Anti-Corrosion Methods and Materials

Monopropylene glycol based heat transfer fluid: effects of green and synthetic corrosion inhibitors on copper and aluminum with ageing tests

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Abstract
Purpose – The purpose of this paper is to understand corrosion behavior of copper and aluminum in monopropylene glycol-based heat transfer fluid by using synthetic and green inhibitors.

Design/methodology/approach – Weight loss, potentiodynamic and impedance measurements were applied to specimens to obtain their electrochemical characteristics and corrosion behaviors. Ageing test was applied to the fluids that contain different corrosion inhibitors to see the effect of inhibitors on fluid structure, and surface morphologies were examined by scanning electron microscopy (SEM).

Findings – The corrosion tests showed that synthetic inhibitors have better anti-corrosion potential than green inhibitors.

Social implications – Like the synthetic corrosion inhibitors, there is growing interest in green inhibitors. Synthetic corrosion inhibitors are expensive and toxic for live beings, but green inhibitors from naturel sources are easy to reach and non-toxic for live beings and environment.

Originality/value – For solar heating systems, there is a need to select the correct heat transfer fluid; corrosion behavior of fluid plays a major role in the operation because the big part of the heating system consists of copper and aluminum close to ferrius metals and stainless steel.

Keywords Corrosion, Aluminium, Copper, Green inhibitors, Heat transfer fluids

Paper type Research paper

1. Introduction

Solar power as a clean and renewable energy source has received increasing attention in recent years. For solar heating systems, there is a need to select the correct heat transfer fluid (HTF), as it has a large effect on the overall performance (Pacio and Wetzel, 2013). The main requirements of an HTF used in thermal solar systems have been identified in earlier researches. Corrosion behavior of fluid plays a major role in the operation because a big part of the heating system consists of copper and aluminum close to ferrius metals and stainless steel. Working temperature is, also, one of the most important properties of the fluid with low pour point to reduce heat tracing requirements (Becker, 1980; Bignon, 1980).

Heat transfer corrosion is one of the biggest problems for HTFs which consists of the combination of dissimilar metal components, including aluminum, cast iron, brass, solder and copper operating on the thermal solar systems at high temperatures (Zhou *et al.*, 2008). HTFs with corrosion inhibitors protect metal parts of the solar water-heating systems. In the earlier studies, the metal structure of a solar water-heater system has been described; the thermosyphonic solar water heater consists of a collector, cylindrical shaped hot water storage tank, an absorber plate and rise tubes. The absorber plate is made of a 30 SWG (0.315 mm) copper sheet, and the rise tubes are made of copper tubes of 12.7 mm diameter, having 22 SWG (0.711 mm) thickness. The hot water storage tank as a double-walled cylinder with 100 mm interspacing is constructed from 22 SWG stainless steel sheet. A support of aluminum frame of size 25 mm × 25 mm × 1.5 mm is provided for the front glazing (Siddiqui, 1997).

Nowadays, among many of the industrial applications where synthetic corrosion inhibitors are used, green inhibitors have a growing interest. In previous studies, the corrosion inhibition properties of *Juglans regia* leaf extracts on aluminum and stainless steel were investigated in acidic media, and water extracts of *J. regia* leaf extracts showed impressively good

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preventing characteristics (Akin *et al.*, 2015), while *Punica granatum* extracts showed good inhibitory activity on the stainless steel in acidic media (Behpour *et al.*, 2012). Corrosion inhibitors with organic acid structure, on the other hand, which are well-known corrosion inhibitors for aluminum and copper, showed better corrosion protection characteristics compared to the green inhibitors. However, these inhibitors are expensive and toxic for live beings (Mansfeld and Jeanjaquet, 1986; Macdonald, 1993).

We investigated a monopropylene glycol-based HTF used in thermal solar water-heating systems, and different formulations have been created by using organic and green corrosion inhibitors. The anti-corrosion properties of these formulations have been detected with various techniques such as gravimetric (weight loss) and potentiodynamic polarization spectroscopy. This is the first study identification of anti-corrosion properties of an HTF used in thermal solar water-heating systems with these techniques. The changes in the structure of the fluid after 20,000 cycles ageing test by FT-IR spectroscopy have been also investigated.

