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ORIGINAL RESEARCH PAPER

Extracts of leaf as green substitute to zinc compound additives in alkyd primer for corrosion inhibition of mild steel

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ABSTRACT

Application of leaf extracts in alkyd extract primer (AEP) to inhibit corrosion of mild steel was compared with performance of alkyd conventional primer (ACP) containing zinc phosphate and zinc chromate as inhibitors. The investigations were carried out through Gasometric technique in 5 °C steps of temperature increase from 25 to 50 °C in 1.0 M HCl as corrodent. The AEP of 34.24 percent actives compared with ACP of 56.57 percent actives gave the same inhibition efficiencies of 76.5%. Extract primer of lower percent active compared with conventional primer were cost effective and more potent than conventional primer.

KEYWORDS

mixed plant leaf extracts, alkyd extract primer, alkyd convectional primer, *Jatropha curas*, *Sida acuta*, mild steel

1. INTRODUCTION

The act of resistance to corrosion by non-contaminated metals are usually better than those having other elements or impurities in their constituents. Apart from been more costly pure metals are of characteristics that does not align with technical requirements [1]. Purely refined metals from its ore are unstable, due to their tendency to react available substances in their environment. The product of such reactions is rust which leads to depreciation of aesthetics and economical value of the metal [2]. It is disadvantageous to the operations of manufacturing and construction industries. In order to stabilize it, there are need for addition of other elements in the form of substances purposely embedded as alloy constituents therefore to fortify for the intended use [3]. It is inevitable to naturally avoid material containing high proportion of iron from corroding. These are prevented by application of appropriate inhibitors based on the corrosion phenomenon in process. It is the industrial maintenance culture practiced in manufacturing industries to allay system failure at unexpected. The corrosion inhibitor requires coating formulations in consonance with the mechanism of the corrosion process in a particular environment [4]. The working principle of an effective inhibitor is to either break the circuit of reaction pathway or supply electron which ought to come from the anode to the cathode of unit cell [5]. The use of coating materials which adhered to the metal surface as an insulator impedes or electrolytically resist corrosion processes aided by corrodent present in the contact fluid [6].

Research works have confirmed extracts from natural plants inhibit corrosion of metals. Investigations are ongoing on extracts of some local plants continually for their suitability as better substitute to toxic inhibitor and dangerous chemicals to handle [7].

Sida acuta (SA) is a plant from the Malvaceae family of origin traced to Central part of America that extended over to the warm tropics. It was used in this region and some other regions of the world to treat headaches and colds which are the symptoms of fever. This plant

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can be added to other native substances to detoxicate a bite of snake as being practiced in countries like Colombia and Gambia, and to apply for disinfection of skin disease of domestic animals. Its stem and leaf have been reported as treatment of whitlow in the rural regions of Nigeria [8]. The leaves on some occasions are mixed with other native concoctions and eaten with food for the purpose of neutralising of gunshot bullets in human body in Nigeria. Asthma and renal inflammation were also treated as well as ulcer and intestinal worms with the plant leaves [9].

Jatropha curcas (JC) plant is propagated by stem and seed. The stem functions as leguminous crops in Gabon – a West African country. Fruits are dried and further processed into powder and taken with food to reduce excessive fat [10].

This work investigated the effect of *Jatropha curcas* and *Sida acuta* mixed extract incorporated into alkyd primer, compared with primers containing chromate and phosphate of zinc as inhibitive agents.

The phytoconstituents in the plant extracts contained heteroatoms in varied proportions. They are adsorption sites by which the heteroatoms in the inhibitor got adsorbed to the metal surface for corrosion inhibition to be effected. These can therefore be used as substitute to the toxic inorganic inhibitive agents.

