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Effect of Bio-composite hybridization stacking sequencing analysis on physical, mechanical and morphological behaviours of glass and Ukam fibre reinforced epoxy composites

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Abstract: *In this study, the compression moulding technique was used to fabricate hybrid composites using different wt. % of the glass and ukam fibers in different stacking sequences. The physical properties studied were density, water absorption, and wear resistance while mechanical properties included tensile strength, hardness, and impact strength and morphology studies were performed for the glass and ukam fibers and the effect of bio-composite hybridization stacking sequencing was studied. A technique for order of preference by similarity to the ideal solution model was used to determine the optimal reinforcement composition in the hybrid composites. The stacking sequence played a crucial role in the physical and mechanical characteristics of the hybrid composites. The GUG (stacking sequence: Glass (4 wt. %) - Ukam (4 wt. %) – Glass (4 wt. %)) hybrid composite showed optimal characteristics; tensile strength: 116 MPa, hardness: 41 Hv, impact energy: 3.55, void content: 1.09% and wear: 67 μm . However, if glass fibers were sandwiched between the ukam fiber layer, the composite showed lower physical and mechanical characteristics; tensile strength: 72 MPa, hardness: 28 Hv, impact energy: 3.1. The glass fiber reinforced composites (GG and GGG) exhibited better water absorption characteristics. The UUU composite's impact strength was comparable with the GGG composites due to the stacking of the composites. Moreover, the glass fiber composites stacking (UU to UGU) exhibited lower tensile strength than ukam fiber composites stacking (GG to GUG). The micro-structural analysis of the fractured composite surface revealed voids, de-lamination, interfacial bonding of the fibers with the matrix, fiber pull-out, and matrix distribution.*

KEYWORDS: Hybrid bio-composites; natural fibers; ukam fibers; epoxy resin

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1. INTRODUCTION

Environmental awareness has motivated researchers worldwide to study the natural fiber-reinforced polymer composite in recent years. Natural fiber composite can replace synthetic fiber reinforced composite owing to its advantages, such as being lightweight, bio-degradable (depending on polymer

matrix) and environment friendly ((Khalid et al. 2021), (Thyavihalli Girijappa et al. 2019)). However, major problems associated with natural fibers are poor moisture resistance, fiber wetting, and adhesion to the polymer matrix, which affect the physical and mechanical properties of composites ((Okoronkwo et al.

2021), (Okoronkwo et al. 2022)). Therefore, to overcome such problems, a combination of natural and synthetic fibers can be used, which can create a balance of natural and synthetic fiber properties in the hybrid composites (Okoronkwo et al. 2025).

It is known that natural fibers possess some limitations compared with those common

fibers such as glass and carbon, where it is having more inferior mechanical properties and a higher water absorption ((Dashtizadeh et al. 2017), (Okoronkwo et al. 2019)). Therefore, an introduction of hybrid bio-composites consists of two or more fibers in one matrix is seen as a solution to enhance the natural fiber-reinforced polymer composites' properties. (Nurazzi et al. 2021) stated that hybridizing one natural fiber with another natural fiber/synthetic fiber in one matrix will improve its thermal and mechanical than the individual fiber composites (Okoronkwo et al. 2024). This has shown that hybrid composites are more reliable for various applications, besides being more environmentally friendly.

The hybridization of natural fiber-based reinforced polymer composites can be done through a combination of natural–natural fibers, natural–synthetic fibers, natural fiber with carbonaceous materials, and natural fiber with metal (Okoronkwo et al. 2022). Due to their varied properties and considerations of interfacial adhesion, hybrid natural fiber composite materials are facing difficulties in fabrication. Composites are manufactured in a variety of ways, such as through basic mixing and open or closed moulding. Many factors can affect the interactions between the fiber and matrix, for example, and could be mild owing to the existing van der Waals forces, hydrogen bonding, and weak electrostatic interactions. In addition, a good interaction could also exist due to the chemical interactions between those materials. Therefore, studies on hybrid natural fiber composites keep increasing in order to discover the ability of hybrids to be a possible alternative, replacing various petroleum-based products.

Several studies have shown better physical properties for glass and natural fiber hybrid composites than their corresponding pure composites ((Aslan et al. 2018), (Giridharan 2019), (Thiyagu et al. 2023)). Natural and glass fiber-reinforced composites are cost-effective when used to make hybrid composites. (Kumar et al. 2018) showed that incorporating ramie fiber into the epoxy matrix substantially enhanced the wear resistance of composites. The sliding wear characteristics of the glass fiber composite increased with fiber reinforcement (Billady et al. 2020). (Ray et

al. 2017) studied the erosion behavior of the glass fiber composite incorporated with granite powder and showed that increasing the filler increased the composites' erosion wear resistance. Natural fiber composites exhibit high water content that may have a detrimental effect on the properties of the composites. However, it can be reduced with fiber loading. (Vedanarayanan et al. 2022) experimentally observed that the void content of hybrid composites of kevlar and Ramie fiber reduced when Ramie fiber loading was increased. The hybridization also improved the mechanical and physical properties of the composites. Hybridization of Kevlar fiber with E-glass fiber significantly improved the impact characteristics of composites in applications such as aircraft, automobile bumpers and bulletproof jackets (Vasudevan et al. 2020).

