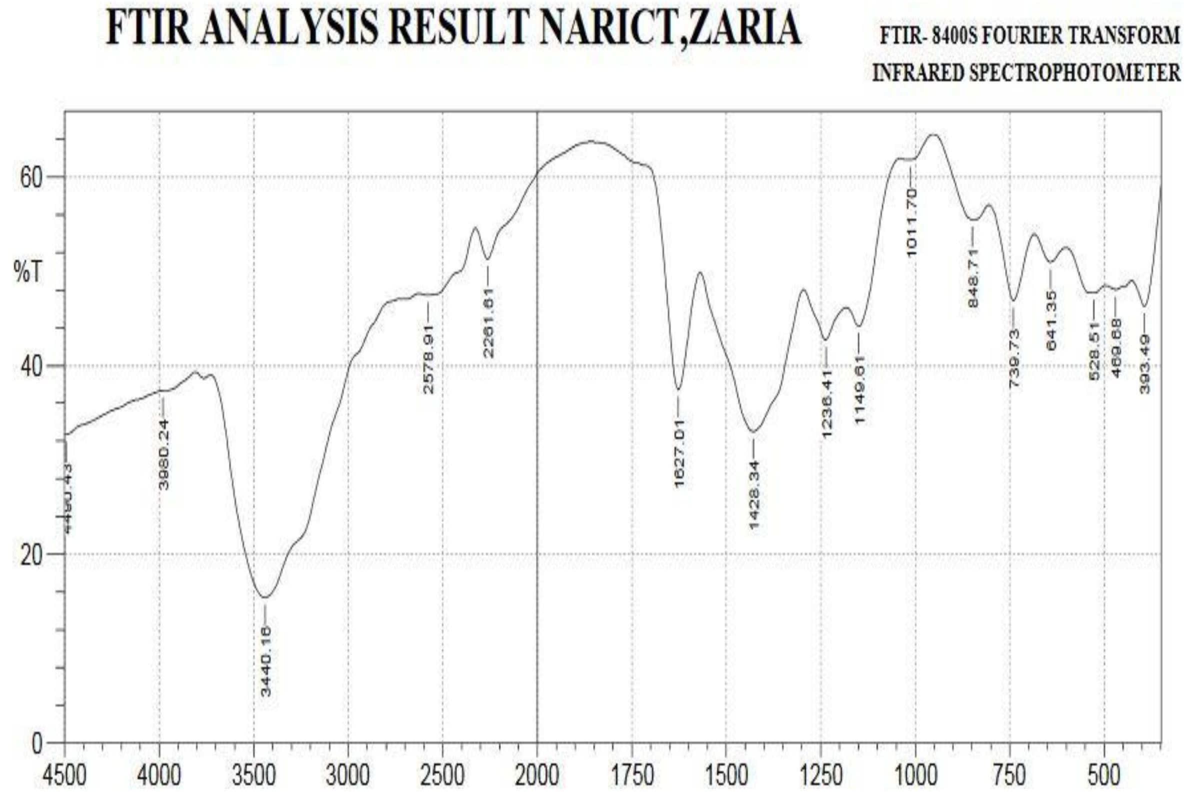


Figure 2: IR Spectrum of Mn (II) Complex

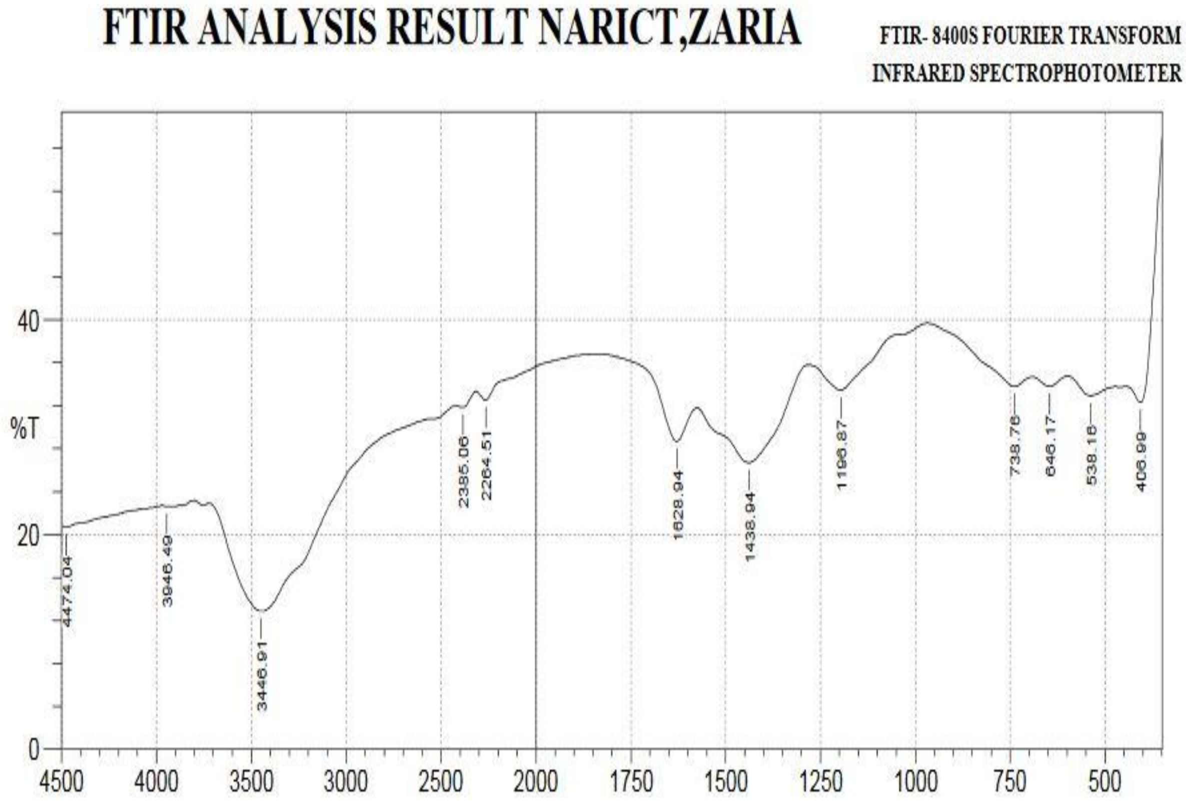


Figure 3: IR Spectrum of Co (II) Complex

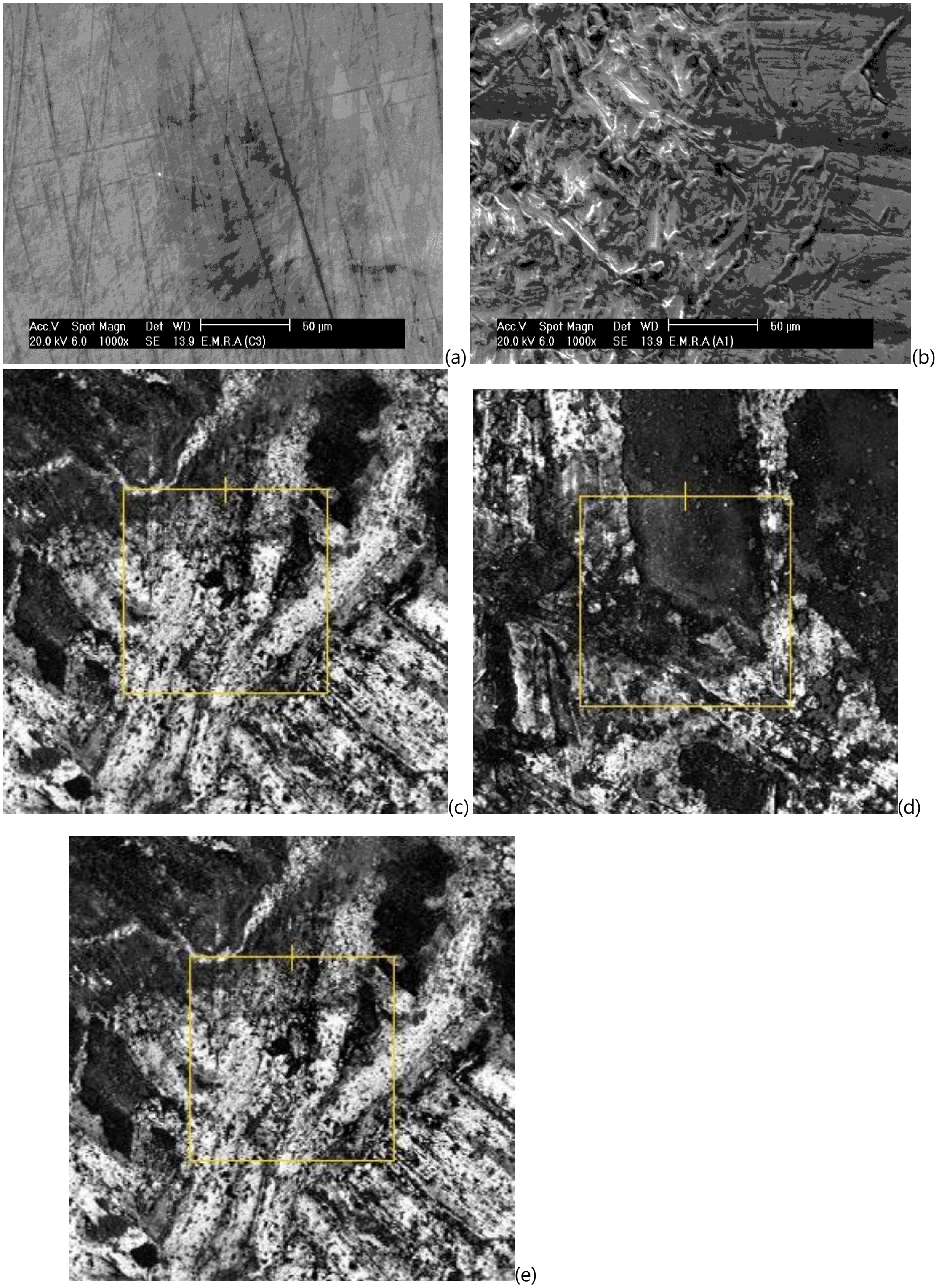


Figure 4: The SEM micrographs of (a) polish copper metal, (b) copper metal in 1M HCl (c) in the presence of 0.02 gL^{-1} ligand, (d) in the presence of 0.02 M Mn (II) complex inhibitor, (e) in the presence of 0.02 gL^{-1} Co (II) complex inhibitor

4.2 Discussion

The Schiff base derived by the condensation of 2-amino phenol and 2-hydroxy-1-naphthaldehyde was orange in colour, stable and non-hygroscopic solid at room temperature. The corresponding product was obtained in good yield (82%). The Schiff base melts at 186^0C reacts with Co and Mn (II) chlorides and formed complexes with a yield of 56-75%. The colors of Mn (II), and Co (II), complexes are cream and brown, respectively. The decomposition temperatures of these complexes were 240^oC for Mn (II) and 286°C for Co (II). The values of decomposition temperatures indicate that the complexes are stable (Aliyu *et al.,* 2013). These results are presented in Table 1.

4.2.1 Solubility Test

