RESEARCH LETTER

Green approach to corrosion inhibition of mild steel in hydrochloric acid medium using extract of spirogyra algae

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ABSTRACT
In the present investigation, a fresh water green algae spirogyra is used as an inexpensive and efficient mild steel corrosion inhibitor. The study is carried out in 0.5 M HCl solution using weight loss measurements, scanning electron microscopy-energy-dispersive X-ray spectroscopy, X-ray diffraction, and Fourier transforms infrared (FT-IR) techniques. The maximum inhibition efficiency was found to be 93.03% at 2 g L⁻¹. The adsorption of extract of spirogyra on mild steel surface obeys the Langmuir adsorption isotherm. Corrosion inhibition mechanisms were inferred from the temperature dependence of the inhibition efficiency as well as from calculation of thermodynamic and kinetic parameters which direct the process. FT-IR analysis of green algae spirogyra revealed the presence of hydroxyl, amino, and carbonyl groups, which are responsible for the adsorption on the mild steel surface. SEM analysis supported the inhibitive action of the spirogyra extract against the mild steel corrosion in acid solution.

ARTICLE HISTORY
Received 19 June 2015
Accepted 29 December 2015

KEYWORDS
Adsorption; corrosion; Spirogyra; SEM; FT-IR

1. Introduction
Mild steel is one of the most frequently used constructional materials in various industries due to its low cost, good ductile strength, and accessibility (1). In the industrial environments mild steel is affected by different corrosion attacks because of using acids like sulphuric acid and hydrochloric acid for various industrial processes (acid pickling, chemical cleaning, oil well acidification) (1, 2). For these reasons inhibitors are employed as one of the most practical methods for corrosion protection (3). Most of the well-known organic corrosion inhibitors are compounds containing nitrogen, oxygen, and sulphur atoms and multiple bonds. These days the uses of many organic corrosion inhibitors have been limited because they are expensive, and hazardous for the environment and human beings as well (4,5). Presently various plant extracts, containing mixture of compounds having oxygen, sulphur, and nitrogen elements, are employed as green corrosion inhibitors from acidic solution. Generally, plant extracts are non-hazardous, friendly, cheap, readily available, and renewable sources (4). Extracts of Artemisia pallens (6), henna (7), Uncaria gambir (8), and Murraya koenigii (9) have been reported as good mild steel corrosion inhibitors. Various investigation shows that plant extracts mainly contain terpenoids, davanone, linalool (6), Lawsone, Gallic acid, a-D-Glucose and Tannic acid (7), catechin (8), β caryophyllene, β gurjunene, β trans-ocimene, β hujene, β bisabolene (9), and other organic nitrogen bases, alkaloids as well as carbohydrates products (4–10). In the present study extract of filamentous green algae Spirogyra was selected as the mild steel corrosion...
inhibitor (6). Polysaccharides, proteins, or/and lipid are present on the surface of their cell walls, so it contains some functional groups such as amino, hydroxyl, and carboxyl which can act as binding sites on mild steel surface (11). *Spirogyra* extracts are viewed as an incredibly rich source of natural chemical compounds that can be extracted by simple procedures. Several researches have been reported using *spirogyra* extracts for different purposes. Pacheco et al. (12) reported that *spirogyra* can be used for the production of pigments and biohydrogen. Yi-Chao Lee and Shui-Ping Chang reported the biosorption of heavy metals from aqueous solution by *spirogyra* because of its high heavy metal ion removal capability (13). Kang et al. (14) reported that gallic acid isolated from *Spirogyra* improves cardiovascular disease through a vasorelaxant and antihypertensive effect. Trifa attar omer studied the isolation and identification of some organic constituents (Gallic acid, Kaempfertin, isoquinoline, coronilagin) and elements (Mn, Cr, etc.) in *spirogyra*. The use of *spirogyra* for the corrosion inhibition of mild steel has not been reported in literature. The extract of *spirogyra* algae contains some organic constituents; these may act as a potential corrosion inhibitor by adsorbing at the metal surface. The main objective of the present work is to estimate the inhibitive effect of extract of filamentous green algae *Spirogyra* as a green corrosion inhibitor on the corrosion of mild steel in a 0.5 M hydrochloric acid solution. The valuation of the corrosion behaviour was studied using weight loss, Fourier transforms infrared (FT-IR), the morphology of the inhibited mild steel surface was examined by scanning electron microscopy (SEM) and X-ray diffraction (XRD). Thermodynamic and kinetic data were obtained from adsorption isotherms and Arrhenius plot, respectively.

