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PHYTOCHEMICAL AND CORROSION INHIBITION OF *CITRUS AURANTIUM* L. LEAF EXTRACT IN AQUEOUS MEDIUM

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ABSTRACT

Objective: To study the morphology, biochemistry and corrosion inhibition of *Citrus aurantium* L. leaf extract. Methods: Morphological studies on *Citrus aurantium* L. leaf extract were carried out by using weight loss method, pH, UV–Vis, and FT-IR. To prepare the extract, the shade-dried leaf of *Citrus aurantium* L. were soaked in ethanol. For antibacterial studies weight loss method was followed by using various biocides. Results: Detailed phytochemical, and corrosion inhibition studies on a medicinal fern *Citrus aurantium* L. showed its inhibition efficiency. The presence or absence of the *Citrus aurantium* L. leaf extract was the key factor for corrosion inhibition of this species. FT-IR and UV showed the presence of various kinds of terpenoids, alkaloids, tannins, saponins and flavonoids in it. Conclusions: The present study shows that *Citrus aurantium* L. leaf extract have several bioactive compounds, can be used as corrosion inhibitor and such rare morphovariant should be conserved in nature. The next step is to isolate the pure compounds and to screen the bioactivity.

INTRODUCTION

Many pharmaceutical innovations are developed from a starting point of knowledge derived from the biological activities of natural organisms. In the case of the random screening of natural product, Aylward et al. [1] argued that the sheer scale of the resource, in the order of 10 to 100 million species, and the continuing evolution of new screens and new disease targets imply that biodiversity will never be fully explored for its pharmaceutical potential. The protection of metal against corrosion is a major industrial problem. The use of inhibitor is one of the best options of protecting metals against corrosion. Several inhibitors in use is either synthesized from cheap raw materials or chosen from compounds having hetero atoms in their aromatic or long chain carbon system. These organic compounds can adsorb on the metal surface, block the active sites and thereby reduce the corrosion rate considerably [2]. Most of the synthetic organic compound shows good anti-corrosive activity, which are highly toxic to cause severe hazards to both human beings and the environment during its applications [3]. The safety and environmental issues of corrosion inhibitors arisen in industries have always been a global concern. The recent trend is to save human being and environment by using eco-friendly inhibitors. Some investigator [4,5] studied the plant extracts and the derived organic species become more important as an environmentally benign, readily available, renewable and acceptable source for a wide range of inhibitors. Several efforts have been made using corrosion preventive practices and the use of green corrosion inhibitors [6]. The plant extract are rich sources of molecules which have appreciably high inhibition efficiency and hence termed as “Green Inhibitors” [7]. These inhibitors are biodegradable and do not contain heavy metals or other toxic compounds [8]. The successful use of naturally occurring substances to inhibit the corrosion of metals in acid and alkaline environment have been presented by some research groups running through references [9-14]. In our present study, we have chosen eco-friendly bio-inhibitor, a green approach to prevent environmental pollution by harmful organic chemicals. The influence of *Citrus aurantium* extract in aqueous medium on carbon using mass loss measurements have been studied. The characterization of alcoholic crystals of inhibitor and the corrosion product on carbon steel in the presence of inhibitor is also reported by UV and IR studies.

EXPERIMENTAL

Preparation of extract

The leaves of *Citrus aurantium* were taken and shade dried and ground well into powder. From this 50 g of sample was refluxed using soxlet in 500 ml of ethanol, the extract and solvent was separated using distillation process. The pure extract was made up to 100 mL using double distilled water.

Preparation of specimens

Carbon steel specimens (0.0267% S, 0.06% P, 0.4% Mn, 0.1% C and the rest iron) of dimensions 1.0 x 4.0 x 0.2 cm were polished to a mirror finish and degreased with trichloroethylene.

Weight loss method

Carbon steel specimens in triplicate were immersed in 100 ml of the solutions containing various concentrations of the inhibitor for one day. The weight of the specimens before and after immersion were determined using Shimadzu balance, model AY 62. The corrosion products were cleansed with Clarke's solution [15]. The inhibition efficiency (IE) was then calculated using the equation;

$$IE = 100\,[1 - (W_2/W_1)]\ \% \qquad (1)$$

where W_1 is the corrosion rate in the absence of the inhibitor, W_2 is the corrosion rate in the presence of the inhibitor.

