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SCANNING ELECTRON MICROSCOPY AND ENERGY DISPERSIVE XRAY SPECTROSCOPY ANALYSIS OF EPOXY-RICEHUSK ASH COMPOSITE

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ABSTRACT

This study presents analysis of microstructure and elemental composition of epoxy-rice husk ash composite using Scanning electron microscope with an attached energy dispersive x-ray spectrophotometer (SEM-EDX) The Scanning Electron Microscopy (SEM) analysis showed that interfacial interactions and adequate dispersion existed between the rice husk ash particles and the epoxy matrix. Energy Dispersive X-ray Spectroscopy (EDX) analysis indicated that interfacial reactions existed between the epoxy matrix and the rice husk ash particles because the composites did not contain homogenous elements. However each of the composites contained C,O ,Si and Cl while the cast neat epoxy(control) contained C,O and Cl. Rice husk ash was observed to be porous giving valuable knowledge on its application in mechanical interlocking and composite material. The SEM-EDX analysis has given useful information on the microstructure –property relationship of the composites, adding to the advancement of green materials for various applications.

Keywords: Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDX), Rice Husk Ash (RHA), Microstructure, Epoxy.

1. INTRODUCTION

The encyclopedia of life support defined composite material as a multiphase material in which the phase distribution and geometry have been controlled in order to optimize one or more properties. The intention of producing a composite material is to make a material that combines the best properties of the components whilst eliminating any poor properties. Composites are also materials that comprise strong load carrying materials known as reinforcement embedded in a weaker material known as matrix (Askeland, 1994).

Reinforcement provides strength and rigidity, helping to support structural load. The matrix or binder (organic or inorganic) maintains the position and orientation of the reinforcement. Rice husk is an agricultural by product from the rice mill. It constitutes about 20% of the weight of rice. Rice husk contains about 50% cellulose, 25%-30 lignin and 15-20% silica (Abubaker et al, 2010). Microstructure is the microscopic description of individual constituents of a material. Microstructure studies of composites show what happens at the atomic and microscopic levels of the interface.

Electron microscopy analysis is used to characterize the microstructure of composites. Scanning electron microscopy shows the morphology and topography of the composites while compositional analysis is conducted using energy dispersive x-ray spectroscopy. Singla and Chawla (2010) applied scanning electron microscopy in the study of mechanical properties of epoxy-fly ash composites. The scanning electron microscope observations showed uniform distribution of fly ash in the matrix and good adhesion.

Energy dispersive x-ray spectroscopy is a chemical micro analytical technique used in conjunction with scanning electron microscopy to determine the elements in the microstructure of a material. Asaka et al (2004) analyzed resin composites with EDX and SEM and discovered that the main element in the resin composite was silicon. The SEM observations showed three different types of filler morphology: - splintered, splintered and pre polymerized and spherical.

Most developing countries like Nigeria are not yet properly utilizing agricultural wastes such as rice husk ash for gainful engineering production. Rice Husk Ash (RHA) constitutes environmental pollution and causes health hazards like silicosis, cancer, tuberculosis, chronic cough and sight disorder in areas where it is dumped. Therefore there is need to develop more ways of reducing the amount of the waste in the environment. One of the easiest ways of solving the problem is to use rice husk ash as filler in epoxy matrix to form epoxy-rice husk ash composite. Microstructural study of the effect of interfacial adhesions between particle and matrix is fundamental in understanding mechanical

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behavior of polymer composites. The knowledge of microstructure and mechanical properties of particle filled composites is vital in describing the behaviors at the interface and the effect of forces on the composites.

1.1. Aim and Objectives

The aim of the study is to observe the microstructure of epoxy –rice husk ash composites using Scanning Electron Microscopy and Energy Dispersive X-ray Spectroscopy method. The objectives are to:

1. To conduct scanning electron microscopy and energy dispersive x-ray spectroscopy analysis on epoxy –rice husk ash composite and study the effect of variation of rice husk ash volume fractions on the microstructure.
2. To carry out scanning electron microscopy and Energy Dispersive X-ray spectroscopy on Adani Rice husk ash.

2. MATERIALS AND METHOD

2.1 Materials

The materials used were cast neat epoxy (Sample one 100% Epoxy,0%RHA),Epoxy –Rice Husk Ash Composites prepared at different volume fractions which are: sample two 90%Epoxy 10% RHA, sample three 80% Epoxy 20%RHA, sample four 70% Epoxy 30%RHA,sample five 60% Epoxy 40% RHA and sample six 50% Epoxy 50%RHA

2.2 Method

Scanning Electron Microscopy and Energy Dispersive X-Ray Spectroscopy Analysis.

The SEM & EDX analysis was done at the Advanced Physics Laboratory, Sheda Science and Technology Complex, (SHETSCO) Abuja. The Carle Zees scanning electron microscope with an attached INCA energy dispersive x-ray microanalysis system was used for the analysis. The scanning electron microscope has the microscope column, specimen chamber and vacuum system on the left with the computer monitor and many other instrument controls on the right. The energy dispersive x- ray spectrophotometer attached to the scanning electron microscope has four set ups which are: the beam source, the x- ray detector made of lithium drifted silicon, the pulse processor and the analyzer. The five composite samples of different composition of rice husk ash particle, the cast neat epoxy and the sample of rice husk ash were loaded in the specimen chamber. Conductive double sided carbon tape was used to hold the specimens in order to make them conductive. The cathode and magnetic lenses in the scanning electron microscope created and focused a beam of electrons on each of the specimen. The bombardment of the specimen with scanning beam of electrons generated backscattered electrons and secondary electrons due to beam specimen interaction. The backscattered electrons and secondary electrons were collected, amplified and displayed on a cathode ray tube. The backscattered electrons and cathode ray tube scanned synchronously to produce an image of the surface of the specimen formed. The analytical lithium drifted silicon detector in the energy dispersive X-ray spectrophotometer meter converted x- ray energy into voltage signals which measure the signal and passed them onto the analyzer for elemental analysis and data display. The morphology and elemental composition of the samples were displayed on the computer screen sequentially with the help of the software.