2. Material and methods

2.1 Materials

Dried walnut leaves (2 gm) were mixed with 50 mL of water and left in a shaker for two hours. The solution was filtered twice by Whatman filter paper No.4. The extract was centrifuged and evaporated in a vacuum evaporator. Finally, the dried extract was stored in the dark at −4°C for further analysis.

Aluminum and copper with 99.8 per cent were purchased from Aldrich. Both copper and aluminum sheets were graded with thick-and-thin emery paper, washed with acetone and ethanol and dried to use. Concentrated solution of HTF prepared with different corrosion inhibitors was diluted with deionized water at the ratio of 1:1. Triethanolamine was used to increase the solubility of dodecanoic acid and decanoic acid with organic acid structure in monopropylene glycol. All experiments were carried out in this solution. The formulations were prepared by using different corrosion inhibitors; the ratio of the components is shown in Table I.

2.2 Weight loss measurements

In these experiments, the effects of different types of inhibitors on weight loss measurements in HTF solutions mixed with proper quantity of corrosive water to give a 1/3 volume test solution, which contains 1 mM sodium sulfate, 2.8 mM sodium chloride and 1.6 mM sodium bicarbonate at pH 8.02, were investigated according to the ASTM D 1384. This method was used for evaluating the effects of engine coolants on metal specimens under the controlled laboratory conditions. The metal specimens of 1 cm^2 were used for weight loss experiments and degreased with acetone, rinsed in distilled water, dried in the air and weighed accurately. The specimens were immersed in 250 ml test solution at 88°C for 336 h in the absence and presence of green and organic corrosion inhibitors (ASTM, 2012).

The corrosion inhibitor properties of test solutions were evaluated on the basis of the weight changes incurred by the specimens. The measured weight loss and the inhibition efficiency (i.e. percentage) were calculated using the formula:

$$IE(\%)x = \frac{\mathrm{Wo} - \mathrm{Wi}}{\mathrm{Wo}} \times 100 \qquad (1a)$$

where W_0 and W_i are the charge weight loss with and without the inhibitor, respectively.

The corrosion rate (CR) was calculated using the following formula:

$$CR = \frac{\Delta \mathrm{m}}{\mathrm{At}} \qquad (1b)$$

where Δm is weight loss in mg, A is the area of exposure in cm^2 and t is the time in hours.

2.3 Electrochemical measurements

The electrochemical experiments were carried out by PARSTAT 2270 potentiostat/galvanostat. These measurements were performed in a conventional three-electrode cell. Saturated calomel electrode (SCE) and platinium (1 cm^2) were used as the reference and counter electrodes, respectively. Copper and aluminum with an exposed area of 1 cm^2 were used as working electrodes. Before measurements, electrode potential was allowed to be stabilized for 30 min. All experiments were conducted at room temperature.

Electrochemical impedance spectroscopy measurements were carried out in a frequency range of 1 kHz to 100 mHz with amplitude of 10 mV at open circuit potential. The charge transfer values were obtained from the diameter of the semicircles of the Nyquist plots. The inhibition efficiency was calculated using the following equation:

$$IE\ (\%) = \frac{\mathrm{Rt} - \mathrm{Rt}^{\circ}}{\mathrm{Rt}} \times 100 \qquad (2)$$

where $Rt°$ and Rt are the charge transfer resistances in the absence and presence of the inhibitor, respectively.

The polarization curves were carried out in the potential range from +250 to −250 mV with respect to open circuit potential at a scan rate of 1 mVs^{-1}. Electrochemical parameters such as E_{corr}, I_{corr} and cathodic tafel slopes (β_a, β_c) were obtained by the Tafel extrapolation method. The inhibition of efficiency was calculated by following equation (Tao *et al.*, 2011):

Table I Chemical contents of the HTF formulations

% (m/m)	Monopropylene glycol	Water	Dodecanoic acid	Decanoic acid	*J. regia* L. extract	Triethanol amine
Witness sample	91.5	6	¬	¬	¬	2.5
HTF 1	87.9	6	3.6	¬	¬	2.5
HTF 2	87.9	6	¬	3.6	¬	2.5
HTF 3	87.9	6	¬	¬	3.6	2.5

$$IE\ (\%) = \frac{I^{\circ}_{corr} - I_{corr}}{I_{corr}} \times 100 \quad (3)$$

where, I_{corr} and I°_{corr} signify the corrosion current density in the absence and presence of the inhibitors, respectively.

