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Research paper

Performance of Piper Nigrum (Black Pepper) as Corrosion Inhibitor of Ductile Iron

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Abstract

The effectiveness of *Piper Nigrum* as green corrosion inhibitor towards ductile iron in 1M Sodium Hydroxide was studied. *Piper Nigrum*, also known as black pepper had undergone extraction process in order to get the final product in the form of liquid which acted as inhibitor. The sample of this inhibitor was analyzed by Fourier Transform Infrared Spectroscopy (FTIR) in determining the bonding and the functional group of the inhibitor. Meanwhile, the capability of *Piper Nigrum* to inhibit corrosion was tested based on concentration of inhibitors by using the gravimetric method. Ductile iron was exposed to 1M Sodium Hydroxide which acted as corrosive medium and inhibitor concentration of 0.1, 0.2 and 0.3 g/L respectively for three hours at constant temperature of 50°C. In addition, there was one ductile iron which was exposed to 1M Sodium Hydroxide in the absence of inhibitor. The ductile iron was then visualized by the microstructure observation through microscope. As a result, FTIR had indicated that the presence of hetero atoms in alkaloids Piperine made *Piper Nigrum* to have an ability to inhibit corrosion. Besides, *Piper Nigrum* could achieve maximum inhibition efficiency of 97.97% while the corrosion rate was only 0.8059 mm/year at 0.3 g/L inhibitor. Furthermore, the concentration of inhibitor gave insignificant effect on the microstructure. However, the morphology of ductile iron microstructure had shown slight difference when the ductile iron was immersed in higher concentration of inhibitor. This had proved that the higher concentration of inhibitor, the less corrosion occurred.

Keywords: *Corrosion; Ductile Iron; Gravimetric Test; Green Corrosion Inhibitor; Piper Nigrum*

1. Introduction

There are two types of metals which are ferrous and non-ferrous metal and both of these can be distinguished by the presence of iron. Ferrous metal contains iron while non-ferrous does not have any. A branch of the ferrous metal is the cast iron family which has high quality of structural and mechanical properties. One of the cast iron family is ductile iron and it has become a subject of renewed interest due to massive burst strength [1]. Ductile iron exhibits a wide range of properties which are obtained through microstructural control by alloying or heat treatment process [2] [3]. The presence of graphite nodules is the most important microstructural feature that gives ductile iron ductility and toughness superior [4]. It is extensively used in industry as the pipeline for the water system, sewer lines and also as material of automotive components [4].

Despite having a great structural and mechanical properties, ductile iron underwent degradation due to vulnerable alkaline medium such as sodium hydroxide and it was also exposed to acidic media like hydrochloric acid which was normally used in industry. The degradation of iron occurred when there was undesirable reaction between metal alloy with environment, which was called as corrosion [4]. This phenomenon had caused some problems such as system failure, increasing cost of maintenance and environmental pollutions. Corrosion of ductile iron can be measured by using gravimetric method [5] [6] and it can occur in non-metallic materials, such as ceramics or polymers. It degraded the useful properties of materials and structures including strength, appearance and permeability to liquids and gases [7] [8]. Gravimetric method which was also known as weight-loss method, was a chemical analysis based on the mass of solid.

The best way to minimize it was by applying inhibitor towards the end formation of corrosion of the material. Basically, there are two commonly used types of inhibitor in industries; organic and inorganic inhibitor. Organic inhibitor can be divided into two types; green and synthetic inhibitor. However, due to high operating cost and hazardous environmental effects, it is suggested not to frequently use synthetic inhibitor even though it has high efficiency towards corrosion [9]. Thus, as an effort in finding the low cost and eco-friendly alternative, the researcher had diverted his attention from synthetic inhibitor to natural products that could be extracted from plant sources and nontoxic organic compound with nitrogen (N), oxygen (O) and sulphur (S) atoms in their molecule [10] [11] [12]. By using extraction method, this kind of solution can be produced from some natural products such as black pepper, ginger and garlic.

