



Received: 23 February 2017  
Accepted: 07 April 2017  
First Published: 19 April 2017

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Reviewing editor:  
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## MATERIALS ENGINEERING | RESEARCH ARTICLE

# Inhibitive characteristics of cetylpyridinium chloride and potassium chromate addition on type A513 mild steel in acid/chloride media

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**Abstract:** Linear polarization resistance and open circuit potential methods were used to assess the inhibitive and adsorption behaviour of Potassium chromate ( $K_2CrO_4$ ) and Cetylpyridinium chloride (CPC) on mild steel (MS) in HCl/NaCl solution. Results obtained show that  $K_2CrO_4$  and CPC inhibit MS significantly against massive degradation. However, under open circuit potential method, inhibition efficiency of  $K_2CrO_4$  decreased at higher concentration. The synergistic effect of both inhibitors is positive on the protection of mild steel against corrosion. An inhibitive efficiency of over 60% was recorded for the combined admixture. Morphological study showed that the exposed steel with the presence of  $K_2CrO_4$  and CPC possess some corrosion product with lesser pitting effect compare to unprotected steel with severe surface deterioration and uniform corrosion degradation. The corrosion inhibition performance of  $K_2CrO_4$  and CPC on mild steel surface was found to follow Langmuir adsorption isotherm model.



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### PUBLIC INTEREST STATEMENT

The detrimental challenge by corrosion is worth studying in sea water applications, since corrosion problem pose a huge cost for new design and maintenance globally. Mild steel is the most available form of steel due to its low and excellent metallurgical properties however its susceptibility to corrosion is alarming. Appropriate efforts have been made to avert this catastrophes ranging from environmental pollution to loss of life. Research has proven that organic or inorganic chemical compounds with active electronegative group are effective in minimizing the corrosion aggressiveness. Meanwhile current trend has affirmed that the easiest way to develop or improve the properties of new inhibitor is via the synergistic application. Hence, this research aimed at investigating the inhibitive synergistic ability of cetylpyridinium and potassium chromate on corrosion behaviour of mild steel in aqueous dominated solution. This work is a contribution towards finding right improves inhibitor to minimize deterioration and extend life span of steel.

**Subjects: Materials Science; Corrosion-Materials Science; Metals & Alloys; Surface Engineering-Materials Science**

**Keywords: mild steel; polarization; interfaces; passivation; micrographs**

**1. Introduction**

Mild steel are industrially important especially in water transportation conveying tank, marine vessels, petrochemical septic tank, automobile, pickling’s plants, construction and oil well oxidizing system (Popoola, Fayomi, & Adeleke, 2013; Solomon, Umoren, Udoso, & Udoh, 2010). However there are impediments resulting from abundance of sea-salt water and sulphide ion during conveyance especially in marine related application (Shanti & Rajendran, 2013). Corrosion by sea water, *aqueous corrosion*, is an electrochemical process, and steel when in contact with sea water have a specific electrical potential at a specific level of sea water acidity or alkalinity which is the pH. The disadvantage of seawater is in the chloride attack to steel structures. Seawater is an aqueous solution of salts with salinity equals 3.5 %. The composition of artificial ocean water according to ASTM D1141–98(2013) standard is described in Table 1 (Zakowski, Narozny, Szocinski, & Darowicki, 2014).

More so the presence of H<sup>+</sup> within sea water salt concentration aids severe degradation and hydrogen embrittlement (Song & Velu, 2007; Zakowski et al., 2014). Thus, the consequence of chloride-acid attack or failure due to general corrosion had been widely reported (Quraishi & Sharma, 2005). Corrosion is a plight that faces everyone who works with metallic materials (Miksic, Boyle, & Wuertz, 2004; Quraishi & Sharma, 2005). Researchers are taken various steps to combat oceanic menaces of chloride and acid attack on steels which covered Inhibitive surface treatment (Miksic et al., 2004), alloy development (Trethewey & Roberget, 1995), use of sacrificial or anode system (Fayomi, Gbenebor, Abdulwahab, Bolu, & Popoola, 2013), cathodic or impressed current system (Loto & Popoola, 2011), organic and inorganic inhibitors (Ping, Cheng, & He, 2010). These methods are often used to mitigate against severe degradation. Moreover replacement of carbon steel has not been successfully achieved due to its importance in manufacturing system such as construction and fabrication industries (Fayomi & Popoola, 2014; Killeen & Killen, 2005).