2.4 Ageing tests – FT-IR measurements

This test method covers a procedure for heating-cooling process effects on solar system fluids. For determination of changes on the fluid's physicial and chemical properties, HTF (contains *J. regia L.* extract) subjected to a test run continuously for 20,000 cycles by using heating and cooling process. Before and after the test, physical properties were determined by using different test methods (Table II) and chemical properties were determined by using the Perkin-Elmer BX 2 Fourier transform infrared spectrophotometer at room temperature.

2.5 Scanning electron microscopy

Surface morphologies were examined by scanning electron microscopy (SEM) (SEM-EDX JOEL 50 A). Copper and aluminum plates of 1 cm^2 were immersed in an inhibitor containing solutions of HTF for ten days. Plates were washed with distilled water and dried in oven at 40°C for 15 min.

3. Results

3.1 Weight loss measurements

Weight loss of copper and aluminum in HTF test solution is performed in the absence and presence of decanoic acid, dodecanoic acid and *J. regia* L. extract according to the ASTM D 1384. The results are shown in Table II. Specification maximum values are taken from ASTM D 3306. The inhibition efficiency of the inhibitors is shown in Table III.

The represented result indicates that the minimum weight loss and maximum corrosion inhibition has been obtained with decanoic acid as the corrosion inhibitors for all metal specimens. Both decanoic acid and dodecanoic acid have shown better corrosion inhibition properties than *J. regia L.* extract. *J. regia L.* extracts contain tannins, chlorogenic acid, caffeic acid, p-coumaric acid, ferulic acid, sinapic acid (hydroxycinnomic acids), ellagic acid and syringic acid (hydroxybenzoic acids), as well as syringaldehyde (hydroxyl benzaldehyde) and juglone in walnut seed, green husk and leaf (Pereira *et al.*, 2007). The inhibiton properties of *J. regia L.* extract may be affected because of the presence of these organic compounds in the extract. Organic compounds that contain O, S or N/or combination of the atoms have been reported as corrosion inhibitors for metals in alkaline or acid solutions (Abiola *et al.*, 2009).

Table III Weight loss data of aluminum and copper and inhibition efficiency of inhibitors

Corrosion inhibitors	Concentration of inhibitor (mg/mL)	Corrosion rate (CR)	IE (%)
Aluminum			
Blank		0.0486	12
Dodecanoic acid	0.14	0.0223	53
Decanoic acid	0.14	0.0217	48
***J. regia* L. extract**	0.14	0.0238	42
Copper			
Blank		0.0154	26
Dodecanoic acid	0.14	0.0034	57
Decanoic acid	0.14	0.0029	65
***J. regia* L. extract**	0.14	0.0046	51

3.2 Electrochemical measurements

Polarization curves of copper and aluminum in HTF test solution in the absence and presence of inhibitors are shown in Figures 1 and 2, respectively. The values of E_{corr}, I_{corr} and cathodic tafel slopes (β_a, β_c) of polarization curves with the concentrations of inhibitors are given in Table IV. Clear reduction of anodic and cathodic currents shown in potentiodynamic curves and tafel plots indicate the cathodic

Figure 1 Polarization curves of copper in HTF test solution for synthetic and green inhibitors

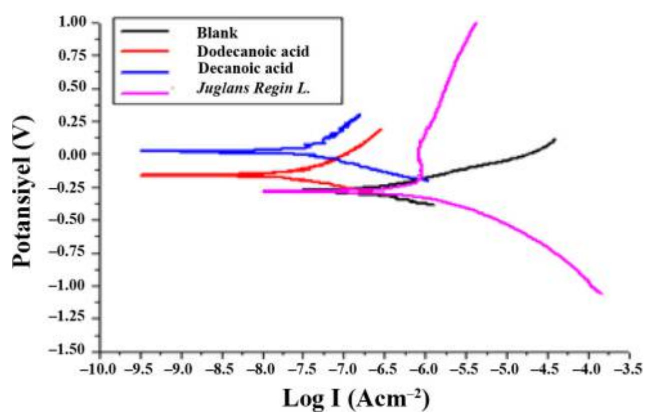
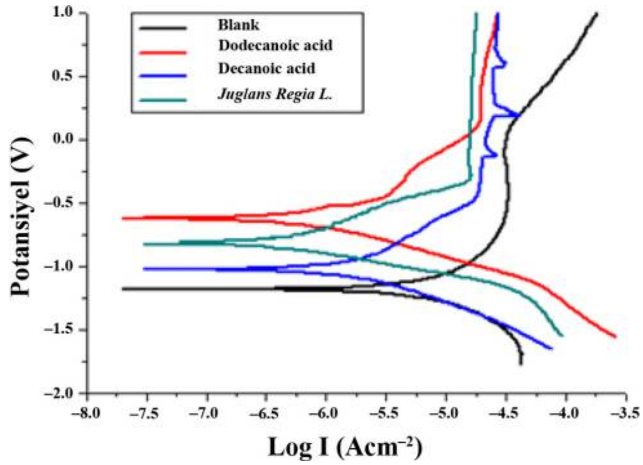