3. RESULTS AND DISCUSSION

3.1 Results and Discussion of Scanning Electron Microscopy (SEM).

Results of scanning electron microscopy are presented in figures 1 to 14

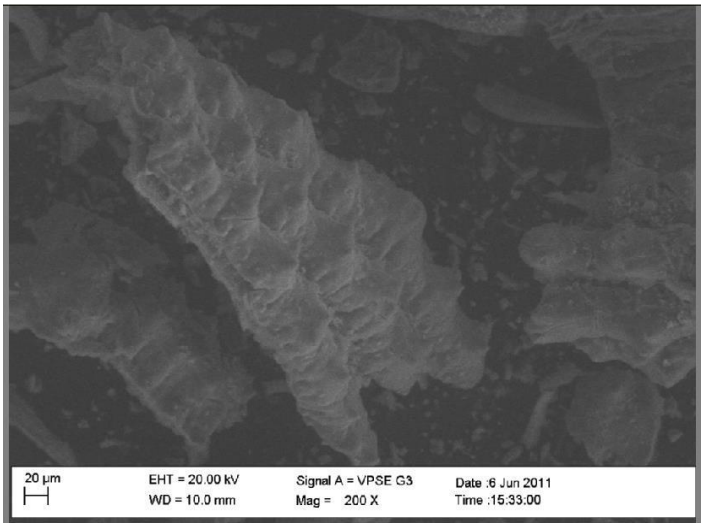

Figure 1: Scanning electron micrograph of epoxy – rice of Adani rice husk ash x200.

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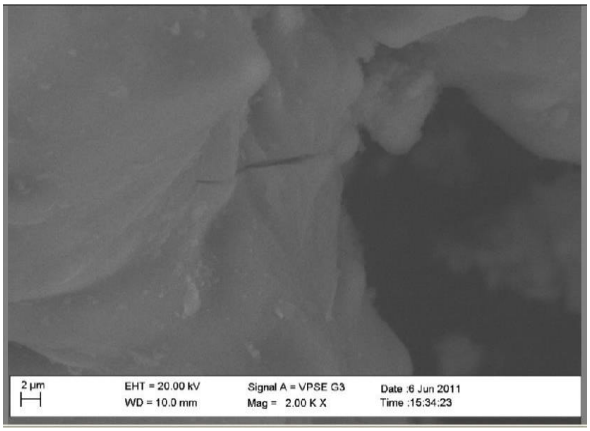

Figure 2: Scanning electron micrograph of Adani rice husk ash x2000.

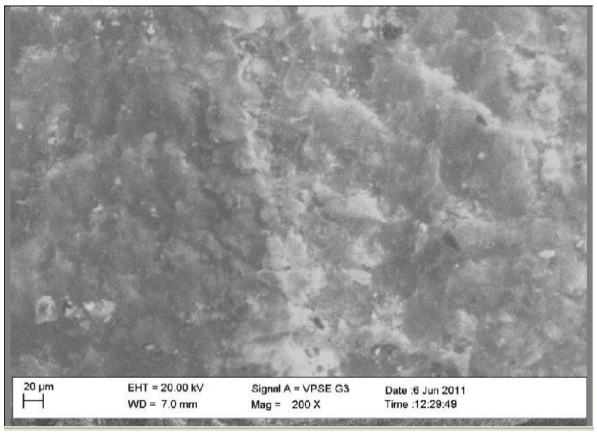

Figure 3: Scanning electron micrograph of cast neat epoxy x200.

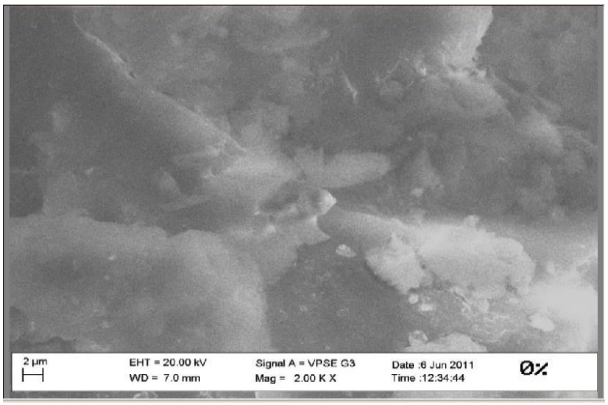

Figure 4: Scanning electron micrograph of cast neat epoxy x2000.

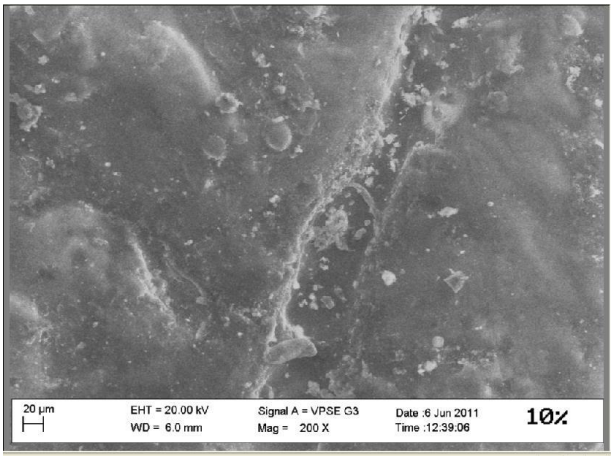

Figure 5: Scanning electron micrograph of epoxy –rice husk ash composite containing 10% rice husk ash x200.

