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## MECHANICAL PROPERTIES OF EPOXY MATRIX COMPOSITES REINFORCED WITH GREEN SILICA PARTICLES

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**Abstract:** Recent developments indicate that a significant improvement in mechanical properties of thermosetting polymers may be obtained by using submicron inorganic fillers. Submicron silica particles with average particle size of 0.5  $\mu\text{m}$  was extracted from rice husk ash (RHA) by sol-gel process and used as reinforcement for epoxy resin. Composites were developed by incorporating the silica particles in the weight fractions of 0.5, 1, 2, 3, 4 and 6wt% in an epoxy resin. Mechanical properties (tensile, flexural, hardness) of the developed composite was investigated. Silica particles incorporation shows beneficiary effect on the modulus of elasticity, flexural modulus and hardness with threshold value experienced at 2 wt%. However tensile strength and flexural strength were found to decrease upon silica addition, the flexural strength values being somewhat higher than their tensile counterparts. SEM images of the specimens revealed the manner of silica dispersion in the matrix. Silica particles extracted from rice husk ash can be used as good reinforcing filler for epoxy resin; However, a compromise must be reached in deciding the content of rice husk silica to be incorporated so as not to sacrifice too much of its tensile and flexural strength.

**Keywords:** Silica, Submicron, Mechanical properties, Epoxy resin, Rice husk ash, Sol-gel process, Rice husk silica

### 1. INTRODUCTION

As advanced engineering materials, composites are used in many applications where excellent performances under hard working conditions are required. Such materials must provide unique mechanical and tribological properties combined with a low specific weight and a high resistance to degradation in order to ensure safety and economic efficiency [1]. Polymers often have advantages over other materials such as metals and ceramics. They are widely used in various technical applications because of their unique advantages such as ease of production, light weight and ductility. Polymer composites have been extensively studied over a long period of time. They are generally organic polymer composites mostly filled with inorganic fillers. Their properties combine the advantages of the inorganic filler material (rigidity and thermal stability) and of the organic polymer (flexibility, ductility and process ability [2]. It is in the view of these expected roles of the organic polymer that epoxy resin was used in this study. The processing of polymer matrix composites need not involve high pressure and doesn't require high temperature. Also equipment required for manufacturing polymer matrix composites are simpler [3].

Several methods have been used to recover silica gel particles from rice husk ash. Acid leaching, gasification, microwave heating, magnesio-thermic reduction and sol-gel methods have all been investigated for recovering silica from rice husk [4,5,6,7,8]. However, sol-gel process consumes low energy and is cost effective compared to the current metallurgical methods.

Epoxy resins have been widely used as impregnating materials, adhesives, or matrices for composites because of their good electric insulating, good chemical resistance, and low shrinkage during cure, good thermal characteristics, and ease in processing. However, the major problems with the epoxy resins for engineering applications are their low stiffness and average mechanical properties when compared with metals. Reinforcement agents such as coconut-shell powder [9], Portland cement and potassium titanate whiskers have already been successfully used to overcome these problems by incorporating it into the epoxy resin matrix. [10, 11]





Rice husk is an agro-waste product produced in large quantities in Nigeria and its being burnt off or dumped in water bodies thereby causing environmental pollution. The utilization of agro-waste products would help solve the problem of environmental pollution which they constitute. It will also serve as a means of turning waste to wealth by utilizing agro-waste products in developing a low cost epoxy resin composite to serve a number of interesting engineering applications. Having discovered from previous works that particulate fillers are very good in reinforcing and enhancing the properties of polymer matrices, it is therefore imperative that we utilize waste agricultural products (e.g. rice husk) and extracts obtained from them (e.g. rice husk silica particles) for material development and engineering applications.

Hence, the main objective of the present study is to evaluate the mechanical properties of an epoxy resin composite reinforced with rice husk silica (RHS) particles.

## 2. MATERIALS AND METHODS

### ☐ Materials

Silica powder of about 0.50  $\mu\text{m}$  particle size which was extracted from rice husk ash was used for this work. The rice husk which was burnt ash was collected from a rice processing mill at Army Barracks along Ondo road, South-West, Nigeria. The Epoxy resin along with its curing hardener (SL 1000 grade) was obtained from a chemical store in Lagos, Nigeria. The reagents (Hydrochloric acid & Sodium hydroxide) and ashless filter paper were purchased from Pascal Chemical Ltd, Akure, Ondo state, Nigeria.

