

Mollification of Mild Steel Corrosion in Hydrochloric Acid Solutions by Sodium carboxymethyl Cellulose and Polyethylene Glycol

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Abstract: The main goal of this study is to extend shelf life of oil and gas pipelines and storage facilities which are essentially made of mild steel from effects of acid corrosion during industrial operation such as acid pickle, cleaning and descaling using cost-effective approach. To accomplish this, mild steel was immersed in 1 M HCl solutions in absence as well as presence of sodium carboxymethyl cellulose (SCMC) solely and in mixture with potassium iodide (KI) and polyethylene glycol (PEG) respectively to ascertain inhibitive capability of the compounds using weight loss measurements at 30–60 °C for 24–120 hr immersion and potentiodynamic polarization technique at 30 °C. Inhibitory performance of SCMC was good (64.12 %) at 500 mg/L which was made better by increasing concentration to 2000 mg/L (80.86 %) and much better on mixing SCMC with KI and PEG to yield 90.91 and 94.02 % respectively at 30 °C for 24 hr immersion. However, extending immersion time and temperature unfavourably affected inhibition efficiency of the compounds. Corrosion inhibition was afforded by physical adsorption of inhibitive compounds on the mild steel surface by thermodynamic considerations. Adsorption followed Langmuir isotherm model. Potentiodynamic polarization study revealed that the compounds behaved as mixed-type inhibitors by curbing both anodic and cathodic reactions on the mild steel immersed in 1 M HCl solution.

Keywords: Mild steel; Hydrochloric acid; Corrosion; Mollification; Adsorption

1. Introduction

Mild steel finds applications in the manufacture of oil and gas pipelines for transport of crude (products) and steel reservoirs / tanks for storage of crude (fractions) owing to its primarily mechanical strength (load-bearing capacity) and affordability, in addition to weldability, machinability, malleability and ductility. These attributes are due to low-carbon content present in mild steel relative to other steel grades. While this low-carbon content may be beneficial as highlighted; however, it makes mild steel prone to corrosion during such applications. Mild steel contains very little carbon and other alloying elements such as molybdenum, chromium etc., needed in considerable proportion to

withstand corrosive influences (Metal, 2016). Corrosion is a manifestation of interaction between mild steel and environmental agents. Effects of corrosion on mild steel structures and equipment are basically loss of the material properties which made it a material of choice in the mentioned areas. This comes with significantly huge consequences such as leakages leading to oil spillage (environmental pollution) and its attendant challenges, material wastage and damage, and structural failures resulting in the collapse of the assemblage be it in part or whole, which calls for in part or whole replacement or repair of the facilities depending on severity of attack (Sastri, 2015). When crude is explored, transported or stored, it comes with potential contaminants which are inherently corrosive such as carbon (IV) oxide (CO_2) and hydrogen sulphide (H_2S) with free water (H_2O) from the well (Lusk *et al.*, 2008). Sweet and sour corrosion is bound to occur over time when CO_2 and H_2S react with H_2O to form carbonic (H_2CO_3) and sulphide $\text{H}_2\text{S}_{(\text{aq})}$ acids respectively which corrode the mild steel pipelines and storage facilities resulting in material failure. These are exacerbated by variation in temperature owing to seasonal changes. The effects of these two types of corrosion can cause unsafe and inefficient transmission, distribution and storage of crude and its fractions, in addition to earlier mentioned corrosion challenges. Thus, acid pickle usually done at temperature up to 80°C is considered necessary (Schmitt, 1984). This is an ongoing maintenance effort to restore the mild steel facilities, and the most common pickling acid used in this regard is hydrochloric acid (Ahamad and Quraishi, 2009, Solmaz, 2014, Heydari *et al.*, 2018). But then, acid corrosion is initiated given its corrosiveness on mild steel. This has called for the need for corrosion inhibitors. But then, these corrosion inhibitors are known to be associated alongside one challenge or the other predicated on either cost, performance, durability or toxicity. Among inhibitor classes, those of organic origin have been commonly employed due to their ability to displace water molecules through formation of protective film occasioned by adsorption on a metal surface against corrosion. Effective ones consist of sulfur, nitrogen, phosphorus and oxygen alongside lone pairs of electrons, as well may comprise structural moieties alongside π -electrons that react with metal surface benefitting adsorption (Bogumil *et al.*, 2017). This was as green counterpart is prone to biodegradation (Kausalya and Hazlina, 2020); inorganics, toxicity (Roy *et al.*, 2014); synthesized organics, costly owing to synthesis process (Krishnaveni and Ravichandran, 2014). Consequently, attention has been shifted to the use of polymers as corrosion inhibitors of metals in aqueous corrosive solutions. Interest in polymers in that regard is owing to their molecular weight and size, constituent, nature as well as presence of multiple functional entities with which polymers use to adsorb on metal surface enveloping significant surface area (Sabirneeza *et al.*, 2015; El Mouaden *et al.*, 2018). Again, polymers are known to have multifunctionality and better film-forming abilities, which could well enhance protective film characteristics (Tiu and Advincula, 2015). Cited in the literature (Umoren and Solomon, 2014, Koulou *et al.*, 2020, Mansri and Bouras, 2014, Ezeh and Chinedu, 2023) were polymers used in the corrosion inhibition of metals in aqueous corrosive solutions. From the literature, our attention has been drawn to the use of (sodium) carboxymethyl cellulose [(S)CMC] as corrosion inhibitor of mild steel in both HCl and H_2SO_4 solutions (Solomon *et al.*, 2010, Umoren *et al.*, 2010, Bayol *et al.*, 2008). In the work of Solomon *et al.*, 2010, carboxymethyl cellulose (CMC) was recorded to have inhibited corrosion of mild steel in 2 M H_2SO_4 solution alongside inhibition efficiency of 64.8 % at 0.5 g/L for 10-h submersion at 30°C by chemical approaches. By extension, the authors investigated effect of halide ions on inhibition efficiency of CMC on the same experiment to which it was reported that 5 mM KCl reduced inhibition efficiency to 51 % whereas inhibition efficiency was boosted to 67 and 89 % on adding 5 m M KBr and KI , respectively to CMC (Umoren *et al.*, 2010). Then, Bayol *et al.*, 2008 noted that SCMC inhibited corrosion of mild steel in 1 M / L HCl with inhibition efficiency of 72 % at 400 ppm for 24-h immersion at 298 K, which reduced to 58

% at 328 K. Again, investigated by weight loss, potentiodynamic polarization and quantum chemical calculations measurements was mixture of CMC and additives [(polyvinylpyrrolidone (PVP), Polyacrylic acid (PAA) as well as polyvinylacetate (PVAc)] to inhibit corrosion of carbon steel in 1 M HCl and KOH solutions, respectively. CMC at low content reportedly inhibited carbon steel corrosion in both solutions, which somewhat improved alongside increase in content. Also, inhibitory performance of CMC was significantly improved alongside the additives. Inhibition efficiency reduced alongside increase in temperature while thermodynamic parameters obtained supported physisorption mechanism. CMC obeyed both Freundlich and Langmuir isotherm models in both solutions. Polarization studies revealed CMC to be mixed-type inhibitor while quantum chemical calculations verified that CMC adsorbed on carbon steel (Nwanonenyi *et al.*, 2018). Nevertheless, examined by weight loss, potentiodynamic polarization and electrochemical impedance measurements, surface morphological observation by scanning electron microscopy as well as molecular dynamics simulation study was inhibitory performance of SCMC-gemini surfactant system composed of mixed solution of varied content (10-700 ppm) of SCMC and fixed content (1 ppm) of synthesized oppositely charged biodegradable cationic di-ester bonded gemini surfactants, that is, ethane-1,2- diylbis(*N,N*-dimethyl-*N*-alkylammoniumacetoxyl)dichloride referred as m-E₂-m, (where m = 12, 14, 16, the number of carbon atoms in alkyl chains and E₂ is the di-ester group in gemini's spacer part), for mild steel immersed in 1 M HCl solution. SCMC solely was noted to yield modest performance of 57.3 % at 500 ppm, which improved with little quantity of synthesized gemini surfactant, synergistically. The enhanced performance was hugely anchored on the surfactant tail length as maximal performance of 90.1 % was recorded alongside 16-E₂-16. The examined systems adsorbed following Langmuir isotherm model. All the studied systems behaved as mixed-type inhibitors by polarization studies while impedance studies corroborated adsorption of molecules of the inhibitors by the presence of protective film on the mild steel surface verified by fourier transform Infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) results. Further provided was correlation between molecular adsorption of the studied systems and their structures by molecular dynamics simulation (Aslam *et al.*, 2019).

