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Synthesis, Characterization and Corrosion Inhibition Study of New Heterocyclic Compounds and Schiff Base with [Co (II), Ni (II), Cu (II) and Hg (II)] Complexes

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ABSTRACT

In the present research, 4-aminoacetophenon reaction with benzoin in ethanol to procure compound [1]. Compound [1] was reacted with 4-aminobenzenesulfonamide and few drops from (glacial CH_3COOH) in EtOH to yield compound [2]. Compound [2] reaction with 2-mercaptobenzoic acid or sodium azide or various anhydrides to give Thiazine [3], tetrazole [4] and 1,3- oxazepine derivatives [5,6] respectively.
The structure of synthesized compounds identified by spectral data (FT-IR), (^{1}H-NMR) spectra and analysis (CHN-S). The Schiff base ligand and synthesis some transition metals complexes [Co (II), Ni (II), Cu (II) and Hg (II) of this ligand were described via FTIR, UV-Visible Spectroscopy and the elemental analysis (CHN-S). in our coclusion we found The corrosion inhibition study of Thiazine [3] tetrazole [4] 1,3- oxazepine[5] ligand and their complexes on mild steel in (0.1M) hydrochloric acid were researched by weight loss .

Keywords: Schiff base4 ,-aminoacetophenon thiazine complexes.

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INTRODUCTION

Schiff bases as well as their metal complexes which have commit applications in medicine as antimicrobial antioxidant and anti-inflammatory, industrial application as corrosion inhibitors [1-5].
Many organic compounds including π-electron either in triple bond or conjugated double bond and hetero-atoms: oxygen, nitrogen sulphur and phosphorou were studied as metal corrosion inhibitors [6,7]. Among these, several tetrazolethiazine and oxazepine were reported as inhibitors of corrosion and found to have good corrosion inhibition effect [8-10].
The efficiency of inhibitors raise in arrange of O < N < S< P it rely on existing electron density hold out around hetero atoms the number of the active centers of the adsorption in a molecule molecular size, their charge densities, adsorption mode and metallic complexes' formation [11-14] .The Schiff base ligands' capability of forming the stable complexes that are closely packed in the domain of the coordination of the metal ion presents another compounds' class for the inhibition of corrosion. The Schiff bases are adsorbed on the surfaces of the metals, result from attendance of >C=N– groups. Such behavior of adsorption conduct to spontaneously forming a monolayer that covers the surface of the metal, due to acting as sufficient inhibitor of corrosion [15-17].

Experimental

Materials: All of the chemicals have been supplied from BDH and CDH.
Instrumentation: FT-IR-Spectra recorded on a Shimadzu – 8400s in the range between (400cm^{-1} and 4,000cm^{-1}) using KBr disk. ^{1}H-NMR spectra were analyzed and characterized. The C.H.N.S were performed on an EuroEA Elemental.

Preparation of compounds

Synthesis of 1-(4-(2-hydroxy-1,2-diphenylethylideneamino) phenyl) ethanone [1] [18]

In a round bottom flask benzoin (0.5 g 0.002 mol) in (15 ml) ethanol and 4-aminoacetophenon (0.3 g0.002mol) in (20 ml) EtOH added three drops of glacial (CH_3COOH) refluxed the mixture for (8hr.) then filtration and recrystallized from the hot EtOH and dried by anhydrous calcium chloride to produce orange precipitate yield (82%) M.P.=153-155C^0 .

Synthesis of 4-amino-N-(1-(4-(2-hydroxy-1,2-diphenylethylideneamino) phenyl) ethylidene) benzenesulfonamide [2][18]

(0.95g 0.003 mol) of compound [1] dissolved in (30 ml) of the ethanol added (3drops) from glacial (CH_3COOH) and refluxing with (0.6g 0.003mol) of 4-aminobenzenesulfonamide for (8hrs.) a brown solution has been acquired. Schiff base Ligand (compound [2]) has been seclude after the mixture volume has been reduced to 50% by evaporation and re-crystallized with the hot (EtOH) and dried over the anhydrous $CaCl_2$ yield (75%) M.P.=180-182C^0

Synthesis of compound 3-(4-aminophenylsulfonyl)-2-(4-(2-(hydroxyl (phenyl) methyl)-4-oxo-2phenyl-2H-benzo[e][1,3]thiazin-3(4 H)-yl)phenyl)-2-methyl-2 H-benzo[e][1,3]thiazin-4(3H)-one [3][19]

(0.3gm0.002mol.) of 2-mercaptobenzoic acid in (30ml) of dry benzene ` added slowly to Schiff base (0.48 gm 0.001mol.). This addition continued for approximately (5 min) with the continuous three-hour stirring. Followed by the refluxing on steam bath for approximately (20 h.). The excess solvent has been evaporated and residue has been treated with the $NaHCO_3$, filtered then re-crystallized in dioxin. yield (72%) M.P.=199-201C^0

Synthesis of compound(1-(4-(1-(4-aminophenylsulfonyl)-5-methyl-2,5-di-hydro-1H-tetrazol-5-yl)phenyl)-5-phenyl-2,5-dihydro-1H-tetrazol-5-yl)(phenyl)methanol [4][20]

To solution of schiff base [2] (0.48 gm0.001mol) in DMF 15ml, Sodium azide (0.13 gm,0.002mol) has been added this mix has been refluxed for 6h., then it has been allowed to cool and the yellow precipitate has been filtered and re-crystallized from petroleum ether. yield (83 %) MP=126-128^{0}C

Synthesis of compound [5,6][21]

A mixture of (0.001mol) of schiff base [1] and (0.002mol) different anhydrides (naphthalic anhydride or 3-Nitro phthalic anhydride) in dry benzene were reflux for 7h. The crystalline solid was filtered and recrystallized from EtOH. yield (88%) (80%), M.P.= (200-202) (190-192) C^0 respectively.

