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## An Investigation into the KSA Pipelines Corrosion Inhibition using an Innovation Schiff Base/Nano Nickel Composites.

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### ABSTRACT

Schiff base (SH) and Schiff/Nano Ni composites of different percentages have been used at various test periods for corrosion resistance of formation water. The surface morphology, of the deposits investigated using the electro-deposition parameters (current density and peak voltage), pH and the concentration of nickel nanoparticles in the bath. The effect of different Schiff compound concentrations on the adsorption of formation water H<sub>2</sub>S, morphology of the deposits will be studied. The adsorption bands (V1, V2, V3 and V4) for the Nano nickel complex (D) is found higher compared to A, C and B complexes respectively. The electrochemical properties of the NNi complexes revealed that the peak voltage tends towards the lower values compared to Schiff base which indicating their effective ability to adsorb H<sub>2</sub>S. The SEM observations of both NNi complexes and Schiff base showed that SH/NNi (D4) exhibit uniform dense microstructure followed by Schiff base and (C4), (B4) and (A4) SH/NNi composites respectively.

**Keywords:** KSA pipelines, corrosion, Schiff Base.

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## INTRODUCTION

Corrosion is one of the major problems faced by the oil and gas industry, especially in wells operating in sour conditions. Billions of dollars are spent on capital replacement and control methods for corrosion infrastructure. Large quantities of hydrogen sulfide ( $H_2S$ ) are produced in gas and petroleum industries. Corrosion of materials occurs because of several factors for example the application environment, operational conditions, presence of non-equilibrium phases, failure of the protective phases or layers in the materials, etc. In addition to the electro-chemical phenomena, temperature could influence the corrosion rates to different degrees depending on the materials involved [1]. Abo-Dief [2], investigated the effect of  $H_2S$  concentrations, flow rates, and test temperatures on the corrosion of the stainless steel specimens at a test periods ranges from 72 hours to 288 hours. The results showed that addition of Nano Zn in the deposition process increases the corrosion resistance. The surface morphology of the zinc deposits was studied by scanning electron microscopy (SEM). Kesavan et al. [3], deduced that corrosion is known as the destruction of materials due to interaction with corrosive environment. Chen et al. [4], introduced the electrochemical corrosion behavior of electrodeposited conductive polypyrrolen (ppy) coatings doped with small size sulfate ( $SO_4$ ), medium size para-toluene sulfonate (pts) and large size dodecyl benzene sulfonate (dbs) counter anions using electrochemical measurements and surface analysis. Ganash [5], illustrated that three approaches are commonly applied to reduce the rate of corrosion including cathodic protection, anodic protection (passivation), and application of barrier coatings. Protective coatings have been widely used for metal corrosion control. The use of conducting polymers for the inhibition of corrosion is an area which is very recently gaining increasing attention.

Composite materials have various properties such as dispersion hardening, self-lubricity, high-temperature oxidation resistance, excellent wear, and corrosion resistance. Because of their importance in many fields, the newer composite materials are synthesized through different existing methods. Among these methods, the electro deposition is considered to be one of the most important techniques for producing composites, owing to precisely controlled near room temperature operation, rapid deposition rates, and low cost, [6]. Abo-Dief et al. [7], deposited Nano zinc coatings on stainless steel by electro-deposition. Khan et al. [8], produced Nano composite coatings with unique magnetic, mechanical and optical properties in the last few decades. Spanou et al. [9], obtained Nano composite coatings by electrochemical code position of  $TiO_2$  Nano-particles (21 nm) with nickel, from an additive-free Watts type bath, by applying direct and pulse current conditions. Ashtiani et al. [10], coated Ck45 Steel with nickel–phosphorus alloy from a bath containing sodium hypophosphite and different complexing agents (sodium citrate, sodium acetate and lactic acid). Girisha and Sharma [11], studied the effect of electrochemical bath parameter like corrosion rate on physical and mechanical properties of Nickel based SiC composite coating on Mild Steel substrate. Also, Khan et al. [8], proved that composite coatings composed of nonmetallic particles or wires dispersed in a metal matrix provide improved corrosion resistance, wear resistance, self-lubrication and high-temperature stability, as well as a better performance as electrical contacts and catalysts compared with the pure metal.

