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CORROSION INHIBITION EFFICACY OF HYBRID ORGANIC EXTRACTS FROM *PROSOPISAFRICANA* AND *CITRULLUSLANATUS* ON MILD STEEL IN ACIDIC MEDIUM

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ABSTRACT

Each of Prosopis Africana pod extract (PAPE) and Citrulluslanatus (WMPE) performed quite well as green inhibitors in the electrochemical corrosion in acidic solution at ambient temperature in previous research. However, the hybridization of these compounds was varied with the concentration ratio in this study to optimize the efficiency of the green inhibitor in a hydrochloric acid medium using mild steel. The extraction process of the inhibitor samples was carried out with the Soxhlet apparatus using n-hexane as the solvent. Furthermore, the potential inhibition efficiency was monitored using various corrosion measurement techniques at room temperature. The inhibitive response of the extracts could be attributed to the adsorption of the extracts' components on mild steel surface by physisorption mechanism according to the Langmuir adsorption isotherm model. The results revealed that inhibition efficiency (IE%) depends on the concentration of the extracts. The peak IE% values were obtained with a hybrid ratio of 3:1 (PAPE: WMPE) for gravimetric technique. The Tafel polarization and gasometrical measurement technique is 96.7%, 98.5% and 83.1% respectively at the concentration of 1.0 g/L, which are above the peak IE% for PAPE (93.7%, 80.96% and 77.8%) and WMPE (92.7%, 80.5% and 75.6%).

KEYWORDS: hybrid, green inhibitor, prosopis africana, citrulluslanatus, inhibition efficiency

1. Introduction

The major degradation mechanism of mild steel in environmental operations is corrosion. Steel corrosion has been the subject of several studies due to its low-cost, wide range of mechanical properties, and industrial applications especially in steel structures. The consequences of this electrochemical reaction affect the safe reliability and effective operation of metal components or equipment and are frequently more severe than the mere loss of a quantity of metal [1]. Organic inhibitors have effectively isolated the metal from corrosion agents [2] since the inorganic compounds endanger the ecosystem [3-5]. One of the challenges of selecting the type of inhibitor has been to develop a scalable particular with approach to identify sustainable bio-material that portends excellent inhibition. Therefore, the inhibitor composition determined the efficiency which includes the size of the molecule, the amount and node of adsorption molecule, the charge density, metallic complexes formation, and the inhibitor's intended area of the metal surface; notwithstanding, characteristics of the environment and the nature of electrochemical potential at the interface [6, 7].

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Based on the hazardous effect of most synthetic corrosion inhibitors, a lot of scientists have considered research on the use of natural biodegradable materials and some agricultural products and wastes can be used as corrosion retardation since they are economical, easily accessible, and renewable sources of materials, as well as being environmentally safe and ecologically tolerable [8-11]. The corrosion inhibitive capacities of Prosopis Africana and watermelon peel in 1 M Hydrochloric (HCl) acid medium for low carbon steel grades have been reported separately. This is monitored with gravimetric and Tafel polarization method in PAPE, analysing the corrosion inhibition and protection of zinc in natural seawater by watermelon peel respectively [2, 12-16]. There are just a few accounts in the specialised literature on using Prosopis Africana and watermelon components for corrosion inhibition. The existing research on plant extracts as corrosion inhibitors mainly focuses on specific plant components such as leaves, stems, bark, roots, and fruits. However, hybridizing research on extracts from diverse components of a certain plant for corrosion inhibition is limited. In furtherance to the studies on the development of green corrosion inhibitors, this study investigates the inhibiting effect of hybridizing Prosopis Africana pod and watermelon peel extract in different ratios on the corrosion of mild steel in 1M HCl solution using gravimetric analysis, gasometric analysis, and Tafel polarization methods.

2. Experiments

2.1. Preparation of inhibitor extracts

The Prosopis Africana pod and the watermelon peels were air dried and ground into a fine powder. The extractions of the samples were carried out in the Department of Chemistry, University of Ilorin, Ilorin, Nigeria. The samples were extracted on a Soxhlet apparatus with n-hexane as the solvent. The extracts were utilized to make stock solutions, which were then used to evaluate the corrosion inhibition properties independently and in combination. Samples of each stage in preparing the inhibitor extract are presented in Figure 1.

2.2. Materials Preparation

Mild steel sourced in the local market with elemental composition analysed at Midwal Engineering Service Limited in Lagos, Nigeria using Spectromaxx LMF06 Spectrometer. The 2.5 x 2.0 x 0.1 cm, coupons for the gravimetric test were prepared using ASTM G1-03 [17] and G4 standards [18] then polished, degreased in ethanol, dried in acetone, and then stored in a desiccator. For the corrosion study, an acidic solution was prepared by dilution of HCl (sp.gr.1.18) with distilled water.

Fig. 1. *The process of extraction of* Prosopis Africana *pod and watermelon peel*

2.3. Weight Loss Measurement/ Gravimetric Technique

The simplest technique of corrosion rate and inhibition efficiency is to use weight loss measurement. The test specimens were pre-weighed and totally immersed in 200 mL solutions of various concentrations prepared in 1.0 M HCl solution at (38 °C), with and without various proportions of Prosopis Africana pod extract and watermelon peel extract with the ratios: 2:1, 1:2, 3:1 and 1:3, sealed from the atmosphere using ASTM NACE/ASTM G31-12a guidelines [19]. The test specimens were exposed to the medium between 24 and 2,160 hours in accordance with guidelines in the ASTM G1 standard [17]. The experiment setup is shown in Figure 2. The specimens were removed after 24 hours, washed in distilled water, rinsed in ethanol and acetone, dried, and reweighted using an electronic weighing balance (HX 302 with 0.01 g accuracy). The process was repeated for other periods of exposure in the medium until 2,160 hours of observations. The corrosion rate was determined using Equation (1),

$$Corrosionrate(mpy) = \frac{kW}{ATD} \quad (1)$$

where W is the weight loss (g), A is the total area of metal specimen in cm^2, k = 3.45 x 10^6 mils per year (mpy), D is the density of steel (g/cm^3) and T is the immersion time (hours).