In view of the foregoing literature, we found relatively common ground on which to premise our comparative analysis. Although, SCMC yielded promising results of which synergism by additives (halides and polymers) enhanced further. However, our exposure duration way exceeded what was employed in those studies for us to settle with those views. Hence, the main objective of this study is to scrutinize mollifying capabilities of sodium carboxymethyl cellulose and its mixture with potassium iodide and polyethylene glycol respectively with a view to obtaining more efficiently cost-effective and environmentally safe inhibitors under more thorough conditions of 24-120 hours of exposure duration, 30 – 60 °C range of temperatures at 0.5 – 2 g/L concentration of SCMC in 1 M HCl by weight loss and potentiodynamic polarization measurements.

2. Methodology

2.1 Sourcing and preparation of materials

Mild steel plate of weight-percentage composition [C (0.04%)], [Si (0.02%)] [Cu (0.06%)], [Cr (0.05%)], [Mn (0.04%)] and [Fe (balance)] was carved into 4x2.5 cm² coupons. The coupons were abraded with emery papers of 240 and 320 grits to remove burrs and make smooth. The coupons were rinsed with acetone to remove impurities, and then washed alongside doubly distilled water to make clean before being kept under shade to dry thoroughly. Perforation at one end of the coupons, before being weighed, was carried out in order to allow for insertion of nylon thread in a way that enabled suspension of the coupons into 300 mL of 1 M HCl solutions (without and with inhibitive compounds)

held in 500-mL beakers and their retrieval as well, accordingly. With the aid of a water bath (thermostatic controlled-STD/GMP model), the system was thermally constrained at a range of temperatures (30 – 60 °C) without perturbation. Stock solution of 1 M HCl (37 %, BDH AR grade) was made ready using doubly distilled water. With the aid of a weighing balance (GF-6100 precision scale), 500, 1000, 1500 and 2000 mg SCMC ($M_w \sim 250,000$; degree of substitution 0.9) were measured into separate measuring cylinders as well as filled to the scaled mark of 1000 mL by 1 M HCl solution, respectively to have varied concentrations of SCMC. Prepared also were test solutions consisting of 500 mg KI and PEG respectively in 1 L of 1 M HCl. For combined study, 500 mg KI and PEG respectively were added into each concentration of SCMC. All the reagents utilized were sourced from Sigma-Aldrich, USA.

2.2 Experiments

Suspended in 300 mL solutions held in 500 mL beakers were weighed coupons of mild steel at room temperature of 30 °C. The coupons were retrieved after 24 h, which unceasingly carried on for 120 h. The coupons were reweighed after having been rinsed alongside doubly distilled water and dried under a shade. Mensuration was carried out three times for accuracy. Similar procedure was followed in the assessment of thermal influence to corrosion control process from 30 – 60 °C as well as in the studying of effect of mixing each concentration of SCMC with KI and PEG respectively on the overall corrosion control process. Mean values of weight loss obtained from difference in weight before-and-after suspension of mild steel in 1 M HCl solutions were made use of to calculate:

$$\text{Corrosion rate } \left(\frac{\text{mm}}{\text{yr}} \right) = \frac{876W}{\rho A t} \quad \text{Eqn. 1}$$

where W , ρ , A and t represent weight loss (g), mild steel density ($\text{g}\cdot\text{cm}^{-3}$), coupon area (cm^2) and exposure duration (h), respectively.

$$\text{Inhibition efficiency (\%)} = 1 - \frac{W_i}{W_o} \times 100 \quad \text{Eqn. 2}$$

where W_o and W_i are weight losses in absence as well as presence of inhibitive compounds at same temperature, correspondingly.

$$\text{Surface coverage } (\theta) = 1 - \frac{W_i}{W_o} \quad \text{Eqn. 3}$$

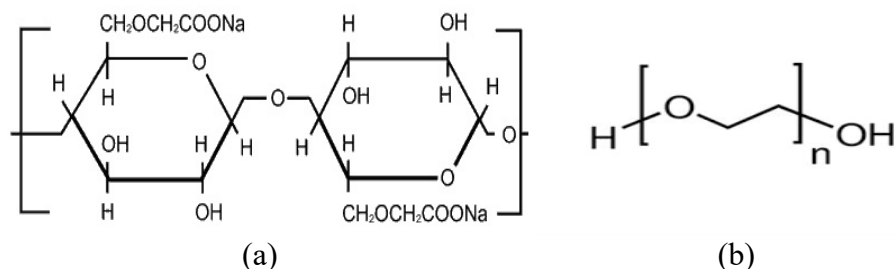
where W_o and W_i are weight losses in absence as well as presence of inhibitive compounds at same temperature, accordingly.

On the other hand, the study of electrochemical corrosion of mild steel in 1 M HCl in absence as well as presence of inhibitive compounds at 30 °C was examined. This technique was carried out in computer-controlled electrochemical workstation (VERSASTAT3 model) attached to by three electrodes of glass electrolytic cell. One of the electrodes was mild steel coupon (working electrode); the other, 99 %–graphite rod (counter electrode) from Aldrich, and then, reference electrode of standard calomel [Ag/AgCl (3 M KCl, Gamry)]. The working electrode of 2 cm^2 prepared as in weight loss measurement was connected to a wire made of copper and dipped into 1 M HCl solution in absence as well as presence of inhibitive compounds respectively for 30 min to set open circuit potential initiated by passage of electric current through the working electrode for mensuration to commence. Uninsulated 1 cm^2 of the working electrode was dipped into test solutions for corrosion to take effect while the other half was insulated alongside epoxy resin. Mensuration was carried out at a range of

potentials -250 to +250 mV at 0.5 mV/s scan rate. Inhibition efficiency of the compounds was calculated using equation (4) (Salhi *et al.*, 2017):

$$\text{Inhibition efficiency (\%)} = 1 - \frac{I_i}{I_o} \times 100 \quad \text{Eqn. 4}$$

where I_o and I_i are corrosion current densities in absence as well as presence of inhibitive compounds, respectively.

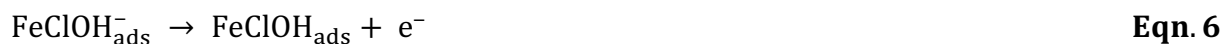
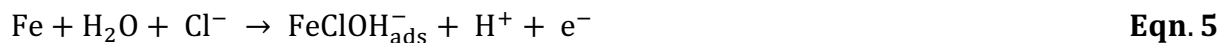


Scheme 1. Schematic structure of (a) SCMC and (b) PEG

3. Results and Discussion

3.1 Effect of immersion time on corrosion rate and inhibition efficiency

Weight loss measurement is done to simulate what happens in industrial environment whether it is acid pickle of a metallic article in acid bath or spraying of the pickling liquor on affected part on a structure, the metal is in contact with a corrosive solution. How long this contact last is regarded as *exposure duration*. For mild steel exposed to 1 M HCl solution in absence of inhibitive compounds, corrosion takes effect according to the following reactions:



In the solution, chloride (Cl^-) ions have affinity for positively charged adsorbed hydroxide, their reaction resulted in the formation of adsorbed hydroxy-chloride entities. On this note, dissolution of iron occurs concurrently to reactions **Eqn. 8-10** (Bockris and Drazic, 1962) according to (Noor and Al-Moubaraki, 2008).



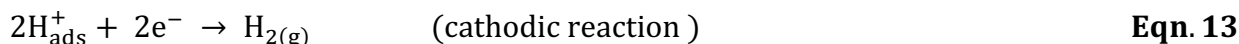
Following this anodic reaction of iron to yield iron hydroxide in aqueous solutions, iron corrodes. For steel in contact alongside dissolved oxygen in water, the usual cathodic reaction is as follows:



Thus, corrosion is remarkably increased in the hydrochloric acid solutions. Again, on the surface attached by H_{ads}^+ , hydroxide and hydroxy-chloride entities at low pH cannot exist thereby making the following reaction to occur:



At this point, iron dissolving and hydrogen evolving are produced by the same reaction. The sites where H_2 evolves create new cathodic sites where the following reaction occurs:



It is because of the reaction in addition to corrosion products yielded that made the metal to lose weight over time as electrons released from the metal interact alongside hydrogen ions adsorbed on the surface of the metal to yield gaseous hydrogen which propound anodic dissolution of the metal (Oguzie, 2007). The resultant effect of this reaction is *metal thinning* made manifest as weight loss thus the metal loses its mechanical strength among other useful material properties— effects of corrosion. **Figure 1** shows effect of immersion time on mild steel immersed in 1 M HCl solution in absence as well as presence of inhibitive compounds, respectively.

