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# Influence of *Piper nigurm*. L on Corrosion Inhibition of Mild Steel in Citric Acid Medium

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The inhibition efficiency of an aqueous extract of *Piper nigurm*. L in controlling corrosion of mild steel at pH 12 has been evaluated by weight loss method in the absence and presence of inhibitor in citric acid medium at different concentration. The result showed that the corrosion inhibition efficiency of these compounds was found to vary with the different time interval at room temperature. It was also found that the corrosion inhibition behaviour of *Piper nigurm*. L is greater in 2N citric acid than 1N citric acid medium. So *Piper nigurm*. L can be used as a good inhibitor for preventing mild steel material. The surface analysis study confirms the corrosion of the mild steel and its inhibition effect by the inhibitor *Piper nigrum* L.

**Key Words: Mild steel, Corrosion inhibitors, Weight loss method, *Piper nigurm* L. Eco-friendly inhibitor.**

## INTRODUCTION

Large amount of sulphuric acid is used in the chemical industry for the removal of undesired scales and rust. The addition of corrosion inhibitors effectively secures the metal against an acid attack. Many studies using organic inhibitors have been reported. Most of the inhibitors are organic compounds with N, S and O hetero atoms have high electron density making them the reaction centers. These compounds adsorbed on the metallic surface and block the active corrosion sites and most of them are highly toxic to both human beings and environment. Hence use of the natural products as eco friendly and harmless corrosion inhibitors, has become popular[1-6].

Pure synthetic chemicals are costly, some of them are not easily biodegradable and their disposal creates pollution problem. Plant extracts are environment friendly, biodegradable, non-toxic, easily available and of potentially low cost. Most of the naturally occurring substances are safe and can be extracted by simple procedure. Recent literature is full of researches which test different extracts for corrosion inhibition applications. The examples are numerous such as fenugreek[7], olive[8], henna[9,10], black pepper[11], jojaba[12], occimum viridis[13], onion, garlic[14] *etc*. Many of these naturally occurring substances proved their ability to act as corrosion inhibitors for the corrosion of different metals and alloys in different aggressive media.

The aim of the present study is to investigate the corrosion inhibition effect of *Piper nigrum*. L (a pepper family) as cheap and environment friendly corrosion inhibitor for mild steel in 1N citric acid and 2N citric acid at different time intervals by weight loss method.

## EXPERIMENTAL

**Weight loss measurement:** According to ASTM method as reported already[15], cold rolled mild steel strips were cut into pieces of 5 cm × 1 cm having the following composition (in percentage) of Fe = 99.437, Ni = 0.019, Mo = 0.016, Cr = 0.050, S = 0.025, P = 0.041, Si = 0.029, Mn = 0.335 and C = 0.048. They were pickled in pickling solution (5 % $H_2SO_4$) for 3 min and washed with distilled water followed by polished with various grades of emery papers and degreased using trichloroethylene. The weight of specimen were noted and then immersed in test solution containing various concentrations of inhibitors at room temperature. After 1 and 2 h in citric acid, the specimens were removed from test solutions and, dried and finally weighed in an electronic balance for an accurate weight. The differences in weights were noted and the corrosion rates were calculated.

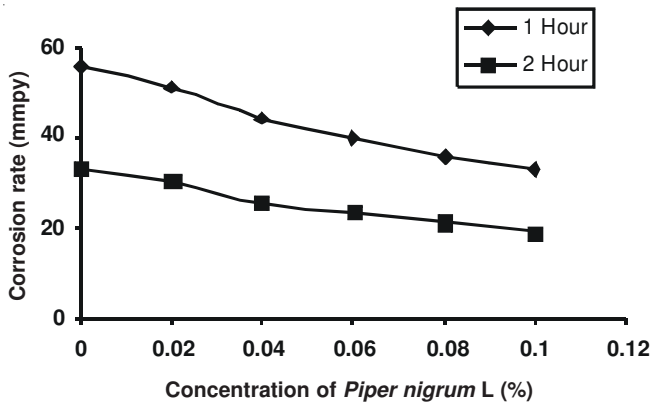
**Inhibitor material:** 5 % Stock solution of the inhibitor material (*Piper nigrum* L extract) was prepared by refluxing 10 g of dry *Piper nigrum* L seed powder with 200 mL citric acid for 2 h. The refluxed solution is allowed to stand over night and then filter through an ordinary filter paper. The filtrate was made upto 200 mL using distilled water. From this solution, different concentrations of inhibitor solution ranging from 0.02-1 % were diluted. All the solutions were prepared using NICE brand AR grade chemicals in double distilled water and