The result of the solubility test of Schiff base ligand and its Mn (II) and Co (II) complexes in water and common organic solvents, presented in table 2 revealed that the Schiff base and its Mn (II), and Co (II), complexes were all soluble in methanol, ethanol, DMSO and DMF. However, they were insoluble in distilled water and tetrachloromethane. Mn (II), and Co (II), complexes found to be slightly soluble in chloroform and n-hexane. The Schiff base was also found to be soluble in chloroform and slightly soluble in n-hexane. This is in agreement with results obtained by Aliyu *et al.,* 2013, Aliyu and Sani, 2011.

4.2.2 IR Spectra

The IR values of the Schiff base, Mn (II) complex and Co (II) are presented in Table 3 and their spectra in figure 1-3. The Schiff base exhibits the following bands in its IR spectrum, 3119 cm^{-1} due to ѵ (-OH) of aromatic, and 1618cm^{-1} is characteristic of the azomethine group. Coordination of azomethine ν(C=N) group to the central metal atom was observed from the shift of the frequency of the Schiff base from 1618cm^{-1} to 1627 and 1628cm^{-1} in the Mn (II) and Co (II) complexes, respectively. The appearance of a new band at 393-459 cm^{-1} was due to ν (M-N) bond (Gomathi *et al.,* 2013) and that of ν (M−O) stretching vibrations were observed at 517-538 cm^{-1} due to the involvement of oxygen atom in the coordination (Siddape and Mane, 2013). Other bands appeared in the spectra of the complexes in the region range (3396-3401cm^{-1}) which may be attributed to Ѵ(H2O) stretching vibration.

4.2.3 Molar Conductivity and Magnetic Susceptibility

The molar conductivity values for the complexes in DMSO were in the electrolytic range of 7.07-7.53 µs. It is clear from the conductivity data that the complexes present seem to be non-electrolytes. Also, the molar conductance values indicate that there is an absence of anions outside or inside the coordination sphere (Moamen *et al.,* 2013). The observed magnetic moment values for Mn (II) and Co (II) complex are 2.11 BM and 3.21 BM, respectively, suggesting a high spin complex with octahedral geometry.

4.2.4 Weight Loss Experiment

The values of percentage inhibition efficiency (ƞ), corrosion rate (CR), and surface coverage (Ɵ) obtained by weight loss method at various concentration of all compounds at 313 K are summarized in table 6.