The replacement of detrimental effect of seawater for general purposes in the industries, has also not be fully minimized due to production processes hence, the need for continuous searching for

**Table 1. Artificial ocean water composition according to the ASTM D1141–98(2013) (Zakowski et al., 2014)**

	Salt concentration (g/dm <sup>3</sup> )	Salt (%)
NaCl	24.53	0.681
MgCl <sub>2</sub>	5.2	0.144
Na <sub>2</sub> SO <sub>4</sub>	4.09	0.114
CaCl <sub>2</sub>	1.16	0.0322
KCl	0.695	0.0193
NaHCO <sub>3</sub>	0.201	0.00558
KBr	0.101	0.00280
H <sub>3</sub> BO <sub>3</sub>	0.027	0.000749
SrCl <sub>2</sub>	0.025	0.000694
NaF	0.003	0.0000833
Ba(NO <sub>3</sub> ) <sub>2</sub>	0.0000994	0.00000276
Mn(NO <sub>2</sub> ) <sub>2</sub>	0.000034	0.000000944
Cu(NO <sub>3</sub> ) <sub>2</sub>	0.0000308	0.000000855
Zn(NO <sub>3</sub> ) <sub>2</sub>	0.0000096	0.000000266
Pb(NO <sub>3</sub> ) <sub>2</sub>	0.0000066	0.000000183
AgNO <sub>3</sub>	0.00000049	0.0000000136

corrosion inhibitor of mild steel in acid/chloride conditions become needful (Dan et al., 2010; Fayomi & Popoola, 2014). The mechanism of corrosion inhibition may be by film formation (Abdulwahab, Popoola, & Fayomi, 2012) on the substrate that could be effective if the film is continuous and tenacious and if otherwise could aggravate localised or pitting corrosion (Zakowski, 2011). Inhibitors could also form salt particles of the aggressive ions in the solution thereby reducing the mobility and the rate of attack of the negative ions on metallic substrate (Abdulwahab et al., 2012).

Cetylpyridinium chloride and potassium chromate are inorganic substances that are available in the market among other metallic salts (Oloruntoba, Abbas, & Olusegun, 2000). The choice of the two substances is on the fact that they are not poisonous in small quantities and when in ionic form can form positive polar region for the attraction of negative ions especially chloride from seawater. Cetylpyridinium and potassium chromate may also serve for neutralisation of other harmful metals in the seawater environment (Fayomi & Popoola, 2013; Oloruntoba et al., 2000). Chloride ions had been reported as being less hydrated than sulphate ions hence provides stronger tendency to adsorb on the metal by creating excess negative charge towards the solution phase, which favours synergetic adsorption on the metal surface (Noor, 2007; Pandiarajan, Rajendran, & Joseph Rathish, 2014; Shylesha, Venkatesha, & Praveen, 2011). Meanwhile current trend has affirmed that the easiest way to develop or improve the properties of new inhibitor is via the synergistic application (Fayomi et al., 2013; Ridhwan, Rahim, & Shah, 2012). Hence, this research is aimed at investigating the inhibitive synergistic ability of cetylpyridinium and potassium chromate on the corrosion behaviour of the mild steel in acid/chloride media.

## 2. Experimental procedures

### 2.1. Sample preparation

Sectioned mild steel test coupons with spectrometer chemical composition in (wt %) is shown in Table 2. The dimensions were (30 × 20 × 2) mm before mounting with an epoxy resin. The coupons were polished to a mirror-like nature with emery papers and cleaned in acetone. It was further dried and weighed with an analytical balance. Each sample weight was recorded and labelled correctly. The chemicals used were of analytical reagent grade which were prepared using distilled water. Concentrations of acid were prepared by using double distilled water and the concentration range of inhibitors was 2–10 ml in each of 40 ml 0.5 M HCl mixed with 3.65% NaCl solution. The molecular structures of the prepared inhibitor are shown in Figures 1 and 2. The electrochemical investigation was performed at 25°C.

