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Effect of Epoxy-Egg Shell Nanoparticle Composite Coating on the Microstructure and Mechanical Properties of Mild Steel

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ABSTRACT

Nanoparticles due to their large surface areas have been shown to be excellent materials as they are good absorbents of coating pigments. In this research, an attempt has been made to develop a novel epoxy-egg shell ash nanoparticle (ESAnp) composite coating on mild steel. Sol-gel method was used to produce the nanoparticles. A 1, 2, 3, 4 and 4wt% ESAnp were added to epoxy. The coating was done using spraying method. The microstructure, coat thickness, hardness values and adhesion strength were determined. Improvement of hardness values and adhesion strength of the mild steel were obtained 4wt% ESAnp at 43.48 % and 190.4%. The results obtained from this work showed that epoxy-4wt% ESAnp has best properties for adhesion strength and hardness values.

1.Introduction

Mild steel which is commonly used in the production of tanks and pipeline finds application in general purpose engineering as a versatile material. However, mild steel possesses low corrosion resistance. Several processes have been employed in the past to protect metallic substances from corrosion attack such as coatings. 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 [1]. 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 [2]. The main function of a protective coating is to isolate structural reactive elements from environmental corrosives. 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 [3]. Metallic coating produce surface coated layer which improves the properties of the substrate compared to uncoated specimen. 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. Nanoparticles are generally considered to be a number of atoms or molecules bonded together with radius of 100nm. A cluster of one nanometer radius has approximately 25 atoms, but most of them are on the surface of the cluster. 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 [4]. 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 [5, 6]. 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 [7]. 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. Some of the animal wastes include feathers, shells (egg, snail, periwinkle, and etcetera), horns, hides and skin, hoofs, bones, etcetera. 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 [8]

Thus, this work evaluates the effect of epoxy-egg shell nanoparticle composite coating on the microstructure and mechanical properties of mild steel.

2. Methodology

2.1 Materials

The eggshells were obtained from a local tea seller in 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 and hardener tri-ethylene-tetramine (TETA) designation HY 951 was purchased from chemical shop in Warri Delta State Nigeria.

Figure 1: Eggshell used in the experiment

2.2 Method

To detach the membranes from the shells, the eggshells were washed using water and thereafter dried and ball milled into eggshell powder particles and then packed in a graphite crucible before firing in a carbolite electric resistance furnace.

Figure 2: Carbolite electric furnace

2.2.1. Mild steel composition

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. 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

	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

2.2.2. Production of the Nanoparticles

The sol gel method was used in the production of PKSAnp. 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.

2.2.3. Production of the Composite coating

The ESAnp 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 and 4wt% nanoparticles was 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 3). The X-ray diffraction (XRD) patterns of the samples were determined by X‟PertPro PANalytical, LR 39487C. XRD diffractometer using Cu Kα radiation (40 kV, 40 mA). The surfaces of the coating specimens were examined directly by scanning electron microscope TESCAN. The specimens were cleaned thoroughly with acetone before being observed under SEM.

