ORIGINAL ARTICLE

Study of the mechanism action of sodium gluconate used for the protection of scale and corrosion in cooling water system

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Received 3 June 2011; accepted 25 October 2011
Available online 7 November 2011

KEYWORDS

EIS;
Corrosion and scale;
Cooling water system

Abstract This work based on the mechanism action study of sodium gluconate (SG) for ordinary mild steel used for cooling water system treatment. In the first time, we evaluated the temperature effect on the scale inhibition of SG using statistic scale inhibition method. Result showed that the inhibition efficiency became more important with increasing temperature, at great concentration (10^{-2} and 10^{-3} M). This can be explained by forming of stable complex SG–Ca^{2+}. In the second time, the present work focuses on the study of operational parameters and corrosion products effect on SG performance using potentiodynamic polarization and electrochemical impedance spectroscopic method. The obtained results show that SG is a very good inhibitor for corrosion and scale and remains effective in the presence of corrosion products. For this study we were proposed a mechanism action for SG on metallic surface. In addition, the SG keeps its effectiveness in a more aggressive medium such as 3% NaCl. Finally, to complete the formulation, we added a not oxidizing biocide (CTAB) to SG. The results obtained show that SG remains its effective.

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1. Introduction

Several problems of cooling water systems are corrosion, scale formation and fouling by micro-organisms. These problems can appear when natural waters are used as thermal fluid and can occur jointly, reducing the thermal efficiency of the circuit with significant economic repercussions (Srisuwan et al., 2008). To attenuate or eliminate these phenomena, corrosion inhibitors are usually associated with chemical reagents. Now days, due to new laws to protect the environment, these compounds must be non-toxic and biodegradable.
In the previous papers (Touir et al., 2008, 2009, 2010a), the sodium gluconate (SG), phosphonate anion (PHOS) and monosaccarides derivatives were used to prevent corrosion and scale of ordinary mild steel in simulated cooling water. It was shown that these compounds act as anodic inhibitors and their inhibition efficiency depended on its concentration. In addition, a synergistic effect was observed for the mixture containing 48 mg L⁻¹ of 2-(2-thio-dodecyl-5-methyl-1,3,4-triazolyl) acetic acid associated with 10⁻⁴ M of MoO₄²⁻, when we were used it for corrosion inhibition in cooling water system (Cenoui et al., 2010). SEM/EDAX revealed that the SG and PHOS act as scale inhibitors. More particularly, SEM analysis revealed that the inhibitive film was formed by the organic molecules of PHOS (Touir et al., 2010a, 2010b).

In this works, for the first time, scale inhibition performance of SG was studied using static scale inhibition method at different temperature. In the second time, we evaluated the operational parameters effect and the corrosion products on the performance of SG in order to explain the mechanism adsorption. Finally, to complete the formulation, we added a not oxidizing biocide (CTAB).

2. Experimental procedure

2.1. Electrochemical cell and materials

The electrochemical study was carried out in a standard three-electrode electrolytic cell. The reference was saturated calomel electrode (SCE). All potentials were given with reference to this electrode. The counter electrode was a platinum plate of surface area of 2 cm².

The working electrode was a rotating disk g of a rod of the ordinary mild steel of 0.8 cm² cross-sectional area, a heat-shrinkable sheath leaving only the tip of the cylinder in contact with the solution and its chemical composition was previously defined (Touir et al., 2008). All experiments were carried out at a rotation rate of 1000 rpm.

Before each experiment, the electrode was polished using emery paper until 1200 grade. After this, the electrode was washed with distillate water and dried. The rotating electrode used was an INF-EL-EDI 101 type. The rotational speed adjustment was made with an independent electronic amplifier of the CTV 101 type.

The simulated solution used here was previously defined (Touir et al., 2008). This composition represents the average concentration present in the waters used in Moroccan cooling waters. Gluconate sodium (SG) and cetyltrimethylammonium bromide (CTAB) were used as corrosion, scale and biofilm inhibitors. The biocide was evaluated using the minimal inhibition concentration (MIC) technique (Choi et al., 2002; Lancini and Parenti, 1988). Their molecular structures are presented in Fig. 1. They are current commercial products. The temperature and pH were adjusted to 32 ± 1°C and 7.3 ± 0.02, respectively.