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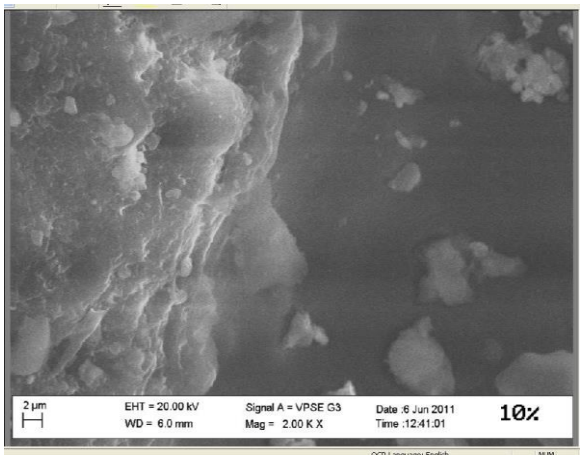

Figure 6: Scanning electron micrograph of epoxy- rice husk ash composite containing 10% rice husk ash x2000.

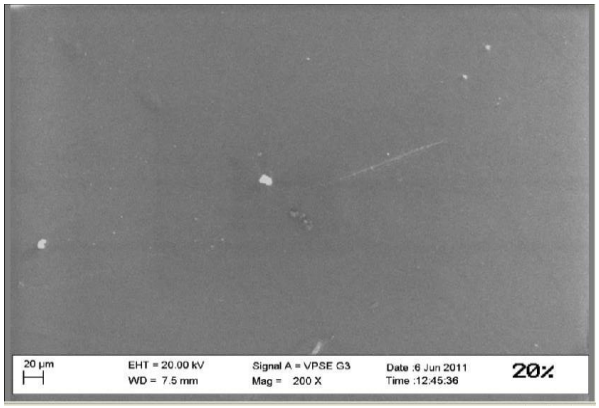

Figure 7: Scanning electron micrograph of epoxy – rice husk ash composite containing 20% rice husk ash x200.

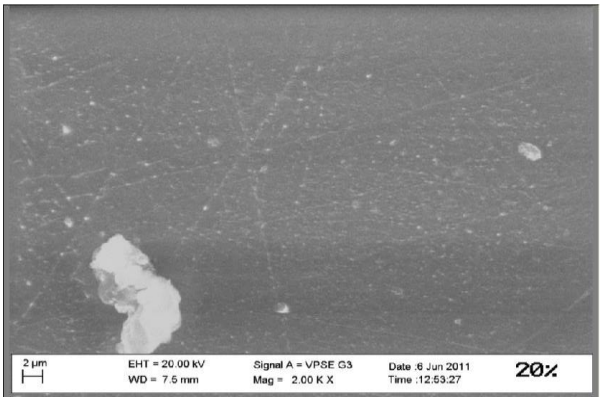

Figure 8: Scanning electron micrograph of epoxy – rice husk ash composite containing 20% rice husk ash x2000.

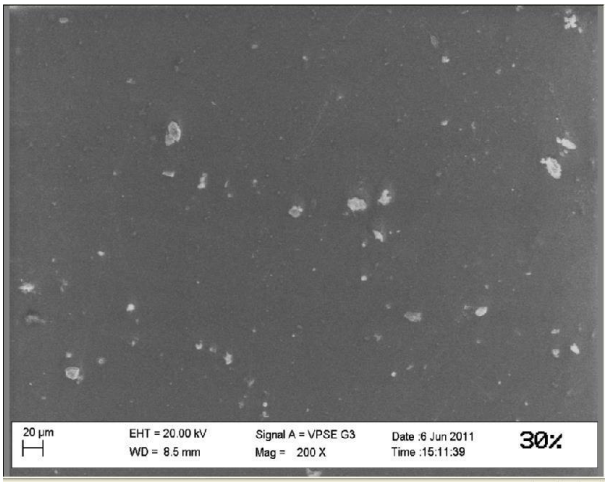

Figure 9: Scanning electron micrograph of epoxy – rice husk ash composite containing 30% rice husk ash x200.

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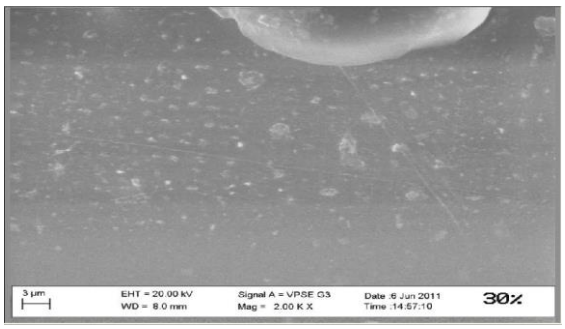

Figure 10: Scanning electron micrograph of epoxy – rice husk ash composite containing 30% rice husk ash x2000.

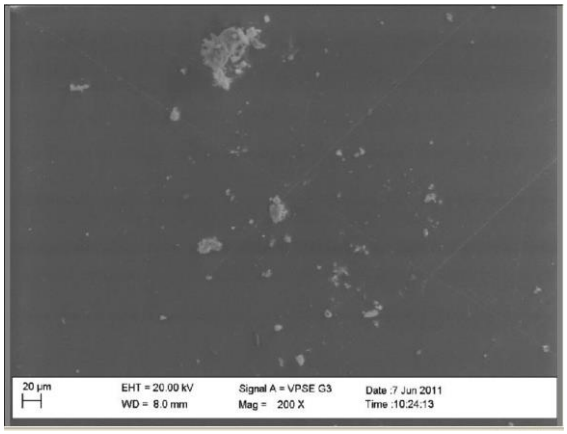

Figure 11: Scanning electron micrograph of epoxy-rice husk ash composite containing 40% rice husk ash x200.