The total phytoconstituents in SA was 5.818% while that of JC was 1.28% [11, 6, 12, 13, 14]. Some of the conventional inhibitive agents contain ionic materials of high potency in some cases. The oxidising potential of Cr^{6+} in the ionic specie $(CrO_4)^{2-}$ contributed to the potency of inhibition of the primer. The primer at pH above 5, the Cr (VI) underwent reduction to Cr(III) per mole of zinc chromate, a process at the anodic sites in which two electrons released from Fe that oxidized it to Fe^{2+} were used to reduce the chromium ion [15, 16]. The combined application of zinc phosphate as a soluble anticorrosive pigment with chromate in this batch aimed at maintaining the activeness of the primer at most pH including acidic region of less than 5 [17, 18].

Coating of metals with primers is one of the methods to inhibit or hinder the already corroding process from advancement [19] Morgans1990. Primers are produced by mixing resin as an active binding medium with other required materials. Resins are formed from specific polymerization process to yield epoxy, alkyd, 2 pack polyurethane or other form of resins. They bind other components in the recipe in solution or paste. Resins are vehicular media that bind other components of primer.

Primers are classified by their constituents. They act as corrosion barrier on metals to hinder the ongoing corrosion process [19]. Primers earlier known are characteristically metals: gold, silver, aluminum, tin and zinc. They have been developed recently from mixture of various compounds such as oxide of iron (Fe_2O_3), chromate and phosphate of zinc ($ZnCrO_4$ or $Zn_3(PO_4)_2$.

The inhibitive additives singly or in a combined form are incorporated into polymeric binders considering the activeness of the inhibitor in relation to the potency required for the primer product. Zinc phosphate a non-toxic and whitely coloured compound has been used for protecting steel by the mechanism of phosphate ions presence for phosphating the moistened steel surface. Zinc chromate is highly inhibitive and impacts yellow colour in the medium but is carcinogenic and toxic to humans. The combination of both zinc materials to form a compound made it effective in several media of wide range of pH [20, 6, 21].

This study investigated the effectiveness of the combined leaf extracts of *Sida acuta* (SA) and *Jatropha curcas* (JC) in alkyd extract primer (AEP) as corrosion inhibitor in comparison with alkyd conventional primer (ACP) that contained zinc compounds of chromate and phosphate as conventional inhibitors. The leaf extract formed part of the recipe constituents by substitution of conventional inhibition additives in the primer for inhibition improvement and environmental greenness.

2. EXPERIMENTAL WORK

2.1. Materials used

Mild steel plate of 3.0 mm thickness was cut into coupons of 18 × 9 mm using metal slicer. The coupons were degreased with ethanol and kept in desiccator [22].

The elemental constituents' analysis of the sample was done for this investigation using Metal Spectrometer Analyser (Optical emission) (Table 1).

2.2. Measurements

2.2.1. Plant extraction. Leaves of JC and SA were processed into powder. 100 g of the powdered leaves were separately measured into conical flask of 2000 mL volume that already contained 1,000 mL ethanol. The flask was well covered and left for 2 days with periodic agitations [23]. The resultant mixture was filtered and extract was obtained from the concentration of filtrate by retrieval of the solvent on rotatory evaporator. The molten extract obtained was evaporated to oily solid on a water bath by heating at 75 °C to expel the remaining ethanol [24].

Table 1. Elemental constituents (%) of mild steel

Element	%
C	0.1551
S	0.0445
P	0.0280
V	0.0169
Cr	0.0626
Mn	0.5571
Fe	98.420
Co	0.0463
Ni	0.1499
Cu	0.0792
Mo	0.0106
Si	0.2378
Nb	0.0823

The resultant extract was characterized using Fourier-transform infrared [FTIR] spectroscopic method. By this analysis, functional groups present in the extract sample were determined.

The two extracts obtained earlier were added together in ratio 1:3 (SA:JC) and then substituted for the two zinc compounds. Alkyd extract primers each containing resultant extract in series of 4.26 g, 5.33 g, 6.39 g, 7.46 and 8.52 g obtained by 25% increase in weight steps: Each was then reduced by 50% to form the recipe below (Table 2):

2.2.2. Coupons coating procedure. Each coupons were coated with the AEP and ACP primers as described by Sodiya and Dawodu 2021.

