A spectroscopic ferrozine-based assay for the calculation of corrosion rate and inhibition efficiency by Schleichera oleosa leaf and bark as an inhibitor

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Received 1 July 2015; Revised 2 September 2015; Accepted 8 September 2015; Published 2 January 2016

Abstract
In the present investigation, corrosion inhibition of mild steel in acidic solutions has been tested primarily via weight loss tests. Further the test solutions were tested by spectroscopic method as ferrozine as a reagent. By using this method, the corrosion rate of mild steel in various time interval and inhibition efficiencies of leaf and bark extracts of Schleichera oleosa were calculated. Ferrous ion chelating ability of extracts and total phenol content in extracts were calculated using the reagents such as ferric chloride and foloin –ciocalteu reagents respectively. The results of both weight loss as well as spectroscopic methods were found to comparable. The result of the present study has a promising effect in both industrial and day to day livings.

Keywords: Corrosion, Schleichera oleosa, Mild steel, Corrosion rate, Inhibition efficiency, Spectroscopic method.

1. Introduction
In many industries, steel is the material of choice in the fabrication of reaction vessels, pipelines for petroleum industries, storage tanks, and chemical batteries etc., those all can be corroded easily in the presence of acids. Continuous usage of acids in industries leads to the metal corrosion and the inhibitor usage is the most practical method for their protection. There are several methods to determine corrosion rate, such as weight loss measurements, electrochemical methods etc. Here, weight loss method together with spectrophotometric method was used to evaluate the inhibitive effect of bark and leaves extract of Schleichera oleosa (SOB and SOL), a member of sapindaceae family on mild steel in 1 M HCl solution. Results obtained for these independent methods were in good agreement.

The weight loss method is the simplest way of measuring the CR. In spectrophotometric (colorimetric) method, the concentration of dissolved iron ion (Fe²⁺) in the test solution was quantified. Spectrophotometric technique is related to the analysis of colored complex formation in solution. Some researchers reported the iron-chelation properties of phenolic groups, which is present in plant extracts (Andjelkovic et al., 2006; Acker et al., 1996). Some of them used ferrozine (FZ) as complexing agent for finding out the iron binding properties of phytochemicals. The aim of this work is to investigate the
inhibiting properties of leaf and bark extracts of Schleichera oleosa on the corrosion process of mild steel in corrosive solutions.

2. Materials and methods
2.1. Materials preparation

The experiments were carried out by MS with the following composition (in weight percentage) C=0.091; Si=0.016; Mn=0.195; S=0.013; P=0.020; Ni=0.018; Mo=0.020; Cr=0.027 and Fe=99.6. The aggressive solution of 1M HCl was prepared. Bark (B) and leaves (L) of Schleichera oleosa (SO) were collected from a moist deciduous forest of Anagan malai, which is a part of Western Ghats, in the northern end of Palakkad district, Kerala, India, cut in to small pieces, air dried and powered. The extracts were prepared by refluxing 25 g of powdered bark or leaves in 1M HCl for 3 h and kept overnight for cooling. The cooled extracts were filtered and made up to 500 ml with 1M HCl to get 5% v/v concentration. The desired concentrations of the inhibitor were prepared by diluting the above stock solution with distilled water. The concentration range of SOL and SOB used was (0.25 to 0.5) volume %.

2.2. Weight loss measurements

Mild steel specimens were sheared from commercially available cold rolled MS sheet into 5 x 1 cm² coupons for immersion studies. The specimens were mechanically polished; their edges were abraded with fine grade emery papers (NO: 400), degreased, rinsed with acetone, stored in desiccator and used for all studies. Mild steel coupons were weighed in a SHIMADZU Digital balance with an accuracy of 0.00001 g and then exposed to the selected aggressive media. In each experiment, samples were supported by a glass-hook and immersed as triplicates in 100 ml of solution (with and without inhibitors) for a predetermined time period viz., ½ h, 1 h, 3 h, 6 h, 12 h and 24 h. The specimens were taken out, neutralized with saturated sodium bicarbonate solution, washed with distilled water, dried and reweighed. The resulted weight loss of the triplicates were averaged and used for further calculations. The corrosion rate (CR) and inhibition efficiency (IE) were obtained using the following equations:

\[
\text{CR (mpy)} = \frac{534 \ w}{\text{DAT}}
\]

\[
\text{IE (%)} = \frac{W_0 - W}{W_0} \times 100
\]

where, \( w \) is the weight loss in g, \( D \) is the density of mild steel in gm/cm² (7.9 gm/cm²), \( A \) is the area of the specimen in cm², \( T \) is the exposure time in hours, \( W_0 \) is the weight loss without inhibitor and \( W \) is the weight loss with inhibitor respectively.

