Article

Study the effect of addition polyaniline Nano magnetic with epoxy resin paint on corrosion rate of carbon steel in HCl media

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Abstract:

Nanomagnetic Fe_3O_4 and polyaniline were prepared and checked by using the FT-IR spectroscopy technique, and the results approved the preparation of these substances. The nanoscale nature of these prepared materials was also examined using the electronic scanning microscopy technique (SEM), and the results indicated that the particle size of the magnetite and the prepared polymer Less than 100 nm. This product was mixed with epoxy resin paint in different percent's nnn (5-25) %. The mixtures were used separately as coating on carbon steel (C1020) alloy. The inhibition efficiencies of coated carbon steel were investigated in 0.1M HCl media using Electrochemical Impedance Spectroscopy EIS. The results showed that good efficiency compare with using epoxy resin paint alone, because the epoxy resin combined with polyaniline and magnetized is highly adherent to the surface of the alloy and increases its resistance to corrosion.

1. Introduction

Magnetic nanoparticles are of great importance to researchers in general, because its applications covered many research fields, including catalysts[1,2], that the successful application of these particles depends on the stability of molecules and in different conditions, such as that of It is worth noting that the performance of magnetic nanoparticles is better when the particle size is less than the critical value، Polymer coatings is used to combat corrosion as a convenient and promising method. these coatings do not make sure that they are free of some problems and faults[3], Such as poor adhesion problems, reduced coating elasticity, and its low resistance to corrosion. Therefore, to get rid of these problems and defects and further improve the performance of the coating, it is done by merging the Nano polymers with the coating, which is considered this method of modern methods, which has a tremendous improvement in properties compared to the traditional coatings, due to the small size of the nanoparticles, in addition to that adding the nanomaterials to the coatings allows Production of coatings with multiple functions, and good scratch resistance, surface hardness of coatings, impact and bend resistance properties can also be improved due to inorganic materials[4,5]. As for the paint used in this study, it is epoxy resin coating with Nano magnetite particles such as magnetite, which has a high magnetic susceptibility and is highly adherent to the metal surface، Magnetite is a magnetic oxide, it can be prepared in a number of ways, including the method of co precipitation, as well as it can be produced from the interaction of iron and tri-oxide as shown by the following equation:

$$\underset{\text{Ferrous Oxide}}{\underset{\uparrow}{FeO}} + \underset{\text{Ferric Oxide}}{\underset{\uparrow}{Fe_2O_3}} \rightarrow \underset{\text{Magnetite}}{\underset{\uparrow}{Fe_3O_4}}$$

The mechanical properties of epoxy can be improved by the introduction of strong inorganic fillers by forming polymer nanocomposites (PNCs), which can also have unique properties such as optical[6], anticorrosive[7], electrical[8,9], and magnetic properties[10], In addition to that its high resistance to corrosion. Electrochemical impedance spectroscopy (EIS) was used to study the effects of this polymer on carbon steel as a corrosion inhibition [10]. Typically, this technique measures the system impedance across a range of frequencies, and therefore the system's frequency response, including energy storage and dissipation properties, is detected.

2. Experimental

2.1 Raw materials

Several chemicals from various companies were used in this study, such as aniline, Ferric chloride hexahydrate & ferrous sulfate heptahydrate, Hydrochloric acid, and Sodium persulfate. epoxy resin, tri-ethylene tetra-amine and carbon steel alloy (C1020) were Supplied from the local market. The physical properties of cured epoxy resin and carbon steel composition are showed in table1, 2 respectively. Aniline monomer were distilled under Reduced pressure before use while other reagents were used without any further treatments.