The calculated corrosion rate inhibition efficiency (I.E.%) was obtained by using the relationship in Equation (2),

$$I.E(\%) = \frac{CR_{Blank} - CR_{Inh}}{CR_{Blank}} \times 100 \quad (2)$$

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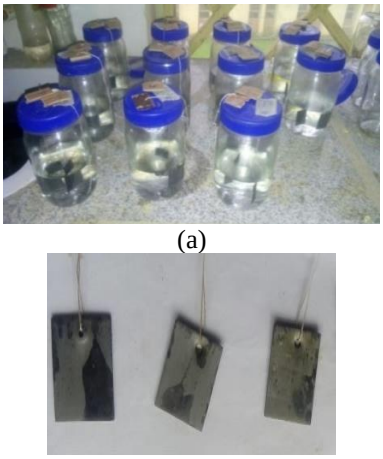
where CR_{Blank} and CR_{Inh} is the corrosion rate of the mild steel specimens in the absence and presence of inhibitor respectively. The experimental setup and some specimens after the corrosion tests are shown in Figure 2.



(a)

(b)

Fig. 2. *(a) Weight loss set-up and (b) cleansed specimen after corrosion test*

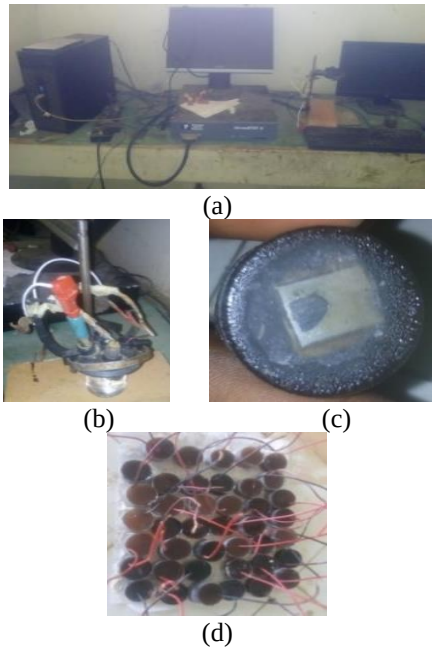
2.4. Hydrogen evolution (Gasometric) measurement

The volume of evolved hydrogen gas from corroded mild steel was measured using the hydrogen evolution measurement setup. A 200 mL of prepared environment (1 M HCl) was put into two-necked conical flasks and the burette beginning volume was set to 50 mL. To prevent gases from escaping, the test specimen from the desiccator was put into the HCl solution and tightly closed. The volume of hydrogen bubbles formed, as a result of the reaction between metal and acidic environment, which increased over 300 minutes. During the reaction, the volume was measured at 10-minute intervals and recorded with the downward displacement of water in the burette. This procedure was carried out for other prepared inhibition test solutions of different concentrations of the hybrid *Prosopis Africana* pod extract (PAPE) and watermelon peel extract (WMPE). The inhibition efficiency (I.E%) from the hydrogen evolution measurement was determined using Equation (3).

$$I.E\% = 1 - \frac{CR_{Inh}}{CR_{abs}} \times 100\% \qquad (3)$$

2.5. Tafel *polarization technique*

Tafel polarization experiments were carried out at room temperature utilizing a three-electrode cell configuration. Using a guillotine machine, mild steel of 1.0 cm^2 was employed as the working electrode, and a platinum electrode was used as an auxiliary electrode dimension. Using aluminium foil to hold it together, a flexible cable was linked to the specimen and placed on a cup mold. In another cup mold, a hardener was applied to a polyester resin and carefully combined. An accelerator was added to the mixture and the two were mixed together to form a solution. The prepared solution was then poured in the mold where the specimens were placed, and left for a period between 15 to 20 minutes to solidify. After solidification, they were removed from the mold. Before being exposed to the atmosphere, the coupon was further polished with several grades of emery sheets to achieve a gleaming reflective surface (like a mirror). The preparation of the specimen samples for the *Tafel* polarization procedures is shown in Figure 3.



(a)

(b) (c)

(d)

Fig. 3. *(a) Electrochemical measurement analysis setup (b) Connection of electrodes inside the medium (c) Mounted* Tafel *sample before polishing (d)* Tafel *samples ready for test*

3. Results and discussion

3.1. Elemental Composition Analysis

Elemental composition analysis of the steel sample results in weight percentage is presented in Table 1. By the American Iron and Steel Institute (AISI) classification of steel, the steel sample used in this study containing 0.0112% carbon falls within the class of low-carbon steel. Low-carbon steel is associated with very low carbon content, less than

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0.10% C [20-23]. Low-carbon steels with a carbon content less than 0.30% are called mild steels [24]. Low-carbon/mild steel contains carbon within the range of 0.05 and 0.25% [25].

Table 1. *Elemental composition of mild steel sample*

Fe	99.700%	**C**	0.0612%	**Mn**	0.1020%
P	0.0222%,	**Si**	0.0052%	**Cr**	0.0342%
Al	0.0134%	**Ni**	0.0015%	**Sn**	0.0053%
Co	0.0159%	**Ti**	0.0002%	**S**	0.0389%

3.2. Phytochemical Analysis

The results of the phytochemical screening of the *Prosopis Africana* pod and watermelon peel extracts are shown in Tables 2 and 3.

Table 2. *Phytochemical analysis result of* Prosopis Africana *pod*

Saponin	108.70	**Alkaloids**	101.60
Tannin	83.80	**Phenol**	9.90
Steroids	7.80	**Flavonoid**	2.10
Cardiac cilycosides		1.06	

Table 3. *Phytochemical analysis result of watermelon peel*

Citrulline	+	**Alkaloid**	+
Flavonoids	+	**Terpenoids**	+
Tannin	-	**Polyphenol**	+
Saponin	+		

It can be deduced from the results in Table 2 that Saponin, Alkaloids, and Tannin are the most abundant constituent in *Prosopis Africana* and are the most powerful components in the pod for inhibiting corrosion, which is in line with the findings in previous studies [2, 13, 26, 27]. For the watermelon peel, out of the constituent tested for, only tannin was found negative while other constituents (citrulline, flavonoids, tannin, saponin, alkaloid, terpenoids, and polyphenol) were present and served as the corrosion inhibiting agents of the extract on mild steel.