### 2.2. Linear polarization resistance

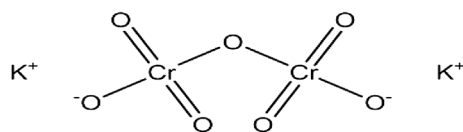
Autolab PGSTAT 101 Metrohm potentiostat/galvanostat was used to obtain linear polarization measurements for potential-current trend. The sectioned mild steel coupon was mounted with resin, with a surface area of 1 cm<sup>2</sup> and connected with electrode cell containing 40 ml of chloride electrolyte, with and without inhibitor. Graphite rod was used as auxiliary electrode and silver chloride electrode (SCE) function as a reference electrode. Linear polarization resistance potential scan range was from –1.5 V to +1.5 mV at scan rate of 0.0012 V/s. The corrosion potential (*E*<sub>corr</sub>), and current density (*j*<sub>corr</sub>) data were evaluated from the Tafel plots. The surface coverage (*θ*) and the percentage inhibition efficiency (% IE) were calculated with Equations (1) and (2).

$$\Theta = 1 - \frac{I_{\text{corr}}}{I_{\text{ocorr}}} \quad (1)$$

**Table 2. Chemical composition of mild steel sample immersed in acid/chloride media**

Element	C	Mn	Si	P	S	Al	Ni	Fe
Wt %	0.18	0.45	0.18	0.01	0.031	0.005	0.008	99.19

Figure 1. Molecular structure of potassium chromate ([https://pubchem.ncbi.nlm.nih.gov/compound/potassium\\_chromate#section=Information-Sources](https://pubchem.ncbi.nlm.nih.gov/compound/potassium_chromate#section=Information-Sources)).



The percentage inhibition efficiency (% IE) was calculated from corrosion current density values using the equation.

$$I.E\% = 1 - \frac{I_{\text{corr}}}{I_{\text{ocorr}}} \times 100 \quad (2)$$

where  $I_{\text{corr}}$  is inhibited corrosion current densities and  $I_{\text{ocorr}}$  is Un-inhibited corrosion current density.

### 2.3. Morphological characterization

The sectioned samples mounted with cold resins sample was prepared mechanically using grade of SiC grit of 80, 150, 400, and 1,200 grits before polishing to 9  $\mu\text{m}$  with Pen Struers diamond paste. Thereafter cleansed with deionized water and placed in a desiccator before used for potentiodynamic polarization according to ASTM standard G1-03(2011). The obtained samples under investigation were then analysed and examined with the help of ME 600T polarising optical microscope with built in camera.

## 3. Results and discussion

### 3.1. Potentiodynamic polarization study

Presented in Figure 3 is the synergistic effect of  $\text{K}_2\text{CrO}_4$  and CPC inhibitive activity on mild steel in 0.5 M HCl + 3.65% NaCl contaminated medium under linear polarization system. The open circuit potential plots of the electrochemical influence of  $\text{K}_2\text{CrO}_4$ /CPC inhibitor on the corrosion of mild steel in its contaminated medium are shown in Figure 4. Table 3 shows linear polarization Tafel data of admix Potassium chromate and Cetylpyridinium chloride in varying concentration. From the observed Tafel plots the corrosion rate values for  $\text{K}_2\text{CrO}_4$ /CPC inhibited steel shows less dissolutions. At 0%  $\text{K}_2\text{CrO}_4$ /CPC spontaneous anodic degradation of the steel sample were seen with obvious possibility of formation of pits within the interfacial surfaces. Although this is expected since metallic corrosion is heterogeneous in nature especially in the presence of aqueous medium (Zakowski et al., 2014). However, in other case of inhibited steel, with 2.5–10% inhibitor admix in 0.5 M HCl + 3.65% NaCl solution a remarkable decrease in corrosion rate occurred from 3.120 mm/years of control sample to 1.020 mm/years for 2.5% inhibited steel. Interestingly  $\text{Cl}^-$  ions within the acid and salt solution are liable for the corrosive activity and deterioration that occurred on the steel interface. In practise corrosion inhibiting complexes intermingle with the cells diffusion or redox system hence, the anode–cathode polarization plots in Figure 3 show passive characteristics as results of the inhibiting activity of the  $\text{K}_2\text{CrO}_4$  and CPC compound. Thus, the corrosion potential “ $E_{\text{corr}}$ ” tend toward more positive region signifying that the contraction of inhibition by  $\text{K}_2\text{CrO}_4$  and CPC is by surface active coverage principle as against the control sample. This surface active compound exhibited by the synergistic admix inhibitor act to prevent infringement of hydrogen and chloride ion thereby limiting the precipitation of H and Cl fruition within the surfaces. The corrosion potential, corrosion rate moves with change in corrosion current values as indicated in Figure 3.