Synthesis of Metal Complexes [Co (II), Ni (II), Cu (II) and Hg (II)] with ligand [2][22]2.3

The (2:2) chelate complexes metal and ligand have been synthesized via dissolving (0.2g) Schiff base [2] in (25ml) of the absolute ethanol then mixed with solution containing hydrated metal chloride salts of ($CoCl_2.6H_2O$, $CuCl_2.2H_2O$, $NiCl_2.6H_2O$, and $HgCl_2$) dissolved in the absolute ethanol (25 mL) . The mixture refluxed a period (5hrs.) on water bath on the cooling of the contents, the complexes have been separated out. The product has been filtered washed by the ethanol and dried under vacuum

RESULTS AND DISCUSSION

In scheme (1) Compound [1] was synthesized through the reacting of 4-aminoacetophenon reaction with benzoin in ethanol. FT-IR of compound [1] [23] display the appearance bands at (1645) cm^{-1} refer to (C=N) and disappearance of two absorption bands at (3270 and 3464cm^{-1}) due to the symmetric and asymmetric stretching of ($-NH_2$) group respectively . Compound [2] has been synthesized by reaction of compound [1] with 4-aminobenzensulfonamide and 3 drops from glacial acetic acid in ethanol. FT-IR of compound [2] shown the vanishing of band at (1732) cm^{-1}due to carbonyl group and appearance of bands at (1630,1642), (1313) and (1147) cm^{-1} which are connected to imine group asymmetric and symmetric of the $\upsilon(SO_2)$ group. ^1H NMR (δ ppm) : a sharp signal at (δ1.25) ppm refer to 3 protons for CH_3 group signal at (δ2.14) ppm that attributed to proton of (OH) signal at (δ6.61) for proton (CH) multiplet signals at (6.91 -8.40) for aromatic protons and signal at δ (10.46) for proton NH_2. Thiazine [3] synthesized by reaction of compound [2] with 2-mercaptobenzoic acid in dry benzene, FTIR of the compound[3] [24] appearance of (C=O)group of thiazine at (1693cm^{-1}) and vanishing of (C=N) group in (1630,1642) cm^{-1}.

Compound [4] produce by reacted compound [2] with Sodium azide in DMF. FTIR of compound [4] display demise of absorption of imine group with appearance of new absorption in (1516, 1365) cm^{-1} that are assigned to (N=N) and (C-N) stretching.^1H NMR (δ ppm): a sharp signal at δ(1.71 ppm) for three protons for CH_3 group signal at δ (2.09) ppm that attributed to proton of (OH) signal at δ (5.35) for proton (CH) signal at δ (5.84) for proton(NH) multiplet signals at (6.95-8.49) for aromatic protons and signal at δ (10.58) for proton NH_2 [25] .

Compounds [5,6] were synthesized through reaction schiff base and different anhydrides in dry benzene. FTIR of compound [5] exhibited appearance of band at (1687 1768) cm^{-1} for carbonyl groups in oxazepine ring (C=O of lactame and lactone respectively)[26] .^1H NMR (δ ppm): a sharp signal at δ(1.24ppm) three protons for CH_3 group signal at δ (2.09ppm) that attributed to proton of (OH) signal at δ (3.51) for proton (CH) multiplet signals at (7.27-8.32) for aromatic protons and signal at δ (10.54) for proton (NH_2).

In scheme (2) Synthesis some transition metals complexes [Co (II), Ni (II), Cu (II) and Hg (II) of this ligand [compound (2)]

Table(1) show the ligand's FT- IR spectrum The band at1642cm^{-1} has been discovered to be shifted to smaller frequency values 1612-1635cm^{-1} in the complexes' spectra indicated donating the lone pair of the electrons through the nitrogen atom at the azomethine to the center of the metal . furthermore, the new band in far infra-red spectra of the complexes ranging between 513 and 565cm^{-1} has been assigned to υ M–N [27].

Table 1: The characteristic infrared band for free ligand and its metal complexes

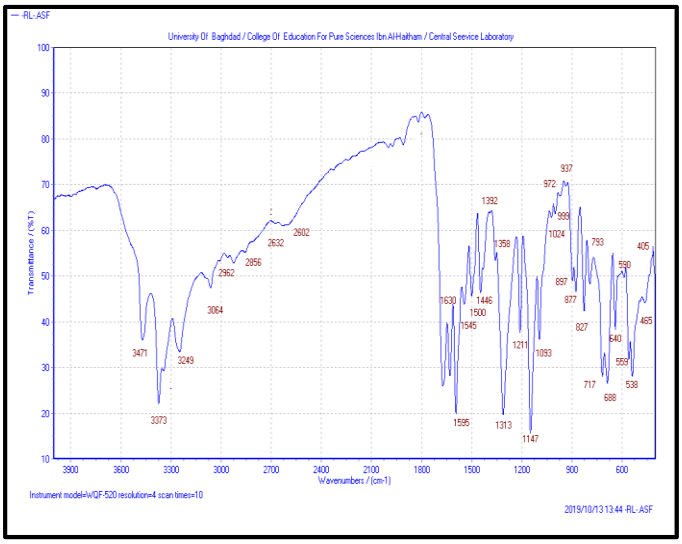
Comp.	Coloer	M.P.	υ(O-H) coordante	υ (NH₂)	υ(C-H) aliph.	υ(C=N)	υ(C=C)	υ (M-N)
L	Brown	180-182	3471	3373 3249	2924-2856	1642	1595	-
L+ Co	Dark green	139-141	3408	3205, 3122	2968-2889	1612	1589	528
L+Ni	Dark brown	132-134	3319	3244 3138	2929-2848	1620	1598	513
L+ Cu	Red brown	163-165	3361	3267 3157	2931-2914	1635	1597	565
L+Hg	Dark yellow	140-142	3473	3260 3150	2935-2908	1616	1597	513

