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RESEARCH ARTICLE

Development and Evaluation of the Corrosion Performance of Epoxy-Palm Kernel Shell Ash Nanoparticle Coating for Mild Steel

[1] Oisakede Maureen Olere, [2] Oisakede Emmanuel Eheledu

[*]University of Benin, Faculty of Engineering, Materials and Metallurgical Engineering Department, Benin City, Nigeria
email. maureen.oisakede@uniben.edu orcid.0000-0002-3795-9915
[2]University of Delta, Faculty of Engineering, Civil and Water Resources Engineering Department, Agbor, Nigeria
email. emmanuel.oisakede@unidel.edu.ng orcid.0009-0007-4088-9042

HIGHLIGHTS

- *Effect and importance of the corrosion performance of epoxy-palm kernel shell ash nanoparticle coating for mild steel*
- *Mild Steel should be coated effectively to prevent corrosion*
- *A Corrosion protection efficiency of 98.62% was obtained for the mild steel when coated with epoxy-5% wt palm kernel ash nanoparticles.*
- *The coating helped to cover the surface of the mild steel and made it passive to corrosion attack*

Keywords:

- Mild steel
- Nanoparticles
- Epoxy
- Palm kernel shell ash
- Corrosion protection

GRAPHICAL ABSTRACT

In this study, the effect of the addition of palm kernel shell ash nanoparticles in epoxy as a coating for mild steel was studied. 1, 2, 3, 4 and 5wt% palm kernel shell ash nanoparticles were added to epoxy. The coating was done using spraying method. Corrosion protection efficiency of 98.62% was obtained for the mild steel when coated with epoxy-5% weight Palm Kernel Shell ash nanoparticle. The substrate showed more tendencies to pitting corrosion than the coated samples. This study has established that epoxy-5wt%PKSAnp have good anti-corrosion properties.

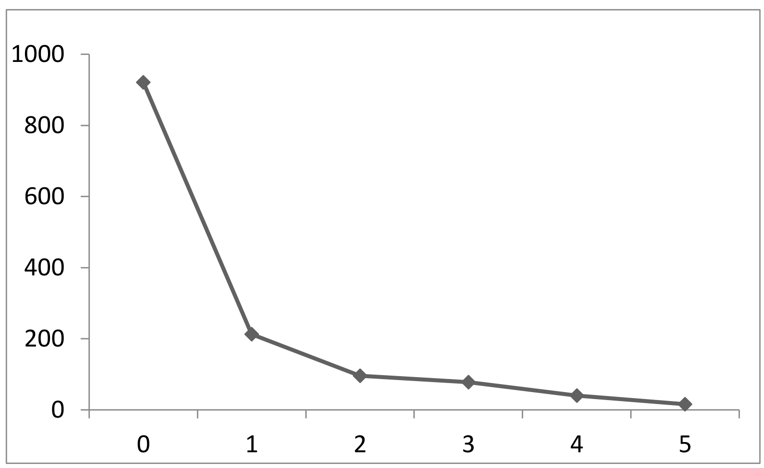

Figure A. Variation of corrosion rate with percentage weight of coating materials

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***Correspondence:**
Maureen Oisakede
maureen.oisakede@uniben.edu
Phone: +23408033632544

Aim of Article : *The aim of this study was to develop and evaluate the corrosion performance of epoxy-palm kernel shell ash nanoparticle coating for mild steel*

Theory and Methodology : *Palm kernel shell ash nanoparticle was produced using the Sol gel method. 1, 2, 3, 4 and 5wt% palm kernel shell ash nanoparticles were added to epoxy. The coating was done using spraying method and the corrosion rate was evaluated.*

Findings and Results: *Coating mild steel with epoxy-5wt%PKSAnp have good anti-corrosion properties.*

Conclusion: *A Corrosion protection efficiency of 98.62% was obtained for the mild steel when coated with epoxy-5% wt palm kernel ash nanoparticles*

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ABSTRACT

The effect of the addition of palm kernel shell ash nanoparticles in epoxy as a coating for mild steel was studied. The protection of steel against corrosion is of importance to the industry and the world at large. Nanoparticles due to their large surface areas have been shown to be excellent materials as they are good absorbents of coating pigments. Palm kernel shells are readily available and easily accessible and they contain calcium which easily combines with epoxy to give some resistance to corrosion attack. Palm kernel shell ash nanoparticle was produced using the Sol gel method. 1, 2, 3, 4 and 5% weight of palm kernel shell ash nanoparticles were added to epoxy. The coating was done using spraying method. Corrosion protection efficiency of 98.62% was obtained for the mild steel when coated with epoxy-5% weight of palm kernel shell ash nanoparticles. The substrate showed more tendencies to pitting corrosion than the coated samples. This study has established that epoxy-5% weight of palm kernel shell ash nanoparticles have good anti-corrosion properties.

