



## RICE HUSK ASH REINFORCED NATURAL RUBBER COMPOSITES: EFFECT OF BENZENE DIAZONIUM SALT TREATMENT.

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### Abstract

Rice husks are a major agricultural residue with growing volume in Nigeria in recent times owing to the country's drive for self-sufficiency in rice production. Rice husks have great potential for use as fillers in biocomposites for applications in the manufacture of engineering products. However, the hydrophilic nature of this agricultural residue limits its choice as fillers in biocomposites. This study focused on the modification of the hydrophilic property of rice husks through treatment with benzene diazonium salts in different media (pH levels of 7, 9, 10.5 and 12) to attain the most suitable treatment media for processing rice husks. The treated samples were burned in a furnace at a temperature of 600°C for 6 hours. The compounding of the rice husk ash, CV-60 natural rubber and additives were done on a two-roll mill at a speed ratio of 1:1.25 with rollers temperature of 70°C and cured in a hydraulic press at 130°C for 20 minutes at 2.5 MPa. The composites were developed using rice husk ash as the filler with loadings of 30, 34, 38, 42, 46 and 50 phr. Characterizations and tests carried out include Fourier transform infrared spectroscopy (FTIRS), X-ray fluorescence (XRF), shore A hardness, compressive strength and tensile strength. The composites developed using pH 10.5 treated rice husks yielded the highest silica content and showed better adhesion characteristics. The sample with 42 phr loading gave better mechanical properties with tensile strength, compressive strength, and tensile modulus of 18.5 MPa, 39.72 MPa and 7.87 MPa respectively with a corresponding shore A hardness of 45.32. The rice husk ash filled natural rubber composites can find application in the development of vibration isolation mounts.

**Keywords:** Rice husk ash, Silica, Biocomposite, Benzene diazonium salt.

### 1.0 INTRODUCTION

Due to the growing demand for biobased materials that are ecologically friendly and meet the sustainability criteria, researchers are increasingly looking for sustainable green materials. With regard to energy conservation, impact on the environment, and even production economics, green materials are increasingly replacing the prevalent materials that have been used in engineering manufacturing for the

past 50 years. These biobased product applications have been steadily growing and controlling the markets not only from a technological and scientific standpoint but also from a socioeconomic and environmental one. It has been asserted that using biobased materials in engineering manufacturing will make the environment safer, encourage energy consumption reductions, be sustainable and provide superior mechanical, physical, and related properties

needed by current products [1,2]. Rice husks have been applied in several applications to enhance the mechanical properties of engineering products due to their intrinsic silica content [3,4].

At present, rice husk as an agricultural residue is underutilized in Nigeria and its current method of disposal through open burning poses a lot of harm to man and the environment [5,6]. Hence the need to utilize explore the utilization of rice husk as fillers in composites. Their use in composites is however limited due to the presence of lignin, hemicellulose, and other related components associated with natural fibres and biobased composites which are responsible for low bonding strength and associated interfacial interactions with their matrix [7,8] which make them hydrophilic. Consequently, there is a need for chemical modifications of agricultural residues and green materials used in engineering manufacture. Several chemical treatments for rice husks have been explored in this regard including mercerisation, permanganate, acid leaching, acetylation, treatment with silane, peroxide treatment, anhydride, and benzoylation among others [9,10]. This research utilized benzene diazonium salt at different pH levels to treat rice husk for the purpose of improving their compatibility with natural rubber thereby improving their properties and overall performance of composites produced from them. Rice husk which when processed under appropriate conditions yields very high purity silica is currently being investigated for use as a viable substitute to commercial silica used for manufacture in the rubber industry [5], hence the need to investigate viable means that will foster the interaction between the rice husk silica and their matrices which will yield favourable engineering properties.