Table 2: The Elemental analysis of some compounds

Com. No.	Theoretical				Experimental			
	C%	H%	N%	S%	C%	H%	N%	S%
[1]	80.24	5.77	4.25	-	80.36	5.69	4.11	-
[2]	69.42	5.16	8.67	6.61	69.39	5.11	8.60	6.58
[3]	66.75	4.37	5.56	12.71	66.66	4.49	6.65	12.92
[5]	60.75	3.56	8.05	3.68	61.73	3.68	8.26	3.79
[L+CO]	58.04	5.00	7.25	5.52	58.01	4.98	7.21	5.50
[L+Ni]	54.66	4.71	6.83	5.20	54.62	4.69	6.80	5.00
[L+Cu]	54.23	4.68	6.77	5.16	54.20	4.63	6.75	5.11
[L+Hg]	44.40	3.83	5.55	4.22	44.39	3.80	5.51	4.18

Electronic Spectral data for the complexes:

The UV-Visible Schiff base ligand spectrum characterized mainly by 2 peaks of absorption at (290nm, 458nm) assigned respectively to (π → π *) & (n → π*). Those electronic transition have been shifted toward higher or lower frequencies in electronic spectra of all of the prepared complexes, verify the ligand's coordination with the ions of the metal. Electronic Co(II) complex spectrum has shown 4 new peaks of absorption, those two peaks at (281nm & 443nm) can be appoint to intra - ligand and other peaks at (635nm) and (780nm) have referred to (d-d) electronic transition Type $^4A_2g^{(F)} \rightarrow {}^4T_3g^{(F)}$ and $^4A_2g^{(F)} \rightarrow 4T_2g^{(F)}$ respectively which suggests octahedral geometry around the ion of the Co(II). The electronic Ni (II) complex spectrum has shown 4 new peaks of absorption, those peaks at (280nm and 441nm) can be given to the intra – ligand. The second peak at (482nm) has been a result of (d-d) type of the electronic transition $^6A_1g \rightarrow 4T_2g$ $^{(G)}$ whereas the final peak at (640nm) $^6A_1g \rightarrow {}^4T_1g$ $^{(G)}$. Those peaks have been in good agreement of the octahedral geometry for the complex of Ni (II). The electronic spectrum of Hg(II) complex showed absorption peaks at (241, 439) nm refers to (π → π *)(n → π*) respectively the metal ion of those types of the complexes belong to d^{10} system and this metal showed no (d-d) electronic transition .[28,29]



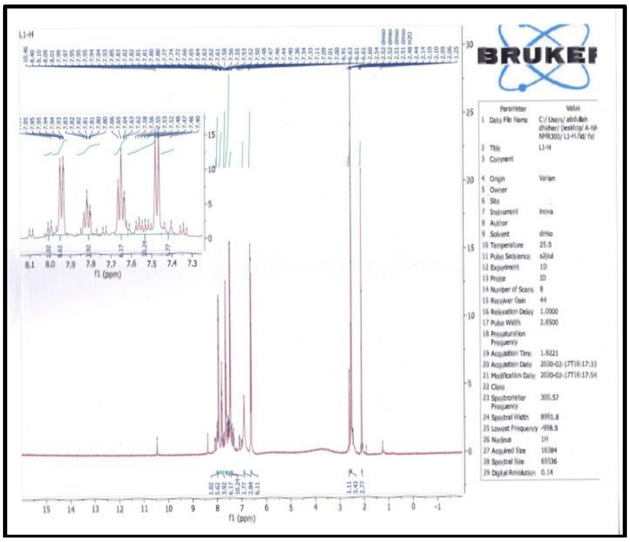
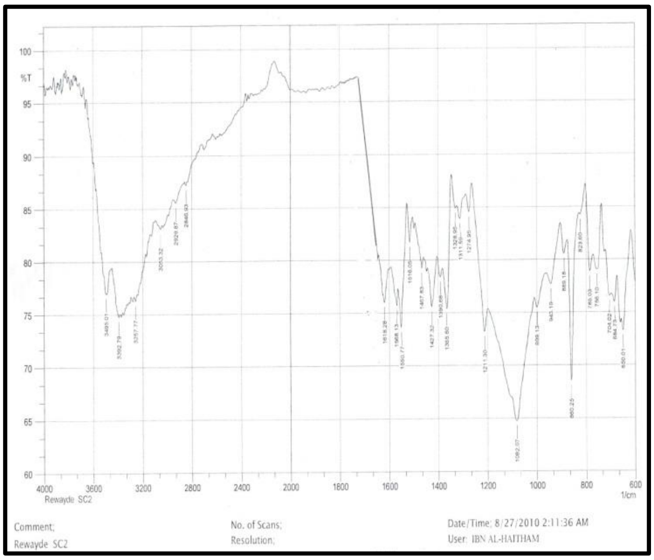


Fig 1: FTIR of compound [2] Fig. (2): ^{1}H-NMR of compound [2]



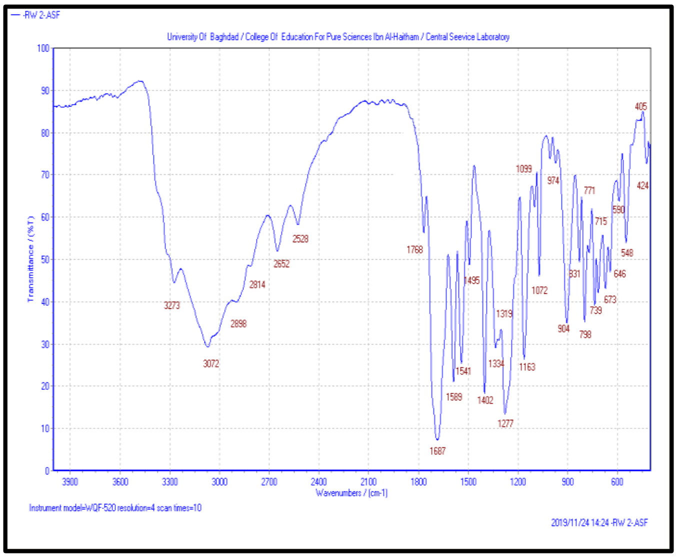
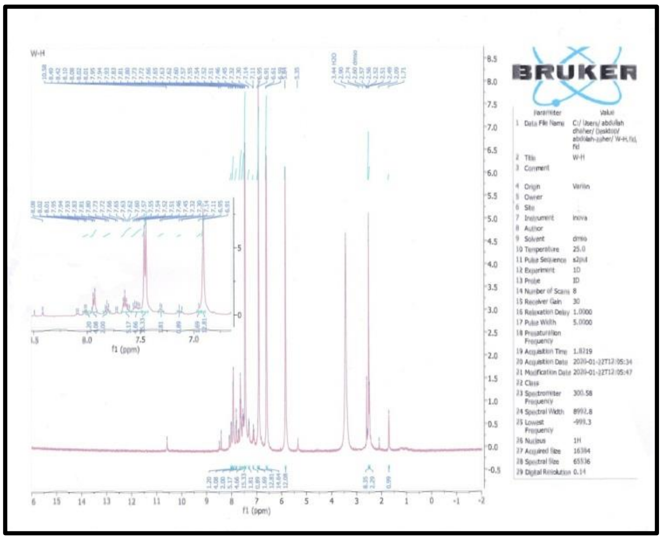