3.3. Gravimetric Technique (Weight loss)

The results of corrosion rate values are plotted against the exposure time for the hybrid concentration of inhibitors in different ratios of PAPE and WMPE (100%, 3:1, 1:3, 2:1, and 1:2 in Figures 5-10). The specimens during the electrochemical tests performed using acidic media are observed without corrosion inhibitor (0.0 g/L) corroded at a higher rate if this behaviour is compared against the specimens. Furthermore, as the concentration of the extract increases, the corrosion rate of the test specimen decreases significantly. The reduction in corrosion rate could be ascribed to the phytochemicals adhering to the metal's surface, forming a barrier to the metal's disintegration in the corrosive medium [26].

At 100% PAPE green inhibitor in Figure 5, after 2160 hours, 1.0 g/L recorded 130.05 mpy. Also in Figure 6, similar trends but slightly lower values were observed for 100% of WMPE. Figure 7, PAPE: WMPE ratio 3:1 result shows a drastic decrease in corrosion rate with time exposure up to 2160 hours. However, the 1:3 concentration ratio of PAPE:WMPE in Figure 8 recorded an increase corrosion rate along the exposure time. This shows that the corrosion rate is highly dependent on the concentration of the inhibitor.

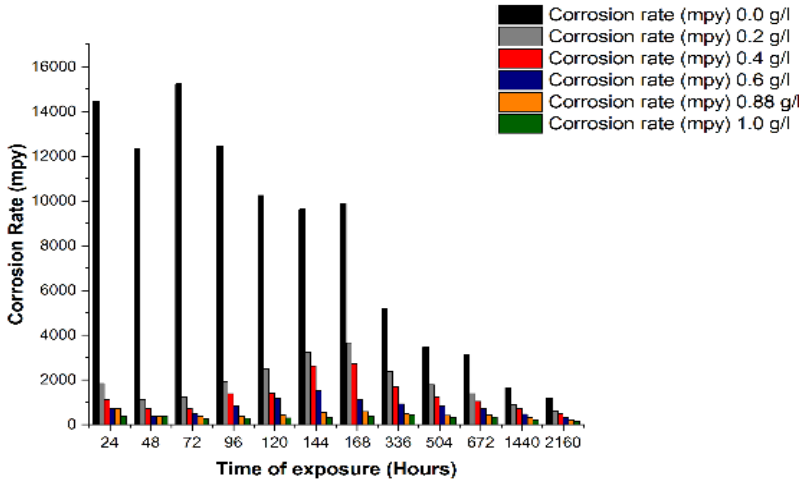

Fig. 4. *Corrosion rate (mpy) for 100% PAPE concentration ratio*

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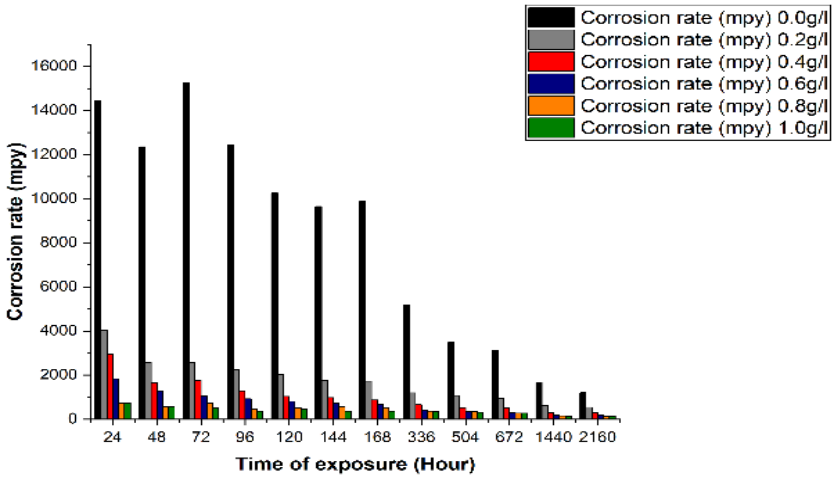