2. Results and discussion

2.1. Weight loss measurements

Values of mild steel corrosion rates $\rho$ ($\text{mg cm}^{-2} \text{h}^{-1}$), inhibition efficiency (\%), and degree of surface coverage (\(\theta\)) obtained with pre-corroded mild steel specimens after 24 h of immersion period in extract solutions in the temperature ranging from 298 to 328 K are summarized in Table 1. The data obtained for the weight loss (g cm$^{-2}$) versus concentration (g L$^{-1}$) measurements for mild steel in 0.5 M HCl of inhibitor at the studied temperature are presented in Figure 1. It is seen from the plot that the amount of material lost (g cm$^{-2}$) decreases in the presence of the inhibitor compared to the blank acid solution and is also found to be dependent on concentrations of the inhibitor. This indicates that the corrosion process on mild steel was inhibited in the presence of inhibitor molecules in 0.5 M HCl solution. Table 1 shows the variation of inhibition efficiency (% $I$) with increase in inhibitor concentrations. It was observed that extract of *spirogyra* inhibits the corrosion of mild steel in 0.5 M HCl solution, at all studied concentrations 0.5–2 g L$^{-1}$ (298–328 K). Maximum inhibition efficiency (93.09%) was shown at the 2 g L$^{-1}$ concentration of the inhibitor in 0.5 M HCl at 308 K temperature. Table 1 also demonstrates the corresponding trend of corrosion rate for various concentrations of algal extract and as estimated, corrosion rate increased gradually with inhibitor concentration in 0.5 M HCl solution. Also, the corrosion rate and inhibition efficiency increases with temperature both in the absence and presence of the inhibitor, and particularly

| Temperature (K) | Concentration (g L$^{-1}$) | Corrosion Rate $\rho$ (mg cm$^{-2}$ h$^{-1}$) | Inhibition Efficiency (%) | $\theta$ |
|----------------|----------------------------|---------------------------------------------|--------------------------|--------|
| 298            | Blank                      | 1.312                                      | 77.82                    | 0.778  |
|                | 0.5                        | 0.291                                      | 77.82                    | 0.778  |
|                | 1.0                        | 0.242                                      | 81.55                    | 0.815  |
|                | 1.5                        | 0.206                                      | 84.29                    | 0.842  |
|                | 2.0                        | 0.142                                      | 89.17                    | 0.891  |
| 308            | Blank                      | 5.011                                      | –                        | –      |
|                | 0.5                        | 1.124                                      | 77.56                    | 0.775  |
|                | 1.0                        | 0.512                                      | 89.78                    | 0.897  |
|                | 1.5                        | 0.444                                      | 91.14                    | 0.911  |
|                | 2.0                        | 0.346                                      | 93.03                    | 0.930  |
| 318            | Blank                      | 5.330                                      | –                        | –      |
|                | 0.5                        | 2.088                                      | 60.82                    | 0.608  |
|                | 1.0                        | 1.371                                      | 74.27                    | 0.742  |
|                | 1.5                        | 1.183                                      | 77.80                    | 0.778  |
|                | 2.0                        | 1.031                                      | 80.66                    | 0.806  |
| 328            | Blank                      | 6.222                                      | –                        | –      |
|                | 0.5                        | 3.703                                      | 40.48                    | 0.404  |
|                | 1.0                        | 2.584                                      | 58.46                    | 0.584  |
|                | 1.5                        | 1.966                                      | 68.39                    | 0.683  |
|                | 2.0                        | 1.382                                      | 77.78                    | 0.777  |

[Figure 1. Plot of weight loss against concentration for mild steel in 0.5 M HCl at different temperatures.]
2.2.1. Adsorption and thermodynamic consideration

By using the weight loss measurements the surface coverage ($\theta$) of different concentrations of the inhibitor in acidic solution has been evaluated and attempts have been made to fit these values to different adsorption isotherms such as Langmuir, Frumkin, Freundlich, and Flory–huggins \(^{15}\). The Langmuir adsorption isotherm fits the data well in the 0.5 M HCl acid solution. Langmuir adsorption isotherm is given by \(^{16, 17}\)