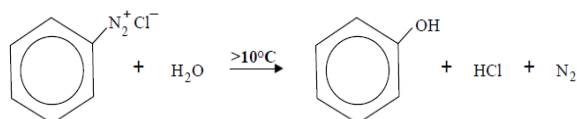
Chemical treatments have the potential to alter the OH groups and other polar groups responsible for the hydrophilic property of natural fibres [11]. Mercerization has been suggested in many studies as a useful technique for altering the cellulose content of natural fibres, improving their interfacial interaction and binding strength with their matrix in composites. Reducing carbonaceous ingredients and treating rice husk have both been shown to improve the filler-to-matrix interfacial interaction and the quality of the silica content generated. According to [12], sodium hydroxide (NaOH) is the most often utilised alkali in the mercerization process. This recommendation might not apply to all natural fibres, as recent studies have revealed that natural fibres treated with NaOH

can have a variety of qualities which are not favourable in all areas of application. In order to improve the adhesion and interfacial filler to matrix bonding properties of rice husk in composites, [13] describe the chemical modification of rice husk using sodium hydroxide solution. Following their examination, it was discovered that the NaOH-treated husk's thermal stability had decreased, suggesting that the concentrated NaOH employed may have caused the rice husk to degrade. Despite the improved mechanical properties of NaOH treated rice husks composites investigated by [14], they observed an adverse effect on the water resistance characteristics of the rice husks when used as fillers in biocomposites. The unfavourable characteristics of rice husks treated with sodium hydroxide may be due to the creation of sodium silicate, which is soluble in water and is created when the sodium hydroxide reacts with the silica in rice husk. The removal of hemicellulose and lignin from the rice husk is more successful with the NaOH/H<sub>2</sub>O<sub>2</sub> treatment [15]. NaOH treatment provides greater delignification effectiveness when compared to H<sub>2</sub>O<sub>2</sub> pretreatment. [16] in their investigation of rice husks in relation to their alkali-treated forms reported that alkali treated rice husks absorbed water higher than their untreated counterpart. The treatment of the fillers with outstanding mechanical properties has been reported to be more favourable with diazonium salt treatments than mercerization and other treatment methods [9,14,17,18]. Hence, the need to subject rice husk to various pH levels of this treatment to ascertain the appropriate concentration that will yield better properties for developed biocomposites. Chemical treatments do not improve all the properties of a given composite material but may influence better properties for one or more and can have a good or bad impact on the characteristics of composites. Therefore, the need to subject natural fibres to various forms of chemical treatment to determine which treatment is more appropriate in the fillers' preparation [10]. As established by [9], benzoylation treatment permits the decrease in hydrophilic properties of rice husk fibres and hence, improves their interfacial interactions with the hydrophobic matrix. Benzene diazonium salt treatment helps in overcoming the issues faced by green composites owing to the hydrophilic nature of their fibres [19].

Haque et al. [20] used benzene diazonium salt to chemically treat coir fibres in alkaline, acidic, and neutral media to boost the compatibility of the coir fibre with the polypropylene matrix in the composite. In their study, they observed that the hydrophilic -OH

groups in the raw coir cellulose were transformed into hydrophobic -O-Na groups during chemical treatments. They employed 10, 15, 20, 25, 30, and 35 wt.% of raw coir fibre and treated coir fibre to reinforce the matrix and found that alkali-treated samples produced the optimum combination of mechanical properties at a loading of 30 wt.% [20,21]. Rahman et al. [21] used benzene diazonium salt in the acid, alkali, and neutral environments in their investigation of the impact of chemically processing rice husk and its application as a reinforcement in polyethylene matrices. The results of their study demonstrated that, in comparison to the acidic medium with a pH of 6 and the neutral media, rice husk treated in the alkaline media at all mixture ratios displayed higher values and greater dispersion of the fillers in the polyethylene matrix. Additionally, they stated that the created composites' mechanical properties had been greatly enhanced by the chemical treatment of the rice husk. According to the loading condition and the water absorption characteristics of the produced composites, the percentage of water absorption increased. It was asserted that the rice husk's hydroxyl group was responsible for the material's capacity to absorb water. The composites made from untreated rice husk had the maximum water absorption capacities, followed by those made from treated rice husk exposed to neutral and acidic environments, respectively. According to their assessments, the polyethylene composites loaded with a 35 percent rice husk filler had the highest mechanical qualities and the best water absorption characteristic in the alkali treated media.

