

Mechanism of Water Absorption Behaviour in Groundnut Shell Powder Filled Waste HDPE Composites

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Received 22 November 2020/Accepted 02 December 2020/Published online: 05 December 2020

Abstract: The mechanism of water absorption and its effect on mechanical properties of groundnut shell powder (GSP) reinforced waste high density polyethylene (wHDPE) was studied at different temperatures. The composite samples were developed via melt mixing and compression moulding techniques respectively. The percentage weight fraction of reinforcement was varied (0, 5, 10, 15, 20 and 25 %). The initial rate of water absorption and the maximum were observed to increase for all GSP filled wHDPE composites samples as the weight fraction of reinforcement increases. The maximum moisture uptake at room temperature was 4.77 % after 720 hours of exposure compared to 6.74 % moisture uptake at elevated temperature showing a 6.73 % increase for 25 % GSP-wHDPE composites. This indicates that sorption at room temperature takes longer time to reach equilibrium than sorption at elevated temperatures. The effects of moisture absorption and temperature on several performance parameters such as tensile strength, flexural strength, hardness value and impact strength revealed a decrease in these properties after 720 hours of immersion in distilled water at 27 °C. This indicates that long term exposure of GSP-wHDPE composites in water affects the interfacial adhesion between the polymer matrix and the fibre, creates de-bonding, leading to decrease in mechanical properties.

Key Words: Groundnut shell powder, mechanical properties, mechanism, water absorption, waste high density polyethylene.

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1.0 Introduction

Adsorption defines sticking of materials to a surface through physical or chemical mechanism (Okwunodulu, and Eddy, 2014; Odoemelam and Eddy, 2009). The significance of adsorption of water is significant in understanding the mechanism of adsorption because a major application of adsorption to remove unwanted materials mostly occurs in aqueous solution (Essien and Eddy, 2015). For example, adsorption of waste materials from polluted water has been widely investigated and found to be one of the best options of removing pollutants from waste water (Odoemelam *et al.*, 2018)). Several adsorbents are currently in use but most of them suffer from the problems of porosity, fast degradability, uneven surface area, thermal instability and other factors (Eddy, 2009). Some of these problems could be overcome if the knowledge of water adsorption is fully comprehended. Natural fibres possess several advantages compared to synthetic fibres, making them attractive reinforcements for composite materials. They are cheap, abundant and renewable, and have good specific properties such as tensile strength and stiffness (Jacob *et al.*, 2018). However, natural fibres have a few disadvantages when used as reinforcement, such as higher moisture absorption which brings about dimensional changes, thus leading to micro-cracking and poor thermal stability (Alomayri *et al.*, 2014).

The poor resistance of natural fibres such as groundnut shell powder (GSP) to water absorption can also have undesirable effects on the physical and mechanical properties of the composites (Jacob *et al.*, 2019). Therefore, it is important to investigate in detail the water absorption behaviour of natural fibre reinforced composites in order evaluate the consequences the water absorbed may have on the adsorption process and the method of minimizing it (Jacob, 2019). Several studies on the use of natural fibre reinforced polymeric composites have shown that the sensitivity of certain mechanical and thermal properties to moisture uptake can be reduced by the use of coupling agents and fibre surface treatment (Tong *et al.*, 2014; Jacob *et al.*, 2018).

Some recent works on the water absorption behaviour of polymeric composites have been reported. Betene *et al.* (2018) studied the diffusion behaviour of water vapour sorption onto natural fibre. The results show that the kinetic adsorption is rapid at the first moments no matter the relative humidity and begin saturation at the seventh hour. The moisture absorption curves obey Fick's law and diffusion coefficients have been deduced. Noutegomo *et al.* (2019) reported the diffusion behaviour of water vapour sorption in natural fibre composite: Plaster/Rhectophyllumcamerunense and the results they obtained indicated that the kinetic of the adsorption is also rapid at first and is also independent of relative humidity but after 28 hours, saturation was observed. However, during this regime, the maximum moisture uptake increases with the relative humidity. Mechanical properties and water absorption characteristics of composites reinforced with cotton fibres recovered from textile waste was studied by Kamble and Behera (2020). The equilibrium water content of the composites was observed to increase with an increment in fibre volume fraction. Considering the scientific significance of reinforcing materials for effective adsorption process, the present study seeks to evaluate the water absorption behaviour and its effect on mechanical properties of GSP reinforced waste high density polyethylene composite with a view in determining its suitable working environment.