2.3. Spectrophotometric (or colorimetric) analysis

2.3.1. Analysis of corrosion rate and inhibition efficiency

Spectrophotometer can be used to measure the amount of iron present in both uninhibited and inhibited experimental solutions in specified periods of time. For determining the iron content in solution, Ferrozine is used as the coloring agent. FZ binds ferrous ion but not ferric ion in to a complex (Fish 1988) and this complex formation is disrupted in the presence of other chelating agents. This could be physically identified with the decreasing of red color of the complex (Dinis et al., 1994; Brittenham 2003). For this study a series of various concentrations of ferrous ion were made and mixed with 0.2 mL of FZ reagent. The color was allowed to develop for 10 minutes. The absorbance of each of the solutions was measured at a wavelength of 560 nm using UV-VIS spectrophotometer-118.

MS strip of 1 X 5 cm² area was immersed in 1 M HCl in the absence and presence of 0.5 % of both extracts. The amount of iron dissolved in the test solution was measured by withdrawing 100 µL of the
solution at various time intervals. The inhibited and uninhibited solutions were also treated with FZ reagent and the colour was compared with the colour of standard solutions in terms of absorption. From the calibration curve, the weight of Fe^{2+} ion was calculated and corrosion rate was estimated using the following formula,

\[ CR = \frac{KW_{Fe^{2+}}}{DAT} \]  

(3)

where, K is rate constant equal to 534 mpy, \( W_{Fe^{2+}} \) is weight of Fe^{2+} ion in gm, D is density of mild steel in gm / cm\(^2\) (7.9 gm / cm\(^2\)), A is area of the specimen in cm\(^2\) and T is exposure time in hours.

2.3.2. Ferrous ion chelating ability of extracts

The chelating ability of acidic extracts of SOB and SOL with ferrous ion was investigated according to the protocol of Dinis et al., (1994). 20 µL of 2 mM FeCl\(_2\) was added to 1 mL of different concentrations of the extract. The reaction was initiated by the addition of 0.2 mL of 5 mM FZ reagent. The mixture was vigorously shaken and left to stand for 10 minutes. The absorbance of the solution was measured at 562 nm. The percentage of chelation effect was calculated as

\[ C = \frac{ABS_{control} - ABS_{sample}}{ABS_{control}} \times 100 \]  

(4)

where, \( ABS_{control} \) is the absorbance of control and \( ABS_{sample} \) is the absorbance of sample. The chelating effect of the extract in presence of MS strip in test solution at various time intervals was calculated following the above procedure.

2.3.3. Determination of total phenol content (TPC) in extracts

Total phenol content in plant extracts were quantified by Folin-Ciocalteu method (Folin and Ciocalteu 1927; George et al., 2005). Aliquots of the extracts were taken in a 10 ml glass tube and made up to a volume of 3 mL with distilled water and add 5 mL of 80 % ethanol. Then 0.5 ml Folin Ciocalteu reagent (1:1 with water) and 2 mL Na\(_2\)CO\(_3\) (20 %) were added sequentially in each tube. A blue color was developed in each tube because the phenols undergo a complex redox reaction with phosphomolibdic acid in Folin Ciocalteu reagent in alkaline medium which resulted in a blue colored complex, molybdenum blue. The test solutions were warmed for 1 min, cooled and absorbance was measured at 650 nm against the reagent used as a blank. A standard calibration plot was generated at 650 nm using known concentrations of gallic acid. The concentrations of phenols in the test samples were calculated from the calibration plot and expressed as mg gallic acid equivalent of phenol/g of sample.