Several studies have reported that the mechanical and physical properties of hybrid composites can be improved by different stacking sequences ((Azlin et al. 2022), (Hassan et al. 2022)). (Portella et al. 2016) found that the tensile strength of the fabricated composites depends upon the layers of the glass fiber. (Kangokar Mukesh et al. 2022) studied the effect of stacking sequence and hybridization and reported that incorporating flax/kevlar in the epoxy matrix increased its mechanical characteristics. (Bharath et al. 2023) found that different stacking sequences of sheep wool and glass fiber composites improved the mechanical and chemical properties of the composite. (Barouni et al. 2022) investigated the fatigue behavior of a flax/glass hybrid composite and found that the fatigue strength increased with the inclusion of laminated flax fiber. The increased filler wt. % increases the vibrational characteristics of the natural fiber-reinforced composites (Prasad et al. 2022). (Thiyagu et al. 2022) showed that the hybrid composite of glass and ramie fiber with two layers of ramie fiber exhibited good tensile properties.

Although hybrid composites of ukam and glass fibers have not been studied, reports on the effect of stacking sequencing of the glass and ukam fibers on the physical and mechanical properties of hybrid composites are scarce in the literature. In the present study, hybrid composites were fabricated by reinforcing ukam and glass fibers (varying the wt. %) in different stacking sequences with epoxy polymer to study the physical and mechanical behaviour of the composite. A total of seven different composites were fabricated to study the physical (density, water absorption, and wear resistance), and mechanical (tensile strength, hardness, and impact strength) characteristics. Scanning electron microscopy (SEM)

micrographs of the fabricated hybrid composites were analyzed to study the morphological characteristics.

II. MATERIALS AND METHODS

2.1. Materials

E-Glass fibers (300 gsm), poly(vinyl alcohol) (PVA), epoxy resin LY556, and epoxy hardener HY951 were procured from Nycil CCRD company Limited and Ikenna chemicals, Nigeria. Ukam fibers were also procured from local sources in Imo State, Nigeria.

2.2. Composite fabrication

The composites were fabricated with the help of a compression-moulding machine (GRIMCO 30 Ton Hydraulic Press, Model 30-2-HT). The epoxy resin and hardener were mixed in a ratio of 10:1 at a curing temperature of 100 °C in a beaker such that no air was entrapped. Glass and ukam fibers were weighed in proportions mentioned in Table 1 and added to this mixture. The glass and woven ukam fibers were layered with 4 wt.% of the matrix as per the sequence shown in Table 1. Glass fibers were selected due to their chemical inertness, availability and low cost. Ukam fibers were selected for their higher mechanical

properties and thermal stability and ukam fibers incorporation in the matrix increased the composite strength significantly. The mylar sheet of PVA was used in the mould (250 mm × 250 mm × 7 mm), and releasing agent was sprayed on the mylar sheet to remove the composite from the sheet effectively. The closed mould was loaded into the compression-moulding machine (50 °C) and subjected to a compressive load. Proper care was taken to minimize the voids formations during the fabrication process of composites as the presence of voids has a detrimental effect on mechanical properties. The mould temperature slightly increased due to the exothermic polymerization reaction, which lowered the viscosity of the matrix system allowing the epoxy impregnation in the fibers. The compressive load was then reduced and maintained at the same load for 1 h to prevent resin flash and minimize fiber disturbance. The mould was cooled under a compressive load for 24 h to inhibit geometrical distortion of the composite plate. Finally, the fabricated composites of dimensions 250 mm × 250 mm × 7 mm were released from the mould. The weight of each fabricated composite was fixed (500 g). The test samples were prepared as per ASTM standards. Three similar samples were prepared for testing and the averages of these reading were reported.

Table 1. Designation, composition and stacking sequence of the fabricated composites.