Table II Weight loss measurements according to the ASTM D 1384

Metals	Witness sample (mg)	Dodecanoic acid (mg)	Decanoic acid (mg)	*J. regia* L. *extract* (mg)	Specification max. (mg)
Aluminum	4.86	2.23	2.17	2.38	30
Cast iron	1.15	0.27	0.21	0.47	10
Copper	1.54	0.34	0.29	0.46	10
Stainless steel	0.96	0.17	0.13	0.22	10

Note: Corrosion weight loss is a positive value, weight gain is a negative value, and if no sign is given to the value it will be interpreted as weight loss

Figure 2 Polarization curves of aluminum in HTF test solution for synthetic and green inhibitors



reduction (H^+ evolution) and the anodic reduction (metal dissolution). The inhibitor molecules are adsorbed onto metal surface, and they decrease the surface area of corrosion by blocking the reaction site of the surface area (Fouda and Elattar, 2012).

Decanoic acid, dodecanoic acid and *J. regia* L. extract, as the corrosion inhibitors, play important role on both aluminum and copper. By the comparison of polarization curves, the results reveal that inhibition efficiency of decanoic acid is better than the inhibition efficiency of dodecanoic acid and *J. regia* L. extract for copper, whereas inhibition efficiency of dodecanoic acid is better than the inhibition efficiency of decanoic acid and *J. regia* L. extract for aluminum. The inhibition efficiency for decanoic acid reaches 99.97 per cent for copper, while inhibition efficiency for dodecanoic acid reaches 95.13 per cent for aluminum. On the other hand, inhibition efficiency of green inhibitor *J. regia L.* reaches 71.52 per cent for copper and 69.74 per cent for aluminum. The inhibitive action of the mucilage extracted from the modified stems of prickly pears, toward acid corrosion of aluminum, was tested in 2 M HCl; the inhibition efficiency was found 65.9 per cent for 2 per cent (w/w) inhibitor concentration (El-Etre, 2003). The effect of the extract of *Phyllanthus amarus* leaves on the corrosion of aluminum in 2 M NaOH solution was studied using chemical technique, the inhibition efficiency was found 66 ± 2.4 per cent for 5 (per cent v/v) concentration (Abiola and Otaigbe, 2009). *Azadirachta indica* leaves extract (AI) was investigated as a copper corrosion inhibitor in 0.5 M H_2SO_4. Inhibition efficiency of AI was found 86.4 per cent at $1gL^{-1}$ inhibitor concentration (Valek and Martinez, 2007). The effect of extract of cannabis plant on the corrosion of copper in aqueous 0.5 M H_2SO_4 was investigated. Inhibition efficiency was found 76 per cent at 5 ppm inhibitor concentration (Abd-El-Nabey *et al.*, 2013). These results seem acceptable for an HTF when compared with the results obtained from literature.

The equivalent Randles circuit model is shown in Figure 3, where RX represents the solution and corrosion product resistance and Rt and Cdl represent corroding interfaces. The double-layer capacitance (Cdl) was calculated as follows:

$$CdI = \frac{1}{2\,\Pi\text{fma} \times \text{Rt}} \tag{4}$$

The effect of inhibitor concentration in HTF test solution on the impedance behavior of copper and aluminum is presented in Figures 4 and 5. Nyquist plots show that the diameter of the semicircles increase with the addition of inhibitor. Tables V and VI show that the value of Rt increases from 96.45 to 122.56 Ω with the addition of dodecanoic acid for copper and 94.52 to 134.6 Ω for aluminum. Rt increases from 96.45 to 384.93 Ω with the addition of *J. regia* L. extract for copper and 94.52 to 267.90 Ω for aluminum. On the other hand, Cdl values decrease with the increase in inhibitor concentration. This can be explained by the decrease in the local dielectric constant and/or with the increase in the thickness of the electrical double layer, suggesting that the inhibitor molecules show adsorption on the metal surface (Singh *et al.*, 2011).