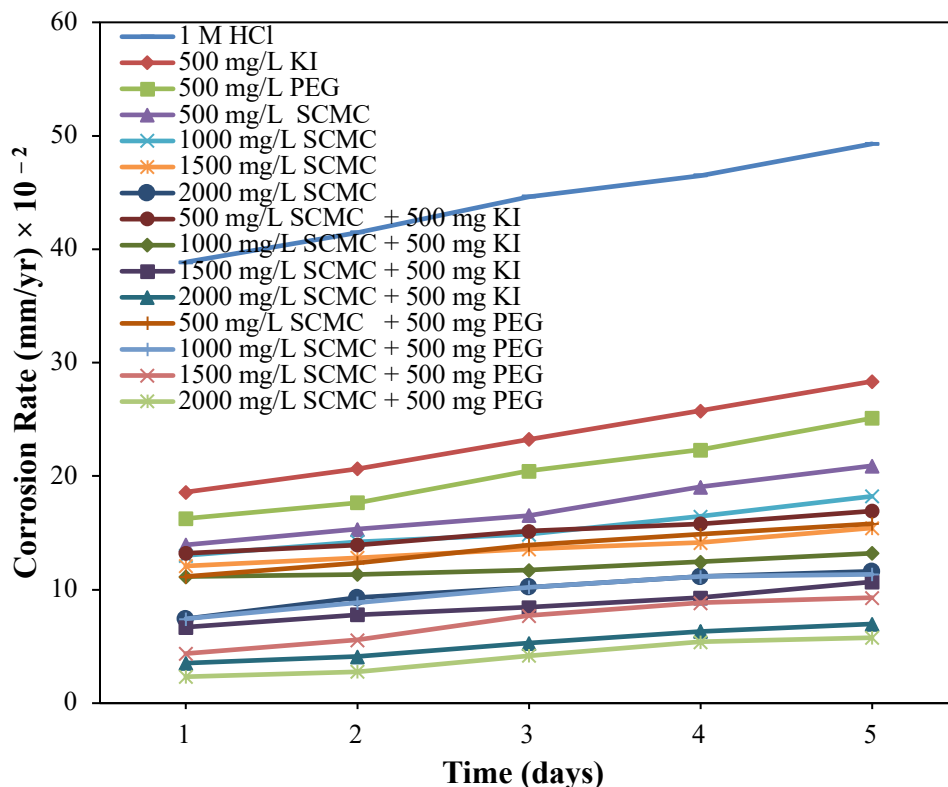


Figure 1. Corrosion rate over time for mild steel immersed in 1 M HCl and inhibited solutions

From the figure, corrosion rate gradually increased as reflected in the plot from 0.3887 mm/yr at day 1, to 0.4464 mm/yr at day 3 and then 0.4929 mm/yr at day 5. However, on introducing 500 mg KI, PEG and SMC respectively into 1 L of 1 M HCl solution, corrosion rate drastically reduced in the order KI < PEG < SMC; least by KI as corrosion rate reduced to 0.1860 at day 1, 0.2325 mm/yr at day 3 and then 0.2836 mm/yr at day 5; most by SMC as corrosion rate minimized to 0.1395 mm/yr at day 1, 0.1655 mm/yr at day 3 and then 0.2092 mm/yr at day 5 while PEG was just in-between with

0.1627 mm/yr at day 1, 0.2046 mm/yr at day 3 and then 0.2511 mm/yr at day 5. These results reflect their respective inhibitory capacities to serve as corrosion inhibitors. For KI, iodide ions (I^-) simply chemisorbed by displacement of chloride ions (Cl^-) attached to the mild steel surface in solution owing to larger atomic radius and higher electronegativity possessed by I^- than Cl^- . For PEG, it has lone pair of electrons in its oxygen atoms which may serve as an anchoring point for it to bond on the d-vacant orbital of iron constituent of the mild steel as noted for d-group transition metals. Moreover, protonation by PEG in acid solution may occur and the positively charged protonated entities conveniently adsorb on mild steel surface through electrostatic interaction (Umoren *et al.*, 2008). For SCMC, its multifunctional approach nailed it as sodium ions took chloride ions down forming table salt (sodium chloride). Meanwhile, protonation by CMC may occur and the polycation from the protonated carbonyl oxygen ($C=O$) of CMC adsorb on the negatively charged mild steel surface as weakly adsorbed hydrated ions of chloride on mild steel inclined it so. Again, hydroxyl ($-OH$) groups may have adsorbed or aided so as it could serve as anchoring point for the compound to adsorb on a metal surface as revealed in the literature (Chengxian *et al.*, 2020, Verma *et al.*, 2018, 2016). Nevertheless, on increasing concentration of SCMC to 2000 mg/L, concentration-dependent performance of SCMC was made manifest as corrosion rate was further curbed to 0.0744 mm/yr at day 1, 0.1023 mm/yr at day 3 and then 0.1162 mm/yr at day 5. Then, on mixing SCMC with KI and PEG respectively, corrosion rate was much further mollified to 0.0353 mm/yr at day 1, 0.0530 mm/yr at day 3 and then 0.0698 mm/yr at day 5 in 2000 mg SCMC+KI/L while 0.0233 mm/yr at day 1, 0.0419 mm/yr at day 3 and then 0.0577 mm/yr at day 5 in 2000 mg SCMC+PEG/L were obtained to which PEG demonstrated to be a more potent additive than KI on that note. The remarkable performances observed on mixing SCMC with KI and PEG respectively had to do with combined effect of separate inhibitive approaches followed by each compound which profoundly boosted their performances thus.

Regarding inhibition efficiency, all compounds wane over time in performance in acid solution. Inhibition efficiency is a measure of how well an inhibitor performs its role usually expressed in percentage. However, the question is how long can effectiveness of an inhibitor last in corrosive solutions? This can impact on shelf life of the structure and also will have significant cost implication. **Figure 2** shows that inhibition efficiency of the compounds doggedly reduced over time as depicted in the plot. This shows that the compounds strongly adsorbed on the mild steel surface. From **Table 1**, the highest values of inhibition efficiency obtained at day 1 were because molecules of the compounds underwent the foregoing inhibition mechanisms elucidated earlier promptly with their initial adsorptive energy on introduction into the test solutions blocking many possible corrosion prone sites on the mild steel surface. However, as molecules of the compounds stay adsorbed on mild steel surface over time, flagging in inhibition efficiency set in probably from day 1 to 5 as anions gained ample time to regenerate in strength and contended at adsorbed-mild steel interface causing resiliently gradual desorption of the adsorbed molecules here and there from the mild steel surface; thus, inhibitory performance doggedly decreased from day 1 to 5 as shown in **Table 1** where inhibition efficiency tenaciously lessened from 64.12 % at day 1 to 62.92 % at day 3 and then 57.55 % at day 5 in 500 mg/L SCMC; 80.86 % at day 1 to 77.08 % at day 3 and then 76.42 % at day 5 in 2000 mg/L SCMC; 90.91 % at day 1 to 88.13 % at day 3 and then 85.85 % at day 5 in 2000 mg/L SCMC+KI; 94.02 % at day 1 to 90.63 % at day 3 and then 88.30 % at day 5 in 2000 mg/L SCMC+PEG. From the highlighted results, it is obvious that increasing concentration enhanced inhibitory performance while mixing two inhibitive compounds much further boosted performances due to combined effect to which PEG was relatively better than KI in that regard.

Comparatively to what was obtained in the work of Bayol *et al.*, 2008 where SCMC inhibited corrosion of mild steel in 1 M/L HCl with inhibition efficiency of 72 % at 400 ppm for 24-h immersion at 298 K, which reduced to 58 % at 328 K. In ours, SCMC curbed corrosion of mild steel in 1 M HCl with inhibition efficiency of 64.12 % at 500 mg/L for 24-h immersion at 30 °C, which reduced to 42.35 % at 60 °C. These results would have been comparable but higher by 5 °C in ours lowered inhibitory performance somewhat at lowest temperature and significantly at highest temperature that difference of 100 mg/L advantage ours would have had was cancelled out by the 5 °C higher in temperature. Then, in the work of Solomon *et al.*, 2010, crucial variable conditions which would have served as a common ground for comparison of results were at variance with ours and thus no meaningful comparison could be drawn.