One notable observation is less change in performance with  $\text{K}_2\text{CrO}_4$ /CPC as concentration of the inhibitor increases. In general 2.5%  $\text{K}_2\text{CrO}_4$ /CPC concentration has inhibition efficiency of 99.95%, while at 5.0%  $\text{K}_2\text{CrO}_4$ /CPC, the inhibition efficiency is 99.94% at 7.5%  $\text{K}_2\text{CrO}_4$ /CPC, the inhibition efficiency is 99.95%, and at 10.0%  $\text{K}_2\text{CrO}_4$ /CPC, the inhibition efficiency is 99.94%. In other word,

Figure 2. Molecular structure of cetylpyridinium chloride ([https://pubchem.ncbi.nlm.nih.gov/compound/cetylpyridinium\\_chloride#section=IUPAC-Name](https://pubchem.ncbi.nlm.nih.gov/compound/cetylpyridinium_chloride#section=IUPAC-Name)).

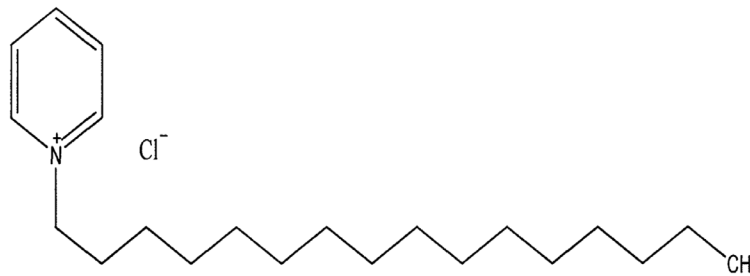
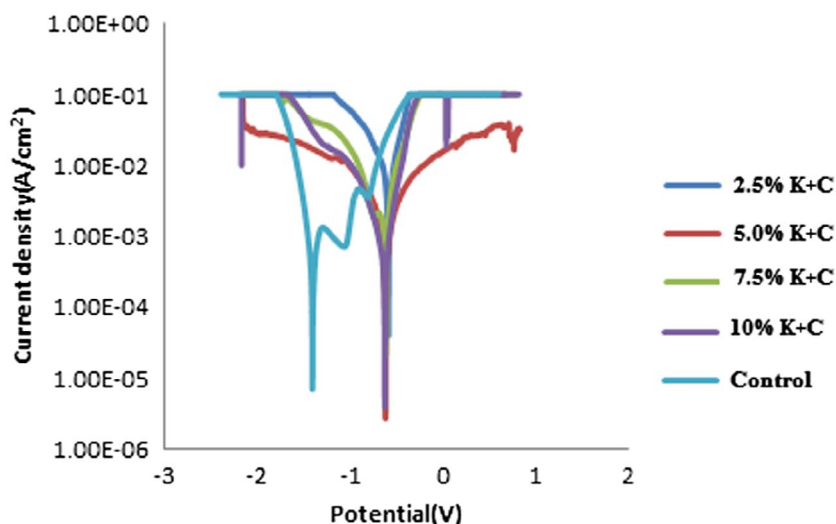


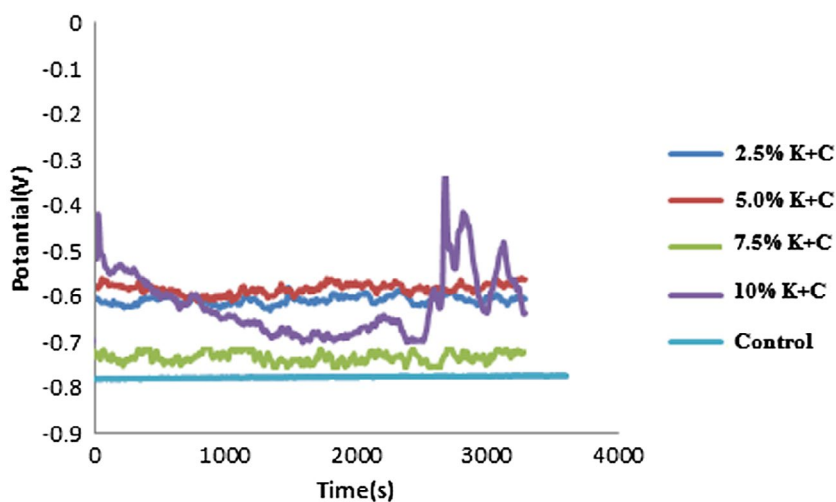
Figure 3. Anodic and cathodic polarization curve for MS in 2.5–10% at 0.5 M HCl + 3.65% NaCl solution.



changes in potentials indicate that  $K_2CrO_4/CPC$  has substantially impacted on the mechanism of the corrosion progression as a result of the film laid on the metal surface.