Table 1: Physical properties of cured epoxy resin used in the research

shade nos/ Colors	Glass transition Temprature (Tg)	Volume solids, %	Thermal degradation	Yield stress
210/ White	50 ˚C	75± 1	340 ˚C	22.8±2.4 Mpa
	Surface dry	flashpoint	VOC content	Impact strength(KJ\m^2)
	12 hours 20C° /68°F	>200 ˚C	1 pound/gallon VOC equals 120 grams/liter	1.2

Table 2: Chemical composition of Carbon Steel alloy

C %	S%	Mn%	Si%	Cr%	Ni%	Cu%	As%	Fe%
0.086	0.028	0.55	0.015	0.023	0.0114	0.0187	0.01	Balance

2.2 Synthesis of Polyaniline (PANI)

(0.054 mol) from aniline was dissolved in (54ml) 1M HCl solution. the mixture was cooled up to (-5 c°) using ice crushed. Then (32 ml) of sodium persulfate(SPS) was added to the mixture gradually as drops with the reaction mixture constantly stirred, and the temperature kept constant at (-5 c°). The residue was collected in a Buchner funnel. After that the dark-green precipitate was washed with dilute ammonia (25%) for a time of (7h). the \Blue-black product was rinsed with deionized water until the filtrate had a pH of 7 and then dried in vacuum at 60°C for 24 h[(11)].

2.4 Synthesis of Magnetite nanoparticles (Fe_3O_4)

Iron oxide nanoparticles were prepared on the basis of co-precipitation Fe^{3+} and Fe^{2+} with a molar ratio of 2:1 respectively[(12,13)]. 2.7 g of $FeCl_3 \cdot 6H_2O$ and1.2 g of $FeSO_4 \cdot 7H_2O$, were dissolved in 100 ml of deionized water under nitrogen. The mixture was stirred vigorously using a magnetic stirrer for one hours at 80°C. Then, (15 ml) of (32) ammonium hydroxide was added to the mixture gradually as dropwise with the reaction mixture constantly stirred. When ammonium hydroxide was added, a precipitate was immediately produced(14,15). The dark precipitate was washed with bid stilled water six times to remove excess of ammonium ions. Finally, the humid suspension was evaporated and dried for 24 hours.

2.5 Synthesis of polyaniline/iron oxide magnetic nanoparticles composite

Fe_3O_4/PANI magnetic nanoparticles composites were prepared(16) by adding (1 mol l^{-1}) of polyaniline (as in paragraph 2.2) to the reaction mixture containing (2 mol l^{-1}) of aqueous ferric chloride, (1 mol l^{-1}) of aqueous ferrous sulfate, and 100 ml of distilled water, the mixture is placed in three-necked round-bottom flask equipped with a mechanical stirrer, the reaction is carried out at a temperature of 80°C,for one hour in an inert atmosphere. after that the mixture was cooled and then added 15 ml ammonium hydroxide solution of concentration (32%) to the mixture as drops gradually, nano magnetic particles were precipitated. The precipitate was washed with bidistilled water six times to remove excess of ammonium ions. Finally, the humid suspension was evaporated and dried for 24 hours. After the drying process, Fe_3O_4/PANI nanocrystals were ground.

3. Preparing the carbon steel alloys

In this study, carbon steel alloy C1020 was used, , whose components are shown in Table2, was used, where it was washed with distilled water several times, then washed with ethanol acetone to remove traces of water, then dried in the electric oven, and also used sanding paper contrasting in smoothness with different numbers from (180 to 3000) For the purpose of polishing these alloys.

