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Investigation of the Inhibitive Properties of Bio-Inspired Starch-Polyvinyl Acetate Graft Copolymer (Ps-Pvagc) on the Acid Corrosion of Mild Steel

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Abstract: *Anticorrosive properties of potato and corn starch, potato starch-polyvinyl acetate graft copolymer (PS-PVAGC) and modified biopolymer. PS-PVAGC was prepared by grafting potato starch into PVA using a crosslinking agent. The modified biopolymer was synthesized by blending starch with NaOH and borax. The weight loss method was used to test the inhibitors on mild steel in 1.0 M HCl at room temperature, the observed changes in functional groups of the inhibitors during the entire experiment were monitored using the FTIR method. The results obtained showed that within 2 hours and a concentration of 4 g of the inhibitor, corn starch produced a very low inhibitor efficiency (IE) of 38.52%, while potato starch yielded a maximum IE of 79.81%. The modified biopolymer performed better with a maximum IE of 87.90% while PS-PVAGC performed best with a maximum IE of 92.5%. The inhibitors all reduced the corrosion rates as their concentrations were increased; however, the poor performance of pure starch can be attributed to its poor solubility in the acid solution and poor adhesive power on the metal surface. All formulations showed OH broad peaks (between 3446cm^{-1}-3287 cm^{-1}) as the major group offering heteroatom (i.e oxygen) for the adsorption of the inhibitor and subsequent suppression of the corrosion of the metal.*

Keywords: *Biopolymer; inhibition efficiency; corrosion rate; graft copolymer*

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1.0 Introduction

Metals are valuable materials in most industrial installations because of their durability, conductivity, mechanical strength and other properties (Eddy *et al*., 2023). However, most metals are prone to corrosion attack, which is an electrochemical process that tends to revert the metal to its natural or original form (Eddy *et al*., 2008, 2010). The corrosion of metals can

be activated in the presence of oxygen, moisture, dissolved gases and aggressive solutions (Kristy *et al*., 2021; Rastogi *et al*., 2017; Marassi *et al*., 2018).

The negative impacts of corrosion in several countries have been widely documented, especially when referring to economic cost. For example, the economic cost of corrosion in the United States is reflected in over 3.4% gross domestic product of the country equivalent to US$2.5 trillion year per year (Dariva and Galio, 2014). Indirect impacts of corrosion may include product contamination, liquid or gaseous goods leakages from transportation pipelines, fire breakup, the cost of remediation, etc. (Koch, 2017).

Scientists have used several technologies for the protection of metals against corrosion damages, such as cathodic protection, the use of corrosion inhibitors, coatings, galvanization, etc. The employment of substances, that tend to retard the rate of corrosion when present in minute concentration forms the basis for corrosion inhibition chemistry and is one of the most efficient techniques. Several organic extracts are effective corrosion inhibitors but some operational limitations have been confirmed for some. The employment of toxic chemical constituents has also been discouraging, irrespective of the high efficiency associated with most of them. Plant extracts are another group of environmentally friendly corrosion inhibitors but some technical factors have also created some limitations on their overall efficiency. The employment of natural polymers has been seen as a good innovation in the corrosion industries because polymers have large adsorption sites, less toxicity, and other unique properties. Efforts in the applications of gums have been widely documented in the literature (Alhaffar *et al*., 2018). However, their efficiencies are widely seen as relatively low but enhanced investigative scope has revealed that the required efficiency can be redeemed using polymer blends in synergistic combinations. In this trend, biopolymers, synthetic polymers, nanomaterials, and polymer blends have received significant research concerning their effectiveness as corrosion inhibitors (Kaczerewska *et al*., 2018). Generally, organic inhibitors are the most well-known, profitable, and economical corrosion inhibition strategies due to their effectiveness, economy, ecology, and environmental friendliness (Alhaffar *et al*., 2018; Kaczerewska *et al*., 2018). Because of this, a variety of biopolymers like starch and polymer mixes are used to prevent corrosion. Utilizing these compounds, however, has a variety of drawbacks. The restricted solubility of starch inhibitors, particularly in polar electrolytes, is one of the trickiest problems with their application (Alhaffar *et al*., 2018).

