# Effect of Woven E-glass and Bamboo Stacking Sequences on the Properties of Laminated Composites Using Polyester Matrix Filled with Eggshell Microparticles

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# Effect of Woven E-glass and Bamboo Stacking Sequences on the Properties of Laminated Composites Using Polyester Matrix Filled with Eggshell Microparticles

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**Abstract**: Bamboo fiber-reinforced composites have been studied for engineering and biomedical materials due to their higher mechanical properties and lower density than other natural fibersThe purpose of this study is to determine how different stacking sequences of woven E-glass fibers (G) and woven bamboo (treated bamboo (B) and untreated bamboo (Ub)) affect the properties (bending, impact, and water absorption) of B, Ub, and G-strengthened polyester filled with eggshell microparticles (EMPs). The bamboo slats were immersed in water for 120 hours and alkalized with 6% NaOH for 36 hours. The composites were fabricated by hand lay-up using various stacking sequences of GUbG, GBG, GBBG, and GBGBG. The composite with a stacking sequence of GBGBG had the best flexural strength and impact toughness, at 177.19 MPa and 88.13 kJ/m$^2$, respectively. It also had the lowest amount of water absorption, at 0.64%. This composite is potentially being developed for biomedical materials.

Keywords: woven E-glass; woven bamboo slats; eggshell; laminated composite; mechanical and physical properties

## 1. Introduction

Polymer composites have become a choice in various applications, including biomedical applications, due to their superior properties, such as biocompatibility, lightweight, and corrosion resistance. Polymer composites reinforced with different components of natural and synthetic fibers have been designed as biomedical applications. To study the orthopedic limb, researchers have used kenaf fiber/epoxy composites [1], laminated carbon and/or glass fiber-reinforced acrylic resin [2], and carbon fiber-reinforced polymer (CFRP) composites [3]. Surgeons utilize natural fiber-reinforced polymer composites derived from bamboo, jute, or flax for surgical meshes and wound dressings because of their biocompatibility and exceptional tensile strength [4].

The bamboo plant is abundantly available, especially in tropical regions, including Indonesia. There are more than 150 types of bamboo in Indonesia. However, the availability of bamboo is not commensurate with its utilization, so there is still a high chance and challenge to develop bamboo into advanced and functional materials. This study used a type of bamboo: i.e., apus bamboo (*Gigantochloa apus)*, which has high fiber tensile strength and modulus of elasticity (MOE) of 178.8 MPa and 7.52 GPa, respectively [5]. The treated fiber showed a higher tensile strength of 335.8 MPa, a MOE of 15.8 GPa, and a flexural strength of 115.9 MPa [6]. Besides, the low density of bamboo fiber (0.6 g/cm$^3$) [5] compared to other fibers such as jute fiber (1.3–1.46 g/cm$^3$), flax fiber (1.5 g/cm$^3$), kenaf fiber (1.3–1.5 g/cm$^3$), and abaca fiber (0.83–1.3 g/cm$^3$) [7] makes it a good choice for reinforcement material in polymer composites.

However, it is well known that natural fibers have lower mechanical properties than synthetic fibers. They are hydrophilic, leading to high moisture content and reduced durability of the composites. This can be a basic issue in fabrication and applications. Therefore, a combination of natural and synthetic fibers would be beneficial.

To improve the mechanical properties of composites reinforced with natural fiber, Islam et. al [8] reviewed the physical and chemical treatments on the jute fiber surface. The chemical treatments used are alkali, silane, and alkali-silane treatments. The mechanical properties of chemically treated jute-reinforced epoxy and polyester did not show substantially high values compared to composites reinforced with physically treated jute fibers. Jute fibers coated with graphene oxide (GO), reduced graphene oxide (rGO), and graphene flake (GF) can significantly improve their mechanical properties. GO and rGO are water-soluble, but rGO has better electrical conductivity. A treatment combination of hot water, alkali, and rGO on jute fiber can achieve a tensile strength of up to ~ 800 MPa.

The improvement of the composite's mechanical strength is due to good interfacial bonding between the modified fiber surface and the matrix polymer. The damping factor, which is the ratio of the loss modulus to the storage modulus, is an important part of dynamic mechanical analysis (DMA). In this case, the damping factor has a correlation effect with interfacial bonding between the fiber and the matrix. Better interfacial bonding results in a higher damping factor value. Researchers have reviewed DMA on natural fiber-reinforced hybrid polymer composites [9]. An example of a DMA on sugar palm yarn/glass fiber/polyester indicated better compatibility between natural fiber and glass fiber and interfacial adhesion with the matrix. It led to a higher value of the damping factor.