bubbling purified by nitrogen gas for 0.5 h to carry out de-aeration of the electrolytes. Citric acid solution (concentration 1N and 2N) was prepared by double distilled water.

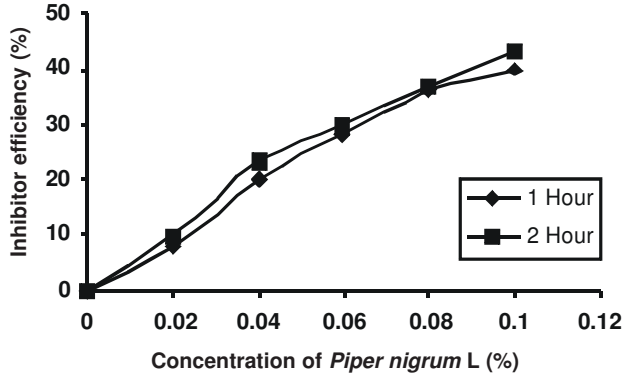
## RESULTS AND DISCUSSION

The corrosion behaviour of mild steel in Citric acid with *Piper nigrum* L was given in Fig. 1, which was studied by weight loss method at different concentration at room temperature. From the graph, it was observed that the weight loss of mild steel in the acid decreases with increasing concentration of additives, which suggesting that the additives are corrosion inhibitor for mild steel in 1 N citric acid and 2 N citric acid from the data of weight lost method, the corrosion rate was calculated using the equation:

$$CR = \frac{(87.6 \times W)}{(D \times A \times T)}$$




(a)

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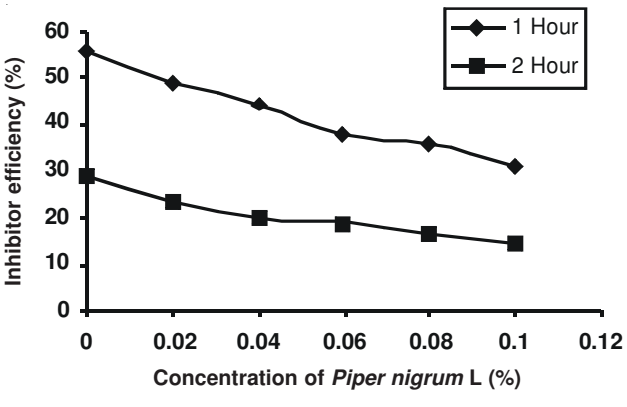


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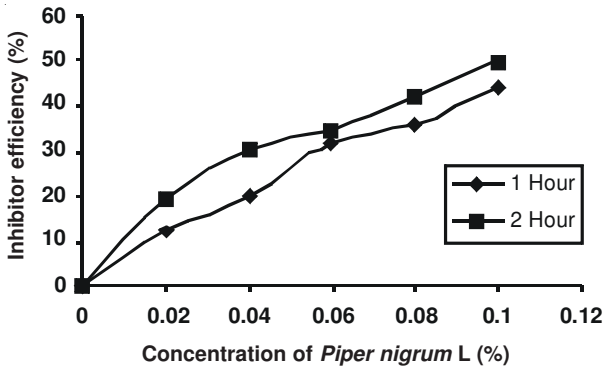



(d)

