Effect of Secondary Species on Inhibition Efficiency of Fatty Amide Mixtures in Dynamic Environment

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Abstract-The corrosion inhibition of carbon steel in 3.5wt% NaCl solution by fatty amides with addition of hydrazine, N_2H_4 in moving condition has been studied using linear polarization resistance measurements and scanning electron microscope technology with energy disperse X-ray spectroscopy (SEM-EDX). The effect of different moving condition was studied using rotating cylinder electrode, RCE at different rotational speeds (0, 1, 5 and 50 rpm). Results have shown that for uninhibited conditions, the corrosion resistance, Rp values decreased drastically as rotation speed increased up to 50 rpm. For inhibited solution, Rp values are highest under static condition and it decreased gradually with increase in rotational speed. It was found that, the inhibition efficiency enhanced by adding hydrazine into the solution containing fatty amide inhibitor due to multiple protections provided by both, fatty amides and hydrazine.

Keyword-Fatty amides, Hydrazine, Moving environment, Corrosion inhibition, Polarization resistance

I. INTRODUCTION

The high cost of corrosion is an important issues worldwide and it is a crucial part to reduce it in petrochemical, oil and gas industries [1]. Most of aqueous environment can promote corrosion which occurs under several complex conditions in oil and gas production. One of the most frequent and corrosive environments found in the oil and gas industries are fluids with high concentrations of chlorides [2].

There are many structural component used in oil and gas extractions and processing are made from carbon steels. However, they are susceptible to corrosion in aggressive environments and then leading to severe damage [3]. Therefore, organizations are looking for a practical and economic alternative way in combating corrosion problem. Presently, the use of corrosion inhibitors has become an alternative practice for protection of metals that are in constant contact with aggressive environment [4, 5].

Most effective inhibitors are organic compounds that consist of heteroatoms like nitrogen, sulfur, phosphorous and oxygen. They function at the interface between the metal surface and aqueous corrosive solution through adsorption mechanism forming a protective film on the metal surface [6, 7]. The inhibition efficiency decreases in the following order, O>N>S>P. The adsorption of organic inhibitors through physical adsorption or chemical adsorption can change the corrosion resistance properties of metals [7].

`Studies have found an occurrence of synergistic inhibition effect when a secondary compound is added into solution containing the organic inhibitor [8, 9]. Synergistic inhibition effect is an improved performance of inhibitors mixture as compared to merely individual inhibitor on the rate of corrosion reaction [10, 11]. It has proven to be an effective way to decrease the amount of organic inhibitor consumption and to expand the application of organic inhibitor in media [12, 13]. The secondary species could be a metallic ion which would increase the inhibition efficiency of organic compound with complex formation [14-16]. Halide ions were also found to be a type of secondary species which pre-adsorb on metal surfaces and then improve the adsorption of organic inhibitor [17-19]. It acts as interconnector between inhibitor species and metal surface [20]. Secondary species also can be other organic molecule which enhances the adsorption of the primary species [21, 22].

The roles of the individual fatty amide organic inhibitor in the inhibition mechanism have been reported by previous studies as conducted by [Ismayilov, *et al.* [2], Zaafarany and Abdallah [23], Hashim, *et al.* [24], Ibrahim, *et al.* [25]] , however, there were no references reported on the effect of fatty amides mixture inhibitor with existence of secondary species in the inhibition system.

The aim of the present study is to investigate the effect of corrosion resistance of fatty amides with addition of secondary species hydrazine (N_2H_4) in static and moving environment. Linear polarization resistance measurement (LPRM) was used to measure the corrosion resistance, Rp. The interest of this research project is to develop an improved inhibition performance when applied in dynamic condition. This study was conducted using rotating cylinder electrode, RCE equipment.

II. EXPERIMENTAL

The elemental composition of carbon steel specimen used in this study was analyzed by using Inductively Coupled Plasma (ICP) with Optical Emission Spectrometer (OES) and CHNS analyzer and had the composition (wt%) 0.04 C, 0.23 Al, 0.74 Mn, 0.056 Ni, 0.059 Cu, 0.060 Cr, 0.54 Zn, 0.02 Pb and the remainder is Fe. Fatty amide mixtures inhibitor was synthesized from palm fatty acid distillate (PFAD) and isopropylamine according to chemical routes described by Cossy and Pale-Grosdemange [26]. Hydrazine is an analytical grade reagent purchased from Merck and was used in as-received condition.