![Figure 1](image-url)  
**Figure 1** Chemical structure of inhibitors: (a) Sodium gluconate and (b) cetyl trimethyl ammonium bromide.

2.2. Polarization measurements

The working electrode was immersed in test solution during 1 h until a steady state open circuit potential (E_{ocp}) was obtained. The cathodic polarization curve was recorded by polarization from E_{ocp} to negative direction under potentiodynamic conditions corresponding to 1 mV/s (sweep rate) and under air atmosphere. After this scan, the anodic polarization curve was recorded from the E_{ocp} in positive direction. The potentiodynamic measurements were carried out using VoltaLab PGZ 100, which was controlled by a personal computer. The polarization curves were corrected for ohmic drop which was measured by electrochemical impedance spectroscopy.

For evaluation corrosion kinetic parameters a fitting by Steren–Gery equation was used. The inhibition efficiency was evaluated from the measured J_{corr} values using the relationship:

\[
IE\% = \frac{I_{corr}^0 - I_{corr}}{I_{corr}^0} \times 100
\]

where \(I_{corr}^0\) and \(I_{corr}\) are the corrosion current densities values without and with inhibitor, respectively.

2.3. EIS measurements

The electrochemical impedance spectroscopy measurements were carried out using a transfer function analyser (VoltaLab PGZ 100), with a small amplitude ac. Signal (10 mV rms), over a frequency domain from 100 KHz to 10 mHz at 32 °C and an air atmosphere. The results were then analysed in terms of equivalent electrical circuit using Bouckamp program (Boukamp, 1993). The polarization resistance \(R_p\) is obtained from the diameter of the semicircle in Nyquist representation. The inhibition efficiency of the inhibitor has been found from the relationship:

\[
IE\% = \frac{R_p^0 - R_p}{R_p^0} \times 100
\]

where \(R_p^0\) and \(R_p\) are the polarization resistance values in the absence and the presence of inhibitor, respectively.

2.4. Scale tests

The inhibition scale was evaluated according to NACE Standard TM 0374-95 (NACE, 1997) in flasks to which calcium and M-alkalinity were added. The protocol consists to mixer two equal volumes of 500 mg/l of solution calcium (like ion) with a total solution of alkalinity 1200 mg/l to prepare starting from a report/ratio 80/20 of bicarbonate/carbonato (NACE solution). The flasks were incubated for 24 h at 55 °C. After the incubation period, the flasks were removed from the heat source (an oven) and an aliquot was filtered through a 0.45 µm membrane. The filtrate was titrated for calcium (the calcium concentration is determined by a chemical dosage with EDTA). The percent of calcium inhibition (%IE_C) was determined according to NACE Standard (Amjad, 1995; Kumar et al., 2010):

\[
IE_C\% = \frac{Ca_t - Ca_b}{Ca_t - Ca_b} \times 100
\]
where $C_a$ is the concentration of $Ca^{2+}$ in the presence of inhibitor after tests, $C_b$ the concentration of $Ca^{2+}$ in the absence of inhibitor after tests, and $C_c$ is the concentration of $Ca^{2+}$ before tests.

3. Results and discussion

3.1. Corrosion inhibition

One of the most simple qualitative methods to monitor the protective layer formation on iron is the measurement of open circuit potential in time (Felhősi et al., 2002). Fig. 2 shows the change of corrosion potential of ordinary mild steel in simulated cooling water containing different concentration of SG during 1 h of immersion. Because of the anodic inhibitory action of SG, the continuous shift of potential towards the anodic direction indicates the spontaneous adsorption of SG compounds on ordinary mild steel surface (Bommersbach et al., 2004).

However, in the absence of SG, we note a decreasing for potential this characterizes of corrosion substrate with corrosion products formation.

The test led to $10^{-1}$ M of inhibitor presents an intermediate evolution, ennoblement then a fall of the corrosion potential after 20 min of immersion, which lets predict an initiation of corrosion. The protective film formed under these conditions would not be likely to protect steel effectively. It can be supposed from the change of open circuit potential values that the rate of ordinary mild steel dissolution is continually decreasing during adsorption. Thus, the change of open circuit potential of ordinary mild steel in time gives direct qualitative information on adsorption, and its influence on ordinary mild steel dissolution rate.