2.0 Materials and Method

2.1 Sample collection and preparation

Waste bottle caps with resin identification code "2" made from commercially high density polyethylene (HDPE) was collected from refuse dumps and plastic waste collection centres in Samaru and Sabon Gari areas of Zaria in Kaduna State, Nigeria. These samples were thoroughly washed with water, dried and shredded into particles of smaller sizes which constitute the polymer matrix (Jacob, 2019).

The groundnut shell powder (GSP) used as reinforcement was also sourced locally, sun-dried, pulverized and sieved to 150 μm . The GSP was initially alkaline pre-treated by suspending the fibres in 5 % NaOH and then benzoyl chloride for 15 minutes. The isolated fibres were soaked in ethanol for 1 hour to remove the excess benzoyl chloride and finally washed with distilled water and dried in oven at 80 $^{\circ}\text{C}$ for 5 hours (Jacob *et al.*, 2018; Jacob, 2019).

2.2 Composite production

The composite samples were produced by compounding process achieved by the addition of the shredded waste HDPE while the rolls were in counter clockwise motion for a period of 10 minutes at a temperature of 170 $^{\circ}\text{C}$. Upon achieving a paste like matrix, treated groundnut shell powder was introduced by gently applying manually as the rolls rotate at a rate of 500 rpm. The % weight fraction of reinforcement was varied from 0-25 % (0, 5, 10, 15, 20, and 25). Curing of the samples was then carried out using hydraulic press at a temperature of 160 $^{\circ}\text{C}$ and a compression pressure of 4 Pa for 10 minutes. Samples obtained were cooled and machined in preparation for characterization tests (Jacob *et al.*, 2018). Plate 1 shows the waste HDPE materials used in this work.



Plate I: HDPE waste used in the study.

2.3 Mechanical Property Test

2.3.1 Tensile test

The tensile testing of the samples was determined using the ASTM D638 (2014) recommended method. The samples were machined to dumbbell shape and then placed in computerized Instron universal tensile testing machine 3369 model, which measured the tensile strength and elastic modulus were evaluated.

2.3.2 Flexural strength

Flexural strength was measured using the ASTM D790 (2015) method. The measurement was carried out under a three-point bending approach using a universal testing machine. The distance between the spans was 40 mm and the strain rate was 5 mm/min. The flexural strength (MPa) was calculated using equation (1):



$$\Sigma = \frac{3Pl}{2bt^2} \quad (1)$$

where l is the length of specimen span between support (mm), P is the maximum deflection force (N)

b = width of specimen (mm) and t is the thickness of specimen (mm)

2.3.3 Test for hardness

The test for shore hardness was conducted with a sample whose dimension was 30 × 30 × 5 mm using a Durometer Shore ‘A’ according to ASTM D2240 (2015) method. Measurements were carried out on five different points on the samples and the average was taken as the hardness of the composites.

2.3.3 Impact strength test

The impact test on the developed composite samples was carried out using a fully instrumented Charpy impact testing CAT NR412 model according to ASTM F2231-02 (2013). The dimensions, gauge length and V-notch were chosen according to the standard.

2.4 Water absorption study

Water absorption test was carried out according to ASTM D570 (2010) method. The test sample was an oven dried specimen of dimension 76 × 25 × 5 mm immersed in water at ambient temperature for 24 hours. After immersion period of 24 hours, the specimens were removed and patted dry with a cloth (lint free) and then reweighed using a Sartorius ED 224S digital Analytical balance. In order to evaluate long term moisture absorption on the composites, the process was repeated at 48, 72, 96, up to 720 hours exposure. The dried weight before ($W_{initial}$) and after weight immersion (W_{final}) were noted. The water absorption was determined as follows:

$$W = \frac{W_{final} - W_{initial}}{W_{final}} (\%) \quad (2)$$

3.0 Results and Discussion

3.1 Mechanism of water sorption behaviour of GSP reinforced RHDPE composites

The percentage of water absorption in the composites was calculated by weight difference between the samples immersed in water and dry samples using the modified equation (equation 3)

$$\Delta W = \frac{w_t - w_o}{w_o} \quad (3)$$

where ΔW is the moisture absorption, W_0 and W_t are the mass of the specimen before and during

aging respectively. Different models have been developed in order to describe moisture absorption behaviour of composite materials (Sreekala and Thomas, 2003; Wang et al., 2006). According to Dhakal *et al.* (2006), for one-dimensional moisture absorption, each sample is exposed, on both sides, to the same environment and the total moisture content (G) can be expressed as follows:

$$G = \frac{w - w_i}{w_s - w_t} = 1 - \frac{8}{\pi^2} \sum_{j=0}^{\infty} \frac{1}{(2j+1)^2} \exp \left[\frac{(2j+1)^2 \pi^2 D_x t}{h^2} \right] \quad (4)$$

where w_i is the initial weight of the moisture in the material and w_s is the weight of moisture in the material when the material is fully saturated and is in equilibrium with its environment. D is the mass diffusivity in the composite. This is an effective diffusivity since all the heterogeneities of the composites have been neglected; h is the thickness of specimen and t is the time while j is the summation index. The diffusion coefficient is an important parameter in Fick’s law. Solving the diffusion equation for the weight of moisture, and rearranging in terms of the percent moisture content, the following relationship is obtained:

$$W = \frac{4W_m}{h} \left(\frac{t}{\pi} \right)^{0.5} D_x^{0.5} \quad (5)$$

where W_m is the equilibrium moisture content of the sample? Using the weight gain data of the composites with respect to time, a graph of weight gain versus time was plotted. The diffusion coefficient was calculated using the following formula:

$$D = \frac{d^2}{\pi^2 t_{70}} \quad (6)$$

where d is sample thickness in mm and t_{70} is the time taken to reach 70% saturation in seconds.

The diffusion parameters defined by Fick’s laws for the composite materials was evaluated through the slope of the first part of the weight gain curve versus square root of time, as described by equation 5 (William *et al.*, 1996). The coefficient of diffusion (D), defined as the slope of the normalised mass uptake and as a function of \sqrt{t} agrees with the model expressed in equation 7

$$D = \pi \left(\frac{kh}{4W_m} \right)^2 \quad (7)$$

where, k is the initial slope of a plot of $M(t)$ versus $t^{1/2}$, W_m is the maximum weight gain and h is the thickness of the composites.



Fig. 1 depicts the percentage of weight gain as a function of square root of time for the control (wHDPE) sample and the composites at various weight fraction of reinforcement of GSP-wHDPE samples immersed in distilled water at room temperature. The maximum percentage of weight gain for 0, 5, 10, 15, 20 and 25% weight fraction of reinforcement immersed at room temperature for 720 hours were 0.39, 1.38, 2.43, 2.92, 3.82 and 4.77%, respectively. The water absorption process for all composite samples (except wHDPE, which was observed not to absorb) was initially linear before slowing down with time towards the point of attainment of saturation after prolonged time. Therefore, the observed trend follows the Fickian diffusion process. Both the initial and maximum rate of water absorption

was observed to increase for all GSP-wHDPE composite samples as the weight fraction of reinforcement increases. This phenomenon can be explained by considering the water absorption

characteristics of natural fibre-based composites. When the composite is exposed to moisture, the hydrophilic GSP fibre swells resulting in micro cracking of the composite occurs (Dhakal *et al.*, 2006). The high cellulose content in GSP might have also contributed to the penetration of moisture to the interface further contributes to more water penetrating into the interface through the micro cracks and enhance can induce fibre swelling and creation of swelling stresses and subsequent composite failure (Bismarck *et al.*, 2002). As the composite cracks and gets damaged, capillarity and transport via micro cracks becomes active. The capillarity mechanisms may most likely involve the flow of water molecules along fibre-matrix interfaces and a process of diffusion through the bulk matrix. Further attack of the interface by the water molecule may finally lead to de-bonding of the fibre and the matrix.