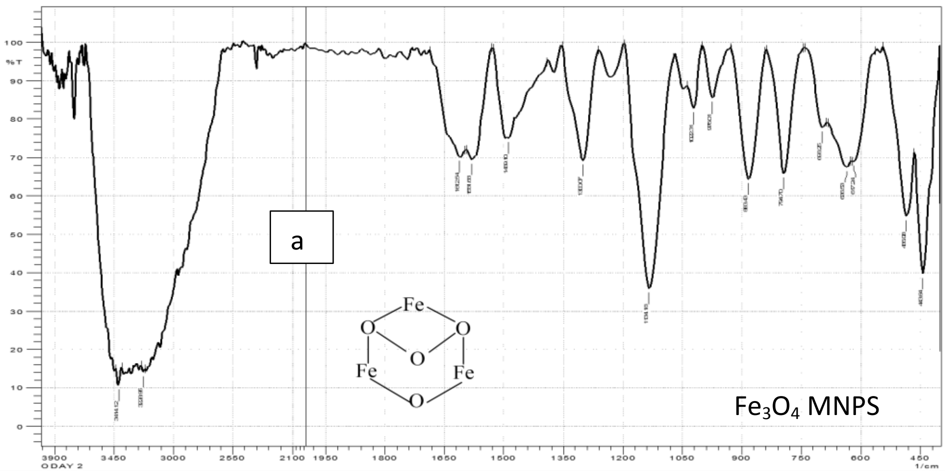
4. Coating of Alloys

In this study the alloy was first coated with epoxy resin and tri-ethylene tetra-amine hardener in a ratio of 3:1, by taking a weight of (0.3 g) of paint then distributed it homogeneously over 1cm^2 area of the alloy's surface. The thickness of the paint layer is 1 mm. A small area of the alloy was left without coating for the purpose of electrical contact with the working electrode. Then the second model was coated with Fe_3O_4, taking a weight as (5%), which corresponds to a weight of (0.015g) of Fe_3O_4, and (0.285 g) of the epoxy resin with the hardener , and mixed it together in a homogeneous way, and so on with the rest of the percentages to obtain 10, 15, 20 and 25% of coating mixture.

5. Results and discussion

5.1. FT-IR analysis

The FT-IR spectra of Fe_3O_4 MNP_S, and polyaniline MNP_S are shown in Figs 1a and 1b respectively.The Figures showed that characteristic Absorption band of Fe-O bond[(15,)(17)] is at 570 cm^{-1}, whereas in the polymerMNP_S was appeared at 617 cm^{-1}. The characteristic band at 3414 cm^{-1} corresponds to the N-H stretching of the primary amine. The Absorption Band at 1582 cm^{-1} is assigned to the quinoid ring stretching For Polyaniline MNPS. The Band at 1489 cm^{-1} corresponds to the presence of C=C stretching vibration in benzenoid ring in the polymer. The sharp peak at 883 cm^{-1} attributes the para-coupled phenyl ring of polyaniline MNP_S and the peak at 1300 cm^{-1} and corresponds to the C-N stretching vibrations of the aromatic primary amine[(13,17)] in PANI MNP_S .



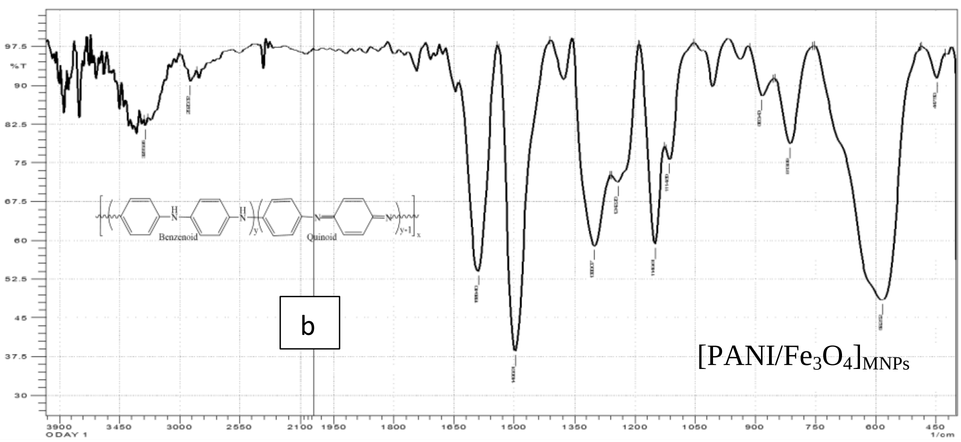
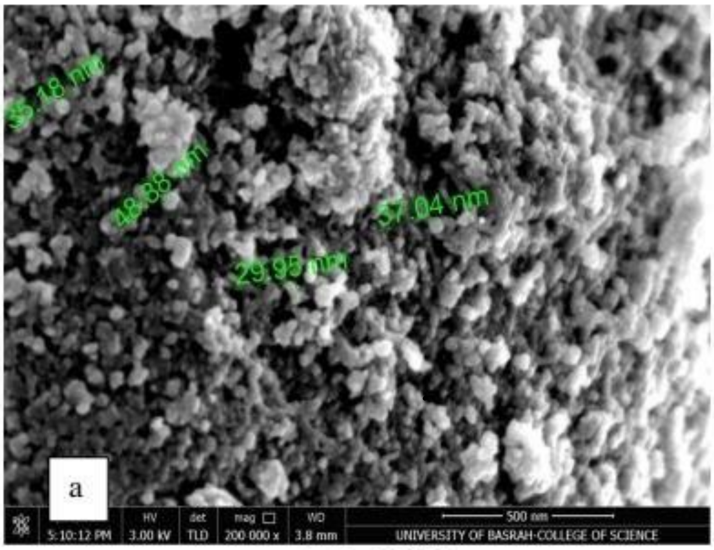