These inhibitors, which are frequently used compounds made up of rings that are aromatic and hydrocarbon chains that are non-polar, are hydrophobic by nature and have limited solubility, which negatively affects the efficacy of their protection (Kaczerewska *et al*., 2018). Recently, research has been concentrated on creating inhibitors with hydrophilic polar functional substituents that are changed in their chemical structures.

The use of grafted polymers has received major attention from corrosion scientists in recent times. This is due to their ability to retard the corrosion of metals in very harmful environments. To create a novel mixture with various physical properties, two or more polymers are combined to form polymer blends (Parameswaranpillai *et al*., 2014). Miscible or homogeneous polymer blends are typically single-phase due to their similar chemical structure. Immiscible polymer blends can also include compatible polymer mixtures because of their obvious similar physical properties and strong relationships between the polymers. Polymer-based blends have been studied and grouped into the following three groups: composites of heterogeneous or immiscible polymers, which typically exist in different phases; mixes of homogeneous or miscible

polymers. The majority of polymer mixtures are naturally immiscible (Jayakumar *et al*., 2022).

According to surface chemistry, the presence of foreign molecules has a significant impact on apparent responses. They can be controlled by using inhibitors that adsorb on the surface of the metal that is reacting because corrosion processes are surface reactions (Arthur *et al*., 2013). Polymer blends are effective inhibitors because of things like their multiple anchoring functional groups or absorption sites, which facilitate easy absorption on the metal surface, their accessibility and stability to metal materials in acidic environments, and their ability to easily form complexes via their functional molecules and on the metal surface. Additionally, the complexes covering the metals' surface protect them from corrosion tools in the solution because they cover a sizable surface area (Umoren and Solomon, 2014).

Biopolymers are a particular class of polymer created by living things. Examples include chitosan, which is present in the shells of insects and crustaceans, and alginate, which is manufactured from naturally occurring anionic polysaccharide that is obtained from seaweeds (Hassan *et al*., 2019). Since biopolymers are renewable and biodegradable, they are known to be environmentally beneficial. Numerous of them have been used for a variety of applications of materials, including coatings, medicine delivery, corrosion inhibitors, etc., because of their availability, biocompatibility, and special features (Umoren and Eduok, 2016).

There have been numerous biopolymers found, and some of them have been the subject of study focusing on corrosion resistance. According to earlier research, biopolymers such as starch, chitosan, polydopamine, cellulose, and carrageenan are effective at reducing the corrosion of metals and alloys (Shanini *et al*., 2021). Numerous adjustments and a range of approaches have been tested to judge and measure their efficacy.

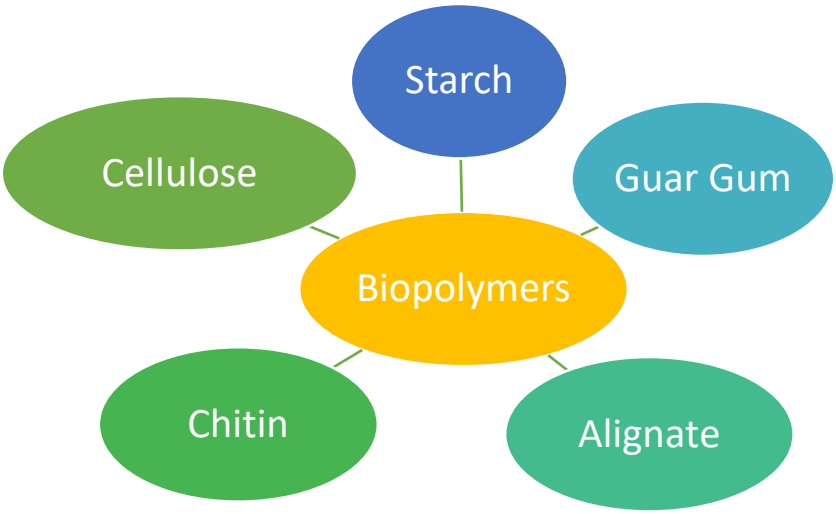

Fig 1: Common Types of Biopolymers Used for Corrosion Inhibition

The naturally occurring carbohydrate-based polymers' restricted solubility in the corrosive solution is one of the problems with using them as corrosion inhibitors, though. They have polymers' high molecular weights, which enable considerable surface coverage, also result in these molecules' poor solubility in the corrosive aqueous medium. In this situation, a practical tactic is to chemically functionalize the polymeric backbone with an appropriate compound to increase solubility and impede performance (Dheeraj *et al*., 2023). The Schiff

base synthesis, crosslinking with synthetic polymers, and chemical species modification are the three most often used techniques for chemically altering certain biopolymers (Haque *et al*., 2018).

