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PRODUCTION AND INVESTIGATION OF THE MECHANICAL PROPERTIES OF POLYPROPYLENE-BIOSILICA COMPOSITES

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ABSTRACT

Polypropylene (PP) as thermoplastic has found use in the production of a variety of items due to its low cost and ease of production. However, its usage is limited to inherent in its mechanical properties. Rice husk which is readily available and underutilize is considered a major source of biosilica (BS) after calcination. Therefore, the incorporation of biosilica into polypropylene resin as a composite will further reduce the cost and improve its properties. This research developed Polypropylene-biosilica composites with varying compositions of BS and their mechanical properties were determined using ASTM standard test methods. The results of the mechanical test showed a significant increase in Young's modulus from131.08 MPa at 0wt% to 224.86 MPa at 20wt% and flexural modulus from 1055 MPa at 0wt% to 1936.36 MPa at 20wt%. However, a decrease in tensile strength from 30.61 MPa at 0wt% to 24.78 MPa at 5wt% and percent elongation from 24 at 0wt% to 12.4 at 5wt% was recorded. Also, a decrease in Impact strength from 0.26 J at 0 wt% to 0.225 J at 5wt% and hardness value from 23.46 HV to 22.00 HV was observed. It was deduced from the results that polypropylene-biosilica composite is suitable for applications where rigidity is required.

1 INTRODUCTION

Engineering materials such as cast iron and alloyed steel that exhibit good mechanical properties have some setbacks on the account of heavyweight, high cost and non-degradability. Technological advancement is geared toward the use of materials that possess a good strength-to-weight ratio in engineering materials which has led to extensive research and development in the field of composites [1]. Reinforced composites with inorganic fillers enhance their mechanical and physical properties [2]. However, these composites have some setbacks because of their non-degradability and eco-friendly [3]. For this reason, natural fibres provide an alternative as reinforcement fillers in thermoset, thermoplastic and elastomer. Particulate composite reinforced with polymer exhibits superior mechanical properties to unreinforced resin [4]. The addition of biofillers with low-density particulate materials like rice husk ash, jute and bones combine into thermoplastic to form polymer composite reduces dependence on the use of mineral particles and could achieve a good strength-to-weight ratio [5]. Organic fillers have become an alternative to traditional inorganic fillers due to their low densities, very low cost, non-abrasiveness, recyclability, biodegradability and renewable nature [6].

Recently, there has been growing environmental concern and the need for sustainable development, which has raised interest in using natural fibres as reinforcement in polymer composite to replace synthetic fibres such as glass [3]. In Nigeria, agricultural by-products such as rice husk, which are in abundance, are not adequately utilized for engineering applications. Rice husks constitute environmental pollution due to its resistance to decomposition in the soil and low nutritional value if used as animal feed [7]. Therefore, the utilization of Rice husks as fillers would help in overcoming these challenges. Rice husk contains 86% - 97% amorphous silica when calcined to ash [8]. The presence of high silica content in rice husk ash makes it a valuable material for use in industrial applications [9]. Hence, this makes it a good candidate for the development of cheap, lightweight and environmentally friendly composite [10]. This research aims at utilizing rice husk by incorporating it into a Polypropylene matrix to improve its mechanical properties.

2 MATERIALS AND METHODS

2.1 Materials and Methods

The materials used for this experiment include rice husk weighing about 3.5 kg sourced from a local mill in Kura, Kura Local Government Area of Kano State, Nigeria, portable water, 1M dilute hydrochloric acid (HCl), distilled water, polypropylene homopolymer with density 0.9 g/cm$^{3.}$

The equipment used includes an aluminium pan of 700 mm^3 volume capacity; a Muffle electric furnace, Carbolite Technology with model number CWF and a capacity of 1100 ˚C. Heating drying oven, model DHG-9053A with a capacity of 900 ˚C. A measuring cylinder of 200 ml capacity. Municipal 4 X-Ray Fluorescence (XRF), the XRF Epsilon analytical spectrometer (Model DY1055) designed for elemental analysis of a wide range of samples; a mettle balance with model number AT400 and a precision of 0.0001 g. An Allen Bradley two-roll milling machine with number 0183; compression moulding machine Model M. Metal mould made from mild steel with a dimension of 120 mm × 120 mm. Hounsfield Monsanto tensometer type W, with model number 9875. A 100 kN Universal Testing Machine; a MicroVickers Hardness machine (model MV1-PC), Charpy Impact Testing Machine.