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C,$$

where $K_{ads}$ is adsorptive equilibrium constant, $\theta$ is the surface coverage, and $C$ is the equilibrium inhibitor concentration. The relation between the surface coverage ($C/\theta$) by the adsorbed molecule and concentration of the inhibitor ($C$) is shown in Figure 2. Linear plots indicate that the experimental results relating the adsorption of algal extract on mild steel can be approached by the Langmuir adsorption isotherm. $K_{ads}$ values were calculated from the intercepts of the straight lines $C/\theta$ versus $C$ axis, and are given in Table 2. The most important thermodynamic adsorption parameter, standard free energy of adsorption ($\Delta G_{ads}$), is related to the adsorption constant, $K$, with the following equation \(^{7}\):

$$\Delta G_{ads} = -RT \ln(55.5K_{ads}),$$

where $K$ is the binding constant, $R$ is the gas constant, and $T$ is the temperature. The negative values of $\Delta G_{ads}$ indicate the stable interactions between the inhibitor molecules and the mild steel surface \(^{3, 18}\). The adsorption of inhibitor molecules on the mild steel surface from 0.5 M HCl solution takes place through both physical, which is the first stage of adsorption, and chemical processes. However, the adsorption of inhibitor molecules on metal surfaces cannot be considered as a purely chemical or physical phenomenon \(^{19}\).

In the studied temperature (298–328 K) values of $K_{ads}$ and $G_{ads}$ are given in Table 2.

2.2. Effect of temperatures

In order to study the effect of temperature on the inhibition activity of algal extract on mild steel, measurements of weight loss were carried out at the different temperatures (298–328 K), in the absence and in the presence of inhibitor at different concentrations. An alternative formulation of Arrhenius equation is \(^{20}\):

$$\log \rho = \log A - \left( \frac{E_a}{2.303R} \right) \frac{1}{T},$$

where $\rho$ is the corrosion rate, $R$ the gas constant, $E_a$ the apparent activation energy, $T$ the absolute temperature, and $A$ the constant. Activation energy ($E_a$) values of mild steel in 0.5 M HCl at different temperatures in the absence and presence of different concentrations of algal extract were calculated from the slope of log $\rho$ versus 1000/$T$ (K) plots. Corresponding values of activation energy ($E_a$) are summarized in Table 3. Arrhenius plots for the corrosion rate of mild steel in 0.5 M HCl are shown in Figure 3. Usually values of activation energy ($E_a$) in the studied inhibitor concentration (0.5–2.0 g L\(^{-1}\)) are higher than that for the uninhibited one, indicating a strong inhibitive action for the algal extract by increasing the energy barrier for the corrosion process \(^{18}\). The transition state equation was used to calculate the enthalpy, $\Delta H^*$ and entropy, $\Delta S^*$ of activation for the corrosion process \(^{18}\):

$$\log \left( \frac{\rho}{T} \right) = \left( \log \left( \frac{R}{Nh} \right) \right) + \left( \frac{\Delta S^*}{2.303} \right) - \left( \frac{\Delta H^*}{2.303RT} \right).$$

| $T$ (K) | $R^2$ | $K_{ads}$ (L mol\(^{-1}\)) | $\Delta G_{ads}$ (kJ mol\(^{-1}\)) |
|---------|-------|-----------------------------|-----------------------------|
| 298     | 0.999 | 6.66                        | −11.79                      |
| 308     | 0.997 | 2.05                        | −9.45                       |
| 318     | 0.997 | 3.39                        | −10.41                      |
| 328     | 0.999 | 2.75                        | −10.03                      |

Figure 2. Linear fitting of spirogyra extract to the Langmuir adsorption isotherm at different temperatures.