Gyawali et al. [12], fabricated Nickel matrix composite coatings by the incorporation of h-BN (hexagonal boron nitride) Nano sheets using the pulse electro deposition technique. Abo-Dief et al. [13], illustrated that some metals basically exhibit high corrosion resistance than others and this can be attributed to several factors like their chemical constituents, the nature of electrochemical reactions itself and others. Kasturibai and Kalaignan [14], deposited  $Si_3N_4$ -reinforced nickel Nano composite coatings on a mild steel substrate using pulse current electro deposition process employing a nickel acetate bath. Karthikaiselvi, and Subhashini [15], studied the efficiency of new water soluble composite for corrosion inhibition of mild steel in 1 M hydrochloric acid (HCl). The use of inhibitors has been found to be one of the best options available for the protection of metals against corrosion. The most efficient corrosion inhibitors are organic compounds containing electronegative functional groups and p electrons in their triple or conjugated double bonds. Karthikaiselvi, and Subhashini [15] concluded that mild steel finds wide application in a broad field of industry and machinery.

Schiff bases is one of the compounds with unique surface activity where it has hydrophobic part (love oil) and another part-loving water (hydrophilic), allowing them to spread among different phases. Adding Schiff bases for Nano Nickel provides a great opportunity for adsorption on the surface of the metal to be protected from corrosion. Schiff base complexes play a vital role in designing metal complexes related to synthetic and natural oxygen carriers [16]. Metal complexes make these compounds effective as stereospecific

catalysts towards oxidation, reduction, hydrolysis, biological activity and other transformations of organic and inorganic chemistry [17]. Schiff bases resulted from aromatic aldehydes ortho-substituted with a hydroxyl group have initially aroused the researchers' interest because of their ability to act as bidentate ligands for transitional metal ions [18-20]. Ha et al. [21], showed that the azomethines obtained from salicylaldehydes gave the best correlation. Xavier et al. [22] and Shah et al. [23], reported that Schiff bases of salicylaldehydes have also been plant growth regulators and antimicrobial or antimycotic activity. Naeimi and Moradian [24] carried an analytical investigation of the Schiff bases. A large number of different Schiff base ligands have been used as cation carriers in potentiometric sensors as they have shown excellent selectivity, sensitivity, and stability for specific metal ions such as Ag(II), Al(III), Co(II), Cu(II), Gd(III), Hg(II), Ni(II), Pb(II), Y(III), and Zn(II), [25]. Schiff bases have been studied for their important properties in catalysis. The aim of the present work is synthesizing and characterizing Nano nickel complexes with Schiff bases using Fourier Transform Infra-Red Spectroscopy and Ultraviolet-Visible Spectroscopy techniques in adsorbing formation water H<sub>2</sub>S. The SEM observations will be used to confirm the experimental results.

## EXPERIMENTAL WORK

### Materials

Nano nickel chloride [NNi (Cl<sub>4</sub>)<sub>2</sub>], methyl amine, ethyl amine, n-propylamine, n-butylamine, n-pentylamine used as synthesis of Nano nickel complexes. Methanol, acetone and ethanol were used as solvents after distillation.

### Synthesis of Schiff Base (SH)

The Schiff base (SH) prepared using a salicyl aldehyde of the following concentrations (0.05 mL, 0.1 mL, 0.5 mL and 1.0 mL) with the following molar ratios 1, 2, 4 and 8 respectively (mL) added to Nano nickel (NNi) to give the Nano nickel complexes D1, D2, D3 and D4 complexes. Then absolute ethanol (50 mL) respectively injected in the flask. Then, (3-trimethoxysilylpropyl)-diethylenetriamine or N'-isopropyl-diethylenetriamine was drop wise introduced in the flask. After concentration of the solution, the precipitate separated, filtered, washed with ethanol, and dried over nickel chloride under vacuum.

### Synthesis of The NNi and NNi Schiff Base Complexes

A mixture of SH in ethanol was added to an aqueous solution of Nano nickel chloride with various molar ratios (1, 2, 4 and 8) named (D1, D2, D3 and D4) respectively. The complexes prepared and monitored by UV-visible spectroscopy. The mixture of reaction was refluxed for 2 hours and then excess solvent was distilled. The precipitated compounds that separated were filtered, washed with ethanol, and dried in vacuum. A series of [NNi (Cl<sub>4</sub>)<sub>2</sub>] and Ethylenediamine of the following concentrations (A1= 0.05 mL, A2= 0.1 mL, A3= 0.5 mL and A4= 1.0 mL), [NNi (Cl<sub>4</sub>)<sub>2</sub>] and N-propyl-ethylenediamine (B1, B2, B3 and B4) and [NNi (Cl<sub>4</sub>)<sub>2</sub>] and N-(salicylaldimine)-(N'-propyltrimethoxysilane)-diethylenetriamine (C1 to C4) complexes with various molar ratios (1, 2, 4 and 8) with the previous concentrations respectively prepared and monitored using UV-visible spectroscopy.

