



## Corrosion

Elixir Corrosion 62 (2013) 17908-17911

Elixir  
ISSN: 2229-712X

# Phytochemical investigation and study on corrosion inhibition properties of *Pongamia pinnata* (L.) pierre leaf extract

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### ARTICLE INFO

#### Article history:

Received: 30 July 2013;

Received in revised form:

28 August 2013;

Accepted: 17 September 2013;

#### Keywords

*Pongamia pinnata* (L.) Pierre;  
Mild steel,  
Corrosion inhibition,  
Mylius thermometric Method.

### ABSTRACT

Plant extracts are excessively used as corrosion inhibitors. Plant extracts contain a variety of organic compounds such as alkaloids, flavonoids, tannins, cellulose and polycyclic compounds. The compounds with hetero atoms-N, O, S, P coordinate with (corroding) metal atom or ion consequently forming a protective layer on the metal surface, that prevents corrosion. These serve as cheaper, readily available, renewable and environmentally benign alternatives to costly and hazardous corrosion inhibitors. This study reports screening of phytoconstituents and also reports the influence of the addition of *Pongamia pinnata* (L.) Pierre leaves extract on the corrosion inhibition of mild steel in 2.0 M H<sub>2</sub>SO<sub>4</sub> solution using the Mylius thermometric technique. Results of the study revealed that *Pongamia pinnata* (L.) Pierre leaves act as corrosion inhibitor for mild steel in the acidic medium. The addition of 0.1 M potassium chloride, potassium bromide and potassium iodide to the inhibitor enhanced the inhibition efficiency. The order of reactivity of halide ions is of the order KI > KBr > KCl. The inhibition efficiency increased 58 % to 78 % at a concentration of 0.1 M KI and 5.0 % of inhibitor concentration. The values of adsorption of *Pongamia pinnata* (L.) Pierre leaves extract onto the mild steel surface show that the process is spontaneous.

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### Introduction

*Pongamia pinnata* is a deciduous Indian Beech Tree, about 15–25 meters tall, belonging to the family of *Fabaceae*. It has a big top with many small flowers in white, pink or violet. *Pongamia pinnata* is a hardy tropical tree, resistant against heat and sunlight. Due to its big root system, it is also tolerant against drought. Naturally, it grows on sandy or rocky soils, including limestone, but in cultivation, it can be successfully grown in nearly all kinds of soil as well as salty soils. The bark is used to make twines or ropes, and the black gum has been used in the past to treat wounds caused by poisonous fish. The leaves, flowers, seeds and stem bark of *P. pinnata* are known to have karanjin, a furanoflavanoid, which is toxic. Phytochemical studies have showed the presence of flavonoid compounds such as furanoflavones, chromenoflavones, simple flavones, chromenoflavanones, chalcones,  $\beta$ -diketone,  $\beta$ -hydroxychalcone. Modern pharmacological experiments show that *Pongamia pinnata* has antinociceptive and antipyretic activities, antibacterial activity, antiviral properly, anti-inflammatory activity, Antine-oplastic activity, etc. they contain both saturated and unsaturated fatty acids such as stearic oleic and palmitic acids. Its root nodules promote nitrogen fixation, a symbiotic process by which gaseous nitrogen (N<sub>2</sub>) from the air is converted into NH<sub>4</sub><sup>+</sup> (a form of nitrogen available to the plant). Thus, it can be used for fertilization of soil that is poor in nutrients. Although the whole plant is toxic, the juice from the plant, as well as the oil, is antiseptic.<sup>1</sup>

The use of inhibitors for the control of corrosion of metals and alloys which are in contact with aggressive environment is an accepted practice. Large numbers of organic compounds

were studied and are being studied to investigate their corrosion inhibition potential. All these studies reveal that organic compounds especially those with N, S and O showed significant inhibition efficiency. But, unfortunately most of these compounds are not only expensive but also toxic to living beings. It is needless to point out the importance of cheap, safe inhibitors of corrosion. Plant extracts have become important as an environmentally acceptable, readily available and renewable source for wide range of inhibitors. They are the rich sources of ingredients which have very high inhibition efficiency. This article gives a vivid account of natural products which are used as corrosion inhibitors for various metal and alloys in aggressive media<sup>2</sup>.