2.3. Gasometric corrosion test

The coated samples of AEP, ACP and uncoated (control) were subjected to gasometric corrosion test as described by [23] Sodiya and Dawodu 2021 [24] Sodiya et al 2016 [11].

Evolved gases were noted every hours for 8 h. It was repeated for 30 °C. 35, 40, 45 and 50 °C in 1.0 M Hydrochloric acid. The experiments were repeated with alkyd conventional primer coated coupon as control.

Table 2. Primer recipe

Materials	wt(g) ACP	wt(g) AEP
Resin (Alkyd)	26.18	26.18
Dichromate Zinc	1.06	–
Phosphate Zinc	1.07	–
Mixed SA & JC	–	2.13
Talc (Magnesium Silicate)	4.84	4.84
CaCO3 (Calcium Carbonate)	4.20	4.20
Methylbenzene	11.56	11.56
Antiskinning agent	0.12	0.12
Drier	0.97	0.97
Total	*50 g*	*50 g*

The plant extract Inhibition Efficiency (I.E.) was determined through Eq. (1) below, [25].

$$\%\ \text{I. E.} = 1 - \frac{V_{inh}}{V_{uninh}}\ \text{x}\ 100 \qquad (1)$$

V_{in} = Volume of hydrogen gas given out from inhibited solution and
V_{uni} = Volume of hydrogen gas given out from uninhibited solution both at time *t*.

$$\text{Surfaced coverage}\ (\theta) = \frac{\%\ I.\ E.}{100} \qquad (2)$$

3. RESULTS AND DISCUSSIONS

3.1. Fourier-transform infrared [FTIR] analysis

FTIR analysis revealed some specific moieties in molecules of compounds present in the combined plant extract in Fig. 1., such as O–H of phenol at 3,420 cm^{-1}, The band at 1,663 cm^{-1} is an indication of stretching vibrational frequency ascribed to C=O of ketone.

The presence of N=O in the extract was inferred from weak bands at 1,458, 1,404 and 1,378 cm^{-1} noted on the spectrum.

While the weak band at 719 cm^{-1} was ascribed to imines and oximes species.

Reports have already been established that the detected heteroatoms contained in the functional groups present in the extract possess structures which are binding centres from the inhibitors with metals [26, 27, 28].

