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Corrosion Pattern of Pipeline Steel in Petroleum Pipeline Water in the Presence of Biomas Derived Extracts of *Brassica oleracea* and *Citrus paradise* Mesocarp

Nnaemeka Chinedu Ngobiri, Kaine Okorosaye-Orubite*

Department of Pure and Industrial Chemistry, University of Port Harcourt, Rivers State, Nigeria
Email: *kainelawsonjack@gmail.com

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Abstract

Corrosion inhibition characteristics of two biomass derived extracts from outer leaves of *Brassica oleracea* (BO) and *Citrus paradise* mesocarps (CPM) on pipeline steel were investigated using modified gravimetric method at ambient temperature (28 ± ˚C). Petroleum pipeline water was used to simulate a pseudo-anaerobic corrosion cell. The result obtained showed that corrosion was a continuous process in the closed system, while BO and CPM showed near equivalence corrosion inhibition efficiency of 91.45% and 89.44% respectively at the concentrations studied. The thermodynamic data suggests inhibition to be through molecular adsorption on metal surface.

Keywords

Adsorption, Corrosion, Modified Gravimetric, Pipeline Water, Pseudo-Anaerobic

1. Introduction

The problem of metallic corrosion is an age long issue and mankind has not ceased to find lasting solution. Though a natural process, the driving force has been attributed to the energy imputed into metallic material during refining of their ores. This thermodynamically inevitable process results to the indirect cost of corrosion damages [1]. Many corrosion mitigation methods abound, one of such is the application of corrosion inhibitors in metallic environments [2] [3]. Steel pipelines are used to transport domestic and industrial fluids because of convenience. However, as metals, they are prone to worsening internal corrosion because of the unique condition of the inner walls of the pipeline. This adversely affects their life span if not controlled or prevented; hence the necessities to develop corrosion inhibitors that are peculiarly suited to control steel pipeline corrosion [4] [5] [6]. Corrosion inhibitors can be organic or inorganic, natural or synthetic. Organic corrosion inhibitors are versatile and function by adsorption on the metallic surface through adsorption sites on their molecules [7] [8] [9]. Plants extracts as corrosion inhibitors are found to be eco-friendly, cheap, and re-newable. They possess active adsorption sites through which inhibition occurs [10] [11], thus are excellent replacement for toxic corrosion inhibitors.

Most plant materials used as corrosion inhibitors also serve as food in some societies and may likely cause a competitive demand in the food chain [11] [12]. Materials regarded as waste, are of less economic importance and would serve the dual purpose of corrosion inhibition and enhance a greener environment. Outer leaves of *Brassica oleracea* (BO) and *Citrus paradise* mesocarp (CPM) are both parts of the fruits which are discarded while other parts are eaten. These parts are therefore considered as waste materials. Pipeline corrosion protection processes include use of sacrificial coating [13], surface coating with inorganic compounds [14] or use of conducting membranes as sensors [15]. These processes may be expensive and materials not ready available hence the advantage of cheap readily available natural material like BO and CPM.

This paper investigates the corrosion behaviour of pipeline steel in petroleum pipeline water in the presence of various concentrations of extracts of outer leaves of *Brassica oleracea* (BO) and *Citrus paradise* mesocarp (CPM) using a modified gravimetric method. The determinations were conducted separately for each additive and the results compared. These biomasses are usually discarded as waste in most homes and food drink industries. The beneficial application of this biomass will contribute to global quest for sustainable development and greener world. Interestingly, the corrosion inhibition characteristics of *Brassica oleracea* and commercial Rutin derivable from *Citrus* mesocarp have been previously reported by our research group, so also, the corrosion inhibition of mild steel corrosion in an acidic medium by juice of *Citrus paradise* [5] [16].

2. Materials and Methods

The test electrolyte was petroleum pipeline water (PPW). This was collected with a sterile container from Shell Petroleum Development Company's (SPDC) Trans Niger Pipeline at Kolo creek in Bayelsa state, Nigeria. The PPW was stored at 4˚C, allowed to normalize to the experimental condition before it was used for the experiments. The composition of the petroleum pipeline water has already been reported [5]. The test coupon/electrode was petroleum pipeline steel (PPS) of composition (C-0.45, S-0.07, P-0.003, S-0.26, Fe-balance) cut into dimensions 3 cm × 2.5 cm × 1 cm and perforated at the edge for hanging with a polymeric thread. The steel coupons for the experiments were polished successively with emery paper from 150 to 2000 grits and rinsed with ethanol. Acetone was applied to remove any residue from polishing and there after air dried. The test electrode has been characterized as previously reported [16]. All other reagents used were of analytical grade.

The extracts were prepared by peeling off the mesocarp of *Citrus paradise* and outer leaves of *Brassica oleracea*, identified by the Plants and Biotechnology Department of University of Port Harcourt. These were dried in an oven below 40˚C and pulverized using electric blender to ensure enhanced extraction. 500 g of both pulverized powders were soaked in separate 1000 ml volumetric flasks using 99% ethanol for 72 hours. Ethanol was poured to slightly cover the surface of the samples during the soaking period. The content of the flasks was thereafter filtered and the filtrate concentrated using rotary evaporator. The concentrated extract was transferred to a sterile air tight analytical container. Appropriate quantities were collect from the container to make desired test corrosion environments.

Modified Gravimetric Experiments

The pre-cleaned and weighed coupons were hanged into 250 ml beakers containing PPW appropriate extract quantities using polymeric threads and glass rods. The tests were conducted under total immersion conditions of the unstirred test solutions at 28˚C ± 1˚C. Pseudo-anaerobic conditions were simulated by tightly sealing the entire corrosion cells using 25 microns-thick aluminum foil and tape. Aluminum foils are known to be impermeable to light, air and moisture. The weight loss was determined with respect to time, by retrieving the coupons after seven days intervals (*i.e.* weekly) for 35 days, scrubbed with bristle brush, washed, dried and re-weighed. The weight loss was taken to be the difference between the weight of the coupons at a given retrieval and its initial weight. All the tests were run in triplicate and the data showed good reproducibility. The average values for each experiment were obtained and used for subsequent calculations and analysis.