When it comes to reactions, benzene diazonium chloride goes through two different types: coupling reactions, wherein nitrogen atoms are kept, and substitution of the diazonium group, where nitrogen is released. The hydroxyl (OH) groups were replaced during the reaction with water (hydrolysis) above 10°C. This is frequently employed because the reaction produces phenol, an antiseptic utilised in the production of polymers. Equation (1) represents the substitution reaction with water as the reagent above 10°C.



Similarly, comparing benzene diazonium chloride with mercerisation, acrylation and permanganate

treatments on abaca fibres, researchers [17,18] reported that the benzene diazonium chloride treated abaca reinforced polypropylene composites showed excellent interfacial bonding characteristics and mechanical properties resulting in the best tensile strength with optimum properties noticed at the 40% fibre loading. [21] similarly utilized diazonium salt in the treatment of palm fibres which was used in reinforcing polypropylene and established that benzene diazonium salt dissolved the hydroxyl groups in the acid media with a pH of 3, the alkaline media with a pH of 10.5 and the neutral media with a pH of 7. However, only the alkaline media dissolved the lignin content in the palm fibre, resulting in excellent mechanical properties exhibited by the developed composites treated in the alkaline media. The treatment of the fibre in the acid media did not dissolve the lignin content of the palm fibre but showed better mechanical properties compared with that of composites developed from neutral media treatment. They reported that treatment done using neutral media was not effective and showed similar mechanical characteristics with composites developed from untreated palm fibres. The young modulus of the developed composites was highest for the alkali-treated rice husk reinforced composites and increased with fibre loading. Their report showed that the stiffness of the composite also increased with increasing fibre content. [22] treated jute fibre using benzene diazonium salts in alkaline and acidic media and reported better tensile strength of fibres treated in the alkaline media compared with those of the acidic media. Composites of banana/luffa strands treated with benzene diazonium chloride were reported to show better impact strength [23] and compressive and flexural properties when compared with those treated using sodium hydroxide [24]. Benzoylation treatment aids in decreasing the hydrophilicity of fibres thereby improving their interaction with the matrix [9]. Benzene diazonium salt treatment using the suitable media and PH level for the treatment of rice husks holds a promising means of improving the interfacial interaction and compatibility of green composites.

## 2.0 MATERIALS AND METHOD

### 2.1 MATERIALS

Rice husk samples obtained from a mill in Kaduna state, Nigeria were treated using benzene diazonium salt at pH levels of 7, 9, 10.5 and 12. The natural rubber used in this study was CV-60 rubber. Aniline, sodium nitrate, sodium hydroxide, hydrochloric acid and distilled water were used in the preparation of the reagents used for the treatment while zinc oxide, stearic acid, sulphur, 2-Mercaptobenzothiazyl Disulfide (MBTS), 2, 2 ,4 – Trimethyl – 1, 2 -

dyhydroquinoline (TMQ), 3-(Triethoxysilyl) propyl tetrasulphide (silane TESPT) and processing oil were used in the mastication and compounding of the composites.

## 2.2 METHOD

The diazonium salt was prepared in accordance with the standard preparation method as reported by [20,21] to obtain diazonium salt in the neutral and basic media corresponding to 7, 9, 10.5 and 12 pH levels. 500 g of rice husk was submerged into the prepared solutions exclusively for each of the media for 10 minutes at 5°C. A freshly prepared diazonium salt was poured slowly into the stated mixtures with constant stirring for 10 minutes. The rice husk was taken out, washed with soap solution followed by thorough rinsing in water and dried in the open air. The treated samples were burned in a furnace at a temperature of 600°C for 6 hours to yield rice husk ash. The rice husks ash was characterized using Fourier transform infrared spectroscopy (FTIRS) and X-ray fluorescence (XRF) equipment. Composites were developed with a filler formulation of 30, 34, 38, 42, 46 and 50 parts per hundred rubber (phr) for the rice husk ash obtained from the chemical treatment done at pH 10.5. The rice husk ash, natural rubber and additives listed in Table 1 were masticated and compounded following the ASTM D3182 standard, on a two-roll mill at a speed ratio of 1:1.25 and rollers temperature of 70°C. During mastication, the compounds were cut and re-banded several times before sheeting out and conditioning. The masticated samples were cured in a hydraulic press at 130°C for 20 minutes at 2.5 MPa. The composites' formulation is presented in Table 1.