Figure 3 Prescribed equivalent circuit schematic by Randles

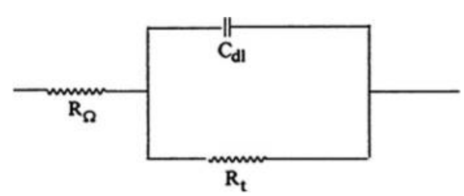
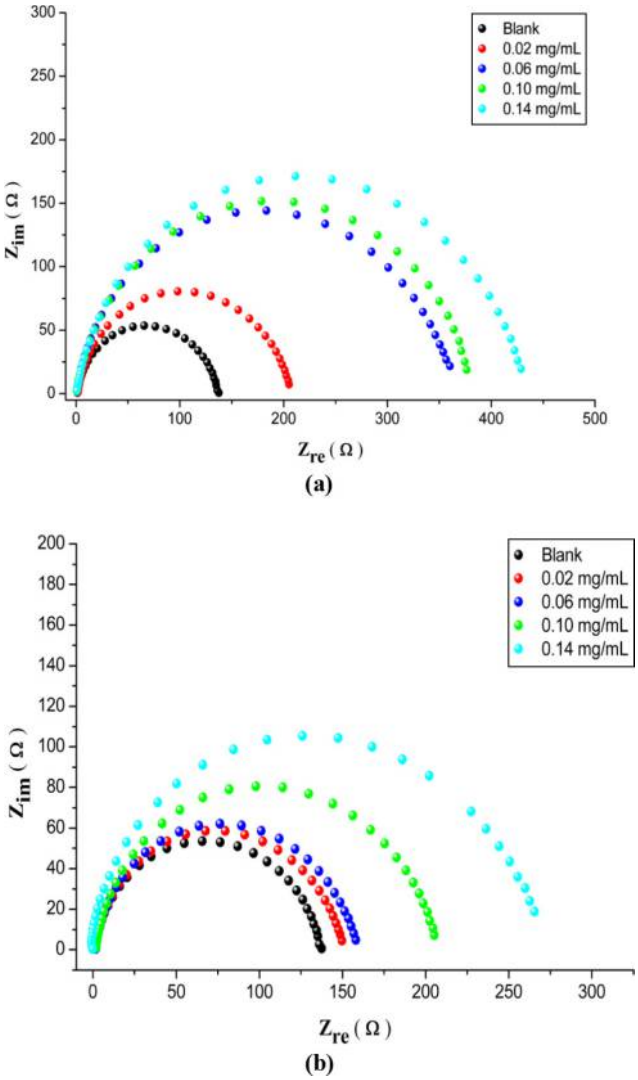


Table IV Tafel polarization parameter values for the corrosion of copper and aluminum in HTF test solution in presence and absence of inhibitors

Inhibitor	I_{corr} (μA cm^{-2})	E_{corr} (mV vs SCE)	b_c (mV/decade)	b_a (mV/decade)	*I.E* (%)
Copper electrode					
Blank	2.184	282.62	138.21	145.89	–
Dodecanoic acid	0.02	165.17	120.04	197.30	99.08
Decanoic acid	0.005	18.91	29.88	32.44	99.97
***J. regia* L. extract**	0.622	255.02	161.25	457.73	71.52
Aluminum electrode					
Blank	3.90	1181.79	190.08	247.26	–
Dodecanoic acid	0.61	624.60	240.95	241.63	95.13
Decanoic acid	0.19	823.17	89.57	126.35	84.36
***J. regia* L. extract**	1.18	1025.65	205.58	249.05	69.74

Figure 4 Nyquist plots for (a) copper and (b) aluminum presence of dodecanoic acid



3.3 FT-IR analysis

The changes of the HTF's chemical and physical properties after cycling in the thermal solar water-heating system have been investigated. To accelerate the ageing of HTF, a 20,000 cycle ageing test that corresponds to about a 6-m period of use was carried out. Changes in chemical and physical properties are shown in Figure 6 and Table VII, respectively. The same FT-IR spectra have been obtained before and after 20,000 cycling ageing test. As it can be seen in Figure 6, no changes have been obtained on the chemical properties of liquid. After ageing test, density and boiling point of the liquid increase and pour point of the liquid decreases because of the evaporation loss during the test, as a result of the changes on the temperatures.