The application range of the formulation was 35 days, coupons were retrieved and washed at & days interval.

3. Results and Discussion

3.1. Gravimetric Behaviour of Pipeline Steel in Petroleum Pipeline Water

Figure 1 shows the weight loss of pipeline steel in petroleum pipeline water. A continuous rise in corrosion of pipeline steel in petroleum pipeline water up to five weeks was observed in the close system. This indicates that the thermodynamic variables tend towards corrosion as a continuous process in the closed system. However the rate of increase in corrosion was not steady with time as shown in the graph. This may be attributed to changes in corrosion mode, probably caused by fluctuation between aerobic to anaerobic attacks. Crude petroleum pipelines operation involves the opening and closing at intervals in

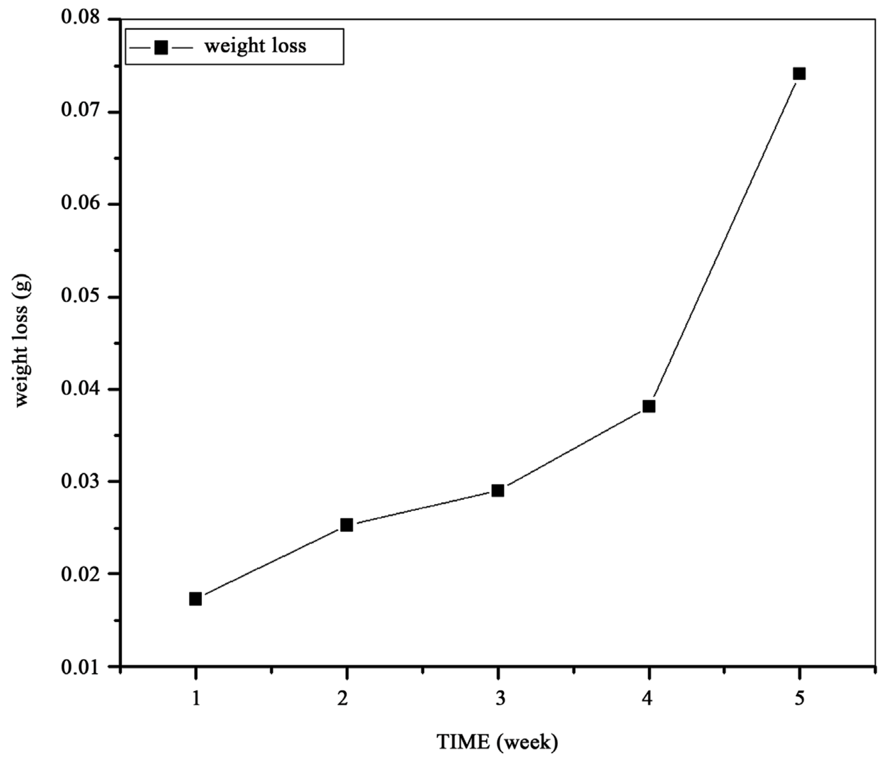

Figure 1. Variation of weight loss with time (weeks) of corrosion of pipeline steel in petroleum pipeline water.

order to carry out certain activities such as pigging and chemical injection. Similarly during this pseudo-anaerobic corrosion experiments, the cells were opened and closed at regular intervals each time weight loss was determined. Aerobic microorganisms may have been introduced into the cell and deplete the oxygen content at such intervals. The observed fluctuations may also be attributed to deficiencies of the weight loss method, where the surface corrosion products have to be brushed off with each determination. However, since the anaerobic condition is a dominate condition, there is need for further studies on the microbial activities in the cell.

3.2. Corrosion Inhibition Studies of Pipeline Steel

The use of weight loss method to measure corrosion is a classical one in measuring corrosion in metallic structures. However, weight loss is closely related to corrosion rate as its data is used to calculate corrosion rate. The weight loss increased steadily with time until fourth week when a pronounced rise was recorded. As expected the highest weight loss and corrosion rate was recorded with 0.0 g/L BO and OM extracts. These results show that *Brassica oleracea* and *Citrus paradise* mesocarp (CPM) extracts have significant capacity to reduce the dissolution of pipeline steel in petroleum aqueous environment. This capacity increased with concentration of extracts and time, a trend that has been reported by many researchers [17] [18] [19].

The corrosion rate (CR) for the pipeline steel in the petroleum pipeline water environment was calculated from the weight loss data using Equation (1). The data obtained was plotted as a function of time and extract concentration. The plots are presented in Figure 2 and Figure 3.

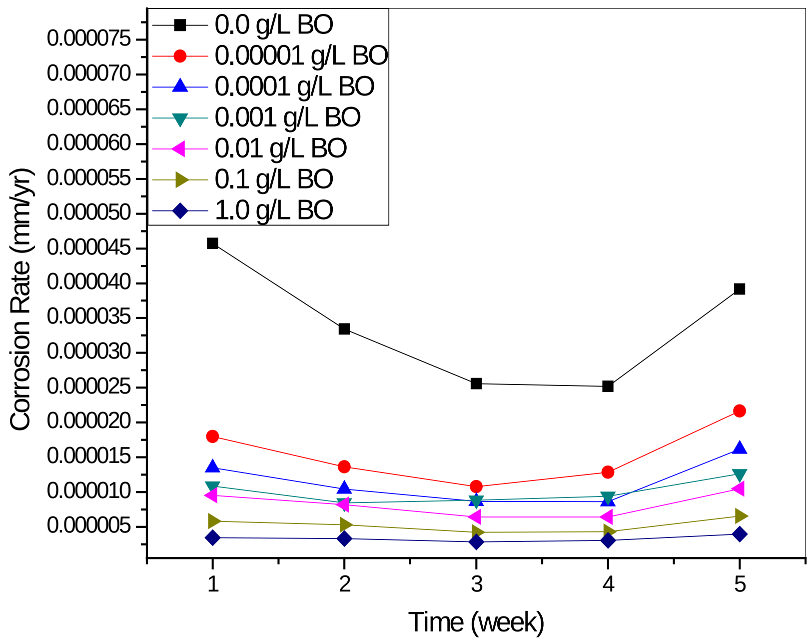

Figure 2. Variation of corrosion rate (mm/year) with time (weeks) of the pseudo anaerobic corrosion inhibition of pipeline steel in petroleum pipeline water with various concentrations of *Brassica oleracea* extract (BO).