### Compositional Analysis

Table 1: Analytical data and melting point of Nano nickel complexes.

Complex	M.P. (°C)	Components %				
		C	H	N	O	Ni
A4	184	72.63	5.78	4.82	6.17	10.6
B4	226	72.00	5.43	5.17	6.31	8.37
C4	217	72.46	5.61	5.00	6.09	10.84
D4	252	71.5	5.24	5.37	6.52	11.38

The Nano nickel (NNi) complexes confirmed by C, H, and N. The FT-IR spectra for the complexes recorded to infer the various stretching frequencies using Perkin Elmer FTIR spectrometer in the range of 4000-400cm<sup>-1</sup>. The electronic absorption spectra of the NNi complexes recorded at an accelerating voltage of

32V at ambient temperature. The analytical, compositional data of complexes A4, B4, C4 and D4 represented in Table 1.

### Preparation of Thin Film

The thin film of Nano nickel complexes prepared by using the thermal method of metallo-organic chemical vapour deposition where alumina served as a deposition chamber. Prior to each deposition, the substrates sonicated for 30 min on an ultrasound sonicator and the reaction chamber cleaned by acetone. This process done to ensure that the deposition surfaces were free from contamination and defects. For the deposition of nickel chloride thin film, the cleaned surface substrates and fine powder of the precursor kept in a quartz boat which was kept in a working chamber. This chamber maintained at 550°C in a digitally controlled furnace. On getting to the hot zone, the precursor kept in the quartz boat sublimed before it thermally decomposed resulting in the coating on the substrates. The whole process left for 2 hours at the deposition temperature of 550°C. The cyclic voltammetry studies were carried out with three electrode assemblies that connected to a personal computer. The three electrode system consisted of glassy carbon (working), platinum wire (counter) and Ag/AgCl (reference) electrodes.

### Fourier Transform Infra-Red Spectroscopy

Fourier Transform Infrared Spectroscopy (FT-IR) is a spectroscopic technique that deals with the middle infrared region of the electromagnetic spectrum (400 - 4000  $\text{cm}^{-1}$ ). Each covalent bond of organic functions and inorganic species exhibits a characteristic frequency of vibration in FT-IR spectroscopy which can be used to identify its component. The vibration forms of molecules are primary two types: (1) stretching vibration ( $\nu$ ), and (2) bending vibration ( $\delta$ ). Generally, stretching vibration modes are observed at higher frequency than that of bending vibrations for a same covalent bond. The absorption bands observed are attributed to the vibrations. The characteristic absorption bands of Nano nickel are the following: bending vibration  $\delta$  (O-NNi-O) of valence angle of octahedral  $[\text{NNi}(\text{Cl}_4)_2]$ , symmetric stretching  $\nu_s$  (NNi-O) of octahedral  $[\text{NNi}(\text{Cl}_4)_2]$  located at a symmetric stretching  $\nu_{as}$  (NNi-O) of octahedral  $[\text{NNi}(\text{Cl}_4)_2]$ , stretching  $\nu$  (O-H) of  $\text{H}_2\text{O}$  of the surface of Nano nickel. In addition, FT-IR spectra also can be used to quantify species by an adequate treatment of spectra.

## RESULTS AND DISCUSSIONS

### Fourier Transform Infra-Red Spectroscopy

The infrared spectral studies show various stretching frequencies and prominent functional groups present in the complexes A4 to D4. The bands at 1797-1808  $\text{cm}^{-1}$  correspond to indicating the involvement of both the nitrogen atoms of the amine groups in the coordination. The bands at 1466-1471  $\text{cm}^{-1}$  occurring for  $\nu$  (phenolic C-O) after complexation indicate the shift due to coordination of phenolic carbon – oxygen in the ligand to the metal atom. The band observed in the region 519-553  $\text{cm}^{-1}$  was assigned (Table 2) to  $\nu$  (NNi-N). The bands in the range 586-598  $\text{cm}^{-1}$  are due to (NNi-O) complexes stretching.