2.1.1 Preparation of biosilica and composite

Rice husk was collected from a milling plant in Kura Local Government Area of Kano State, Nigeria. It was washed to remove sand particles and dust. The clean Rice husk was leached with 1M HCl by heating it in an oven at 70 ˚C for three hours. Thereafter, it was washed thoroughly with distilled water and air-dried. The sample was calcined into ash in a furnace at about 700 ˚C for six hours.

Composite preparation involves compounding the constituent substances (polypropylene and biosilica) and pressing the mixture. The formulation of the composite was in weight percent as shown in Table 1.

Table 1. *Formulation of composite*

Sample ID	Polypropylene (wt%)	Biosilica (wt%)
A	100	0
B	95	5
C	90	10
D	85	15
E	80	20
F	75	25

The compounding process was done by two-roll milling to mix and ensure the homogeneity of the mixture. The milling machine was switched on and set for preheating at a temperature of 180 °C for one hour. The speed of the front and rear rolls of the machine was set at 40 and 30 rpm respectively. It was ensured that the clearance between the rolls was minimal. Mixing was done in between the roll by the rollers, and manually turning the mixture was done to achieve good dispersion. The final mixture was then transferred quickly into a mould to avoid heat loss. The mixture was then compressed for two minutes without pressure to stabilize and allow trapped air to escape. The pressure was then raised gradually to 50 bar at 150°C and maintained for eight minutes. These parameters were chosen carefully to avoid degradation of the sample. Thereafter, the mould was removed from the machine and allowed to cool with its content to room temperature. The same procedure was repeated for the remaining four samples.

2.1.2 Testing of mechanical properties

Tensile Test: The test was conducted as per ASTM D3039 using a specimen of dumbbell shape with a width of 10 mm, length of 100 mm and thickness of 3 mm [11]. Two fine marks were made on the samples with a distance of 40 mm apart as the gauge length. A crosshead speed of 5mm/min at room temperature was used. The sample was mounted on the tensometer and the mercury indicator was set. It was ensured that the ends of the test piece were fitted into the grip of the

tensometer. The grip was held between the tension head and the operating screw. The force applied to the specimen was transmitted to the chart roller which is attached to a graph paper. Turning of the operating handle leads to a gradual increase of the load at the middle span until a fracture occurs. The force and extension were obtained from the load and extension curve from the graph paper. Two specimens from each composition were used. Figure 1 shows two samples of the composite materials cut into the dumbbell shape for tensile testing.

Figure 1*. Samples for Tensile Testing*

Flexural Test: Flexural test was conducted according to ASTM D 790-10 Standard Test Methods [11]. The composite sample was cut to 100 mm by 30 mm by 3 mm dimensions using a hacksaw. The experiment was performed with a three-point fixture where samples fracture in the middle. The sample was placed horizontally over two points of contact (lower support span). A lever was gradually pulled down on the sample through one point of contact (upper loading span) until the sample failed. The flexural force that fractures the sample and the deflection was recorded from the machine scale. Two specimens from each composition were used for this test.

Toughness Test: The test was carried out according to ASTM D6110 standard method [11]. The specimen was cut to 100 mm by 10 mm by 5 mm dimensions using a hacksaw. A V-notch of 1.5 mm deep was cut in the middle of the specimen to provide an area of stress concentration for crack initiation. The hammer was raised and the gauged length was adjusted to zero reference position. The specimen was placed horizontally between the two anvils of the machine with the notch away from the hammer. The swinging pendulum harmer was released from a fixed height to strike and fracture the specimen. At the point of fracture, the energy required to

break the specimen by a single impact was recorded from the machine scale. The energy value is a measure of the toughness of the sample. Two specimens from each composition were used to measure the impact test.