Fig 3: FTIR of compound [4] Fig 5: FTIR of compound [5]



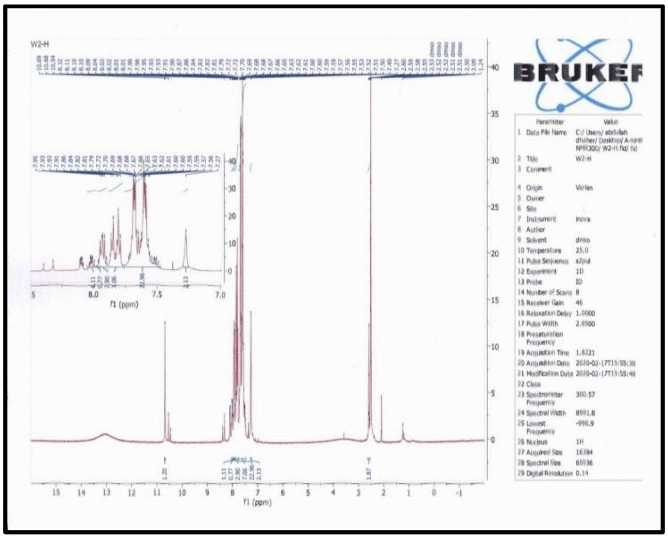


Fig 4: ^{1}H-NM R of compound [4] Fig 6: ^{1}H-NM R of compound [5]

Corrosion inhibition (12,30)

Mild steel samples with the chemical composition: C, 0.20 ; Fe, 99.22 ; Cu, 0.19; Si,0.28; Mn, 0.04; Ca, 0.01; and S, 0.06; The gravimetric approach (i.e. the weight loss) is possibly the most commonly utilized inhibition assessment approach. The simplicity and reliability of measurement that have been presented by weight loss approach is such that the approach is forming a base-line manner for the measurement in several of the programmed corrosion monitoring. The measurements of the weight loss which have been carried out under the total immersion with the use of (250ml) beakers that contains (100ml) of the testing solution at the temperature of the room. The coupons of the iron weighed and hanged in beaker using a hook and a rod. The coupons have been covered for a 6h period, cleaned by the use of washed distilled water, dried then reweighed. After that, the loss of weight, in grams, has been considered as a weight difference of the coupons of iron prior to and post the immersion in a variety of the testing solutions.

The % inhibition efficiency was calculated by using following formula:

$$I.E. = \frac{Wu-Wi}{Wu} \times 100$$

Where, I.E. stands for the Inhibition efficiency in inhibitor solution

Wi represents the Loss in weight in the solution of inhibitor,

Wu stands for the weight loss in control solution

Table 3**:** Weight loss and the efficiency of the percentage inhibition which has been obtained for the mild steel has been immersed in 0.1M HCl solutions of some compounds ligand and its complexes at a variety of the concentration values of all compounds at room temperature

Beaker No.	Compound	Concentration	Initial Weight	Final Weight	Loss in weight	% Loss in weight	I.E (%)
1	Control [HCl]	0.1	6.524	4.588	1.936	29.675	-
2	HCl+compound[3]	1×10^{-3}	6.293	6.012	0.281	4.465	85.485
3	HCl+compound[3]	1×10^{-2}	6.754	6.495	0.259	3.834	86.621
4	HCl+compound[4]	1×10^{-3}	6.522	6.198	0.324	4.967	83.264
5	HCl+compound[4]	1×10^{-2}	6.613	6.300	0.313	4.733	83.832
6	HCl+compound[5]	1×10^{-3}	6.443	6.190	0.253	3.926	86.931
7	HCl+compound[5]	1×10^{-2}	6.601	6.399	0.202	3.060	89.566
8	HCl + Ligand	1×10^{-3}	6.392	6.202	0.19	2.972	90.185
9	HCl + Ligand	1×10^{-2}	6.572	6.395	0.177	2.693	90.857
10	HCl + Co (II) complex	1×10^{-3}	6.501	6.411	0.090	1.384	95.351
11	HCl + Co (II) complex	1×10^{-2}	6.631	6.595	0.036	0.542	98.140
12	HCl + Ni (II) complex	1×10^{-3}	6.495	6.384	0.111	1.709	94.266
13	HCl + Ni (II) complex	1×10^{-2}	6.453	6.414	0.039	0.604	97.985

14	HCl + Cu (II) complex	1×10^{-3}	6.683	6.576	0.107	1.601	94.473
15	HCl + Cu (II) complex	1×10^{-2}	6.593	6.489	0.104	1.577	94.628
16	HCl + Hg (II) complex	1×10^{-3}	6.761	6.590	0.171	2.529	91.167
17	HCl + Hg (II) complex	1×10^{-2}	6.799	6.652	0.147	2.162	92.407

Mechanism of Corrosion Inhibition (10,31)

Synthesis compounds[3,4,5] schiff base and its complexes have shown good level of the corrosion inhibition against the mild steel corrosion in the acidic medium that can be a result of to the existence of the p electrons in the aromatic systems and several bonds existence of the azomethine group and electro-negative atom (N) In structures of inhibitor molecules.The chemisorption of inhibitor molecules, has been possible through the the unoccupied (d) orbital of Fe-atoms, acting as electron acceptor. Which is why, the coordinated bonds are produced through an overlap of unoccupied 3-d-orbitals of Fe with the p-orbital electrons in inhibitor. The impact of additional substituent groups of the hydroxyl on the aromatic ring. This group shows an inductive impact, resulting in the increase of the density of the electron and aromatic ring activation that can have an influence of more sufficient absorptivity to inhibitor, improving the protection as well as the adsorption. Which suggests that the corrosion inhibition results from the adsorption of the inhibitors on metal surface and compounds play the role of adsorption inhibitors. In addition to that it has to be pointed out that large sizes and high molecular weights of compounds may play a role in a higher efficiency of inhibition.