3.4 Scanning Electron Microscopy

SEM images of aluminum and copper are given in Figure 7. Figure 7(a) and 7(d) are aluminum and copper plates immersed in HTF solution without any inhibitors. There are many voids formed as a result of corrosion and many particles

Figure 5 Nyquist plots for (a) copper and (b) aluminum presence of *J. regia* L.

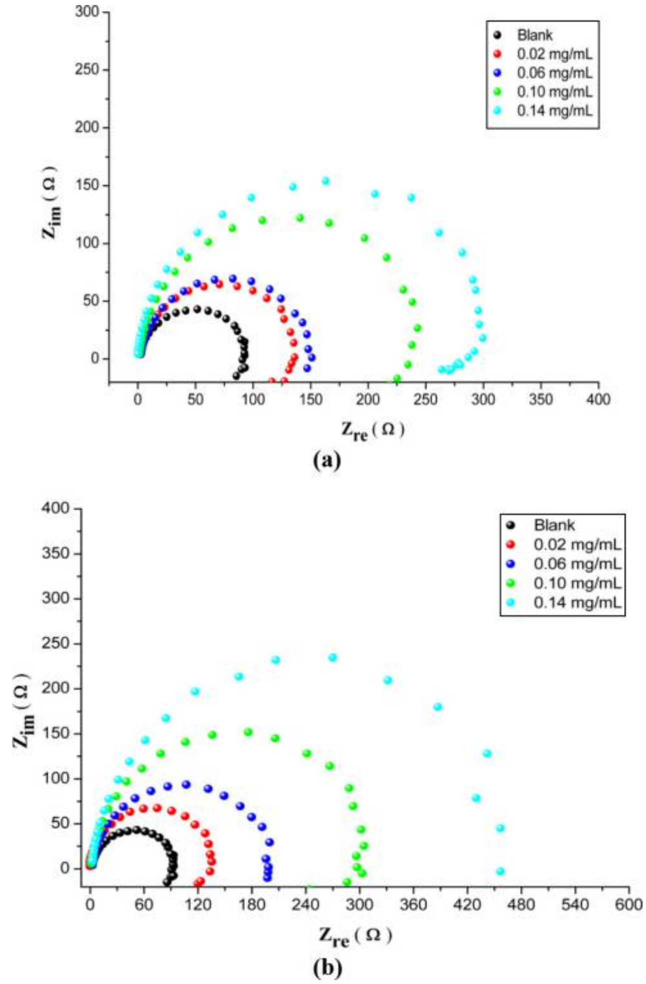


Table V Impedance parameters of dodecanoic acid on copper and aluminum in HTF test solution

Inhibitor	*Rt* (Ω)	*fmax* (Hz)	C_{d1} (μF cm^2)	*I.E* (%)
Copper electrode				
Blank	96.45	1.134	272.17	–
0.02	103.5	1.134	256.35	11.45
0.06	111.24	1.134	174.82	31.91
0.1	116.4	1.134	204.58	48.32
0.14	122.56	1.134	212.86	52.56
Aluminum electrode				
Blank	94.52	18.34	77.43	–
0.02	114.96	16.04	54.67	27.84
0.06	101.24	16.04	61.23	39.35
0.1	121.74	16.04	39.2	44.69
0.14	134.6	16.04	45.67	58.76

Table VI Impedance parameters of *J. regia* L. on copper and aluminum in HTF test solution