Hardness Test: The test was carried out using a micro Vickers hardness machine and conducted as per ASTM E 384-17 standard methods [12]. The specimen dimensions used for the test were 10 mm x 10 mm with 3 mm thickness. The Vickers machine was switched on and the specimen sample was placed on the anvil of the machine. It was ensured that the diamond indenter in the form of a pyramid was on the surface of the sample. Ten (10) kgf was selected as the appropriate load. Thereafter, the test button was pressed and there was an automatic indentation of 30 seconds on the specimen. Reading was taken directly from the dial gauge at the end of the indentation. An average of three points was taken for each composition.

3 RESULTS AND DISCUSSION

3.1 Chemical Analysis

The chemical analysis of rice husk ash was carried out using X-Ray Fluorescence (XRF). Table 2 shows the chemical composition of various oxides from the analysis of the rice husk ash. From the table, it can be seen that silicon dioxide (SiO_2) otherwise known as Silica is the constituent with the highest percentage composition (92.8%) followed by sulphur oxide (SO_3) at 2.40% and ZrO_2 being the least constituent oxide (0.01%) in the composition.

From the result of the chemical composition of rice husk ash in Table 2, the XRF analysis result reveals silicon dioxide (SiO_2) otherwise known as Silica is the constituent with the highest percentage composition (92.8%) followed by thirteen oxides components as impurities. The high percentage of silica in the rice husk ash may be attributed to the treatment of rice husk with HCl (calcination), studies in literature have shown that calcination improves silica content[13]–[15]. This finding shows that rice husk is a good source of silica which is an important mineral that can be used as filler in polymer composite as observed by [16].

***Table 2**. Chemical analysis of rice husk ash*

Components (Oxide Element)	Composition (%wt)
SiO_2	92.8
SO_3	2.4
K_2O	1.43
CaO	1.4
Fe_2O_3	0.8
Cr_2O_3	0.23
MnO	0.21
TiO_2	0.12
CuO	0.058
ZnO	0.055
BaO	0.0098
V_2O_5	0.008
Al_2O_3	0.002
P_2O_5	0.001
MgO	0.001

3.2 Mechanical Properties of Polypropylene-Biosilica Composite

The results of ultimate tensile strength (UTS) tests conducted on the composites are presented in Figure 2.

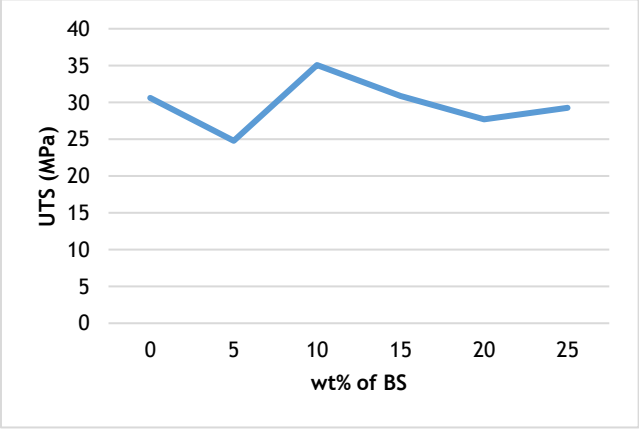

***Figure 2**. Variation of UTS with wt% BS additions*

From Figure 2, it can be seen that when the pure polypropylene (0wt% loading of BS) was tested, its UTS was found to be 30.61 MPa, this agrees with what is documented in literature as the UTS of pure polypropylene at room temperature [17]. When BS loading in the composite was at 5wt% it was found that the UTS had dropped to 24.78 MPa. A further increase in the BS loading to 10wt% showed that the UTS of the composite increased to 35.07 MPa. As can be observed, there seems to be an oscillatory pattern to the UTS of the composite as the BS content is gradually increased, however, the average UTS for composites with BS loading of 0wt% to 25wt% hovered between approximately 28 and 30 MPa. This shows that BS has a minimal effect on the UTS of PP, this is corroborated by the findings of [18] who also found that the UTS of their PP composite declined as the biosilica was increased even though they used a different variety of rice husk ash. This finding shows that if PP is to be used to manufacture an object whose desired UTS is supposed to be a bit higher than the UTS of commercially available PP, then a BS load of about 10 wt% is most appropriate.