S. no.	Composite	Epoxy polymer (wt.%)	Stack-1	Stack-2	Stack-3
1	UUU	88	4 wt.% ukam	4 wt.% ukam	4 wt.% ukam
2	UGU	88	4 wt.% ukam	4 wt.% glass	4 wt.% ukam
3	GUG	88	4 wt.% glass	4 wt. % ukam	4 wt.% glass
4	GGG	88	4 wt.% glass	4 wt.% glass	4 wt.% glass
5	UG	92	4 wt.% ukam	4 wt.% glass	-
6	UU	92	4 wt.% ukam	4 wt.% ukam	-
7	GG	92	4 wt.% glass	4 wt.% glass	-

2.3. Characterization of physical, mechanical and morphological properties

2.3.1. Density

The density affects the component’s mechanical and physical properties. The theoretical density (ρ_{th}) of composites in terms of weight fraction was obtained using the relation (Prasad et al. 2020):

$$\rho_{th} = \frac{1}{\sum (W_f/\rho_f) + W_m/\rho_m}$$

where W_f - weight fraction of fiber, W_m - weight fraction of matrix, ρ_f - density of fiber, ρ_m - density of matrix. The composites’ actual density (ρ_{exp}) was

calculated with the simple water immersion technique of the composites. The volume fraction of voids (V_v) in the composites was determined using the ρ_{th} and ρ_{exp} density of composites using the following equation (Prasad et al. 2020):

$$V_v = \frac{\rho_{th} - \rho_{exp}}{\rho_{th}}$$

where ρ_{exp} – experimental density of composite, respectively.

2.3.2. Water absorption test

The water absorption test in the present study of the composite sample (50.8 mm diameter and 7 mm thick) was performed as per ASTM standards. The sample weight was recorded using a digital weighing machine with an accuracy of ±1 mg. The test samples were immersed in water for 24 h. After 24 h, the water over the sample’s surface was cleaned with tissue paper, and the weight was recorded. This was repeated until a steady state (no more water uptake by samples) was obtained for the test samples. The samples for the water absorption test are shown in Fig. 1a. The percentage of water content (m %) was determined using the following equation (Keerthi Gowda et al. 2022):

$$m = \left(\frac{w_t - w_d}{w_d} \right) \times 100$$

where: w_t - the weight of the sample in time t , w_d - the sample’s initial or dried weight.



Fig. 1(a). Water absorption test samples.



Fig. 1(b). Pin-on-disc wears test samples.



Fig. 1(c). Hardness test samples.



Fig. 1(d). Impact test samples.



Fig. 1(e). Tensile test samples.



Fig. 1(f). Ukam fiber mat.



Fig. 1(g). Glass fiber.

2.3.3. Wear test

A pin disc tribometer was used to assess the fabricated composites’ wear behaviour. In this test, the test samples were loaded against a rotating pin. The test samples (length: 32 mm, diameter: 6 mm with spherical tip) were prepared, and testing was done as per ASTM G99 standard. The rotating disc speed and load were kept at 500 rpm and 10 N. Fig. 1b shows the wear test samples.

2.3.4. Hardness test

The Vickers hardness test was conducted per the ASTM standard. The hardness samples (Fig. 1c) were polished before applying the load (test load: 5 kgf). The square base pyramid-shaped diamond indenter was used. The hardness of the fabricated composites was

measured by a diamond indenter with an apical angle of 136°.

2.3.5. Tensile test

The tensile test was performed as per ASTM D3039 standard on a universal testing machine (UTM). The test sample dimensions were taken as 115 × 6 × 7 mm. Fig. 1e shows the tensile test samples.

2.3.6. Impact test

The ability to quantify the impact properties of composites leads to an advantage in product liability and safety. The Charpy test was used to conduct the impact test (Fig. 1d) using a Charpy impact tester. The sample (L × W × t = 55 mm × 10 mm × 2 mm, and with a V notch at the center) was freely rested, and the impactor (projectiles) was stuck at the center of the sample.

2.3.7. Morphological test

A scanning electron microscope (SEM) was used to study the morphological behaviour of the tested composites. SEM analysis was performed using Carl Zeiss EVO 18 instrument. For SEM analysis, the fractured sample's surfaces were gold-sputtered using an ion-sputter device. The SEM images were used to evaluate the fractured surfaces, bonding between fibers and the epoxy, and provide information about voids.

III. RESULTS AND DISCUSSION

3. Results and discussion

3.1. Physical characteristics of composites

Table 2 shows the fabricated composites' theoretical density, experimental density, and voids fraction. The experimental density values are lower than the theoretical density values due to the presence of voids and pores. A high void fraction (>5%) affects the mechanical properties of the composites (Prasad et al. 2019). The lowest void content (0.18%) was in the UU composite, while the maximum void content (1.64%) was in the GGG composite. The GGG composite was prepared using chopped glass fiber. The matrix material could not penetrate the chopped glass fiber mat properly, leading to voids in the GGG composite. The ukam layer consisted of a woven mat where warp and weft were at 90°. Hence, matrix material easily penetrated the ukam fiber mat, resulting in lesser voids. As the fiber loading (glass or ukam fiber) increased, the void content increased. Similar trends have been reported in previous studies (Wang et al. 2019; Zainudin et al. 2020). However, the increment rate in the ukam

Table 2. Density and void % of different composite samples.