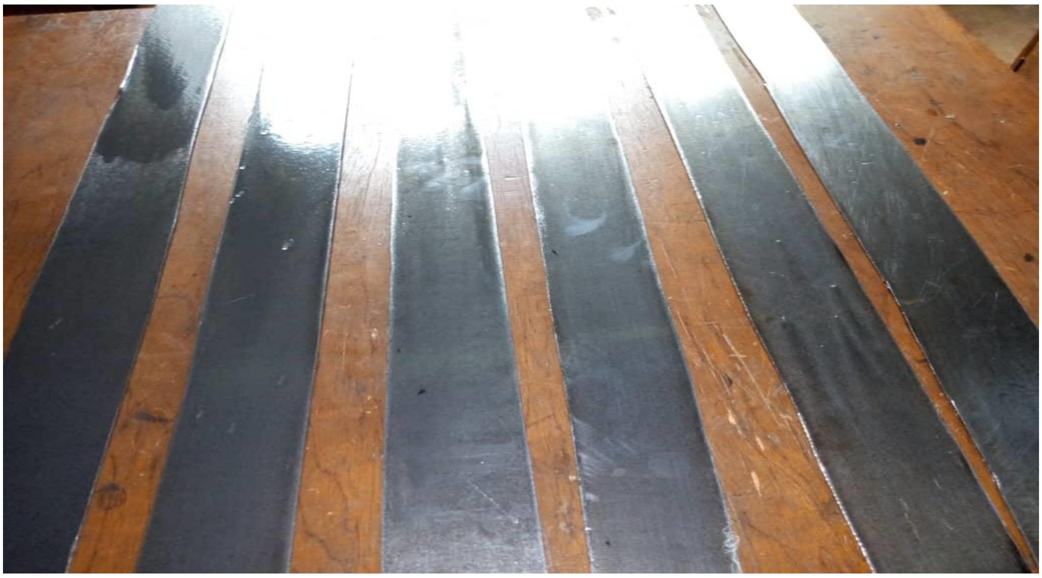

Figure 3: Photograph of the coated samples

2.2.4. Determination of Coating Thickness of the Specimen

With the aid of a TechCut 4 low speed saw, each sample was cut into two and the coat thickness was obtained using a coating thickness gauge and the average thickness was computed by calculating the mean of three values of thickness obtained from each sample in order to obtain uniformity and accuracy of the results (Figure 4).

Figure 4: Coating thickness gauge

2.2.5. Determination of the Hardness Values of the Specimen

The portable Rockwell hardness machine was used to determine the hardness values of the samples. A Rockwell hardness machine with 1.56mm steel ball indenter, minor load and major load of 10kg and 100kg respectively was utilized to determine the hardness values. A load of 10kg was initially applied to the sample to establish zero datum position and then the major load of 100kg was applied. The weight was detached by returning the crank handle to the latched spot and the hardness value computed from the semi-automatic digital scale. The average of three readings was recorded.

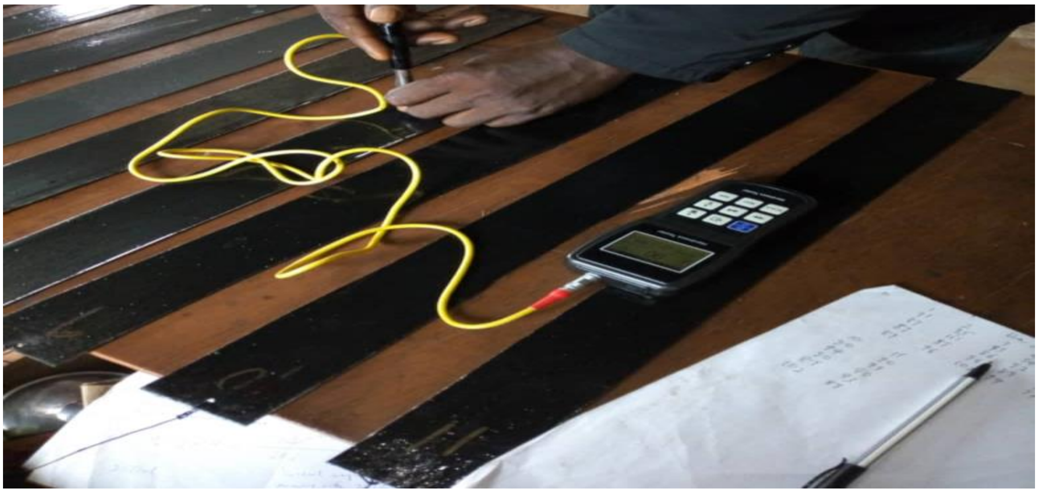

Figure 5: Rockwell hardness machine used for the Hardness test

2.2.6. Determination of the Adhesion strength of the Specimen

The coating adhesion strength was determined using universal testing machine PC-2000 Testometric testing machine. A universal testing machine (PC-2000 Testometric testing machine) was utilized for this procedure Figure 6. The pull-out method which involves two cylindrical samples being used with the surface of one of the cylinders coated with the material being examined and glued with the epoxy resin to the surface of the other cylindrical sample which is not coated.