Fig. 5. *Corrosion rate (mpy) for the 100% WMPE concentration ratio*

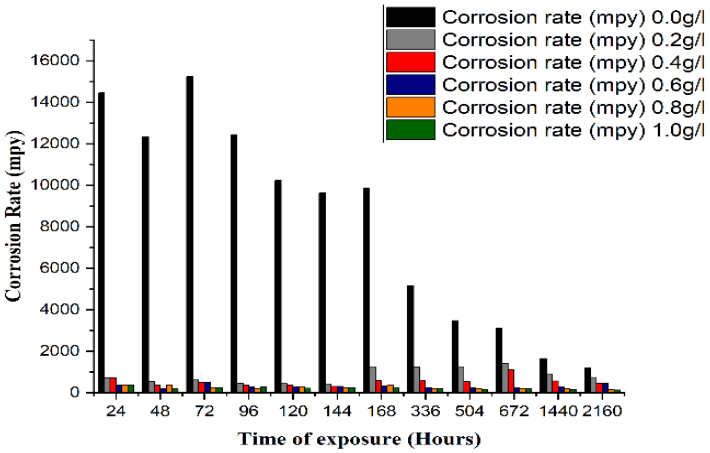

Fig. 6. *Corrosion rate (mpy) for the 3:1 concentration ratio of PAPE:WMPE*

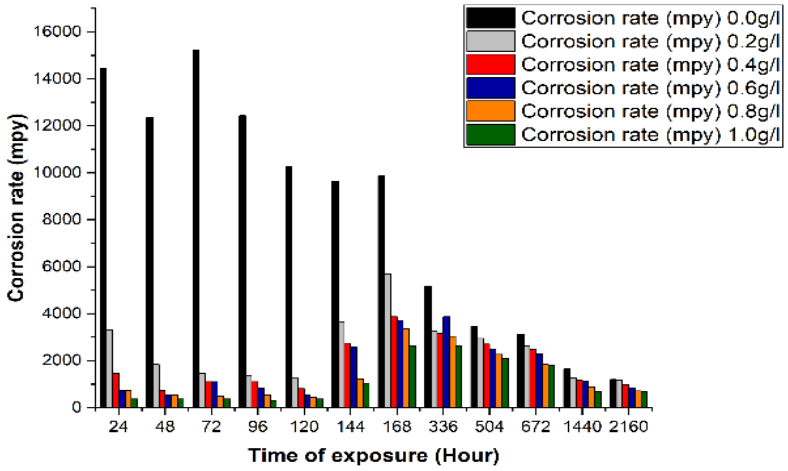

Fig. 7. *Corrosion rate (mpy) for the 1:3 concentration ratio of PAPE:WMPE*

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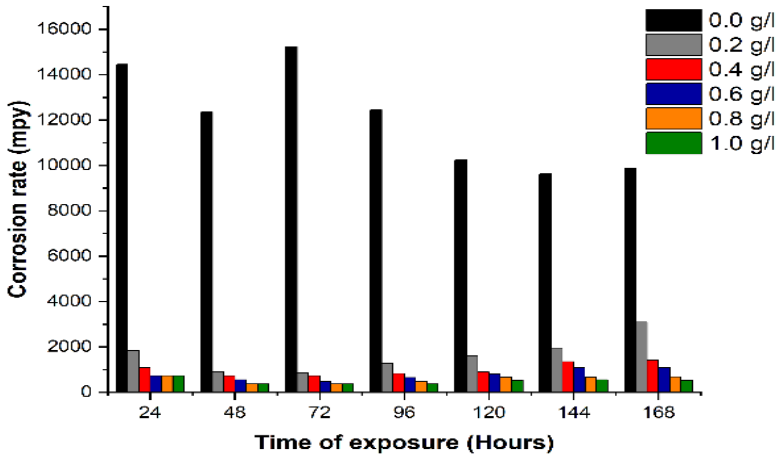

Fig. 8. *Corrosion rate (mpy) for the 2:1 concentration ratio of PAPE:WMPE*

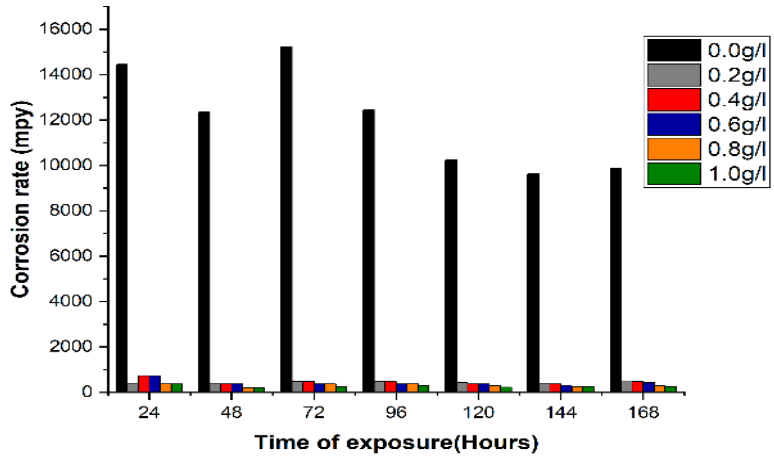

Fig. 9. *Corrosion rate (mpy) for the 1:2 concentration ratio of PAPE:WMPE*

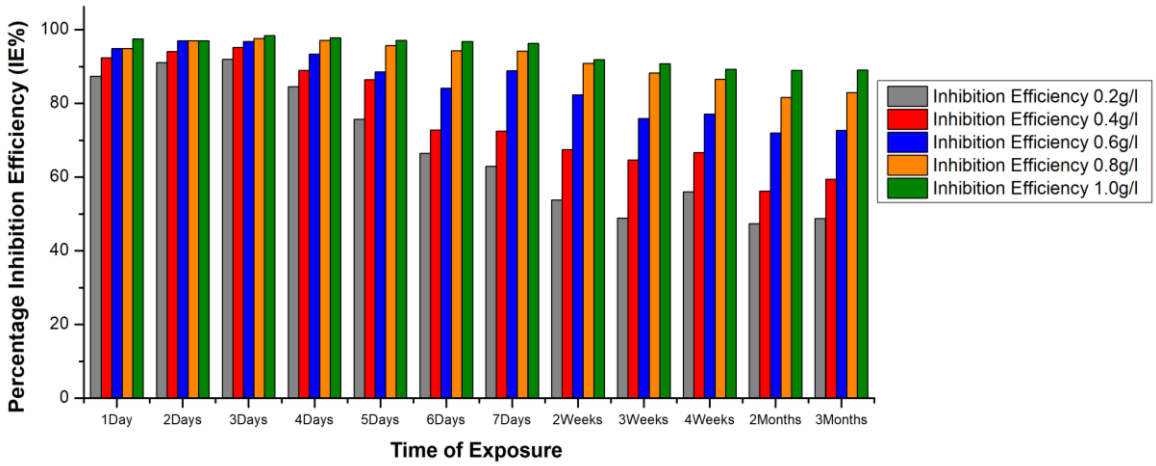

Fig. 10. *Percentage Inhibition Efficiency for the 100% PAPE concentration ratio*

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The trends of inhibitory efficiency (IE%) for hybrid concentrations of PAPE and WMPE are illustrated in Figures 11-16. This was used to examine the extracts' potential for inhibitive behavior. It is shown that increasing the inhibitor concentration increases inhibition efficiency. However, this was not consistent with the period of exposure. The efficiency is attributed to the development of a protective coating due to the change of the metal/solution interface from an active to a passive dissolution state [26].