**Table 1:** NR-RHA Formulation

Material	Phr
Natural Rubber (NR)	100
Zinc Oxide	2
Stearic Acid	3
TMQ	2
MBTS	3
Rice Husk Ash (RHA)	30, 34, 38, 42, 46, 50
Sulphur	5
Processing Oil	10

The tests carried out on the developed biocomposites include hardness test following the ASTM D2240, flexural test using the ASTM D790 standard, and compression test in line with the ASTM D795.

## 3.0 RESULTS AND DISCUSSION

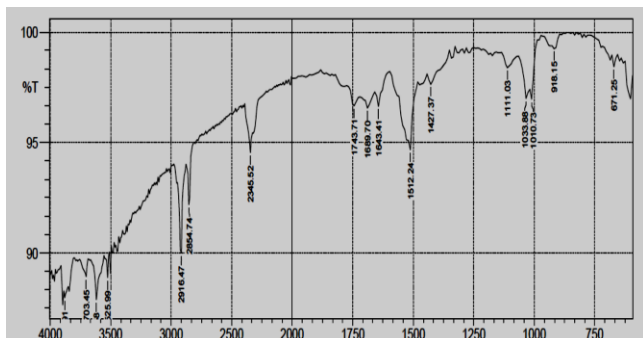
The x-ray fluorescence results of the benzene diazonium treated rice husks showed that the samples treated using the pH 10.5 solution yielded the highest percentage concentration of silica when compared with samples treated with pH values of 7.0, 9.0 and 12.0. At a higher alkali concentration of pH 12, the silica concentration was noted to drop and resulted in a value of 86.321% which was the least among the treated samples considered. The drop in the silica concentration at a higher pH value of 12 in the alkaline media is likely due to the enhancement in the bond strength of O-Si-O of the rice husk ash which led to a reduction in the silica content as asserted by Halip et al. [9] who stated that alkaline treated natural fibres enhances the bond strength of O-Si-O of the silica thereby reducing the quantity of amorphous silica. Based on the concentrations obtained from the rice husk, it was noticed that variation in the silica content of the oven-burned rice husks was not so significant among other treated samples and that silica is the predominant compound found in rice husk ash as reported by [25]. Also, the XRF result as presented in Table 2 shows that benzene diazonium salt treatment of rice husks improves the silica yield. Physical examination of the treated rice husk before burning in the furnace showed decreasing brightness in colour of the husk from pH 7 which was the brightest with a light brown colour to pH 12 which had a very dark brownish colour.

**Table 2:** Compounds present in rice husk

Compound (%)	Untreated RHS (Wt. %)	pH 7.0 RHS (Wt. %)	pH 9.0 RHS (Wt. %)	pH 10.5 RHS (Wt. %)	pH 12.0 RHS (Wt. %)
SiO <sub>2</sub>	65.440	87.096	88.871	89.744	86.321
Al <sub>2</sub> O <sub>3</sub>	0.006	1.495	1.502	1.236	1.504
Fe <sub>2</sub> O <sub>3</sub>	9.910	0.580	0.504	0.458	0.439
MgO	1.040	0.937	0.821	0.619	1.002
CaO	17.230	4.256	0.961	1.350	3.487
Na <sub>2</sub> O	4.200	0.809	2.134	2.157	2.162
TiO <sub>2</sub>	0.030	0.073	0.057	0.053	0.069
P <sub>2</sub> O <sub>5</sub>	0.010	2.662	2.899	2.429	2.967
MnO	0.020	0.161	0.147	0.163	0.148
K <sub>2</sub> O	2.020	0.895	1.075	0.889	0.809
TOTAL	99.906	98.964	98.971	99.098	98.908

The qualitative characterization of the rice husk surface functional group was carried out on the Rice husk silica samples. As seen in Figure 1, the untreated sample showed a band around 3525cm<sup>-1</sup> which is attributed to the O-H stretch of the hydroxyl groups (adsorbed water). The absorption band of O-H group

shifted from larger wave number of  $3525\text{cm}^{-1}$  (untreated sample) to between  $3400$  and  $3200\text{cm}^{-1}$  (treated samples) with weak band intensity which ascertained the reaction between the cellulose hydroxyl group with the diazonium salt [10].