Electrochemical tests were carried out in a rotating cylinder electrode, RCE (Model 636) connected to a Gamry potentiostat shown in Fig 1(a). The specimens which act as working electrode had a dimension as shown in Fig 1(b), with an exposed area of 3.02 cm^2. The specimens for corrosion test were prepared prior to the experiment by grinding with SiC paper (#200, #600 and #1000) and then washed with distilled water and acetone. Then, specimens were dried in air and immediately immersed in the test solution. The reference electrode was a saturated calomel electrode and a platinum electrode was used as an auxiliary electrode.

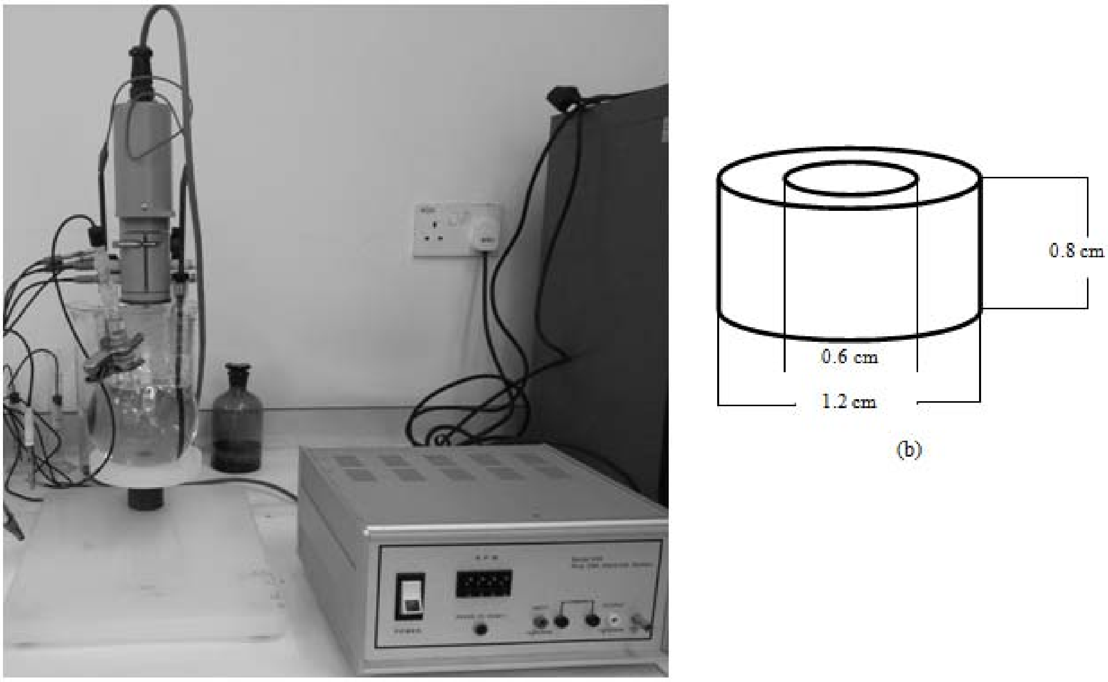


Fig 1. (a) Rotating cylinder electrode (RCE) set up (b) Dimensions of the working electrode for RCE tests

The working electrode was immersed in test solution for 60 minutes to a establish steady state open circuit potential (E_{ocp}). After measuring the E_{ocp}, linear polarization resistance (LPR) measurements were performed at 298 K to determine the inhibition performance on carbon steel specimens in 3.5 wt. % sodium chloride solution with and without inhibitors. Constant concentration of fatty amides mixture (20 ppm) with addition of different concentration of hydrazine (500, 1000, and 2000 ppm) were tested in the 3.5 wt. %, NaCl solution. The systems were tested under different dynamic conditions that is, 0, 1, 5 and 50 rpm to represent static, laminar, transition and turbulent conditions in pipe-line flow. The surface morphology of the polarized samples was investigated using scanning electron microscope equipped with an Energy dispersive X-ray spectrometer for chemical analysis.

III. RESULTS AND DISCUSSION

A. Electrochemical test

Fig. 2(a), 2(b), 2(c) and 2(d) show the Polarization resistance, Rp values evolution changes over 1 hour immersion time on different speed rotations; 0, 1, 5, and 50 rpm. The solution consists of addition of 20 ppm fatty amide mixtures together with different concentration of hydrazine (500, 1000 and 2000 ppm) in 3.5wt% NaCl solution.