### Experimental

#### Phytochemical Screening Test for the Extracts:

Fresh leaves of *Pongamia pinnata* were washed under running water, shade dried and ground into powder. The powdered leaves were extracted using 95% ethanol. 10g of the powdered leaves was soaked in 200 ml in ethanol for 4 days and filtered thereafter. The filtrate was subjected to evaporation using a rotary evaporator to remove ethanol. A small portion of the dry extract was subjected to the phytochemical test.

**Test for alkaloids:** Each plant sample (0.5 g) was dissolved in 5 ml dilute HCl in a steam bath and filtered. A few drops of Dragendoff's reagent is added. Orange red precipitate indicates the presence of alkaloids.

**Test for tannins:** About 0.5 g of the dried powdered samples was boiled in 20 ml of water in a test tube and then filtered. A few drops of 0.1% ferric chloride was added. A dark green solution indicates the presence of tannins.

**Test for steroids:** Exactly 2ml of acetic anhydride added to 0.5g of the dried powdered sample with 2ml of H<sub>2</sub>SO<sub>4</sub>. The colour changes from violet to blue or green indicated the presence of steroids.

**Test for terpenoids:** About 0.2g dried powdered sample was mixed with 2ml chloroform and 3 ml concentrated H<sub>2</sub>SO<sub>4</sub> was carefully added to form a layer. A reddish brown colouration at the interface formed indicated the presence of terpenoids.

**Test for reducing sugar:** Dried powdered sample was shaken with distilled water and filtered. The filtrate was boiled with Fehling's A and B, an orange and red precipitate indicates the presence of reducing sugar.

**Test for carbonyls:** 1ml of dried powdered sample was taken in a test tube and few drops of 2, 4 DNPH solutions was added and shaken. Instant appearance of yellow crystals indicates the presence of aldehyde.

**Test for flavonoids :** 0.5 g of the sample is shaken with water and 5 ml of dilute ammonia solution was added followed by addition of concentrated H<sub>2</sub>SO<sub>4</sub>. A yellow colouration observed in each extract indicated the presence of flavonoids. The yellow colouration disappeared on standing.

**Test for Saponin :** 2 ml of each of the Fehling's A and B solution was added to 3 ml of the extract. The mixture was boiled for 5 min. Brick red colour indicates the presence of saponin

Mild steel metal (the percentage elemental composition was found to be, C (0.048%), Mn (0.335%), Si (0.029%), P(0.041%), S (0.025%), Cr (0.050%), Mo (0.016%), Ni (0.019%) and Fe (99.437%) having a surface area of 5x1cm<sup>2</sup> were cut from a large sheet. The specimens were polished successively with emery sheets, degreased and dried. Sulphuric acid used for preparing solutions was AR grade<sup>3</sup>.

Fresh *Pongamia pinnata* (L.) Pierre leaves were shade dried, hand crushed, washed with distilled water and dried in an oven at 80 °C. The extract was prepared by refluxing 10 g of powdered dry leaves in 0.5 N sulphuric acid for 5 h and kept overnight. Then it was filtered and the volume of the filtrate was made up to 50 mL using the same acid and this was taken as stock solution. The concentrations of the inhibitor from 1.0 % to 5.0 % was prepared and used in the study. The specimens in triplicate were immersed in 2.0M acid solutions containing various concentrations of the inhibitor (*Pongamia pinnata* (L.) Pierre leaves extract) for one hours at 300K. The specimens were removed washed with water and dried. The mass of the specimens before and after immersion was determined using an electronic digital balance<sup>4</sup>. The concentrations of H<sub>2</sub>SO<sub>4</sub> (blank) used were in the range 0.5 M – 2.0 M and Synergistic effects was studied in the presence of 0.05 M halide additives namely potassium chloride, potassium bromide and potassium iodide. The corrosion rate for room temperature with various concentrations of inhibitor was obtained from the following formula,

$$\text{Corrosion Rate (mpy)} = \frac{436.095 \times 1000 \times W}{A \times T}$$