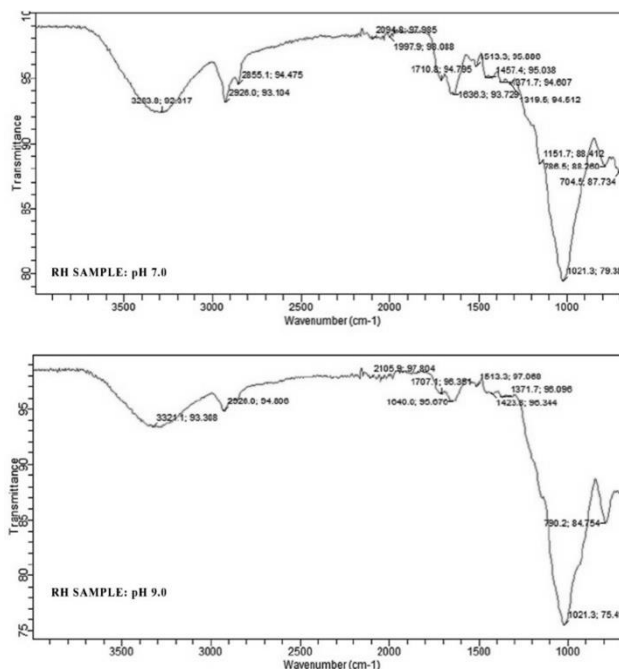


**Figure 1:** FTIR Spectrum for untreated RHS

The treatment of the rice husk with benzene diazonium salt showed significant modification in the functional groups present in the samples corresponding to a reduction in the lignin and hemicellulose content present in the rice husks. Significant alterations were noticed at all peaks for the treated samples with varying pH when compared with the untreated rice husk sample. Figures 2 and 3 show the results of the FTIR for the various treatment levels. The region  $2800$  to  $3000\text{cm}^{-1}$  in the spectra from the untreated rice husk samples showed sharp and strong bands which correspond to C-C and C-H bond vibrations. Variation in the fingerprint region ( $600$  to  $1400\text{cm}^{-1}$ ) with respect to the percentage transmittance is observed from the infrared spectra after treatment with diazonium salt carried out at the various pH levels. Transmittance for the range  $95$  to  $98\%$  gave a wave number of  $1021.3\text{cm}^{-1}$  for the chemically treated samples. The bands at the fingerprint region are strong and narrow for both the chemically treated and untreated samples.

The prominent broad bands in the infra-red spectra for wave numbers between  $3000$  and  $3700\text{cm}^{-1}$  after treatment with benzene diazonium salt as shown in Figures 2 and 3 resulted in effective modification of the rice husk and confirms the presence of alcohol due to O-H bond. Typically, broad bands represent the presence of O-H bonds found in alcohols and carboxylic acid.

The strong broadband alongside the sharp bands confirms the presence of carboxylic acid with the stake-shaped band around  $1700\text{cm}^{-1}$  in the spectra for the pH 7.0 and 12.0 treated rice husk and corresponds to C=O stretch. The carboxylic functional group com-