In Figure 4, the change in corrosion potential against time was observed from the open circuit potential curve. The electrochemical performance of  $K_2CrO_4/CPC$  in 0.5 M HCl + 3.65% NaCl indicated that there were similar corrosion progressions of inhibitive performance of OCP with linear polarization behaviour of steel. This implies that  $K_2CrO_4/CPC$  instigate passivation through adsorption by reducing the active surface area prone to corrosive medium environment.

Figure 4. Open circuit polarization for MS in 2.5–10% at 0.5 M HCl + 3.65% NaCl solution.



**Table 3. Linear polarization data for MS in 2.5–10% at 0.5 M HCl + 3.65% NaCl solution**

Sample	Ecorr, Obs (V)	jcorr (A/cm <sup>2</sup> )	CR (mm/year)	Surface coverage	Inhibitor efficiency
Control	-1.532	2.10E-02	3.120	-	-
2.5	-0.843	1.03E-05	1.030	0.9995	99.95
5.0	-0.850	1.10E-05	1.098	0.9994	99.94
7.5	-0.843	1.03E-05	1.031	0.9995	99.95
10	-0.852	1.11E-05	1.099	0.9994	99.94

### 3.2. Mechanism of inhibition efficiency and adsorption study

Figure 5 displays the comparative chart of inhibitory efficiency (IE) on mild steel substrate against synergistic inhibitive concentration in 0.5 M HCl + 3.65% NaCl solution. Observed on the chart are the potentiodynamic polarization (PP-Ecorr), Potentiodynamic-corrosion current (PP-Icorr), and polarization-corrosion rates (PP-CR). It was apparent that the inhibitory efficiency from all the processes agreed. More so this confirms the phenomena of adsorption to be linked with the molecular chemical activities of the molecule, ion, and the hydroxyl present.

The corrosion rates efficiency increases with increasing concentration of K<sub>2</sub>CrO<sub>4</sub>/CPC as inhibitor between 2.5 and 7% but slightly decreased at 5 and 10% concentrations. Icorr rises to about 100 percent while the PP-Ecorr ranges around 40% efficiency with increasing concentration of K<sub>2</sub>CrO<sub>4</sub>/CPC. The Langmuir plot (Figure 6) on the surface features shows a linear relationship with the increase in the concentration of the inhibitor this confirms a continuous adsorption of the corrosion product on the mild steel substrates and a protection on the mild steel substrate. The linear regression coefficient correction factors (R<sup>2</sup>) is almost unity – 0.998 which means all the surfaces of the mild steel substrate is covered with adsorbed corrosion products hence, the corrosion protection is achieved.

The adsorption characteristics of this study are centred on the molecular structure and the atom of the adsorbents inhibitor which are traced to physical adsorption between metal surface/charge atoms of the inhibitor (Abdulwahab et al., 2012). The change in the current at both anodic and cathodic region acted increasingly with the concentration of the mixed inhibitor at the metal interface reactions. The mechanism of these mixed inhibitory complex compounds is a blend of metal surface blockage, possible salt formation and the electrostatic force repulsion between the main adsorbed species and the medium ion. Hydrogen evolution generation that could have produced a massive impact and deterioration is distorted as a result of inhibitor effort. In an attempt to understand the metal inhibitor interaction and the metallic-complex activities on the coverage site, an adsorption mechanism facilitated according (Fayomi & Popoola, 2013) were computed for C/θ and C for potentiodynamic polarization system using Langmuir isotherm and a linear relationship was obtained with correlation regression

**Figure 5. Synergistic inhibitory efficiency (IE) for MS in 2.5–10% at 0.5 M HCl + 3.65% NaCl solution obtained from potentiodynamic polarization (PP-Ecorr), Potentiodynamic-corrosion current (PP-Icorr), polarization-corrosion rate (PP-CR).**