The overall decrease in the tensile strength may be attributed to the weak interfacial bonding caused by the hydrophobic nature of the PP matrix and the hydrophilic nature of BS filler, resulting in the inability of the BS filler to transfer stress from the matrix. A similar observation was reported by [19] where the addition of inorganic silica filler in polypropylene showed no significant change in the UTS and the maximum value obtained was less compared to BS filler. The increase in the tensile strength at 10 wt% BS loading may be due to the stability of reinforcement to support stresses transferred from the polymer matrix.

The Young’s modulus of the composite samples with varying compositions of biosilica obtained by experimentation is presented in Figure 3.

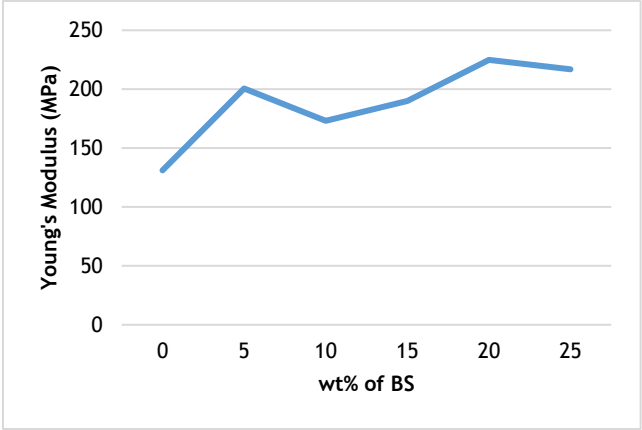

Figure 3. Variation of Young's modulus with wt% BS addition

Looking at the data for the Young's modulus with respect to the weight percent of BS in the composite samples as presented in Figure 3, it can be seen that the Young's modulus of the composite material increased with an increase in BS loading up to 5% and slightly decreased with an increase in loading from 10% to 15%. An increase in the value of Young's modulus was noticed with an increase in the loading of the BS to 25%. The maximum and minimum value of Young's modulus is 224.86 MPa at 20 wt% loading of BS and 131.08 MPa at 0 wt% loading respectively. From the data presented in figure 3, it can be seen that there is a general increase in the Young's modulus of the composite material as the BS content is increased. This trend has been described in similar studies and this is because filler particles of high modulus and aspect ratio increase the modulus of the composite [20], [21]. This shows that a component made of this composite material that has a higher content of biosilica will be able to resist plastic deformation better. Therefore, it can be stated that polypropylene-biosilica composite with a high content of biosilica is most suitable for use in the manufacture of materials that need to resist plastic deformation. It is worthy of mention that the reason why there was a decrease in the young modulus of the composite between 5-10% addition of BS cannot be explained. Also, such trend has not been noted in literature.

The percentage elongation of the composite samples with varying compositions of biosilica obtained by experimentation is presented in Figure 4.