Before the gluing, the uncoated face was grit blasted. Both cylinders were then controlled with steady tensile load. The adhesion strength of the coat was computed by dividing the maximum load applied at the rupture by the cross sectional area of the sample.

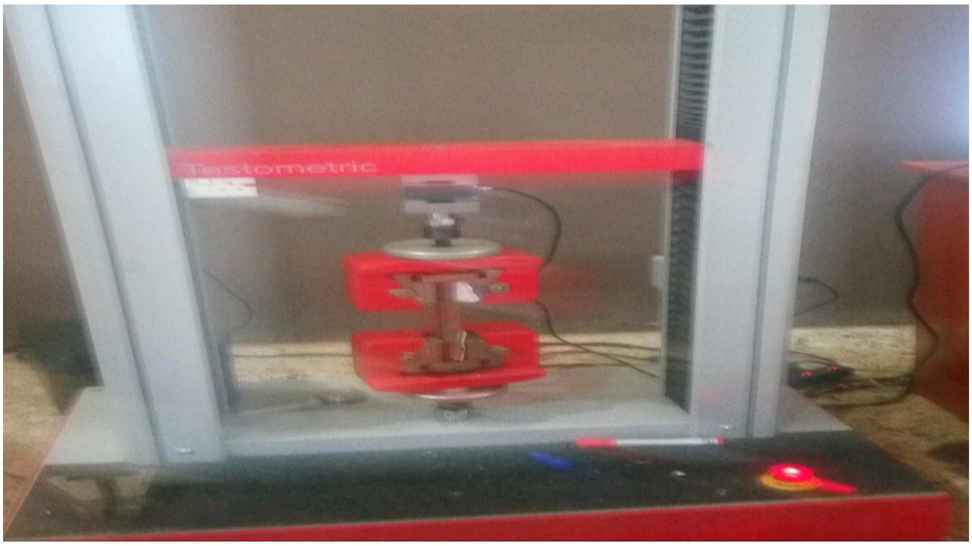

Figure 6: PC-2000 Testometric testing machine

3. Results and Discussion

The morphology of the ESAnp by TEM is shown in Figure 7. 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 is 91.34nm was obtained.

It was observed that the micro-analysis of the EDS of the ESAnp revealed the presence of C, Al, Ca and O. The higher peak of calcium (Ca) and in Figure 7 was as a result of the fact that the major constituent of ESAnp which is calcium. Also the presence of high peak of oxygen (O) may confirm that the various elements in the egg shell ash and palm kernel ash are not pure. The high carbon obtained in both samples is due to the effect of carbonization at high temperature. This is similar to the work of Hassan et al [9].

Figure 8 displayed the percentage by weight gain of the coating samples, while figure 9 gives the coating thickness of the coated samples. In figure 8, it was observed that the weight increases as the wt% of ESAnp was increased, that is weight gained of: 1.92, 2.36, 2.38 2.71, 2.83% was obtained with epoxy/ESAnp coating. Similar pattern was obtained for the coating thickness. A 99.6, 130, 135, 170, 184.6 µm was obtained with epoxy/ESAnp coating. Increases in both weight gain and coating thickness could be attributed to the facts that ESAnp was able to cover the surface of the mild steel.