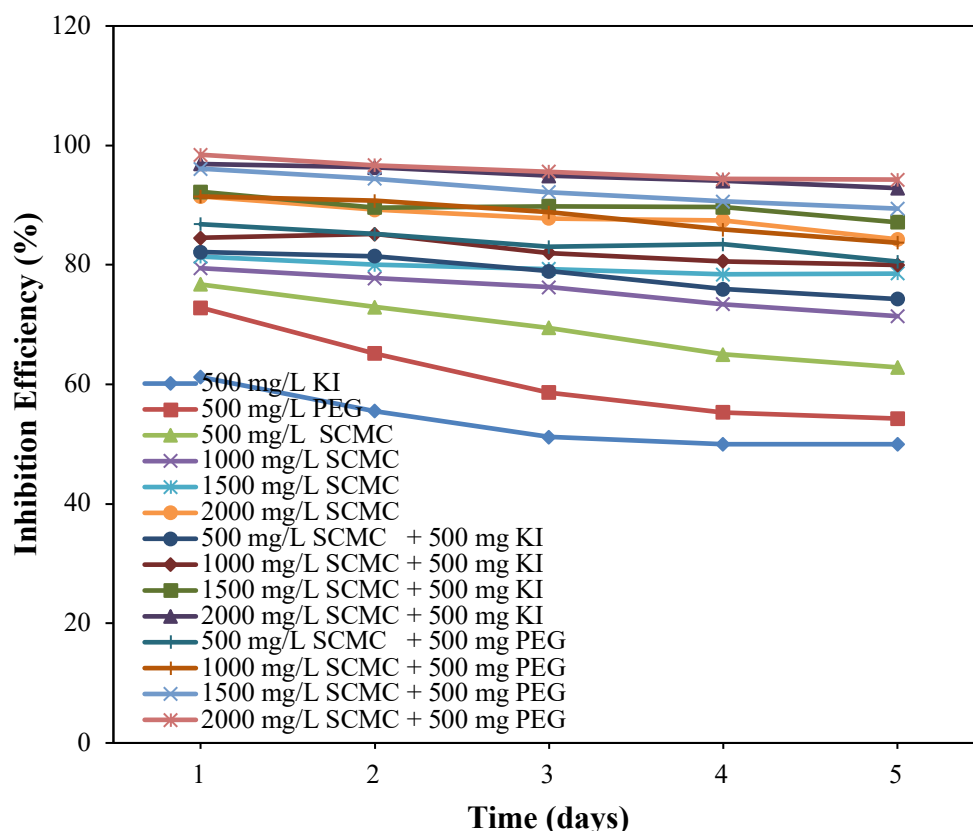


Figure 2. Inhibition efficiency over time for mild steel immersed in 1 M HCl and inhibited solutions

For further clarification, stronger oxidizing acid H_2SO_4 of 2 M was used in theirs while a relatively less corrosive strength type of acid HCl at lower concentration of 1 M was employed in ours. Exposure duration of 10 h was used in theirs while the least in ours on that note was 24 h, a significant difference of 14 h that is huge; thus, making modest performance of 57.3 % obtained in theirs and 64.12 % performance obtained in ours incomparable for concentration of 500 mg/L SCMC. Nevertheless, in the work of Aslam *et al.*, 2019, where SCMC mitigated mild steel corrosion in 1 M HCl with inhibition efficiency of 57.3 % at 500 mg/L for 6 h immersion. SCMC mollified corrosion of mild steel in 1 M HCl solution with inhibition efficiency of 64.12 % at 500 mg/L for 24 h immersion in ours; a weighty difference of 18 h enough time to run theirs four times for one of ours. Perhaps, the weighty immersion time difference gave inhibitory mechanisms adopted by SCMC ample time to take effect resulting in the better inhibitory performance of 64.12 % obtained. Again, 100 mg/L (1 ppm) synthesized

oppositely charged biodegradable cationic di-ester bonded gemini surfactants, that is, ethane-1,2-diylbis (*N, N*-dimethyl-*N*-alkylammoniumacetoxo) dichloride referred as m-E₂-m, (where m = 12, 14, 16, the number of carbon atoms in alkyl chains and E₂ is the di-ester group in gemini's spacer part), whose performance was significantly hinged on the surfactant tail length greatly impacted SCMC performance to 90.1 % alongside 16-E₂-16. This may suggest that resources put in that synthesis was worth it. Regarding ours, inhibitory performance of 71.29 % was obtained on adding 500 mg PEG into 500 mg/L SCMC which is considerable though; but then, it would have been inferred that the gemini surfactants had more combined effect on SCMC than PEG but for immersion time. Perhaps, a further study should be done in that regard to establish a common ground for more meaningful comparison.

Table 1. Calculated corrosion rate and inhibition efficiency for mild steel immersed in 1 M HCl and inhibited solutions respectively at varied days

| System/Day | Corrosion Rate (mm/yr) x 10 ⁻² | | | | | Inhibition Efficiency (%) | | | | |
|-----------------------------|---|-------|-------|-------|-------|---------------------------|-------|-------|-------|-------|
| | 1 | 2 | 3 | 4 | 5 | 1 | 2 | 3 | 4 | 5 |
| 1 M HCl | 38.87 | 41.48 | 44.64 | 46.50 | 49.29 | - | - | - | - | - |
| 500 mg/L KI | 18.60 | 20.65 | 23.25 | 25.76 | 28.36 | 52.15 | 50.22 | 47.92 | 44.60 | 42.45 |
| 500 mg/L PEG | 16.27 | 17.67 | 20.46 | 22.32 | 25.11 | 58.13 | 57.40 | 54.17 | 52.00 | 49.06 |
| 500 mg/L SCMC | 13.95 | 15.34 | 16.55 | 19.06 | 20.92 | 64.12 | 63.01 | 62.92 | 59.00 | 57.55 |
| 1000 mg/L SCMC | 13.02 | 14.23 | 14.88 | 16.46 | 18.23 | 66.51 | 65.70 | 66.67 | 64.60 | 63.02 |
| 1500 mg/L SCMC | 12.09 | 12.83 | 13.58 | 14.14 | 15.44 | 68.90 | 69.06 | 69.58 | 69.60 | 68.68 |
| 2000 mg/L SCMC | 7.44 | 9.30 | 10.23 | 11.16 | 11.62 | 80.86 | 77.58 | 77.08 | 76.00 | 76.42 |
| 500 mg/L SCMC + 500 mg KI | 13.21 | 13.95 | 15.16 | 15.81 | 16.93 | 66.03 | 66.37 | 66.04 | 66.00 | 65.66 |
| 1000 mg/L SCMC + 500 mg KI | 11.16 | 11.35 | 11.72 | 12.46 | 13.21 | 71.29 | 72.65 | 73.75 | 73.20 | 73.21 |
| 1500 mg/L SCMC + 500 mg KI | 6.70 | 7.81 | 8.46 | 9.30 | 10.69 | 82.78 | 81.17 | 81.04 | 80.00 | 78.30 |
| 2000 mg/L SCMC + 500 mg KI | 3.53 | 4.09 | 5.30 | 6.32 | 6.98 | 90.91 | 90.14 | 88.13 | 86.40 | 85.85 |
| 500 mg/L SCMC + 500 mg PEG | 11.16 | 12.37 | 13.95 | 14.88 | 15.81 | 71.29 | 70.18 | 68.75 | 68.00 | 67.93 |
| 1000 mg/L SCMC + 500 mg PEG | 7.44 | 8.83 | 10.23 | 11.16 | 11.35 | 80.86 | 78.70 | 77.08 | 76.00 | 76.98 |
| 1500 mg/L SCMC + 500 mg PEG | 4.37 | 5.58 | 7.72 | 8.83 | 9.30 | 88.76 | 86.55 | 82.71 | 81.00 | 81.13 |
| 2000 mg/L SCMC + 500 mg PEG | 2.33 | 2.79 | 4.19 | 5.39 | 5.77 | 94.02 | 93.27 | 90.63 | 88.40 | 88.30 |

3.2 Effect of temperature on corrosion rate and inhibition efficiency

Mild steel has low carbon content and other alloying elements which made its iron constituent prone to oxidation in absence of proper protection. It also implies that its low carbon content and other alloying elements will not be sufficient to block dislocation in its crystal lattice generally resulting in less tensile strength. Thus, increasing temperature strains and widens dislocation in mild steel which makes its grains susceptible to corrosion. **Figure 3** illustrates influence of increasing temperature on

mild steel exposed to 1 M HCl solution in absence as well as presence of inhibitive compounds, respectively.