At static condition (0 rpm) Rp values in every system are highest as shown in Fig 2(a**)**. It reaches maximum value 7084 ohm.cm^2 (at addition of 20 ppm fatty amide with 2000 ppm hydrazine), compared to the Rp values at rotation speed conditions; 1 rpm, 5 rpm and 50 rpm which reached a maximum value, 974, 883 and 527 ohm.cm^2 , respectively.

At static condition, when 20 ppm fatty amides mixture was added to the 3.5 wt% NaCl solution (blank solution), it shows definite trend of inhibition as the Rp values were slightly increased compared to the Rp values in blank solution. Even though 20 ppm concentration of fatty amides seems a small concentration used but it is able to exhibit surface protection on specimen surface at static condition. From the result obtained, small concentration (20 ppm) of fatty amides gives a constant corrosion resistance about 1394 ohm.cm^2. This result is supported by study from Chen, *et al.* [27] which reported it required small amide inhibitor concentration to give constant corrosion resistance as well as constant corrosion rate.

The surface protection provided by fatty amides mixture consists of mixture of amides which includes acetamide (C_2H_5NO) , acetylamide (C_2H_4NO), butyramide (C_4H_9NO), myristamide ($C_{14}H_{29}NO$), stearamide ($C_{18}H_{37}NO$), oleoamide ($C_{18}H_{35}NO$) [25]. These amides consist of nitrogen element in their molecules. The free electron pair in nitrogen atoms can adsorb onto iron surface that consist of vacant d-orbital in the structure then forming protective layer. Besides that, the various length of hydrocarbon chain in amides mixture formed hydrophobic film on the surface thus, providing strong surface resistances to the inward diffusion of dissolved oxygen molecules to the metal surface. When different concentration of hydrazine is introduced into the inhibited solution that contained 20 ppm amides mixture, the corrosion resistance, Rp drastically increased. Rp-values were increased when an increased in the concentration of hydrazine was made.

However, in dynamic condition, addition of only 20 ppm fatty amides mixture cannot exhibit corrosion resistance on the metal surface as shown by the variation of Rp with time in Fig 2(b), 2(c) and 2(d). It shows the addition of an arbitrarily chosen amount of fatty amides mixture could not exhibit corrosion resistance on the steel surface in a dynamic condition. Performance of fatty amide mixtures in more vigorous surrounding (e.g rotation speed of 50 rpm) became worst with measured Rp values declining to about126 ohm.cm^2.

When some concentrations of hydrazine were introduced into the inhibited system at different dynamic conditions, a remarkable increasing in Rp values were observed. The Rp values were increased when concentration of hydrazine was increased up to 2000 ppm. Hydrazine acts as an oxygen scavenger in the test solution.

Hydrazine is a reducing agent reacts with the dissolved oxygen molecules available in the mixture by following chemical reaction (equation 1) [28];

$$N_2H_4 + O_2 \rightarrow N_2 + 2H_2O \qquad \text{(Equation 1)}$$

The dissolved oxygen is an important element in order to complete the hydrogen evolution reaction at cathode part in corrosion process as following reaction (equation 2) [29];