Keywords: Mild steel, Nanoparticles, Epoxy, Palm kernel shell ash, Corrosion protection

I. INTRODUCTION

Several processes have been employed to protect metallic substances from corrosion attack. Polymeric coatings are widely used to control corrosion of metal structures, however, due to weak resistance of polymer coating against penetration of electrolyte and thus penetration of corrosive solution to the metal/coating interface, voids, defects and blistering can occur and corrosion resistance of the coating is reduced. Due to weak resistance of polymer coating against penetration of electrolyte and thus penetration of corrosive solution to the metal/coating interface, voids, defects and blistering can occur and corrosion resistance of the coating is reduced [1]. The effectiveness of the coating is typically dependent on the fundamental properties of the sacrificial pigments, barrier effect, organic film, presence of inhibitors and the interface interaction as regards observance [2].

Epoxy resins, a common polymeric coating is susceptible to damage by surface abrasion and wear and also act as pathways accelerating the ingress of water, oxygen and aggressive species onto the metallic substrate, resulting in its localized corrosion [3]. These can be enhanced by the incorporation of a second phase that is miscible with the epoxy polymer, for instance, inorganic filler particles at nanometer scale can be dispersed within the epoxy resin matrix to form an epoxy nano composite. The incorporation of nanoparticles into epoxy resins offers environmentally benign solutions to enhancing the integrity and durability of coatings, since the fine particles dispersed in coatings can fill cavities and cause crack bridging, crack deflection and crack bowing. Nanoparticles can prevent disaggregation of epoxy during curing, resulting in a more homogenous coating. They tend to occupy small hole defects formed from local shrinkage during curing of the epoxy resin and act as a bridge to connect more molecules. This results in a reduced total free volume as well as an increase in the cross-linking density. In addition, epoxy coatings containing nanoparticles offer significant barrier properties for corrosion protection and reduce the trend for the coating to blister or delaminate [3].

Nanoparticles are generally considered to be a number of atoms or molecules bonded together with radius of 100 nm. Nanoparticles find relevance in corrosion resistant, erosion-resistant and wear-resistant environments [4].

Nanoparticles are being incorporated into epoxy matrices as fillers to improve the mechanical, rheological, anticorrosive, and light-resistance properties. Especially nano metal oxides such as TiO_2, Fe_2O_3, ZnO, SiO_2, Al_2O_3, $CaCO_3$ and zirconia have been used as nano filler for corrosion protection on mild steel for more than a decade. The anticorrosive property of these coatings provides a barrier protection against the penetration of aggressive environmental constituents and prevents the cathodic reaction ($2H_2O + O_2 + 4e^- \rightarrow 4OH^-$) occurring on the substrate/coating interface [4].

Nanoparticles can prevent disaggregation of epoxy during curing, resulting in a more homogenous coating. They tend to occupy small hole defects formed from local shrinkage during curing of the epoxy resin and act as a bridge to connect more molecules. This results in a reduced total free volume as well as an increase in the cross-linking density [2].

The use of biomaterials in general and agro-waste in particular is a subject of great interest nowadays not only from the technological and scientific points of view, but also socially, and economically, in terms of employment, cost and environmental issues[5]. Bio-wastes are produced from a large variety of sources and agro-wastes are a class of these wastes. Agro-wastes are gotten from animal and plant sources. These wastes contribute to the problem of environmental pollution and the growing cost of handling the problems of environmental pollution is a world problem being tackled by various organizations around the world. [6] suggested that a wise alternative is to utilize these wastes and extract useful substances from them and therefore reduce the cost of disposing the wastes and also the environmental damages imposed on our environment by these wastes.

Palm kernel shells are waste products of oil palm and previous researches have shown that palm kernel ash contains hard silica. Bearing these in mind, this research focuses on the development and evaluation of the corrosion performance of epoxy-palm kernel shell ash nanoparticle coating for mild steel. The nanoparticles were produced using the sol-gel method and were characterized using XRF (X-ray fluorescence), FT-IR (Fourier transform infrared spectroscopy), XRD (X-ray diffraction), and SEM (Scanning electron microscopy) techniques. The composite coating was then produced by mixing epoxy with the nanoparticles of Palm kernel shell and then the mild steel was coated with it using a spray gun.