### ☐ Silica Powder Extraction

The silica gel was extracted from RHA as described by Daramola *et al.*, (2015b) [12]. 80g of RHA was added to sodium hydroxide (NaOH) solution with a concentration and volume of 2.5 M and 990 ml respectively in a beaker. The beaker containing the mixture was placed in a water shaker bath and heated at 100 °C for 1 hour. The solution was then allowed to cool to room temperature and then filtered through a Whatman No. 42 ashless filter paper. The filtrate and the carbon residue were both collected in separate containers. Concentrated Hydrochloric acid was then added to the obtained filtrate and then stirred continuously. The PH of the solution was checked until a value of 7.0 was reached to allow precipitation of silica gel. Ageing was done for 48 hours to promote silica gel formation. The silica gel produced was separated from the soluble solution with the aid of the vacuum filtration pump. The silica gel was dried in an air blast oven for 48 hours. The obtained white silica gel was then pulverized into silica powder. Figure 1 shows the pulverized rice husk silica powder.



Figure 1: The Rice Husk Silica Powder (RHSP)

### ☐ Chemical Composition Analysis of the Rice Husk Silica Powder (RHSP)

The extraction yield and purification parameter of rice husk silica from the rice husk ash (RHA) was estimated using the Energy Dispersive X-ray (EDX) spectrometer.

### ☐ Particle Size Analysis

The particle size of the rice husk silica powder was analysed using Horiba dynamic light scattering (DLS) particle size analyser. About 0.5 g of the silica powder was dispersed in deionised water in the sample dispersion unit of the instrument, vigorously mixed for about two (2) minutes at speed of 2100 rpm, and sonicated for 45 seconds. The ultrasonic waves were used to break or minimise any particle agglomerates that may be present in the suspension. Measurements were taken and the diffraction data and graphs recorded by the software program of the equipment.

### ☐ Composite Production

Manual mixing method and hand lay-up (open moulding technique) were used for the composite production. The production was carried out at room temperature. The composite samples were prepared using the 0.5, 1, 2, 3, 4 and 6 wt. % fractions of the rice husk silica powder.

The matrix material (epoxy resin and curing hardener) was prepared in the ratio of 2 parts of epoxy resin to 1 part of the curing hardener (2:1). The silica powder was first added to the epoxy resin and mixed thoroughly before adding the hardener. All forms of weight measurements were carried using an electronic weighing balance





The mixture was stirred to ensure homogeneity and poured into an aluminium mould pre-coated with a lubricant to aid easy removal of the cured samples. For neat epoxy cast sample, only the epoxy and hardener was mixed and poured into the mould. Levelling was done to uniformly fill the mould cavity. The mixture was then left at room temperature to cure for two hours before removal of the composite samples. Table 1 shows the proportion of the constituents utilized in the composite production.

Table 1: Composition, designation and weight of the reinforcement and matrix used for the fabrication of the composite samples (Tensile, Flexural and Hardness samples)

Composition	Designation	Reinforcement(g)	Resin(g)	Hardener(g)
Neat Epoxy	EPS0	-----	160.0	80
0.5 wt% SiO <sub>2</sub>	EPS1	1.2	159.2	79.6
1.0 wt% SiO <sub>2</sub>	EPS2	2.4	158.4	79.2
2.0 wt% SiO <sub>2</sub>	EPS3	4.8	156.8	78.4
3.0 wt% SiO <sub>2</sub>	EPS4	7.2	155.2	77.6
4.0 wt% SiO <sub>2</sub>	EPS5	9.6	153.6	76.8
6.0 wt% SiO <sub>2</sub>	EPS6	11.4	150.4	70.2

