



MICROSTRUCTURE AND MECHANICAL PROPERTIES OF EPOXY-PALM KERNEL SHELL NANOPARTICLE COMPOSITE COATING ON MILD STEEL

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ABSTRACT

An attempt has been made in this work to develop a novel epoxy-palm kernel shell ash nanoparticle (PKSAnp) composite coating on mild steel. One to five weight percent PKSAnp were added to epoxy. The coating was done using spraying method. The microstructure, coating thickness, hardness values and adhesion strength were determined. A 51.69% and 275.7% improvement in hardness values and adhesion strength of the composite coating on mild steel were obtained at 5wt%PKSAnp. This work showed that epoxy-5wt%PKSAnp has best properties for adhesion strength and hardness values.

KEYWORDS: *Mild steel, Epoxy, Nanoparticles, Palm kernel shell ash, Hardness values, Adhesion strength.*

1. INTRODUCTION

Steels are vital construction materials in our modern society; the corrosion protection of steel is of great importance, both to ensure safety and to reduce costs associated to corrosion. Functional coatings are applied to change the surface properties of the substrate, such as corrosion resistance, wear resistance and hardness. Protective coatings are possibly the most widely used products for corrosion mechanism (Roberge, 2000). The main function of a protective coating is to isolate structural reactive elements from environmental corrosion. A coating must provide a continuous barrier to a substrate, and any imperfection can become the focal point for degradation and corrosion of the substrate (Smith and Yirmani 2000). Metallic coating produce surface coated layer which improves the properties of the substrate compared to uncoated specimen.

Epoxy resin is the most common polymeric coating that inhibited the process of metal corrosion due to high tensile strength and modulus, low

shrinkage in cure, good chemical and corrosion resistance, high adhesion and dimensional stability. Protection of steel by epoxy coating is based on the principles of acting as both a physical and electrochemical barrier. The successful application of epoxy coatings is often hampered by their susceptibility to damage by surface abrasion and wear. They also show poor resistance to the initiation and propagation of cracks. Such processes introduce localized defects in the coating and impair their appearance and mechanical strength. The defects can also act as pathways accelerating the ingress of water, oxygen and aggressive species onto the metallic substrate, resulting in its localized corrosion. Furthermore, being hydrophilic in nature epoxy coatings experience large volume shrinkage upon curing and can absorb water from surroundings. The pores in the cured epoxy coating can assist in the migration of absorbed water and other species to the epoxy-metal interface, leading to the initiation of corrosion of the metallic

substrate and to the delamination of the coating (Min Zhi et al, 2001).

Nanoparticles are generally considered to be a number of atoms or molecules bonded together with radius of 100 nm. A cluster of one nanometer radius has approximately 25 atoms, but most of them are on the surface of the cluster. In typical nanomaterials, the majority of the atoms are located on the surface of the particles, whereas they are located in the bulk of conventional materials. Thus, the intrinsic properties of nanomaterials are different from conventional materials since the majority of atoms are in a different environment (Gleiter, 2000). Nanoparticles tend to occupy small hole defects formed from local shrinkage during curing of the epoxy resin and act as a bridge interconnecting 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 (Min Zhi et al, 2001)., These materials can be blended with epoxy matrix with low concentrations due to their unique properties. The challenges to apply these materials are based on toxicity, dispersability and homogeneity in epoxy matrix due to their high reactivity (Gia Vu et al, 2014; Winterer et al, 2003).

Nanoparticles are being incorporated into epoxy matrices as filler to improve the mechanical, rheological, anticorrosive, and light-resistance properties. Nano metal oxides such as TiO₂, Fe₂O₃, ZnO, SiO₂, Al₂O₃, CaCO₃ and zirconia have been used as nano filler for corrosion protection on mild steel for more than a decade. The high cost of current nanoparticles compared to epoxy has inhibited production of the coating on industrial scale. In attempts to overcome this limitation, this research will focus on the development of the nanoparticles using

low cost agro-waste by-products (palm kernel shell) in the production of the epoxy-agro-waste.

2. MATERIALS AND METHODS

2.1 materials

The palm kernel shell was obtained from Nigeria Institute for Oil Research near Benin City, Nigeria, The epoxy (LY 556), chemically belonging to the epoxide family was used in the present work. Its common name is *Bisphenol-A-Diglycidyl-Ether*; hardener tri-ethylene-tetramine (TETA) designation HY 951 was purchased from Chemical shop in Warri Delta State Nigeria.

2.2 Method

The palm kernel shell was cleaned and dried. They were packed in graphite crucible and heated in a muffle furnace at 1200°C to obtain palm kernel shell ash (PKSA). The PKSA was pulverized in high intensity ball milling machine. 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/cm² using alumina grits having size of around 60 µm size. 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.