Fig. 1. (a) Comparison of corrosion rate (CR) with concentration of *Piper nigrum* L (%) in 1N citric acid solution at 1 and 2 h, (b) Comparison of inhibition efficiency (IE) with concentration *Piper nigrum* L (%) in 1N citric acid solution at 1 and 2 h, (c) Comparison of corrosion rate (CR) with concentration *Piper nigrum* L (%) in 2N citric acid solution at 1 and 2 h, (d) Comparison of inhibition efficiency (IE) with concentration of *Piper nigrum* L (%) in 2N citric acid solution at 1 and 2 h

where W, D, A and T are weight loss (mg), density of mild steel (7.86 g/cc), area of the specimen in cm square and exposure time in hours, respectively.

Similarly, inhibition efficiency (IE) was calculated using the equation,

$$IE\ (\%) = \left[\frac{[(W_o - W_i)}{W_o}\right] \times 100$$

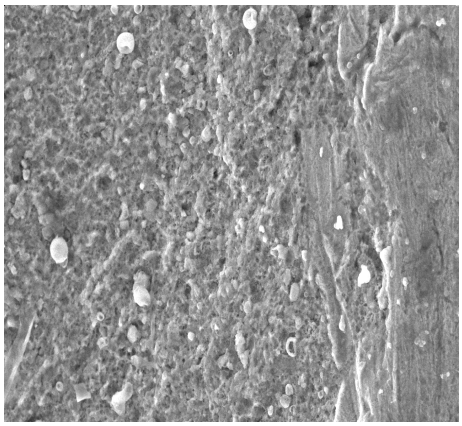
where $W_o$ and $W_i$ are the values of the weight loss (g) of mild steel in the absence and presence of inhibitor, respectively. The values of corrosion rate and inhibition efficiency in absence and presence of inhibitor in difference concentration of 1N citric acid and 2N citric acid are used for 1 and 2 h were given in Table-1.

From Table-1, it was clear that the corrosion rate was decreased with increasing concentration of inhibitor and inhibition efficiency increased with increasing the concentration of the inhibitor. In addition, the maximum corrosion inhibition efficiency of *Piper nigrum* L was 40.00 and 43.33 % in 1N citric acid at 0.10 % solution at 1 and 2 h, respectively. Where as in 2N citric acid the inhibitor efficiency was 44 and 50 % at 0.10 % solution at 1 and 2 h, respectively at room temperature. It was also concluded that *Piper nigrum* L was best inhibitor in mild steel. So that *Piper nigrum* L acts as a good inhibitor in citric acid medium. When the concentration increases the inhibitor efficiency is also increases.

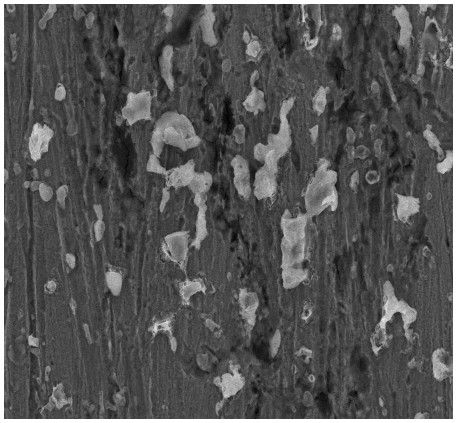
**Surface analysis:** The polished specimen (mild steel) and the test specimens which are immersed in the blank (1N citric acid and 2N citric acid) and in the inhibitor *Piper nigrum* L for 48 h, then the specimens were observed under scanning electron microscope are shown in the plate 1 and 2. Plate 1 (Fig. 2a-b) shows polished specimen which is kept in the blank solution of (1N citric acid and 2N citric acid), which is associated with polishing scratches. Plate 2 (Fig. 2c-d) shows specimen which is kept in the (0.10 % concentration of inhibitor solution with (1N citric acid and 2N citric acid) depends upon the concentration of the inhibitor solution suggesting that the presence of adsorbed layer of the inhibitor on mild steel surface which impedes corrosion rate of metal appreciably.