II. METHOD

Mild steel (ASTM/SAE 1013 Steel plate/sheet) was used, palm kernel shells (figure 1) were used and they were obtained from Nigeria Institute for Oil Palm Research in Edo State, Epoxy (LY 556) also called *Bisphenol-A-Diglycidyl-Ether* and hardener tri-ethylene-tetramine (TETA) with commercial designation HY 951 was also used and they were purchased from a chemical shop in Warri Delta State Nigeria.

Figure 1: Palm kernel shell

Preparation of mild steel and Palm kernel shell ash

The palm kernel shell was cleaned and dried, they were packed in graphite crucible and ashed in a Carbolite electric resistance furnace at 1100ºC for 4 hrs as reported by [7] to obtained palm kernel shell ash (figure 2). A high intensity planetary ball mill pm 400 machine was used in pulverizing the palm kernel shell ash.
Mild steel with compositions shown in Table 1 was used in this work. The mild steel was grit blasted at a pressure of 3 kg/cm^2 using alumina grits having size of around 60 µm size in order to achieve a good coating adherence. The grit blasted sample was cleaned in an ultrasonic cleaner and the weight of each cleaned specimen was taken by using a precision electronic balance with ± 0.1 mg accuracy. This procedure was adopted from [8]

Figure 2: Palm kernel shell particulate

Table 1: Chemical composition of the mild steel

Metal Elements	Percentage (%)
1. C	0.130
2. Si	0.153
3. Mn	0.630
4. P	0.060
5. Cu	0.040
6. Al	0.030
7. S	0.010
8. Cr	0.010
9. Ni	0.020
10.Mo	0.01
11.W	0.088
12. Fe	balance

Production of the nanoparticles

The sol gel method was applied in producing the palm kernel shell ash nanoparticles. 50 g of Sodium hydroxide mixed in one dm^3 of water was added to 100 g of Palm kernel ash, put in an Erlenmeyer flask and stirred for about 2 hours before the solution was filtered to remove the residue which is carbon (Figure 3). The filtrate was then allowed to cool at room temperature, then hydrochloric acid of weight 0.5 mols was added and stirred for hydrolysis – condensation reaction to occur until pH of 7 was attained with a pH meter and ageing was done at 65ºC for 8 hours in an oven to obtain the gel. The above procedure was adopted from [9].

Figure 3: The palm kernel shell ash filtrate

Characterization of the nanoparticles

The size of the nanoparticles as well as their form and structure were observed with Transmission Electron Microscope using Jeol, JSM2010, X-ray fluorescence spectrometer was utilized for the composition analysis of the produced nanoparticles, X-ray diffractometer , XPertPro PANalytical, LR 39487C was used to obtain the X-ray diffraction patterns of Palm kernel ash nanoparticles.

Production of the Composite Coating

The production of the composite coating was done with accordance with ASTM D3878-20 (2020) Standard Terminology for Composite Materials, ASTM International, West Conshohocken, PA, 2020, these procedures are applied standards on composites containing high-modulus fibers. In the procedure the

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palm kernel shell ash nanoparticles were sonicated in ethanol solvent using sonicator equipped with a titanium probe having a diameter of 13 mm) for 15 minutes, the uncured epoxy (LY556) of density 1.15 – 1.20 g/cm^3 and its corresponding hardener (HY 951) of density 0.98g/cm^3 were then combined by weight in a ratio of 2:1 and the nano particles were added in a percentage weight of 1 to 5 because after the preliminary experiments it was observed that addition of palm kernel Shell beyond 5% made the mixture slurry too thick and could not pass through the nozzle of the spraying machine. Thereafter, the mixture was stirred up to 1200 rpm speed for 15 minutes before the steel was coated with the mixture using spray gun and then kept at room temperature to allow for full curing for seven (7) days. (Figure 4).

Figure 4: Photograph of the coated samples

Characterization of the coated samples

The coating thickness was done in accordance with ASTM E376 - 19 using a coating thickness gauge, XPertPro PANalytical, LR 39487C was used to obtain the X-ray diffraction patterns The microstructure of the samples was conducted using Scanning electron microscope model: TESCAN SEM, Siemens D-500 diffractometer model: Co-Kc radiation (Kc = 1.79026 A) was used to determine the various phases formed in the samples.scanning electron microscope (TESCAN)
A universal testing machine (PC-2000 Testometric testing machine) was utilized to test the adhesion strength, the test was performed as per ASTM C-633,
Electrochemical tester Model: CHI604E was used to perform the corrosion test, this investigation was carried out in accordance with ASTM G19 Standard.