$$O_2 + 2H_2O + 4e^- \rightarrow 4OH^- \qquad \text{(Equation 2)}$$

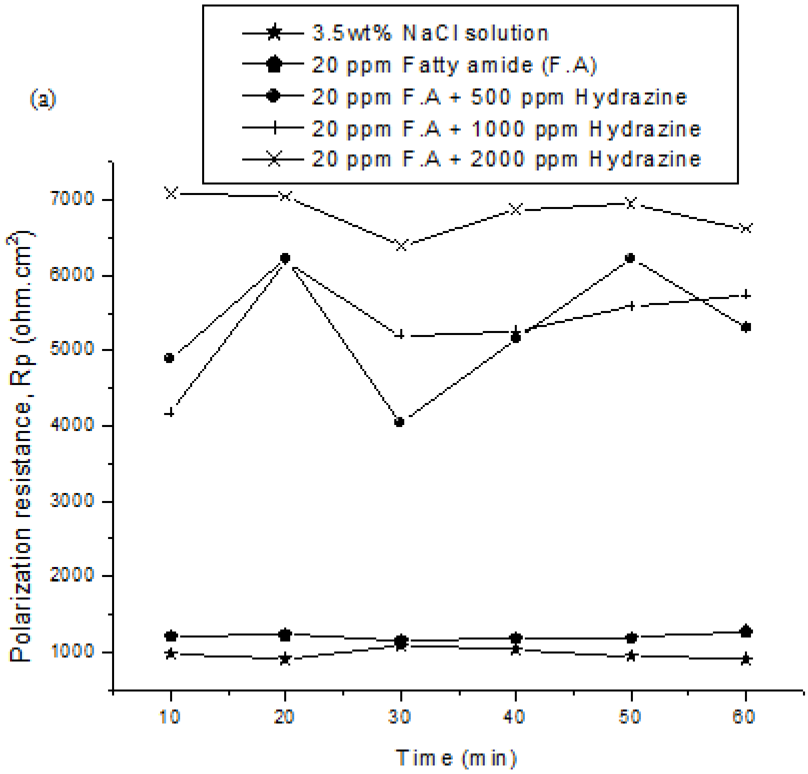
When amount of dissolved oxygen molecules had depleted in the test solution due to reaction occurred between hydrazine and oxygen, the cathodic reaction would be decreased and which in turn also would decrease the anodic reaction [30]. Thus, it results high in corrosion resistance and low in corrosion rate which in turns is being shown by an increase in Rp values.

These results suggest that addition of synthesized inhibitor and a secondary element, hydrazine retard the hydrogen evolution reaction and reduce anodic dissolution. It shows that fatty amide mixtures and hydrazine provide multiple inhibition protection. It is well known that the corrosion inhibition provided by fatty amides mixture is an adsorption process that occurred on steel surface. While by introducing hydrazine in the inhibited solution, it tended to give interaction in the medium solution by eliminating the dissolved oxygen in the test solution that induced to corrosion process.

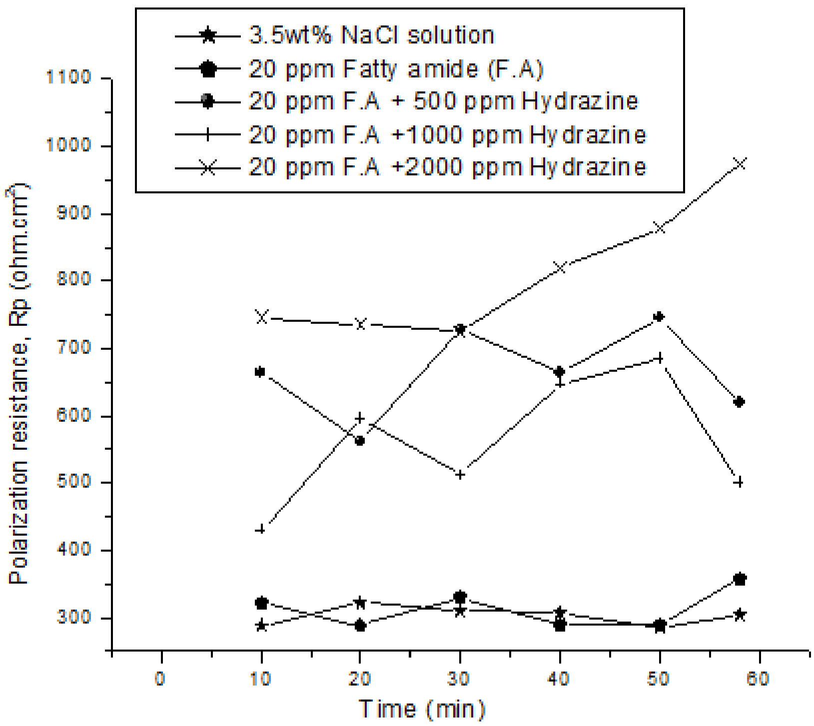
Fig. 3 and Table 1 shows the inhibition efficiency, IE (%) calculated by the polarization resistance after 1 hour immersion according to the following equation (Equation 3):

$$IE = \frac{R_p - R_{po}}{R_p} \times 100\% \qquad \text{(Equation 3)}$$

(a)


3.5wt% NaCl solution
20 ppm Fatty amide (F.A)
20 ppm F.A + 500 ppm Hydrazine
20 ppm F.A + 1000 ppm Hydrazine
20 ppm F.A + 2000 ppm Hydrazine
Polarization resistance, Rp (ohm.cm2)
Time (min)
1000
2000
3000
4000
5000
6000
7000
10
20
30
40
50
60

(b)