Figure1:

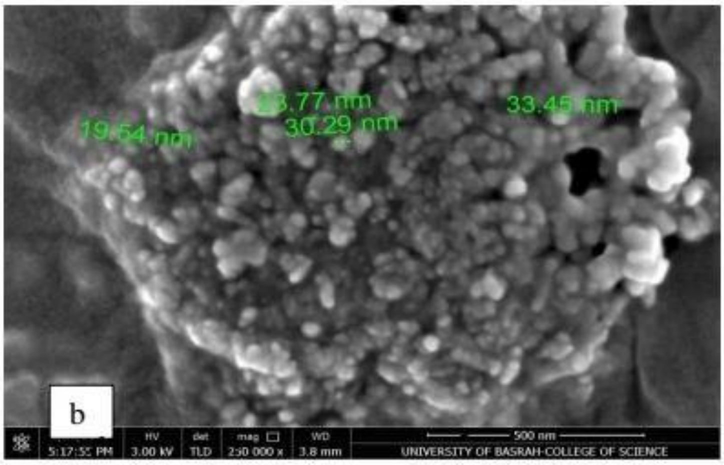
a. *(a) FT-IR spectrum pattern of as-prepared Fe_3O_4 /MNPs ;*
b. *(b) FT-IR spectrum pattern of as-prepared Polyaniline MNPs.*

5.2. SEM analysis

The scanning electron microscope (SEM) images of Fe_3O_4 MNP_S, and Polyaniline MNP_S are shown in figures 2a and 2b respectively. Where it was found that the size of nanoparticles ranges (19.54 - 48.88)nm, It has a narrow size distribution with high morphology.



Fe_3O_4 MNPS



[PANI / Fe_3O_4] $_{MNPs}$

Figure2. *(a) SEM image of Fe_3O_4 MNPS; (b) SEM image of Polyaniline MNPS*

5.3. EIS analysis

Data obtained by EIS is graphically expressed in a Nyquist or body diagram as shown in Figures 3 to 5, In order to better analyze the characteristics of the electrochemical impedance EIS, the measurement data was processed by CS Studio specifically using Z view to obtain the rest of the relevant information. There are two types of drawings obtained from this technique, namely Nyquist curve and Bode curve for carbon steel alloys coated with a mixture of specific weight ratios of Fe_3O_4/MNP_S and polyaniline MNP_S combined with epoxy resin in the presence of a solution (0.1 M) of hydraulic acid, as shown in Figure 3A, 2A and 3A. EIS data for the inhibitors was calculated using an electrical equivalent circuit in a frequency range of (0.01-105) Hz, with an AC current signal of 10 mv. there are many variables that appear during the analysis of experimental EIS results, and all EIS data have been included in table (3), such as the solution resistance Rs[(18)], and the polarization resistance of the metal surface Rp, which reflects the property Protection [(18)], and the constant phase element CPE, which is called Y_0 measured by units ($\Omega^{-1} S^n$), and (n) is the phase shift, which can be used as a measure of the roughness of the metal surface, as for the capacity of the double layer double layer- capacitance (C_{dl}), which is the layer formed by the coating on the surface of solution [(18-21)] measured by ($\mu F.cm^{-2}$) and calculated from the equation (1) below:

$C_{dl} = 1/(2\pi R_P F_{max})$ ------ (1)

IE% damping efficiency can be calculated using the equation below:

$\boldsymbol{IE\%} = \frac{\mathbf{Rp(inh)-Rp}}{\mathbf{Rp(inh)}} \times \mathbf{100}$ ------ (2)

Where $R_{p(inh)}$ and R_p are polarization resistance with the presence and absence of Inhibitor respectively. While the relaxation time (τ)[(22)] values can be calculated using the following equation:

$\tau = C_{dl} R_P$ --------- (3)

The addition of magnetite, PANI and MNP_S with different percentage of weight to the epoxy coating does not change the corrosion mechanism but it prevents corrosion by increasing the surface coverage, while increasing the efficiency inhibition by the adsorbent diaphragm membrane. It improves the barrier properties of the coating and enhances the corrosion protection of the coating It increases the adhesive force of the coating on the surface of the metal alloy and thus works on the physical separation of the metal alloy from the corrosive medium[23, 24]. This indicates the efficiency of the coating during its process of combining it with the magnetic nanoparticles of the inhibitor, as it prepares all corrosive types such as water molecules, oxygen, and other ions[25], so it is very difficult to penetrate the coating layer by the electrolyte solution. When observing the figure 5A, we observe that node amplitude in the Nyquist plote of $PANIMNP_S$ and it combined with epoxy paint is greater by comparison with the node amplitude of polyaniline NP_S and Fe_3O_4. The node's capacity of polymer is also larger compared to epoxy paint alone, As this capacity increases, the percentage of inhibitor increases, this indicates the efficiency of the paint while it is combined with Fe_3O_4, It prevents all types of corrosive substances such as water molecules and other ions[26]. It was also observed that by increasing the percentage of an inhibitor, the inhibitory efficiency of it increased, as indicated in the table (3), It is also clear from this table that the values of (R_P) also increase with the percentage increase of Fe_3O_4 MNPs and Polyaniline MNP_S, This increase in Rp values leads to a decrease in corrosion and an increase in efficiency inhibition[25]. The protective layer of retarder on the wear surface is made of carbon steel alloy, which constitutes an obstacle to the transport of the charge. While, the decrease in the damping efficiency of some inhibitors may be attributed to the occurrence of a deterioration of the coating layer due to roughness and heterogeneity of the coating surface and its exposure to some structural deformation as a result of saturating the electrolyte coating and the occurrence of irregular electrochemical corrosion reactions on the alloy surface [27]. On the other hand, adsorption of Fe_3O_4 MNPs inhibitor, and the polymer MNPs on the corrosive surface of the alloy, constitute an impediment to the transport of the charge and the closure of active sites subject to corrosion. As for the values of A, they vary between increase and decrease as indicated in Table (3), where this difference leads to the formation of a protective layer on the surface of the metal alloy and reduces the process of transfer of charge and displacement of water molecules by the inhibitor molecules at the interface of the solution (metal / acid), and this means that Cdl was used To determine the damping ability, it indicates that these inhibitors inhibit the rate of corrosion in alloys by means of the adsorption mechanism, That is, to reduce the extent of dissolution of the metal. The decrease in the cdl value of the magnetite and polymer inhibitor at the highest percentage may be attributed to the decrease in the value of the dielectric constant and an increase in the thickness of the double layer as a result of absorption of the damper on the surface of carbon steel, as the value of C_{dl} is related to the thickness of the protective layer by adopting the following equation:

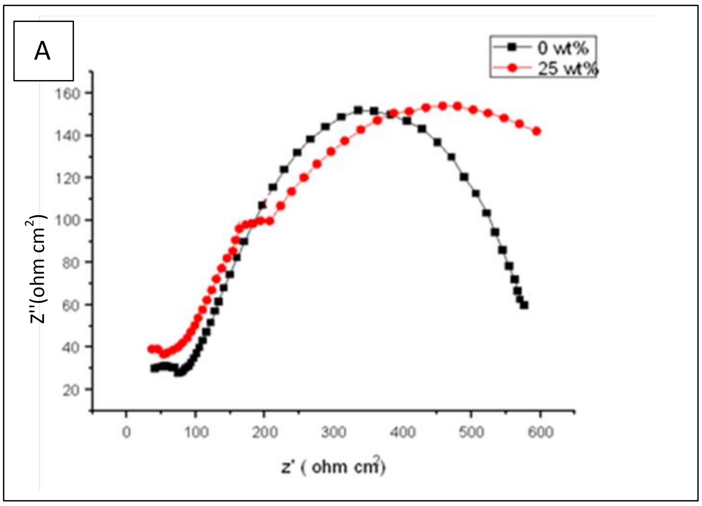
$$\mathrm{Cdl} = \frac{\varepsilon\, \varepsilon_{\circ} A}{d} \quad \text{-----------(4)}$$

Where (ε) is local dielectric constant of solution, ($\varepsilon_{\circ}$) is permittivity of free space (8.8542× 10-12 F m^{-1}), (d) is protective layer thickness and (A) is electrode surface area[28].

As for the values of **n** (phase displacement) it gives information about the smoothness and roughness of the surface of the coating, where if the value of n is small this means that the surface of the coating is rough, while if a high value indicates that the surface is smooth and more uniform [29, 30], From Table (3) we note that the value of (n) at the percentage 25 % is the highest value in the polymer compound, in the magnetite compound at the percentage of 25 % the value of (n) is the highest value. As this increase in the values of n is due to the dense absorption of inhibitor particles on the alloy surface, which leads to a slow process of alloy corrosion, moreover an increase in the inhibition efficiency due to an increase in the percentage of inhibitor weight. As for the Bode plote as in Figures 3B, 4B and 5B, the simulation results are considered according to the proposed electrical circuit in Figure 4, and the excellent relationship is shown in the data processed and measured (separate points), and it also shows the maximum phase angles of the samples measured in the presence of inhibitors, where we note At the angle value of the phase which gives excellent anti-corrosion inhibition efficiency [31]

Table3: EIS parameters for the coated alloy in the presence of a mixture of polyaniline MNPs and magnetite with epoxy resin in 0.1M HCl at 298K.

Inhibit.	Wt. % of inhibit	Rp ($K\Omega.cm^2$)	n	$Y_\circ$*10^6 ($\Omega^{-1} s^n cm^{-2}$)	C_{dl} ($\mu F\ cm^{-2}$)	τ*1000 (s)	F_{max} (Hertz)	% IE
Epoxy resin	-	687	0.5 010	0.000583	1.7211E-5	0.1182	13.467	-
Polyaniline	25	991.2	0.9071	0.001618	1.4617E-7	0.1448	1099	31%
Fe_3O_4 MNPs	5	1564	0.3269	7.5938 E-	1.7553E-7	0.2745	580	56%
	10	1672	0.4371	1.0877E-4	1.2651E-7	0.2115	752.76	59%
	15	1838	0.4493	1.1424E-4	2.2217E-7	0.4083	389.94	63%
	20	2419	0.4138	1.936E-4	9.6982E-8	2.3459	678.75	72%
	25	1771	0.6097	4.7207E-4	3.006E-7	0.5324	299.1	61%
Polyaniline MNPs	5	1351	0.5239	0.046485	1.3169E-7	0.1779	895	50%
	10	1958	0.8246	4.6816E-6	8.1336E-7	1.5925	99.987	65%
	15	2658	0.9201	8.8555E-6	1.0422E-6	2.7701	57.481	74%
	20	2868	0.8428	0.4764E-6	2.8312E-7	0.8119	196.1	76%
	25	4992	1.028	0.5069E-6	4.6165E-7	2.3045	690.95	86%