Black pepper had been chosen in this research due to the presences of amide and alkaloids components in their major compounds which contributes in inhibiting corrosion [13]. There are several types of peppers which are black, green and white pepper. These peppers were produced in different ways where black and green peppers were made from unripe fruits while green pepper was made from ripe fruits. In details, active components of black pepper were produced from green unripe drupes of pepper plant as shown in Table 1. They were harvested, soaked in hot water to clean and dried either under direct sun or in machines for several days. After a certain period, pepper will be darkened and become wrinkled black layer peppercorn as shown in Figure 1.

It is well known that the addition of inhibitor can restrict the corrosion rate [14] [15] [16], in fact it contributes to the improvement of ductile iron surface. Thus, the purpose of this study was to determine the potential extract of *Piper Nigrum*, or also known as black pepper, to become a green corrosion inhibitor on ductile iron in alkaline solution.

Table 1: Components of Black Pepper

Component	Amount
Water	9.5-12.0g
Protein	10.9-12.7g
Starch	25.8–44.8g
Fiber	9.7–17.2g
Ash	3.4-6.0%
Piperine (pungent)	4.9-7.7%
Essential oils (odiferous)	1.0–1.8%

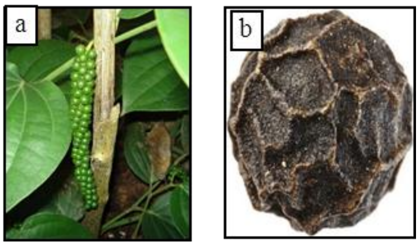


Fig. 1: Peppers: (a) Unripe Peppers; (b) Black Pepper

2. Methodology

2.1. Preparation of Sample

Ductile iron sample was casted by using CO_2 sand casting method and Ferrosilicon Magnesium was used as graphite nucleating agent. The samples were in the form of double cylinder with dimension of 300 mm long and 25±2 mm diameter. The chemical compositions of the samples were obtained through spectrometer test as shown in Table 2.

Table 2: Chemical Compositions of Ductile Iron (wt. %)

C	Si	Mn	P	S	FE
3.49	2.62	0.55	0.069	0.0074	Balance

2.2. Preparation of Specimen

The specimens of gravimetric test were prepared in 30 mm diameter and 1 mm thickness. The specimens were ground by using metallography silicon carbide sand paper with grade of 180, 240, 320, 600 and 800 subsequently.

2.3. Plant extraction

The raw *Piper Nigrum* was washed and rinsed with tap water and distilled water respectively, then it was allowed to air dry at room temperature. The dried black pepper was ground before they were extracted by using 2-propanol as solvent for two hours. The extract was filtered and the solvent was removed by using a rotary evaporator. The extract of *Piper Nigrum* was stored in the chiller at 3°C.

2.4. Gravimetric Test

Gravimetric test was performed to analyze the corrosion behavior of ductile iron at different corrosion medium. Weighed gravimetric specimen was immersed in 250 mL beaker containing 100 mL of 1M NaOH with additional different concentration of *Piper Nigrum* extract (0.1 g/l, 0.2g/l and 0.3g/l) and without *Piper Nigrum* extract. The beakers were covered with parafilm and were gently shaken for three hours in the incubator shaker at 50 °C. All specimens were weighed by using an electronic scale. The specimens were later washed with distilled water and dried. The inhibition efficiency (IE), surface coverage (θ) and corrosion rate (CR) were calculated by using formulae (1), (2) and (3) respectively.

$$\text{Inhibition Efficiency (\%)} = IE = \frac{(W_0 - W_i)}{W_0} \times 100\% \quad (1)$$

$$\text{Surface Coverage, } \theta = \frac{(W_0 - W_i)}{W_0} \quad (2)$$

$$\text{Corrosion Rate (mm/yr), } CR = \frac{876W}{PAt} \quad (3)$$

2.5. Fourier Transform Infrared Spectroscopy (FTIR) Test

Fourier Transform Infrared (FTIR) Test was used to analyse the chemical bond and functional groups of molecule in *Piper Nigrum* extract.