Additionally, researchers have previously investigated bamboo-based composites and woven bamboo-glass hybrid composites [7–11]. As mentioned above, moisture in natural fibers, including bamboo fiber, becomes a significant factor that must be considered in the use of bamboo-based composites. Immersing bamboo fibers in water at room temperature for 60 days and at 80°C for two days revealed a decrease in the composite's tensile strength of ~6% at room temperature and ~13% at 80°C [10]. The reduction in tensile strength was possibly due to debonding or delamination between the fiber and matrix during water immersion [14]. However, there was a contradictory opinion on improving the mechanical properties of water-immersed composites [15]. Researchers conducted a study on hybrid composites, fabricating them in three layers where woven bamboo fibers were positioned in the middle to reinforce polyester. Bamboo content was varied by 3, 6, 9, 12, and 15 wt.%. The tensile strength, Young modulus, flexural strength and flexural modulus reached by 9 wt.% bamboo content were 108 MPa, 757 MPa, 255 MPa, and 760.2 MPa, respectively [11]. Other research groups studied bamboo fiber/glass fiber mesh/epoxy resin by varying the three types of fiber stacking sequences: GBBGBBG, BGBGBGB, and BBGGGBB, in which B and G were bamboo fiber and glass fiber mesh, respectively. The composite with the BGBGBGB sequence achieved the highest flexural strength of 92.22 MPa. While the composite with the GBBGBBG sequence reached the highest impact energy of 150 J and the lowest water absorption of 1.3%[12]. Manik et al. [13] investigated laminated composites made of epoxy resin reinforced with apus bamboo. The laminations were varied by 3, 5, and 7 layers with five types of laminated board joints called butt joints, finger joints, scarf joints, tongue and groove joints, and desk joints. The composite was fabricated in seven layers, with desk joints exhibiting the highest tensile strength of 118.62 MPa and bending strength of 475.89 MPa. The lamination technique had a crucial effect on improving the composite's bending strength. In addition, Guadua angustifolia, a type of bamboo was investigated as particles of ~ 150 μm to reinforce low-density polyethylene (LDPE). Bamboo particles were varied in concentrations of 1, 2, 3, 4, and 5% and mixed with 1% of maleic anhydride (MA) as a coupling agent [16]. Adding bamboo particles decreases the composite's mechanical properties (average modulus and compressive strength), and the highest properties were achieved by adding 1% bamboo particles.

Investigations of laminated woven bamboo slats and E-glass reinforced polyester matrix filled with eggshell microparticles (EMPs) have not been reported. Our previous work [17] compared the properties of abaca fiber/EMPs/polyester hybrid composites to those of hybrid composites with different uses of polymer matrix: i.e., polymethyl methacrylate (PMMA) and epoxy. Incorporating EMPs in polyester resulted in substantially reducing the water absorption compared to that in PMMA and epoxy. In addition, a study on EMPs/polyester composites [18] indicated that the tensile and flexural strengths and hardness of the composites increase with EMPs concentrations. Scanning electron microscopy (SEM) confirmed the enhanced interfacial bonding between EMP and polyester.

In this work, a combination of bamboo, glass fiber, and EMPs is beneficial in improving the composite's properties and can be developed as an advanced and functional material. Bamboo is abundantly available and has a low density. Glass fiber has high mechanical strength and is hydrophobic. EMP, typically considered waste, can serve as a valuable filler. This study aims to fabricate hybrid laminated composites of B/G/EMPs and polyester by varying the stacking sequences of B and G. The concentration of EMPs used was constant at 5 vol% [17]. The properties (bending, impact toughness, and water absorption) of the present composites were evaluated and compared with those of biomedical materials to understand their potential applications.

## 2. Materials and methods

### 2.1 Materials

Reinforcement materials including Indonesian apus bamboo slats, E-glass woven roving (WR 200) Matt (Fig. 1) and commercial eggshell powder were purchased locally. A local shop supplied unsaturated polyester (157 BTQN) for the polymer matrix material. NaOH and $CH_3COOH$ (glacial) 100% supplied by Merck were used to modify the bamboo slat surfaces.