Surface examination

The carbon steel specimens were immersed in various test solutions for a period of one day, taken out and dried. The nature of the film formed on the surface of metal specimens was analyzed by Perkin-Elmer 1600 FTIR spectroscopy and fluorescence spectra was recorded with Hitachi F-4500 fluorescence spectrophotometer.

RESULTS AND DISCUSSION

Analysis of results from weight loss method

Table 1 shows the values of corrosion rates and inhibition efficiencies obtained from mass loss measurements of different concentrations of *Citrus aurantium* extract (CAE). 2 ml of the CAE offered 79.2% corrosion inhibition efficiency to carbon steel immersed in 100 ml solution. When the concentration of CAE was increased, the inhibition efficiency was decreased. This is due to the fact that when higher concentrations of *Citrus aurantium* are added the protective film (Fe^{2+}–

Citrus aurantium complex) formed on the metal surface goes into the solution and thus destroying the protective film. It may be considered that the protective film formed may go into transpassive state, where the film is broken [16].

Table 2 shows the values of corrosion rates and inhibition efficiencies obtained from mass loss measurements using *Citrus aurantium* extract (CAE) and of different concentrations of biocides (*Ocimum sanctum*). When the concentration of biocides was increased, the inhibition efficiency was also increased.

Table 3 shows the values of corrosion rates and inhibition efficiencies obtained from mass loss measurements of using *Citrus aurantium* extract (CAE) and of different pH value. When the pH was increased, the inhibition efficiency was decreased.

Table 1. Corrosion rates (CR) of carbon steel immersed in the presence and absence of inhibitors and the inhibition efficiencies (IE) obtained by weight loss method. Inhibitor: CAE; Period: 1 day.

***Citrus aurantium* (ml)**	**Corrosion rate (ppm)**	**Inhibition efficiency (%)**
0	38	-
1	15.2	60
2	7.9	79.2
3	9.6	74.7
4	10.9	73.4

Table 2. Corrosion rates (CR) of carbon steel immersed in the presence of biocides. Inhibitor: CAE; Period: 1 day.

S. No.	***Citrus aurantium* (ml)**	**Mn^{2+} (ppm)**	**Biocides (*Ocimum sanctum*) (ml)**	**Corrosion rate (ppm)**	**Inhibition efficiency (%)**
1	2	20	-	7.9	79.2
2	2	20	1	6	84.2
3	2	20	2	5	86.8
4	2	20	3	8	78.9
5	2	20	4	4	89.5
6	2	20	5	1	97.4