Inhibitor	*Rt* (Ω)	*fmax* (Hz)	C_{d1} (μF cm^2)	*I.E* (%)
Copper electrode				
Blank	96.45	1.134	272.17	–
0.02	198.65	3.78	175.46	33.02
0.06	214.3	3.78	123.72	52.07
0.1	341.04	3.78	104.68	71.43
0.14	384.93	3.78	95.32	88.41
Aluminum electrode				
Blank	94.52	18.34	77.43	–
0.02	163	29.17	32	41.04
0.06	198.51	29.17	28.23	51.85
0.1	224.85	29.17	22.15	63.85
0.14	267.90	29.17	18.54	74.03

in dark-gray contrast. It is also possible to see the deformation of surface in Figure 7D with copper. *J. regia* extract-containing samples [Figure 7(b) and 7(e)] also have voids and deformations on the metal surfaces. Solutions containing dodecanoic acid have better surface properties [Figure 7(c) and 7(f)] for both copper and aluminum. The formation of voids is inevitable because of the chemical interaction between the metal and the solution, which results in the dissolution of the matrix in the course of corrosion.

4. Discussion

The corrosion test results showed that synthetic and green inhibitors acted as the efficient corrosion inhibitors in monopropylene glycol-based HTF for aluminum and copper. According to the test results, organic corrosion inhibitors have showed better anti-corrosion effect than the green inhibitor. However, it can clearly be said that there is no significant differences between the values obtained from weight loss measurements in organic and green inhibitors. If the toxic effects and prices of organic inhibitors are considered. It is obvious that green inhibitors have an impressive inhibition effect on corrosion.

In previous studies of anti-corrosion properties, extracts of carob tree seeds on low alloy carbon steel (Jano *et al.*, 2012), adsorption behavior of caffeine as a green inhibitor on copper (De Souza *et al.*, 2012) and anti-corrosion properties of *Coriandrum sativum* L. on aluminum (Prabhu and Rao, 2013) have been investigated. All these experiments showed that plant extracts contain many organic compounds and adsorption of these compounds on metal surface reduces the surface area that is available for the attack of the aggressive ion from the solution.

In industries, the known hazardous effects of most synthetic organic inhibitors and the need to develop cheap, non-toxic and environmentally benign process now urge researchers to develop new green and good performance inhibitors. *J. regia L.* is a plant that grows anywhere in the world in terms of easily attainable and non-toxic corrosion inhibitor in HTF solution. Docecanoic acid and decanoic acid acted as efficient corrosion inhibitors in the HTF solution of aluminum and copper better than *J. regia L.* extract by adsorption on metal surface. However, green inhibitors are more eco-friendly and cheaper than the synthetic ones.

Table VII Changes in the physical properties of HTF containing *J. regia* L. extract before and after ageing test

Tests	Before ageing test	After ageing test	Test method
Appearance	Clear, blue	Blurry, light blue	Visual
Density at 20°C, gr/mL	1.024	1.036	ASTM D 1122
Boiling point, °C	105	107	ASTM D 1120
pH	7.56	8.23	ASTM D 1287
Pour point, °C	−37	−33	ASTM D 97