Hence, this research is aimed at investigating the differences between PS-PVAGP, modified biopolymer, potato starch and corn starch in the corrosion protection of metals.

2.0 Materials and Methods

2.1 Materials

The metals were collected from the Panteka metal market, Kaduna. They were degreased by washing with ethanol, rinsed with acetone and mechanically press-cut into the dimension of 6.73 by 2.21cm. The coupons were polished using an emery paper of P220 and P1000 (ISO/FEPA Designation). They were dried in a desiccator before further weighing was carried out (Agbogo *et al*., 2020).

NaOH, Borax, 1.18 g/ml HCl and PVA were obtained from Sigma-Aldrich. Potato and corn were purchased from the market, cleaned to remove sand and stored in a dry and clean place before further processing. FTIR characterization was carried out at the National Research Institute for Chemical Technology (NARICT), Kaduna, using Shimadzu FTIR-8400S Fourier Transform Infrared Spectrophotometer.

2.2 Extraction of starch

Fresh samples of starch source (corn, potato, cassava) were washed, peeled, chopped into approximately 1 cm cubes and then ground in a high-speed blender for 5 min. The pulp was suspended in ten times its volume of water, stirred for 5 minutes and filtered using double-fold cheesecloth. The filtrate was allowed to stand for 2 hours for the starch to settle and the top liquid was decanted and discarded. Water was added to the sediment and the mixture was stirred again for 5 minutes. Filtration was repeated as before and the starch from the filtrate was allowed to settle. After decanting the top liquid, the sediment (starch) was dried at 55^{o}C for one hour (Kundu *et al*., 2011).

2.3 Preparation of potato starch-polyvinyl acetate graft copolymer (PS-PAGC)

0.5 M of NaOH was prepared by dissolving 5 g of NaOH pellets in 250 ml of distilled water. 100 ml of the solution was added to the PVA in a beaker and stirred while heating at a constant temperature of 70 ℃ on the heating mantle. This is to ensure that the polyvinyl acetate dissolves completely in NaOH. Meanwhile, 100 ml of distilled water was added to 10 g of raw starch and 0.5 g of borax and stirred in a beaker on a heating mantle to form a homogenous solution. The dissolved solution of PVA was transferred to the starch solution and stirred while heating continuously at 70 ℃. This formed a very viscous and sticky substance. 2 ml of tetra ethylamine was added as a crosslinking agent to balance the blend. 1 g of citric acid was added which acts as a preservative. The sticky substance formed is then cooled, covered with aluminium foil and kept in a cool and dry place (Agbogo *et al*., 2021).

2.4 Preparation of modified biopolymer inhibitor

10g of cassava starch was transferred into a 250 ml beaker with 100 ml of 0.5M NaOH solution. The mixture was stirred vigorously to ensure homogeneity. The starch solution was then heated on a heating mantle at 70 ^{o}C with continuous stirring. After a few minutes, 0.5 g of borax was added and stirred continuously. The mixture was then allowed to cool down and citric acid was added for preservation (Agbogo *et al*., 2021).

2.5 Weight loss analysis

The coupons were worked on using emery paper of different grades, cleaned with a clean piece of cloth, splashed with distilled water and finally degreased using acetone. The coupons were air-dried and weighed using an analytical balance.

The coupons were then dipped in 100ml of 1.0M of HCl in a beaker and removed at 2-hour intervals (up to 8 hours). The removed coupon was then splashed with distilled water, rinsed with methanol for degreasing wrapped in a filter paper and placed in a desiccator to dry. It is then weighed immediately after drying.

The coupon was then placed in a solution of the acid with the inhibitor for 2 hours. It was then removed, splashed with distilled water rinsed with methanol and then wrapped in a filter paper and placed in a desiccator to dry. It was weighed after drying. This procedure was repeated while varying the concentration of various inhibitors from 1g to 4 g respectively (Agbogo *et al*., 2020).