From the results, in 1M HCl medium, the Schiff base and its Mn (II) and Co (II) showed significant corrosion inhibition for copper.

The inhibition efficiency seems to have improved with increased inhibitor concentration, and this is due to the copper surface interaction of the inhibitor molecule, allowing it to be adsorbed (Aouniti *et al.,* 2016; Nassar *et al.,* 2015)..

The inhibitor's ability to prevent the copper's corrosion ability is due to its propensity to form bonds using the lone pair of electrons present in the O atom and the π-electrons present in both the azomethine moiety and the benzene ring (Nassar *et al.,* 2015).

4.2.5 Adsorption Isotherms

Adsorption isotherms provide data on the interaction between inhibitors and metal surfaces (Hussaini *et al.,* 2020). The surface coverage values at 313 K for different concentrations have been used to describe the best isotherms that assess the mechanism of adsorption. Attempts were made to fit (Ɵ) values to different isotherms, like Langmuir, Frumkin, Freundlich and Temkin.

Langmuir: $Log\left(\frac{C}{\theta}\right) = Log\ K_{ad} - \ LogC$

Frumkin: $\frac{\theta}{1-\theta}\exp(-2f\theta\theta) = KC$

Temkin: $\exp(f\theta) = KC$

Freundlich: $Log(\theta) = Log\ K + \frac{1}{n}LogC$

Ɵ is the surface coverage, K is the adsorption-desorption equilibrium constant, C is the concentration of inhibitor and f is the factor of energetic inhomogeneity. Among the several tested adsorption isotherms, Langmuir isotherm gave the best fit with values of the regression coefficient (R^2) very close to unity. The values of the regression coefficient for Langmuir adsorption isotherm (Tables 7) were 0.958, 0.997 and 0.998 for ligand, Mn (II) complex and Co (II) complex, respectively. The negative ΔG°ads comply with the spontaneity of the adsorption system and the stability of the adsorbed layer on the copper metal surface (Mahross *et al.,* 2019). ΔGads values around -20 kJ mol-1 or lower are well known to be associated with the physisorption phenomenon where the electrostatic interaction between the charged molecule and the charged metal is assembled, whereas those around -40 kJ mol-1 or higher are associated with the phenomenon of chemisorption (Fekry and Ameer 2010).

The calculated values of ΔG^0_{ads} are given in Table 7. The value of ΔG^0_{ads} for the ligand, Mn (II) complex and Co (II) complex are -9.421KJmol^{-1}, -10.445KJmol^{-1} and -10.379KJmol^{-1} respectively, indicating that the adsorption of the inhibitors is physical adsorption (Verma *et al.,* 2015).

4.2.6 Surface Morphological Examination

The surface analysis was carried out by using Scanning Electron Microscope (SEM) for the copper surface before immersing in acidic solution, after immersing in acidic solution in the absence of inhibitors, and after immersing in acidic solution in the presence of inhibitors. The results obtained were presented as scanning electron micrographs in Figures 4a, 4b, 4c, 4d, and 4e. The SEM image of polished copper metal (blank) surface was obtained and shown in Figure 4a. The micrographs of the copper metal surface showed an aggressive attack after immersion without inhibitor in 1M HCl solution was shown in Figure 4b. In the absence of an inhibitor, the surface appeared to be strongly damaged because of some cracks and pits as a result of exposure to the corrosive solution. In the presence of the inhibitor, the SEM micrograph shows the formation of a thick film copper metal surface and reduction of surface roughness compared to the micrograph of copper metal without inhibitor, and the result is presented in figure 4c, 4d and 4e. Therefore, it is understood that the inhibitor molecules adsorbed on the copper metal surface, forming an efficient protective layer. This formed layer protects the copper surface against the corrosion process in 1M HCl.

5. Conclusion

The present work describes the synthesis and characterization of Co (II) and Mn (II) complexes of 1-{(Z)-[(2-hydroxyphenyl)imino]methyl}naphthalen-2-ol. The ability of this Schiff base and its metal (II) complexes to inhibit the corrosion of copper in 1M HCl solution using the weight loss method was then investigated. The results showed that corrosion rate increases with increasing inhibitors concentration. Also, corrosion rate was found to be in the order: Ni (II) > Co (II) > Ligand. The adsorption of studied inhibitors on the copper metal surface obeys Langmuir adsorption isotherm. The values of ΔG° obtained show that the adsorption of all inhibitors was spontaneous and physisorption. The surface morphology of copper was analyzed by the SEM method. The SEM images show that inhibitors adsorb to the copper metal surface.

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Conflicts of Interest: The authors declare no conflict of interest of any form.

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