III. Results and Discussion

Transmission Electron Microscopy Analysis of the Nanoparticles

The morphology of the Palm kernel shell ash nanoparticles (PKSAnp) by transmission electron microscope/ energy dispersive spectrometry (TEM/EDS) is shown in Figures 5. The nanoparticles were observed to be solid in nature, but irregular in size. Spherical shape particles can also be seen. The average particle size obtained was 71.67 nm.

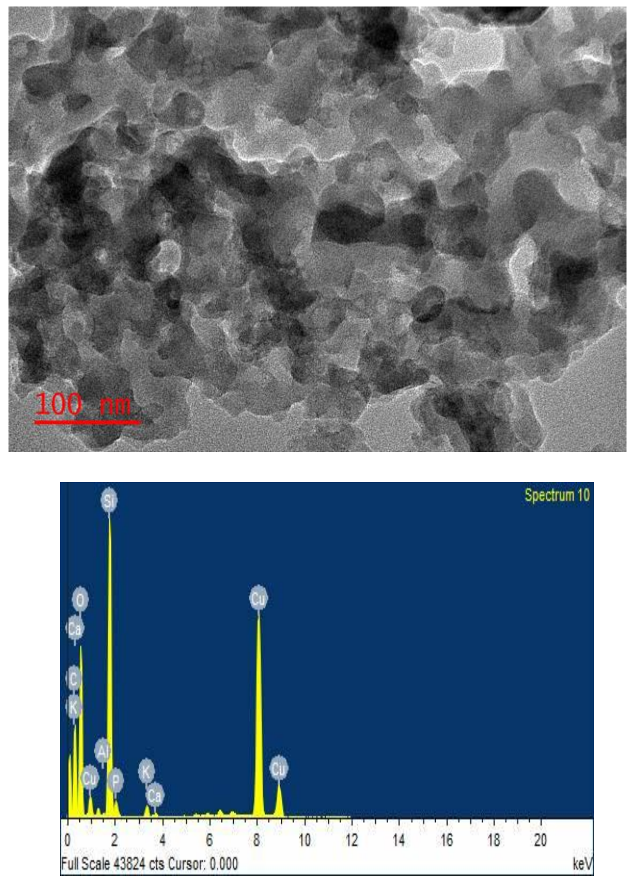

Figure 5: TEM/EDS of the Microstructure of the PKSAnp

It was observed that the micro-analysis of the EDS of the PKSAnp revealed the presence of Si, C, K, Ca, O and Al. The high peak of silicon (Si) in Figure 5 was as a result of the fact that the major constituent of Palm kernel ash nanoparticles is silica. Also the presence of high peak of oxygen (O) may confirm that the various elements in the palm kernel ash are not pure. The high carbon obtained is due to the effect of carbonization at high temperature. This is similar to the work of [10].

Weight gain, coating thickness and Adhesion of the coated samples

Figure 6 displayed the percentage by weight gain of the coating samples, while Figure 7 and give the coating thickness of the coated samples. Figure 6 it was observed that the weight increased as the wt% of PKSAnp was increased, that is weight gained of: 1.92, 2.24, 2.56. 3.01, 3.21, 3.84% was obtained with epoxy/PKSAnp coating. Similar pattern was obtained for the coating thickness. A 99.6, 147.3, 154.3, 181.7, 194.0, 237.3μm was obtained with epoxy/PKSAnp coating. Increases in both weight gain and coating thickness could be attributed to the fact that PKSAnp was able to cover the surface of the mild steel.