### Ultra Violet-Visible Spectroscopy

A series of Nano nickel complexes (A, B, C and D) prepared and monitored by UV-visible spectroscopy. The corresponding data are gathered and showed in Figures 1-6. All the  $[\text{NNi}(\text{Cl}_4)_2]$  complexes presented two absorption bands  $\nu_3$  and  $\nu_2$  in the range of 25962 to 34131 and 15648 to 20777  $\text{cm}^{-1}$  respectively (Figures 1 and 2), and a third large band,  $\nu_1$ , appeared in the NIR region (Figure 3). The bands  $\nu_3$ ,  $\nu_2$ , and  $\nu_1$  can be attributed to the spin-allowed electronic transitions of  $\text{Ni}^{2+}$  ions in an octahedral environment. From the absorption bands  $\nu_3$  and  $\nu_2$ , the crystal field,  $\nu_1$ , and the nephelauxetic parameter,  $\alpha$ , can be obtained according to Kettle [26]. However, absorption bands  $\nu_3$  and  $\nu_2$  of B-complexes decrease compared to A-complexes with same ligand/ $[\text{NNi}(\text{Cl}_4)_2]$  molar ratio in absolute ethanol and the spectra show a systematic decrease in  $\nu_4$  values (Figure 4).

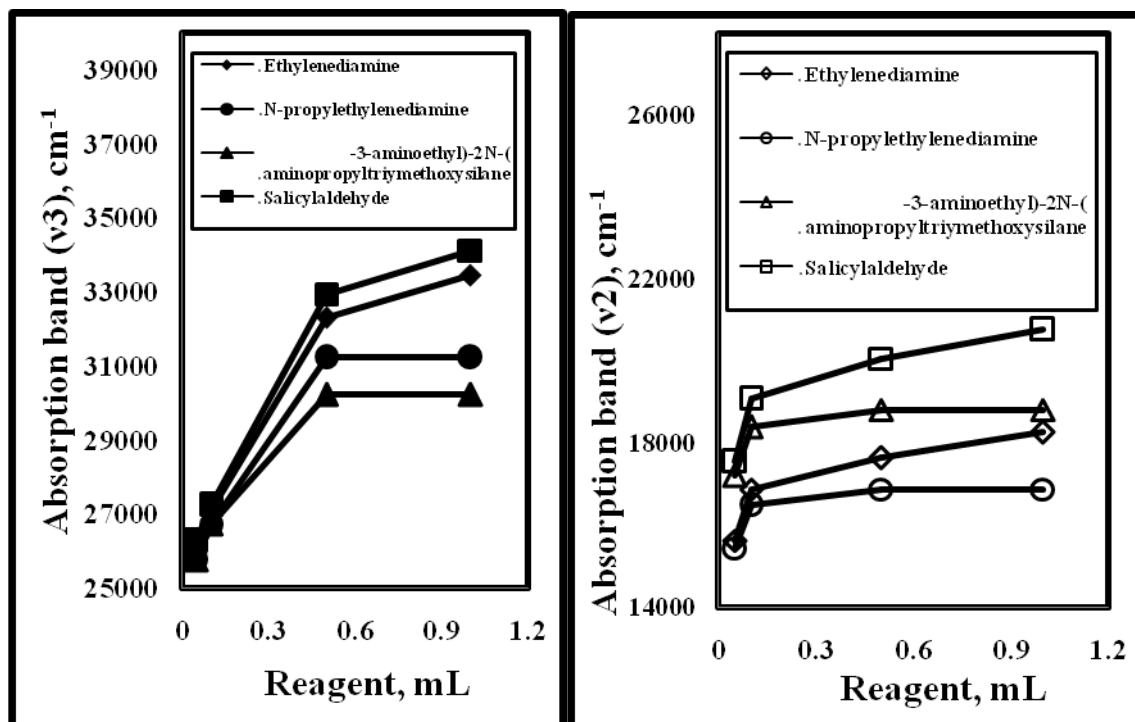
Furthermore, Incorporation of Nano nickel-B complexes in the wall of a nano structured increase of ligand/ $[\text{NNi}(\text{Cl}_4)_2]$  complexes, the difference of  $\nu_4$  values between B1, B2 and B3 and A1, A2 and A3 become progressively great indicating that the steric effect is more important than the inductive effect. The ratio of the

energy splitting in the complex to the free Ni<sup>2+</sup> ion value is the nephelauxetic parameter  $\alpha$ .  $\alpha$  correlates the extent to which the d-electrons of the metal are delocalized on the ligand orbitals. The more electrons delocalized, the smaller nephelauxetic parameter  $\alpha$ . The  $\alpha$  values of both the B-complexes and A-complexes decrease with the increase of ligand/[Ni(Cl<sub>4</sub>)<sub>2</sub>] molar ratio (=1, 2, 3, 4). When B-complexes were compared to A-complexes in the same ligand/[Ni(Cl<sub>4</sub>)<sub>2</sub>] molar ratio, similar  $\alpha$  values are observed, Fig. 5. Thus the nephelauxetic parameter seems less sensitive to steric interaction than the  $\nu_4$  value. Therefore,  $\alpha$  is primarily dependent on inductive and polarization factors.