The total phenolic content in 0.5 % concentration of the extract in presence of MS was measured at various time intervals adopting the procedure described above. Total phenolic content was estimated as mg gallic acid equivalents (GAE) / g of extract (Rekha et al., 2012). The dried weight of plant material was calculated using the linear equation obtained on the calibration curve:

\[ y = mx + c \]  

(5)

where y is the absorbance and x is gallic acid equivalents mg / ml. Total content of phenolic compounds in solution, studied extracts in gallic acid equivalents (GAE) was calculated by the following formula (Folin and Ciocalteu1927):

\[ T = \frac{C \times V}{M} \]  

(6)

where T is the total content of phenolic compounds in mg/g GAE, C is the concentration of Gallic acid (mg/ml) established from calibration curve, V is the volume of the extract and M is the weight of pure extract.
3. Results and discussion

3.1. Corrosion rates and Inhibition efficiency from weight loss method

The results of the weight loss analysis were shown in Table 1, which showed the CR and IE for SOB and SOL in 1 M HCl at room temperature ([28±2]°C). It is clear from the table that the addition of SOB or SOL extract strongly suppressed the corrosion rate and this leads to an increase in inhibition efficiency. The increase in inhibition efficiency with the extract concentration indicated that the inhibitor molecules get adsorbed on the metal surface and it prevents further corrosion.

The effect of immersion time on MS corrosion protection of SOB and SOL showed that the corrosion rates in blank solution increased with increasing immersion time up to 6 h and then decreased. The variation in corrosion rates with the time of exposure between 1/2 h and 6 h may be due to the increase in conductance of the solution as a result of continuous addition of Fe²⁺ ion, is formed by the corrosion of mild steel. The decrease in corrosion rate after 6 h may be due to either a decrease in solubility of the electrolyte as it slowly becomes saturated with the corrosion product or the formation of some surface film which retards the corrosion of mild steel. It also understandable from the Table 1 that the inhibition efficiency increased with increasing immersion time, which can be attributed to the fact that the protective film formed on the metal surface tend to become more compact, stable, persistent and uniform by the increase of time (Ajmal et al., 2000).

3.2. Corrosion rates and inhibition efficiency from spectrophotometric method

Table 2 clearly described the results of this technique, which is clear that the absorbance increased enormously in blank solution, indicating that the Fe²⁺ ion concentration was more and hence high corrosion rate. But by comparing the absorbance of blank solution with the presence of extract solution, the absorbance decreased which point out the running down of Fe²⁺ ion concentration in solution; and so the corrosion rate reduced in the presence of extracts at each interval of time. It is clear that if SOB is used as inhibitor, CR value decreased from 252.94 to 52.33 mpy and IE increased with increase in immersion time. Competing forces of adsorption and desorption may actually explain the occasional discrepancies in IE values in presence of SOL extract. A comparative study of inhibition efficiency obtained from weight loss and spectrophotometric methods in 1 M HCl were shown in figure 1. The inhibition efficiency had same trend in both methods at 0.5 % concentration of each extract.

3.3. Metal chelating ability of bark / leaves extracts of Schleichera oleosa in 1 M HCl

The chelating capacity of 0.5 % extracts were measured in the corroding solutions in presence of mild steel at various time intervals, the results were presented in figure 2. Chelating ability of the extracts increased with increase in time of immersion. This showed a continuous Fe²⁺- extract complex formation with time. This may lead to the increase in film thickness on metal surface and this explains persistent inhibiting capacity of the extracts up to 6 h.

3.3. Estimation of total phenolic content in Schleichera oleosa extracts in 1 M HCl

Most of the oxygen containing compounds of the plant extracts is hydroxyl aromatic compounds such as poly phenolic compounds. In polyphenols, especially tannin present in plant extracts have been reported for inhibition of acid corrosion of metals (Tan and Kassim, 2011). The amount of total phenolic content present in bark/leaves extracts of Schleichera oleosa was determined with Folin-Ciocalteu reagent. Gallic acid was used as a standard compound. The calibration curve showed linearity to gallic acid in the range of 20 to 220 mg/mL with a correlation coefficient (R²) of 0.998. The total phenol content was expressed as mg/g gallic acid equivalent.
Table 1. Weight loss data for the corrosion inhibition of MS in 1 M HCl at various concentrations of SOB and SOL at room temperature.