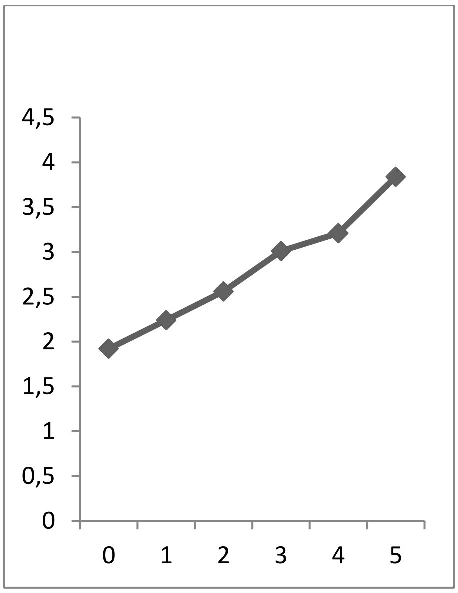

Figure 6: Percentage by weight gain of the sample

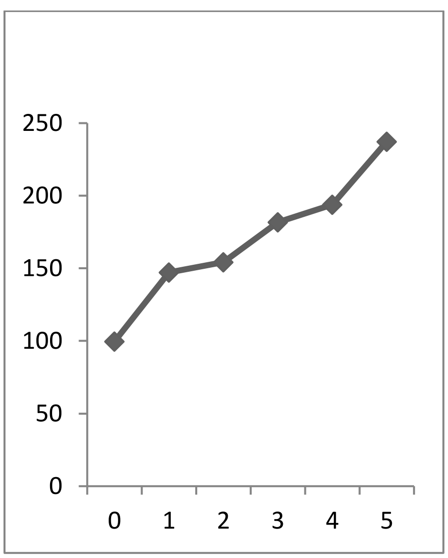

Figure 7: Coating thickness of the sample

Figure 8 shows that adhesion strength rises as the percentage weight of Palm kernel shell ash nanoparticles increased in the epoxy. Values of 5.32, 7.96, 10.248, 11.559, 12.355, 19.986MPa were obtained for the epoxy- palm kernel shell ash nanoparticles at 0, 1, 2, 3, 4 and 5wt%. Improvement of 275.7% was obtained for epoxy-5wt% Palm kernel shell ash nanoparticles, this can be attributed to the good interfacial bond between the mild steel and coating material used. This was achieved by the stirring done during the mixing of the composite coating and also the good surface preparation before the coating of the mild steel.

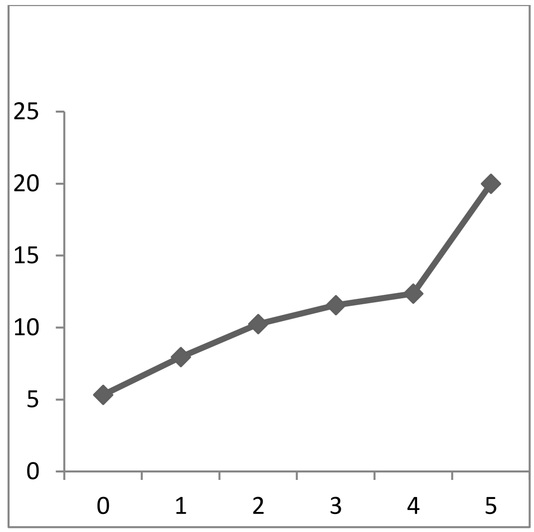

Figure 8: Adhesion strength with percentage by weight coating formulation

Corrosion Rate

Open-Circuit Potential Measurements

In the Open-Circuit Potential plot shown in Figure 9, the mild steel without coating was observed to have the lowest potential of all the samples. Disparity was observed in the potential of the mild steel and coated specimens, with that of the coated specimens tending with respect to the positive axis and that of mild steel tending with respect to the negative axis. Establishing that the developed coating has the ability to protect the specimens against corrosion attack. Also, as the percentage by weight of Palm kernel shell ash nanoparticles increased, the samples' potential also increased with 5wt% Palm kernel shell ash nanoparticles having the highest potential, considering the fact that Palm kernel shell ash nanoparticles was able to cover the entire surface of the mild steel.

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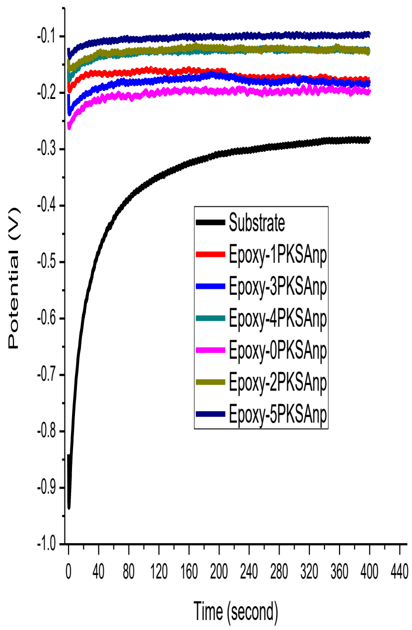