Table 2. Thermodynamic parameters for the adsorption of green algae spirogyra extract in 0.5 M HCl on the mild steel surface at different temperatures (298–318 K).
where $\Delta S^*$ is the entropy of activation, $\Delta H^*$ the enthalpy of activation, $h$ the plank constant, and $N$ the Avogadro’s number. The transition state plot of $\log \rho/T$ against $1000/T$ (K) is shown in Figure 4. Straight lines were obtained with an intercept of $\log \left( \frac{R}{Nh} \right) + \left( \frac{\Delta S^*}{2.303} \right)$, and a slope of $-\frac{\Delta H^*}{2.303R}$ from which the values of enthalpy of activation ($\Delta H^*$) and entropy of activation ($\Delta S^*$) were calculated and values are shown in Table 3. The positive sign of enthalpies $\Delta H^*$ reveals the endothermic nature of dissolution of mild steel, indicating that dissolution of mild steel is difficult (20). The shift towards the positive values of the entropy of activation ($\Delta S^*$) in the presence of the inhibitor suggests that the activated complex in the rate-determining step represents dissociation rather than association (21), revealing that a decrease in disorder takes place on going from reactant to the activated complex (18).

### 2.3. FT-IR analysis

FT-IR spectrum analysis is a powerful tool that can be used to identify the type of bonding predominantly functional groups present in the inhibitor. The spectra of algal extract and scratched samples are shown in Figure 5. In Figure 5(a) the broad absorption band observed at 3448.97 cm$^{-1}$ is associated with hydroxyl or/and N–H groups. The band at 1636.97 cm$^{-1}$ indicates the presence of C=O groups (4, 22). The band around

![Figure 3](image-url)  
**Figure 3.** Arrhenius plot for mild steel corrosion rates in 0.5 M HCl in the absence and presence of different concentrations of inhibitor at temperatures 298–328 K.

![Figure 4](image-url)  
**Figure 4.** Transition state plot for mild steel corrosion rates in 0.5 M HCl in the absence and presence of different concentrations of inhibitor at different temperatures (298–328 K).

![Figure 5](image-url)  
**Figure 5.** FT-IR spectra of (a) spirogyra extract and (b) scrapped samples.

| Conc. (g L$^{-1}$) | A (mg cm$^{-2}$ h$^{-1}$) | Ea (kJ mol$^{-1}$) | $\Delta H^*$ (kJ mol$^{-1}$) | $\Delta S^*$ (kJ mol$^{-1}$) |
|------------------|--------------------------|-------------------|-----------------------------|-----------------------------|
| Blank            | 10.96                    | 38.50             | 18.78                       | $-21.13$                    |
| 0.5              | $7.64 \times 10^4$        | 84.03             | 46.18                       | $-12.36$                    |
| 1.0              | $8.15 \times 10^4$        | 85.09             | 46.68                       | $-12.29$                    |
| 1.5              | $19.0 \times 10^4$        | 83.16             | 49.39                       | $-11.44$                    |
| 2.0              | $24.49 \times 10^4$       | 80.99             | 56.67                       | $-08.89$                    |

Table 3. Activation parameters $E_a$, $\Delta H^*$ and $\Delta S^*$ of the dissolution of mild steel in 0.5M HCl in the absence and presence of different concentrations of green algae spirogyra extract.
at 1250 cm$^{-1}$ can be assigned to the stretching mode of the C–N group. The FT-IR spectrum of the scratched samples is shown in Figure 5(b). Comparison of Figure 5(a) and 5(b) shows that significant change was observed in the FT-IR spectrum of the scratched sample in comparison to that of the inhibitor. The shift in the absorption bands of the inhibitor on the mild steel surface strongly supports the interaction between the phytochemical compounds of the inhibitor and mild steel surface (6).

2.4. SEM energy-dispersive X-ray spectroscopy analysis

SEM microphotographs and the corresponding energy-dispersive X-ray spectroscopy (EDS) spectra of the surface of the mild steel coupons after in 0.5 M HCl solution in the absence and presence of algal extract are revealed in Figures 6 and 7. The surface morphology of mild steel surfaces immersed into 0.5 M HCl solutions in the absence and presence of optimum concentration of algal extract after 24 h of immersion were examined using SEM. The microphotographs of mild steel surface are given in Figure 6. Figure 6(a) represents the polished metal sample, the whole surface is a plane. Figure 6(b) shows the corroded sample exposed for 24 h in 0.5 M HCl solution, which corresponds to the maximum corrosion rate on mild steel surface. Figure 6(c) represents the SEM image of the surface of mild steel specimen immersed for 24 h in the 0.5 M HCl solution containing optimum concentration of the inhibitor (2 g L$^{-1}$). Surface analysis of Figure 6(c) proved a significant improvement on surface morphology of mild steel coupons in the presence of algal extract (8).