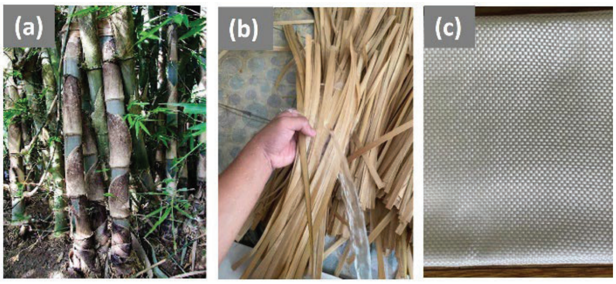



**Fig. 1:** (a) Profile of apus bamboo plant (*Gigantochloa apus*), (b) apus bamboo slats, and (c) woven E-glass.

### 2.2 Fiber treatments and laminated composite fabrication

Apus bamboo slats were first washed in flowing water and then immersed in water for five days to avoid dirt from the slat surfaces and dried at 60°C for 30 minutes, which reduced the water content by ~ 10%. Next, the bamboo slats were chemically treated by immersing them in a NaOH solution at 6% concentration for 36 hours and at room temperature to avoid non-cellulosic compounds and improve interfacial adhesion between the fillers and the matrix [17]. Subsequently, the alkali solution was neutralized with $CH_3COOH$, and the bamboo slats were washed with flushing water and then immersed in distilled water for 24 hours. Thereafter, bamboo slats were dried at 105°C for 12 hours (Fig. 2 a). and then woven and cut according to the molding size (170 mm x 90 mm) (Fig. 2 b). This research utilized untreated E-glass.

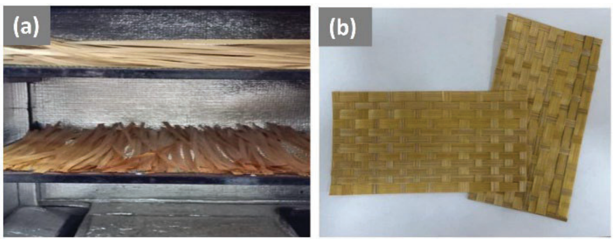



**Fig. 2:** (a) Apus bamboo slats during drying in a drier cupboard and (b) the woven bamboo slats.

The laminated composites were designated with four different stacking sequences: GUbG, GBG, GBBG, and GBGBG (Fig. 3). G is woven E-glass. B and Ub represent treated and untreated woven apus bamboo, respectively. In this case, the laminated composite using Ub made with a stacking sequence GUbG was used as a reference.

In the composite fabrication, polyester (P) was mixed with 5 vol% of eggshell microparticles (EMPs), which were sieved to 200 mesh (~ 74 μm) [17]. The hand lay-up technique was used to fabricate the laminated composites in the hot press molding at 100°C for 30 minutes. Table 1 summarizes the laminated composite specimens and their compositions, based on the densities of G = 2.5 g/cm$^3$, B = 0.6 g/cm$^3$, P = 1.2 g/cm$^3$, and EMPs = 1.13 g/cm$^3$.

### 2.3 Mechanical and physical tests, and characterization

All laminated composites were subjected to three-point bending, a Charpy impact test, and water absorption tests according to ASTM D790-10, ASTM D6110-10, and ASTM D-570-10, respectively. Each case required at least five specimens. After bending and impact tests, the fracture surface of the composite specimens was examined using an optical microscope (SZ61 OLYMPUS). In addition, EMPs were examined by scanning electron microscopy-electron dispersive x-ray spectroscopy (SEM-EDS, JEM-6510LA).

Table 1. The laminated composite specimens and their component compositions

| Stacking sequence | Composition (vol%) | | | |
|---|---|---|---|---|
| | G | B/Ub | P | EMPs |
| GUbG | 6 | 14 | 75 | 5 |
| GBG | 6 | 14 | 75 | 5 |
| GBBG | 6 | 28 | 61 | 5 |
| GBGBG | 9 | 28 | 58 | 5 |

Woven E-glass: G, woven untreated bamboo: Ub, woven treated bamboo: B, polyester: P, and eggshell microparticles: EMPs