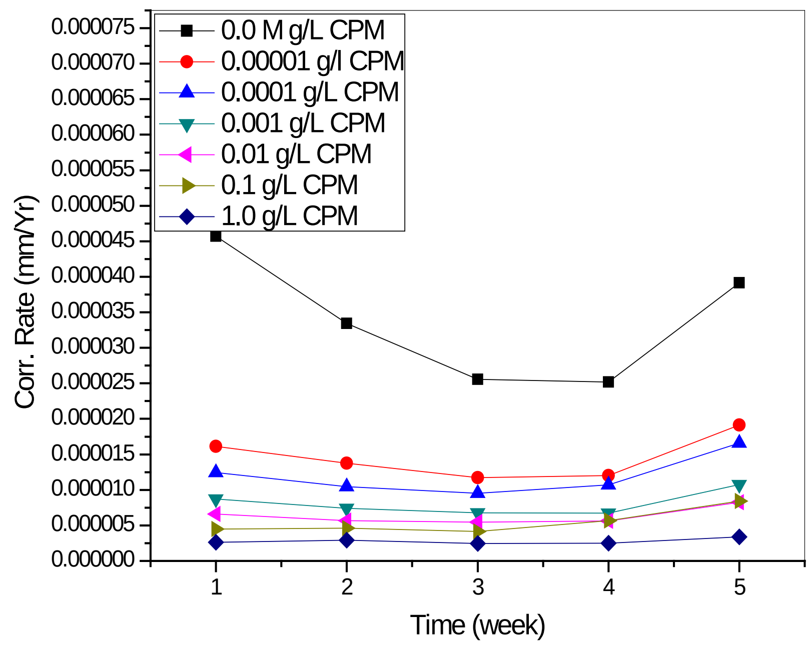

Figure 3. Variation of corrosion rate (mm/year) with time (weeks) of the pseudo anaerobic corrosion inhibition of pipeline steel in petroleum pipeline water with various concentrations of *Citrus paradise* mesocarp extract.

$$CR_{(\text{mm per year})} = 87.6\Delta w/DAT \tag{1}$$

Δw is the weight loss, D the density of steel (g/cm^3), A the area of the coupon in (cm^2) and T exposure time (h).

Figure 2 and Figure 3 show the variation of corrosion rate (mm/year) with time (weeks) of the pseudo anaerobic corrosion inhibition of pipeline steel in petroleum pipeline water with various concentrations of *Brassica oleracea* and *Citrus paradise* mesocarp extracts respectively. Though the corrosion rate initially seemed to decrease with time, the highest corrosion rate was recorded after three weeks of immersion in both extracts, an indication that corrosion can get worse with time after initiation. This may be partly explained by the presence of chloride ion which initiates pitting and is usually autocatalytic [7] [20]. The probable introduction of another form of corrosion may have increased the corrosion rate with time.

3.3. Corrosion Inhibition Characteristics of CPM and Bo Extracts

The corrosion mitigation capacities of both extracts on pipeline steel corrosion was evaluated by quantifying the corrosion inhibition efficiency IE (%) using Equation (2)

$$IE\% = \left(1 - \frac{CR_{inh}}{CR_{blank}}\right) \times 100 \tag{2}$$

where CR_{inh} and CR_{blank} represent the corrosion rates in inhibited and uninhibited solutions, respectively. Figure 4 and Figure 5, present the variation of

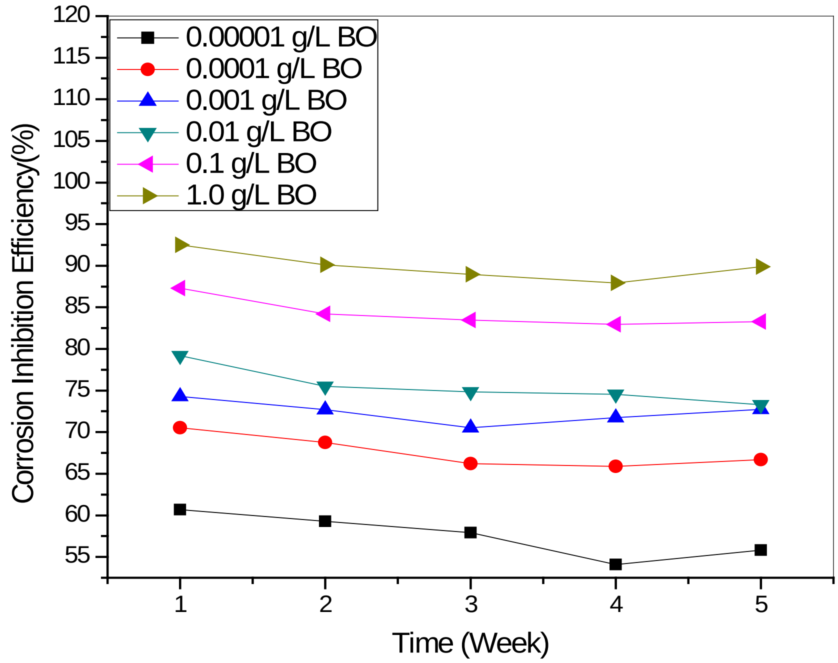

Figure 4. Variation of corrosion inhibition efficiency (%) with time (week) for the pseudo anaerobic corrosion inhibition of pipeline steel in petroleum pipeline water with various concentrations of *Brassica oleracea* extract (BO).