Figure 9: Open circuit potential for epoxy- palm kernel shell ash nanoparticles coating

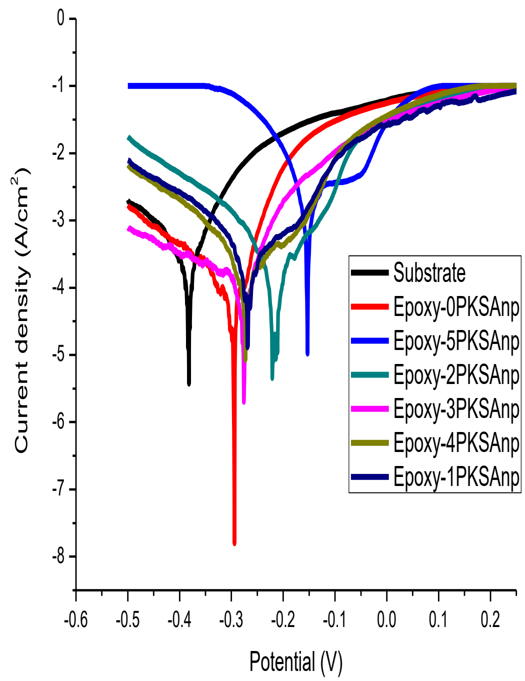

Figure 10: Tafel polarization curves for epoxy-palm kernel shell ash nanoparticles composites coating

Potentiodynamic polarisation studies

The investigation of the corrosion rate of the coating was carried out in simulated sea water using Tafel polarization curves. The Tafel polarization curves are shown in Figure 10 and the outcome obtained are shown in Table 2. From Table 2, it was observed that corrosion current of the samples coated was lesser than that of mild steel alone, and the potential samples coated tending with respect to the positive axis and that of mild steel tending with respect to the negative axis of the graph. This established that the developed coating can improve the corrosion protection of the sample. From Figure 9, the observation showed that the mean potential for mild steel moved towards a lesser potential and elevated current density. With increase in percentage weight of Palm kernel shell ash nanoparticles, the corrosion potential as well as the corrosion resistance of the samples also increased, this could be due to the various microstructures obtained in the work.

From Figures 11- 14 shows the potential, current density, polarisation resistance and corrosion rate of the samples, it can be seen that there is a comparable drift, meaning the higher the polarisation and potential resistance the lesser the corrosion rate and current density. This trend was anticipated since the lesser the potential the higher the predisposition for corrosion to occur. For mild steel, the corrosion potential was -0.385 the values increased to -0.289, -0.16, -0.156, -0.143, -0.136, -0.103V for epoxy-palm kernel shell ash nanoparticles at 0, 1, 2, 3, 4, and 5wt% respectively. Furthermore, the corrosion rate of the mild steel was 1150 mpy this decreased to 921, 213.1, 95.82, 78, 40.1, 15.87mpy for epoxy-palm kernel ash nanoparticles at 0, 1, 2, 3, 4, and 5wt% respectively. A Corrosion protection efficiency of 98.62% was obtained for the mild steel when coated with epoxy-5wt% Palm kernel ash nanoparticles respectively.

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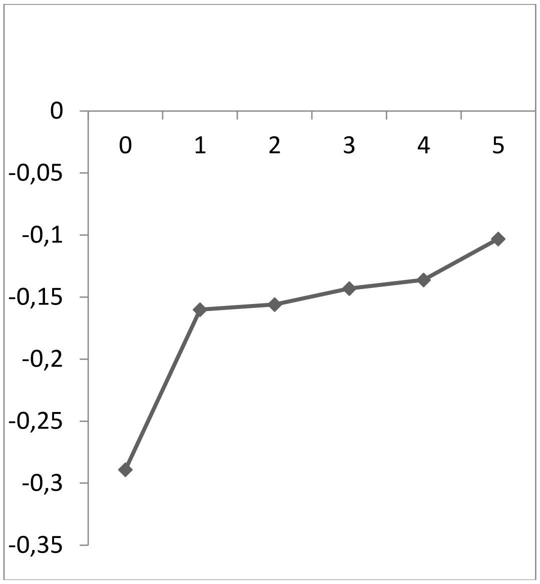

Figure 11: Variation of corrosion potential with percentage weight of coating materials