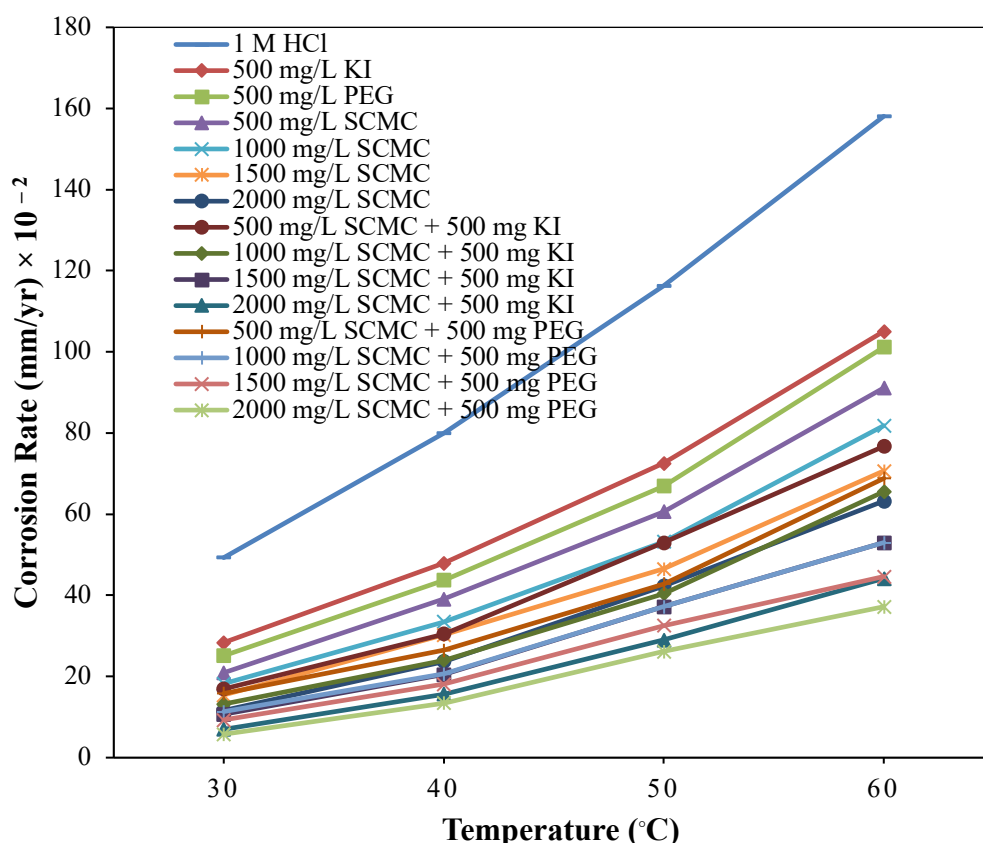


Figure 3. Corrosion rate against temperature for mild steel immersed in 1 M HCl and inhibited solutions

From **Table 2**, in absence of inhibitive compounds, corrosion rate increased from 0.4929 mm/yr at 30 °C to 0.7998 mm/yr at 40 °C, 1.1624 mm/yr at 50 °C and then 1.5809 mm/yr at 60 °C attesting to the foregoing premises and also to elementary chemistry where it is stated that increase in temperature increases rate of chemical reaction and corrosion is a chemical reaction even though it is also electrochemical too. However, on adding 500 mg KI, PEG and SCMC respectively into 1 L of 1 M HCl solution, corrosion rate was curtailed to 0.2836 mm/yr at 30 °C, 0.4789 mm/yr at 40 °C, 0.7254 mm/yr at 50 °C and then 1.0508 mm/yr at 60 °C in 500 mg/L KI; 0.2511 mm/yr at 30 °C, 0.4371 mm/yr at 40 °C, 0.6696 mm/yr at 50 °C and then 1.0118 mm/yr at 60 °C in 500 mg/L PEG; 0.2092 mm/yr at 30 °C, 0.3906 mm/yr at 40 °C, 0.6063 mm/yr at 50 °C and then 0.9113 mm/yr at 60 °C in 500 mg/L SCMC. For KI, this was as iodide ions (I^-) displaced perching chloride ions (Cl^-) on the surface of mild steel which over time would have infiltrated through the cracks into the grains wreaking havoc in mild steel. This was done swiftly as increase in temperature increased the kinetics of such displacement reaction. For PEG and SCMC respectively, their respective inhibition mechanisms were quickened as a response to influence of increasing temperature on the dynamics of corrosion control process. This was as the compounds plugged possible points of dislocation on the mild steel surface. This plugging became more effective alongside increase in concentration of SCMC to 2000 mg/L as corrosion rate was further dampened to 0.1162 mm/yr at 30 °C, 0.2371 mm/yr at 40 °C, 0.4231 mm/yr at 50 °C and then 0.6324 mm/yr at 60 °C. Much further dampening in corrosion rate was recorded on mixing SCMC with KI as well as PEG respectively to yield 0.0698 mm/yr at 30 °C, 0.1562 mm/yr at 40 °C, 0.2901

mm/yr at 50 °C and then 0.4417 mm/yr at 60 °C in 2000 mg/L SCMC+KI as well as 0.0577 mm/yr at 30 °C, 0.1348 mm/yr at 40 °C, 0.2613 mm/yr at 50 °C and then 0.3720 mm/yr at 60 °C in 2000 mg/L SCMC+PEG. In the case of SCMC+KI, I⁻ served as a *flooring* for polycation of carbonyl oxygen of CMC to adsorb which thickened the adsorbed species on the surface of mild steel and possibly spread considerably in the combined effect. This was as sodium ions (cations) had pulled out leaving CMC molecules while trapping anions (Cl⁻) as explicated earlier. For SCMC+PEG, combined effect was afforded by miscibility of the two polymers which made for efficient plugging on the dislocated sites on the mild steel surface. This is made manifest in the best results obtained on that note.

On the other hand, while the compounds mollified corrosion rate of mild steel in 1 M HCl solution under thermal influence as illustrated in Figure 3, it does not imply that inhibitory performance by the compounds was favourable alongside rise in temperature. This was as inhibition efficiency decreased with rise in temperature from 42.45 % at 30 °C to 40.12 % at 40 °C, 37.60 % at 50 °C and then 33.53 % at 60 °C in 500 mg/L KI; from 49.06 % at 30 °C to 45.35 % at 40 °C, 42.40 % at 50 °C and then 36.00 % at 60 °C in 500 mg/L PEG; from 57.55 % at 30 °C to 51.16 % at 40 °C, 47.84 % at 50 °C and then 42.35 % at 60 °C in 500 mg/L SCMC. Figure 4 illustrates declines in inhibition efficiency of the compounds alongside increasing temperature on mild steel immersed in 1 M HCl solution.