### ☐ Mechanical Testing

Tensile, Flexural and Hardness testing were used to characterize the mechanical properties of the composites produced. Tensile tests were carried out according to ASTM D303-08 standard procedures (2008) [13]. It was performed on flat dog-bone shaped specimens at room temperature using an INSTRON 1195 at a fixed crosshead speed of 10 mm/min. The flexural strength of the composites was evaluated by performing flexural three-point bending tests on the composites. The test was performed at room temperature using a Tensiometric universal testing machine operated at a crosshead speed of 0.3 mm/min. The testing procedure and flexural strength and modulus evaluation were performed in accordance with ASTM D7264M-07 standard (2007) [14]. All the results presented are the average of three individual test samples so as to obtain a reliable data. The composite samples were indented using the INDENTEC hardness testing machine on Rockwell hardness 'A' scale following ASTM .D2240 procedure [15]. The test was carried out by indenting the sample with the instrument for about 5 seconds before taking the reading that was displayed on the calibrated scale. Five repeat indents were carried on each sample and the average value was taken as a measure of the specimen's hardness.

### ☐ Scanning Electron Microscopic Examination

The surface morphology of the samples was studied using a PHENOM PRO 6 Scanning Electron Microscope (SEM) with an accelerating voltage of 15 kV which was also equipped with an EDS for detailed study of the qualitative composition of the composites produced. The fractured surfaces of the tensile-moulded samples were mounted on aluminium stubs and were sputter coated with gold using a QUORUM Q150R gold sputter before being subjected to SEM analysis.

## 3. RESULTS AND DISCUSSION

### ☐ Chemical Composition of the Rice Husk Silica Powder

EDS analysis was to confirm the presence of silica, ascertain the silica extraction yield and evaluate the purification parameter effectiveness. Figure 2 shows the EDS spectrum and spectrometric data of silica particles extracted from RHA with 2.5M NaOH concentration. Peaks of silicon and oxygen alongside small concentrations of chlorine, sodium and potassium were observed. The silicon and oxygen peaks confirm the presence of silica. The spectrometric data however shows that the content of silica is 96.28 wt % and 97.80 atm %.

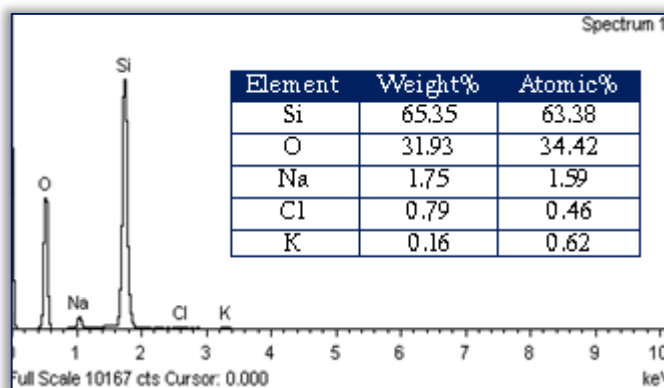


Figure 2: EDS spectrometric data of silica extracted from RHA with 2.0 M NaOH

### ☐ Particle Size Analysis

The chemical composition of silica powder extracted from RHA has been shown to compose of mainly silica (SiO<sub>2</sub>). The particle size of filler materials usually has a significant effect on the properties of the composite produced. Hence, the cumulative particle size distribution of the silica powder extracted was





analysed with Horiba dynamic light scattering particle size analyser and is shown in Figure 3. The particle size distribution of the rice husk silica powder is approximately  $0.50\ \mu\text{m}$ .

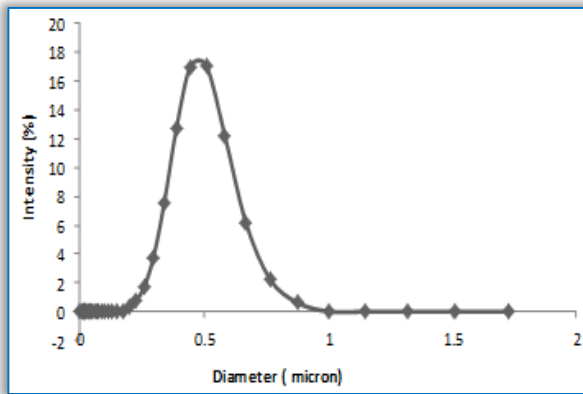


Figure 3: The Particle Size Distribution of the Rice Husk Silica powder (RHS) with average particle size of  $0.5\ \mu\text{m}$