Composites	Theoretical density (g/cm^3)	Experimental density/ g/cm^3	Void content (%)
UUU	1.173	1.176	0.60
UGU	1.189	1.185	1.12
GUG	1.204	1.201	1.09
GGG	1.221	1.210	1.64
UG	1.177	1.182	0.44
UU	1.162	1.170	0.18
GG	1.202	1.196	0.52

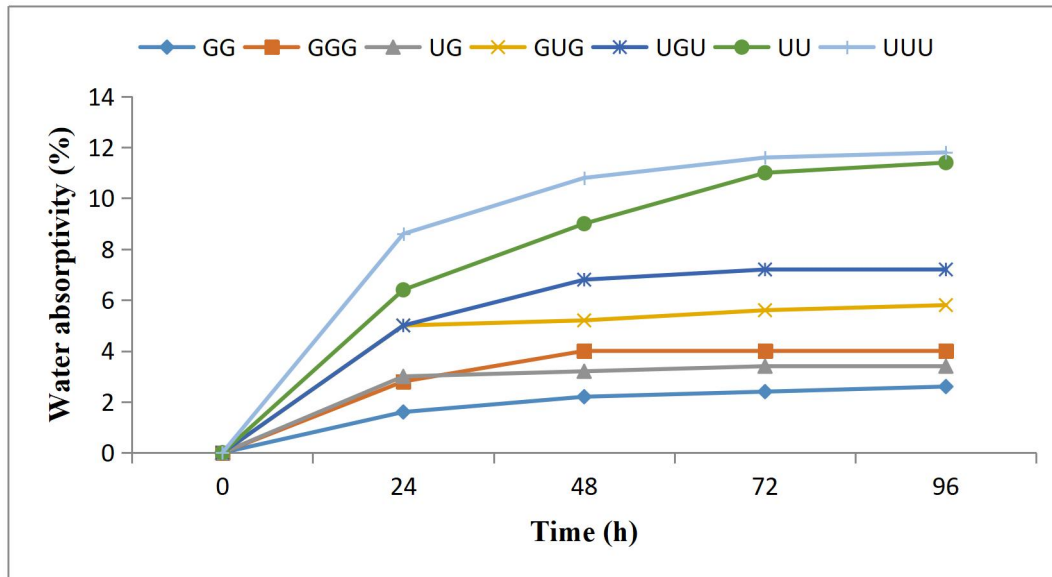


Fig. 2: Water absorption behaviour of the composite samples

fiber composites (UU with 8 wt.% and UUU with 12 wt.%) was lesser than in glass fiber composites (GG with 8 wt.% and GGG with 12 wt.%). This can be attributed to the epoxy polymer matrix compatibilization of ukam fiber and the low packing ability of the glass fiber (Prasad et al. 2020).

The water absorption in the composites was less when glass fiber wt. % in the hybrid composites was increased since glass fiber is hydrophobic. The maximum water absorption was obtained for the UUU composite, and the minimum water absorption was obtained in the GG sample. Again, natural fibers (ukam) absorb more moisture than synthetic (glass). While comparing the UU and UUU composites, the water absorption in the former was lower, as it has lower ukam fiber content. This can be attributed to the ukam fiber containing many polar hydroxide groups, resulting in a high moisture absorption level (Bonniau and Bunsell 1981; Deo and Acharya 2010). Fig. 2 shows the water absorption behaviour of composites. All the composite samples achieved a steady state after 72 h of immersion. Other researchers have reported

similar trends for natural fiber-reinforced composites (Negi et al. 2022; Prasad et al. 2020). The steady states for glass fiber reinforced composites (GG and GGG) were achieved much earlier (about 24 h).

3.2. Mechanical characteristics of composites

The fiber reinforcement, fiber loading and stacking affect the composite's hardness. The hardness was measured on both sides of the composites at random places and the average hardness values are reported. Fig. 3 shows the hardness behaviour of the fabricated composites. The maximum hardness was found for the GGG composite, followed by the GUG and GG composite. This was due to the higher wt. % of glass fiber, which is harder than the polymer matrix. The hardness of the composite UG improved by 34 % when 4 wt. % of ukam fiber was replaced by the 4 wt.% of the glass fiber. The average hardness on ukam fiber and glass fiber sides of composite UG was 34 and 37 HV. The hardness increased by 48 % (GG) when the glass fiber was used in place of ukam fiber (UU). This was due to the higher hardness of the glass fibers compared to ukam fibers. There was an increment in the hardness of the composite when the fiber loading wt.% increased (UU and UUU, 8 to 12 wt.%).