Where, W = Weight loss in grams, A = Area of specimen in cm<sup>2</sup> T = Exposure time in hours. The unit of the corrosion rate is in mills per year (mpy).

$$\text{IE \%} = \left[ \frac{\text{weight loss without inhibitor} - \text{weight loss with inhibitor}}{\text{weight loss without inhibitor}} \right] \times 100$$

The corrosion rate was calculated by measuring the amount of mild steel dissolved in the solution analytically.

$$\text{Surface coverage } (\Theta) = \frac{CR_{\text{Blank}} - CR_{\text{inh}}}{CR_{\text{Blank}}}$$

Mild steel specimens were completely immersed 2 M hydrochloric acid which is blank solution. The volume of the test solution was kept at 100 ml. The initial temperature in all experiments was kept at 303 K. The temperature was measured to ±0.05 °C on a calibrated thermometer (0-100 °C). This method allowed for the evaluation of the reaction number (RN). The RN is defined as

$$\text{Reaction number (k/min)} = \frac{T_m - T_i}{t}$$

T<sub>m</sub> =Maximum temperature attained by solution. T<sub>i</sub> =Initial temperature of solution. t = time required to attain maximum temperature. The inhibition efficiency (IE %) was evaluated from percentage reduction in the reaction number using equation<sup>5,6</sup>.

$$\text{IE \%} = \left[ \frac{RN_f - RN_i}{RN_f} \right] \times 100$$

Where, RN<sub>f</sub> is the reaction number in free solution, RN<sub>i</sub> is the Reaction number in inhibited solution.

## Results and discussion

Phytochemical screening of the *Pongamia pinnata* (L.) Pierre extract given in table-1 revealed the presence of alkaloids, flavonoids, saponins, steroids and tannins. Analysis of the chemical structures of these phytochemical constituents reveal that these compounds are easily hydrolysable and the compounds can be adsorbed on the metal surface via the lone pair of electrons present on their oxygen atoms (i.e. they contain multifunctional group) which make a barrier for charge and mass transfer leading to decrease the interaction of the metal with the corrosive environment. As a result, the corrosion rate of the metal was decreased. The formation of film layer essentially blocks the discharge of H<sup>+</sup> and dissolution of the metal ions. Due to electrostatic interaction, the protonated constituent"s molecules are adsorbed (physisorption) and high inhibition is expected.

**Table 1. Analysis of phytochemical contents of the ethanolic extract of the leaves of *Pongamia pinnata* (L.) Pierre**

Phytoconstituents	Tests	ethanolic extract
Alkaloids	Dragendroff's	+
Tannins	FeCl <sub>3</sub>	+
Steroids	Liebermann-Burchard	+
Terpenoids	H <sub>2</sub> SO <sub>4</sub>	+
Reducing sugar	Benedict's Reagent	-
Carbonyls	2, 4 DNPH s	-
Flavonoids	Alkaline reagent	+
Saponin	Frothing	+

+positive; - Negative

**Effect of sulphuric acid concentration:** In this part the weight loss-time curves of mild steel are constructed, under weight loss method. Figure-1 represents the variation of weight loss with time of mild steel immersed in H<sub>2</sub>SO<sub>4</sub> of different concentrations range (0.5 to 2.0 M). Analysis of these results show that the weight loss (g/cm<sup>3</sup>), increases with time along a period of 60 minute (immersion time), and also upon increasing the concentration of all solutions under test. This may be attributed to the presence of water, air and H<sup>+</sup> which accelerate the corrosion process.