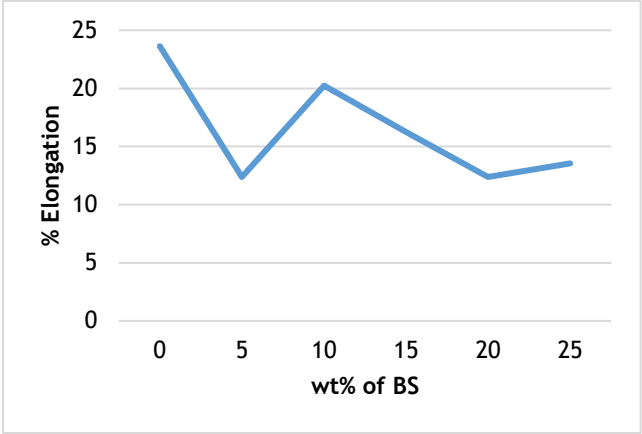

***Figure 4**. Variation of percentage elongation with wt% BS addition*

As can be seen from the data presented in Figure 4, there is a general decrease in the percentage elongation of the composite as the BS content of the composite is increased. At 5 %wt BS loading, the elongation of the PP decreased and as the BS loading was increased to 10 %wt there was a slight increase in the elongation. The value of the percentage elongation gradually decreased with the increase of BS loading from 15% to 25%. The maximum value of percentage elongation of the composite material is 24% at 0% composition and the minimum value is 12.4 at 5% and 20% BS loading. The average decrease in percent elongation with an increase in BS content of the composite might be attributed to the stiffness or rigidity of the composite caused by friction between the BS particles and the polypropylene composite matrix as observed in literature [18], [19]. This finding reinforces what has been stated earlier that biosilica increases the stiffness of PP materials, therefore, this means in instances where PP is to be used to manufacture components that need to be a bit stiffer than the stiffness regular commercial PP provides, then a PP-BS composite can be considered. However, it must be noted that an increase in rigidity/stiffness makes the material more brittle.

The flexural modulus of the composite samples with varying compositions of biosilica obtained by experimentation is presented in Figure 5.

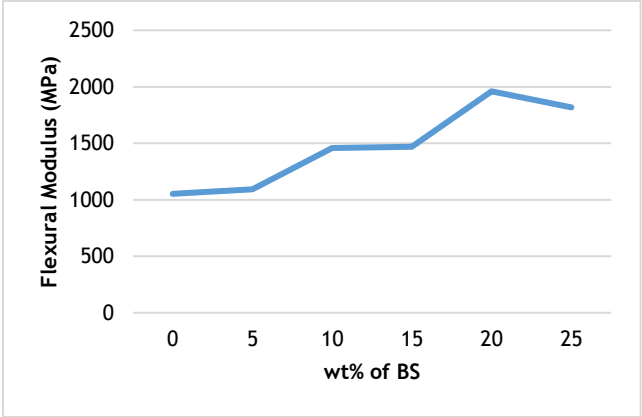

Figure 5. Variation of Flexural modulus with wt % BS addition

The data presented in Figure 5 shows that the modulus of elasticity increases with an increase in BS loading. It can be seen that the modulus of elasticity of the composite material increased from 5% to 20% and slightly decreased to 25% with an increase in the BS loading. That notwithstanding, it can be noted that there is an average increase in the modulus of elasticity of the composite material as BS content is increased. The steady increase in the flexural modulus as biosilica content is increased has equally been observed by other researchers though they used a different rice husk ash [18], [20], [21].

The results obtained are in agreement with the result obtained from percentage elongation at break. The increase in flexural modulus with an increase in BS loading may be attributed to the inability of the polymer chains to move freely because of the restriction caused by filler particles. This implies that BS filler provides stiffness to the polypropylene and therefore it is most suitable in applications where rigidity is required like domestic appliances and automobiles.