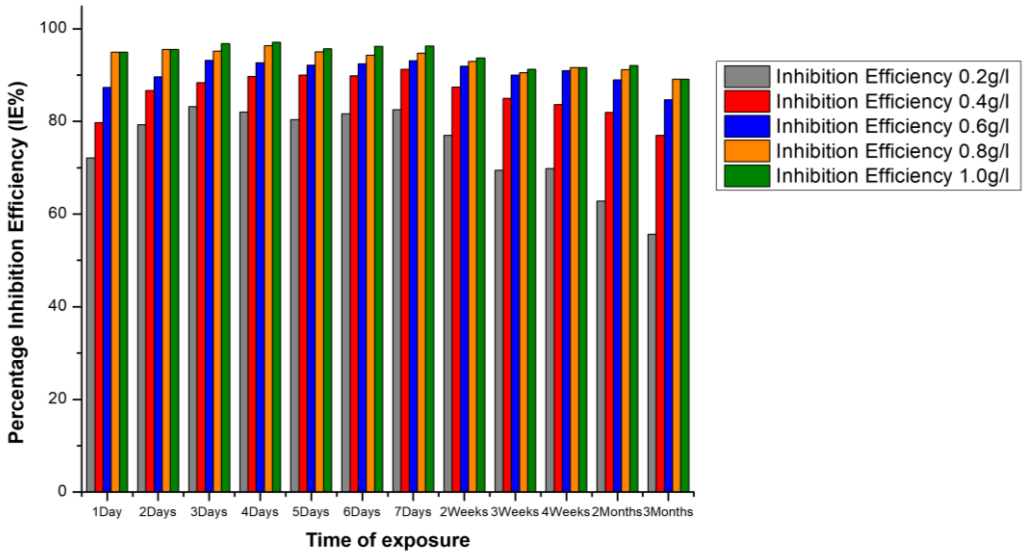

Fig. 11. *Percentage Inhibition Efficiency for the 100% WMPE concentration ratio*

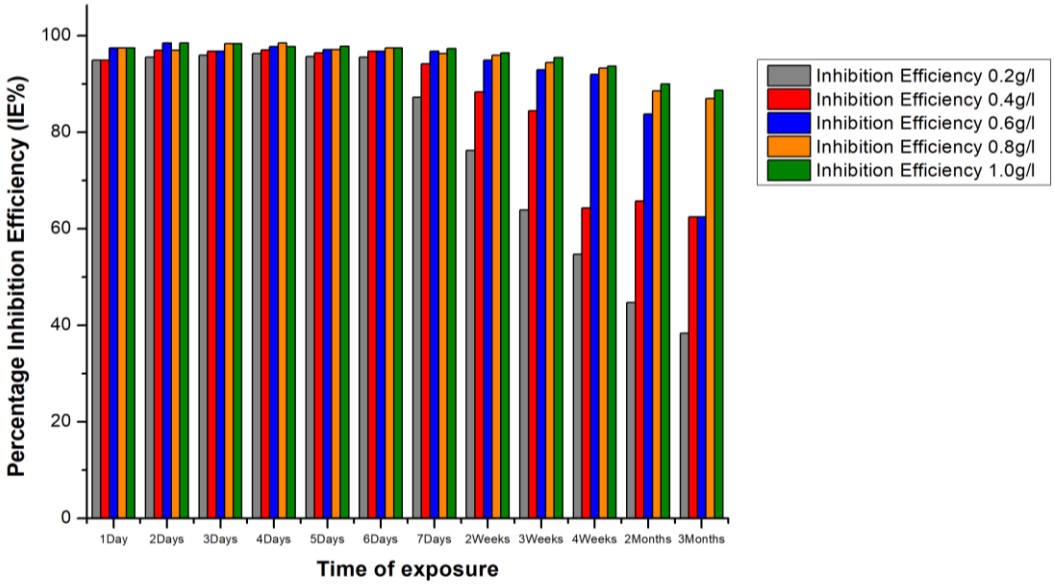

Fig. 12. *Percentage Inhibition Efficiency for the 3:1 concentration ratio of PAPE:WMPE*

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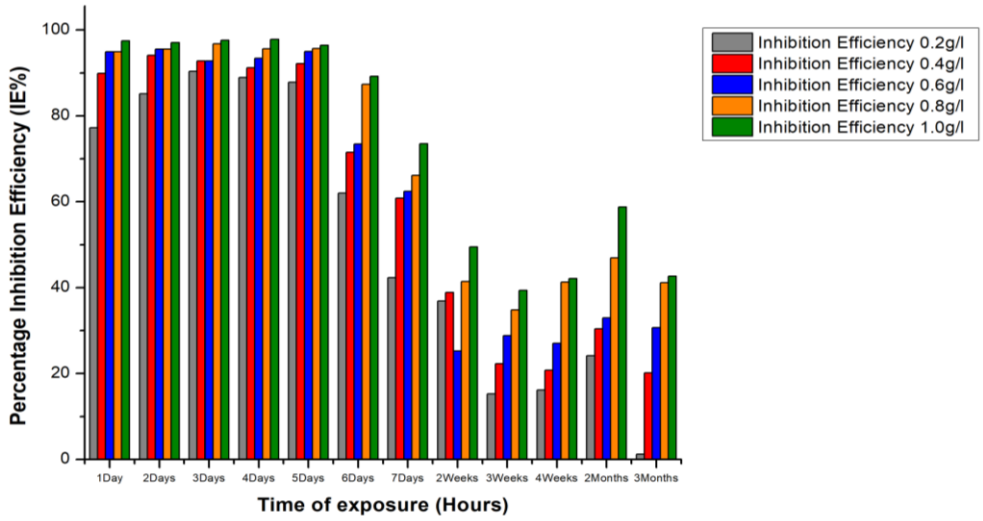

Fig. 13. *Percentage Inhibition Efficiency for the 1:3 concentration ratio of PAPE:WMPE*