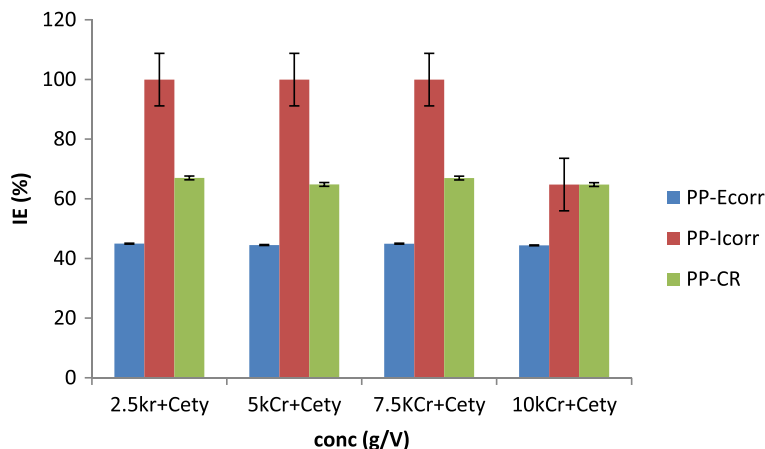
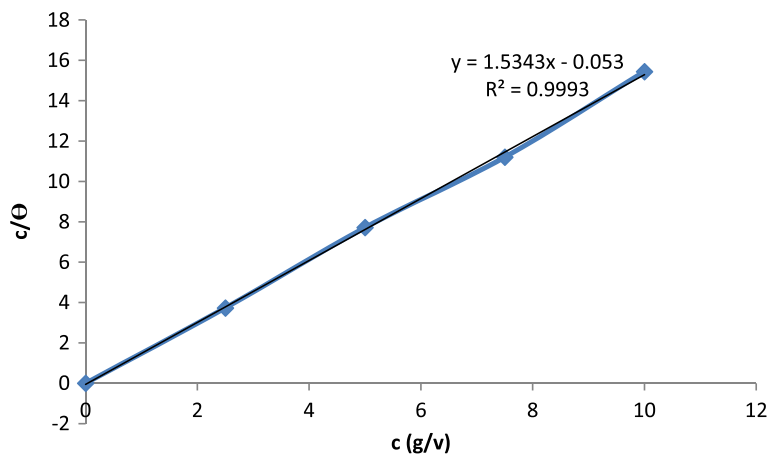




Figure 6. Langmuir isotherm adsorption for MS in 2.5–10% at 0.5 M HCl + 3.65% NaCl solution.