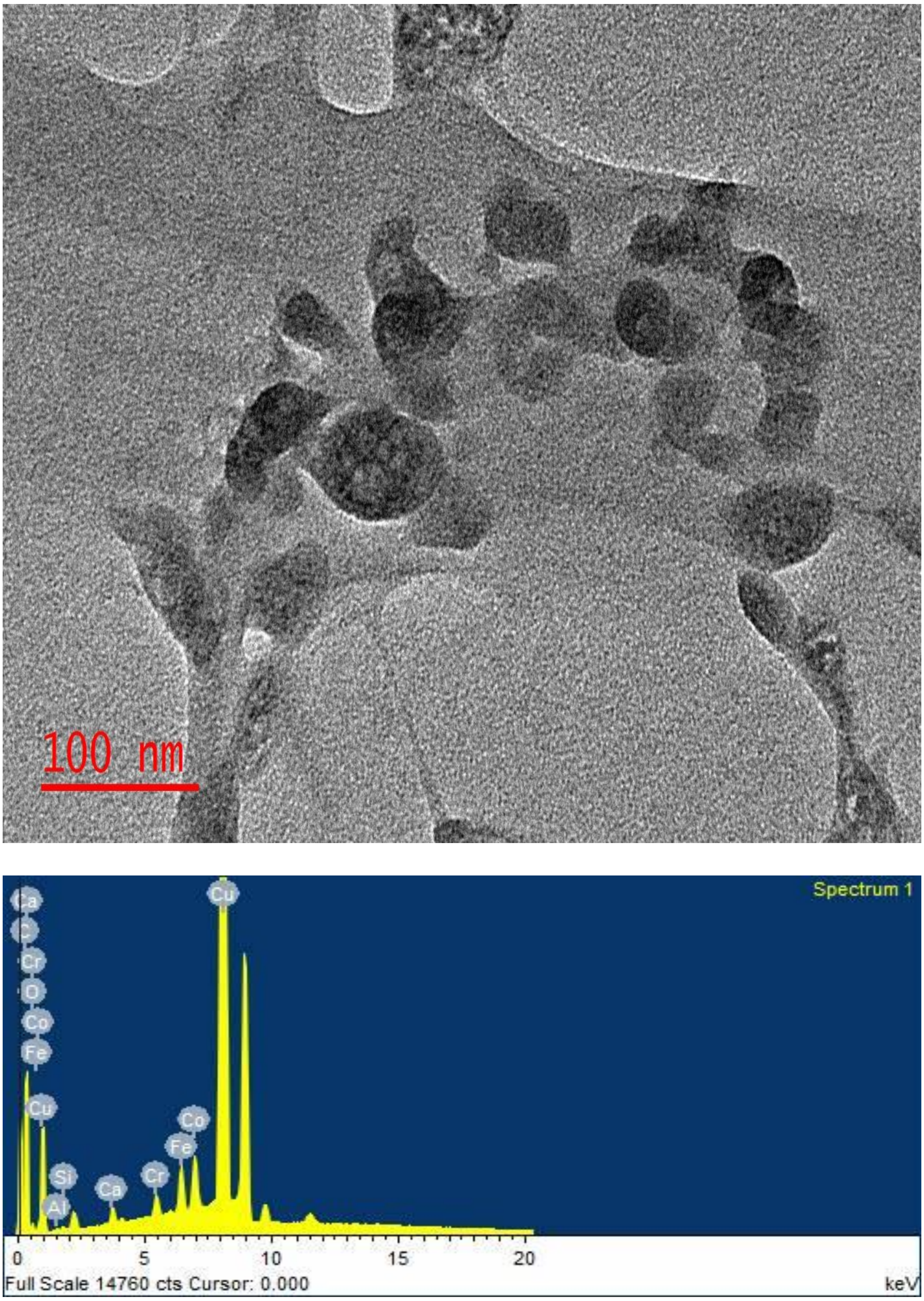

Figure 7: TEM/EDS of the Microstructure of the ESAnp

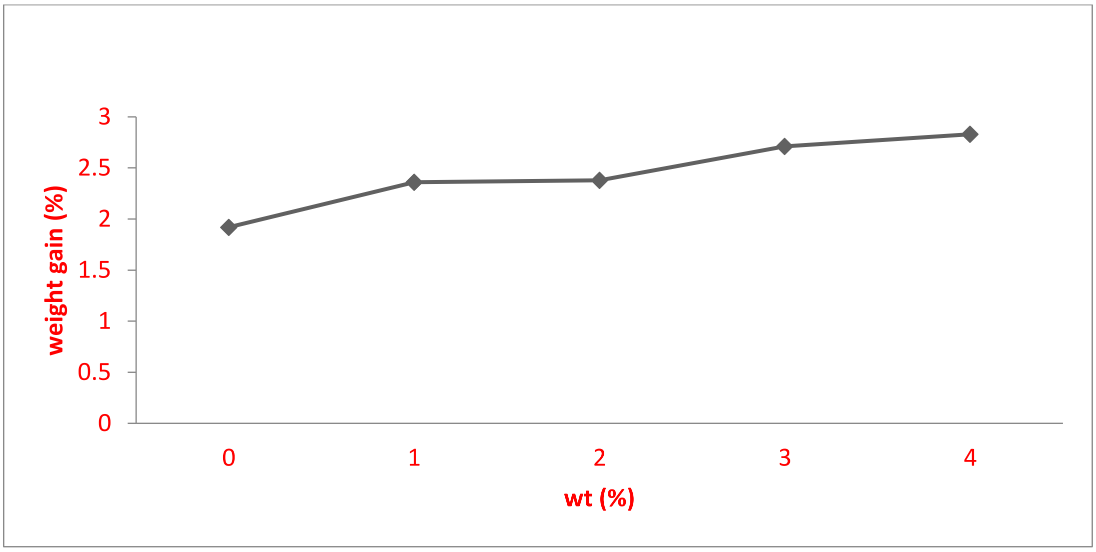

Figure 8: Percentage weight gain of the sample

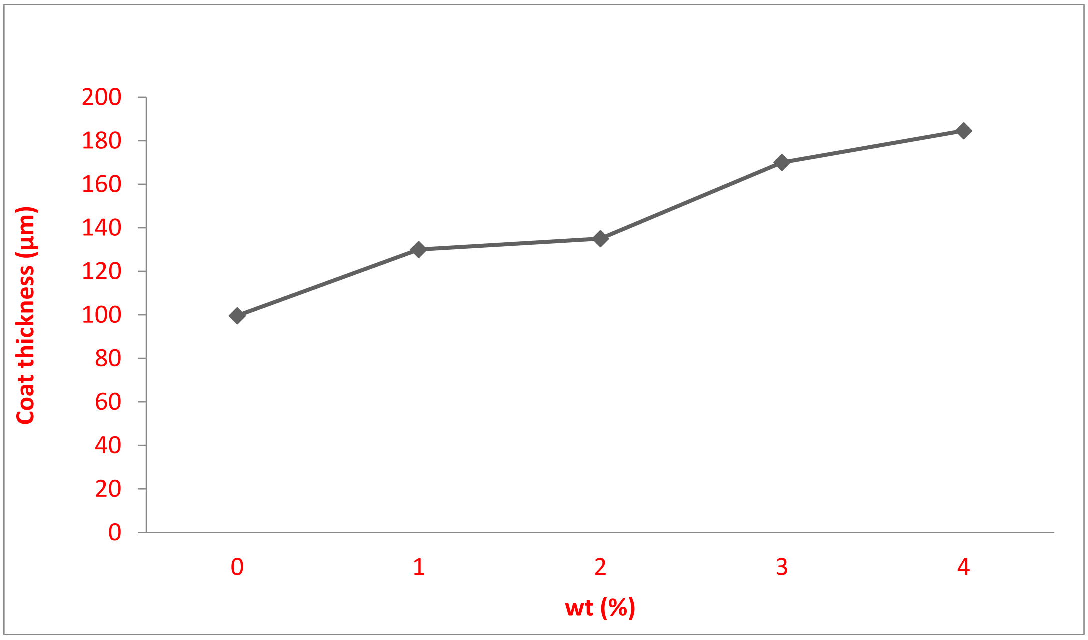

Figure 9: Coating thickness of the sample

The chemical composition of Egg shell ash nanoparticles was determined by X-ray fluorescence to identify the chemical makeup as represented in Table 2.

Table 2: Chemical constituents of the nanoparticles

Compounds	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**CaO**	**MgO**	**Na_2O**	**K_2O**	**L OI**
Egg shell ash nanoparticles	5.89	1.23	0.01	85.56	1.67	0.50	0.13	4.02

The X-ray fluorescence investigation verified that CaO has the highest percentage (85.56%) of the constituent of the Egg shell ash nanoparticles because Calcium is the major constituent of egg shell. The Eggshell ash nanoparticles also has a reasonable amount of SiO_2 (5.89%) and MgO (1.67%). The presence of other compounds such as K_2O, Na_2O, Fe_2O_3 contains percentage less than one.