In the previous studies, the corrosion inhibition of ordinary mild steel in simulated cooling water solution by SG inhibitor using weight loss and electrochemical measurement was investigated. It was shown that SG is a good corrosion inhibitor (Touir et al., 2008).

3.2. Effect of concentration of inhibitor on scale inhibition

Table 1 shows scale inhibition efficiency of SG at different temperature range in simulated cooling water system. For the blank solution, the results show that the solution became more scale with increasing temperature.

![Figure 2](image-url) Open circuit potential evolution vs. time of ordinary mild steel in simulated solution containing different concentration of SG ($T = 32{\, ^\circ C}$ and $\Omega = 1000$ rpm).

![Table 1](table-url) Scale inhibition efficiency containing different concentration of SG for 24 h of immersion at different temperature.
In the presence of SG, the inhibition efficiency generally increased with increasing its concentration for all temperature range. At great concentration ($10^{-2}$ and $10^{-3}$ M), we note also that the inhibition became more important with increasing temperature. These results indicate that higher temperature demands higher concentration to keep the scale forming ions into solution. Plummer and Busenberg showed the general behavior of CaCO$_3$ solubility as a function of temperature (Moghadasi et al., 2004). Calcium carbonate becomes less soluble as temperature increases. At hot water the more likely the CaCO$_3$ precipitation. Hence, water, which is no scaling at the down whole temperature is sufficiently high. Therefore, a higher concentration of inhibitor was required at higher temperature to prevent super saturation of the test solution.

Two common calcite scale inhibitors such as PPCA and BHPMP has been investigated and inferred that thermodynamic inhibitors decrease super saturation by lowering the ionic activity product through either chelating of the metal ions or by decreasing solution pH (Yean et al., 2008). The SG can evade scale by forming complex with Ca$^{2+}$ ions and avoids their precipitations in the salts form. The shift may indicate linking of carboxylate groups with calcium ion via electrovalent linkages as demonstrated by Kumar et al. using FTIR spectroscopy.

3.3. Parameters effect on the inhibition efficiency  
3.3.1. pH solution effect 

The pH is one the influencing parameters on corrosion and scale tendency of natural water. For this purpose, the evaluated the SG inhibition efficiency at various pH values have been carried out using the potentiodynamic polarization after 1 h of immersion in simulated solution containing $10^{-2}$ M of SG at 32 °C. The obtained results are presented in Fig. 3. It shows that the rise in the pH values does not change the kinetics type process. The electrochemical data resulting from these curves are illustrated in Table 2. It shows that the efficiency increases with increasing pH values. This behavior was explained by the presence of hydroxide ions which favors the ionic form of the molecule. The ionic form combines with Fe$^{2+}$ to give the corresponding chelate making a performance effect. These results are in very good agreement with those obtained by literatures (Touir et al., 2010a; Moghadasi et al., 2004; Gonzalez et al., 1996).

3.3.2. Influence of corrosion products on the inhibitor action

To collect more information on the mechanism of SG, the possible interaction between the corrosion products and adsorbed film by SG has been studied using electrochemical impedance spectroscopy.

In the First, we added $10^{-2}$ M of SG after 1 h of immersion of electrode in corrosive solution (after being formed a layer of corrosion products on metallic surface), then we carried out a second addition after 24 h of immersion.

The obtained results are presented in Figs. 4 and 5. It is not noted that the additions of SG ennoble remarkably the corrosion potential (Fig. 4), which returns the adsorption of SG on covered surface with corrosion products. We can still consider a probable interaction between SG molecules and corrosion products leading to the reinforcement of adsorbed film on metallic surface. In order to confirm this result, the impedance diagrams were carried out under the same conditions. The obtained diagrams (Fig. 5) can be simulated by the equivalent circuit presented in Fig. 6, which gives an account of:

- A small loop (at high frequency), which did not appear in the case of electrode immersion in simulated solution containing $10^{-2}$ M of SG (Touir et al., 2008). This effect, which