The weight loss, corrosion rate, inhibition efficiency and surface coverage were calculated using the following equations,

$$I.E = 1 - \frac{W1}{W2} \times 100 \quad (1)$$

$$C.R = \frac{W2 - W1}{AT} \quad (2)$$

$$\theta = 1 - \frac{W1}{W2} \quad (3)$$

Where W_1 and W_2 are the weight before and weight after in grams, A is the area of the coupon, T is the total time of immersion in hours, CR is the corrosion rate in gcm^2h^{-1} and Θ is the surface coverage.

3.0 Results and Discussion

In Tables 1 to 4, corrosion data concerning the weight loss, corrosion rate, degree of surface coverage and inhibition efficiencies of various concentrations of potassium starch, corn starch, PS-PVAGC and the modified at various periods of immersion are presented respectively.

Table 1: Weightloss analysis results for potato starch inhibitor measured at room temperature

Time (hr)	C (g/ml)	Weight loss (g)	Inhibition efficiency (IE%)	surface coverage (θ)	CR corrosion rate in $gcm^{-2}h^{-1}$
	Blank	0.322			0.00727
	1.0	0.121	62.42	0.624	0.00276
2	2.0	0.096	70.18	0.701	0.00221
	3.0	0.089	72.36	0.723	0.00206
	4.0	0.065	79.81	0.798	0.00152
	Blank	0.160			0.00445
	1.0	0.384	44.79	0.447	0.00247
4	2.0	0.212	51.88	0.518	0.00215
	3.0	0.184	55.48	0.554	0.00200
	4.0	0.170	67.86	0.678	0.00146
	Blank	0.412			0.00322
	1.0	0.294	28.64	0.286	0.00231
6	2.0	0.207	49.60	0.496	0.00163
	3.0	0.197	52.09	0.520	0.00156
	4.0	0.146	64.51	0.645	0.00117
	Blank	0.435			0.00257
	1.0	0.321	26.21	0.262	0.00191
8	2.0	0.251	42.18	0.421	0.00152
	3.0	0.219	49.64	0.496	0.00132
	4.0	0.170	60.71	0.607	0.00104

	Blank	0.482			0.00231
	1.0	0.389	19.29	0.192	0.00189
10	2.0	0.332	30.95	0.309	0.00163
	3.0	0.268	44.27	0.442	0.00131
	4.0	0.227	52.90	0.528	0.00113

Table 2: Weightloss analysis result for corn starch inhibitor measured at room temperature

Time (hr)	**Conc. (g/l)**	**Weight loss (g)**	**IE% Inhibition efficiency**	**Θ surface coverage**	**CR corrosion rate in gcm^{-2}h^{-1}**
	Blank	0.306			0.0069
	1.0	0.253	30.87	0.308	0.0057
2	2.0	0.246	32.79	0.327	0.0056
	3.0	0.230	37.16	0.371	0.0052
	4.0	0.225	38.51	0.385	0.0051
	Blank	0.390			0.0049
	1.0	0.323	17.18	0.171	0.0037
4	2.0	0.315	19.26	0.192	0.0036
	3.0	0.299	23.33	0.233	0.0034
	4.0	0.290	25.64	0.256	0.0032
	Blank	0.464			0.0036
	1.0	0.386	16.81	0.168	0.0030
6	2.0	0.375	19.10	0.190	0.0029
	3.0	0.362	21.98	0.219	0.0028
	4.0	0.348	25.00	0.250	0.0027
	Blank	0.499			0.0029
	1.0	0.427	14.42	0.144	0.0025
8	2.0	0.411	17.63	0.176	0.0024
	3.0	0.397	20.44	0.204	0.0023
	4.0	0.392	21.44	0.214	0.0023
	Blank	0.546			0.0025
	1.0	0.475	13.00	0.130	0.0022
10	2.0	0.467	14.47	0.144	0.0022
	3.0	0.450	17.58	0.175	0.0021
	4.0	0.448	17.95	0.179	0.0021