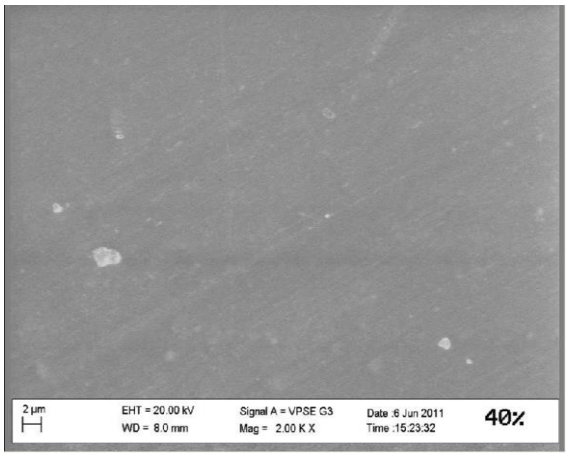

Figure 12: Scanning electron micrograph of epoxy-rice husk ash composite containing 40% rice husk ash composite x2000.

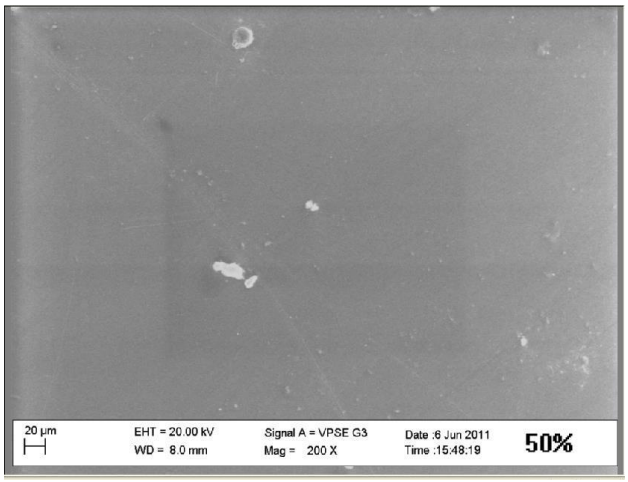

Figure 13: Scanning electron micrograph of epoxy –rice husk ash composite containing 50% rice husk ash x200.

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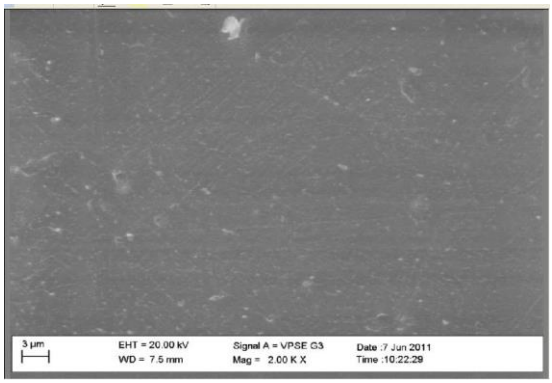

Figure 14: Scanning electron micrograph of epoxy- rice husk ash composite containing 50% rice husk ash x2000.

Scanning Electron microscopy analyses was done to observe the distribution and interaction of rice husk ash fillers in the epoxy matrix and to determine the morphology of the cast neat epoxy and Adani rice husk ash. Two magnifications of 200x and 2000x were used in order to adequately view the specimens. The explanations of the scanning microscopy analysis are given thus:

Figures 1 and 2 show the photomicrographs of Adani rice husk ash at 200x and 2000x magnifications. At 200x magnification the rice husk ash had a porous structure with the particles looking a bit rough and dispersed. The increase in magnification to 2000x gave a hollow structure of the rice husk ash. Nnaji etal, (2024) in their work on characterization pig bone ash and hamburger seed shell wastes from Umuahia,Abia State found that pig bone ash has porous structure.

Figures 3 and 4 showed that the cast neat epoxy had a rough surface and there were poor cohesion between the hardener and the resin in some regions in the microstructure. An observation of the composite with 10% RHA in figures 5 and 6 showed that the surface of the composite was rough and the rice husk ash particles were fairly distributed in the matrix. The rice husk ash particles were interacting at the interface.

Figures 7 and 8 showed the scanning electron microscopy analysis of the composite containing 20%RHA. It can be deduced from the photomicrographs that the composite had a smooth surface with the particles of rice husk ash evenly distributed and there were interactions at the interface. However the particles did not bond well with the matrix. Looking at the microstructure of the composite containing 30% RHA in figures 9 and 10. It can be seen that interfacial interactions existed between the rice husk ash particles and the epoxy matrix. At 2000x magnification the particles formed a cluster at the edge and the particles did not bond well with the matrix. The microstructure appeared strained at 2000x magnification while at 200x magnification a smooth surface was observed.

From figures 11 and 12 the composite containing 40% RHA had a smooth surface at 200x and fairly strained surface at 2000x. The rice husk ash particles were uniformly distributed. Figures 13 and 14 showed the microstructure of the composite containing 50% RHA. It was observed that the rice husk ash was not well distributed in the matrix. At 200x magnification a smooth surface was noticed with clusters of rice husk ash while at 2000x magnification the surface looked a bit strained. Conclusively it was observed in the scanning electron microscopy analyses that interfacial interactions existed between rice husk ash particles and epoxy matrix. Scanning electron microscopy analysis equally showed that there was poor interfacial adhesion (bonding) between the rice husk ash particles and the epoxy matrix in the microstructure and poor dispersion of rice husk ash particles in the epoxy matrix.