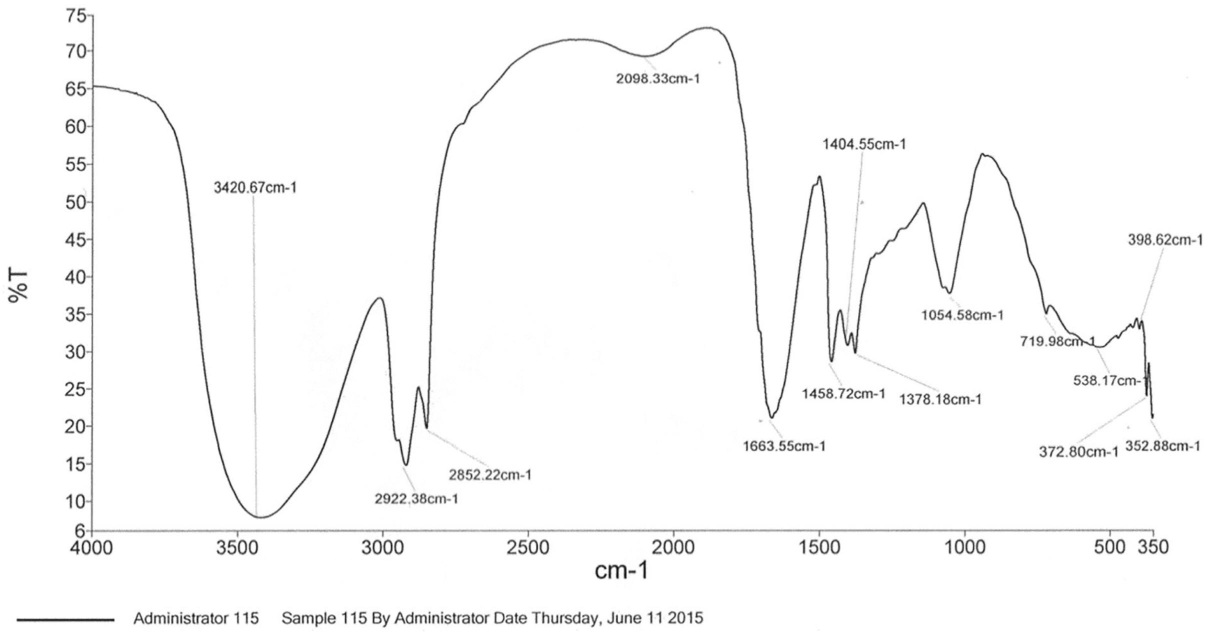

Fig. 1. Spectrum of leaf extract

3.2. Corrosion rate (mL cm^{-2} hr^{-1})

$$\text{From } R\ \alpha\ \frac{\partial \Delta W_m}{\partial t}\ \alpha\ \frac{\partial \Delta V_{H2}}{\partial t}\ \left(mg/\ cm^2hr^{-1}\right)\ \alpha\ \left(ml/cm^2hr^{-1}\right) \tag{3}$$

Regression analysis of polynomials was applied to deduce volume V of H_2 gas evolved at time t [29].

$$V_{in} = l \pm kt \pm jt^2 \tag{4}$$

Differentiating Eq. (4) gave $R = \frac{\partial v}{\partial t} = b + 2at$

$$\text{Rewritten as } R = 2jt \pm k \tag{5}$$

Therefore, the mild steel in corrodent, conventional and extract primers, indicated in Eqs (4) and (5) were employed to obtain corrosion rate R. Where x = t

i) HCl acid (corodent) $y = 0.144x^2 + 1.842x + 1.033$; $\frac{\partial v}{\partial t} = 0.288x + 1.842$;
ii) Alkyd convectional primer $y = -0.033x^2 + 0.513x + 0.191$; $\frac{\partial v}{\partial t} = -0.066x + 0.513$
iii) Alkyd extract primer coated steel $y = -0.016x^2 + 0.412x + 0.16$; $\frac{\partial v}{\partial t} = -0.032x + 0.412$

3.3. Extract weight increase effect on inhibition efficiencies

In Fig. 2, the Percentage Inhibition Efficiency of ACP that contained conventional inhibitive agents was constant at 76.5%. Successive weight increase of plant extracts as components primer samples resulted in increase in their respective % I.E. at 30 °C. The I.E. of both ACP and AEP