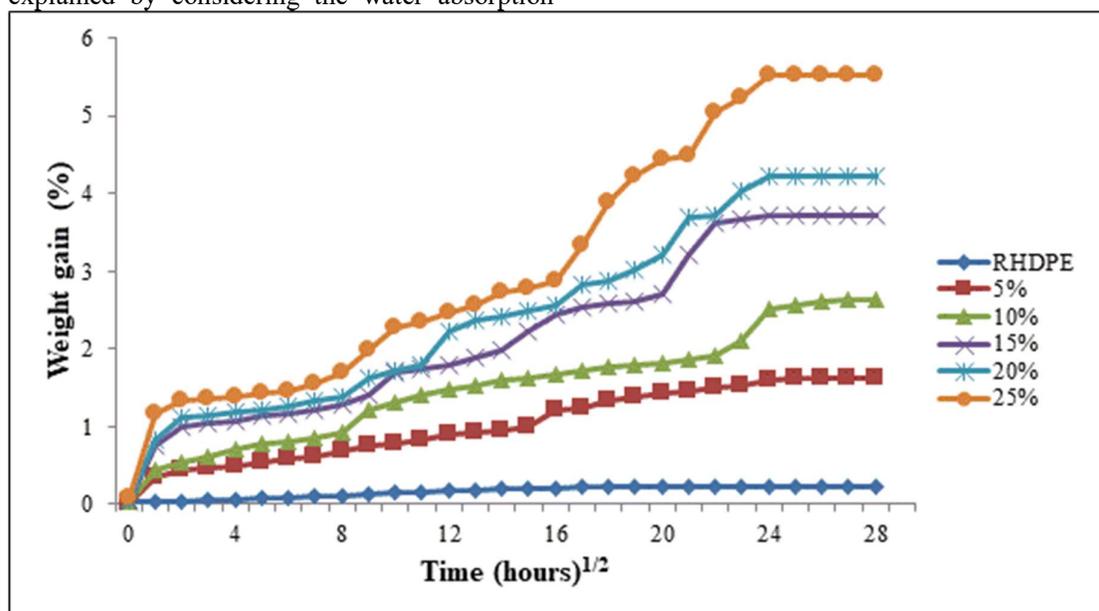


Fig. 1: Water absorption curves at room temperature (RT) for GSP-RHDPE composites

Fig. 2 shows the plots for the variation of percentage of weight gain for RHDPE, 5, 10, 15, 20 and 25% GSP reinforced wHDPE composites (immersed in water at 100 °C) with time. The respective percentage water absorption were 0.46, 1.78, 2.86, 3.38, 4.94 and 6.74% respectively. From the plots, it is evident that weight fraction of reinforcement is affected by time. The rate of approach to equilibrium is clearly more rapid for the 100°C specimens than the samples immersed at room temperature. Higher temperatures seem to

accelerate the moisture uptake behaviour. When the temperature of immersion was raised to 100 °C, the moisture saturation time (MST) is greatly

shortened. For the sample containing 25%wt of PPP at room temperature, it takes 720 hours to reach MST whereas at 100 °C the MST is 432 hours. The MST in this case was shortened by 288 hours. This shows that sorption at room temperature takes far longer period to reach equilibrium than sorption at elevated



temperatures. In addition to the increase in weight gain percentage, it also shows that weight gain is higher for sample immersed in boiling water than at room temperature. For 25%wt samples, the weight gain percentage at moisture saturation point at boiling temperature is approximately 29% higher than at room temperature. It is also evident that there are different sorption behaviours for immersion at room temperature than for elevated temperature indicating different aging mechanisms (Jacob, 2019). The higher and faster weight gain upon exposure to boiling water may be attributed to different diffusivity of water into the material leading to moisture induced interfacial cracks at an accelerated rate as a result of degradation in the fibre-matrix interface region as well as the state of water molecules existing in

the GSP-wHDPE composites. Other studies reported a similar trend for ageing of polymer composites at elevated temperatures (Dhakal *et al.*, 2006; Srubar *et al.*, 2012; Jacob, 2019).

The moisture uptake at elevated temperatures compared to RT seems to obey non-Fickian behaviour showing a 6.73% higher moisture uptake for 25% fibre reinforced composites. The moisture uptake results in this study show Fickian behaviour at room temperature and non-Fickian behaviour at boiling temperature. This is attributed to the moist, high temperature environment, and micro cracks developed on the surface and inside the materials (Zhou and Lucas, 1995). After the occurrence of damage in the composites, water transport mechanisms become more active (Jacob, 2019).