**Table 2. Effect of inhibitor concentration on corrosion rate (CR), inhibition efficiency (IE %) and surface coverage ( $\theta$ ) of mild steel in 2.0 M H<sub>2</sub>SO<sub>4</sub> at 27 °C**

Sl.No.	Concentration (%)	weight loss(g)	Inhibition efficiency (%)	Degree of surface coverage ( $\theta$ )	Corrosion rate (mpy)
1	Blank (2.0 MH <sub>2</sub> SO <sub>4</sub> )	0.063	-	-	5494.80
2	1.0 %	0.048	23.81	0.24	4186.51
3	2.0 %	0.044	30.16	0.30	3837.64
4	3.0 %	0.041	34.92	0.35	3575.98
5	4.0 %	0.038	39.68	0.40	3314.32
6	5.0 %	0.024	61.90	0.62	2093.26

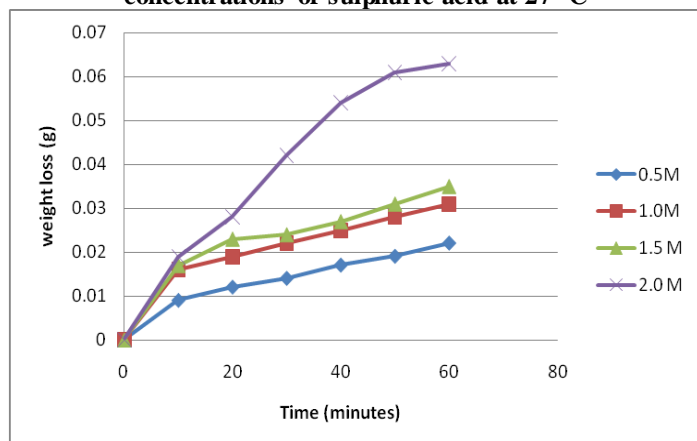
**Table 3. Effect of Pongamia pinnata (L.) Pierre concentration on the parameters of temperature for mild steel in 2.0 M sulphuric acid**

Concentration of inhibitor	Initial temp.(°C)	Max temp.(°C)	Time (minutes)	Reaction Number RN <sub>r</sub>	Inhibition efficiency (%)
Blank (2.0 M H <sub>2</sub> SO <sub>4</sub> )	27.6	63.8	36	-	-
1.0 %	27	62	42	0.17	17.13
2.0 %	27	59.5	48	0.33	32.67
3.0 %	27	54.5	49	0.44	44.19
4.0 %	27.1	52.3	50	0.50	49.88
5.0 %	27	49.5	53	0.58	57.78

**Table 4. Synergistic effect of halides and Pongamia pinnata (L.) Pierre concentration on the parameters of temperature for mild steel in 2.0 M sulphuric acid**

System concentration	Initial temp. (°C)	Max temp. (°C)	Time (minutes)	Reaction Number RN	Inhibition efficiency (%)
Blank (2.0 MH <sub>2</sub> SO <sub>4</sub> )	27.8	70.5	36	-	-
5.0% inhibitor	27.0	37.2	53	0.58	57.78
5.0% Inhibitor +0.1M KCl	27.0	36.1	56	0.66	66.26
5.0% Inhibitor+0.1 M KBr	27.0	34.6	57	0.71	70.69
5.0% Inhibitor +0.1 % KI	27.1	31.6	48	0.95	78.6

This indicates that the corrosion rate of mild steel in test solutions is a function of the concentration of acid solution. This observation agrees with the fact that the rate of a chemical reaction increases with increasing concentrations.

**Figure 1. Weight loss verses time for mild steel at the given concentrations of sulphuric acid at 27 °C**

The values of C.R, IE% and  $\theta$  at different inhibitor concentrations are listed in Table-2. The data in the table reveals that as the inhibitor concentration is increased, the corrosion rate decreases while the efficiency percent and surface coverage increases<sup>7</sup>. This behaviour may be attributed to be the increased