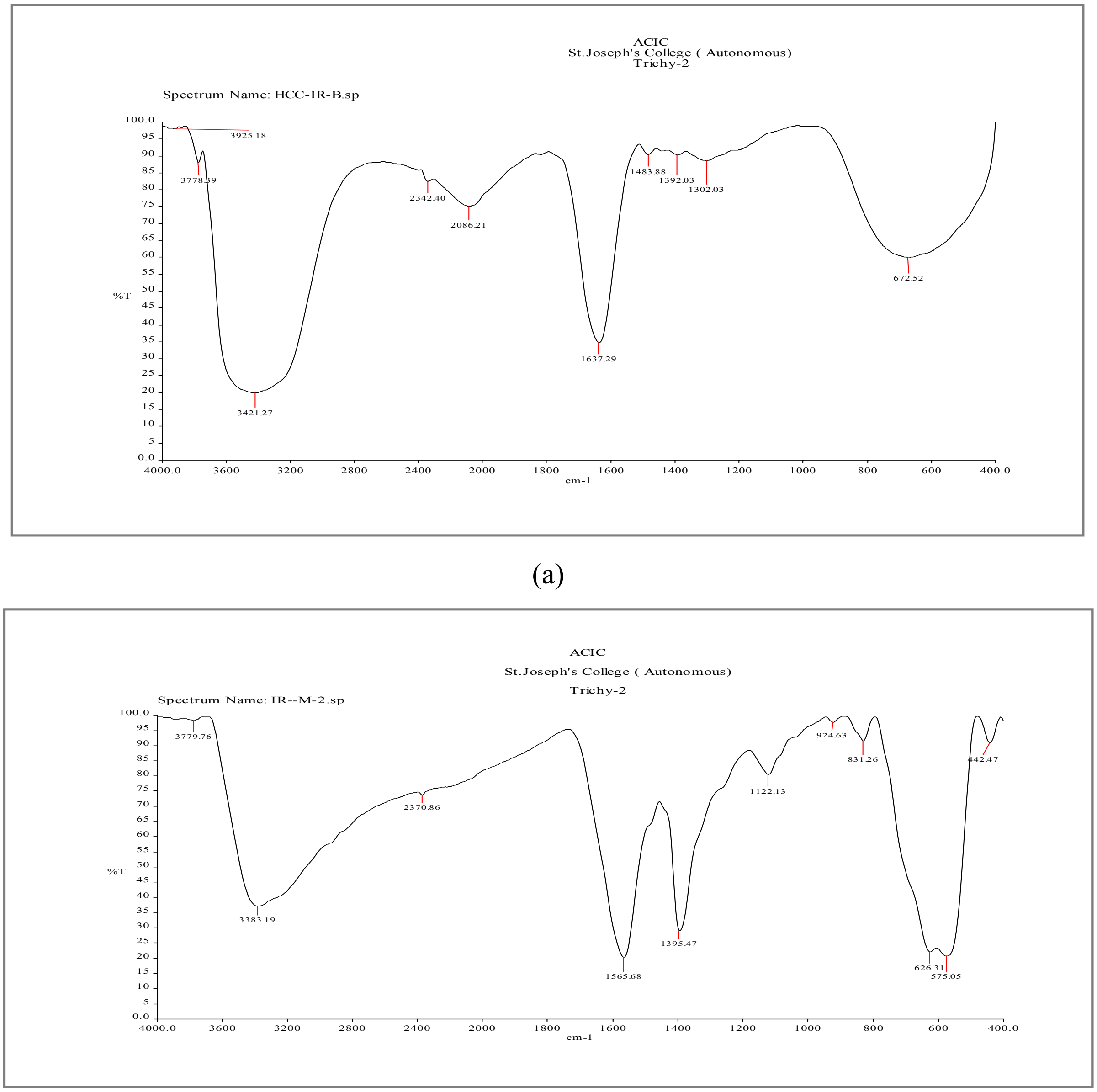
Table 3. Corrosion rates (CR) of carbon steel immersed with different pH. Inhibitor: CAE; Period: 1 day pH

pH	***Citrus aurantium* (ml)**	**Mn^{2+} (ppm)**	**Corrosion rate (ppm)**	**Inhibition efficiency (%)**
1	2	20	21.85	42.5
3	2	20	21.29	43.93
5	2	20	7.47	80.47
7	2	20	20.89	45.1
9	2	20	21.62	43.1

Fourier transfer infrared spectra (FTIR)

Citrus aurantium extract FTIR spectrum is shown in Figure 1(a), –C=C stretching frequency appeared at 2086.21 cm^{-1}. The aliphatic –CH stretching frequency appeared at 3421.27 cm^{-1}. The ring oxygen stretching frequency at 1302.03 cm^{-1}, $-OCH_3$ stretching frequency appeared at 1637 cm^{-1} (Figure 1(b)) [19,20]. The FTIR spectrum of the protective film formed on the surface of the metal after immersion in the aqueous solution containing 2 ml of *Citrus aurantium* is shown in Figure 1(b). It is found that the –C=C stretch has shifted from 1395.47 cm^{-1} to 2370.86 cm^{-1}. The aromatic –CH_2 stretching frequency appeared at 2370 cm^{-1} to 3383 cm^{-1}. The ring oxygen stretching frequency at 924.63 cm^{-1} to 1122.13 cm^{-1}. It is inferred that the oxygen atoms of ring oxygen and –OCH_3 group have coordinated with Fe^{2+} resulting in the formation of Fe^{2+}–CAE complex formed on the anodic sites of the metal surface.