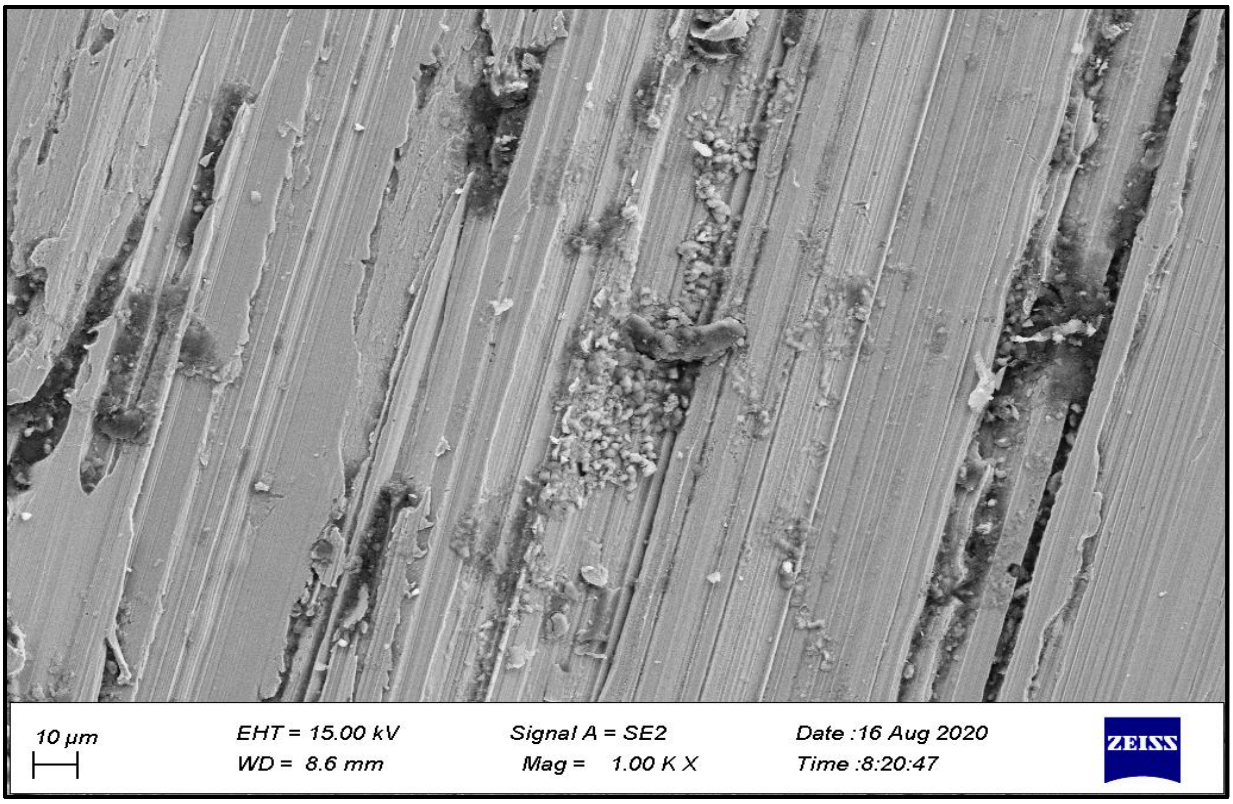
Scanning Optical Microscope (32,33)

The optical mild steel sample images in Figure (7) show the degradation of the carbon steel with an absence of the inhibitors. The coupon surface in which it may be seen that surface has been rough and corroded in corrosive solution in corrosion inhibitor absence.

This degradation seems more at the grain boundary, due to the fact that those areas have the highest susceptibility to the corrosions and could have the responsibility for high corrosion rates. Optical images in Figure (8) of mild steel following the corrosion in the acidic medium that contain inhibitors exhibit adsorbed inhibitor molecules' layer on the surface of the metal, which protects this metal.

CONCLUSION

The present study shows Thiazine [3], Tetrazole [4] 1,3-Oxazepine[5] Schiff base and its complexes that researched are sufficient inhibitors of corrosion for the mild steel in 0.1M of the HCl . The metal complexes have shown higher efficiency of inhibition compared to free ligand and synthesis compounds. The increase in the metal complexes' efficiency in comparison with schiff base can be a result of their molecular planarity and larger size. which is why, the efficiency order is as [CoL] > [NiL] >[CuL] >[HgL] >[L]> [5]> [3]> [4]. The measurements of the weight loss exhibit that the surface coverage level and the efficiency of the inhibition are increased with increasing the concentration values of the compounds [34].