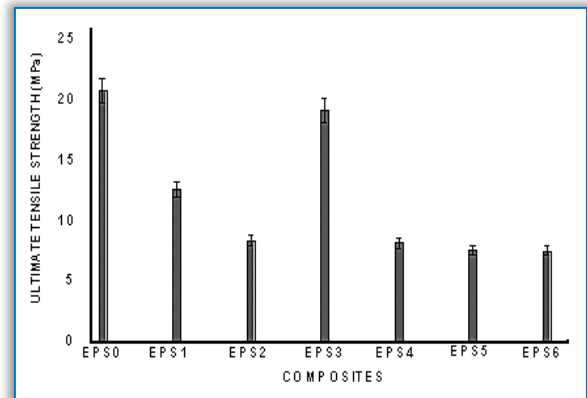


Figure 4: Variation in Ultimate Tensile strength of the Neat Epoxy and Developed Composites

#### ☐ Tensile Properties - Ultimate Tensile Strength

The ultimate tensile strength is the maximum **stress** that a material can withstand while being stretched or pulled before failing or breaking. Figure 4 shows the variation of the tensile strength for the neat epoxy and the composite samples.

#### ☐ Composites

The ultimate tensile strength of the neat epoxy (EPS0) is higher than that of the composites indicating that the addition of silica particles decreases the tensile strength of the matrix. Similar results were obtained in other works where particulate composites were developed [9, 16, 17]. The decrease in tensile strength of the epoxy/silica composite can also be attributed to the power law in case of poor matrix/filler bonding [18]. EPS3 showed the highest tensile strength (19.2MPa) amongst other composite samples and afterwards, a linear decrease in tensile strength was noticed as the rice husk silica content increases. This is attributed to agglomeration of silica particles at higher loading which led to poor interfacial bonding at the matrix-particle interface.

#### ☐ Modulus of Elasticity

Young Modulus is the ratio of the stress to strain in the linear region of the stress-strain curve. It is also the stiffness of a material at the elastic stage of a tensile test. Modulus of Elasticity for the Epoxy/SiO<sub>2</sub> composite samples are shown in Figure 5. The composite sample with 2wt% SiO<sub>2</sub> revealed the best modulus. In general, all the composite samples produced possess higher modulus than the neat epoxy samples since hard dispersed phases possess higher values of stiffness and rigidity than the matrix material which is in agreement with Fu *et al.*, (2008) [19]. The drastic reduction of the stiffness values between 2-3 wt% SiO<sub>2</sub> can be attributed to lowering in cross linking density caused by increasing the silica particles. Due to the type of loading condition, the epoxy matrix around these silica particles breaks quickly due to the high stresses, which resulted in the decrease in tensile strength of the composites. The decrease in tensile strength however leads to an improvement in the young modulus of the neat epoxy matrix. It is understandable that a highly stiffer material such as silica, when added to relatively softer resin such as epoxy, will contribute to the improvement of the young modulus value of the composites. This is in good agreement with Mudr diet *al.*, (2014) [11].

#### ☐ Flexural Strength

The variation in the flexural strength of the composites as well as the neat epoxy is shown in Figure 6. The highest flexural strength (33.46MPa) was observed in EPS1 which is a 24% improvement over the strength of the neat epoxy (EPS0). This increment is due to the lower particle loading and even

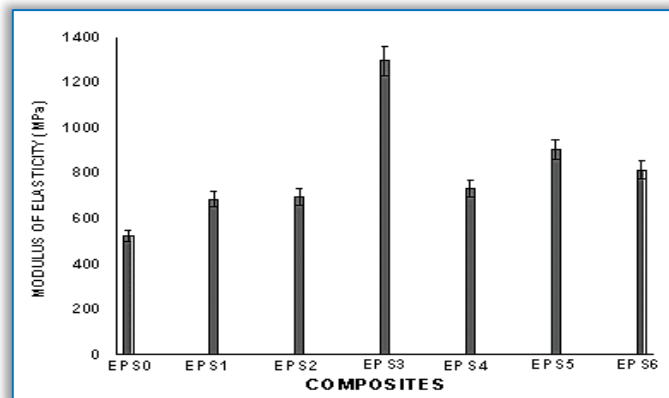


Figure 5: Variation of Young Modulus of Neat Epoxy and the Composite samples





dispersion of the particles in the matrix. By increasing the filler content up to 3wt% SiO<sub>2</sub>, a gradual decrease in flexural strength was experienced. This can be defined by the fact that overloading the resin phase with the silica particles will increase the viscosity of resin phase that subsequently reduce its flexural strength. As the filler content increased up to 6wt %, the flexural strength improved but was still lower than that of the neat epoxy (26.98MPa). The influence of rigid and hard fillers on the stress-stain behaviour of polymers under flexural loading is a well-known phenomenon. Rigid fillers commonly increase the stiffness, but on the other side they may have a devastating effect on the flexural strength [20].