The SEM analysis in Figure 10 shows the structure of the mild steel, the scratches on the surfaces shows the lines made by the abrasive cutter. No structure was observed. Figure 11 shows the SEM of the coated sample. From Figure 11, one can observe morphological differences of the structure when compared with the SEM of the substrate. It was observed in Figure 11, that with the coated sample of ESAnp, 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 ESAnp. 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 good adhesion that occurred within the interface is as a result of the stirring and spraying applied during production. Similar observation was obtained in the work of Khodair et al [10].

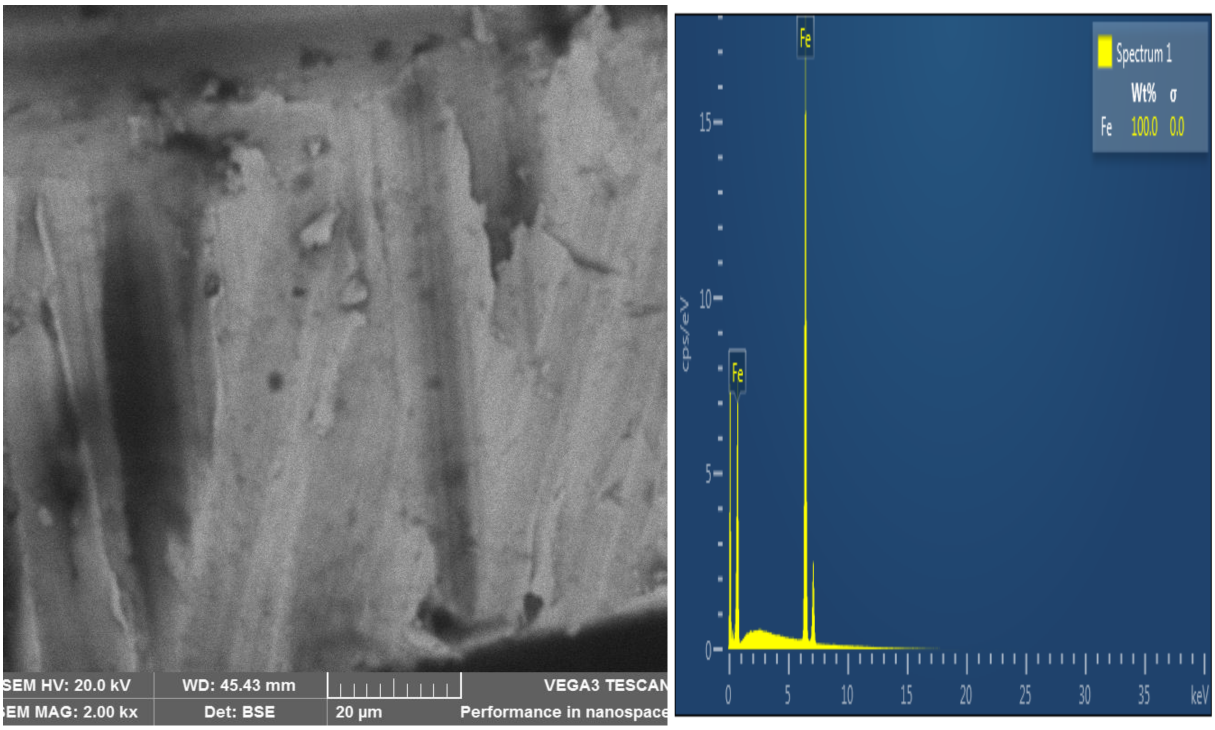

Figure 10: SEM/EDS of the mild steel