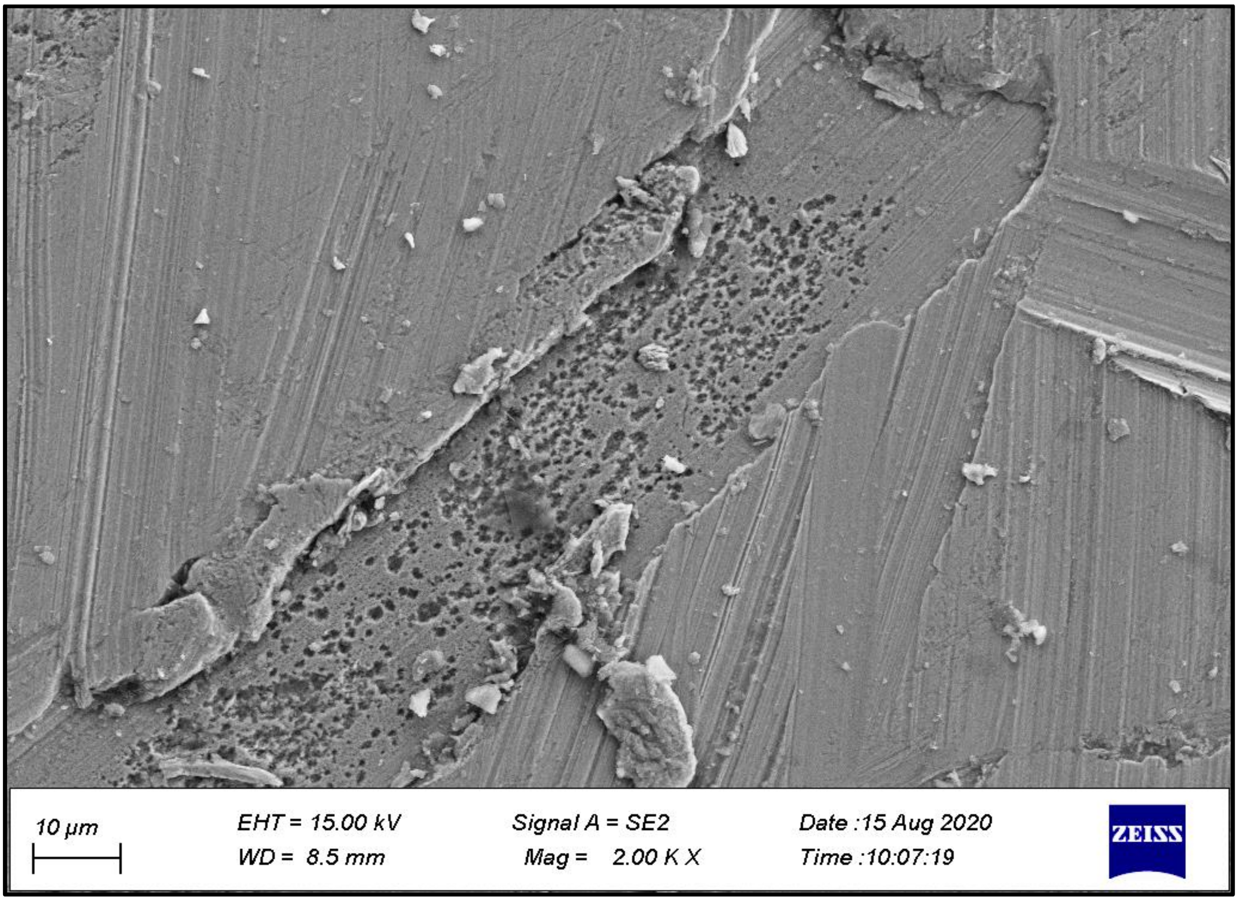
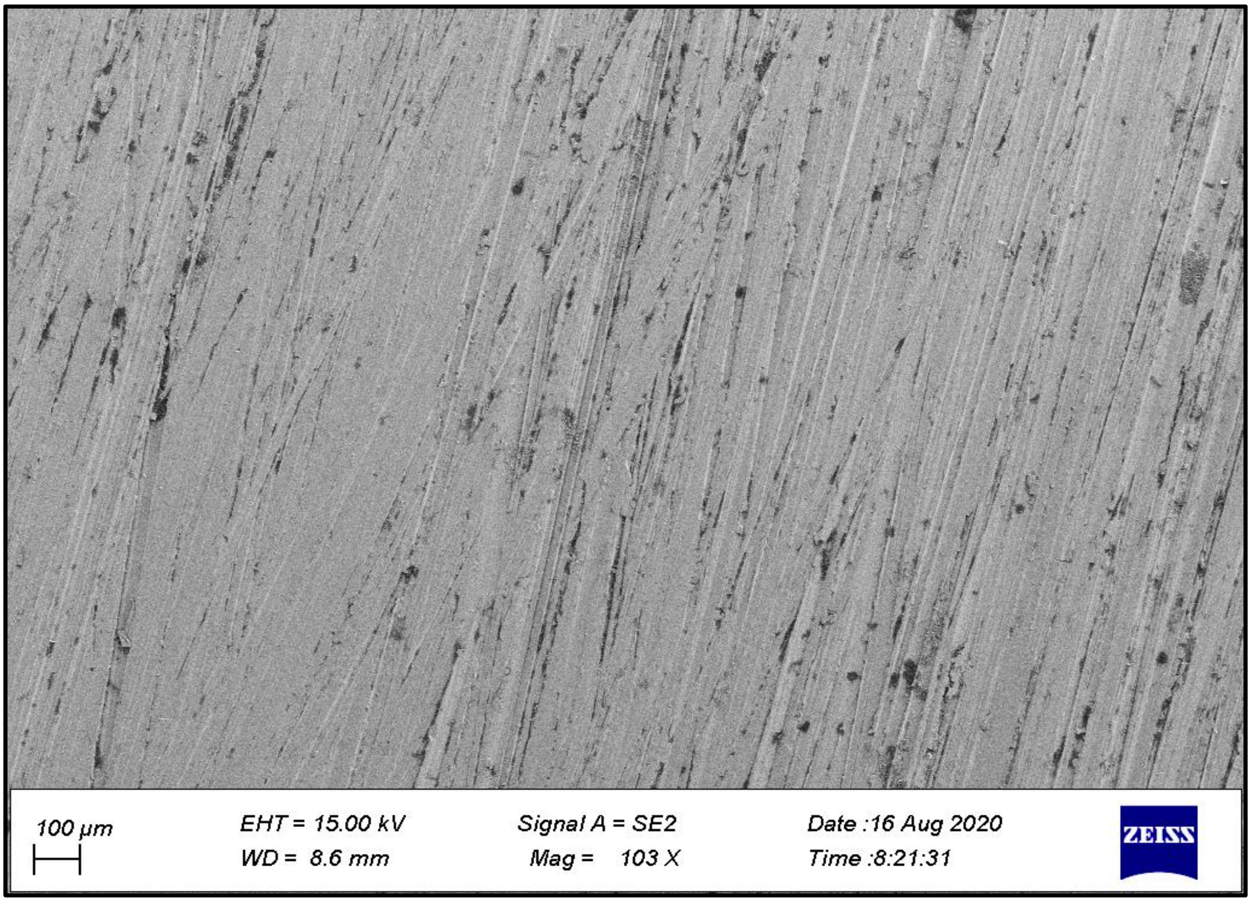