Table 3: Weightloss analysis result for ps-pvagc measured at room temperature

Time (hr)	C (g/l)	Weight loss (g)	Inhibition efficiency (IE%)	Surface coverage (θ)	CR ($gcm^{-2}h^{-1}$)
	Blank	0.120			0.00286
	1.0	0.020	83.33	0.833	0.00067
2	2.0	0.012	90.00	0.900	0.00040
	3.0	0.010	91.67	0.916	0.00032
	4.0	0.009	92.50	0.925	0.00021
	Blank	0.160			0.00285
	1.0	0.083	48.13	0.481	0.00135
4	2.0	0.061	61.88	0.618	0.00230
	3.0	0.038	76.25	0.762	0.00072
	4.0	0.027	83.13	0.831	0.00046
	Blank	0.188			0.00276
	1.0	0.101	46.28	0.462	0.00111
6	2.0	0.087	53.72	0.537	0.00100
	3.0	0.061	67.55	0.675	0.00074
	4.0	0.036	80.85	0.808	0.00040
	Blank	0.216			0.00199
	1.0	0.131	39.35	0.393	0.00116
8	2.0	0.105	51.39	0.513	0.00095
	3.0	0.083	61.57	0.615	0.00069
	4.0	0.063	70.83	0.708	0.00057
	Blank	0.290			0.00197
	1.0	0.187	35.517	0.355	0.00129
10	2.0	0.154	46.90	0.468	0.00106
	3.0	0.114	60.69	0.606	0.00080
	4.0	0.087	70.00	0.700	0.00063

Table 4: Weightloss analysis result for modified biopolymer at room temperature

Time (hr)	Conc(g/l)	Weight loss (g)	Inhibition efficiency (IE%)	Surface coverage (θ)	CR ($gcm^{-2}h^{-1}$)
2	Blank	0.066			0.00236
	1.0	0.014	78.8	0.788	0.00051
	2.0	0.012	81.8	0.818	0.00044
	3.0	0.010	84.8	0.848	0.00036
	4.0	0.008	87.9	0.879	0.00029
4	Blank	0.089			0.00159
	1.0	0.074	16.9	0.169	0.00141
	2.0	0.036	59.5	0.595	0.00069

	3.0	0.029	67.0	0.670	0.00056
	4.0	0.023	74.0	0.740	0.00042
6	Blank	0.172			0.00196
	1.0	0.155	9.9	0.099	0.00185
	2.0	0.037	78.5	0.785	0.00047
	3.0	0.030	82.6	0.826	0.00035
	4.0	0.029	71.1	0.831	0.00034
8	Blank	0.276			0.00250
	1.0	0.171	38.0	0.380	0.00155
	2.0	0.109	60.5	0.605	0.00097
	3.0	0.106	61.6	0.616	0.00096
	4.0	0.102	63.0	0.630	0.00093
10	Blank	0.337			0.00236
	1.0	0.204	39.5	0.395	0.00146
	2.0	0.165	51.0	0.510	0.00117
	3.0	0.160	52.5	0.525	0.00116
	4.0	0.136	59.6	0.596	0.00094

3.1 Effect of concentration on the inhibitor efficiency (IE%)

From the gravimetric results, at a maximum concentration of 4g of inhibitor, corn starch gave the poorest inhibition efficiency of 38.33%, followed by potato starch which yielded an IE of 79.81%, then modified biopolymer with an IE of 87.90% and lastly, PS-PVAGC with an IE of 92.50%.

As evident in the weight loss studies, the increase in the inhibitor's concentration reduced the corrosion rate for all inhibitors. The inverse relationship between the inhibitor's concentration and corrosion rate yielded a higher IE for all inhibitors. However, the extent by which the corrosion rate was reduced was different for the various kinds of inhibitors used. The pure starch performed poorly than their modified and blended counterparts.

However, we observed that potato starch performed better than corn starch in this experiment. Potato starch and its modified products have high molecular weight as a result of their high amylose content; hence, yielding to its increased aqueous solubility as compared to corn starch. It thus will possess a higher surface coverage than corn starch because it has a higher adhesive power which helps it to encapsulate the surface of the metal when added to the corrosive media (Yu *et al*., 2015). Also, it has a larger granule size that enhances its coating abilities (Johnston, 1971).

Charitha and Padmalatha (2016) conducted a similar experiment using starch dissolved in 0.1 M HCl on Al-alloy. Their weight loss experiment at room temperature showed that the corrosion rate of the coupon reduced as the starch inhibitor was increased. However, at the highest concentration of 800 ppm, their highest IE was 41.72% (Charitha and Padmalatha, 2016). While their experiment showed that starch had inhibiting properties, it however depicts the limited power it has and as such, agrees with our evaluations.