3.5wt% NaCl solution
20 ppm Fatty amide (F.A)
20 ppm F.A + 500 ppm Hydrazine
20 ppm F.A +1000 ppm Hydrazine
20 ppm F.A +2000 ppm Hydrazine
Polarization resistance, Rp (ohm.cm2)
Time (min)
300
400
500
600
700
800
900
1000
1100
0
10
20
30
40
50
60

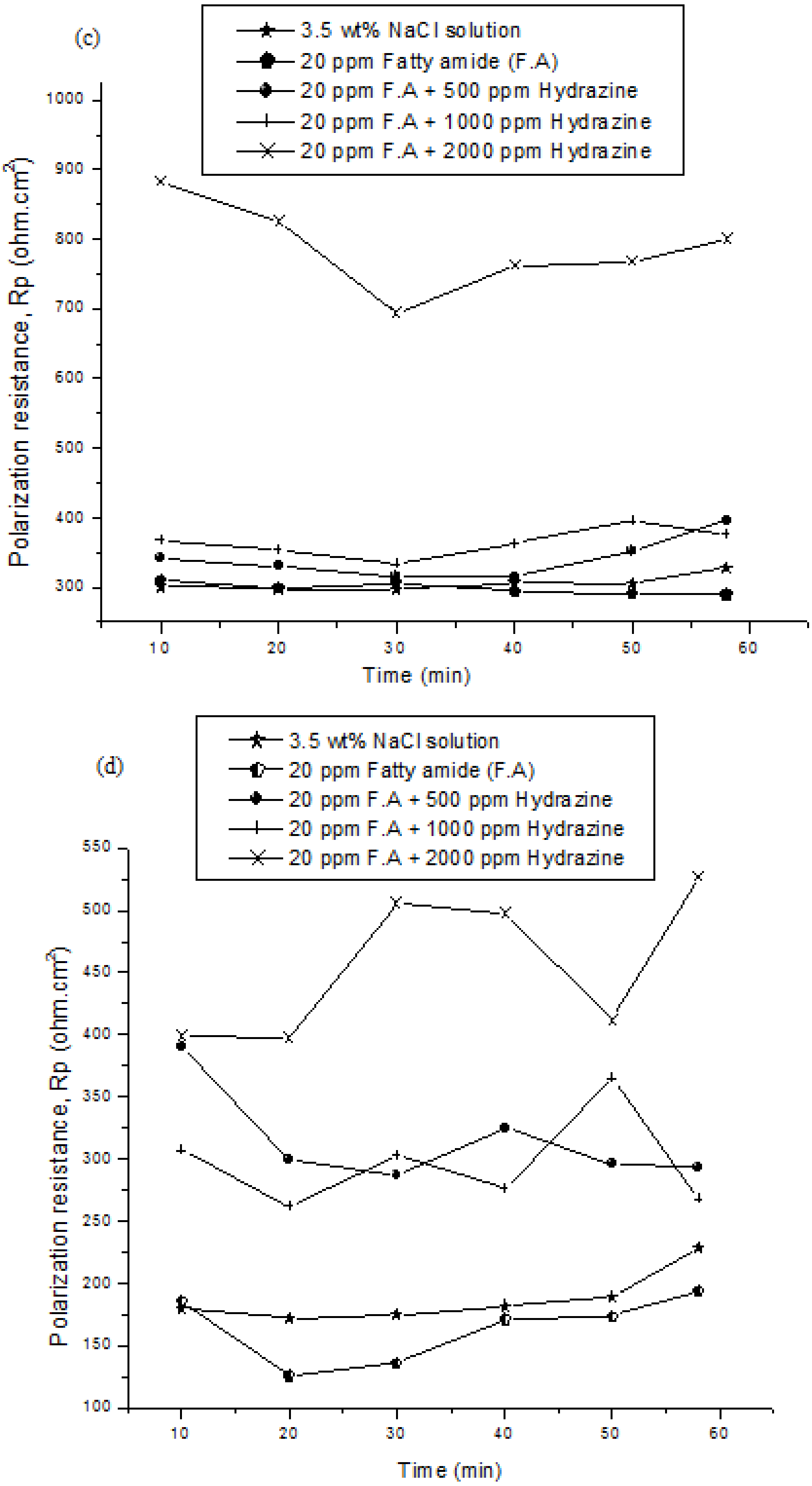


Fig 2. Variation of polarization resistance, Rp with immersion time containing 20 ppm fatty amide and different concentration of hydrazine (500, 1000, 2000 ppm) in (a) 0 rpm (static) (b)1 rpm (c) 5 rpm (d) 50 rpm rotational speed.