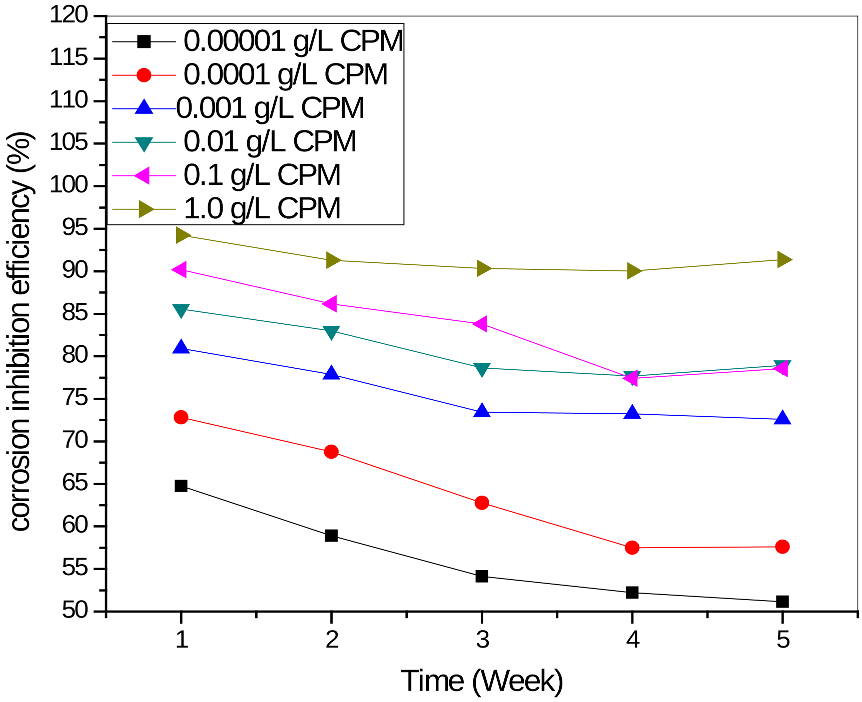

Figure 5. Variation of corrosion inhibition efficiency (%) with time (week) for the pseudo anaerobic corrosion inhibition of pipeline steel in petroleum pipeline water with various concentrations of *Citrus paradise* mesocarp extract.

corrosion inhibition efficiency (%) with time (weeks) and concentration (g/l) for the pseudo anaerobic corrosion of pipeline steel in petroleum pipeline water with various concentrations of BO and CPM. Figure 4 and Figure 5 reveal a general reduction in corrosion inhibition efficiency with time up to the third week when there was a slight increase. The corrosion rate and concentration have inverse relationship within the study concentrations. This implies that increased application of BO and CPM reduced corrosion rate within the study condition. The corrosion inhibition behavior of most plant extracts follows this pattern [12] [21] [22]. Worthy of note is that both extracts showed comparable corrosion inhibition capacity of 91.45% and 89.88% respectively at equivalent concentrations. Also corrosion inhibition efficiencies increased with increasing concentrations of both extracts [23] [24]. Corrosion inhibition efficiency trend is therefore concentration dependent.

The tendency of corrosion inhibition efficiency dependence on concentration is related to the surface coverage by the inhibitors because corrosion inhibitors act by getting to the metal surface, covering it and getting adsorbed on the metal surface [25] [26].

Different mechanisms have been proposed to explain the activity of inhibitor molecules on metallic surface. The inhibitors either block the corrosion active sites by geometrically blocking the attacking corrodent from getting to the active sites or by catalytically altering the corrosion environment or the corrosion products to minimize corrosion [27]. The mechanism followed is usually dependent on physio-chemical properties and interaction between the metal, inhibitor and the environment. The extent of these interactions is usually concentration dependent. The concentration of the inhibitor is an important factor in determining the surface coverage (θ) on the metal and consequently the corrosion inhibition efficiency.

$$\theta = 1 - \frac{CR_{inh}}{CR_{blank}} \quad (3)$$

The surface coverage characteristics of the pseudo anaerobic corrosion inhibition of pipeline steel in petroleum pipeline water in the presence of various concentrations of *Brassica oleracea* and *Citrus paradise* mesocarp extracts were calculated using Equation (3). The results obtained are presented in **Figure 6** and **Figure 7**. Sel vi *et al.*, [28] previously reported that inhibitors act by separating metal surface from the corrodent covering its surface. The surface coverage of BO and CPM extracts increased with increase in their concentrations. This trend must have resulted from the availability of more inhibitive phyto molecules which covered the surface of pipeline steel as concentration increased. The drastic increase in surface coverage from concentration of 0.00 g/l to 0.2 g/l of both extracts further confirms the effectiveness BO and CPM as corrosion inhibitors.