<table>
<thead>
<tr>
<th>Conc. (%)</th>
<th>1/2 h</th>
<th>1 h</th>
<th>3 h</th>
<th>6h</th>
<th>12 h</th>
<th>24 h</th>
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<tr>
<td></td>
<td>CR (mpy)</td>
<td>IE (%)</td>
<td>CR (mpy)</td>
<td>IE (%)</td>
<td>CR (mpy)</td>
<td>IE (%)</td>
</tr>
<tr>
<td>0.00</td>
<td>2940.15</td>
<td>3101.95</td>
<td>3204.16</td>
<td>3407.97</td>
<td>2748.24</td>
<td>1800.00</td>
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<td>478.01</td>
<td>84.59</td>
<td>373.15</td>
<td>88.35</td>
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<td>450.71</td>
<td>85.47</td>
<td>328.35</td>
<td>89.75</td>
</tr>
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<td>342.79</td>
<td>88.95</td>
<td>308.07</td>
<td>90.39</td>
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<tr>
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</tr>
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<td>219.17</td>
<td>92.93</td>
<td>219.78</td>
<td>93.14</td>
</tr>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.25</td>
<td>1263.48</td>
<td>57.03</td>
<td>756.36</td>
<td>75.62</td>
<td>243.26</td>
<td>92.41</td>
</tr>
<tr>
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</tr>
<tr>
<td>0.35</td>
<td>937.59</td>
<td>68.11</td>
<td>693.58</td>
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<td>93.12</td>
<td>97.09</td>
</tr>
<tr>
<td>0.40</td>
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<td>83.73</td>
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<tr>
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<td>601.23</td>
<td>80.62</td>
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<td>97.77</td>
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Table 2. Absorbance, CR and IE of inhibited and uninhibited solutions after exposure for predetermined periods.

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<tr>
<th>Time (h)</th>
<th>0.5</th>
<th>1</th>
<th>1.5</th>
<th>2</th>
<th>2.5</th>
<th>3</th>
<th>3.5</th>
<th>4</th>
<th>4.5</th>
<th>5</th>
<th>5.5</th>
<th>6</th>
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</thead>
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<tr>
<td>Blank</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Absorbance (nm)</td>
<td>0.11</td>
<td>0.18</td>
<td>0.26</td>
<td>0.34</td>
<td>0.36</td>
<td>0.51</td>
<td>0.6</td>
<td>0.72</td>
<td>0.82</td>
<td>0.96</td>
<td>1.04</td>
<td>1.12</td>
</tr>
<tr>
<td>CR (mpy)</td>
<td>462.25</td>
<td>353.25</td>
<td>366.34</td>
<td>351.06</td>
<td>299.15</td>
<td>347.40</td>
<td>346.54</td>
<td>369.88</td>
<td>379.95</td>
<td>390.21</td>
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</tr>
<tr>
<td>SOB</td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Absorbance (nm)</td>
<td>0.053</td>
<td>0.058</td>
<td>0.071</td>
<td>0.075</td>
<td>0.081</td>
<td>0.092</td>
<td>0.096</td>
<td>0.094</td>
<td>0.103</td>
<td>0.108</td>
<td>0.118</td>
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<tr>
<td>CR (mpy)</td>
<td>292.94</td>
<td>143.91</td>
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<td>91.58</td>
<td>80.24</td>
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<td>68.53</td>
<td>58.87</td>
<td>57.18</td>
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<td>52.33</td>
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<td></td>
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<tr>
<td>Absorbance (nm)</td>
<td>45.28</td>
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<td>73.18</td>
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<td>CR (mpy)</td>
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<td>0.058</td>
<td>0.07</td>
<td>0.12</td>
<td>0.15</td>
<td>0.05</td>
<td>0.098</td>
<td>0.108</td>
<td>0.152</td>
<td>0.179</td>
<td>0.183</td>
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<tr>
<td>IE (%)</td>
<td>279.1</td>
<td>148.27</td>
<td>119.2</td>
<td>156.99</td>
<td>155.24</td>
<td>43.61</td>
<td>72.27</td>
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<td>85.63</td>
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<td>81.13</td>
<td>77.04</td>
<td>76.07</td>
<td>78.03</td>
<td>74.19</td>
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</tbody>
</table>
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3.3. Estimation of total phenolic content in *Schleichera oleosa* extracts in 1 M HCl

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![Fig. 1. IE of SOB and SOL inhibitors for MS corrosion in 1M HCl solution by weight loss and spectrophotometric methods.](image-url)
Fig. 2. Iron chelating activity of 0.5 % *Schleichera oleosa* extracts in presence of MS.