In reinforced polymers, it is commonly observed that flexural strengths are higher than tensile strengths [9, 21]. Same trend is observed in this current study. Under a flexural loading situation, rather than acting as stress raisers as is the case in tensile loading, the fillers apparently aid the load bearing capability of a composite.

This load bearing capability is due to the fact that compressive stress tends to close cracks and flaws that are perpendicular to the applied stress, opposite to the crack opening mechanism occurring in a tensile strength situation [22]. Hence the flexural strengths tend to be higher.

#### Flexural Modulus

The variation in flexural modulus of the neat epoxy and the composite samples is presented in Figure 7. The flexural modulus was found to increase linearly with silica loading up to 2% wt. SiO<sub>2</sub>. The maximum value of flexural stiffness (764MPa) was observed in the EPS3 sample which is a two fold increase when compared to EPS0 (378MPa). This is due to the high surface energy possessed by the small size fillers [23]. However, after the EPS3 composite sample, the silica particles get agglomerated, thus the modulus decreases due to poor dispersion during processing. The decrease can also be attributed to the small size of the filler particles (about 0.50 μm) such that the aspect ratio is high. Summarily, an increase in modulus was observed in good number of composite samples when compared to the neat epoxy matrix and this shows that the silica provides good reinforcement properties under flexural loading.

#### Hardness

Hardness is a property which measures the resistance of a material to surface indentation and abrasion. The hardness is also a critical parameter which denotes the durability and life span of the composite [17]. Figure 8 shows the hardness of the different compositions of composites produced as well as the neat epoxy sample.

It is noted that all the composites produced possess high hardness than the neat epoxy. This increased hardness is linked to the high rigidity and hardness of the dispersed phase particles which allows strengthening due to its load carrying capacity [16].

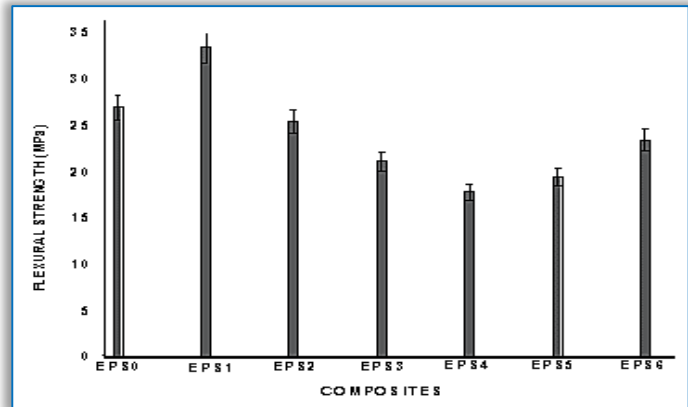


Figure 6: Variation in Flexural Strength of the Neat Epoxy and the Composite samples.

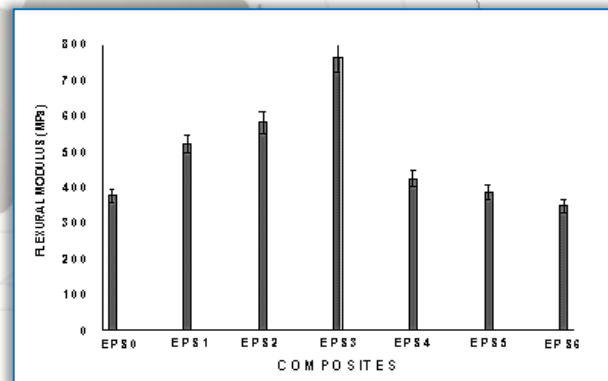


Figure 7: Variation in Flexural Modulus of the Neat Epoxy and the Composite Samples