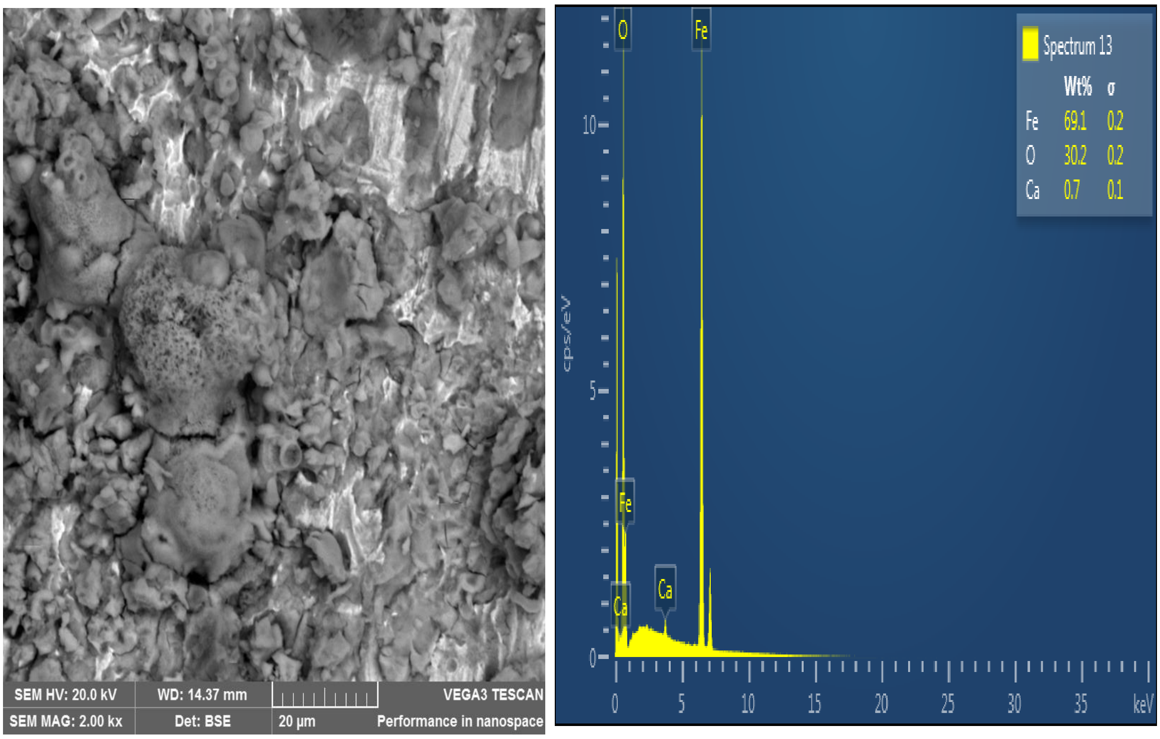

Figure 11: SEM/EDS of the epoxy-4wtESAnp coated mild steel

From the energy dispersion spectrum (EDS analysis) of the composite coating, it was observed in Figures 10 and 11 that there is great difference in the EDS of the substrate from that of the coated samples. The EDS of the mild steel revealed high peak of Fe (Figure 6). The EDS of the ESAnp coated samples have high peak of Ca (Figure 11). This result was at par with the work of Tolumoye et al [11].

Figure 12 displayed the results of the hardness values. From Figure 12, it was clearly seen that the hardness values of the mild steel improved with increase in the wt% ESAnp. Values of 90, 94, 96.7, 102.3 and 127.7HRB were obtained for epoxy-ESAnp at 0, 1, 2, 3 and 4wt%. Improvement of 43.48 % was obtained for the hardness.