The change in phenolic content of the test solution containing 0.5 % of the extract in the presence of MS was studied at various time intervals (Fig. 3). The phenolic content of these extracts decreased with time. In the case of SOB extract TPC varied from 26 to 11 mg/g and 25 to 14 mg/g in presence of SOL extract. This suggests that the polyphenols may either adsorb directly on the metal surface or formed complexes with Fe²⁺ ions in solution.

Fig. 3. Total phenol content in bark / leaves of *Schleichera oleosa* extracts of 0.5 % concentrations in presence of MS at various immersion time.

The hydroxyl groups of polyphenols with abundant π electrons, can capture H⁺ ions by hydrogen bonding which reduces the concentration and corrosiveness of acid solutions. The hydroxyl groups of the polyphenols readily coordinate with Fe²⁺ ion (Chen et al., 2012). These complexes block the micro anode / cathode on the metal surface and hence retard the subsequent dissolution of the metal.

3.4. Mechanism
Inhibition of MS corrosion in 1 M HCl in the presence of the extracts can be explained on the basis of molecular interaction of the inhibitor with the metals. The inhibitive effect of bark / leaves extracts of *Schleichera oleosa* is ascribed to the presence of number of organic compounds in the extracts. The organic compounds include terpenes, polyphenols, tannins etc. along with acid hydrolysis products. The first stage in the reaction mechanism of inhibitor in acid media is adsorption of inhibitor molecules on the metal surface. The adsorption of main constituents of the extracts can be attributed to the presence of hetero atoms, π electrons and aromatic / heterocyclic rings. In aqueous acidic solution main constituents exist either as natural molecules or as protonated (cation) molecules. The mode of adsorption can be visualized as follows. The steel surface bears positive charge in the acid solution (Mu et al., 1996). Chloride / sulphate ions get adsorbed on the surface. These ions should bring excess of negative charges in the vicinity of metal / solution interface and far or adsorption of the positively charged inhibitor molecules. Synergism between Cl⁻ ions and protonated inhibitor molecules showed that the inhibitors
adsorbed on the metal surface through electrostatic interaction. Literature survey on the phytochemical analysis of barks and leaves of Schleicheria oleosa revealed the presence of various constituents as lupeol, lupeol acetate, betulin, betulanic acid, beta-sitosterol, scopoletin, taraxerone, tricadenic acid A and tannin (Ghosh et al., 2011, Dan and Dan1986). If the adsorbed molecular ion on the surface is stable the corrosion of mild steel is inhibited. To be stable the component of the plant extract should lead to the formation of complex on the surface. The protection efficiency of inhibitors with longer immersion time showed the formation of insoluble complex on the mild steel surface. The chelating capacity of the extracts with Fe\(^{2+}\) ions was proved spectrophotometric method.

4. Conclusion
The corrosion control of metals is an important activity in big industries like petrochemical, fertilizer, nuclear establishment as well as in smaller industries like food processing, pharmaceuticals etc. Necessary steps to prevent corrosion are important from the point of view of economy and conservation of metals for a better and longer utilization. The corrosion protection capacity of the Schleicheria oleosa extract was evaluated using weight loss and spectrophotometric methods. In weight loss method, the experiments were carried out in varying immersion time and concentrations. Inhibitor efficiency was found to increase with inhibitor concentration. Spectrophotometric studies showed a decrease in adsorption and as such the corrosion rates found to increase with time showing high inhibition efficiency of the extracts. The decrease in Fe\(^{2+}\) ion concentration and depletion of polyphenol content in corroding medium with increasing time indicated the formation of complex in solution. The results obtained from both the techniques were in good agreement.

Conflict of interest statement
We declare that we have no conflict of interest.

Acknowledgements
The authors wish to acknowledge Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore, India, for providing the necessary facilities to carry out the studies.

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