The result of weight loss data with time can be related to the kinetics of corrosion. **Figure 8** and **Figure 9** present the plot of $\log(W - W_1)$ versus time (weeks)

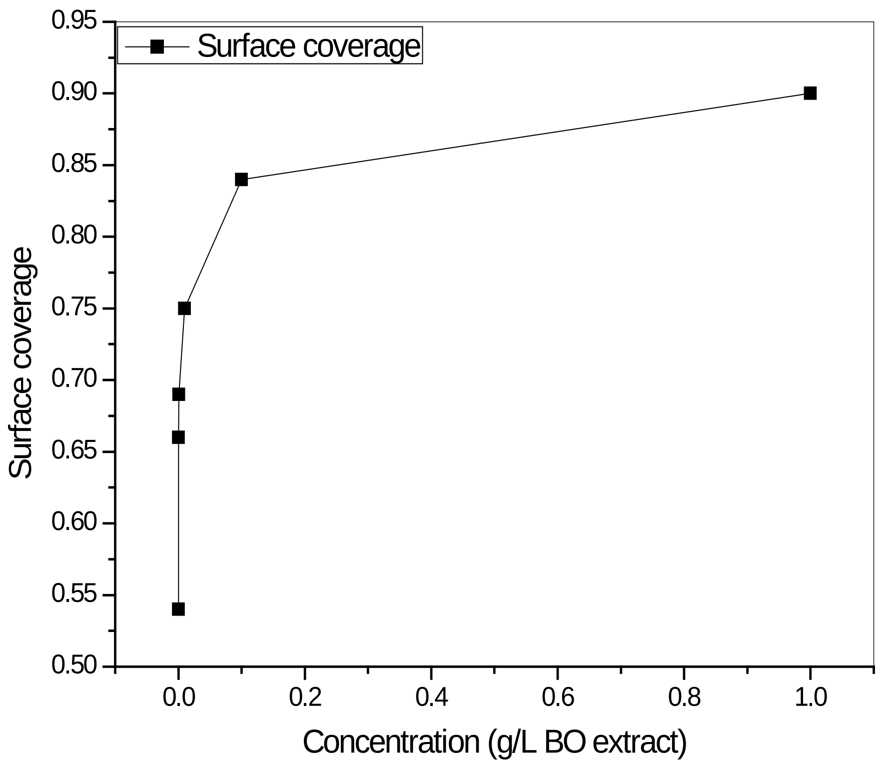

Figure 6. Variation of surface coverage (θ) with concentration (g/l) for the pseudo anaerobic corrosion inhibition of pipeline steel in petroleum pipeline water with various concentrations of *Brassica oleracea* extract (BO).

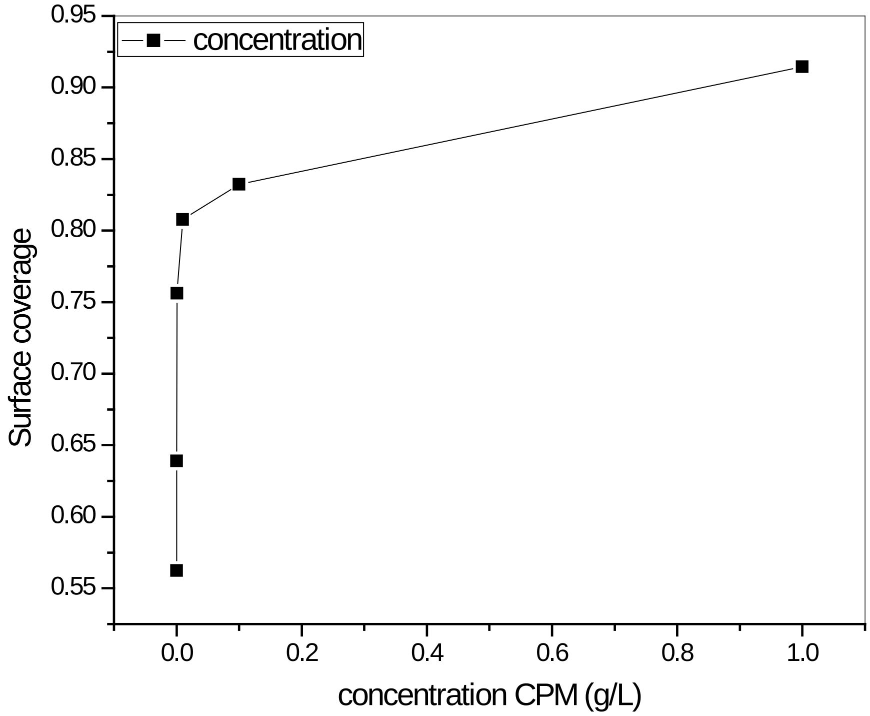

Figure 7. Variation of surface coverage (θ) with concentration (g/L) for the pseudo anaerobic corrosion inhibition of pipeline steel in petroleum pipeline water with various concentrations of *Citrus paradise* mesocarp extract.

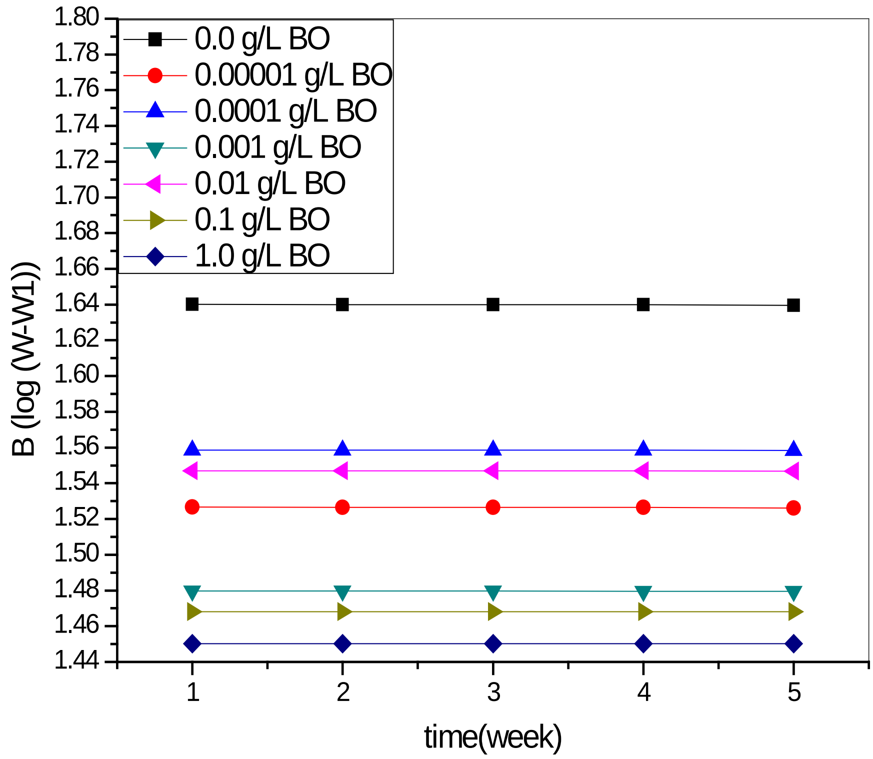

Figure 8. Plot of log(W − W_1) versus time (week) for pipeline steel in petroleum pipeline water in the presence of various concentrations of *Brassica oleracea* extract (BO) extract under pseudo anaerobic condition.