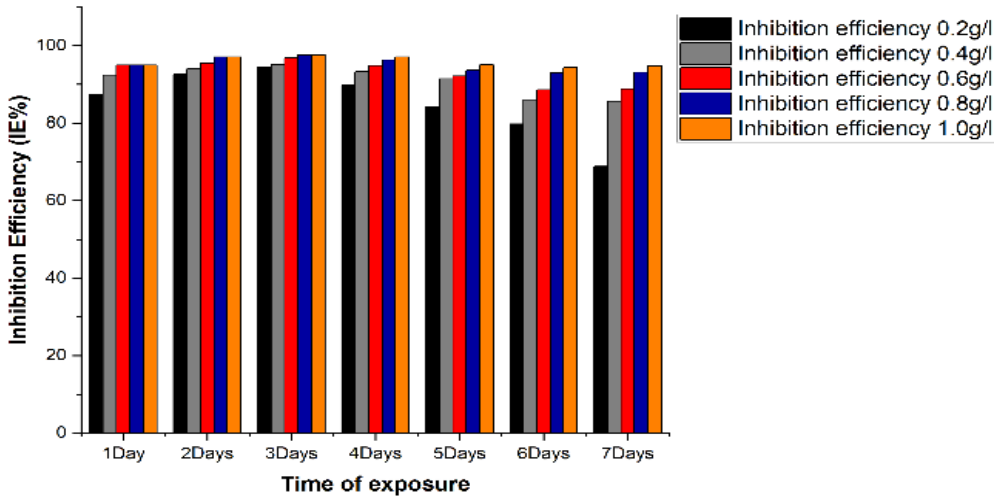

Fig. 14. *Percentage Inhibition Efficiency for the 2:1 concentration ratio of PAPE:WMPE*

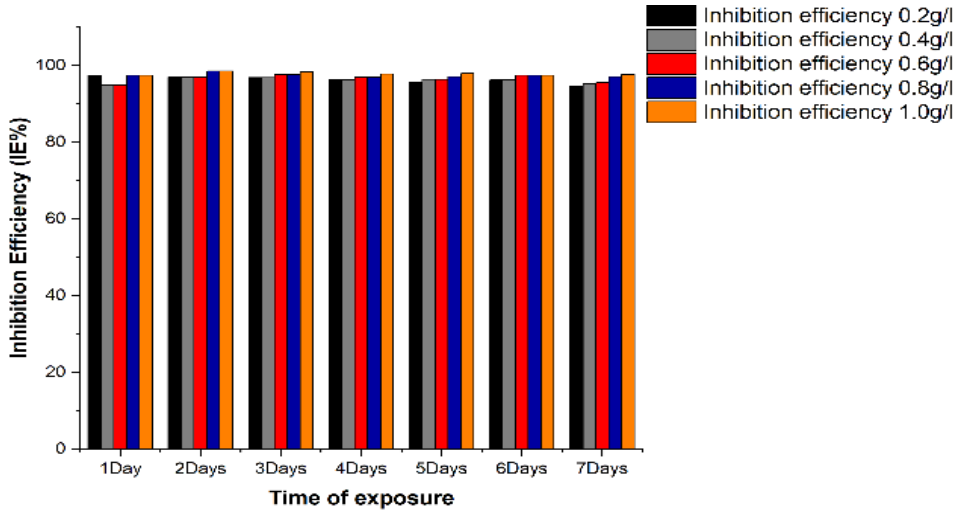

Fig. 15. *Percentage Inhibition Efficiency for the 1:2 concentration ratio of PAPE:WMPE*

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Through the gravimetric measurement technique, the peak inhibition efficiency value of 96.7% was obtained at the concentration of 1.0 g/L with 3:1 PAPE:WAPE, which is above the inhibition value obtained with PAPE (93.7%) and WMPE (92.7%).

3.3. Hydrogen evolution measurement

The volume of hydrogen gas evolved in the presence and absence of different concentrations of the hybrid extracts that are presented in Figures 16-20. The results showed that the volume of evolved hydrogen gas increases with time but decreases with an increase in the concentration of the extracts. For example, after 300 minutes of observation, the highest and lowest reading for evolved hydrogen was recorded for PAPE, WMPE, 3:1 of PAPE: WMPE and 1:3 of PAPE:WMPE in 0.0 g/L (27.00 for all considerations) and 1.0 g/L (5.20, 7.10, 4.10 and 4.90 respectively). The result affirms the view of Odusote *et al.* [26] that the evolution rate of gas decreases as the concentration increases. This may be a result of the formation of a passive layer on the surface of the metal in the extract inhibitor.

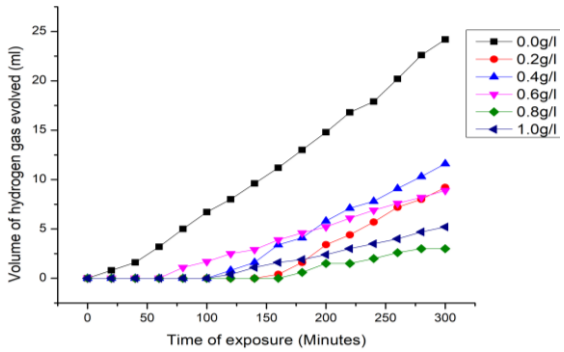

Fig. 16. *Rate of evolution of hydrogen gas in 1 M HCl at different concentrations of the 100% PAPE*

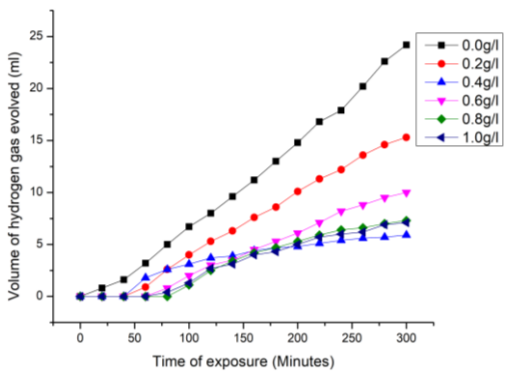

Fig. 17. *Rate of evolution of hydrogen gas in 1 M HCl at different concentrations of the 100% WMPE*