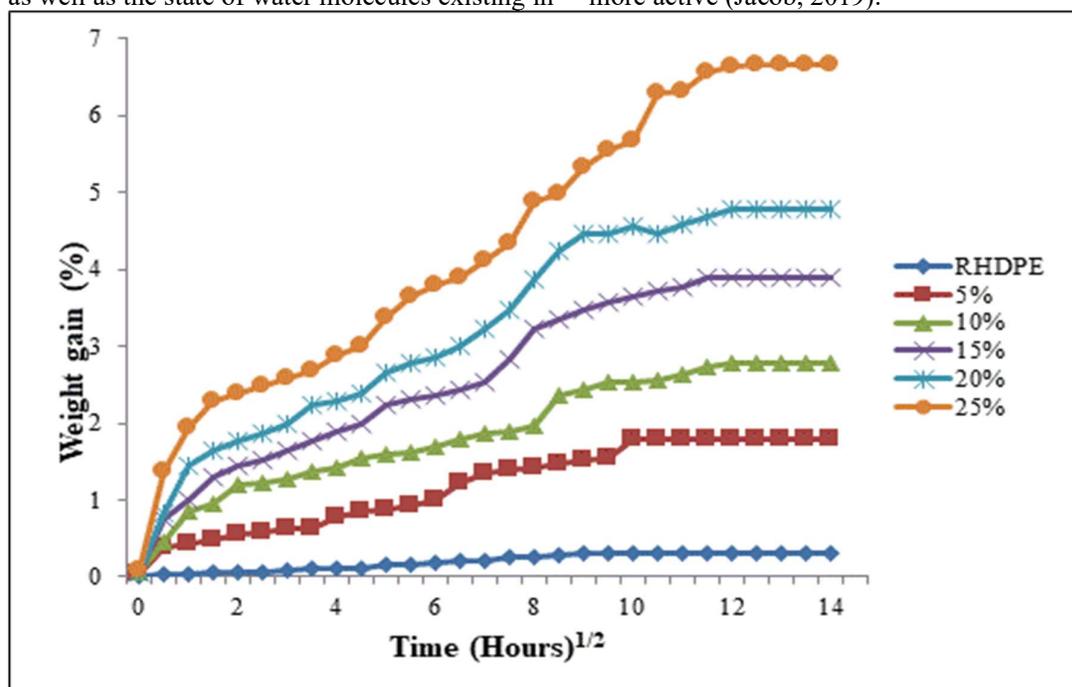


Fig. 2: Water absorption curves at boiling temperature for GSP-wHDPE composites

3.2 Effect of long term moisture absorption on mechanical properties

3.2.1 Tensile characteristics

Variation of the ultimate tensile strength as a function of weight fraction of reinforcement for GSP-wHDPE samples (exposure time up to 720 h at 27°C) is shown in Fig. 3. It is interesting to note that there was an observable increase in tensile strength at 5% weight fraction of reinforcement by 36.4% after immersion in water. This increase in tensile strength for 5wt% PPP reinforced sample implies that further cross-linking or other mechanisms which took place resulted in the

enhancement of the material strength. The tensile strength, however, drops by 26.3%, 36.9%, 25.8%, and 23.0%, respectively, for 10wt%, 15wt%, and 20wt% and 25wt% GSP reinforced specimens. Generally, for composites with higher weight fraction of reinforcement immersed in water, it is expected that the relative extent of decrease in tensile properties should be greater compared to dry samples. This could be due to the fact that high amounts of water causes swelling of the fibres, which could fill the gaps between the fibre and the polymer matrix and eventually lead to a decrease in the mechanical



properties of the composites (Dhakal *et al.*, 2006; Jacob, 2019).

3.2.2 Flexural strength

The variation of the flexural strength as a function of weight fraction of reinforcement for dry and water immersed (exposure time 720 h at 27°C) GSP-wHDPE composites is shown in Fig. 4. The

flexural strength drops as the weight fraction of reinforcement increases. This could be due to the fact that the immersion of composite samples in water affects the interfacial adhesion process between the fibre and the matrix. This can create de-bonding, leading to a decrease in mechanical properties (Alomayri *et al.*, 2014).

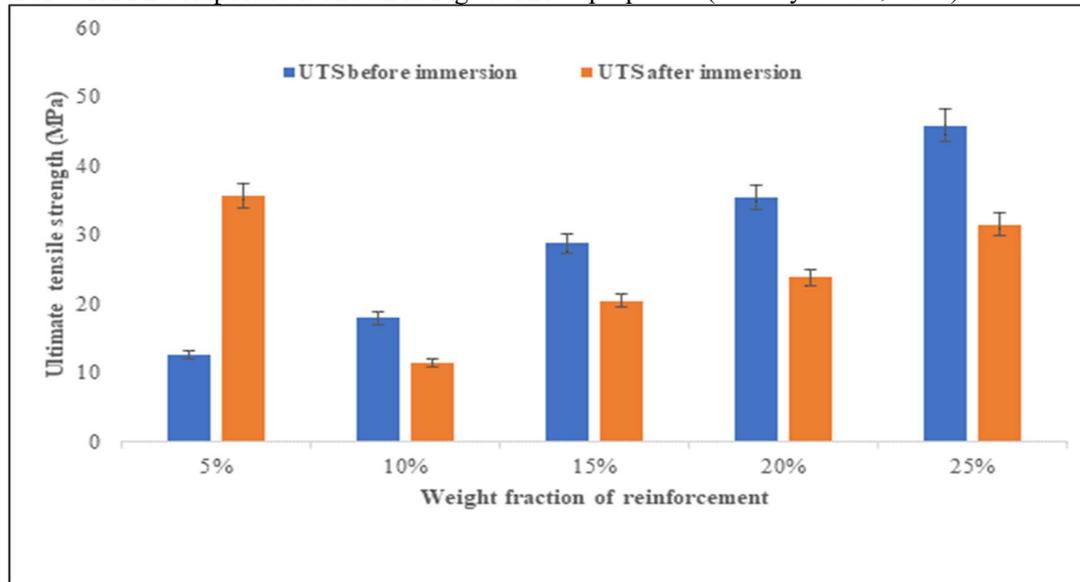


Fig. 3: Effect of water absorption on the tensile strength of GSP-wHDPE composites