Figure 1. FTIR spectra of (a) pure CAE (b) film formed on the metal surface after immersion in aqueous solution + CAE (2 ml)



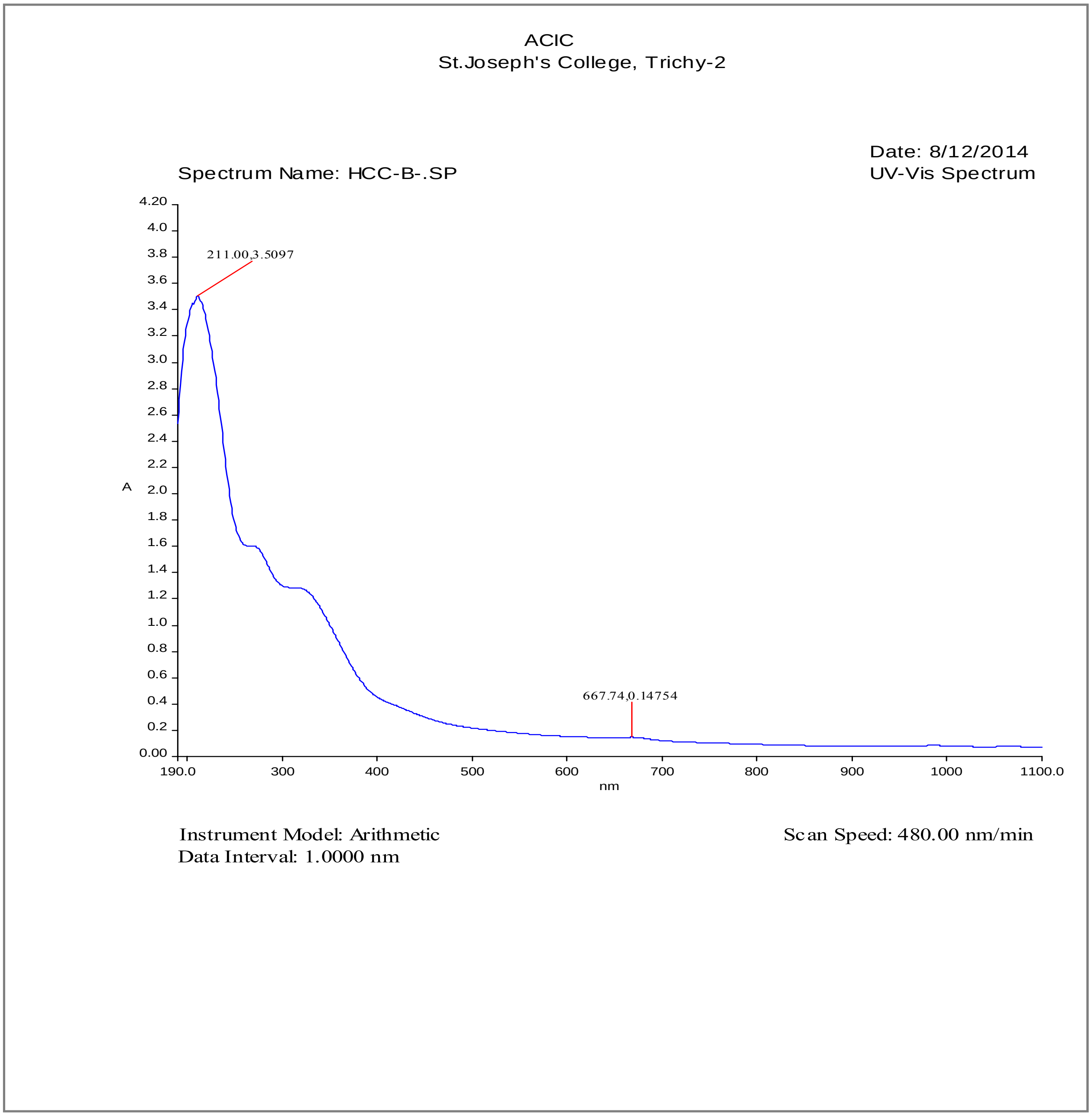
(a)

(b)