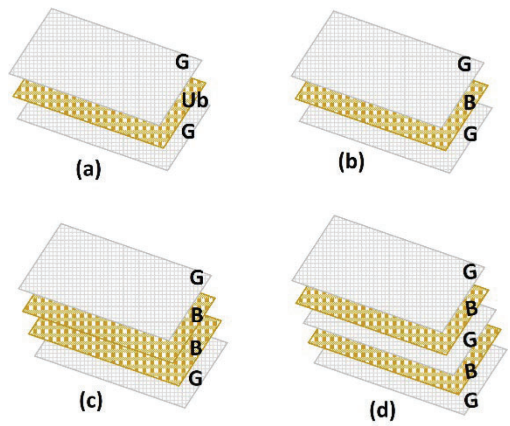



**Fig. 3**: Stacking sequences used in laminated composite specimens. (a) GUbG, (b) GBG, (c) GBBG, and (d) GBGBG.

## 3. Results and Discussion

### 3.1 Flexural properties

The result confirmed that the flexural strength and modulus of composites using alkali-treated bamboo were significantly higher than those using untreated ones (Fig. 4). With the different stacking sequences, this figure shows a significant increase in the composite's flexural strength and modulus, especially with the stacking sequences GBBG and GBGBG. The higher amount of woven glass in GBGBG contributes to the highest flexural strength and modulus of the composite. The interfacial bonding between woven glass and woven bamboo seemed to be stronger than that between woven bamboo and woven bamboo. This meant that the composite was stronger when stacked in the GBG sequence than when stacked in the GBBG sequence. Furthermore, the previous study [19] demonstrated that while the number of laminates positively correlates with the mechanical strength of the composites, the stacking sequences also play a crucial role. They investigated the laminated composites of woven jute fiber, perlon, glass, and carbon fabric-reinforced polyester with various stacking sequences. The composite stacked in the sequences of 2 perlons + 3 jute + 4 carbon + 2 perlons (11 layers) had higher flexural strength than that in the lower layer number.

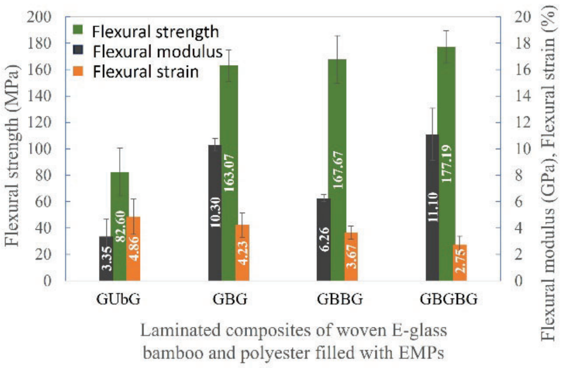



**Fig.4**: Flexural properties of woven E-glass bamboo polyester filled with EMPs.

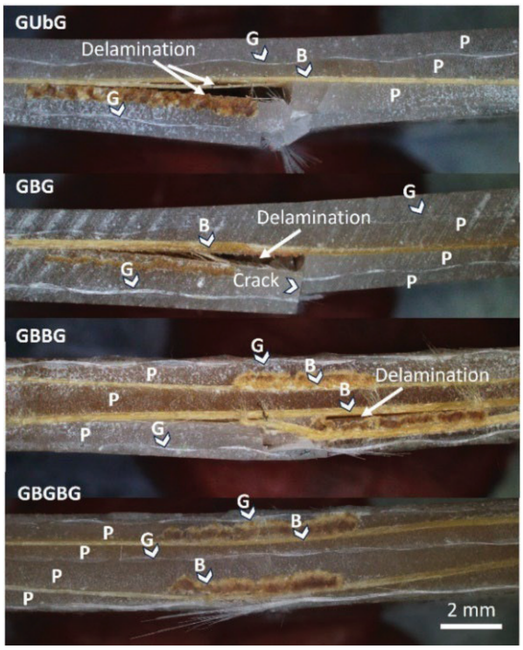



**Fig. 5:** Optical micrographs of all laminated composite specimens after the bending test that were obtained from side views.

In this study, no composite specimen broke after the bending test. Figure 5 depicts an optical microscope examination of the bending test fracture areas for all composites observed from side views. The stacking sequences in all composites demonstrated success in composite fabrication. Delamination occurred after the bending test, particularly in the stacking sequences of GUbG, GBG, and GBBG, but not in that of GBGBG. However, only the GUbG and GBG composites, which have lower layer numbers than the other two composites, exhibited cracks. Close observations of the delamination areas all happened on the bamboo side. This suggests that the interface bonding between the bamboo surface and the polyester matrix was weaker than that of the glass surface and polyester interface. The GBGBG composite exhibited the strongest laminated bonding, resulting in the highest strength.