Where R_p and R_{po} are inhibited and uninhibited polarization resistance values, respectively. The graph pattern (Fig. 3) shows the influence of rotation speed of fluid on the inhibition efficiency in each system having 20 ppm fatty amide mixtures with different concentration of hydrazine (500, 1000 and 2000 ppm).

When the rotation speed of fluid was increased from 0 rpm up to 5 rpm, the inhibition efficiency was reduced to some point but when rotation speed was increased up to 50 rpm, the inhibition efficiency was increased. This behavior was seen for systems; 20 ppm fatty amide with 500 ppm hydrazine and 20 ppm fatty amide with 1000 ppm hydrazine.

It could be said that in rotation speed of fluid at 5 rpm, the chemical adsorption of fatty amide on the specimen surface is unstable making them break and detach from the metal surface. This phenomenon was also reported by other researcher [31].

However, when rotation speed of fluid was increased up to 50 rpm the inhibition efficiency is increased. This phenomenon could be explained by the increase of inhibitors mass transfer onto the specimen surface [31]. Larger amount of inhibitors were physically or chemically adsorb and react on the specimen surface in 50 rpm rotation speed.

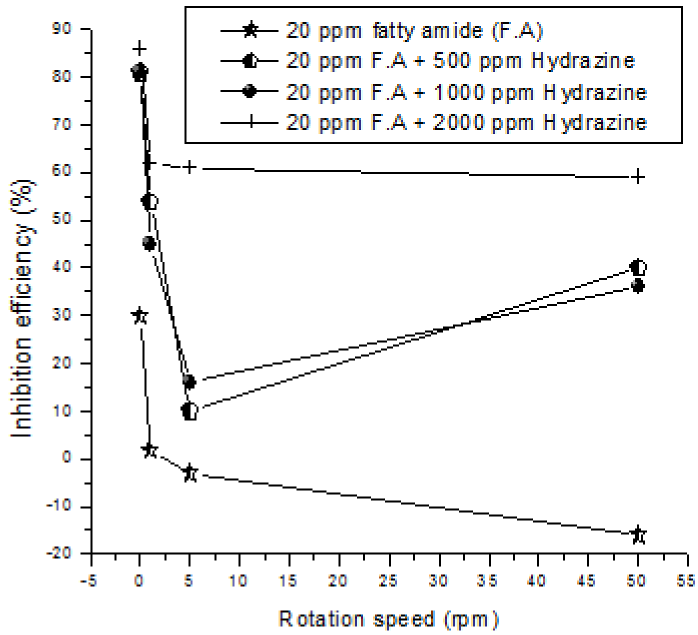


Fig 3. Influence of rotation speed on inhibition efficiency

B. Surface analysis

The SEM micrographs in Fig. 4 (a) shows carbon steel surface were severely damaged with surface cracking after immersions in 3.5wt% NaCl solution without inhibitor addition (blank solution) at 50 rpm rotation speed. This is due to the penetration of aggressive species onto the unprotected metal surface in dynamic condition. Fig. 4(b) and (c) show the sample surface in presence of 2000 ppm hydrazine in the inhibited solution containing 20 ppm fatty amide. At 1 rpm rotation speed, the sample surface is in good condition without corrosion products and surface cracking as shown in Fig. 4(b). At higher rotational, the surface morphology shows a moderate inhibition with a few formations of corrosion spot as shown in Fig. 4(c). The above differences between in surface morphologies as shown in Fig. 4(b) and (c) explained a small decreased in the inhibition efficiency, 62% to 59% due to the effect of imposed dynamic condition.

The EDX spectrums are shown in Fig. 5 (a) and (b) show the elemental composition of samples. The composition of each sample is tabulated in the Table 2. These data shows the percentage of Fe, O, Na, and Cl of which indicate the condition of the metal. In the corroded sample (Figure 5a), the percentage of oxygen is higher due to formation of ferrous hydroxide as well as other oxide precipitates [32, 33]. The presence of hydrazine as oxygen scavenger in the inhibited solution has reduced the oxygen content as shown in Fig. 5(b) indicates the decreasing in corrosion product.