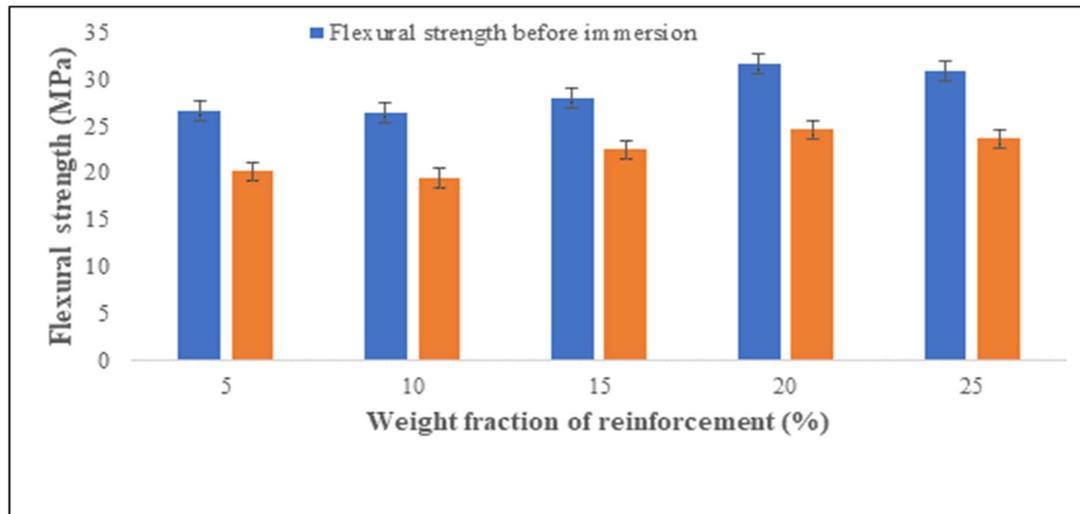


Fig. 4: Effect of water absorption on the flexural strength of GSP-wHDPE composites.

When the fibre-matrix interface was accessible to moisture in the environment, the GSP fibres swelled up. This resulted in development of shear stress at the interface, and led to the ultimate de-bonding of the fibres, delamination and loss of structural integrity. This is at par with observation reported by other authors (Dhakal *et al.*, 2006; Jacob, 2019).

3.2.3 Influence of moisture absorption on the modulus

Table 1 presents values of tensile and flexural modulus for both dry and water- immersed specimens at room temperature. It can be seen from the results that moisture absorption causes change in the modulus as determined by tensile and flexural tests. The tensile modulus decreases



for all GSP reinforced samples. The reduction in tensile modulus for 5%wt-25%wt GSP-wHDPE specimens compared to dry specimens were 50.15%, 64.21%, 67.35%, 71.05% and 78.28% respectively. A plausible explanation for this would be that the elastic modulus is a fibre sensitive property in composites and is affected by moisture absorption. This effect is particularly greater for composites with higher fibre content, which stress transfer capability between fibre and matrix interface gets sharply reduced due to moisture content. Similar observations have been reported by other authors (Dhakal *et al.*, 2006; Alomayri *et al.*, 2014; Marinho *et al.*, 2015).

It was also observed that the flexural modulus was not significantly affected by moisture absorption. However, the increase in flexural modulus was more significant in samples with higher fibre and moisture contents. It would be intuitive to assume that the effect of fibre reinforcement to be less critical for the flexural failure stress than in tensile failure mode. This is because the flexural samples fail in combination of compression, shear and tension mode (Dhakal *et al.*, 2006; Jacob, 2019).