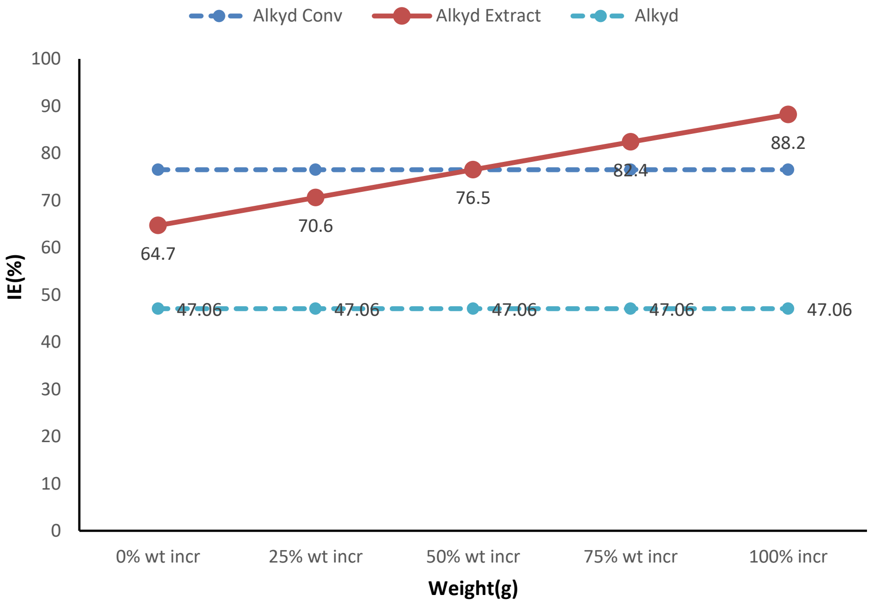

Fig. 2. Graphs of % I.E. of primed steel with AEP, ACP and primer without inhibitor in corrodent at 30 °C versus various extract weights

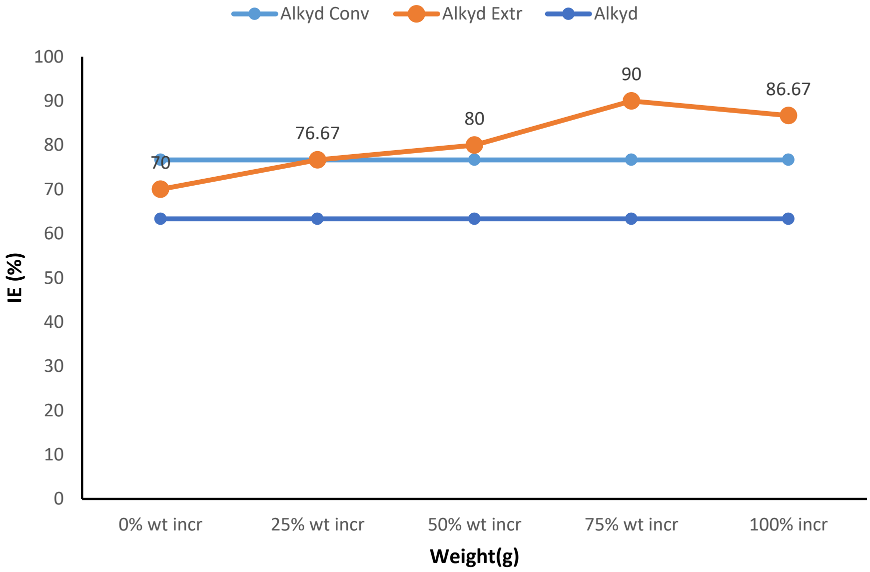

Fig. 3. Graphs of % I.E. of primed steel with AEP, ACP and primer without inhibitor in corrodent versus various extract weights at 50 °C

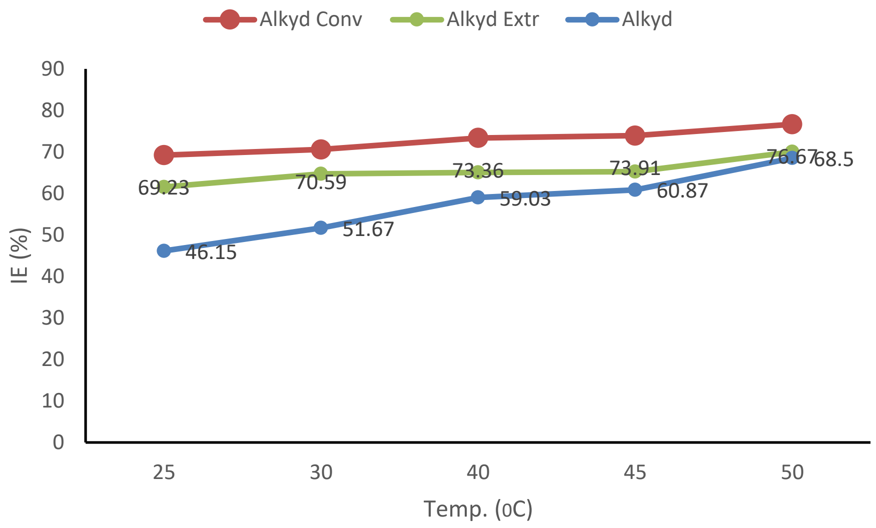

Fig. 4. Graphs of % I.E. of primed steel with AEP, ACP and primer without inhibitor in corrodent versus various extract weights at 50 °C at various temperatures of equivalent weight

were the same at 76.5 of 50% weight increase in AEP. At double extract weight 100% increase (8.53 g) in alkyd primer gave I.E. of 88.2% which was higher compared with 76.5% of ACP.