Figure 6 IR Spectra of HTF (contains *J. regia* L.) before and after ageing test

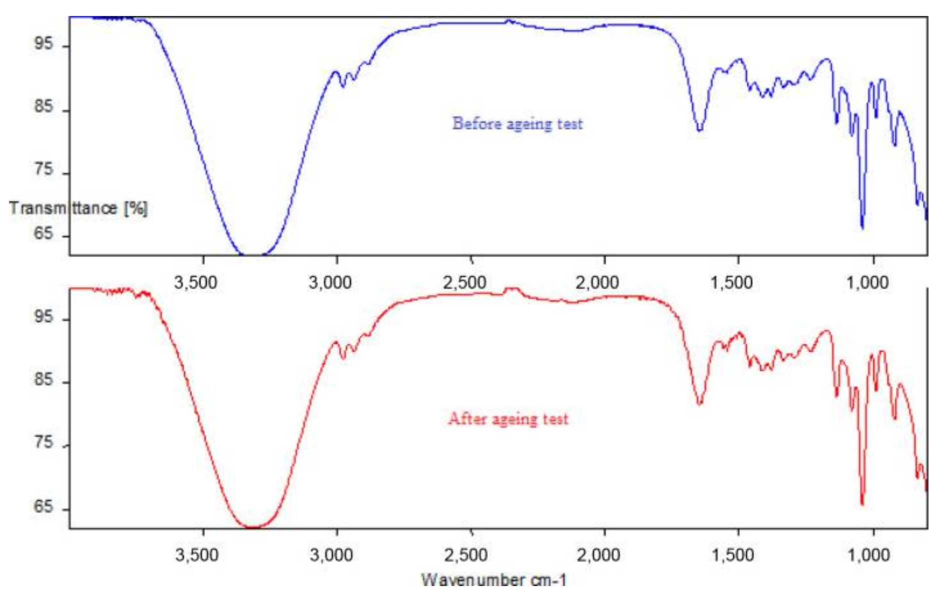
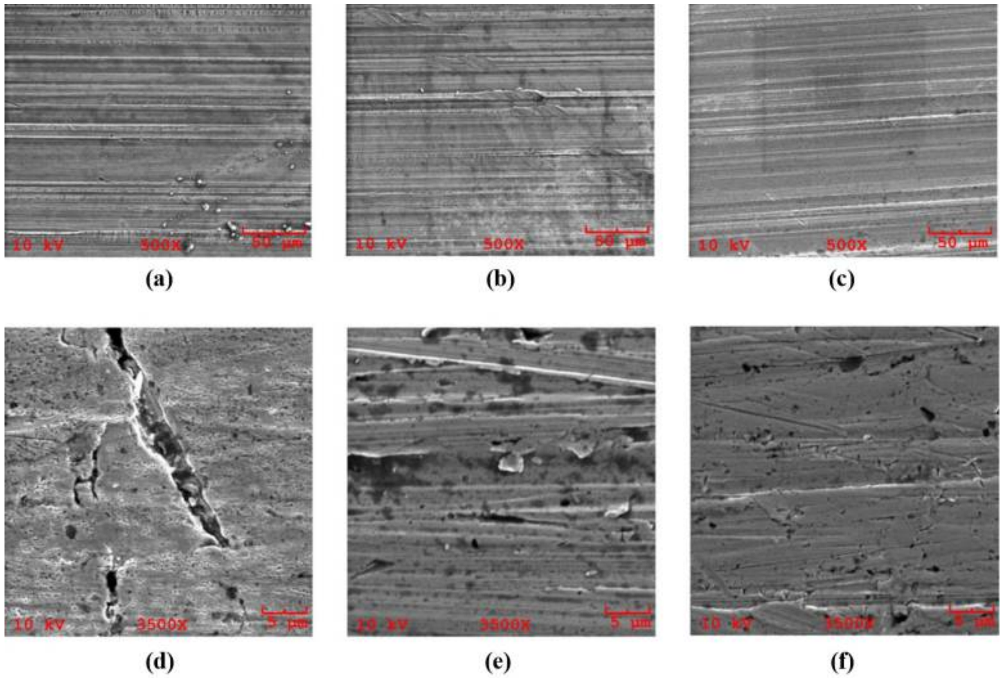


Figure 7 SEM images of Aluminium



Notes: (a) Blank; (b) 0.14 mg/mL *J.* regia extract; (c) 0.14 mg/mL dodecanoic acid; and Copper; (d) Blank; (e) 0.14 mg/mL *J.* regia extract; (f) 0.14 mg/mL dodecanoic acid

5. Conclusions

Decanoic acid, dodecanoic acid and *J. regia L.* extract acted as efficient corrosion inhibitors in monopropylene glycol-based HTF for aluminum and copper. Higher inhibition efficiencies are obtained with the dodecanoic acid and decanoic acid. Length of the carbon chain decreased the corrosion inhibition directly in proportion, and thus, decanoic acid showed better corrosion inhibition effect than dodecanoic acid and *J. regia L.* extract for copper, while dodecanoic acid showed better corrosion inhibition effect than decanoic acid and *J. regia L.* extract for aluminum. A significant reduction in the corrosion rate was observed with synthetic corrosion inhibitors; 99.97 per cent for copper for decanoic acid, 95.13 per cent for aluminium for dodecanoic acid while *J. regia L.* extracts showed moderate reduction with 71.52 per cent copper and 69.74 per cent for aluminium. There were no noticeable changes on the chemical properties of the fluid on the ageing test results after 20,000 cycling run of the HTF and FT-IR analysis also supported this result. SEM analysis also supported the results obtained from electrochemical measurements. Therefore, organic corrosion inhibitors and green inhibitor *J. regia L.* can be used as anti-corrosion agents for HTF.

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