Table 1: Chemical composition of the mild steel

S/N	Elements	Percentage (wt. %)
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

2.3.3 Production of the Nanoparticles

The sol gel method was used in the production of PKSA_{np}. Particle size and morphology of produced nanoparticles was examined by TEM (Jeol, JSM2010) using a 200 keV electron beam. Mini Pal compact energy dispersive X-ray spectrometer (XRF) was used for the elemental analysis of the produced nanoparticles. The PKSA nanoparticles were continuously sonicated in ethanol solvent by ultrasonic waves using sonicator equipped with a titanium probe with a diameter =13 mm) for 15 minutes. The uncured epoxy (LY556) and its corresponding hardener (HY 951) were mixed in a ratio of 2:1 by weight as per recommendation. Then 1, 2, 3, 4 and 5wt% nanoparticles were added to the epoxy mixture and stirred up to a speed of 1200 rpm for 15 minutes, then the coating mixture was applied to the steel substrate by using spray gun and then kept in a dry place at room temperature for 7 days to allow full curing (Figure 1).



Figure 1: Photograph of the coated samples

The thickness of the coatings was measured by cutting each sample into two halves (perpendicular to its length) using a TechCut 4 low speed saw from Allied High Tech. Productions Inc. The thickness of the coatings was calculated by taking the mean of three thickness values on each sample. The X-ray diffraction (XRD) patterns of the samples were determined by X^{PERT}Pro PANalytical, LR 39487C XRD diffractometer using Cu K α radiation (40 kV, 40 mA). The surfaces of the

coated specimens were examined directly by scanning electron microscope TESCAN. The specimens were cleaned thoroughly with acetone before being observed under SEM. The portable Rockwell hardness machine was used to determine the hardness values of the samples. The coating adhesion strength was determined using universal testing machine PC-2000 Testometric testing machine.

3. 0 RESULTS AND DISCUSSION

The morphologies of the PKSA_{np} obtained using TEM are shown in Figures 2. 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 of 71.67 nm was obtained.

Figure 3 shows the percentage weight gain of the coating samples, while Figure 4 give the coating thickness of the coated samples. In Figure 3 it was observed that the weight increases with increasing wt% of PKSA_{np}. For example weight gain of 1.92, 2.24, 2.56, 3.01 and 3.21 mg were observed for the 1-5wt.% epoxy/PKSA_{np} coating, respectively. Similar pattern was obtained for the coating thicknesses. The thicknesses of 99.6, 147.3, 154.3, 181.7, 194.0, 237.3 μ m were also observed for the epoxy/PKSA_{np} coating, respectively. Increases in both weight gain and coating thickness could be attributed to the facts that PKSA_{np} was able to cover the surface of the mild steel.

The XRD patterns of the substrate and coated samples are shown in Figure 5. From Figure 5, it can be seen that the substrate has two major peaks which corresponded to α -Fe and Fe₃C around 45 and 65° respectively. The composite coating has Fe, C₁₁H₆O₂ and C phases.

The presence of C₁₁H₆O₂ and C in the epoxy coated samples is attributed to the facts that epoxy was used as the matrix materials for the coating.