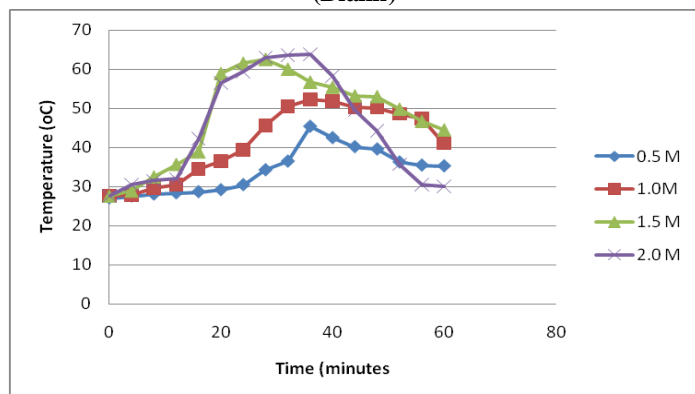
surface coverage ( $\theta$ ) due to the increase in the number of adsorbed molecules on mild steel surface. A good efficiency is observed at constant concentration of inhibitor (5.0%).

The effect of sulphuric acid concentration on the corrosion of mild steel is illustrated in the figure-2 for temperature (°C) against time (min) at different concentrations of H<sub>2</sub>SO<sub>4</sub>. It is observed that the dissolution of mild steel begins after a time lag from the immersion of the coupons in the test solution. Also the temperature rises gradually with time and then decreases after reaching a maximum temperature (T<sub>m</sub>). It is also observed that as the concentration of the sulphuric acid increases, temperature (T<sub>m</sub>) increases and the time required to reach the maximum temperature decreases. This may due to the fact that increase in H<sub>2</sub>SO<sub>4</sub> concentration gives rise to a corresponding increase in the concentrations of active species as well as increase in the rate of chemical reaction.

Temperature change of the system involving mild steel in 2.0 M H<sub>2</sub>SO<sub>4</sub> is a function of time in the absence and presence of given concentrations of Pongamia pinnata (L.) Pierre leaves extract (table-3). The maximum temperature (T<sub>m</sub>) measured in the free acid solution is 63.8 °C and was attained after time (t) of 32 min. This corresponds to a reaction number (RN). Addition of inhibitor caused a decreased in the maximum temperature and an increase in the time required reaching it. This indicates that the inhibitor retards the dissolution rate of mild steel in the acidic solution, may be due to adsorption on the metal surface.

The extent of inhibition depends on the degree of coverage of the metal by the adsorbed molecules. Adsorption is noted for inhibitor, since a simultaneous increase in time and decrease in  $T_m$  takes place, and both the factors cause a large decrease in the reaction number RN of the system (Table-4). Increasing the inhibitor concentration in the acid solution decreases the RN of mild steel and consequently the inhibition efficiency is increased<sup>10</sup>.

**Figure-2. Variation of temperature with time, for the dissolution of mild steel in the given concentrations of  $H_2SO_4$  (Blank)**



The synergetic effects caused by halide ions are given in Table- 4, shows the dissolution of mild steel in different concentrations of Pongamia pinnata (L.) Pierre leaves extract and halides mixture<sup>8</sup>. This shows that Pongamia pinnata (L.) Pierre leaves extract in combination with iodide ions further retards the dissolution rate of mild steel in acidic medium when compared to Pongamia pinnata (L.) Pierre leaves extract alone. The order of reactivity of halide ions is of the order  $KI > KBr > KCl$ . The inhibition efficiency increased 86 % to 94% at a concentration of 0.1 M KI and 5.0 % of inhibitor concentration.

#### Conclusion

Extracts from parts of plants such as roots, stems, and leaves contain some extraordinary phytochemicals that are used as pesticides, antimicrobials, drugs and herbal medicines. Apart from these plant extracts have become important as an environmentally acceptable, readily available and renewable source for wide range of inhibitors<sup>9</sup>. They are the rich sources of ingredients which have very high inhibition efficiency. In this study Pongamia pinnata (L.) Pierre leaves extract is found to be an inhibitor for mild steel corrosion in sulphuric acid medium. Inhibition efficiency increased with increasing inhibitor

concentration. The addition of halides to Pongamia pinnata (L.) Pierre leaves extract enhances the inhibition efficiency. The inhibition efficiency increased 58 % to 78% at a concentration of 0.1 M potassium iodide and 5.0 % of inhibitor concentration.

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