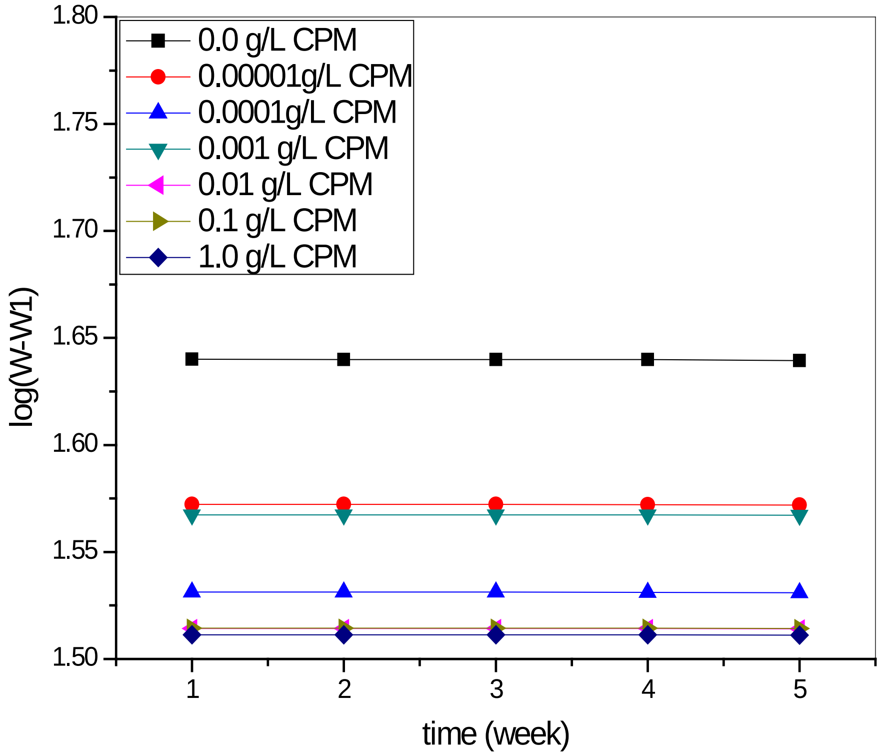

Figure 9. Plot of log(W − W_1) versus time (week) for pipeline steel in petroleum pipeline water in the presence of various concentrations of *Citrus paradise* mesocarp (CPM) extract under pseudo anaerobic condition.

for pipeline steel in petroleum pipeline water in the presence of various concentrations of BO and OM extracts under pseudo anaerobic condition. (W and W_1 are initial and final weight of pipeline steel in uninhibited environment respectively.)

The straight line plots indicate that the corrosion reaction is of first order kinetics [29].

The corrosion rate constant k for the pseudo anaerobic corrosion of pipeline steel in petroleum pipeline water with various concentrations of *Brassica oleracea* extract (BO) and *Citrus paradise* mesocarp (CPM) were calculated using equation 4 and presented in **Figure 10** and **Figure 11**.

$$K = \frac{2.303}{\text{Time}} \log \frac{w_i}{w_f} \tag{4}$$

where w_i and w_f are initial and final weight respectively.

Figure 10 and **Figure 11** present the variation of corrosion rate constant with time for the pseudo anaerobic corrosion of pipeline steel in petroleum pipeline water with various concentrations of *Brassica oleracea* extract (BO) and *Citrus paradisi* mesocarp (CPM). The weekly average corrosion rate constant seemed to be steady. The rate constant decreased with increase in concentration of BO and OM extracts. This indicates that *Brassica oleracea* and *Citrus paradise* mesocarp extracts can reduce the corrosion rate of pipeline steel in pipeline water.