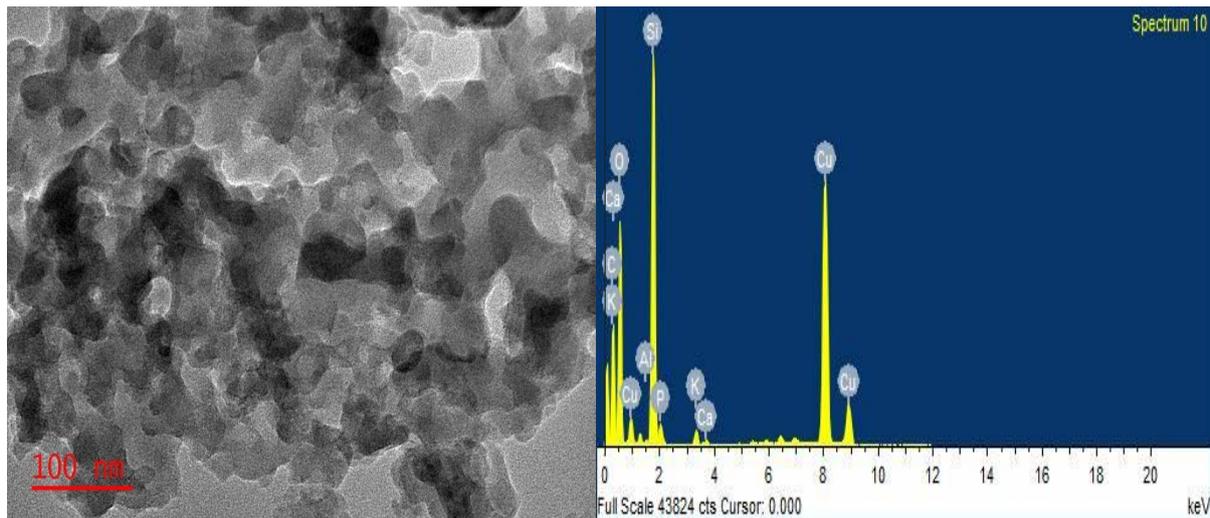


Figure 2: TEM/EDS of the Microstructure of the PKSAnp

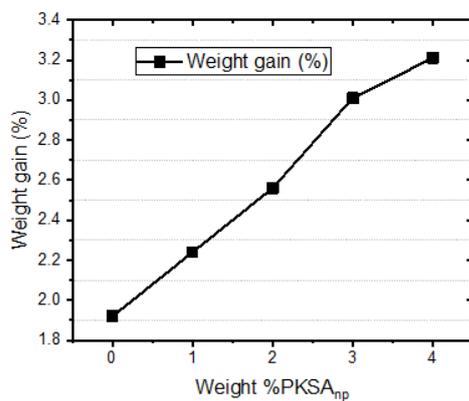


Figure 3: Percentage weight gain of the samples

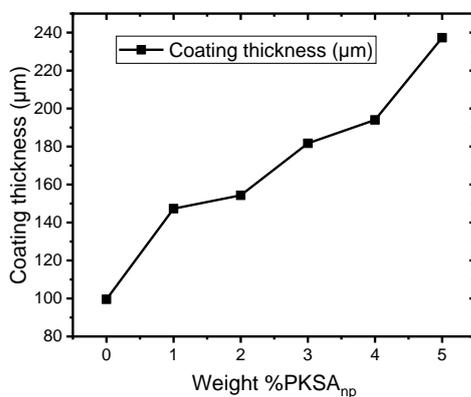


Figure 4: Coating thickness of the samples

Similar observation was obtained in the work of (Zaarei et al, 2010).

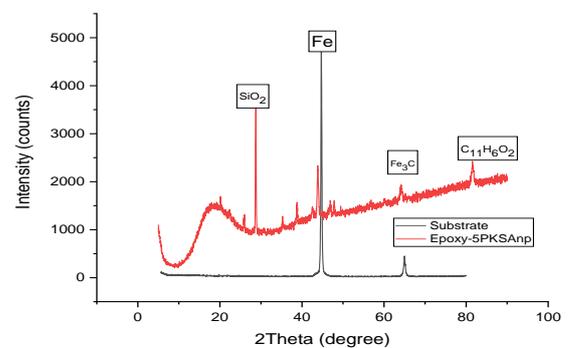


Figure 5: XRD analysis of the coated sample

The SEM analysis in Figure 6 shows the structure of the mild steel, the scratches on the surfaces show the lines made by the abrasive cutter. Figure 7 shows the SEM of the coated samples. From Figure 7, one can observe morphological differences of the structure when compared with the SEM of the substrate. It was observed in Figure 7 that with the coated samples of PKSAnp, dense, pack and smaller grains

were formed. This could be attributed to the fact that the stirring applied during production was able to mobilize the dispersed PKSAnp. There were no external surface features of particles such as contours, defects and damage and surface layer in the SEM images of the coated samples. The coating surface layers play an important role in the properties of the mild steel. The better adhesion that occurs within the interface is as a result of the stirring and spraying applied during production.

From the energy dispersion spectrum (EDS analysis) of the composite coating, it was observed in Figures 6-7 that there is great difference in the EDS of the substrate from that of the coated samples. The EDS of the mild steel substrate revealed high peak of Fe (Figure 6). The EDS of the

PKSAnp coated samples have high peak of Si (Figure 7). The absence of Si in the EDS of uncoated sample was attributed to the facts that PKSAnp were not added at this formulation, this result was at par with the work of Tolumoye et al (2014).