**Table 2: FT-IR frequencies of [Ni(Cl<sub>4</sub>)<sub>2</sub>] complexes.**

Band	Wave length, cm <sup>-1</sup>			
	A4	B4	C4	D4
$\nu$ (C=O)	1469	1470	1471	1466
$\nu$ (C=N)	1799	1804	1808	1808
$\nu$ (NNi-O)	594	598	586	586
$\nu$ (NNi-N)	514	553	519	521

The absorption bands  $\nu_3$  and  $\nu_2$  of both B-complexes and C-complexes shift to lower energy values in aqueous solutions similar absorption bands  $\nu_3$  and  $\nu_2$  and  $\nu_4$  values and nephelauxetic parameter  $\alpha$  are observed (Figures 1-5), indicating that there are no effect on the trimethoxysilane in C-ligand. Comparing the C1 complex with C2, absorption bands  $\nu_3$  and  $\nu_2$  are significantly shifted, from 25770 cm<sup>-1</sup> and 26731 cm<sup>-1</sup>



**Figure 1: Effect of reagent volume on the UV absorption band ( $\nu_3$ ) at various reagent values of the Nano nickel complexes.**

**Figure 2: Effect of reagent volume on the UV absorption band ( $\nu_2$ ) at various reagent values of the Nano nickel complexes.**

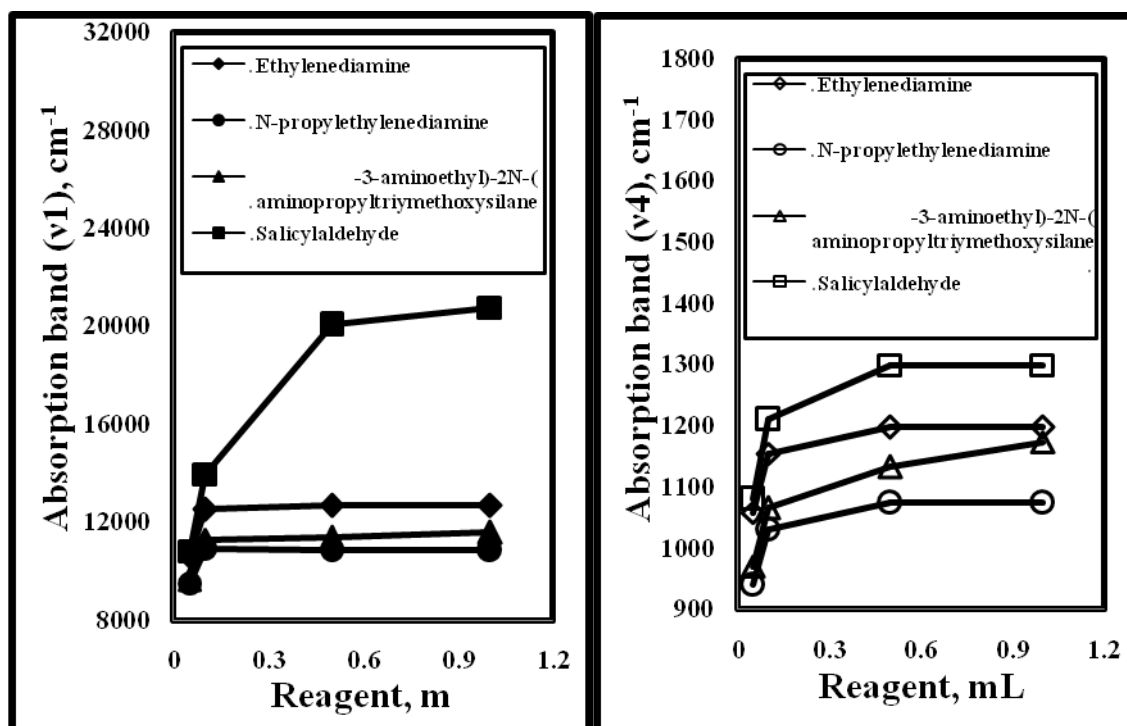


Figure 3: Effect of reagent volume on the UV crystal field (V1) at various reagent values of the Nano nickel complexes.

Figure 4: Effect of reagent volume on the UV absorption band (V4) at various reagent values of the NNI complexes.