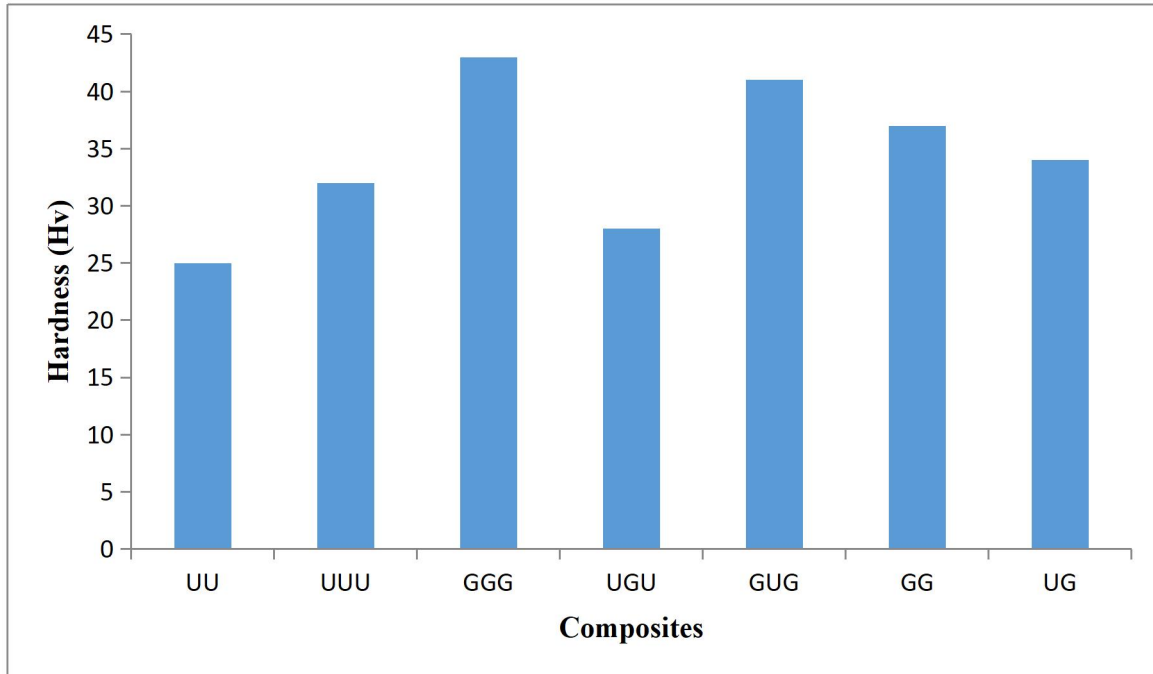


Fig. 3: Hardness behaviour of the composite samples.

The increase in hardness was also recorded when the glass fibers wt. % increased from 8 to 12 wt. % (GG and GGG). From the above results, it can be concluded that the hardness improved as the fiber content increased. Moreover, the addition of glass fibers improved the hardness compared to the addition of ukam fibers, due to hard glass fiber. Similar trends have been reported in other studies (Rudresh and Ravikumar 2017).

Fig.4 shows the tensile behaviour of the fabricated composites. As the fiber content of glass fiber increased, the tensile strength of the composites increased. However, excessive glass fiber content led to voids forming in fabricated composite samples. Consequently, the GGG with 12 wt. % fiber loading slightly decreased tensile strength than GUG. The results showed a better tensile strength of composites with only glass fiber as reinforcement than with only ukam fiber. This was attributed to the better tensile properties of the glass fibers (Giridharan 2019). With higher wt. % of fiber content, tensile strength increased. This can be attributed to the availability of more fibers to resist the same amount of deformation. From these results, it can be concluded that the hybridization

showed better tensile strength compared to natural fiber and synthetic fiber alone. The fabricated composites showed better tensile strength compared to other studies. The maximum tensile strength was 116 MPa for GUG composite and 71 MPa for UUU. It was concluded that the hybridization of glass fiber enhanced more tensile strength than ukam fiber hybridization. Compared to existing literature, the composites without hybridization also showed good tensile behaviour (Giridharan 2019).

The material's impact strength shows its toughness or the ability to resist high-speed stresses. Fig. 5 shows the impact behaviour of the fabricated composites. The impact strength of the composites increased with the fiber content irrespective of the fibers types (glass and ukam fibers). This was because more fibers resist high-speed loading (Prasad et al. 2020). The maximum impact energy was 4.05 Jm^{-1} for the GGG composites, while the minimum was recorded for the UU composite (2.05 Jm^{-1}). At the same time, comparing the same amount of fiber by wt. % of glass and ukam fiber, as in the case of GGG and UUU and UU and GG, the glass fiber composites showed better impact strength.