EDS spectra were recorded to determine the percentage of oxygen and chlorine in mild steel in the absence and presence of the inhibitor; corresponding results are shown in Table 4. EDS analysis on the blank solution sample (Figure 7(b)) surface shows a high amount of oxygen (27.81%), while less amount oxygen (8.90%) was detected on the sample immersed in the solution with algae extract (1, 4, 7). The mild steel surface corroded in 0.5 M HCl solution clearly shows a high chloride concentration (6.04%) compared to the surface in the presence of algae extract (0.12%). These values confirm the formation of a protective film by algal extract on the mild steel surface in acid solution.

2.4.1. XRD analysis

XRD was used to study the film formation on the mild steel in the absence and presence of inhibitor solutions (23). XRD patterns (Figure 8) of the scratched sample of mild steel in 0.5 M HCl and inhibitor adsorbed on the mild steel surface were recorded to confirm the formation of protective film. Mild steel specimens were scratched in both the cases and the resultant scratched samples were used for XRD spectral analysis. The mild steel specimen had undergone corrosion, leading to the formation of magnetite peaks in the 0.5 M HCl solution.
Figure 7. EDS spectrum of (a) mild steel, (b) mild steel in 0.5 M HCl, and (c) mild steel in the presence of inhibitor.

Table 4. EDS analysis results of mild steel specimens in 0.5 M HCl in the absence and presence of algae extract.

| Medium          | Fe  | O   | C   | S   | Cl  | P   | Cr  | Ni  | Mn  | Zn  |
|-----------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Mild steel (MS) | 97.04 | 1.99 | 0.23 | 0.01 | —   | 0.02 | 0.03 | 0.03 | —   | 0.36 |
| MS in 0.5 M HCl | 59.07 | 27.81 | 6.19 | —   | 6.04 | —   | —   | —   | —   | —   |
| MS in inhibitor | 79.09 | 8.90 | 11.5 | 0.08 | 0.12 | 0.04 | 0.02 | —   | 0.15 | —   |
solution due to iron oxides (Fe$_3$O$_4$ and FeOOH) appearing at $2\theta = 28.33^\circ$, 35.20$^\circ$, and 36.19$^\circ$ (Figure 8(a)) (21). The XRD spectra of the mild steel surface immersed in the 0.5 M HCl acid solution containing optimum concentration of the inhibitor extract are shown in Figure 8(b). The intense iron peak appears at $2\theta = 44.69^\circ$. From Figure 8(b) it is observed that the peaks due to iron oxides are found to be absent.

2.5. Comparison of inhibition efficiency of spirogyra extract with inhibitors surveyed in literature

In literatures analysed in the present work related to experimental inhibitors, relevant information was found, whose data are summarized in Table 5. From Table 5 it is evident that several inhibitors (entries 1, 3, 4) showed higher inhibition efficiencies than spirogyra (entry 8). Moreover, spirogyra extract showed higher inhibition efficiency in comparison to the inhibitors (entries 2, 5, 6) at relatively same temperatures. From Table 5 it has been observed that a lower concentration of spirogyra extract was used (2.0 g L$^{-1}$) than Artemisia pallens (40 g L$^{-1}$), whereas higher concentration of spirogyra is used than the inhibitors of entries 2, 3, 4 and 7. In conclusion, spirogyra possesses relatively good corrosion inhibition for mild steel in acidic solution.

3. Experimental

3.1. Materials preparation

The chemical composition wt.% of mild steel used for all experiments was as follows:

| C    | O    | Si   | P    | S    | Cr   | Ni   | Fe   |
|------|------|------|------|------|------|------|------|
| 0.23 | 1.99 | 0.58 | 0.02 | 0.01 | 0.03 | 0.03 | Balance |

Mild steel coupons were cut into 5 × 2 × 0.1 cm sizes from a steel sheet and polished consecutively using different grades of emery papers. Next, it were degreased with acetone and washed with double distilled water, then dried before and after the experiment. About 0.5 M HCl solutions were prepared using AR grade HCl (Merck).