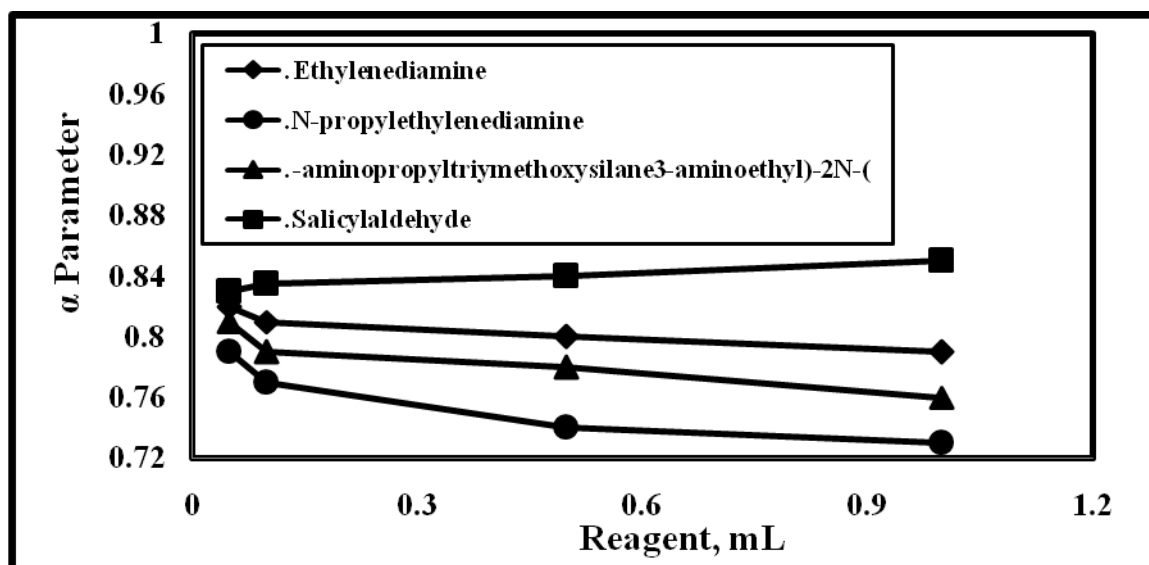
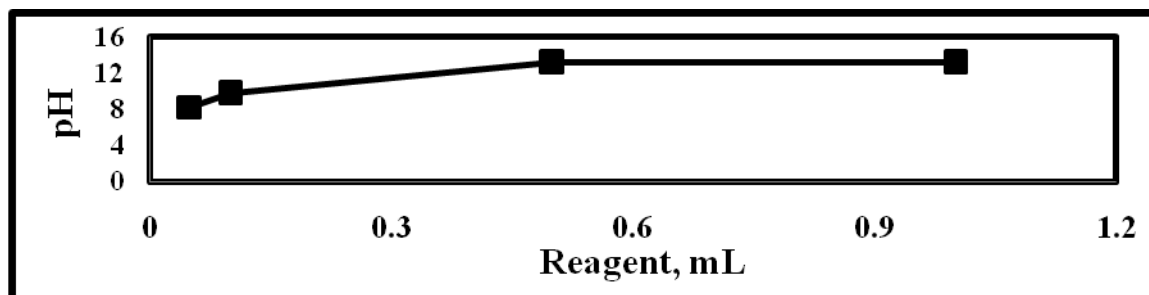


Figure 5: Effect of reagent volume on the UV nephelauxetic parameter (V4) at various reagent values of the NNI complexes.

to  $15648 \text{ cm}^{-1}$  and  $168960 \text{ cm}^{-1}$ , respectively. Concomitantly, the crystal field  $V1$  increases from  $10614 \text{ cm}^{-1}$  to  $20777 \text{ cm}^{-1}$  and the nephelauxetic parameter  $\alpha$  decreases from 0.85 to 0.81 with the increase of the ligand number, indicating that the electron of Ni (II) delocalize to the ligand. Absorption bands  $V3$  and  $V2$  could probably be assigned to that of C-complexes. This progressive shift is also observed in that of B-complexes in aqueous solution, whereas, the absorption bands  $V3$  and  $V2$  in absolute ethanol. Therefore, the general shift of the absorption bands is most probably due to the complexity of aqueous solutions of amine coordination

compounds or the pH effect of solution. So, Fig. 6 showed that as reagent concentration increases, pH increases.