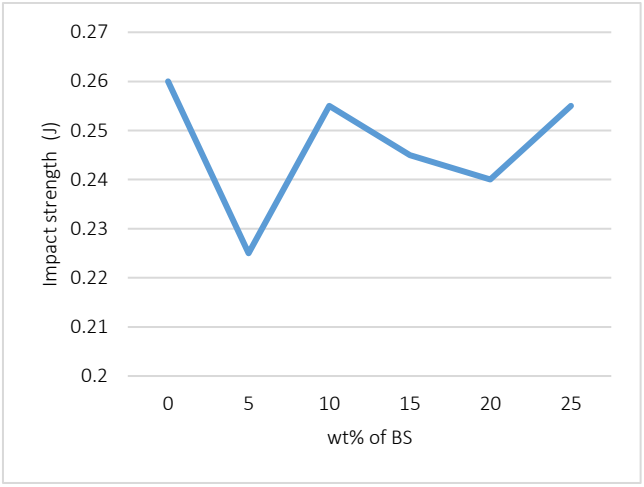

***Figure 6**. Variation of Impact strength with wt% BS addition*

From the data of the Charpy impact tests conducted to determine the toughness of the specimens as presented in Figure 6, it can be noticed there is a decrease in toughness as the BS content is increased. [22] also reported that the addition of inorganic silica in PP reduces the toughness and impact strength of the composite. However, the addition of compatibilizers has improved the impact strength. It can be noted that there is a significant decrease in the toughness of the PP when it is formed into a PP-BS composite with 5 %wt BS content. However, at 10 %wt BS loading, the value increases and then starts to gradually decline up until the last trial which is 25%wt BS loading. It was noted that the maximum and minimum value of the composite impact strength was 0.26 J at 0% composition and 0.225 J at 5% composition respectively.

The decrease in impact strength may be attributed to the weak interface between the BS filler and polypropylene matrix which would have stress concentration and generate cracks when there is an impact. The sharp decrease in toughness at 5 wt% BS loading may be attributed to the poor adhesion between the BS filler and the polypropylene matrix. This could be a result of improper mixing resulting in uneven dispersion of the fillers on the matrix.

The result of the microhardness test conducted on the specimens is presented here in Figure 7.

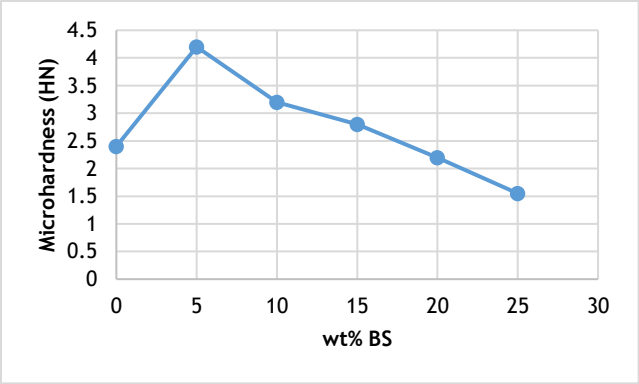

***Figure 7**. Hardness Value of the Composite Samples*

From the data presented in Figure 7, it can be seen that as BS loading was increased to 5%, the microhardness spiked and thereafter witnessed a steady decline as the BS loading was continuously increased until the final loading was done. The microhardness value is dependent on the complex surface properties of the material and the condition in which measurements are taken [23]. When this pattern is compared to studies done by other researchers [18], [20], [24], [25] it was found that there is a similarity in the initial increase in the hardness of the composites and then a gradual decrease as the biosilica loading was increased.

The optimum value of hardness was found to be 4.2 at 5% BS loading. This, therefore, implies that the microhardness valve is independent of the biosilica particles.

4 CONCLUSIONS

The research work is centred on the development and mechanical characterization of polypropylene-biosilica composite. It was found that the addition of BS to PP does not significantly change its ultimate tensile strength. It was also found that the addition of biosilica to PP causes an average rise in other mechanical properties of the PP matrix-like Young's Modulus and Flexural Modulus. However, other mechanical properties such as toughness, elongation at break and hardness, and the addition of biosilica to the PP matrix caused an average decrease in these properties.

These findings suggest that BS filler provides stiffness to PP and therefore it is most suitable in applications where rigidity is required like domestic appliances, the automobile and the aviation industry, hence could be a replacement for inorganic silica.

Conflict of interest

There is no conflict of interest between the authors.

Authors contributions

Sudi conducted the laboratory experiments and proofread the write-up, while Mshelia conceptualized the research and authored the research article. Sudi's responsibilities encompassed performing the laboratory experiments, ensuring their proper execution, and meticulously proofreading the write-up for errors and improvements. Mshelia, on the other hand, played a key role in conceptualizing the research, crafting the overall structure, and writing the research article.

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Research and publication ethics

The study is complied with research and publication ethics

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