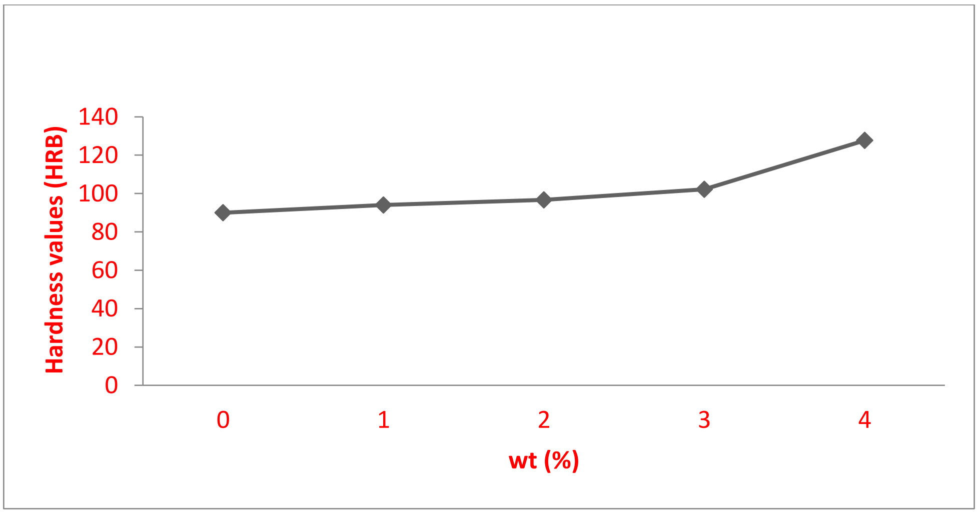

Figure 12: Variation of Hardness values with weight %

The adhesion strength is displayed in Figure 13. It was seen that adhesion strength rises as the wt% ESAnp increases in the epoxy. For example, the values of values of 5.32, 7.303, 9.016, 12.32 and 15.45MPa were obtained for epoxy-ESAnp at 0, 1, 2, 3 and 4wt%. Improvement of 190.4%. This increase in the adhesion strength can be attributed to the good interfacial bonding between the coating materials and the mild steel. 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.

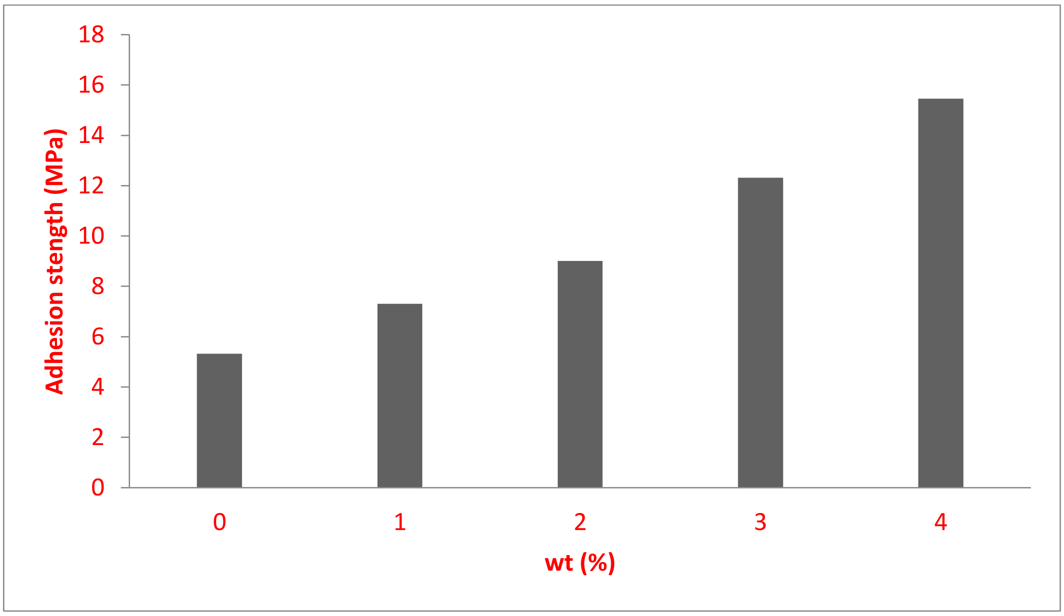

Figure 13: Adhesion strength with weight % coating formulation

4. Conclusion

The results obtained showed that coating thickness increases with increases in wt% of ESAnp. The adhesion strength rises as the wt% ESAnp increases in the epoxy with an improvement of 190.4%. 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. A 43.48 %. improvement of hardness values of the mild steel was obtained at 4wt%ESAnp. It has been established that enhanced hardness values and adhesion strength can be obtained with the formulation.

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