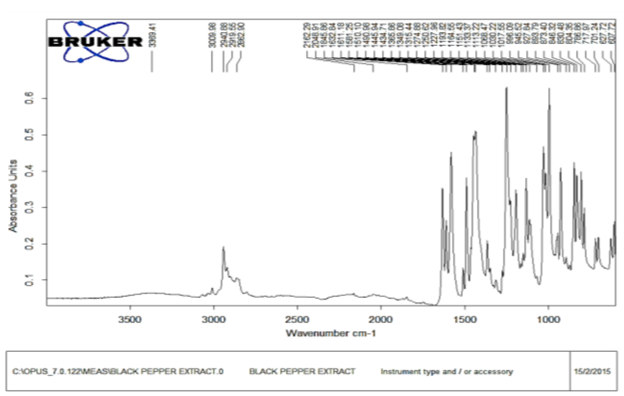
2.6. Microstructure Observation

The surface of ductile iron specimens were observed by a series of standard microstructure observation method, using Huvitz HRM-300 microscope and CapturePro software.

3. Result and Discussion

3.1. Characterization of Extract

There were many compounds presented in the *Piper Nigrum* extract which contributed to the effectiveness of the inhibitor. The FTIR analysis of *Piper Nigrum* had shown the presence of alcohols, alkaloids and free compounds (N, O and F) which gave *Piper Nigrum* its inhibitory effect. Figure 2 clearly shows the presence of alcohol group at O-H stretching vibrations and amide group at N-H stretching vibrations on wave number of 3369.41 cm-1. Besides, the existence of other functional groups such as C-O and C-F attribute to the corrosion inhibition efficiency of *Piper Nigrum*. The presence of hetero atoms especially oxygen and nitrogen in these functional group gave more evidence on inhibitory effect of *Piper Nigrum*. This result was supported by [11] which used Piper Longum (long pepper) fruit as green inhibitor. It also contained oxygen and nitrogen atoms in functional groups (O–H, C=C, C=O, C–N, C–O) and aromatic ring, which met the general consideration of typical corrosion inhibitors [11].



Adsorption peak (cm^{-1}): 3369.41, Bond: N-H (Amide), Intensity: Medium.
Adsorption peak (cm^{-1}): 1274.88, Bond: C-O (Benzoates), Intensity: Strong.
Adsorption peak (cm^{-1}): 1193.82, Bond: C-O (Ethanoates), Intensity: Strong.
Adsorption peak (cm^{-1}): 1151.43, Bond: C-O (Propanoates), Intensity: Strong.
Adsorption peak (cm^{-1}): 1017.55, Bond: C-F (Halogen), Intensity: Strong.

Fig. 2: FTIR Spectra for extraction of *Piper Nigrum*

3.2. Gravimetric Test

The average weight loss and surface coverage of ductile iron immersed in 1M NaOH with and without additional different concentration of *Piper Nigrum* extract was tabulated in Table 3. These results indicated the average weight loss decreased with the increment degree of surface coverage and concentration of the *Piper Nigrum* extract. Similar results were obtained for inhibition of metal in acidic and alkaline media by *Areca catechu* seed [10], *Brugmansia Suaveolens*, *Cassia Roxburghii* [7] and fresh porous silicon [8]. This trend was most likely due to the number of available reaction sites that had been reduced and covered by complex chemical composition extracts.

Table 3: Average Weight Loss and Surface Coverage

Concentration (ml/L)	Average Weight loss (g)	Surface Coverage (θ)
Blank	0.4468	-
0.1	0.3011	0.9520
0.2	0.2128	0.9698
0.3	0.1300	0.9797

Figure 3 reveals the concentration of inhibitor which had affected the inhibition efficiency and corrosion rate of ductile iron. Increasing inhibitor concentration had increased the inhibition efficiency but decreased the corrosion rate of ductile iron. The presence of Nitrogen and Oxygen atoms in the inhibitor functional group had contributed to the effectiveness of the inhibitor for the corrosion of ductile iron in the media [6]. The similar trends were also reported by [12] and [15] when *Nephelium Lappaceum* peel and *Annona Muricata* leaf were used as inhibitor. The corrosion process occurred rapidly when there was no inhibitor as compared to the presence of inhibitor. High adsorption level of active inhibitor molecules from extract occurred at higher inhibitor concentration. [16] also found that corrosion rate decreased in accordance with the inhibitor's concentration.