3.2 Results and Discussion of Energy Dispersive X-ray Spectroscopy Analysis (EDX).

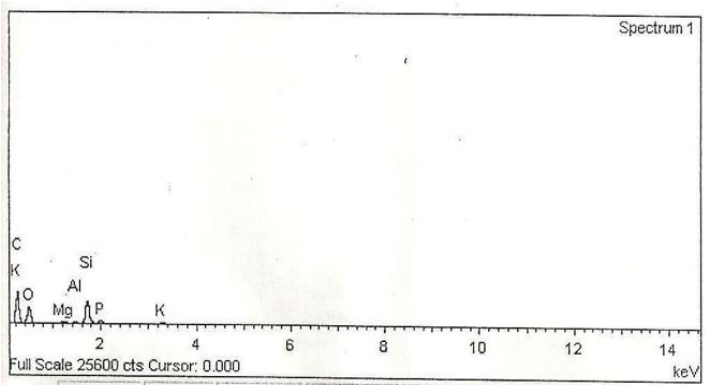

Figure 15: EDX spectrum Of Adani Rice Husk Ash (RHA)

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Table 1: Elemental Composition of Adani Rice Husk Ash.

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
C K	125.27	0.6880	50.27	1.08	59.67
O K	73.53	0.5126	39.61	0.97	35.30
Mg K	2.17	0.7690	0.78	0.07	0.46
Al K	0.89	0.8564	0.29	0.05	0.15
Si K	23.53	0.9232	7.04	0.20	3.57
P K	5.37	1.2144	1.22	0.09	0.56
K K	2.94	1.0221	0.79	0.07	0.29
Totals			100.00		

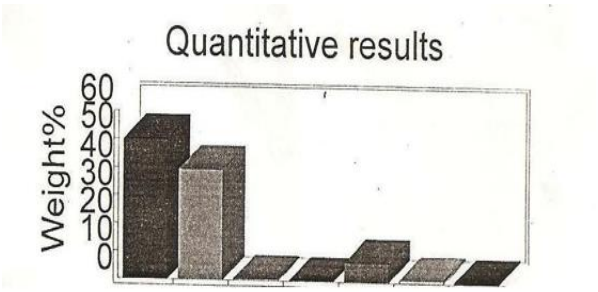

Figure 16: Quantitative Results for Adani Rice Husk Ash

Table 2: Elemental composition of cast neat epoxy.

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
C K	342.57	1.5885	81.35	0.39	85.46
O K	16.44	0.3395	18.27	0.39	14.41
Cl K	0.86	0.8356	0.39	0.04	0.14
Totals			100.00		

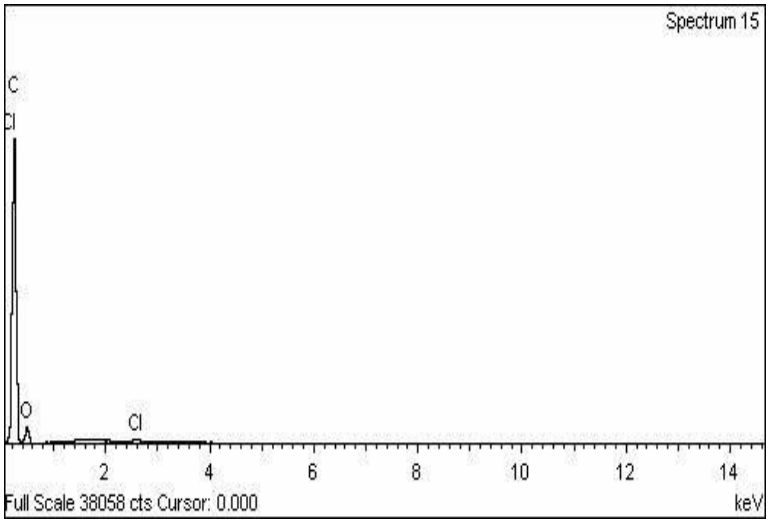

Figure 18: EDX spectrum of Cast neat epoxy.

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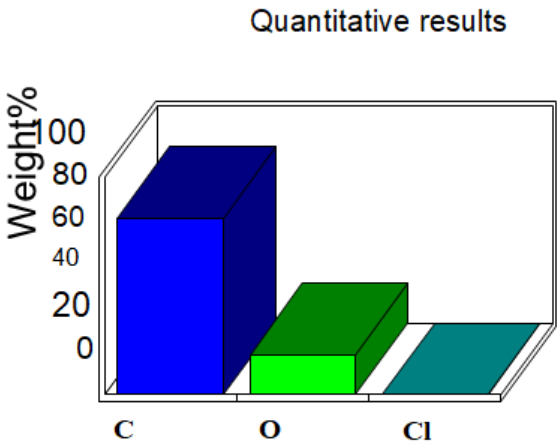

Figure 18 **Quantitative results for cast neat epoxy showing elements**

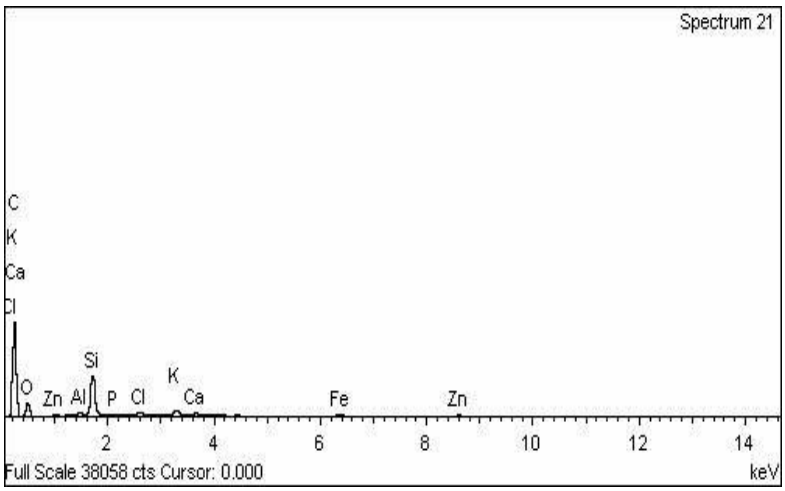

Figure 19: EDX spectrum of epoxy- rice husk ash composite (90% Epoxy, 10% RHA)