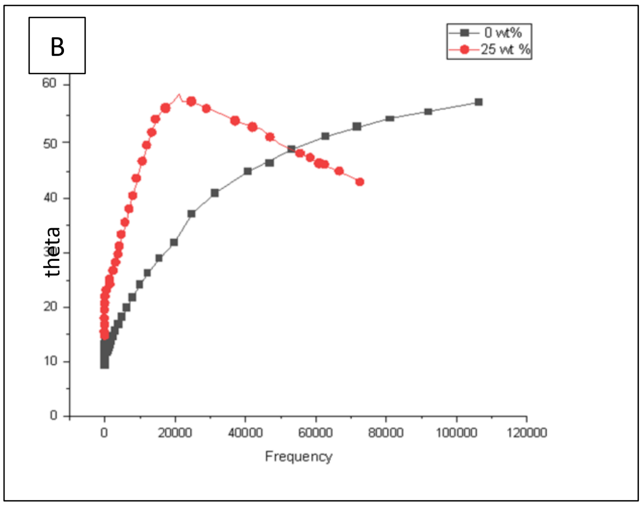


Figure 3.*(A) Nyquist plote, (B) Bode plote of Polyaniline NPS with epoxy resin against of 0.1 M HCl at 298K.*

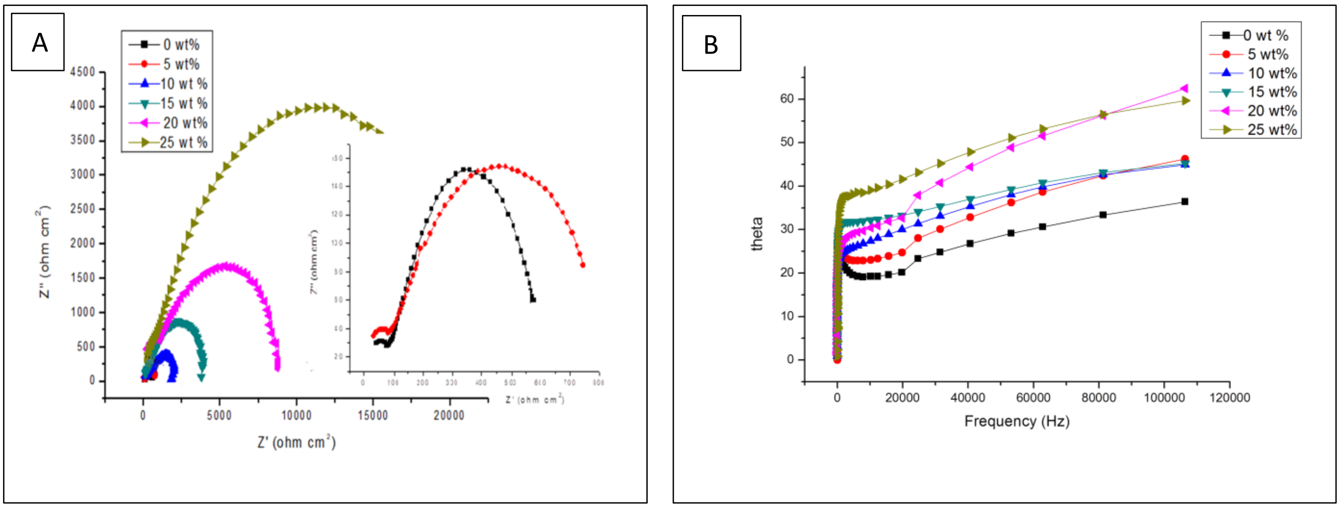


***Figure 4**: (A) Nyquist plot; (B) Bode plot of Fe_3O_4 MNPS with epoxy resin against of 0.1 MHCl at 298K .*

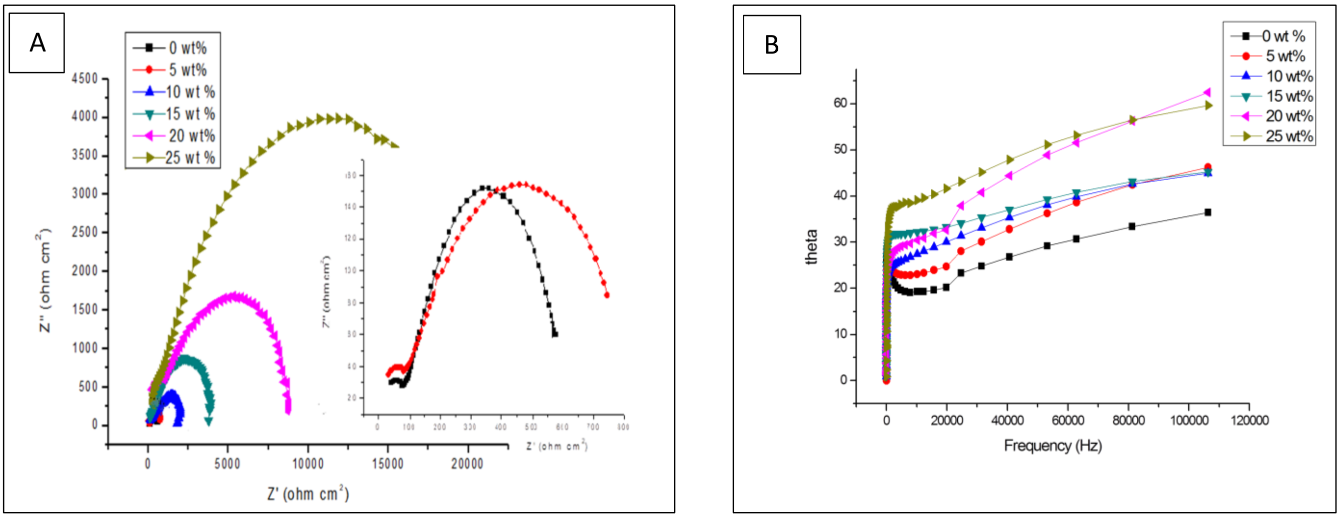


***Figure 5**: (A) Nyquist plot, (B) Bode plot of Polyaniline MNPS with epoxy resin against of 0.1 M HCl at 298K*

6. Conclusion.

Epoxy resin, polyaniline and Fe_3O_4, were used to obtain magnetic nano-coating, as the carbon-steel alloy was once coated with epoxy resin paint alone, and again the polymer or magnetite was combined with the epoxy resin in percentage ranges (5-25%). The EIS results demonstrated that the epoxy coating combined with the polymer magnetic nanoparticles and magnetite compound has an effective and strong barrier against the corrosive environment compared to epoxy paint alone. Also, the paint combined with polyaniline was found to have higher inhibition efficiency than the epoxy paint mixture with magnetite, as this increase in the inhibition efficiency is due to the high R_p value of the (epoxy resin - polymer) MNPs coating, especially at the highest percentage of the inhibitor. As increasing the percentage of the inhibitor improves the wear resistance of the coating, and leads to the formation of a protective layer of the inhibitor on the surface of the carbon steel alloy, as the coating reduces the electrical capacitance by displacing water molecules or any ions adsorbed on the surface of the metal. However, the (epoxy - polymer) coating improves the properties of the coating, enhances the adhesive force of the coating on the surface of the metal alloy, and therefore works on the physical separation of the alloy from the corrosive medium, so that it is very difficult to penetrate the coating by electrolyte solution, thus the coating efficiency increases due to the increase of the inhibitor efficiency in the damping. We also notice a clear decrease in the values of the C_{dl} inhibitor of polyaniline from the values of C_{dl} in the magnetite inhibitor, and this indicates an increase in the inhibition efficiency of the inhibitor polymer due to the increase in the thickness of the protective layer that reduces the wear rate of the metal alloy.

7.Referencess

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