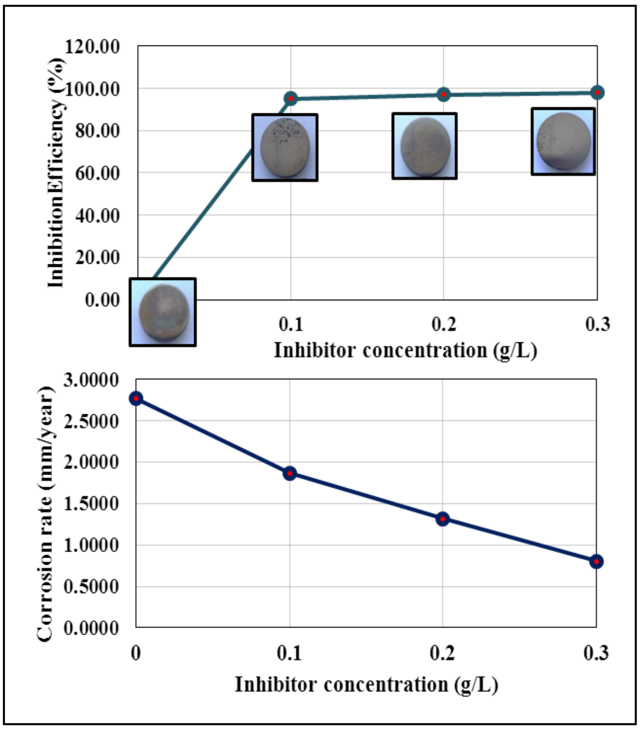


Fig. 3: Effect of inhibitor concentration to inhibition efficiency (IE) and corrosion rate (CR).

3.3. Microstructure observation

Figure 4 indicates the microstructures of ductile iron after immersing in solution that contained different concentration of inhibitor. Both microstructures contained the three basic microconstituents of ductile iron; graphite, ferrite and pearlite. The concentration of inhibitor gave no significant effect on the microstructure element. However, the morphology of ductile iron microstructure had shown slightly difference when the ductile iron was immersed in higher concentration of inhibitor. The pearlite and graphite microconstituents were obviously represented because less corrosion occurred to the sample immersed in higher inhibitor concentration.

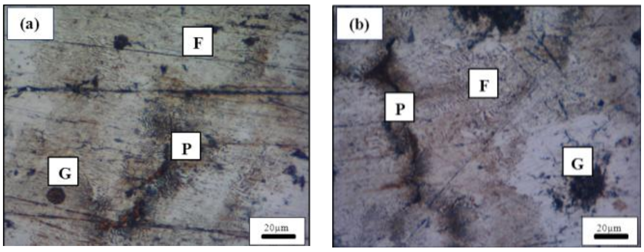


Fig. 4: Microstructure of Ductile Iron at 500 Times Magnification: (a) 0.1g/L Inhibitor Concentration (b) 0.3g/L Inhibitor Concentration. G: Graphite Nodule; F: Ferrite; P: Pearlite

4. Conclusion

In this study, the natural corrosion inhibitor extracted from *Piper Nigrum*, or black pepper was tested its potential in terms of its inhibition efficiency in preventing ductile iron corrosion. The results indicated that *Piper Nigrum* extract had been successfully acted as effective corrosion inhibitor since the inhibition efficiency (%) increased and corrosion rate of ductile iron decreased with the increment of inhibitor concentration. In addition, the existing components in the extract were nitrogen and oxygen which proved that it could be used as inhibitor. Microstructure observation also confirmed there were adsorptions of extracted inhibitor molecules onto ductile iron surface when the inhibitor was mixed with the corrosive medium. Thus, it is concluded that *Piper Nigrum* extracts have the potential to be an effective natural alternative to function as corrosion inhibitor of ductile iron in corrosive solutions.

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