**Figure 6: Effect of Reagent volume on the degree of acidity pH at various reagent values of the Nano nickel complexes.**

### Electrochemical Polarization results

The electrochemical response of the Schiff base was studied by recording the values of minus peak voltage and peak current in the potential range +1.0 V to -1.0 V. Fig.7. showed the peak voltage of both Schiff base and SH/NNi (D4) at a scan rate of 0.1 V/s. It can be observed that there are two trends for SH peaks and two trends for SH/NNi Peaks. In the case of Schiff base shown in Fig. 7, the peak values of the anode voltage is higher than the cathode voltage since the ordinate (-V). This can be attributed to the two successive oxidation processes of the reduced form of the Schiff base. Thus the original molecule regenerates at a higher oxidation potential - 0.337 V. While the cathodic peak appeared at - 0.592V due to the involvement of two electrons was assumed, since the height of this cathodic peak was significantly higher than the other normal peaks in agreement with Shaju et al [27].

While in the case of Schiff base/Nano nickel (D4) shown in Fig. 7, it is clear that both the anodic and cathodic peak values are lower than that of the Schiff base values which means that they directed into the positive zone which induced the effectiveness of the Schiff base/Nano nickel (D4) composite on the adsorption of H<sub>2</sub>S over the Schiff base effect. Also, the cathode peak trend values are shown higher than the anode peak trend that illustrate the higher adsorbent of the cathodic effect. The figure showed that as the scan rate increases, the Epc values are shifted to more negative potentials for both cases. For the counter anodic peak was lower and it may be concluded that the reduction and oxidation process occurred in the potential range -1 to +1 V for the Schiff base were lower than the composite in nature.

Fig. 8 illustrated the variation of I peak values for both Schiff base and Schiff base/NNi (D4) compositewith the scan rate. The peak current-scan responses are nearly straight lines which exhibit slight deviations from linearity. These deviations suggest a change from a diffusion-controlled process to a mixed controlled process. In the case of Schiff base, the anode and cathode trends are higher which means the adsorption on the anode while in the case of the composite, the adsorption will be on the anode.

Figure 9 illustrated that both  $\Delta$  peak voltage of both Schiff base and Schiff base/NNi composite are nearer to each other and that the composite trend is higher due to its effective on the adsorbent of H<sub>2</sub>S. While Fig. 10 illustrated  $\Delta$  peak current on which there is a clear difference between both Schiff base and Schiff base/NNi composite on which the Schiff trend is shown higher which illustrated the effectiveness of the composite over the Schiff base in the adsorption of H<sub>2</sub>S.

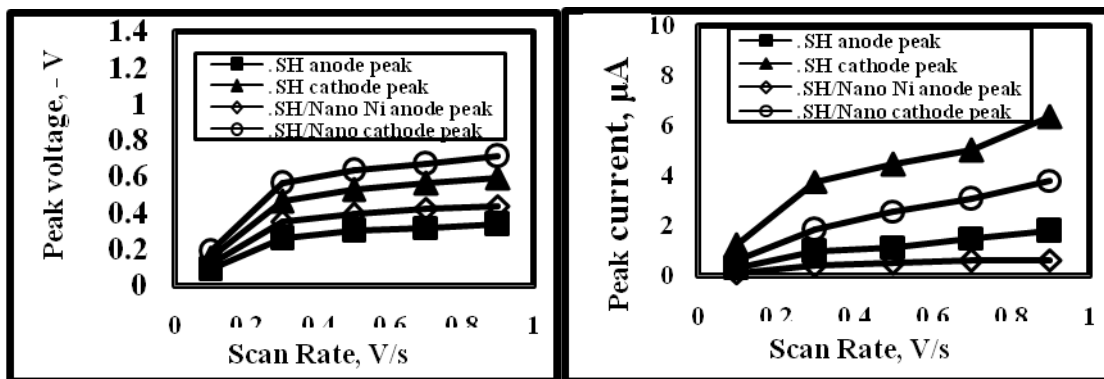


Figure 7: Variation of the peak voltage with the scan rate of both shiff base and Shiff/Nano nickel complex (D4).

Figure 8: Variation of the peak current with the scan rate of both shiff base and Shiff/Nano nickel complex (D4).