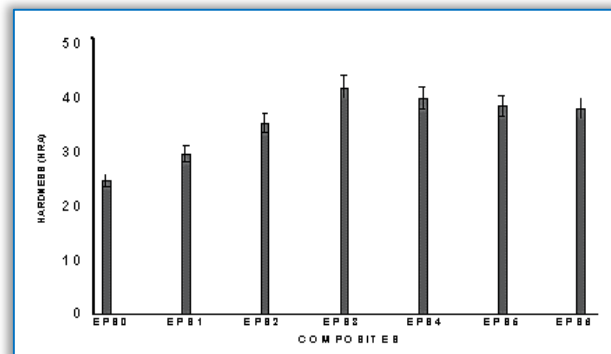


Figure 8: Hardness values of the Neat Epoxy and the Composite samples





It was observed from the plot that EPS3 has the highest hardness value which is a 70% improvement over the matrix material (EPS0). However, hardness of a composite depends on the homogeneous dispersion of the particulate into the matrix. The hardness of the composite showed an increasing trend with an increase in silica content up to 2wt% but reduces slightly and gradually as particulate loading increases. The increase in hardness value at lower particulate loading may be attributed to the homogeneous dispersion of silica particles into the matrix with minimisation of voids and stronger interfacial adhesion between the matrix and the particulate while the gradual reduction in hardness at higher particulate loading may be due to the agglomeration of particles which encourages voids formation and poor interfacial adhesion between the matrix and the particulate.

It is noteworthy to mention that even though the incorporation of the silica has reduced the tensile strength of the composite, it has increased the hardness of the epoxy matrix. The difference is attributed to the loading conditions. The tensile loading condition tends to cause debonding of the silica particles from the epoxy matrix due to poor interfacial adhesion between the two. In the case of the hardness test, a vertical and downward indentation load is exerted on the composite. This makes the matrix to be pressed firmly onto the silica particles.

The pressing action causes smooth transfer of the load from the matrix onto the silica particles even though their interfacial adhesion is poor. All these result in improved hardness of the composites. Similar property improvement has been reported for hard and rigid phases dispersed into soft polymer matrices [11, 24].

### ☐ Surface Morphology

Scanning Electron Microscopy (SEM) was used to investigate the microstructure, observe the distribution of the rice husk silica powder in the epoxy matrix and also assess miscibility between the rice husk silica particles and polymer matrix at the fractured surfaces of the tensile specimens. Figure 9 shows the SEM micrograph of the neat epoxy matrix. Smooth appearance in conjunction with delta-like markings was observed on the fractured surface of the neat epoxy (EPS0) specimen. This is similar to the findings of Yüstraet *al.*, (2015) [25]. This is an indication of the brittle nature of the neat epoxy and it also show that it has been subjected to plastic deformation. The SEM image of the EPS3 sample in Figure 10 reveals the embedded rice husk silica particles in the matrix. Uniform silica particle dispersion was more effective in this case which is evident in the highest value of tensile strength it possessed compared to other composites. However, little clustering of the particles was observed in some regions in the matrix. The EDS profile of the composite showed peaks of Silicon (Si), Carbon (C) and Oxygen (O) atoms confirming the presence of silica particles as the dispersed phase in the epoxy matrix.

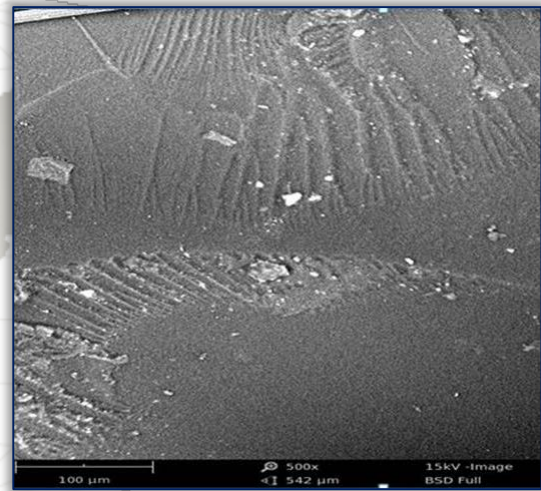


Figure 9: SEM Image of the Neat Epoxy (EPS0) Sample

When the rice husk silica content was increased to 4wt%, morphological changes were observed in the EPS5 sample. The surfaces were observed to be rough. Due to the high amount of fillers, large agglomerates were formed as depicted by the arrow in Figure 11.