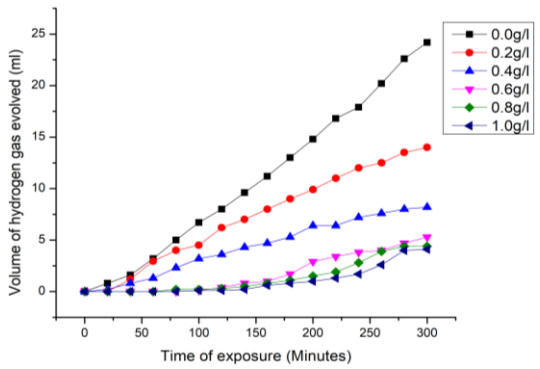

Fig. 18. *Rate of evolution of hydrogen gas in 1 M HCl at different concentrations of the 3:1 hybrid extract*

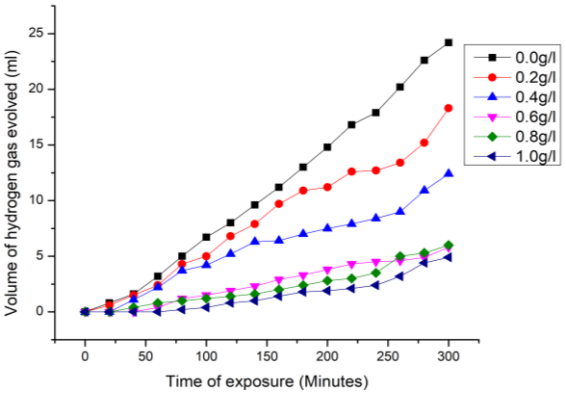

Fig. 19. *Rate of evolution of hydrogen gas in 1 M HCl at different concentrations of the 1:3 hybrid extract*

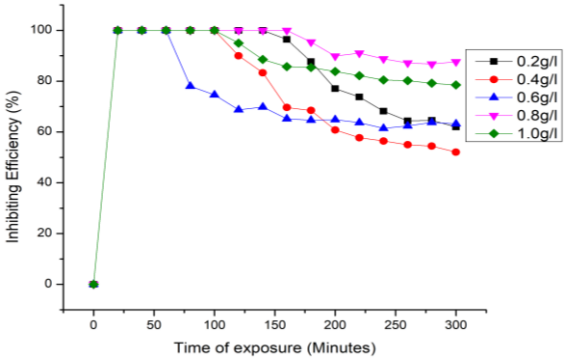

Fig. 20. *Variation of inhibition efficiency with time of exposure at different concentrations of 100% PAPE*

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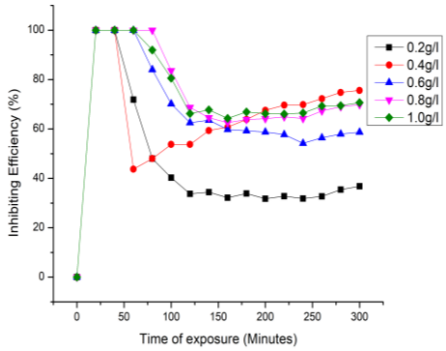

Fig. 21. *Variation of inhibition efficiency with time of exposure at different concentrations of 100% WMPE*

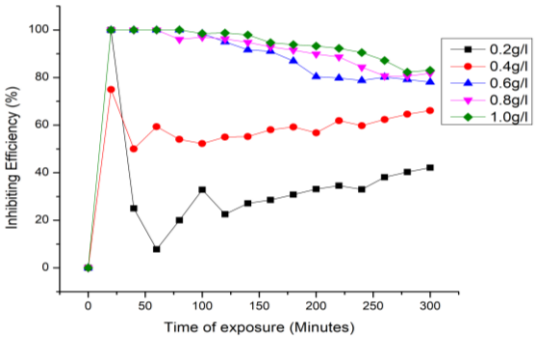

Fig. 22. *Variation of inhibition efficiency with time of exposure at different concentrations of 3:1 Hybrid*

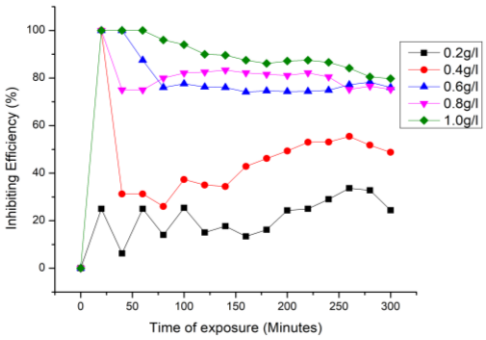

Fig. 23. *Variation of inhibition efficiency with time of exposure at different concentrations of 1:3 Hybrid*

Figures 21-24 show the results obtained by the variation of percentage inhibition efficiency with the time of exposure. The results show that when the concentration of the inhibitors' extracts increases, the inhibition efficiency increases. The extracts' physiochemical components may be responsible for corrosion inhibition. At 1.0 g/L, the maximum inhibitory efficiency (83.1%) was recorded.

3.4. Tafel *polarization*

Figures 24-27 show the curves derived from the *Tafel* polarization measurements of mild steel dissolution in 1 M of HCl in the absence and presence of various amounts of hybrid inhibitors. In the presence of PAPE, WMPE, and hybrid inhibitors, both anodic and cathodic current densities were reduced, indicating a mixed-type inhibitor activity [2, 15]. As the concentration of the extracts increases, the Icorr values are reduced. It can be seen that as the extract concentration rises, the inhibition efficiency rises as well. The influence of the extract on both anodic and cathodic reactions is seen in this result. The highest inhibition efficiency was obtained at 1.0 g/L of the hybrid inhibitors of the ratio of PAPE to WMPE in ratios of 3:1, 1:3, 2:1, and 1:2 which give the result of 98.52%, 80.95%, 82.35% and 97.41% respectively. This implies that the most appropriate ratio in the formulation of an effective hybrid organic inhibitor comprising of PAPE and WMPE was achieved at the ratio 3:1 PAPE:WMPE.