3.2. Inhibitor preparation

Green algae Spirogyra was collected from the pond located near the place of the National Institute of Technology, Raipur, India. It is an unbranched filamentous green algae with coiled chloroplasts. For corrosion studies, the algal biomass was washed in running tap water for 30 minutes and then exhaustively washed with double distilled water, and ultimately dried in a hot air oven then crushed. Powdered green algae (400 g) were extracted in boiled water for 3 h. Then the mixture was filtered off and concentrated until the water from the filtered solution evaporates. This solid extract was used to prepare the required concentrations (0.5–2.0 g L$^{-1}$) of the algal solution and study the corrosion inhibition properties (7).

3.3. Weight loss measurements

The weight loss measurement is probably the most extensively used method for mild steel corrosion inhibition assessment. Weight loss measurements were conducted under total immersion of mild steel specimens using 100 mL capacity beakers at 298–328 K maintained in a thermostated water bath. Specimens of mild steel were weighed and dipped in the beaker. Experiments were performed at different concentrations of green algae Spirogyra extract for 24 h of immersion time. Specimens in uninhibited and inhibited solutions were measured using metteler Toledo AL204 electronic...
The corrosion rate ($\rho$) in mg cm$^{-2}$ h$^{-1}$ was calculated from the equation given below (26):

$$\rho = \frac{\Delta W}{At},$$  

(1)

where $\Delta W$ is the weight loss in mg, $A$ the total area of metal specimen in cm$^2$, and $t$ the immersion time (24 h). Inhibition Efficiency ($\%I$) was calculated by using the equation given below (26):

$$\%I = \left( \frac{\rho_1 - \rho_2}{\rho_1} \right) \times 100,$$  

(2)

where $\rho_1$ and $\rho_2$ are the corrosion rates of the mild steel specimens in the absence and presence of the inhibitor, respectively. The degree of surface coverage ($\theta$) was calculated as follows:

$$\theta = \frac{\%I}{100}.$$  

(3)

### 3.4. FT-IR and XRD spectral studies

A mild steel specimen was immersed in a 0.5 M HCl acid solution containing optimum concentration of inhibitor for 24 h. The mild steel specimen was removed and dried, then the surface film of the dried mild steel specimen was scratched and the resultant scraped powder sample was used for spectroscopic study. FT-IR spectra were obtained using the Thermo Nicolet, AVATAR-370-FTIR (USA), over a range of 500–4000 cm$^{-1}$ with a resolution of 4.000 cm$^{-1}$. XRD spectrum analysis was recorded by using PANalytical 3 kW $\alpha$-pert Powder – Multifunctional, the Netherlands.

### 3.5. SEM–EDS analysis

The surface morphologies of the uninhibited and inhibited mild steel in 0.5 M hydrochloric solution were investigated by SEM. For SEM analysis mild steel specimens were immersed in 0.5 M HCl solution in the absence and presence of inhibitor for 24 h. EDS analysis determined the composition (wt. %) of chlorine concentration in the uninhibited and inhibited 0.5 M HCl solution for a 3 h period. The surface morphology and EDS of mild steel specimens was examined by using a scanning electron microscope ZEISS EVO SEM 18 model equipped with an INCA 250 EDS with X-MAX 20 mm Detector system, oxford.

### 4. Conclusions

The results clearly indicate that the algal extract is a good green corrosion inhibitor of mild steel in 0.5 M HCl solution. The inhibition efficiency ($\%I$) was found to increase with increase of the inhibitor concentrations due to the adsorption of the *spirogyra* extract components on the metal surface and found to decrease as reaction temperature increased (298–328 K). The corrosion inhibition was supposed to occur by adsorption of the algal extract components on the mild steel surface, which depends on the functional groups present in the extract. The adsorption of the algal extract on the mild steel surface obeys the Langmuir adsorption isotherm. An inhibition mechanism is inferred from the temperature dependence of the inhibition efficiency and was further supported by the values of activation energy and thermodynamic parameters obtained from the experimental data. Surface analysis (FT-IR, SEM, and XRD) shows the improvements on the metal surface morphology after being added with the algal extract.

### Disclosure statement

No potential conflict of interest was reported by the authors.

### Funding

The authors are thankful to the Ministry of Human Resource Development (MHRD) and Govt. of India for financial support in the form of JRF.

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