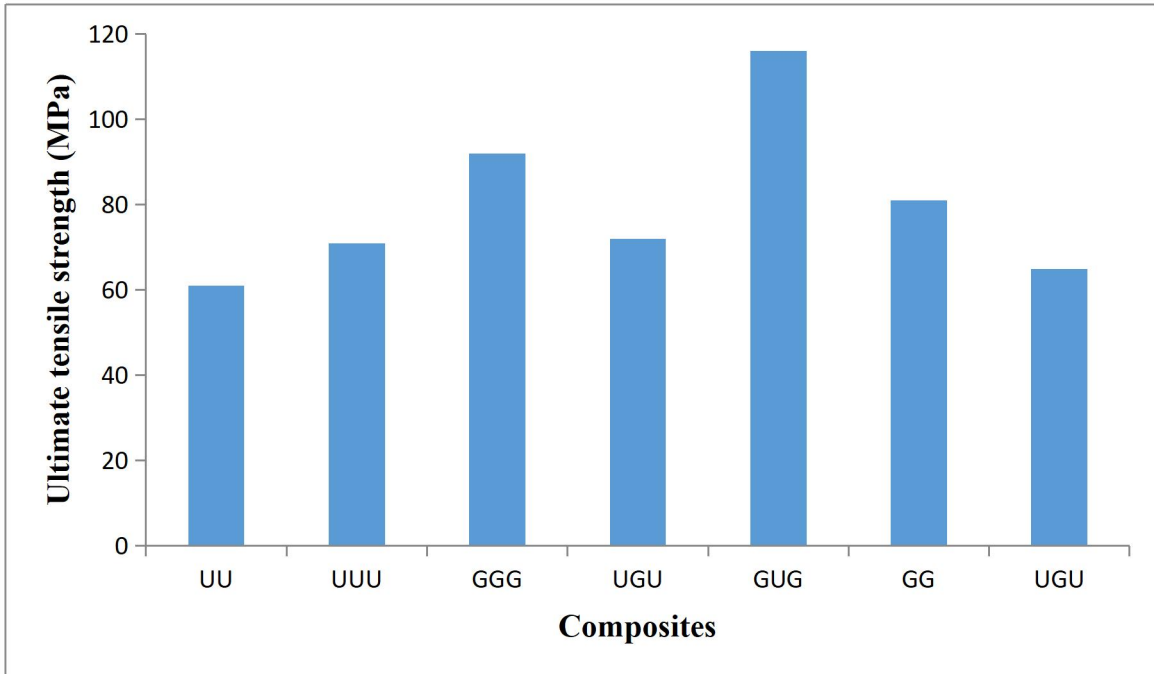


Fig. 4: Tensile behaviour of the composite samples.

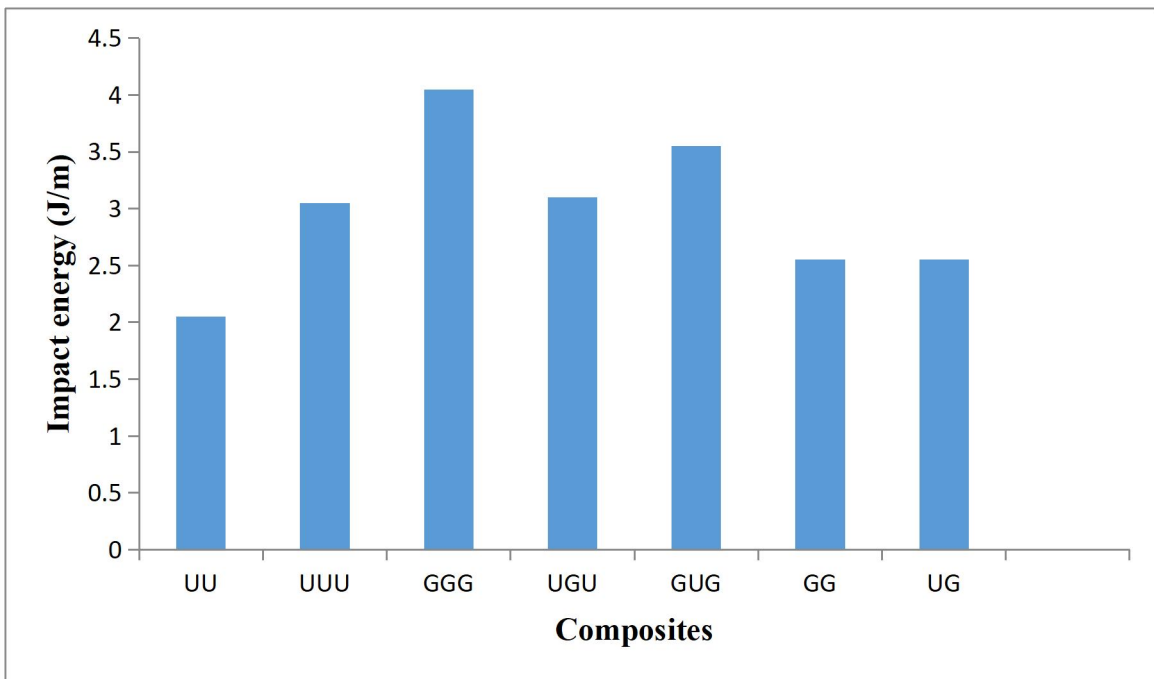


Fig. 5: Impact behaviour of the composite samples.