<table>
<thead>
<tr>
<th>Table 2</th>
<th>Electrochemical parameters of ordinary mild steel in simulated cooling water containing $10^{-2}$ M of SG at different pH values ($T = 32$ °C and $\omega = 1000$ rpm).</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td>$E_{corr}$ (mV/s)</td>
</tr>
<tr>
<td>7</td>
<td>−403</td>
</tr>
<tr>
<td>7.35</td>
<td>−404</td>
</tr>
<tr>
<td>8</td>
<td>−418</td>
</tr>
<tr>
<td>8.5</td>
<td>−429</td>
</tr>
<tr>
<td>9</td>
<td>−380</td>
</tr>
</tbody>
</table>

![Figure 3](image3.png)  
Figure 3  Polarization curves of ordinary mild steel in simulated solution with $10^{-2}$ M of SG at various pH values ($T = 32$ °C and $\omega = 1000$ rpm).

![Figure 4](image4.png)  
Figure 4  Open circuit potential evolution vs. time of ordinary mild steel after different adding of $10^{-2}$ M of SG ($T = 32$ °C and $\omega = 1000$ rpm).
has a capacity value 20 \mu \text{F cm}^{-2} (Table 3), may be devoted to the reinforcement of adsorbed inhibition film remained constant though shifted to the left due to the decrease in \( R_s \) as a result of presence corrosion product.

- A capacitive semicircle in the intermediate frequency range, characteristic of the double-layer relaxation in parallel with charge transfer resistance (\( C_{ct} \) between 167 and 138 \mu \text{F cm}^{-2}).
- A Warburg impedance observed at low frequency, representing the diffusion of SG through a film of corrosion products formed on metallic surface, and/or which is due the presence of porous layer. A similar phenomenon was observed by other authors (Duprat et al., 1985; Deslouis et al., 1984).

These attributions are similar to the complex behavior of copper in ammoniacal medium (Trachli et al., 2002). In fact, a capacity value of about 10 \mu \text{F cm}^{-2} was calculated at low frequency and was allotted to accumulation of corrosion products which take part in the process redox and completely disturb the kinetics of transport process.

In the second time, we added 10^{-2} \text{M of SG} after 24 h of the immersion of electrode in corrosive medium without SG (Fig. 7). It is noted a significant increase in the diameter of smaller semicircle at high frequency (can be viewed on the HF Zoom in the right figure), which confirms its attribution to the formation of film inhibitor-corrosion products. This effect did not appear in the case of the 24 h of immersion in blank solution. The inhibition efficiency remains always important and reaches 78% (Table 4).

### 3.3.3. Adherence capacity of formed film by SG

In order to test the adherence capacity of formed film by SG molecules, a new experiment was carried out using electrochemical impedance spectroscopy. It consists to immerse the electrode in simulated solution containing 10^{-2} \text{M of SG} for 1 h after this to retre and plunge it immediately in corrosive solution.

The obtained diagram is represented in Fig. 8. The proposed equivalent circuit for this system is shown in Fig. 9. It is note that the diagram is composed of two capacitive loops. The high frequency loop presumably is attributed to the adsorbed species molecules. The low frequency loop is assigned to the charge transfer resistance. The effectiveness obtained is about 90% testify the adherence of the formed film (Table 5).

### Table 3

<table>
<thead>
<tr>
<th>Curves</th>
<th>( R_s ) (\Omega \text{ cm}^2)</th>
<th>( R_{ad} ) (\Omega \text{ cm}^2)</th>
<th>( C_{ad} ) (\mu \text{F cm}^{-2})</th>
<th>( R_{ct} ) (\Omega \text{ cm}^2)</th>
<th>( C_{ct} ) (\mu \text{F cm}^{-2})</th>
<th>( R_w ) (\Omega \text{ cm}^2)</th>
<th>( C_w ) (\mu \text{F cm}^{-2})</th>
<th>IE%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>222</td>
<td>205</td>
<td>20</td>
<td>470</td>
<td>167</td>
<td>568</td>
<td>1330</td>
<td>1242</td>
</tr>
<tr>
<td>2</td>
<td>184</td>
<td>304</td>
<td>18</td>
<td>562</td>
<td>138</td>
<td>842</td>
<td>744</td>
<td>1708</td>
</tr>
</tbody>
</table>
3.3.4. Mechanism action for SG

The inhibitors usually act on metallic surface by forming an adsorption film or by implication of products corrosion layer. Adsorption depends on metallic surface charge density, the charge (or the dipole moment) of inhibitor molecules and the adsorption of the other ionic species present in solution (Ma et al., 2001; Luo et al., 1998). In the previous work (Touir et al., 2008), we showed that the metal surface is positively charged with respect to potential of zero charge (PZC).