TABLE 1. Effect of Rotation Speed of Fluid on Inhibition Efficiency (%)

Fluid velocity (rpm)	Inhibition efficiency (%)			
	20 ppm Fatty Amide (F.A)	20 ppm F.A + 500 ppm N_2H_4	20 ppm F.A + 1000 ppm N_2H_4	20 ppm F.A + 2000 ppm N_2H_4
0	30	81	81	86
1	2	54	45	62
5	-3	10	16	61
50	-16	40	36	59

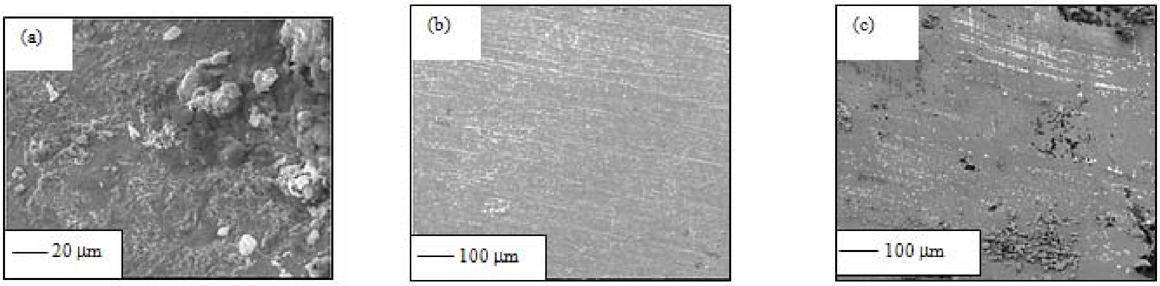


Fig 4. SEM micrographs of sample polarized in (a) 3.5wt% NaCl at 50 rpm, (b) 20 ppm F.A + 2000ppm hydrazine at 1 rpm rotational speed, and (c)) 20 ppm F.A + 2000 ppm hydrazine at 50 rpm rotational speed.

TABLE II. EDX Analysis on polarized samples

	Element (%)			
Sample	Fe	O	Na	Cl
Carbon steel in 3.5wt% NaCl solution (blank) at 50 rpm	82.35	13.87	3.08	0.72
Carbon steel in 20 ppm fatty amide + 2000 ppm hydrazine at 50 rpm	90.48	2.92	4.76	1.84

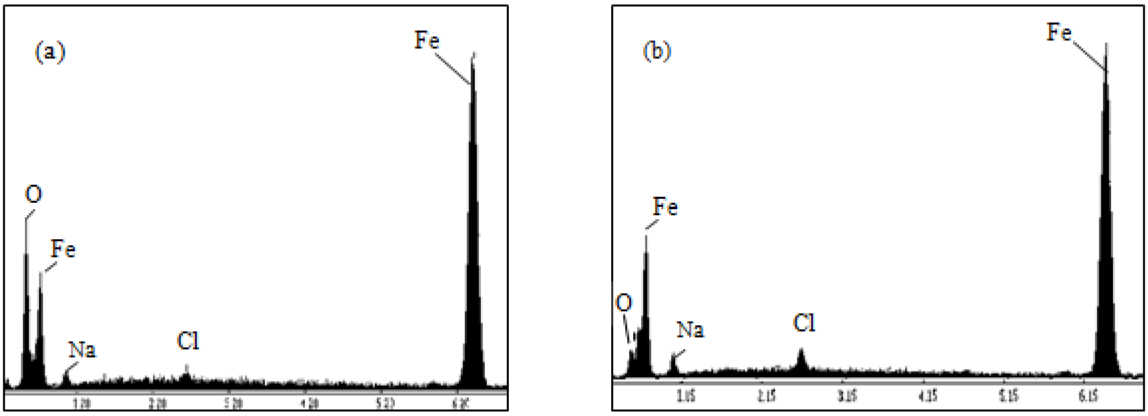


Fig 5: EDAX analysis on polarized carbon steel sample in (a) 3.5wt% NaCl solution at 50 rpm (b) 3.5wt% NaCl containing 20 ppm fatty amide + 2000 ppm hydrazine at 50 rpm

IV. Conclusion

It was observed that the inhibition property was enhanced by adding hydrazine into the test solution containing fatty amide inhibitor. For solution without inhibitors, corrosion resistance, Rp decreased with increasing the rotation speed up to 50 rpm. On the other hand for inhibited solution, the Rp value has its highest value under static condition (0 rpm) and it decreased with increasing in rotational speed.

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