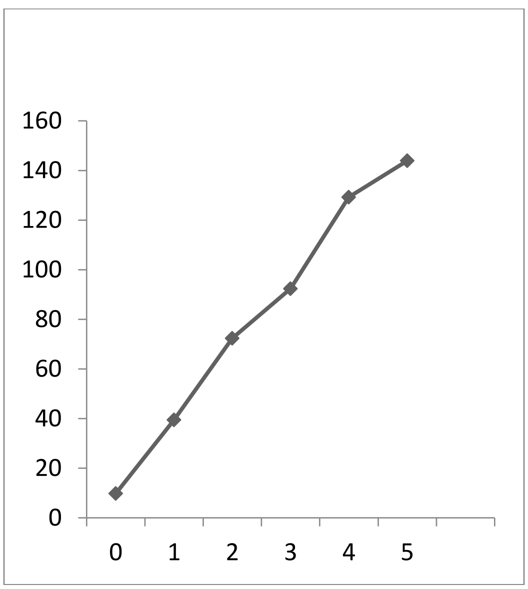

Figure 12: Variation of corrosion resistance with percentage weight of coating materials

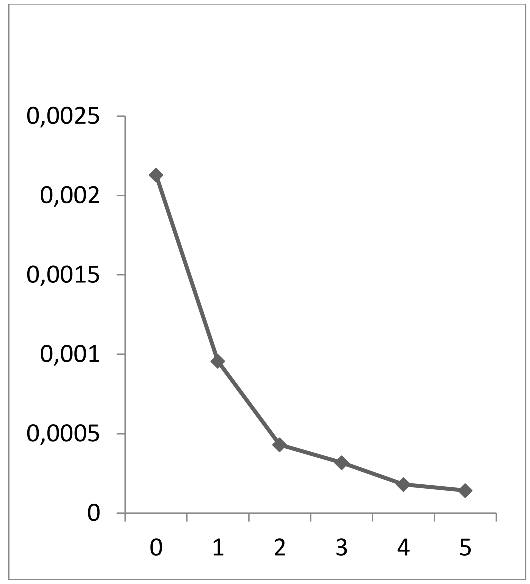

Figure 13: Variation of corrosion current density with percentage by weight of coating materials

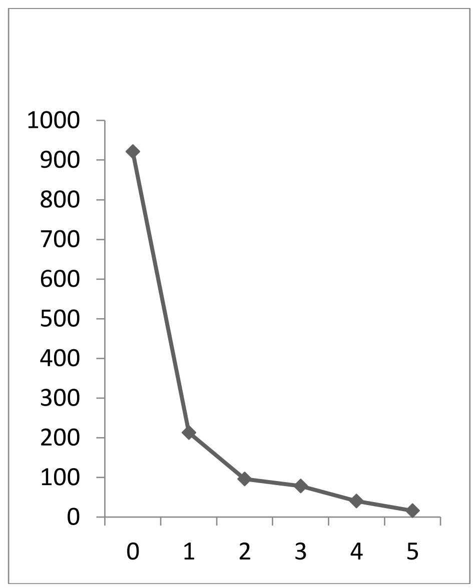

Figure 14: Variation of corrosion rate with percentage weight of coating materials

In studying the corrosion surface of mild steel as well as its composite coatings at 0wt% and 5% wt palm kernel shell ash nanoparticles formulation, scanning electron microscope was used. It was seen that mild steel showed higher tendency to pitting corrosion than the samples that are coated (Figure 15).

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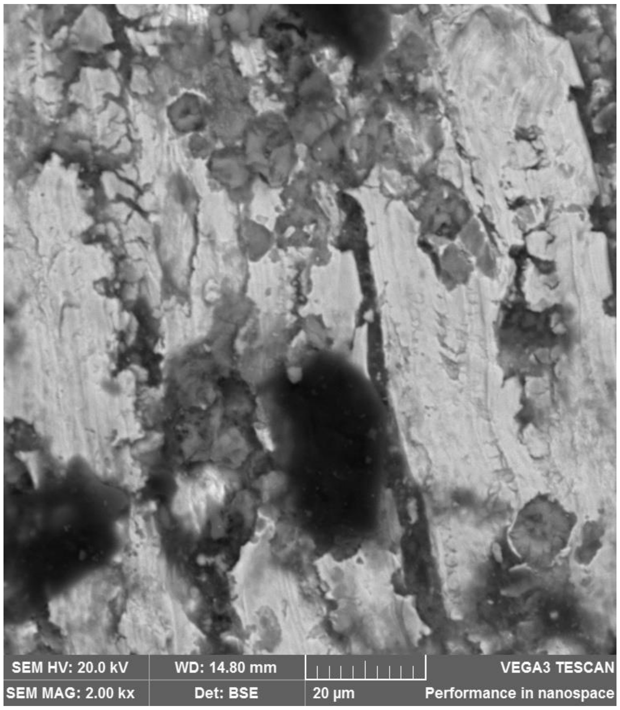