This can be attributed to the better bonding characteristics of glass fibers than ukam fibers. Consequently, better energy dissipation was obtained with the glass fiber composites. The same reason was responsible for the better impact behaviour of the GUG composite compared to the UGU composite. It can be concluded from the above results that as the fibers

content increased, the impact loading resistance of the composite increased. The glass fiber reinforced composites showed better impact resistance than ukam fiber reinforced composites.

3.3. Tribological behaviour of the composites

The maximum wear was for the glass fiber reinforcement compared to ukam fiber reinforcement,

as shown in Fig. 6. The composite's epoxy and fibers eroded during the test, and the average wear of two sides of the composites was reported. The maximum wear (85 μm) was for GGG, followed by the GG (75 μm) and GUG (67 μm). This can be attributed to the voids in the GGG composites. It can be concluded from these results that the limitation of glass fiber of poor abrasive resistance can be overcome by using hybrid ukam and glass fiber reinforced composites, as in the case of UGU (55 μm) composite, which showed lower wear. However, the minimum wear was recorded for UUU (39 μm), but the hardness was less.

3.4. Fractography of the hybrid composites

The SEM micrographs of the fractured hybrid composites are shown in Fig. 7, depicting the damage caused to the fibers due to fracture. The extensive damage (fiber pull out and fiber breaking) was caused to the ukam fiber. The damages led to fiber breaking which was visible from the SEM images. De-bonding of fiber and matrix was also visible. The mechanism for crack propagation, and consequently the composite mechanical strength, depends on the fiber/matrix adhesion (Djafar, Renreng, and Jannah 2020). The fibers were embedded in the matrix. However, some cavities formed in the composite.

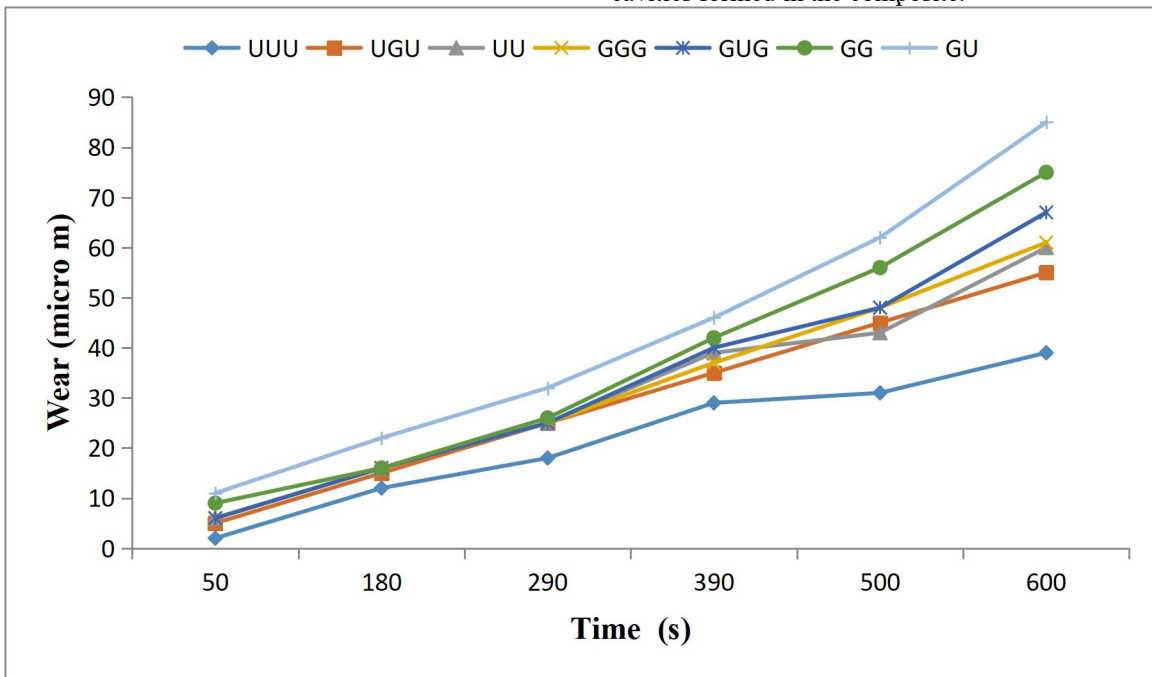


Fig. 6: Wear characteristics of the composite samples.