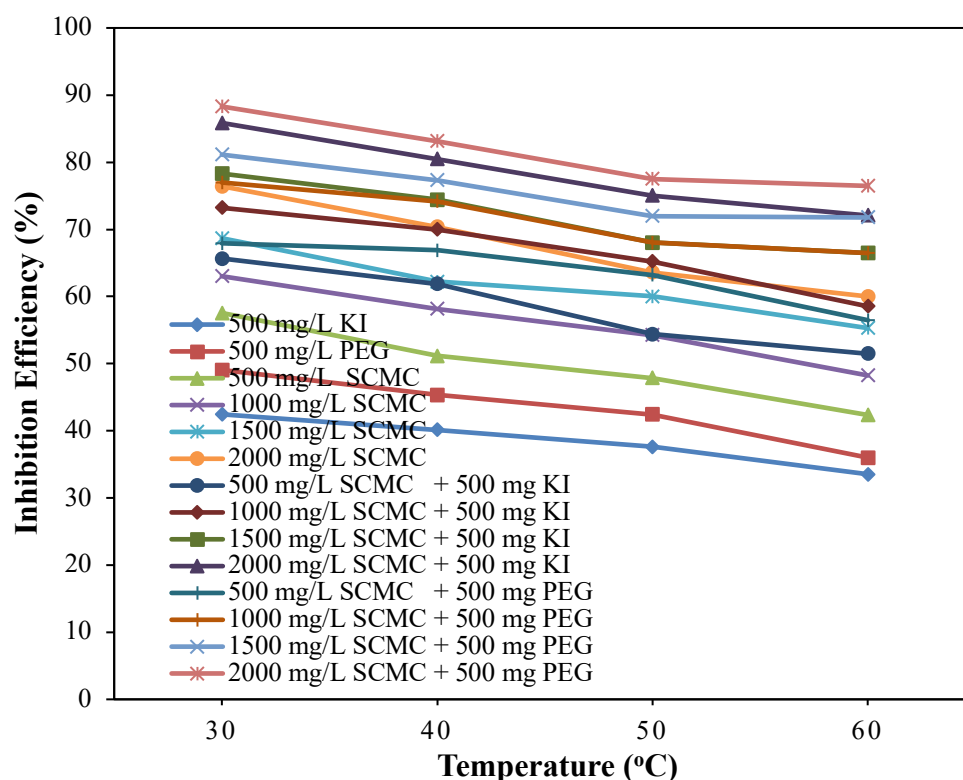


Figure 4. Inhibition efficiency against temperature for mild steel immersed in 1 M HCl and inhibited solutions

From the figure, it shows that adsorbed molecules gradually desorbed or pulled away from the surface of mild steel as its surface temperature began to scorch with rise in temperature resulting in decrease in surface coverage and unvaryingly decrease in inhibition efficiency of the compounds. However, this pulling away of the adsorbed molecules from the scorching surface of mild steel due to rise in temperature was delayed at 2000 mg/L SCMC due to thick presence of adsorbed molecules on mild steel surface thereby resulting in improved inhibitory performance recorded as inhibition efficiency increased to 76.42 % at 30 °C, 70.35 % at 40 °C, 63.60 % at 50 °C and then 60.00 % at 60 °C. Although,

thermal influence still reduced the performance but its effect was only minimal. Nevertheless, on mixing SCMC with KI and PEG respectively, the delay in desorption was further prolonged as thicker adsorbed molecules were formed on the mild steel surface resulting in much better inhibitory performances due to combined effect as inhibition efficiency further improved to 85.85 % at 30 °C, 80.47 % at 40 °C, 75.04 % at 50 °C and then 72.06 % at 60 °C in 2000 mg/L SCMC+KI and 88.30 % at 30 °C, 83.14 % at 40 °C, 77.52 % at 50 °C and then 76.47 % at 60 °C in 2000 mg/L SCMC+PEG thereby making influence of increasing temperature less minimal to which PEG edged KI in that regard for reasons explicated earlier. Presented in **Table 2** are calculated corrosion rate and inhibition efficiency for mild steel immersed in 1 M HCl and inhibited solutions respectively at varied temperatures.

Table 2. Calculated corrosion rate and inhibition efficiency for mild steel immersed in 1 M HCl and inhibited solutions respectively at varied temperatures

| System | Corrosion Rate (mm/yr) x 10 ⁻² | | | | Inhibition Efficiency (%) | | | |
|-----------------------------|---|-------|--------|--------|---------------------------|-------|-------|-------|
| | 30 °C | 40 °C | 50 °C | 60 °C | 30 °C | 40 °C | 50 °C | 60 °C |
| 1 M HCl | 49.29 | 79.98 | 116.24 | 158.09 | - | - | - | - |
| 500 mg/L KI | 28.36 | 47.89 | 72.54 | 105.08 | 42.45 | 40.12 | 37.60 | 33.53 |
| 500 mg/L PEG | 25.11 | 43.71 | 66.96 | 101.18 | 49.06 | 45.35 | 42.40 | 36.00 |
| 500 mg/L SCMC | 20.92 | 39.06 | 60.63 | 91.13 | 57.55 | 51.16 | 47.84 | 42.35 |
| 1000 mg/L SCMC | 18.23 | 33.48 | 53.19 | 81.83 | 63.02 | 58.14 | 54.24 | 48.24 |
| 1500 mg/L SCMC | 15.44 | 30.22 | 46.50 | 70.68 | 68.68 | 62.21 | 60.00 | 55.29 |
| 2000 mg/L SCMC | 11.62 | 23.71 | 42.31 | 63.24 | 76.42 | 70.35 | 63.60 | 60.00 |
| 500 mg/L SCMC + 500 mg KI | 16.93 | 30.50 | 53.01 | 76.72 | 65.66 | 61.86 | 54.40 | 51.47 |
| 1000 mg/L SCMC + 500 mg KI | 13.21 | 23.99 | 40.45 | 65.56 | 73.21 | 70.00 | 65.20 | 58.53 |
| 1500 mg/L SCMC + 500 mg KI | 10.69 | 20.46 | 37.20 | 53.01 | 78.30 | 74.42 | 68.00 | 66.47 |
| 2000 mg/L SCMC + 500 mg KI | 6.98 | 15.62 | 29.01 | 44.17 | 85.85 | 80.47 | 75.04 | 72.06 |
| 500 mg/L SCMC + 500 mg PEG | 15.81 | 26.50 | 42.78 | 68.82 | 67.93 | 66.86 | 63.20 | 56.47 |
| 1000 mg/L SCMC + 500 mg PEG | 11.35 | 20.65 | 37.20 | 53.01 | 76.98 | 74.19 | 68.00 | 66.47 |
| 1500 mg/L SCMC + 500 mg PEG | 9.30 | 18.13 | 32.55 | 44.64 | 81.13 | 77.33 | 72.00 | 71.77 |
| 2000 mg/L SCMC + 500 mg PEG | 5.77 | 13.48 | 26.13 | 37.20 | 88.30 | 83.14 | 77.52 | 76.47 |

4. Adsorption consideration

From the foregoing, it is evident that corrosion rate was minimized through the inhibitory mechanisms employed by the compounds. However, mode and pattern of adsorption by molecules of the compounds on the mild steel surface need be ascertained. This can be achieved by exploring

adsorption isotherm models to which Langmuir isotherm model became agreeable on that note. This was achieved as values of degree of surface coverage were installed into different adsorption isotherm equations such as Temkin, Langmuir as well as Freundlich and graphically appraised for values of correlation coefficients which came close to one (unity). Equation of Langmuir isotherm model is written thus:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad \text{Eqn. 14}$$

Where θ , C and K_{ads} symbolize proportion of surface coverage, concentration of inhibitive compounds and binding strength of molecules of inhibitive compounds, respectively. K_{ads} is connected to Gibb's free energy change of adsorption (ΔG_{ads}) by [Eqn. 15](#).

$$K_{ads} = \frac{1}{55.5} \exp[-\Delta G_{ads}/RT] \quad \text{Eqn. 15}$$

Where T , 55.5, R and K_{ads} symbolize absolute temperature (K), H_2O content in solution (mol/L), universal gas constant (J/mol.K) and binding strength of molecules of the compounds (L/g) deduced from adsorption isotherm plots ([Figure 5](#)), respectively.

[Figure 5](#) shows the plots of C/θ against C for mild steel exposed to 1 M HCl solution alongside varied concentrations of the compounds SCMC, SCMC+KI and SCMC+PEG at different temperatures, respectively. Befitting correlation coefficients and slope values of unity were derived from the linearized plots proposing that the compounds adsorbed on mild steel in 1 M HCl solution following Langmuir isotherm model. Binding strength conformed to the trend of inhibition efficiency by the compounds as reported earlier in the order SCMC < SCMC+KI < SCMC+PEG thereby suggestive of the link between binding strength and inhibitory performance ([Refaey et al., 2004](#)). ΔG_{ads} values calculated from [Eqn. 15](#) yielded negative values ranging from – 12.65 to – 14.51 KJ/mol thereby verifying spontaneous physisorption mechanism by the compounds on mild steel ([Obot et al., 2009](#)). Presented in [Table 3](#) are Langmuir adsorption parameters.