The % I.E. alkyd primers without any inhibitive agent, be it extract or conventional, remained constant at 47.06%. In this case the C=O of ketone group in the gyptal resin have both σ sigma and л bonds, thus implies richness in л electrons that tends to reside more on the oxygen atom. These were available for donation into the empty orbital of the metal Fe^{2+} for adsorption.

At 50 °C in Fig. 3, the increase in temperature lead to desorption of primers from the mild steel. This, however, made the % I.E. to be lower in values at corresponding weights compared with the primers characteristics at 30 °C in Fig. 2.

I.E. of 76.67 and 63.33% were respectively attained for ACP with 4.26g of conventional inhibitor and alkyd primer batch without inhibitor at temperature of 50 °C.

At that same 4.26g of extract weight in primer (AEP), an optimum I.E. of 90% was achieved at 75% weight, further increase of extract weight led to reduction in efficiency at that same temperature of 50 °C. At attainment of 63.33% by alkyd resin without inhibitors, no increase in I.E was obtained.

In Fig. 4, the primers' I.E. % increased with increase in temperature at the same weight content. ACP have higher corrosion inhibition efficiencies over AEP at all temperatures.

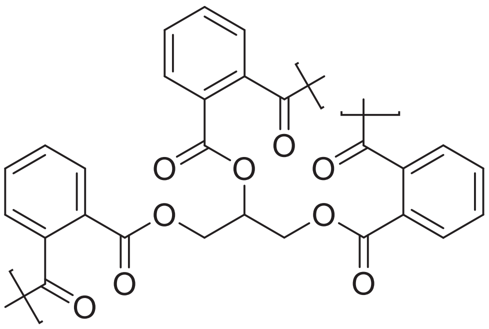

Fig. 5. Alkyd chemical structure

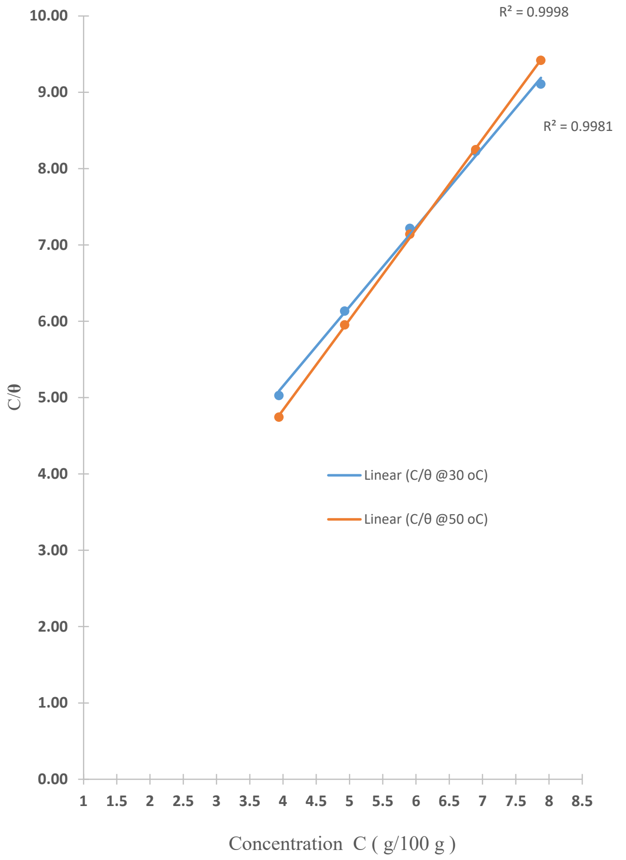

Fig. 6. Langmuir isotherm graphs of C/θ versus concentration C (g 100 g^{-1}) of AEP at 50% extract weight increase (6.39 g) in primer adsorbed on mild steel at 30 and 50 °C in corrodent

The primer without any form of inhibitor was lowest. This implies that alkyd extract primer was not as efficient as its conventional counterpart under certain conditions.