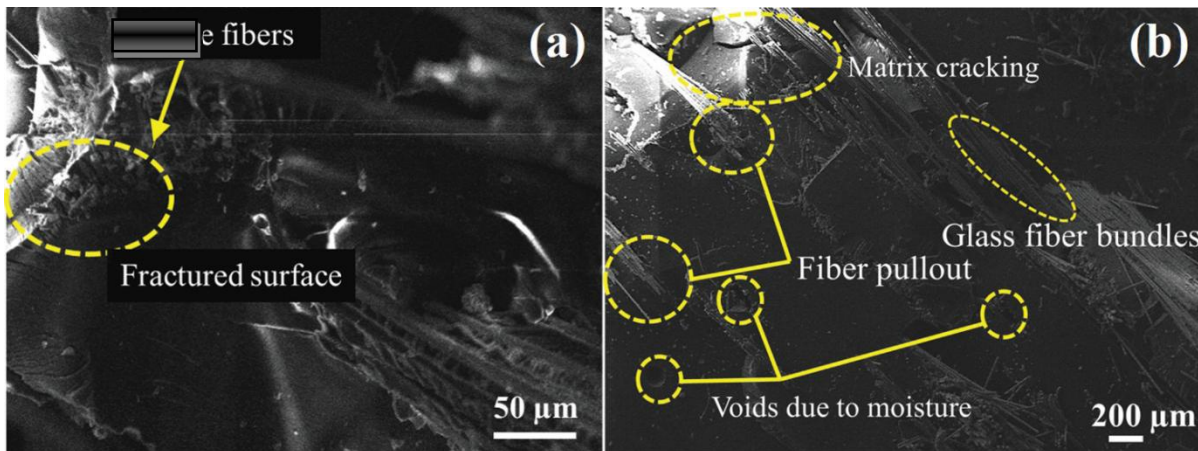


Fig. 7: SEM micrographs of fractured hybrid composites.

IV. CONCLUSIONS

Using different amounts of epoxy resin and hardener, efforts were made to improve the inter-laminar adhesion of glass/ukam hybrid composites. Glass fiber being synthetic fiber showed a lower water absorption percentage than ukam fiber reinforced composites. The UUU composites showed 1.16% water absorption compared to only 0.24% for the GGG composite. The tensile strength of composites increased with glass fiber content and fiber loading. The tensile strength of the GGG composite (pure glass fiber reinforced) was 29.58 % more than the UUU composite (pure ukam fiber reinforced). This was due to the high strength of the glass fiber than ukam fibers. Ukam fiber is a natural fiber, but with composites of ukam fiber, the impact strength is comparable to glass fiber's composites. Sample with pure ukam UUU showed impact energy of 3.05 Jm^{-1} , whereas the sample with pure glass GGG had impact energy of 4.05 Jm^{-1} . The results of composites prepared with one type of fiber were lower than the hybrid composites. Glass fibers reinforced composites showed excellent mechanical properties. However, glass fiber's abrasive resistance is lower. The addition of natural fiber in glass fiber composite increased its wear resistance; simultaneously, there were lesser decrements in other properties of glass fiber composites. UUU composite showed higher wear resistance than GGG composite leading to such composites as bearing components. Glass fiber-reinforced composites absorbed very little moisture content, while the ukam fibers composites showed higher moisture content. At the same time, the hybrid composites showed better moisture characteristics. Therefore, a mixture of natural and glass fiber can be used to reduce glass fibers content leading to greener composites. The good bonding of ukam and glass fibers was visualized in SEM analysis of glass-ukam samples. From this study, it can be concluded that it is possible to assume that epoxy polymers could be substituted with ukam/epoxy composites for improved environmental and mechanical efficiency. Moreover, the findings show that the physical and mechanical characteristics of natural fibers composites are significantly affected by natural fiber hybridization and varying the fiber stacking sequence. The fabricated composites are suitable for different structural and non-structural applications.

4.1. Highlights

- Hybrid composites using different wt. % of the glass and ukam fibers in different stacking sequences were fabricated.

The stacking sequence played a crucial role in the physical and mechanical characteristics of the composites.

- For composites with ukam fiber layer sandwiched between glass fiber layers, the composite showed optimal characteristics (tensile strength: 116 MPa, hardness: 41 Hv, impact energy: 3.55 Jm^{-1} , void content: 1.2 % and wear: $67 \mu\text{m}$).
- For composites with glass fiber layer sandwiched between ukam fiber layers, the composite showed lower physical and mechanical characteristics (tensile strength: 72 MPa, hardness: 28 Hv, impact energy: 3.1 Jm^{-1}).

Disclosure statement

The authors declare that there is no conflicting interest in the publication of this paper.

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