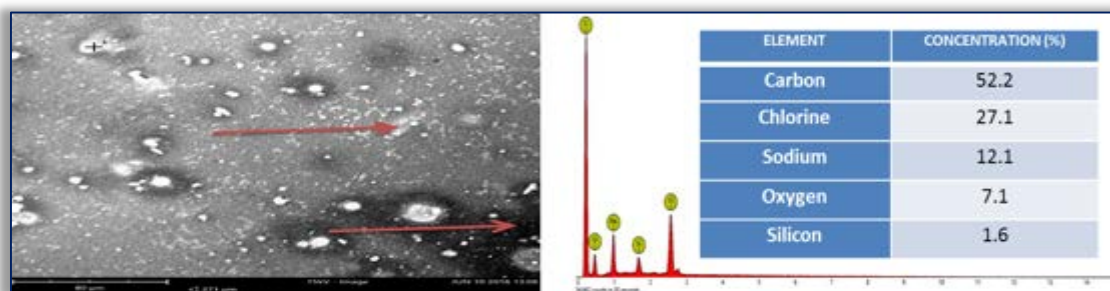


Figure 10: SEM Image /EDS profile of the 2wt% SiO<sub>2</sub>/Epoxy Composite (EPS3) showing the Spectrometric

### ☐ Data as detected by an EDS-equipped Scanning Electron Microscope

The agglomerated nature of the rice husk silica particles acted as stress concentration sites under the applied tensile stress. Thus, cracks were able to penetrate through these silica agglomerates resulting





in weak points and initiating failure. This is reflected in the poor tensile strength of the EPS5 composite. In general, this scenario was experienced at higher filler loading and this results in a linear reduction in the tensile strength of the composites with higher silica loading.

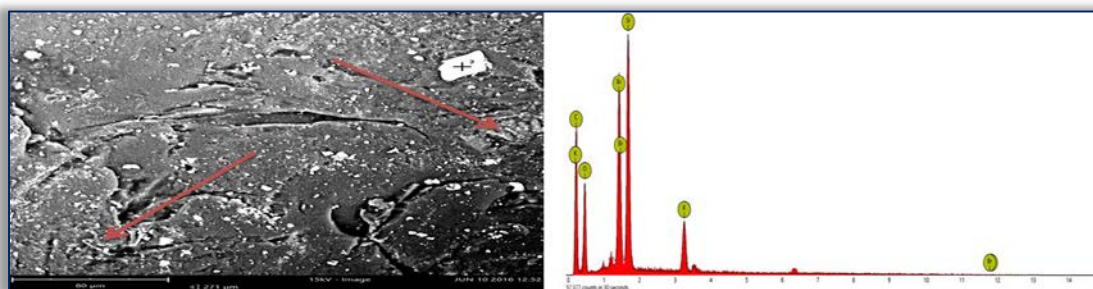


Figure 11: SEM Image /EDS Profile and Spectrometric Data in 4wt% SiO<sub>2</sub> /Epoxy Composite (EPS5)

#### 4. CONCLUSIONS

The mechanical properties (tensile, hardness and flexural properties) of epoxy matrix composite reinforced with rice husk silica (RHS) particles has been studied. From the test results obtained, the following conclusions were drawn:

- i. The silica particles extracted from RHA was used to produce an Epoxy/SiO<sub>2</sub> composite using hand lay-up technique.
- ii. Some mechanical properties such as tensile strength and flexural strength were found to suffer under higher silica loading. However, the flexural strength values obtained were greater than their tensile counterparts. Properties such as hardness, modulus of elasticity and flexural modulus were enhanced in the composites developed up to a threshold value of 2 wt% silica particles loading. This shows that low silica content is good for enhancement of mechanical properties
- iii. The surface morphology of the tensile fractured surface reveals particle agglomeration which resulted in decreased tensile strength in the composites with higher silica loading
- iv. Summarily, rice husk silica particles can be used as good reinforcing filler for epoxy resin. However, a compromise must be reached in deciding the content of rice husk silica to be incorporated so as not to sacrifice too much of its tensile and flexural strength.

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