Table 3: Elemental composition of epoxy- rice husk ash composite (90% epoxy 10% RHA)

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
C K	112.01	0.7330	64.95	0.72	75.96
O K	19.53	0.3915	21.21	0.57	18.62
Al K	1.08	0.8643	0.53	0.04	0.28
Si K	13.56	0.9253	6.23	0.14	3.12
P K	0.52	1.2301	0.18	0.05	0.08
Cl K	1.51	0.8015	0.80	0.06	0.32
K K	3.08	1.0299	1.27	0.07	0.46
Ca K	1.46	0.9646	0.64	0.06	0.22
Fe K	2.26	0.8001	1.20	0.12	0.30
Zn K	5.26	0.7465	2.99	0.23	0.64
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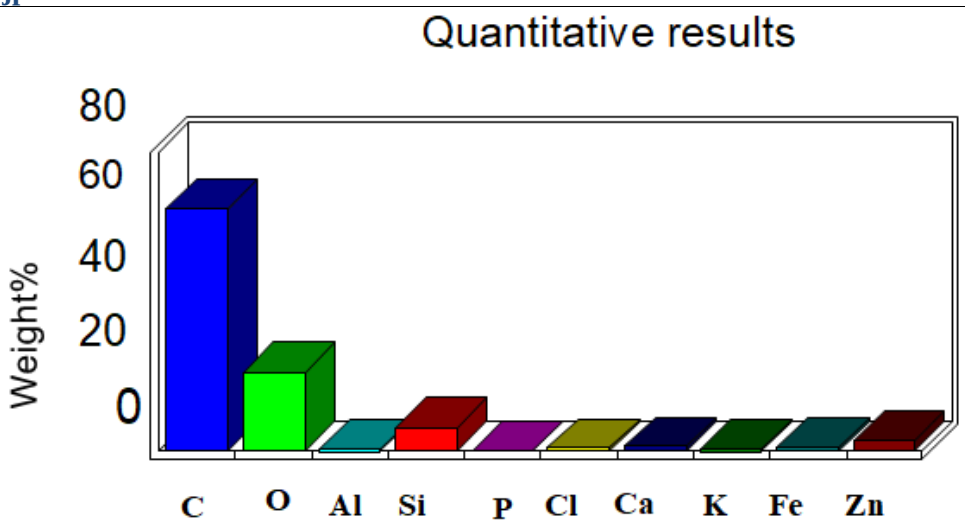

Figure 20: Quantitative results for epoxy – rice husk ash composite (90% Epoxy 10% RHA) $_{showing}$ weight % of elements.

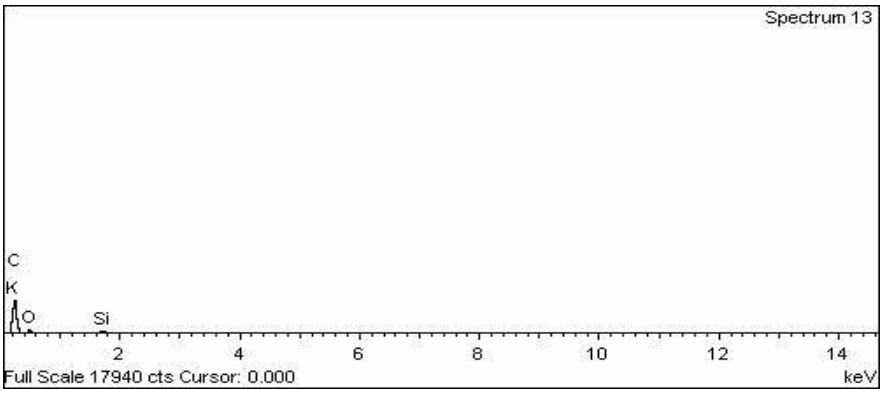

Figure 21: EDX spectrum of epoxy- rice husk ash composite (80% Epoxy, 20% RHA)

Table 4: Elemental composition epoxy-rice husk ash composite (80%Epoxy 20%RHA)

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
C K	226.60	1.2025	71.37	1.51	77.97
O K	25.90	0.3866	25.37	1.48	20.80
Si K	3.83	0.9459	1.53	0.16	0.72
Cl K	1.28	0.8261	0.59	0.14	0.22
K K	0.73	1.0381	0.27	0.13	0.09
Fe K	1.79	0.7767	0.88	0.26	0.21
Totals			100.00		

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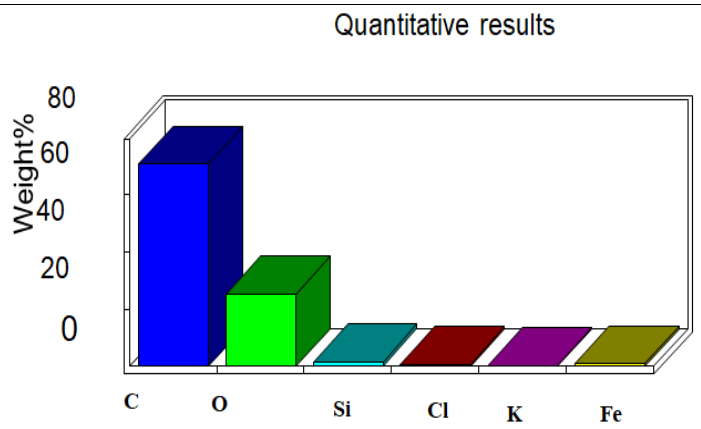

Figure 22: Quantitative results for epoxy – rice husk ash composite (80%Epoxy 20%RHA) showing weight% of elements.