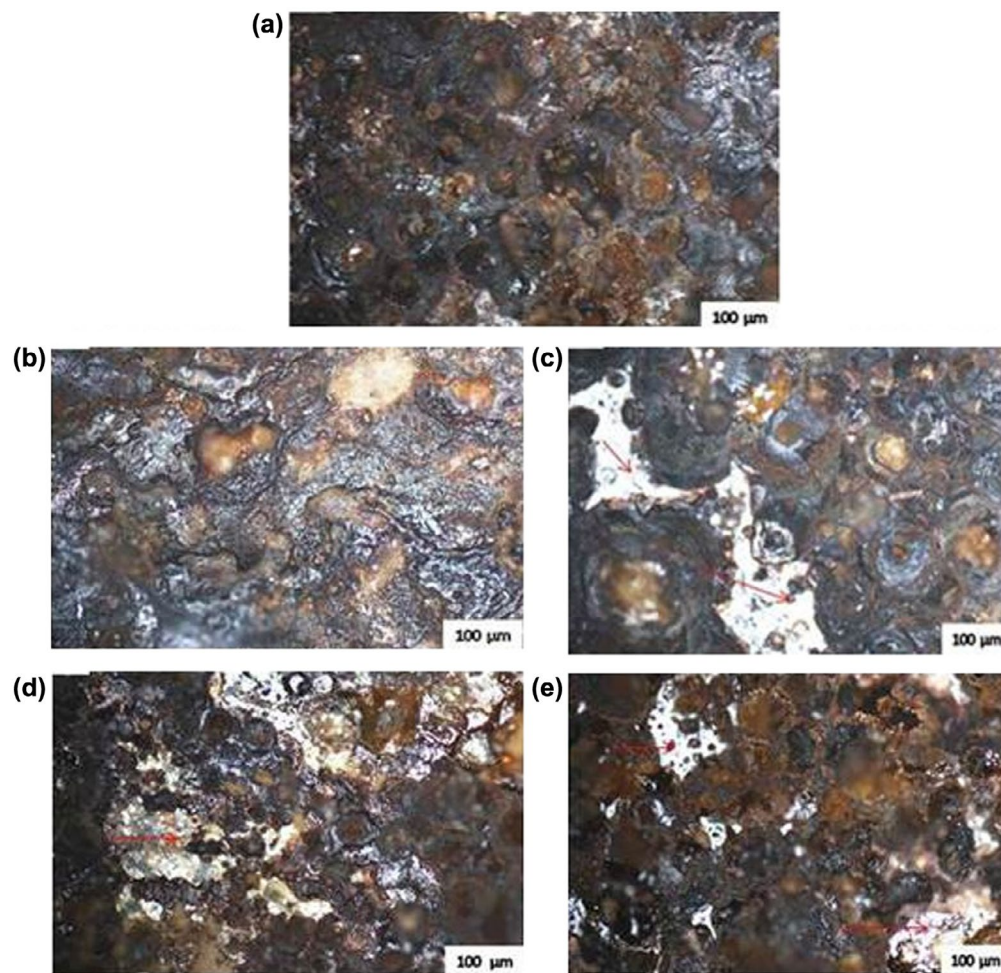


coefficient of  $R^2 = 0.998, 0.9595$  and  $0.9992$  approximate unity for the addition of potassium chromate, cetylpyridinium chloride and synergistic effect of both inhibitor respectively.

### 3.3. Microstructural examination

Figure 7(a)–(e) shows the micrograph of mild steel after exposure to 0.5 M HCl + 3.65% NaCl in the presence of  $K_2CrO_4$ /CPC varying concentration. The images show that there is a film formation on the

Figure 7. Micrographs of MS after corrosion test with (a) as-received (b) 2.5% inhibitor (c) 5.0% inhibitor (d) 7.5% inhibitor (e) 10% inhibitor in 0.5 M HCl + 3.65 NaCl medium.



samples of the inhibited steel (see Figure 7(b)–(e)) resulting to the protection of the mild steel against severe corrosion attack. Figure 7(a) shows the micrograph of the uninhibited mild steel after the corrosion test. Surface deterioration was observed due to the redox chemical reactions of corrosive ions emanating from metal solution interaction. It is obvious that the activity of medium spell negatively on the metal ions causing the release of valence electrons of  $\text{Fe}^{2+}$  cations into the acid medium thereby weakening the grey interfacial lattices. It is good to mention that a clear illustration of the micrograph especially for the as received sample in Figure 7(a) shows that pitting effect and intergranular corrosion is invariably accountable for the corrosion of MS. Little micro-pits but scale of film along metal surface were noticed from the inhibited metals in Figure 7(b)–(e), this thus indicated that synergistic action of  $\text{K}_2\text{CrO}_4/\text{CPC}$  ions and its heteroatoms adsorbs onto the mild steel.

#### 4. Conclusions

- $\text{K}_2\text{CrO}_4/\text{CPC}$  synergistic admix performed effectively well in the contaminated acid-chloride medium thereby inhibiting the mild steel.
- $\text{K}_2\text{CrO}_4/\text{CPC}$  was found to be mixed type due to functional groups and heteroatoms of the compound.
- Maximum corrosion inhibitor efficiency of 99.95% was obtained for  $\text{K}_2\text{CrO}_4/\text{CPC}$  concentrate.
- Langmuir isotherm of a linear relationship with correlation regression coefficient of  $R^2 = 0.9992$  approximately unity was obtained for the addition  $\text{K}_2\text{CrO}_4/\text{CPC}$  on mild steel.

#### Funding

This work is based upon the financial support of National Research Foundation and effort by surface engineering research centre (SERC), Department of Chemical Metallurgical and Materials Engineering, the Tshwane University of Technology, Pretoria, South Africa. Covenant University is acknowledged for providing the funds for open access publication.

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#### Citation information

Cite this article as: Inhibitive characteristics of cetylpyridinium chloride and potassium chromate addition on type A513 mild steel in acid/chloride media, O.S.I. Fayomi, A.P.I. Popoola, T. Oloruntoba & A.A. Ayoola, *Cogent Engineering* (2017), 4: 1318736.

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