3.2.3 Influence of moisture absorption on the modulus

Table 1 presents results of tensile and flexural modulus for both dry and water-immersed specimens at RT. It can be seen that moisture absorption causes change in the modulus as determined by tensile and flexural tests. The tensile modulus decreases for all GSP reinforced samples. The reduction in tensile modulus for 5 %wt-25 %wt GSP-wHDPE specimens compared to dry specimens are 50.15 %, 64.21 %, 67.35 %, 71.05 % and 78.28 % respectively. A plausible explanation for this would be that the elastic modulus is a fibre sensitive property in composites and is affected by moisture absorption. This effect is particularly greater for composites with higher fibre content, which stress transfer capability between fibre and matrix interface gets sharply reduced due to moisture content. Similar observations have been reported by other authors (Dhakal *et al.*, 2006; Alomayri *et al.*, 2014; Marinho *et al.*, 2015).

The flexural modulus, however, is not adversely affected by moisture absorption. The increase in flexural modulus is more pronounced with higher fibre content specimens and hence higher moisture content. It would be intuitive to assume that the effect of fibre reinforcement to be less critical for the flexural failure stress than in tensile

failure mode. This is because the flexural samples fail in combination of compression, shear and tension mode (Dhakal *et al.*, 2006; Jacob, 2019).

Table 1: Elastic and flexural modulus for dry and wet GSP-RHDPE samples

Weight of fibre (%)	Elastic modulus (MPa)		Flexural modulus (MPa)	
	dry	wet	Dry	Wet
5	65.60	50.15	125.19	128.21
10	75.42	64.21	128.72	136.50
15	79.56	67.35	138.61	147.65
20	85.45	71.05	166.65	177.11
25	90.70	78.28	176.77	192.24

3.2.4 Hardness

The effect of GSP contents on the hardness of RHDPE composites is presented in Fig. 5. There was gradual increase in hardness of RHDPE composites reinforced with 5, 10, 15, 20, and 25%wt GSP relative to the unreinforced polymer. This enhancement in hardness may be attributed to the distribution of the test load on the fibres, which decreased the penetration of the indenter on the surface of the composite material and consequently improved the hardness of the material. Hardness decreases in all GSP reinforced samples in wet condition, the decrease being dependent upon the amount of GSP incorporated. This behaviour is associated with the weakening of interface between the wHDPE matrix and the GSP fibre caused by the water absorption. Similar observations have been reported by other researchers who worked with natural fibre-based composites. Dhakal *et al.* (2014) reported that as water absorption increased, the hardness of flax fibre-reinforced composites decreased, and found that the deformation depth increased for water-immersed specimens compared to dry ones, due to the hydrophilic nature of the fibres. These changes could lead to the formation of a weak fibre-matrix interface. In the case of GSP reinforced HDPE composites, when water uptake reaches saturation level, the bound water and the free water remain in the composite as a reservoir. This leads to softening of the fibres and weakening of the fibre-matrix adhesion, resulting in reduced materials' properties. Similar observation has been reported by Alomayri *et al.* (2014) who investigated the effect of water absorption on the mechanical properties of

cotton fabric-reinforced geopolymer composites.

3.2.5 Impact strength



Impact strength of fibre-reinforced polymer is governed by the matrix-fibre interfacial bonding, and properties of both matrix and fibre. When the composites undergo a sudden force, the impact energy is dissipated by the combination of fibre pullouts, fibre fracture and matrix deformation (Wambua *et al.*, 2003). Normally, in fibre-reinforced polymer composites, impact strength increases as fibre content increases because of the

increase in fibre pull out and fibre breakage (Alomayri *et al.*, 2014).

The effect of fibre contents on the impact strength of dry and wet GSP reinforced wHDPE composites are shown in Fig. 6. It is evident from Fig. 6 that the impact strength significantly increased as GSP content increased in dry composites up to 25%wt in which decrease in impact strength was observed.