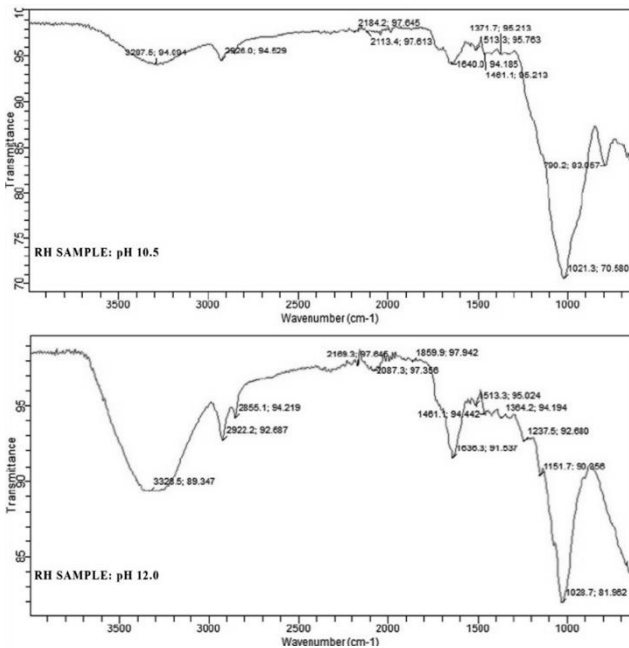


**Figure 2:** FTIR Spectrum for pH levels 7.0 and 9.0 Treated RHS

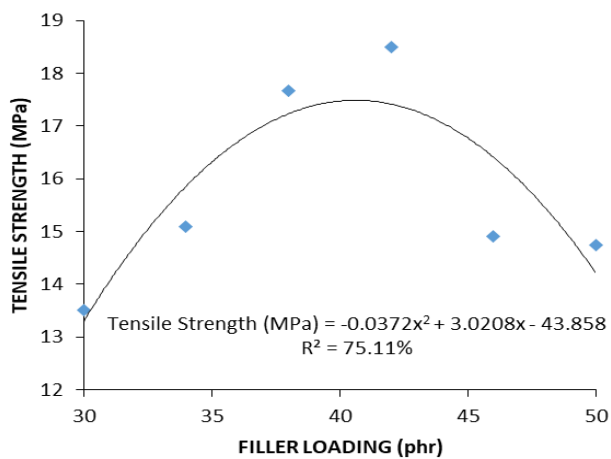
binates the features of ketones and alcohols due to its combination of the C=O and O-H bonds. The spike observed in the infra-red spectrum of all treated rice husks is a characteristic of amines due to N-H bond stretch and is broad though usually not as broad as the hydroxyl bond. The bands with higher intensity indicate a strong bond strength while bands with a lower intensity suggest a low bonding strength. As established by [26], the low vibration observed depict the development of crystals in the recovered ashes and the higher bonding strength depicts the increment of silica in its crystalline form.

The sample with 42 phr rice husk ash loading exhibited better mechanical properties and confirms the reports of [18] who asserted that the best mechanical properties for benzene diazonium treated natural fibres are obtained with filler loadings around 40% wt. [18] reported a tensile strength of 50.24 MPa for the abaca-filled polypropylene composites which they studied. In this research, a tensile strength of 18.5 MPa was obtained at the 42 phr loading and a correlation of determination ( $R^2$ ) of 75.11% as illustrated in Figure 4 was obtained. This suggests that about 75% of the tensile strength of the developed composites is dependent on the rice husks ash filler loading. The remaining 25 % may likely be dependent on other parameters such as the percentage weight in phr of the additives and the processing conditions such as curing pressure, temperature and time.