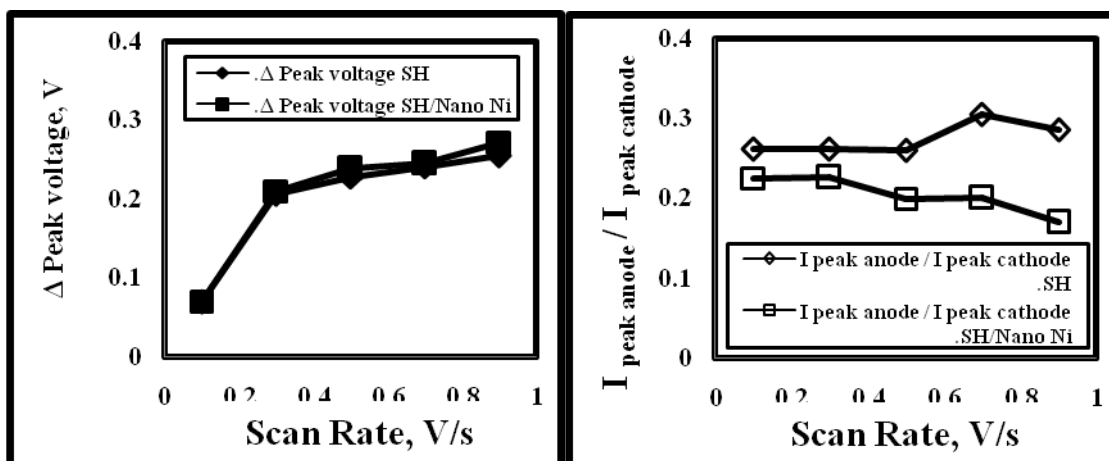


Figure 9: Variation of  $\Delta$  peak voltage on the scan rate of both shiff base and Shiff/Nano nickel complex (D4).

Figure 10: Variation of  $\Delta$  peak current on the scan rate of both shiff base and Shiff/Nano nickel complex (D4).

### Metallographic Examination

The surface morphology of the thin films shown in Fig. 11 of the deposited films reveals a progressive grain growth. The SEM images of both Shiff base/Nano composites nickel thin and Shiff base films. It is clear that SH/NNi (D4) exhibit uniform dense microstructure followed by Shiff base and (C4), (B4) and (A4) SH/NNi composites respectively. These SEM pictures of thin films indicate that the film grown on the substrate shows more particle formation on the surface.

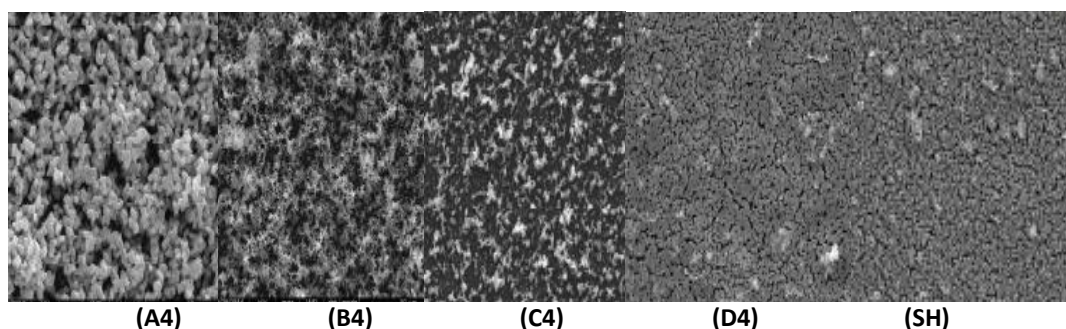


Figure 11: Metallographic examination of SH/NNi thin film coatings.



## CONCLUSIONS

Up to now, the following conclusions are obtained;

1. Nano nickel complexes (A1 to A4, B1 to B4, C1 to C4 and D1 to D4) with Schiff bases have been synthesized and characterized by Fourier Transform Infra-Red Spectroscopy and UltraViolet-Visible Spectroscopy techniques.
2. Nano nickel and N-propylethylenediamine (B) complexes shift to low energy values compared to Nano nickel and Ethylenediamine (A) complexes, due to the steric effect indicated by  $V_4$  values.
3. The absorption bands ( $V_1$ ,  $V_2$ ,  $V_3$  and  $V_4$ ) for the Nano nickel complexes D are found higher compared to A, C and B respectively.
4. As the reagent concentration increases, the degree of acidity and alkalinity increases.
5. The nephelauxetic parameter ( $\alpha$ ) seems less sensitive to steric interaction than the  $V_4$  value. Therefore,  $\alpha$  is primarily dependent on inductive and polarization factors.
6. The absorption stretching band ( $V_3$ ) followed by ( $V_2$ ), ( $V_1$ ) and ( $V_4$ ) respectively for all Nano nickel complexes.
7. The electrochemical properties of the NNi complexes revealed that the peak voltage tends towards the lower values compared to Schiff base that indicating their effective ability to adsorb  $H_2S$ .
8. The SEM observations of both NNi complexes and Schiff base showed that SH/NNi (D4) exhibit uniform dense microstructure followed by Schiff base and (C4), (B4) and (A4) SH/NNi composites respectively.

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