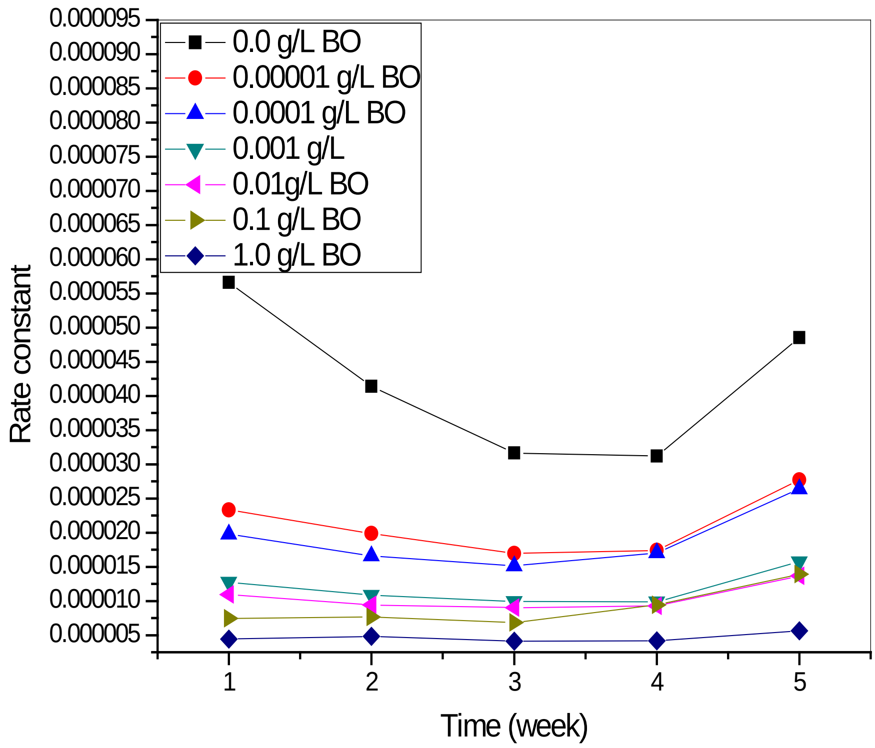

Figure 10. Variation of corrosion rate constant with time (week) for the pseudo anaerobic corrosion of pipeline steel in petroleum pipeline water with various concentrations of *Brassica oleracea* extract (BO).

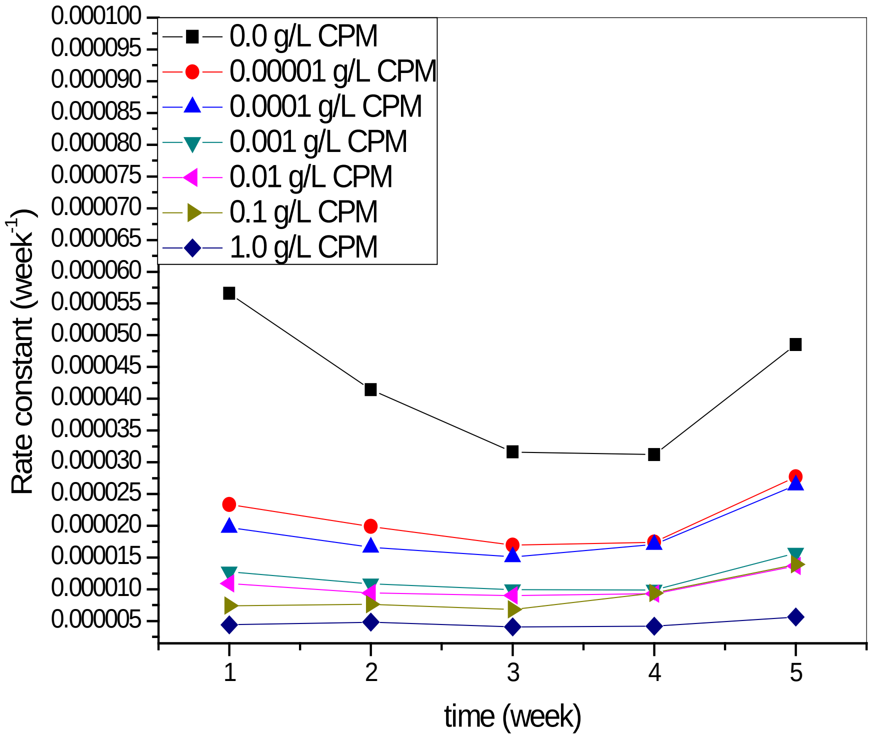

Figure 11. Variation of corrosion rate constant with time ($week^{-1}$) for the pseudo anaerobic corrosion of pipeline steel in petroleum pipeline water with various concentrations of *Citrus paradise* mesocarp extract.

The corrosion rate constant decreased with increase in concentration of BO and CPM extracts. This may lead to longer half-life as extracts concentration increases [5].

The material half-life $t_{1/2}$ of petroleum pipeline steel in petroleum pipeline water with various concentrations of BO and CPM extracts were calculated from k using Equation (5). The results obtained were presented in Figure 12 and Figure 13

$$t_{\frac{1}{2}} = \frac{0.693}{k} \tag{5}$$

As shown in the graphs the half-life of the pipeline steel increased with increase in concentration of the extracts. However both are inversely related to the corrosion rate constant. They are therefore useful parameters as tools in material integrity assessment.

Gibbs free energy of adsorption is another good measure of the propensity of a chemical process under specific conditions. The Gibbs free energy was calculated using Equation (6). The results obtained were presented in Table 1

$$-2.303RT\log\left[\frac{55.4\theta}{Co(1-\theta)^{n}}\frac{\{\theta+(1-\theta)n\}^{n-1}}{n^{n}}\right] \tag{6}$$

where Co is the concentration of inhibitor in the bulk of the solution; n is the size factor (9 for flat adsorption on the surface and 3 in the perpendicular direction to the surface), R is gas constant, T is the absolute temperature.