Figure 7: images of mild steel sample in acid medium without inhibitor



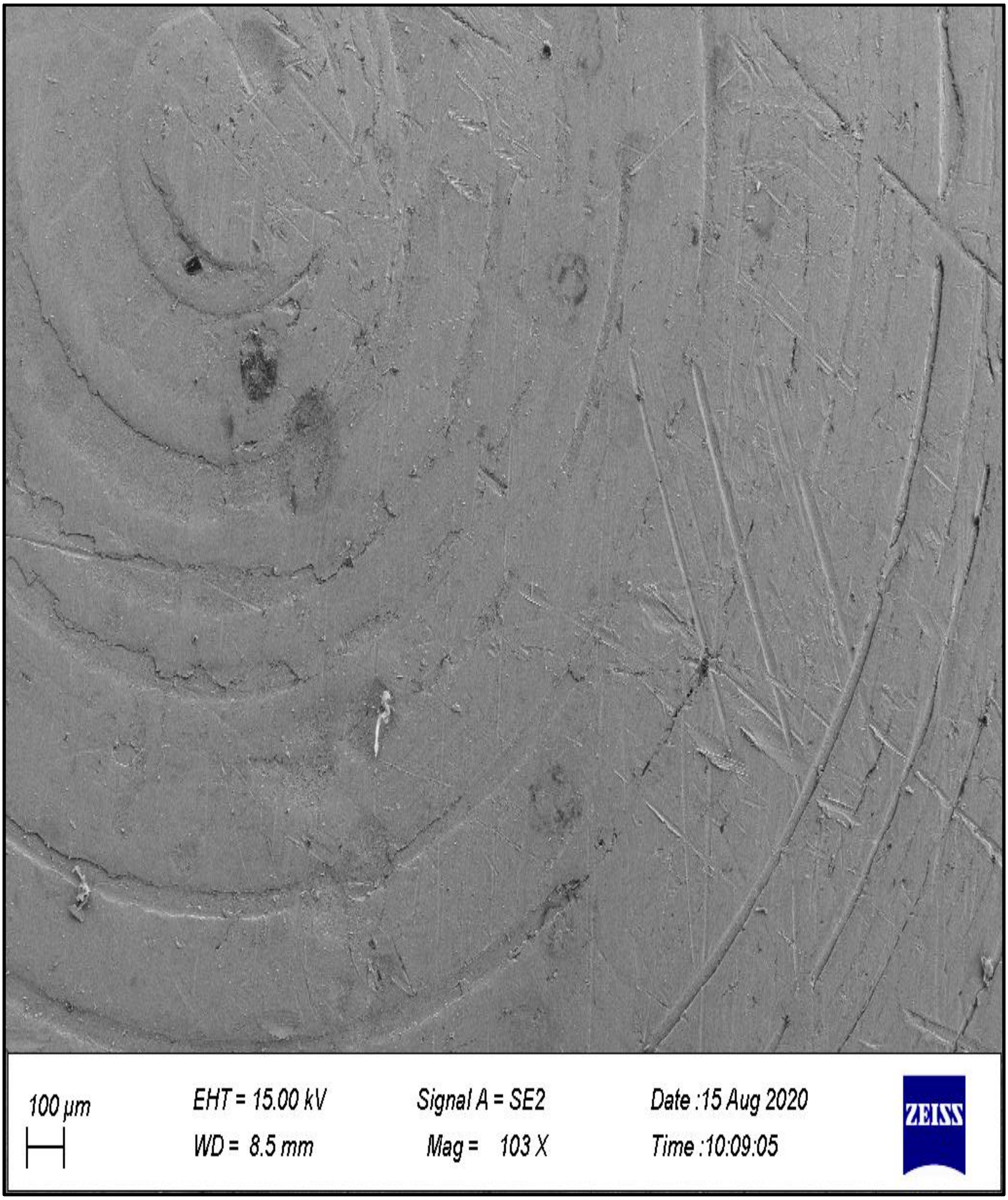
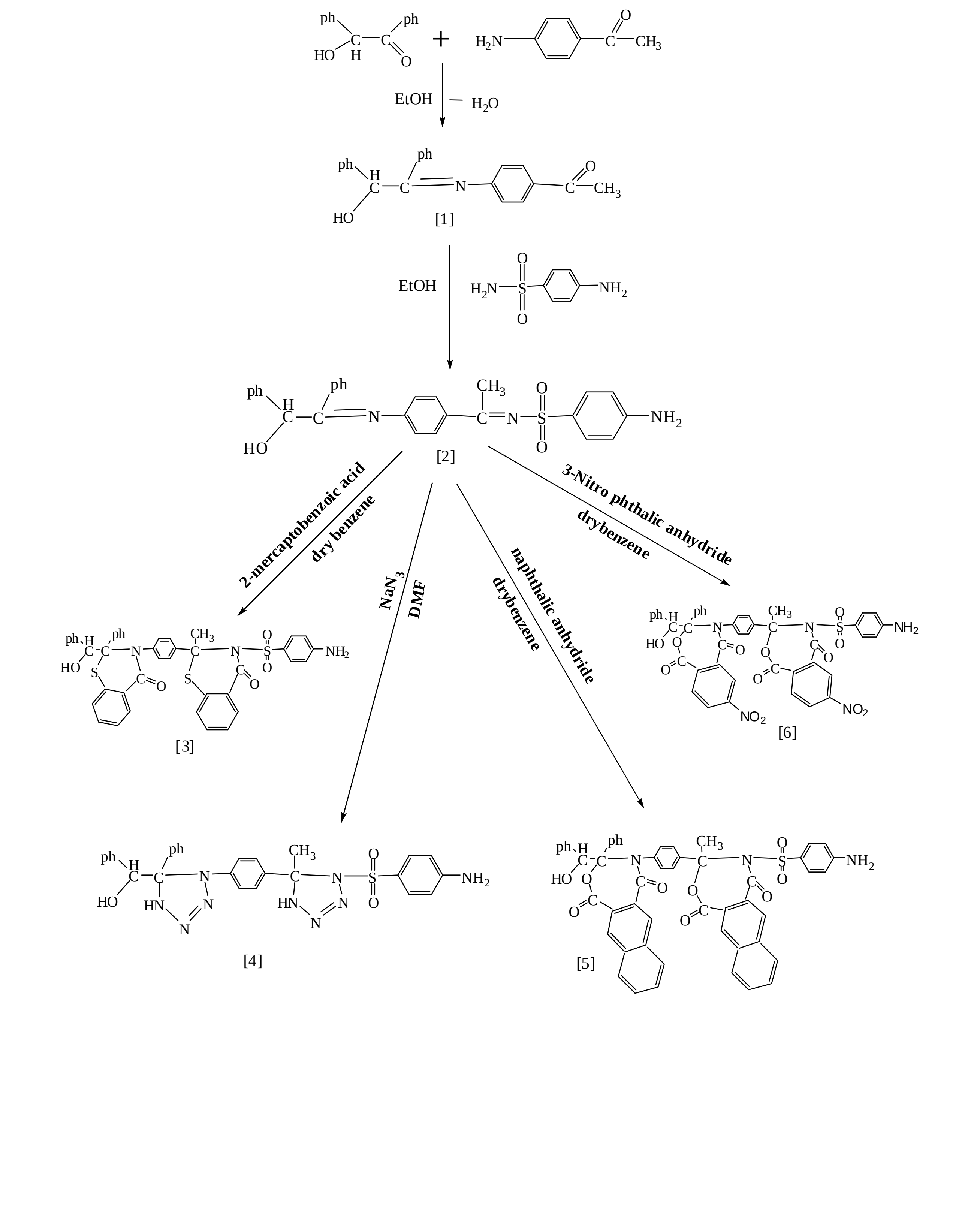