The combination of *JC* and *SA* plant extracts utilized in this study as anti-corrosion additives in the alkyd extract primer consisted of several organic compounds of plant origin [30]. The plants contain, among others, tannins, flavonoids, alkaloids and saponins which have heteroatoms at which electrons can be donated in their structures. Tannins which are esters of aliphatic acids in combination with that of phenolic acids or oligomers were water-soluble compounds. Flavonoids are polyphenolic molecules and polyhydroxyflavan-3-ol units of molecular weights that range between 500 and 3,000 [31, 32]. Alkaloids are compounds of organic origin of nitrogen-containing bases. They can be quinine, morphine, nicotine, ephedrine, and strychnine [33, 34]. Saponins are triterpene glycosides characterized by bitter taste and soap-like foam when agitated in water in aqueous solutions in general. They structurally contain both hydrophilic glycone species with lipophilic triterpene or steroid derivative.

Alkyd is an esterification product of polybasic acids and polyhydric alcohols that contain a monobasic fatty acid in its molecule as indicated in Fig. 5.

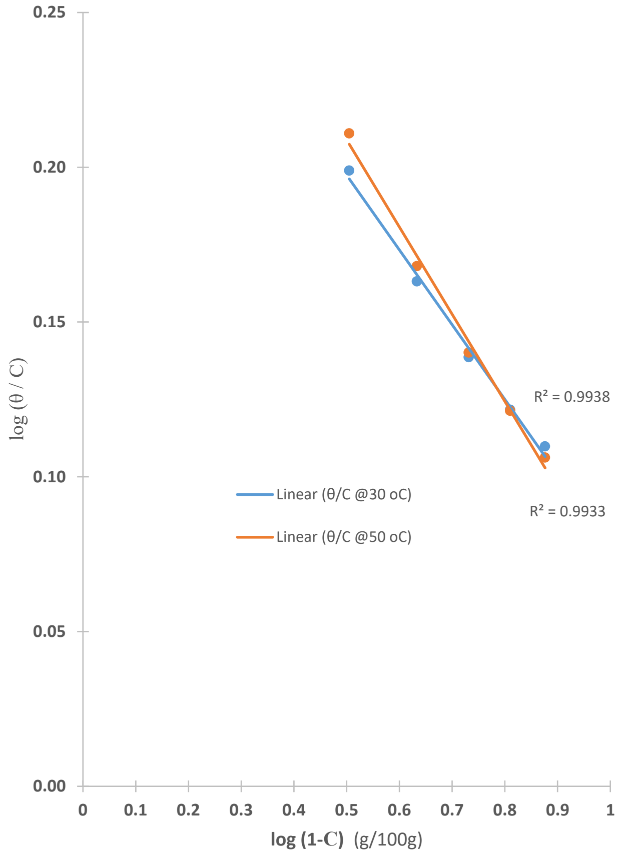

Fig. 7. Florry-Huggins isotherm graphs of log θ/C versus log (1–C) (g 100 g^{-1}) of AEP at 50% extract weight increase (6.39 g) in primer adsorbed on mild steel at 30 and 50 °C in corrodent

3.4. Primer adsorption isotherms

The effectiveness of zinc compounds combination of chromate and phosphate combination as anti-corrosive agent in primers is based on the ionic characteristics of the compounds. These compounds chemically adsorbed unto the surface of the mild steel.