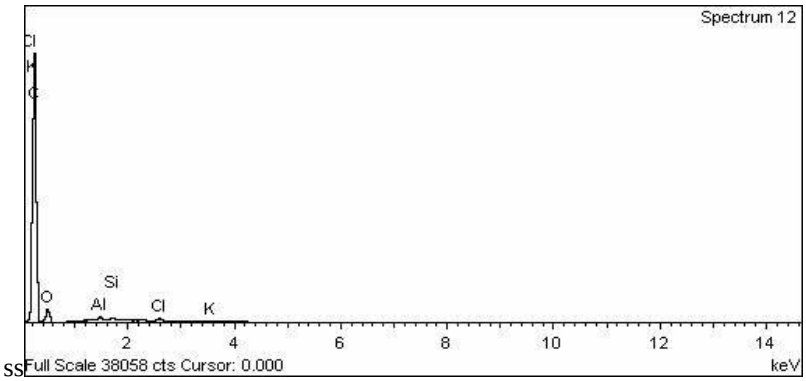

Figure 23: EDX spectrum of epoxy – rice husk ash composite (70% Epoxy, 30% RHA)

Table 5: Elemental composition of epoxy –rice husk ash composite (70%Epoxy30% RHA)

Element	App	Intensity	Weight%	Weight%	Atomic%
	Conc.	Corrn.		Sigma	
C K	286.07	1.5090	80.07	0.41	84.57
O K	15.43	0.3442	18.93	0.40	15.01
Al K	0.75	0.9089	0.35	0.03	0.16
Si K	0.45	0.9585	0.20	0.03	0.09
Cl K	0.72	0.8334	0.37	0.04	0.13
K K	0.21	1.0399	0.09	0.03	0.03
Totals			100.00		

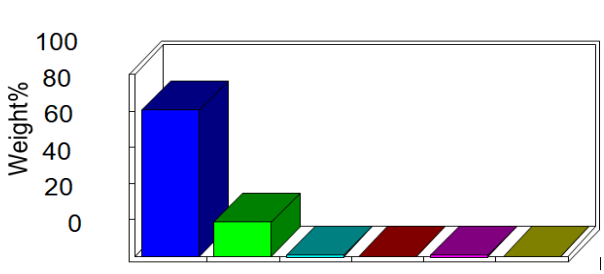

Figure 24: Quantitative results for epoxy –rice husk ash composite (70%Epoxy 30%RHA) showing weight % of elements.

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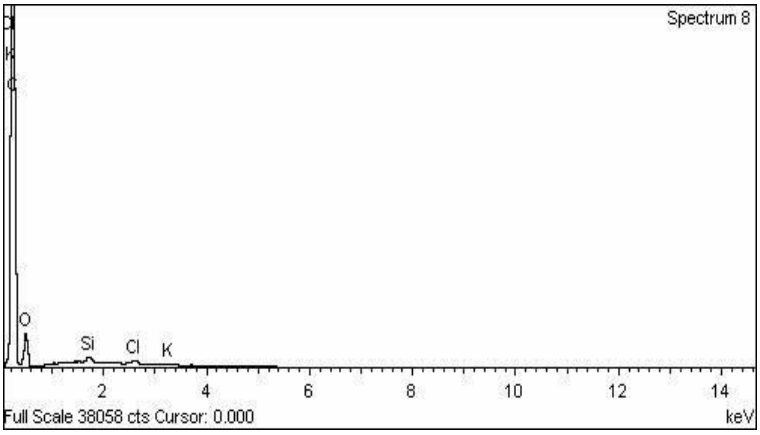

Figure 25: EDX spectrum of epoxy rice husk ash composite (60% Epoxy, 40% RHA)

Table 6: Elemental composition of epoxy –rice husk ash composite (60%Epoxy 30%RHA)

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
C K	307.99	1.5354	78.77	0.29	83.37
O K	18.48	0.3519	20.63	0.29	16.39
Si K	0.76	0.9588	0.31	0.02	0.14
Cl K	0.51	0.8331	0.24	0.02	0.09
K K	0.13	1.0406	0.05	0.02	0.02
Totals			100.00		

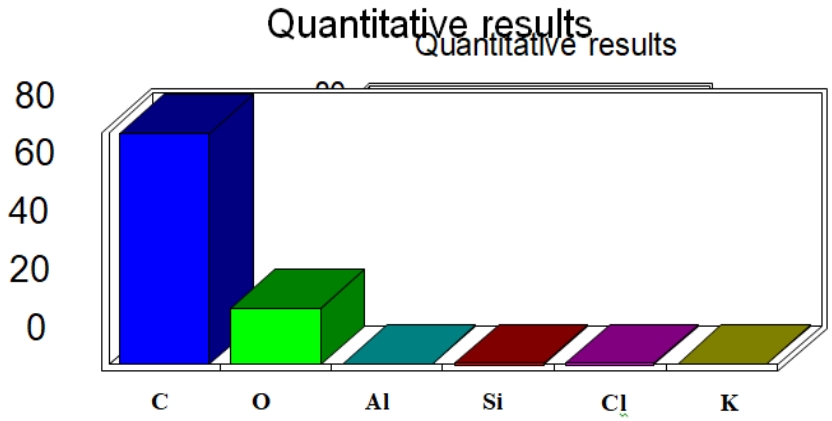

Figure 26: Quantitative results of epoxy –rice husk ash composite (60%Epoxy 40%RHA) showing weight % of elements.