Figure 15: Scanning Electron Microscopy corrosion worn surface of the uncoated mild steel

The corroded surface of the coated sample revealed that the coating was not detached from the surface of the mild steel as a result of corrosion attack. The coating helped to cover the surface of the mild steel and made it passive to corrosion attack. (Figures 16).

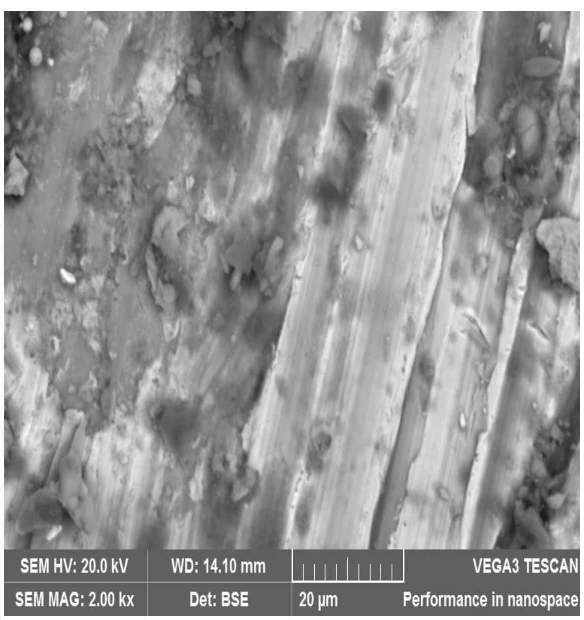

Figure 16: Scanning Electron Microscopy corrosion worn surface of the epoxy-5wt% Palm kernel ash nanoparticles coated mild steel

The method of the corrosion of materials in sea water involves vigorous disbanding of mild steel in subsea water as well as electron discharge [11]. This is shown in equations 1 and 2

$$Fe + H_2O + Cl^- \text{---------------------} [FeClOH]_{ads} \quad (1)$$

$$[FeClOH] + H^+ \text{--------------------} Fe^{2+} + Cl^- + H_2O \quad (2)$$

There was a huge amount of pits enveloped by iron oxide film showing that pit creation under these circumstances occurred at some point in the exposure time while iron oxide built up on the surface (Figure 14).
The sample of the composite coating contained hard phases and made the degree of damage on the sample surface low (Figure 16). These hard phases gave some resistance to the corrosion attack. Therefore, this study provides credibility to the controversy that the existence of Palm kernel ash nanoparticles allows low corrosion of materials [12]

IV. CONCLUSION

Palm kernel shell ash was successful developed as nanoparticles using the sol-gel method and from the transmission electron microscope analysis, they were seen to be solid, irregular in size and sphere-shaped with particle size of 71.67 nm. Characterization of the nanoparticles as well as the coating of the mild steel was also successfully done and the following conclusions were obtained:
I. The weight of the coating as well as coat thickness increased with the increase in weight percent of Palm kernel shell ash nanoparticles up to 5%, after which there was a decrease.
ii. From the scanning electron microscope analysis of the microstructure of the coated samples with Palm kernel ash nanoparticles, it was observed that dense, packed and smaller grains than the uncoated samples were formed and there were no external features of particles such as contours, defects or damages seen on the images. This could be attributed to the effectiveness of the stirring done before the spraying which was able to disperse the nanoparticles as well as the spraying method applied.
iii. Improvement of adhesion strength of 275.7% was obtained for epoxy-5% weight Palm kernel ash nanoparticles
iv. The coating helped to cover the surface of the mild steel and made it passive to corrosion attack
v. A Corrosion protection efficiency of 98.62% was obtained for the mild steel when coated with epoxy-5% weight palm kernel ash nanoparticles.

CONFLICTS OF INTEREST

They reported that there was no conflict of interest between the authors and their respective institutions.

RESEARCH AND PUBLICATION ETHICS

In the studies carried out within the scope of this article, the rules of research and publication ethics were followed.

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