In addition, inhibition efficiency depends on several factors, such as the number of adsorption site and their charge density, molecular size, heat of hydrogenation, interaction mode with metallic surface and the formation of metallic complexes (Wang et al., 2001). Most organic inhibitors contain at least one polar group with a nitrogen atom, sulfur or oxygen, each of them might be a chemisorption center. The higher the electron density at the center, the more effect is inhibitor.

The following explanations are postulated: SG interferes in the dissolution reaction by adsorption on metallic surface in two different ways. First, the inhibitor competes with Cl\(^{-}\)/CO\(_3\)^{2-} ions for sites at the water covered anodic surface as shown in Fig. 10.

Moreover, the inhibitor electrostatically adsorbed onto the anion covered surface. It thus plays a dynamic role at the interface and interferes with the dissolution reaction by participating in a certain number of adsorption–desorption stage, rather than solely by a blockage of sites (Hackerman and McCafferty, 1972; Hackerman et al., 1966; Murakawa and Hackerman, 1964). The two modes of adsorption are schematized in Fig. 10.

In addition, the study carried out by the corrosion products effect shows that a possibility of interaction between these products and the inhibitor molecules leading to the consolidation of protective layer. Indeed, it is extremely probable to consider the complexes formation between Fe\(^{2+}\) and SG ions such as shown in the case of scale inhibition.

3.3.5. Influence of Cl\(^{-}\)/CO\(_3\)^{2-} ions on SG performance: Study in 3% NaCl medium

In order to evaluate the inhibition potential of SG, we choose another more aggressive medium such as NaCl 3%. The electrochemical impedance spectroscopy was carried out at E\(_\text{corr}\) in turn to characterize the corrosion behavior of ordinary mild steel immersed after it had been immersed for 1 h in 3% NaCl medium containing 10\(^{-2}\) M of SG. The obtained result are given in Fig. 11.

<table>
<thead>
<tr>
<th>Time (h)</th>
<th>R(_s) (Ω cm(^2))</th>
<th>R(_{ad}) (Ω cm(^2))</th>
<th>C(_{ad}) (µF cm(^{-2}))</th>
<th>R(_{ad}) (Ω cm(^2))</th>
<th>C(_{ad}) (µF cm(^{-2}))</th>
<th>C(_{ct}) (l F cm(^{-2}))</th>
<th>R(_{ct}) (Ω cm(^2))</th>
<th>IE%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>222</td>
<td>205</td>
<td>20</td>
<td>470</td>
<td>167</td>
<td>568</td>
<td>1330</td>
<td>1242</td>
</tr>
<tr>
<td>24</td>
<td>265</td>
<td>119</td>
<td>76.5</td>
<td>322</td>
<td>1150</td>
<td>518</td>
<td>6310</td>
<td>959</td>
</tr>
</tbody>
</table>
The high frequency (HF) loop shows linear portion having a slope close to $50\,\text{C}^2\text{m}^{-2}$, suggesting the existence of a porous behavior in agreement with De Levie theory (De Levie in: Delahay and Tobias (1967)). However, the intermediate frequency loop is generally assigned to a slow transport process like diffusion.

In the presence of SG, the Nyquist plots show two capacitive loops. It can be also observed that the diameters of semicircles increase with increasing of SG concentrations comparatively to blank solution (Table 6). This means that the corrosion rate became lower by the presence of this compound.