TABLE-1
CORROSION INHIBITION BEHAVIOUR OF MILD STEEL IN 1 N CITRIC ACID AND 2 N CITRIC ACID SOLUTION IN ABSENCE AND PRESENCE OF *Piper nigrum* L BY WEIGHT LOSS MEASUREMENT AT 1 h

| Corrosion inhibitors (*Piper nigrum* L) | Conc. of inhibitor (%) | 1 N Citric acid (1 h) | 1 N Citric acid (2 h) | 2 N Citric acid (1 h) | 2 N Citric acid (2 h) |
|---|---|---|---|---|---|
| Corrosion rate (mmpy) | Blank | 55.725 | 33.435 | 55.725 | 28.977 |
| | 0.02 | 51.267 | 30.091 | 49.038 | 23.404 |
| | 0.04 | 44.580 | 25.633 | 44.580 | 20.061 |
| | 0.06 | 40.122 | 23.404 | 37.893 | 18.946 |
| | 0.08 | 35.664 | 21.175 | 35.664 | 16.717 |
| | 0.10 | 33.435 | 18.946 | 31.206 | 14.488 |
| Inhibitor efficiency (%) | Blank | – | – | – | – |
| | 0.02 | 8.000 | 10.00 | 12.00 | 19.23 |
| | 0.04 | 20.00 | 23.33 | 20.00 | 30.76 |
| | 0.06 | 28.00 | 30.00 | 32.00 | 34.61 |
| | 0.08 | 36.00 | 36.66 | 36.00 | 42.30 |
| | 0.10 | 40.00 | 43.33 | 44.00 | 50.00 |



(a)



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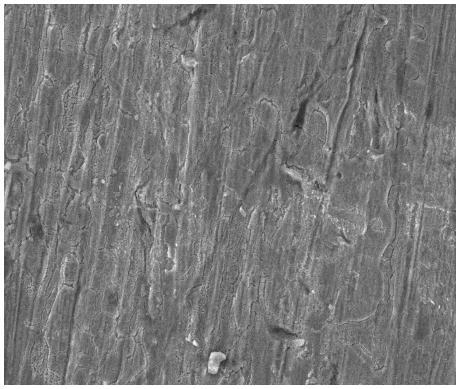

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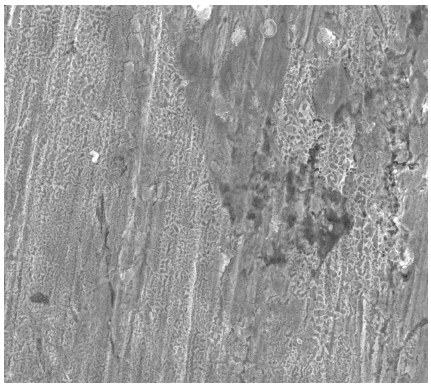

(d)

Fig. 2. Mild steel samples immersed in blank solution and inhibitor solution. (a) 1 N citric acid (blank), (b) 2 N citric acid (blank), (c) 1 N citric acid (with inhibitor), (d) 2 N citric acid (with inhibitor)

## Conclusion

The *Piper nigrum* L showed good performance as corrosion inhibitor in citric acid solution. The inhibition efficiency increased with increase in concentration of inhibitors for 0.02-0.10 % at 1 and 2 h at room temperature. The maximum corrosion inhibition efficiency of *Piper nigrum* L was 40.00 and 43.33 % in 1N citric acid at 0.10 % solution at 1 and 2 h, respectively. Where as in 2N citric acid the inhibitor efficiency was 44.00 and 50.00 % at 0.10 % solution at 1 and 2 h immersion time. From the comparative studies, it was concluded that the inhibitor efficiency is better in 2N citric acid than 1N citric acid. Surface analysis study confirms the corrosion of mild steel and its inhibition effect by the inhibitor *Piper nigrum* L.

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