In another investigation using starch-graft copolymer, their weight loss experiment gave a maximum IE of 90.6%, which is in close range with the result obtained from this investigation (Deng *et al*., 2020). Their research agrees that polymer blends in the form of graft copolymers are significantly effective because they adsorb efficiently on the surface of the metal. In another research, Sihem et al. (2019) reported the synthesis of a modified biopolymer from corn starch and glycerin. The modified corn

starch yielded a maximum IE of 94% at a concentration of 300 mg/L at room temperature. Meanwhile, the pure corn starch used in our experiment yielded a poor IE of 38.33%.

Table 5: Maximum I.E (%) at 4g of each Inhibitor

Inhibitors	IE at 2hrs	IE at 4hrs	IE at 6hrs	IE at 8hrs	IE at 10hrs
Corn starch	38.33	25.64	25.00	21.44	17.95
Potato starch	79.81	67.87	64.51	60.71	52.90
Modified biopolymer	87.90	74.00	71.10	63.0	59.60
PS-PVAGC	92.50	83.125	80.85	70.83	70.00

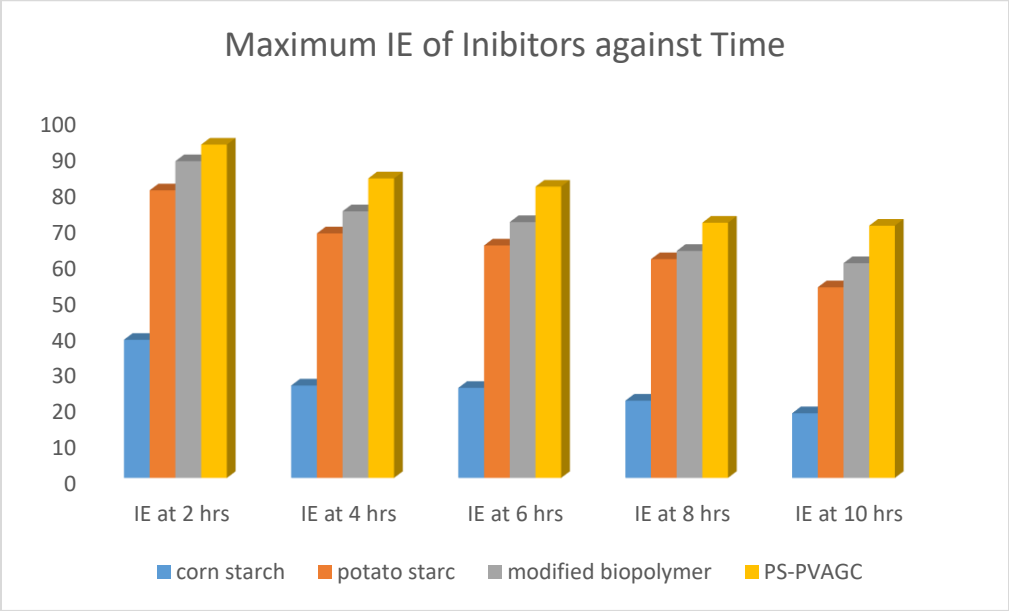

Fig 2: Graph showing maximum IE of inhibitors at different time variations

3.2 FTIR Characterization

FTIR spectra of PS-PVAGC A strong broad peak at 3287 cm^{-1} indicates the presence of OH functional groups. The medium peak at 1636 cm^{-1} indicates C=C stretching. FTIR spectra of starch also showed a strong broad peak at 3446 cm^{-1} showing the presence of OH stretching, while medium peaks at 2824 cm^{-1} indicate C-H stretching. Peak at 1643 indicates C-H bending. Weak peaks at 1417 cm^{-1} and 995 cm^{-1} show OH bending and CO bending respectively. FTIR of modified biopolymer with a strong broad peak at 3318 cm^{-1} indicates the presence of OH group which attached to a medium CH stretching peak at 2922 cm^{-1}. Pronounced peaks at 1029 cm^{-1} show the presence of C-O stretching.

The FTIR graphs provided information about the kinds of heteroatoms contained in the inhibitors. One of the reasons why the inhibitor molecules adsorb on the metal surface and prevent it from corroding is due to the presence

of heteroatoms like OH group. The characterization reveals that all the inhibitors contain OH groups. These OH groups are the active sites that adsorb on the metal surface, reducing the corrosion rate (Agbogo *et al*., 2020).