Figure 8: images of mild steel in acid medium witht inhibitor


ph
ph
HO
H
C
O
H2N
CH3
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− H2O
[1]
EtOH
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2-mercaptobenzoic acid
dry benzene
NaN3
DMF
naphthalic anhydride
drybenzene
3-Nitro phthalic anhydride
drybenzene
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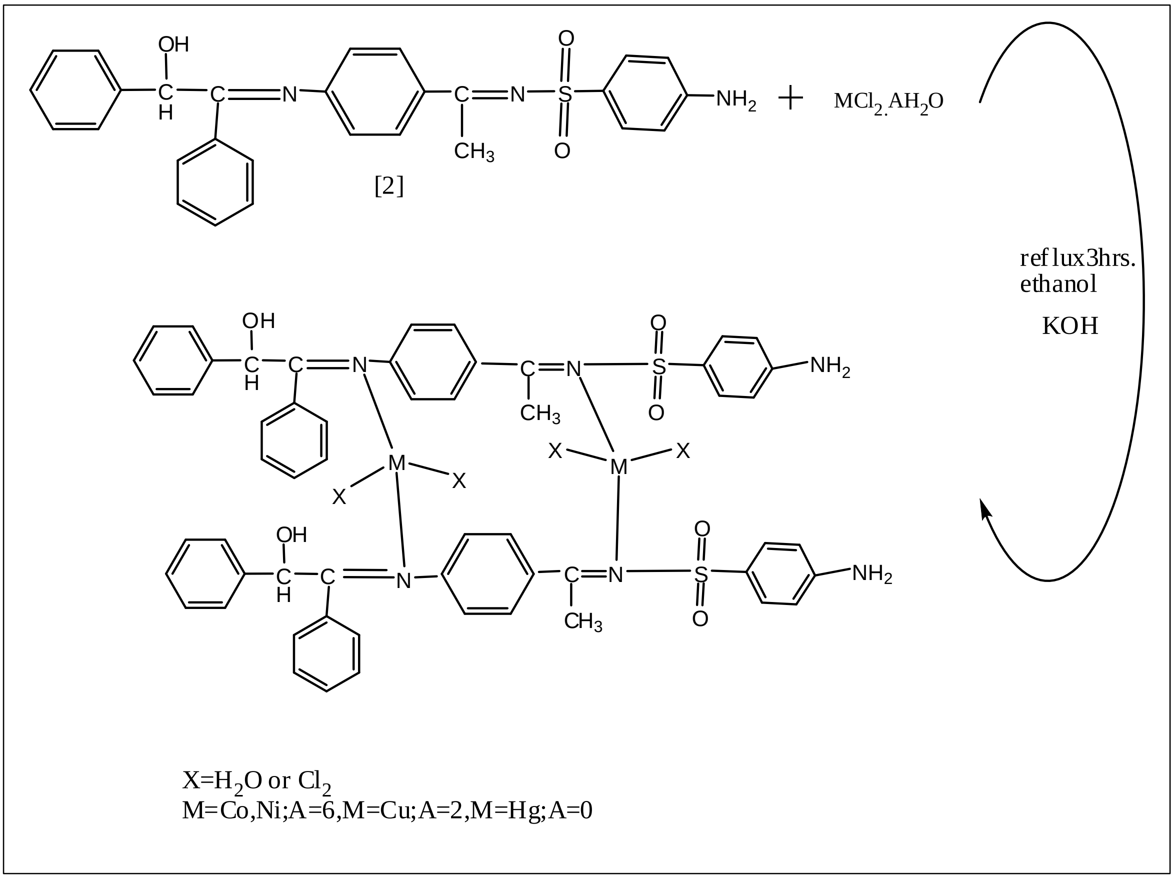
Scheme (1)



Scheme (2)

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