**Figure 3:** FTIR Spectrum for pH levels 10.5 and 12.0 Treated RHS

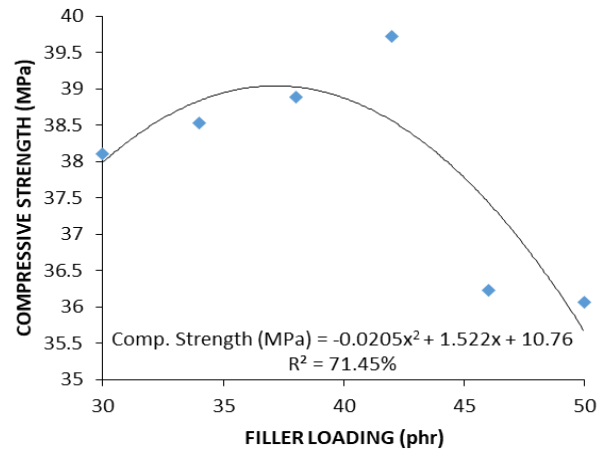


**Figure 4:** Tensile strength of the RHA-NR composites

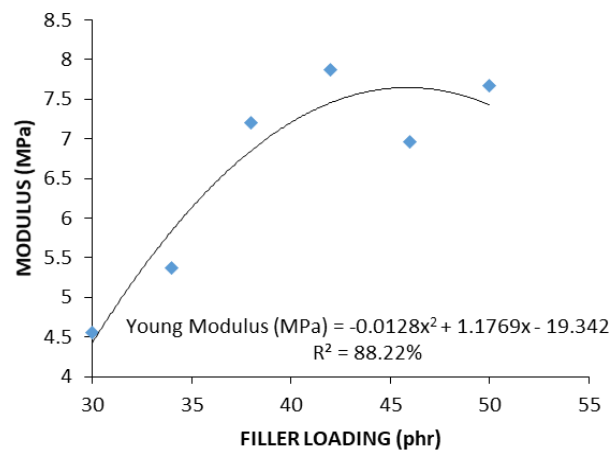
The compressive strength similarly had an  $R^2$  value of 71.45% as illustrated in Figure 5 and a value of 39.72 MPa at 42 phr filler loading. A similar study by [27] on the behaviour of the compressive strength of waste plastic-rubber composites reported compressive strength values of between 3.5 and 12 MPa which is low when compared with the compressive strength obtained in this research.

An  $R^2$  value of 88.22% was obtained for the tensile modulus (Figure 6) which had a value of 7.84 MPa while an  $R^2$  value of 98.12% was obtained for the shore A hardness property which means that the regression equation presented in Figure 7 largely

explains the relationship that exists between the filler loading level and shore A hardness.

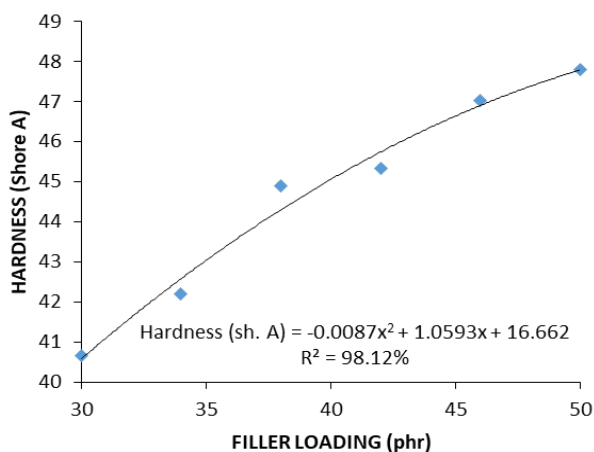


**Figure 5:** Compressive strength of the RHA-NR composites



**Figure 6:** Tensile modulus of the RHA-NR composites

The introduction of fillers typically results in an increase in hardness, particularly when the particle sizes of the fillers are high [28]. When the rubber composite is indented, large filler particles inhibit the mobility of the matrix, thereby leading to an increase in the indentation hardness. The shore A hardness results obtained in this study conform to the hardness characteristics of rubber composites as reported by several researchers. According to [28], RHA silica has the potential to be used as a reinforcing filler in natural rubber products where hardness is not the most important factor but other mechanical characteristics are desired.



**Figure 6:** Shore A hardness of the RHA-NR composites.

#### 4.0 CONCLUSION

The utilisation of rice husks treated in various benzene diazonium salt media was investigated. The alkaline media at a pH of 10.5 gave a better silica yield and composites produced from it exhibited good mechanical properties, hence establishing that rice husk ash can be a viable substitute for traditional commercial silica used in the manufacture of rubber-based products. The composites developed using the rice husk ash filler have the potential for use in the manufacture of anti-vibration mounts and related elastomeric products used in the automobile industry.

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