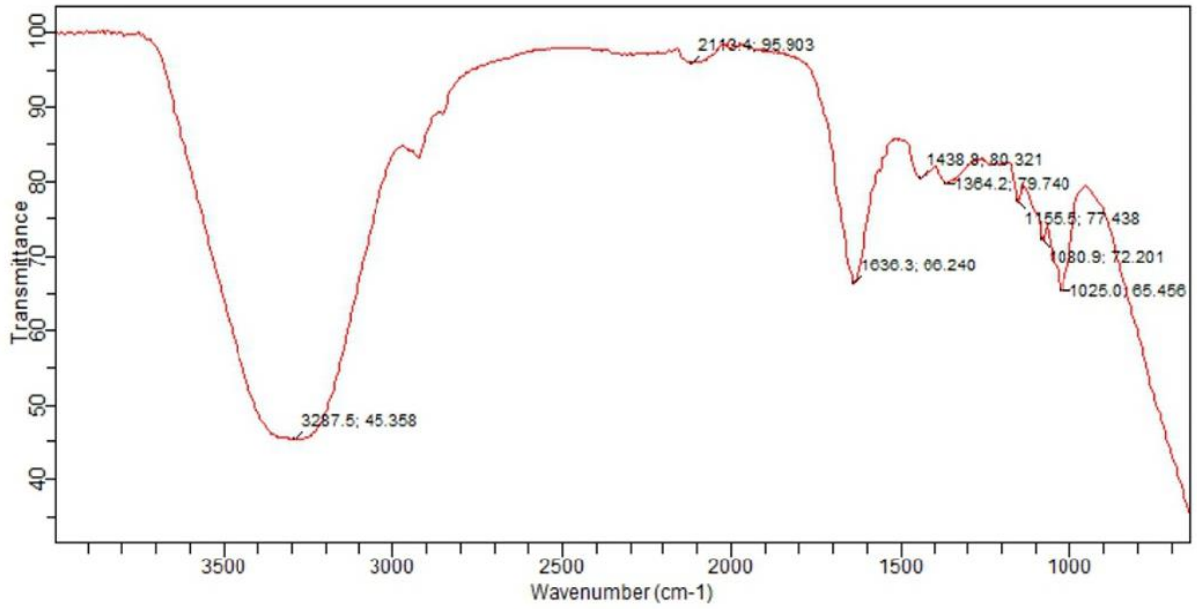

Fig 3: FTIR Spectra of PS-PVAGC

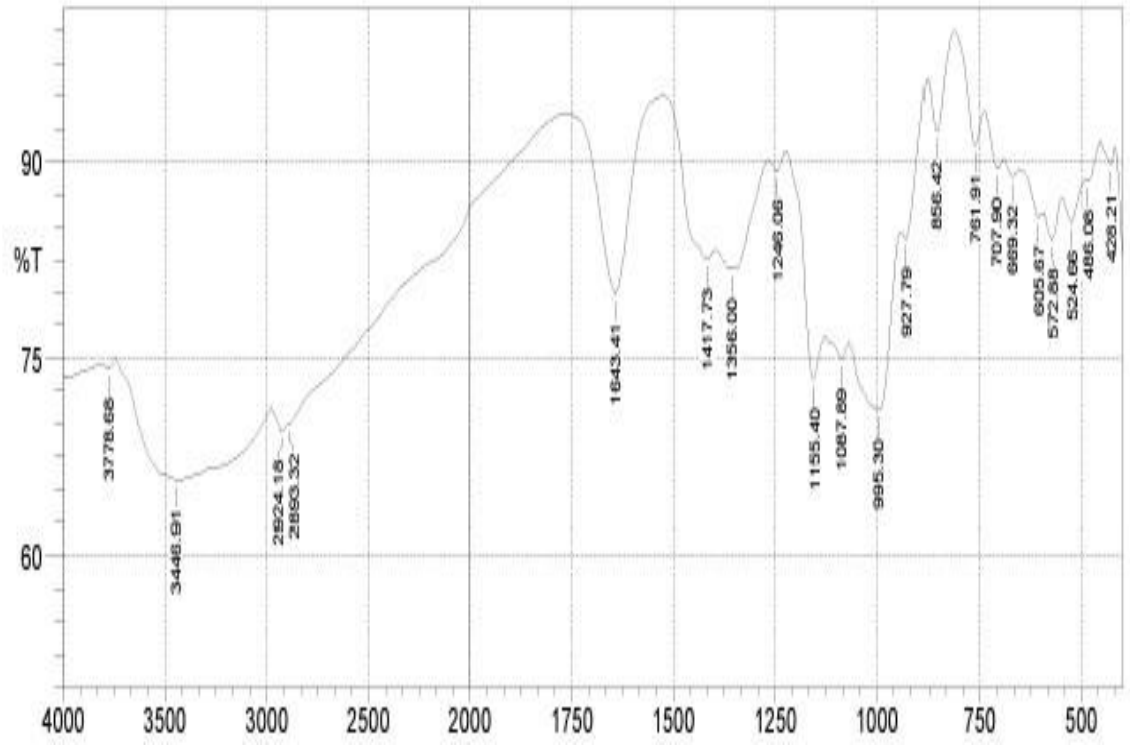

Fig 4: FTIR Spectra of Starch

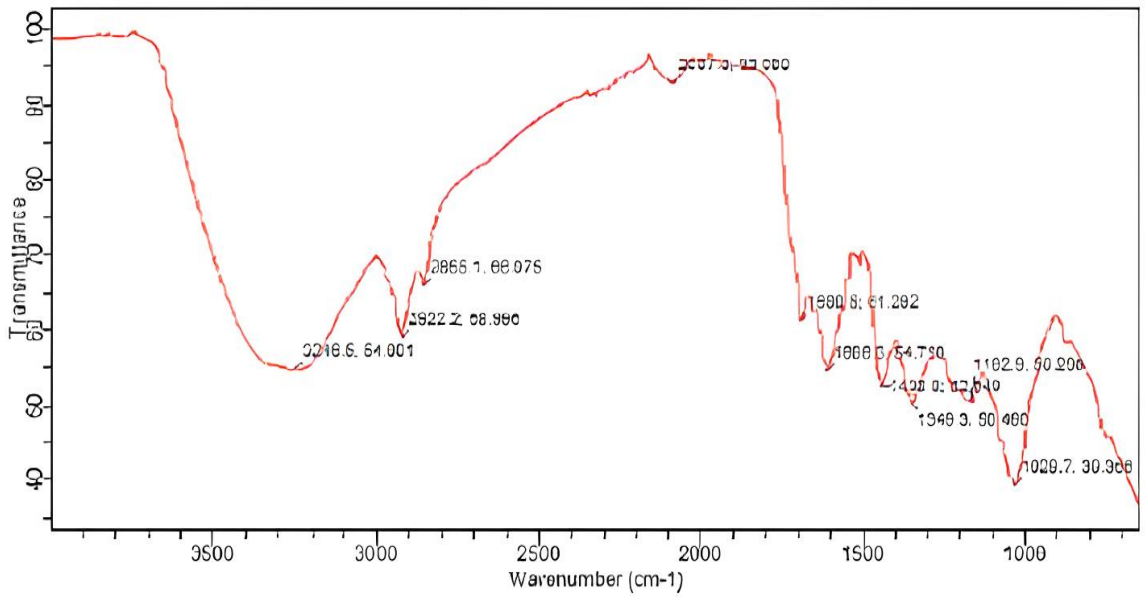

Fig 5: FTIR Spectra of Modified Polymer

4.0 Conclusion

The results of this research have provided experimental evidence that biopolymers have corrosion-inhibitive abilities but are not as effective as modified biopolymers and grafted biopolymers. The modified biopolymers and grafted biopolymers have been proven to possess a higher surface coverage, higher adhesive power and great solubility in a wide range of aqueous mediums. This further helps us to infer that it would be more efficient and sustainable to use polymer blends like PS-PVAGC and modified biopolymer to the purpose of inhibiting corrosion.

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Compliance with Ethical Standards

Declarations

The authors declare that they have no conflict of interest.

Data availability

All data used in this study will be readily available to the public.

Consent for publication

Not Applicable

Availability of data and materials

The publisher has the right to make the data Public.

Competing interests

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Authors' contributions

Ugbetan Victor Agbogo provided the methodology, and assisted with manuscript reviewing and research supervision. Rifore Belief Silas assisted with conceptualization, original draft preparation, and investigation, while Victor Olaoye Inioluwa, Philip Ifeanyi Jerome and Mathew Joshua carried out manuscript reviewing and editing, validation of resources and investigations.