### 3.3.6. Effect of the biocide addition (CTAB) on the inhibition of SG

Since the cooling water system is affected by different types of bacteria and other microorganisms, it was necessary to evaluate the ability of the inhibitor to control the specific growth of SG.
different test microorganisms. Cetyl trimethyl ammonium bromide (CTAB) is well known as a potential biocide and a cationic surfactant. The biocide was evaluated using the minimal inhibitory concentration (MIC) technique (Sharma et al., 1988; Lancini and Parenti, 1988). To test the influence of inhibitor along with biocide, the interference effect between them and to complete the formulation of cooling water, we added simultaneously 20 ppm of CTAB to the solution containing $10^{-2}$ M of SG. The result obtained is shown in Fig. 12. As can be seen, the Nyquist plot always appears as a capacitive semicircle in the high to the intermediate frequency range and an inductive loop in the low frequency range. The equivalent circuit for this system is shown in Fig. 13, where $R_s$ is the solution resistance, $R_{ct}$ is the resistance transfer charge, $C_{ct}$ is the interfacial capacitance of the Helmholtz double layer, $L$ is the inductance. The impedance ($Z$) of this system can be written as:

$$Z(j\omega) = R_s + \frac{R_{ct}(R + j\omega L)}{R_{ct} + R_0 + j\omega(L + R_0 R_{ct} C_{ct}) - \alpha R_{ct} C_{ct}}$$

At the high frequency range, the impedance can be simplified as:

$$Z(j\omega) = R_s + \frac{R_{ct}}{1 + j\omega R_{ct} C_{ct}}$$

The state of the adsorbed inhibitors species and the inhibition mechanism can be reflected by the change of capacitance and resistance after addition inhibitors. Table 7 shows the electrochemical parameters calculated from EIS measurement with different inhibitor concentrations. It is note that CTAB behaves like a corrosion inhibitor and has inhibition efficiency about 52%. This result is in agreement with literature work (Ramesh and Rajeswari, 2004; Gopi et al., 2007). On the other hand its presence with the SG does not affect practically its performance which decreases only from 96% to 88%. This shows that SG resists to the biocides addition and can serve as a basic element of formulation for cooling water system.

In the continuation of this work, we studied the effect of operational parameters on the inhibition capacity of the formulation ($10^{-2}$ M of SG + 20 ppm of CTAB).

4. Conclusion

This study points out to investigate the effect of operational parameters for SG inhibition in order to prevent the several problems reencountered in cooling water system can present.

The principal results are:

- SG a very good inhibitor for corrosion and scale and remains effective even in the presence of corrosion products.

<table>
<thead>
<tr>
<th>Solution</th>
<th>$R_s$ (Ω cm$^2$)</th>
<th>$R_{ct}$ (Ω cm$^2$)</th>
<th>$C_{ct}$ (μF cm$^{-2}$)</th>
<th>$R_0$ (Ω cm$^2$)</th>
<th>IE%</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaCl 3%</td>
<td>5</td>
<td>46</td>
<td>450</td>
<td>112</td>
<td>–</td>
</tr>
<tr>
<td>$10^{-2}$ M of SG</td>
<td>18</td>
<td>1476</td>
<td>190</td>
<td>2084</td>
<td>94</td>
</tr>
<tr>
<td>$20$ ppm of CTAB</td>
<td>112</td>
<td>319</td>
<td>238</td>
<td>440</td>
<td>52</td>
</tr>
<tr>
<td>$20$ ppm of CTAB + $10^{-2}$ M of SG</td>
<td>146</td>
<td>1132</td>
<td>165</td>
<td>1753</td>
<td>88</td>
</tr>
</tbody>
</table>

**Table 6** Impedance parameters of ordinary mild steel in 3% NaCl solution in the absence and presence of $10^{-2}$ M of SG ($T = 32^\circ$C and $\Omega = 1000$ rpm).

**Table 7** Impedance parameters of ordinary mild steel in simulated solution containing $10^{-2}$ M of SG with and without 20 ppm of CTAB ($T = 32^\circ$C and $\Omega = 1000$ rpm).
The static study of scale inhibition performance of SG at different temperature showed that the inhibition became more important, when the temperature increased, at great concentration (10^{-2} and 10^{-3} M) and this result can be explained by forming of complex SG–Ca^{2+}.

The mechanism action for SG is based by competition or by co-operation adsorption by the already adsorbed ions presented in simulated solution.

SG keeps its effectiveness in a more aggressive medium such as 3% NaCl.

SG remains its effective in the presence of CTAB.

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