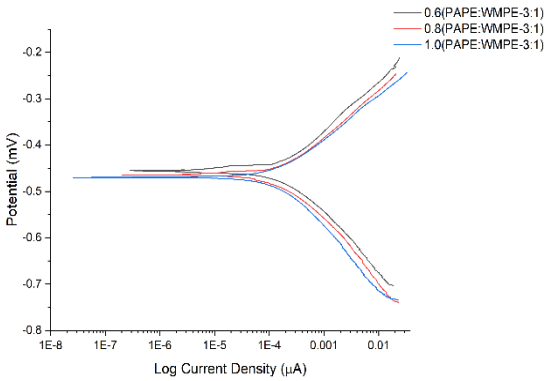

Fig. 24. *Tafel Polarization curves of mild steel in 1 M HCl with and without both PAPE and WMPE Inhibitor in ratio of 3:1*

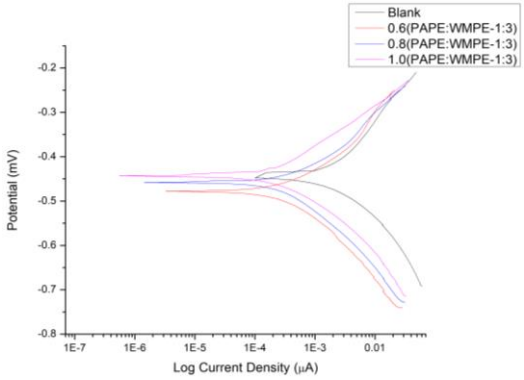

Fig. 25. *Tafel Polarization curves of mild steel in 1 M HCl with and without both PAPE and WMPE Inhibitor in ratio of 1:3*

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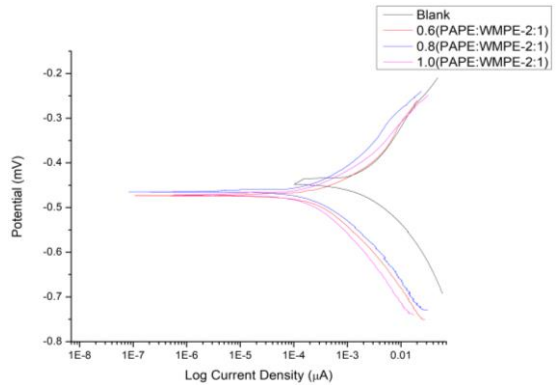

Fig. 26. *Tafel Polarization curves of mild steel in 1 M HCl with and without both PAPE and WMPE Inhibitor in ratio of 2:1*

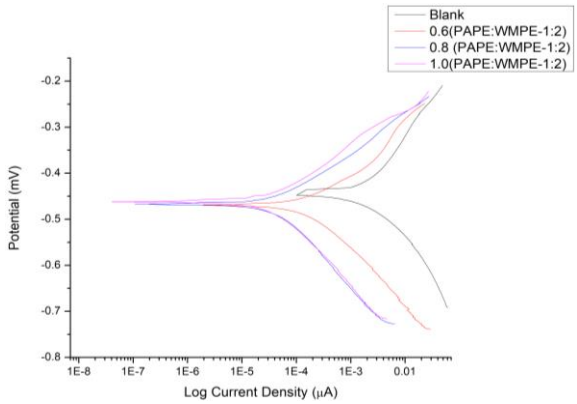

Fig. 27. *Tafel Polarization curves of mild steel in 1 M HCl with and without both PAPE and WMPE Inhibitor in ratio of 1:2*

From Figures 24-28 showing the results of PAPE and WMPE *Tafel* polarization, it is observed that the corrosion potentials (Ecorr) for the mild steel in the presence of PAPE all shifted toward the negative potentials. As the concentration of the extracts increases, the corrosion current density (Icorr) values decreased. However, the inhibition efficiency value computed from the corrosion current density in the absence (Icorro) and presence (Icorr) of the inhibitor increased. The highest inhibition efficiency of 98.52 % was obtained at 1.0 g/L concentration. The shift in the *Tafel* slopes of both the cathodic reaction (βc) and anodic reaction (βa) as shown in Figures 24-28 in the presence and absence of the extract suggests that the inhibitor affects both the cathodic and anodic reactions. This also implies that the hybrid extract is a mixed inhibitor. Some of the components included in the examined extracts may be protonated in the HCl solution, and these protonated species may adsorb directly on the mild steel surface's cathodic sites. According to Odusote *et al.* (2016), the inhibitory mechanism was activated by simply blocking the available cathodic and anodic sites on the metal surface.

4. Conclusions

The following conclusions were drawn from the investigation into the inhibition of mild steel corrosion in hydrochloric acid solution by hybridizing Prosopis Africana pod and watermelon peel extract using weight loss measurement, hydrogen evolution measurement, and *Tafel* polarization techniques:

i. The corrosion rate of the mild steel in the HCl solution was found to decrease with an increase in the concentration of the entire ratio considered.
ii. The inhibiting efficiency of the entire ratio considered is dependent on the concentration of the extract and it increased with the increase of concentration of the extract in the acidic medium irrespective of the corrosion measurement technique used.
iii. All ratio hybrid inhibitor extracts could serve as an effective inhibitor of corrosion of mild steel in hydrochloric acid solution. Meanwhile, the hybrid of the two green inhibitors of a ratio of 3:1 PAPE: WMPE displayed the best IE% (98.52%), followed by a hybrid ratio 1:3 with IE% of 97.79%, at a 1.0 g/L concentration after exposure in the 1M HCl medium for mild steel.
iv. The *Tafel* polarization technique results showed that extracts of each ratio hybrid inhibitor products acted as mixed-typed inhibitors via simple adsorption of the phytochemicals present in the extract on the mild steel surface in HCl solution, thereby influencing both cathodic and anodic mild steel dissolution reactions as revealed by the *Tafel* polarization measurements.

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