5. Thermodynamic considerations

Earlier, effect of temperature to corrosion control process on mild steel immersed in 1 M HCl solution in absence as well as presence of inhibitive compounds had been explained. Two thermodynamic parameters crucial here are energy of activation (E_a) and heat of adsorption (Q_{ads}). The former gives evidence of adsorption and effect of increasing temperature on the adsorbed molecules; the latter, information about mode of adsorption as well as influence of increasing temperature on the adsorbed molecules. To achieve the former, Arrhenius equation in [Eqn. 16](#) was employed from which plot of $\log CR$ against $1/T$ for mild steel immersed in 1 M HCl solution at varied temperatures 30 – 60 °C in absence as well as presence of inhibitive compounds, respectively was deduced ([Figure 6](#)).

$$\log CR = (-E_a/2.303RT) + \log A \quad \text{Eqn. 16}$$

Where CR , T , E_a , A and R symbolize corrosion rate (mm/yr), absolute temperature (K), activation energy (KJ.mol⁻¹), pre-exponential factor and universal gas constant (J.K⁻¹.mol⁻¹), respectively.

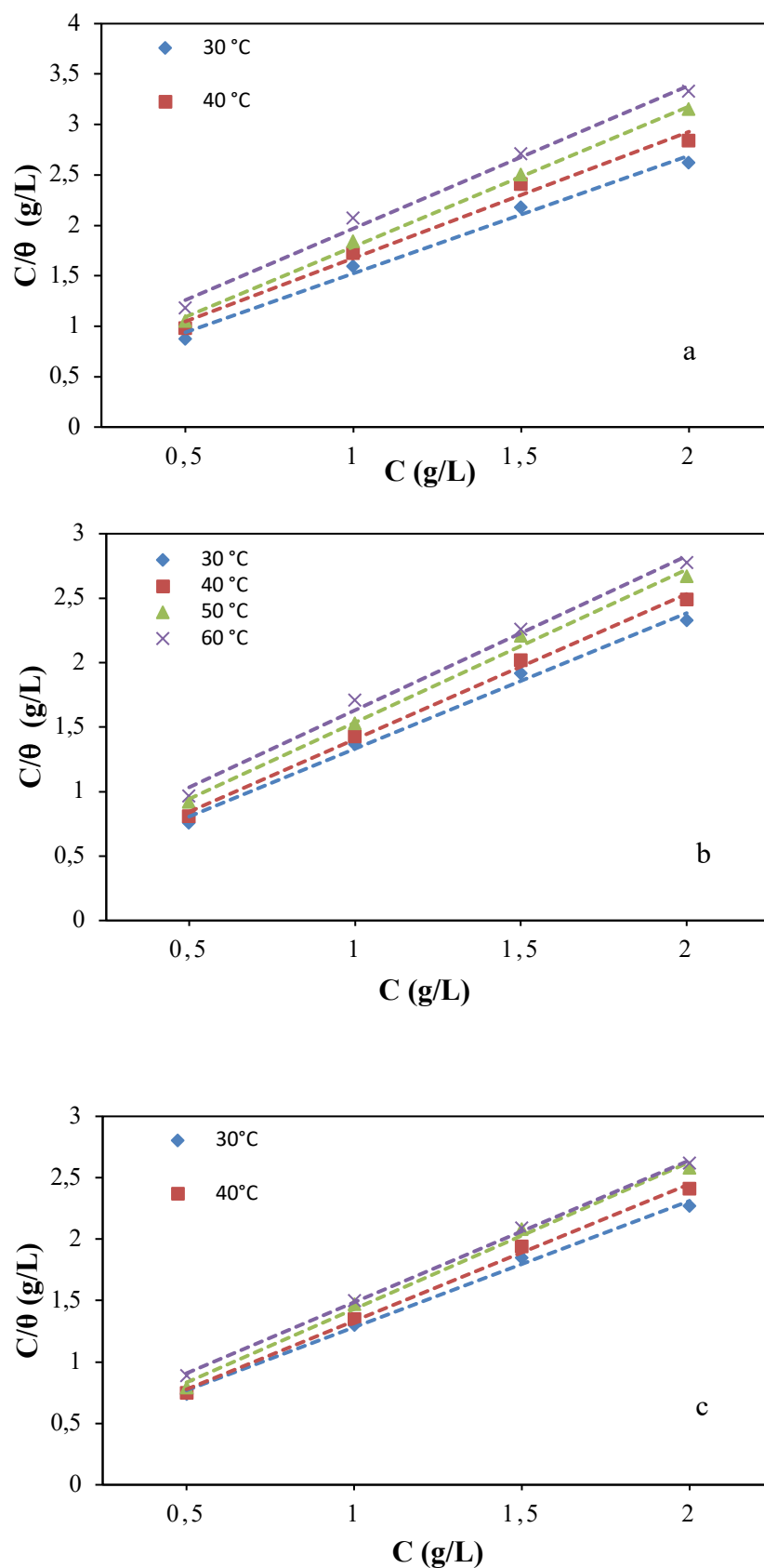


Figure 5. Langmuir isotherm for the adsorption of varied concentrations of (a) SCMC (b) SCMC+KI and (c) SCMC+PEG at different temperatures on mild steel immersed in 1 M HCl

Table 3. Adsorption parameters for inhibitive compounds on mild steel immersed in 1 M HCl at varied temperatures

| System | Temperature (°C) | Langmuir adsorption isotherm (log θ vs log C) | | |
|----------|---------------------|--|--------|-----------|
| | | Slope | R^2 | Intercept |
| SCMC | 30 | 1.1667 | 0.9886 | 0.3667 |
| | 40 | 1.3250 | 0.9868 | 0.3500 |
| | 50 | 1.4000 | 0.9977 | 0.4000 |
| | 60 | 1.4250 | 0.9918 | 0.5500 |
| SCMC+KI | 30 | 1.0750 | 0.9926 | 0.2625 |
| | 40 | 1.1450 | 0.9962 | 0.2600 |
| | 50 | 1.1750 | 0.9944 | 0.4000 |
| | 60 | 1.1750 | 0.9926 | 0.5000 |
| SCMC+PEG | 30 | 1.0667 | 0.9958 | 0.2000 |
| | 40 | 1.1133 | 0.9969 | 0.2500 |
| | 50 | 1.2000 | 0.9955 | 0.2500 |
| | 60 | 1.1533 | 0.9990 | 0.3700 |

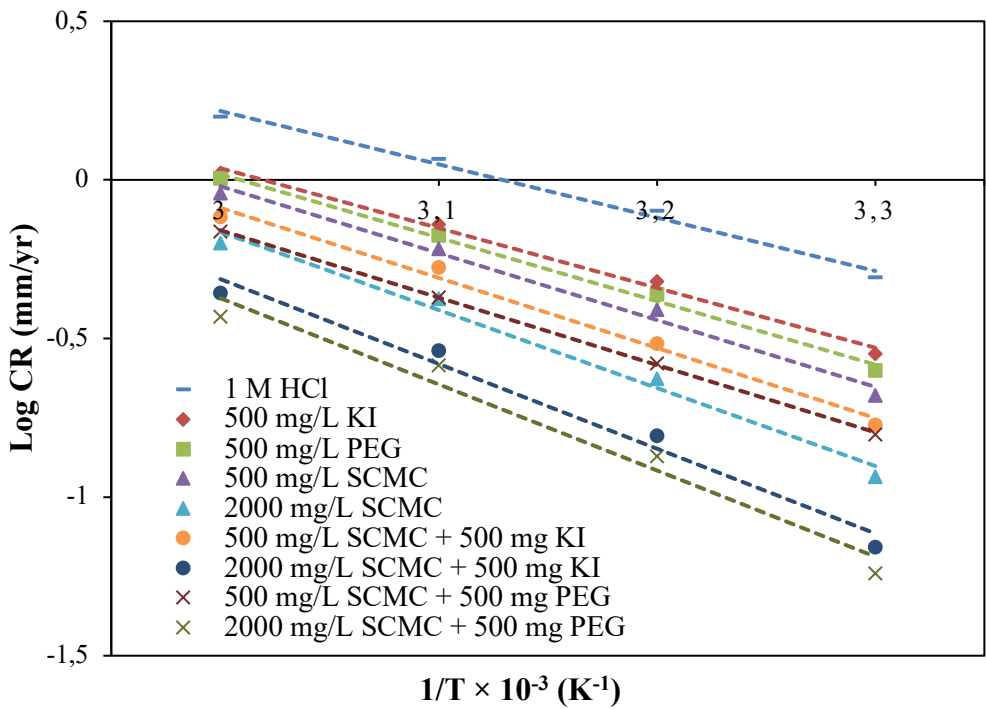


Figure 6. Log CR against $1/T$ for mild steel immersed in 1 M HCl and inhibited solutions