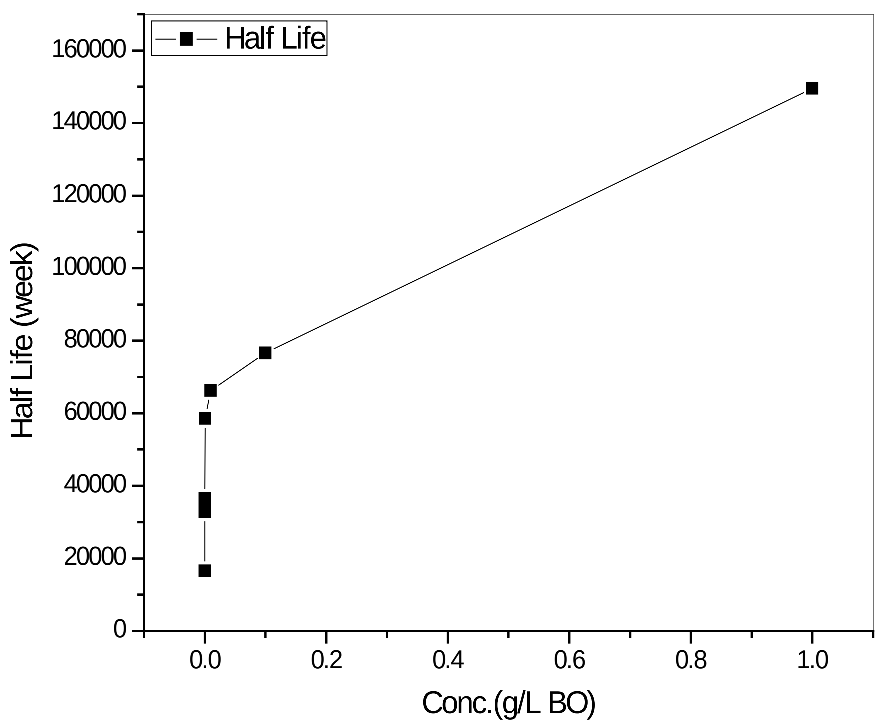

Figure 12. Variation of half-life with concentration (g/l) for the pseudo anaerobic corrosion inhibition of pipeline steel in petroleum pipeline water with various concentrations of *Brassica oleracea* extract.

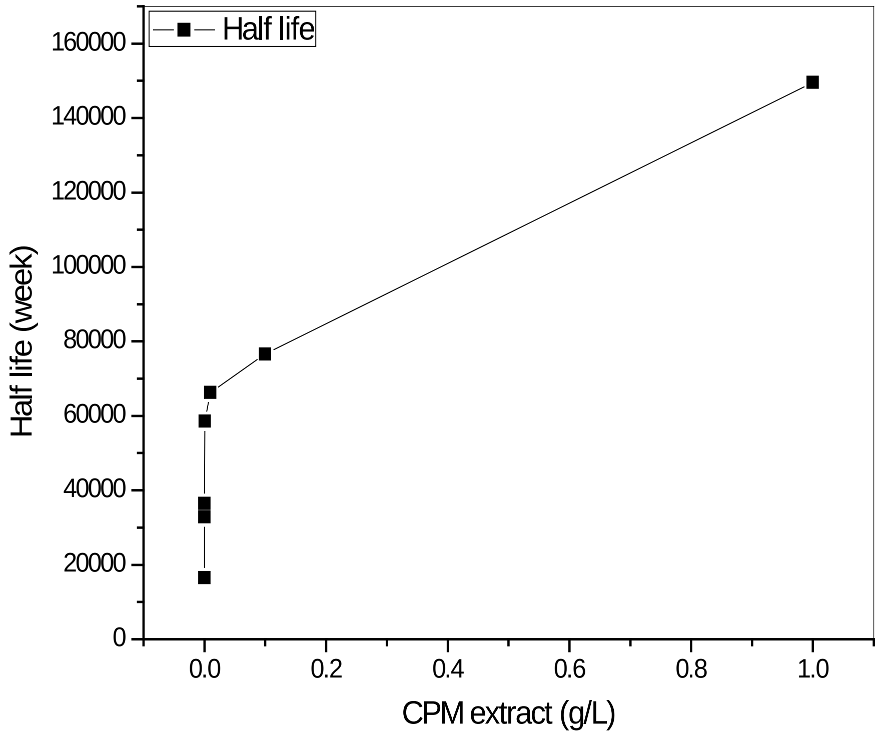

Figure 13. Variation of half-life with concentration (g/L) for the pseudo anaerobic corrosion inhibition of pipeline steel in petroleum pipeline water with various concentrations of *Citrus paradise* mesocarp extract.

Table 1. Calculated values of ΔG_{ads} for BO and CPM.

Concentration (g/l)	ΔG_{ads} (BO)	ΔG_{ads} (CPM)
0.00001	−31.24	−8.72
0.0001	−27.86	−7.45
0.001	−23.07	−4.87
0.01	−19.05	−1.24
0.1	−17.23	−7.11
1.0	−19.93	−1.78

Table 1 presents the variation of Gibbs free energy of adsorption with concentration (g/l) for the pseudo anaerobic corrosion inhibition of pipeline steel in petroleum pipeline water with various concentrations of BO and CPM. The values in the table are in the negative region showing that adsorption of BO and CPM was spontaneous. However, the values of ΔG_{ads} increased with decreased concentration suggesting a possible steric hindrance due to the bulky and many phyto molecules in BO and CPM extracts [30]. Researchers [31] [32] have previously attributed adsorption potentials of a citrus species-orange mesocarp extract to Rutin, which is a bulky molecule.

The higher values of ΔG_{ads} above −25 kJ/mol suggest that the molecules of the extracts are chemisorbed on the pipeline steel surface [33].

4. Conclusions

A modified gravimetric procedure was used to measure the pseudo anaerobic corrosion inhibition of pipeline steel in petroleum pipeline water by extracts of BO and CPM. The work has shown that BO and CPM successfully inhibit the pipeline corrosion in the aqueous petroleum environment. Weight loss data revealed that corrosion rate decreased with increased concentration of both extracts. Half-life and surface coverage data confirm these extract as inhibitors for pipeline steel in the environment under investigation. The Gibb's free energy calculated values that suggest possible adsorption of inhibitor molecules by chemisorption on pipeline steel surface. However, the ΔG_{ads} values were higher at lower concentrations which were attributed to steric hindrance as phyto molecules from the extracts increase. Inhibition efficiencies of 91.45% and 89.88% were obtained for BO and CPM respectively. Therefore, the extracts of BO and CPM can be added to the list of safe, non toxic and environmental friendly corrosion inhibitors.

Due to some limitations experienced during this work, the researchers are unable to present SEM images of the extracts presently. However, the research is still ongoing where SEM images as well other methods of affirming the effectiveness of these two plant extracts as corrosion inhibition will be presented. The authors also suggest further work on developing of surface coating paint using BO and CPM as additives.

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