Adsorption isotherms of Langmuir and Florry-Huggins plotted in Fig. 6 at 30 °C and Fig. 7 at 50 °C respectively for alkyd extract primer were the best fitted for the adsorption of primer on the mild steel. The correlation coefficients (R^2) were deduced from the plot of concentration C and surface coverage θ relationships for five (5) different adsorption isotherms. These were considered at the % I.E. of ACP = AEP = 76.5% at extract weight of 6.39 g in primer.

It was deduced on Table 3, that Langmuir isotherm best explains the adsorption of primer on the mild steel at 50 °C with R^2 0.999. This depicted that physisorption took

Table 3. Adsorption isotherms of AEP when its % I.E. equals ACP

			AEP ←R^2 →	
	Isotherms	Relationship plots	30 °C	50 °C
1	Langmuir	C/θ versus C	0.998	0.999
2	Freundlich	log θ versus log C	0.989	0.945
3	Frumkin	ln{θ/C (1–θ)} versus θ	0.114 0.512	0.280 0.879
4	Temkin	θ versus log C	0.247	0.945
5	Florry-Huggins	log θ/C versus (1–θ)	0.993	0.993

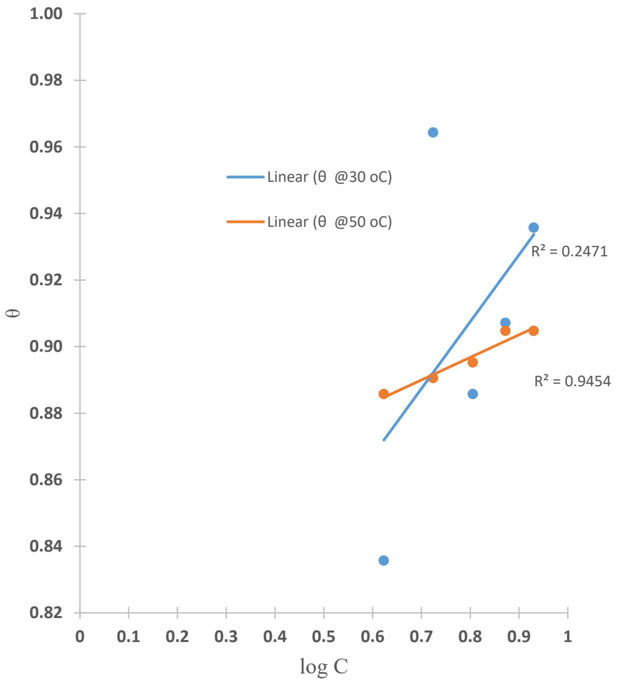

Fig. 8. Temkin isotherm graphs of θ versus log C for (g 100 g^{-1}) of AEP at 50% extract weight increase in (6.39 g) primer adsorbed on mild steel at 30 and 50 °C in corrodent

place predominantly. Florry-Huggins isotherm at 30 °C with R^2 0.993 in AEP gave its contributory roles to the adsorptions. Temkin adsorption isotherm, which is synonymous to chemisorption, also shows some degree of activities in AEP with R^2 0.945 at 50 °C also indicated in Fig. 8 as their Determination Coefficient (R) significantly tends to 1.0.

4. CONCLUSION

From this research of conventional inhibitors substitution with leaf extract to formulated extracts primers, the following conclusions were made on the inhibition characteristics of the extract primer:

i) Inhibition efficiency of alkyd primers increased with the increase in temperature due to both chemisorption and physisorption of the primer that concurrently adsorbed on the mildsteel.
ii) The weight amount 2.188g of extract required to form alkyd primer that gave same I.E. of 76.5% is less than 2.41g of conventional additives required.
iii) The alkyd extract primer of 34.24 percent active and conventional primer of 56.57 percent active gave the same 76.5% inhibition efficiency. With this, the alkyd extract primer AEP was of higher inhibitory effectiveness compared with alkyd conventional primers ACP at at temperature below 30 °C.
iv) The substitution of polarised additives in primers with plant leaf extract eliminated toxicity potential of the conventional inhibitors to environment and also available at reduced cost.

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