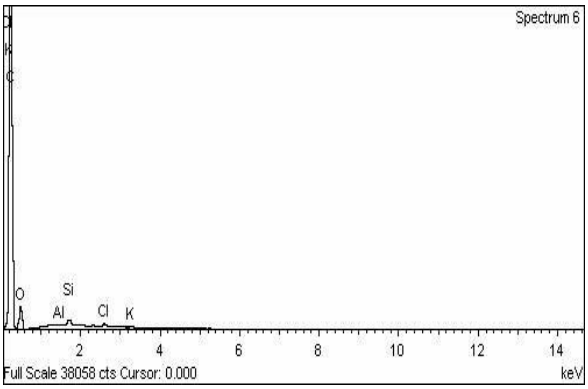

Figure 27: EDX spectrum of epoxy rice husk ash composite (50% Epoxy, 50% RHA)

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Table 7: Elemental composition of epoxy – rice husk ash composite (50%Epoxy 50%RHA)

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
C K	291.96	1.5144	79.69	0.32	84.22
O K	16.29	0.3464	19.43	0.32	15.42
Al K	0.21	0.9071	0.09	0.02	0.04
Si K	1.03	0.9609	0.44	0.03	0.20
Cl K	0.57	0.8328	0.28	0.03	0.10
K K	0.17	1.0400	0.07	0.02	0.02
Totals			100.00		

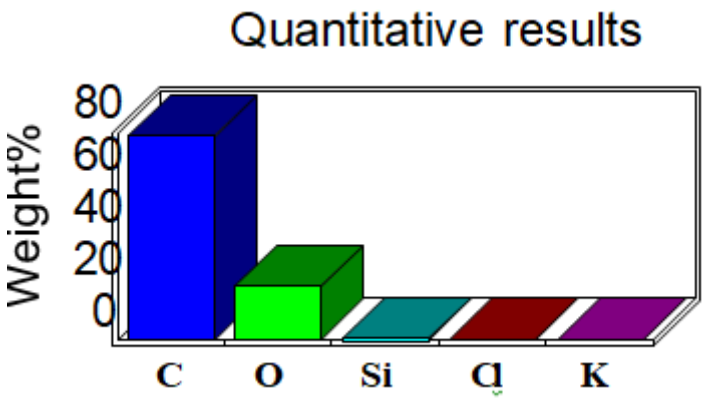

Figure 28: Quantitative results for epoxy – rice husk ash composite (50%Epoxy 50%RHA)

Energy Dispersive X-Ray Spectroscopy Analysis

Energy dispersive x-ray spectroscopy analysis showed the elements in the microstructure of the composites and the cast neat epoxy .The elements and their composition were found from the EDX attached to the SEM with the help of a software. The EDX results showed that interfacial reactions existed between the rice husk ash particles and the epoxy matrix because the composites did not contain homogenous elements in their microstructure. In an EDX result, the EDX spectrum showed the elements and their peaks with corresponding energy on the horizontal line of the spectrum. The elemental composition was shown in the tables. The elemental composition was given in atomic%, weight%, approximate concentration and intensity. Figure 15 and table 1 highlights the EDX results of Adani rice husk ash showing the elemental composition as C,O,Mg, Al, Si,P and K. Carbon has the highest composition which could be attributed these reasons : incomplete combustion due to insufficient oxygen(Mansaray etal.,2015) and presence of organic matter like cellulose, hemicellulose and lignin(Shen etal.,2016) The results of the energy dispersive x-ray spectroscopy analysis for the cast neat epoxy and the composites are explained as follows: Table 2 and figure 17 show the elemental composition and the EDX spectrum of the cast neat epoxy respectively It showed that the cast neat epoxy contained these elements C, O,Cl. Table 3 and figure 19 gave the elemental composition of the composite with 10% RHA as C,O, Al, Si, P, Cl,K, Ca, Fe, Zn. The surface of this composite allowed more reactions between the epoxy matrix and rice husk ash particles which accounted for the highest elements in its microstructure than other composites.(Nnaji etal,2024) Table 4 and figure 21 showed that the composite with 20% RHA contained C, O, Si, Cl, K and Fe. From table 5 and figure 23 the composite with 30% RHA was found to contain C, O, Al, Si, Cl and K.

Table 6 and figure 25 showed that the elemental composition of the composite with 40% RHA is C, O, Si, Cl and K. Table 7 and figure 27 showed that the elements in the microstructure of the composites with 50% RHA are C,O,Al, Si, Cl and K.

It became evident from the analysis that all the composites had C, O, Cl and Si in their microstructure. Nnaji etal (2024) equally observed polypropylene/pigboneash/hamburger seed shell composite to contain C,Si,K,O All the composites had higher composition of carbon in them and their peaks overlapped.

4. CONCLUSION

Successful investigation of the morphology and elemental composition of epoxy-rice husk ash composite was done in this study.

This study showed from the Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDX) microstructural analysis that the microstructure and elemental composition of the composites varied at the interface of the epoxy matrix and rice husk ash particles indicating interfacial interactions. SEM analysis showed that Adani rice husk ash has a porous structure. The investigated microstructures suggests that Adani rice husk ash can be used as adsorbents ,construction materials ,insulation materials while epoxy-rice husk ash composites can be applied in coatings/adhesives and automotive parts.

5. RECOMMENDATIONS

1. Further studies should be done on the application of rice husk ash as adsorbents and water treatment.

(2) Utilization of natural abundant materials like rice husk ash for production should be explored to save cost due to its sustainability.

(3)Industrial uses of epoxy –rice husk ash composite like in construction, automotive, plastic should be investigated.

6.0 Contributions to Knowledge

(1) SEM –EDX analysis of Adani rice husk ash has shown the structure and elemental composition providing insights to its potential application

(2) EDX analysis on the epoxy-rice husk ash composites have revealed that Si,O and C are there key elements.

(3) SEM analysis has shown the morphology and distribution of RHA particles in epoxy matrix clearly.

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