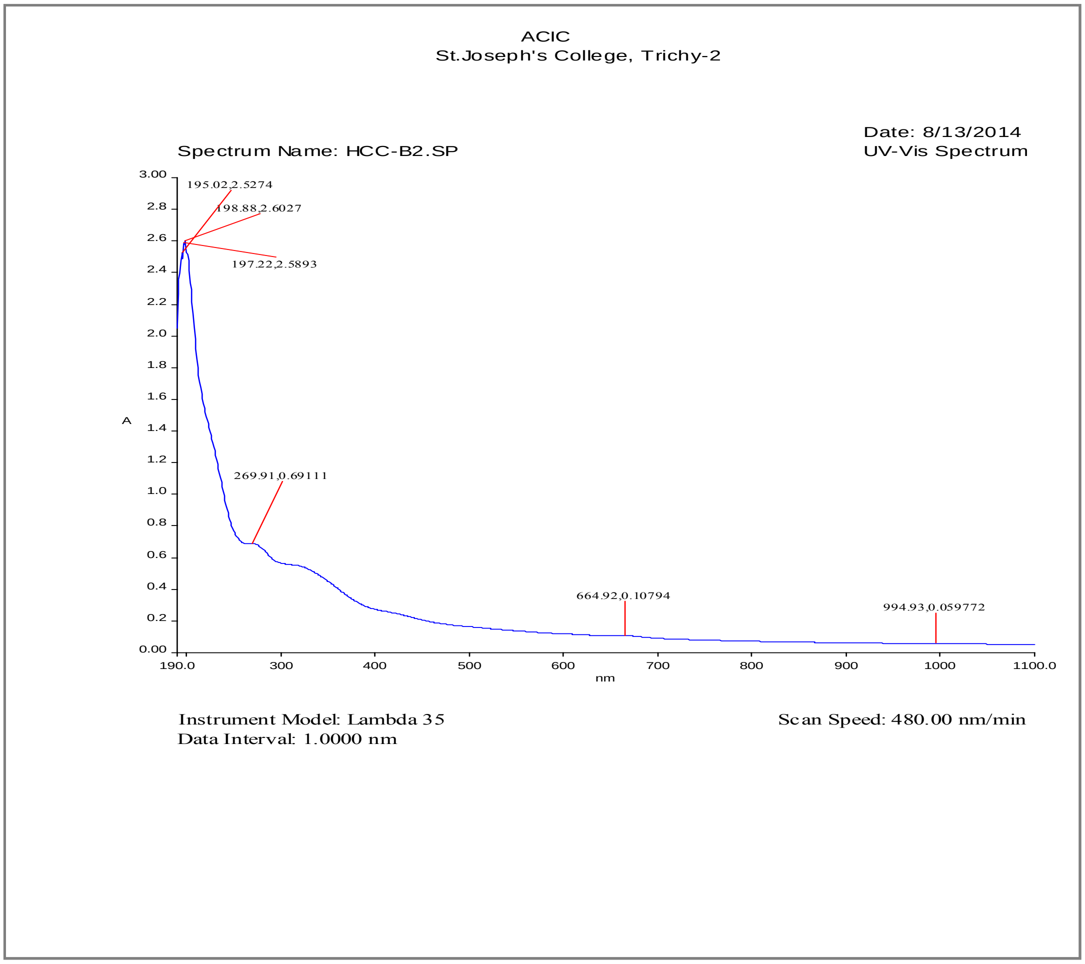
Fluorescence spectra

The UV–Vis adsorption spectrum of aqueous solution of *Citrus aurantium* is shown in Figure 2(a) and (b). Peak appears at 228 nm, 268 nm. When the Fe^{2+} is added to the aqueous solution of *Citrus aurantium*, peaks appear at 225 nm and 265 nm.

Figure 2. UV-spectra (a) aqueous solution of CAE (b) aqueous solution of CAE + Fe^{2+} + 8 ml *Citrus aurantium* extract.



(a)



(b)

CONCLUSIONS

1. Inhibition efficiency is high at 2 ml of *Citrus aurantium*, so it is found to best system.

2. Corrosion rate decreases with increase in biocide concentration.

3. An aqueous extract of (*Citrus aurantium*) has excellent is inhibition efficiency in controlling corrosion of carbon steel in aqueous solution.

4. The protective film consists of Fe^{2+}–*Citrus aurantium* complex.

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