From the linearized plot, E_a values obtained from slope ($-E_a/2.303R$) and presented in **Table 4** were greater in inhibited solutions relative to uninhibited one. This shows that the compounds adsorbed on the mild steel surface (Oguzie, 2005). The noticeable alteration in E_a values alongside increasing concentration of the compounds is linked to perturbation of the adsorbed molecules with rise in temperature. On the other hand, heat of adsorption was calculated from **Eqn. 17**.

$$Q_{ads} = 2.303R \left[\log \left(\frac{\theta_2}{1 - \theta_2} \right) - \log \left(\frac{\theta_1}{1 - \theta_1} \right) \right] \times \left(\frac{T_1 T_2}{T_2 - T_1} \right) \quad \text{Eqn. 17}$$

Where Q_{ads} , θ_1 and θ_2 at T_1 and T_2 symbolize heat of adsorption, proportion of surface coverages at lowest and highest temperatures, respectively. From the results presented in **Table 4**, calculated values of Q_{ads} were negative and lay below -40 KJ/mol proposing the compounds underwent physisorption mechanism on the mild steel (Bouyanzer *et al.*, 2006). Also, the observed fluctuation in Q_{ads} values alongside increasing concentration of the compounds is associated with agitation of adsorbed molecules alongside increasing thermal influence.

Table 4. Thermodynamic parameters for mild steel immersed in 1 M HCl and inhibited solutions

| System | E_a (KJ mol ⁻¹) | Q_{ads} (KJ mol ⁻¹) |
|----------------------------|-------------------------------|-----------------------------------|
| 1 M HCl | 32.55 | – |
| 500 mg/L KI | 36.38 | – 10.67 |
| 500 mg/L PEG | 37.66 | – 14.97 |
| 500 mg/L SMC | 40.21 | – 18.06 |
| 1000 mg/L SMC | 41.49 | – 17.12 |
| 1500 mg/L SMC | 40.85 | – 16.76 |
| 2000 mg/L SMC | 47.87 | – 20.90 |
| 500 mg/L SMC + 500 mg KI | 42.44 | – 16.31 |
| 1000 mg/L SMC + 500 mg KI | 45.00 | – 17.64 |
| 1500 mg/L SMC + 500 mg KI | 44.68 | – 15.59 |
| 2000 mg/L SMC + 500 mg KI | 51.38 | – 24.36 |
| 500 mg/L SMC + 500 mg PEG | 40.85 | – 13.20 |
| 1000 mg/L SMC + 500 mg PEG | 44.36 | – 13.99 |
| 1500 mg/L SMC + 500 mg PEG | 44.68 | – 14.14 |
| 2000 mg/L SMC + 500 mg PEG | 51.70 | – 21.93 |

6. Potentiodynamic polarization technique

This is a well-known method employed in quantifying corrosion of metals by measuring potential difference between the reference and working (metal under study) electrodes. By application of the potential difference to the two electrodes, current is induced and measured. This measured current is related to corrosion rate on the working electrode expressed as current per unit area simply known as *current density*. **Figure 7** shows polarization plot for mild steel immersed in 1 M HCl solution in absence as well as presence of inhibitive compounds at 30 °C, respectively.

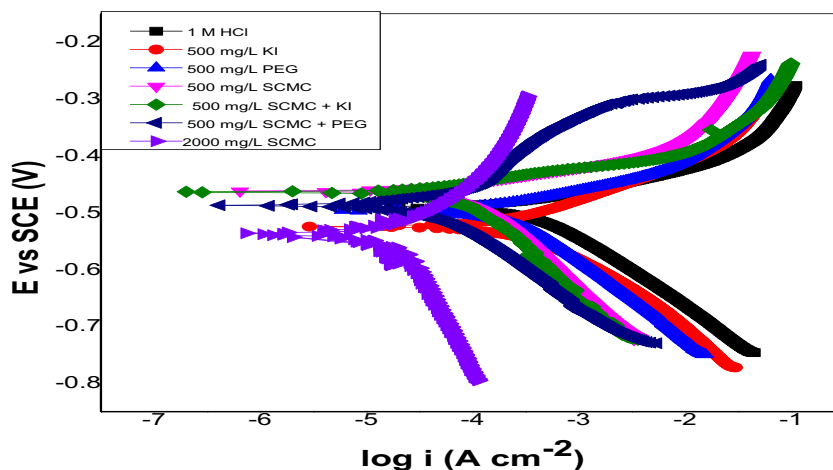


Figure 7. Polarization plot for mild steel immersed in 1 M HCl and inhibited solutions at 30 °C

From the figure, corrosion current density was recorded to be 749.30 $\mu\text{A}/\text{cm}^2$ in 1 M HCl solution as both anodic and cathodic reactions occurred complying to Tafel's. However, on introducing 500 mg KI, PEG and SCMC respectively into 1 L of 1 M HCl solution, corrosion current density was abated as both anodic and cathodic branches of the curves diverged to relatively lower values of corrosion current density according to the degree of inhibitive effect by the compounds in the order KI (355.20 $\mu\text{A}/\text{cm}^2$) < PEG (348.20 $\mu\text{A}/\text{cm}^2$) < SCMC (334.90 $\mu\text{A}/\text{cm}^2$) < SCMC+KI (245.60 $\mu\text{A}/\text{cm}^2$) < SCMC+PEG (214.70 $\mu\text{A}/\text{cm}^2$) as shown in **Table 5**. Moreover, corrosion current density was further abated to 204.70 $\mu\text{A}/\text{cm}^2$ in 2000 mg/L SCMC which stressed the concentration dependent performance of the compound to corrosion control process. These results showed that weight loss and potentiodynamic polarization measurements are correlational.

On the other hand, referring to E_{corr} of 1 M HCl solution, presence of inhibitive compounds veered corrosion potential to anodic direction in an inert sense within 500 mg/L SCMC, SCMC+KI as well as SCMC+PEG respectively and to cathodic direction within 500 mg/L KI, 500 mg/L PEG and 2000 mg/L SCMC. Observably, lowest concentration of 500 mg/L SCMC was anodically inclined whose anodicity tilted towards cathodicity on mixing alongside cathodically inclined additives KI and PEG, respectively. However, on increasing concentration of SCMC to 2000 mg/L, the system became cathodically inclined. This shows that more efficient inhibitive compounds would be inclined to mollifying corrosion of mild steel in 1 M HCl solution through cathodic engagement. Nevertheless, the compounds have proven to be good inhibitors by dampening both anodic and cathodic reactions on mild steel immersed in 1 M HCl solution and as a result are deemed as mixed type inhibitors (Li *et al.*, 2008). Presented in **Table 5** are polarization parameters for mild steel immersed in 1 M HCl solution in absence as well as presence of inhibitive compounds, respectively.

Table 5. Polarization parameters for mild steel immersed in 1 M HCl and inhibited solutions

| System | I_{corr} ($\mu\text{A}/\text{cm}^2$) | E_{corr} (mV vs SCE) | IE (%) |
|---------------------|--|----------------------------------|--------|
| 1 M HCl | 749.30 | – 500.80 | – |
| 500 mg/L KI | 355.20 | – 521.40 | 52.60 |
| 500 mg/L PEG | 348.20 | – 502.40 | 53.53 |
| 500 mg/L SCMC | 334.90 | – 448.60 | 55.31 |
| 500 mg/L SCMC + KI | 245.60 | – 468.50 | 67.22 |
| 500 mg/L SCMC + PEG | 214.70 | – 482.10 | 71.35 |
| 2000 mg/L SCMC | 204.70 | – 550.60 | 72.68 |

Conclusion

SCMC performed as a good inhibitor (64.12 %) which was made much better on mixing alongside PEG to yield 94.02 % at 30 °C for 24 hr immersion. Conversely, increasing immersion time and temperature decreased inhibition efficiency of the compounds. Thermodynamic considerations conveyed that the compounds curbed corrosion of mild steel in 1 M HCl solution through physical adsorption. Adsorption obeyed Langmuir isotherm model. The compounds were noted to have demonstrated mixed-type behaviour by mollifying both anodic and cathodic reactions on mild steel in 1 M HCl solution. Lastly, inhibitory performance by the compounds was hinged on immersion time, temperature, concentration of the compounds and possibly combined effect.

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Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

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