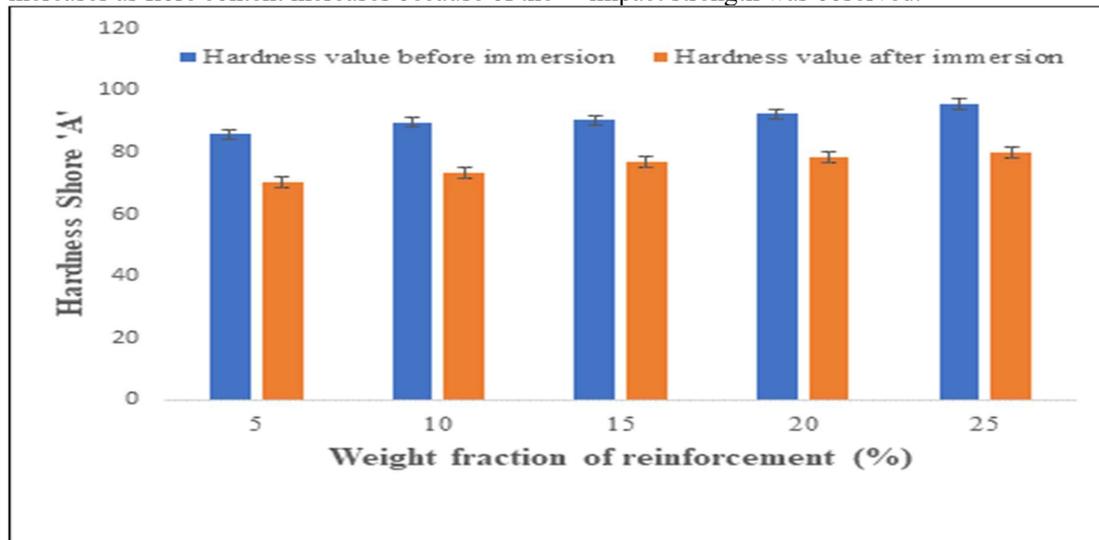


Fig. 5: Effect of water absorption on the flexural strength of GSP-wHDPE composites

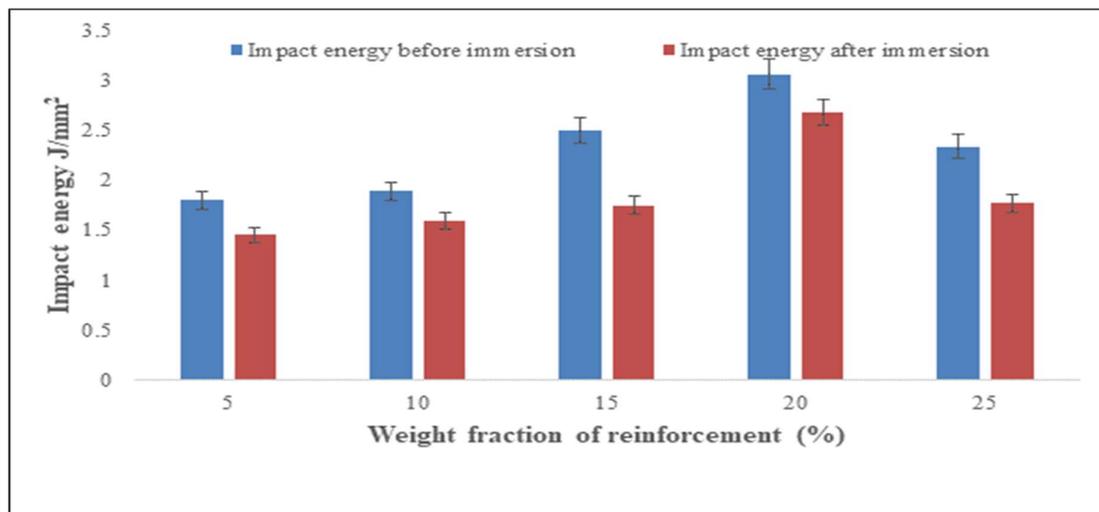


Fig. 6: Effect of water absorption on the impact energy of GSP-wHDPE composites

The presence of GSP in the matrix increases the ability of these composites to absorb impact energy. In dry conditions, the addition of GSP with contents of 5, 10, 15, 20 and 25%wt increases the impact strength from 1.80 to 2.56, 3.67 and 3.45J/mm², respectively, compared to unreinforced wHDPE. A similar remarkable improvement in impact strength was reported by

Alomayri *et al.* (2014). The decrease in impact properties after water immersion can be related to the weak fibre-matrix interface, which resulted in a reduction of the mechanical properties and dimensional stability of composites.

4.0 Conclusion

The mechanism of water absorption behaviour and its effect on mechanical properties of



groundnut shell powder reinforced RHDPE was successfully investigated and the following conclusions are made:

- The water sorption process for all composite samples at RT except unreinforced wHDPE which hardly absorbs water is linear in the beginning, then slows and approaches saturation after prolonged time, following Fickian diffusion process.
- The moisture uptake at elevated temperatures compared to sorption at RT seems to obey non-Fickian behaviour showing 6.73% higher moisture absorption for 25 % GSP reinforced composites.
- Moisture absorption at RT takes longer time to reach equilibrium than sorption at elevated temperatures.
- With the exception of flexural modulus, all the mechanical properties of the composites investigated were observed to be adversely affected by long term moisture absorption.

Conflict of Interest

The authors declare that there is no conflict of interest.

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The authors declare no conflict of interest.