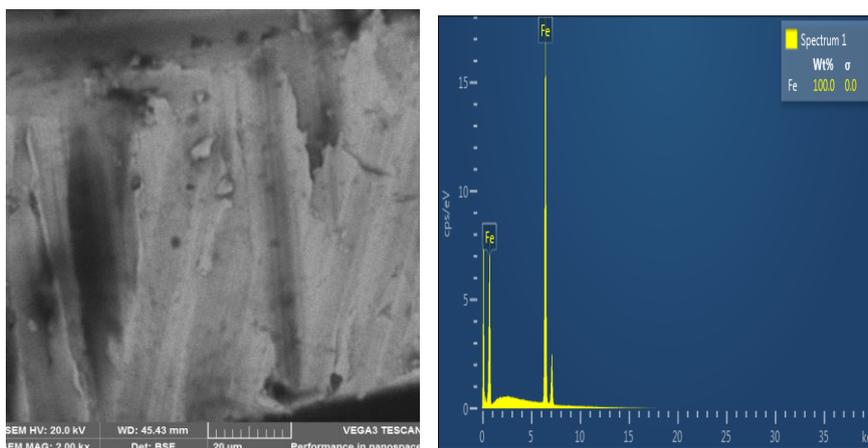


Figure 6: SEM/EDS of the mild steel substrate

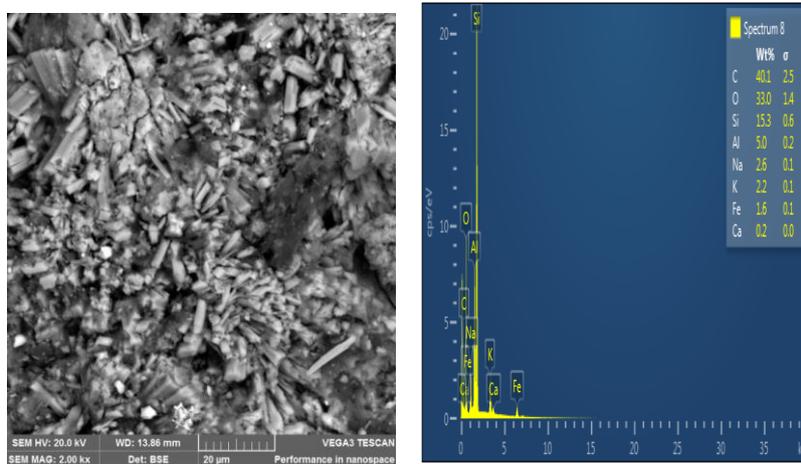


Figure 7: SEM/EDS of the epoxy-5wtPKSAnp coated mild steel substrate

Figure 8 shows the results of the hardness values. From Figure 8, it is clearly seen that the hardness values of the mild steel improved with increase in the wt% PKSA_{np}. The high hardness values of the epoxy-PKSA_{np} composites can be attributed to the presence of hard phase of SiO₂. Similar observation was observed in the work of Tiwari et al, (2011). An improvement of 51.69% in hardness values of the mild steel was obtained at 5wt%PKSA_{np}.

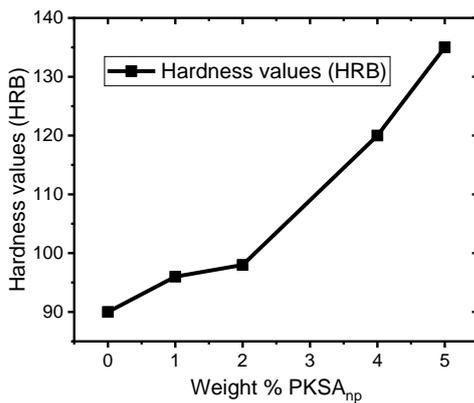


Figure 8: Variation of Hardness values with weight % PKSA_{np}

The adhesion strength is displayed in Figure 9. It was seen that adhesion strength increased as the wt% PKSA_{np} increased in the epoxy. For example the values of 5.32, 7.96, 10.248, 11.559, 12.355, 19.986 MPa were obtained for epoxy-PKSA_{np} at 0, 1, 2, 3, 4 and 5wt%

leading to overall improvement of 275.7% at 5wt% PKSA_{np}. This general increase in the adhesion strength can be attributed to the good interfacial bonding between the coating materials and the mild steel substrate. This interfacial bonding was achieved with help of the stirrer used in the mixing of the coating mixture and good surface preparation of the mild steel before coating.

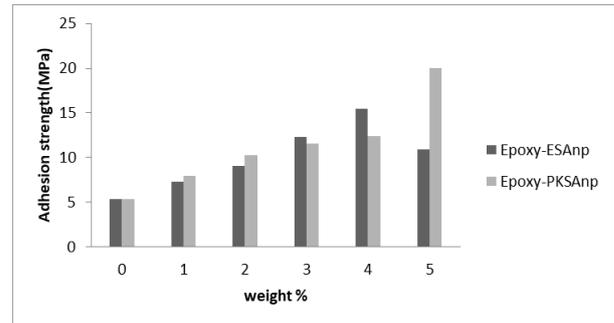


Figure 9: Adhesion strength with weight % coating formulation

4. CONCLUSIONS

From the above results, the following conclusions are made:

1. The coating thickness increases with increase in wt% of PKSA_{np}.
2. The adhesion strength rises as the wt% PKSA_{np} increases in the epoxy.
3. There were no external surface features of particles such as contours, defects and damage and surface layer in the SEM images of the coated samples
4. A 51.69% improvement in hardness values of the mild steel were obtained at 5wt%PKSA_{np}.
5. It has been established that enhanced hardness values and adhesion strength can be obtained with the formulation.

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6. Conflict of Interest

There is no conflict of interest associated with this work.

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