The bonding between the bamboo surfaces happened in the glue between the hydroxyl ($OH^-$) and functional groups while the bamboo was being pressed. The polymeric matrix, typically composed of monomers, facilitates matrix wetting and penetration when applied to bamboo components. When the polymeric matrix produces a stiff, strongly cross-linked polymer, the monomers eventually grow into huge molecules (polymers) [20].

The flexural strength obtained in this research (177.19 MPa) was significantly lower than those in the previous studies, i.e., 255 MPa with a similar matrix material of polyester [11] and 475.89 MPa with epoxy resin [13], but higher than the laminated composite's flexural strength, i.e., 92.22 MPa using the epoxy resin matrix [12]. Composition, lamination type, and stacking sequence influence these differences. Additionally, the use of woven and non-woven fibers as reinforcement materials affects the mechanical properties of the composite. Although the hybrid composites of sisal/carbon/polymethylmethacrylate (PMMA) used carbon fiber, which has a higher mechanical strength than glass fiber, they showed lower flexural strength (108 ± 6.61) MPa [21] than woven-bamboo/woven-glass/polyester's flexural strength of 160–180 MPa (this study). The non-woven carbon fiber in the sisal/carbon/PMMA composites is the root cause.

### 3.2 Impact toughness

Figure 6 shows the impact toughness of all the laminated composite samples. The composite with a stacking sequence of GBGBG has the highest value of 88.13 kJ/m$^2$. This is in line with the flexural strength (Fig. 4). The changes seen in the microstructure of the composite specimens after the impact test may be linked to the changes in impact toughness caused by the different stacking sequences. All composite specimens were unbroken after the impact test, showing laminate failures obtained from the opposite side that did not hit the hammer impact. (Fig. 7).

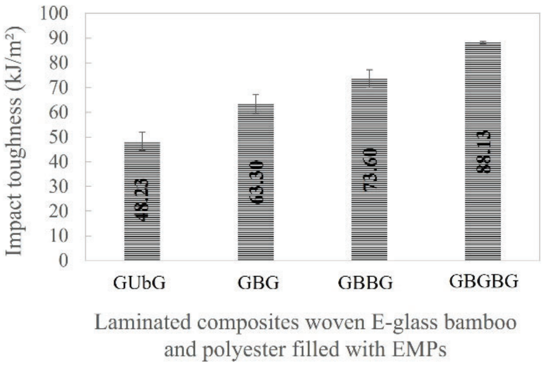



**Fig. 6:** Impact toughness of woven E-glass apus-bamboo polyester filled with 5 vol% EMPs.

The composite with a stacking sequence GBG shows that the bamboo surface bonding with polyester was stronger than that of a stacking sequence GUbG (Fig. 7). This confirmed the significant role of alkalization in improving impact toughness. The composite with the stacking sequence GBBG demonstrates a weak layer sequence between B and B, leading to lower impact toughness compared to the stacking sequence GBG. In this case, the stacking sequence GBGBG has the highest layer number, making its impact toughness the highest, although the B layer broke after the impact test. The impact toughness of 88.13 kJ/m$^2$ was higher than the highest value of 73 kJ/m2. This made the laminated composite of polyester reinforced with 2 layers of perlon, 3 layers of jute, 4 layers of glass, and 2 layers of perlon fibers [19].

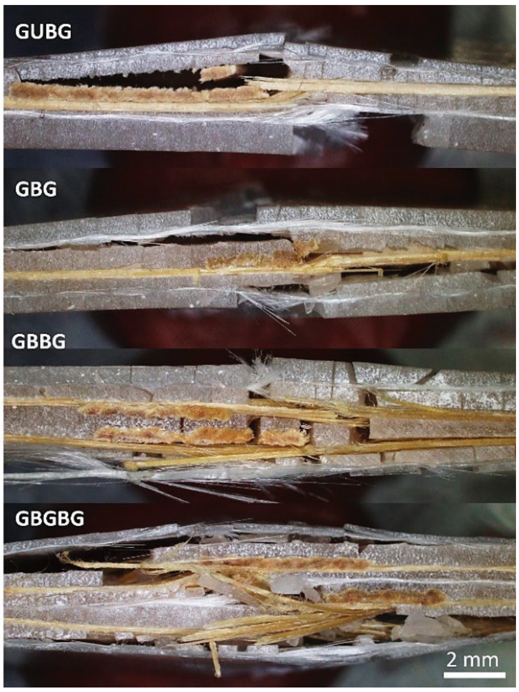
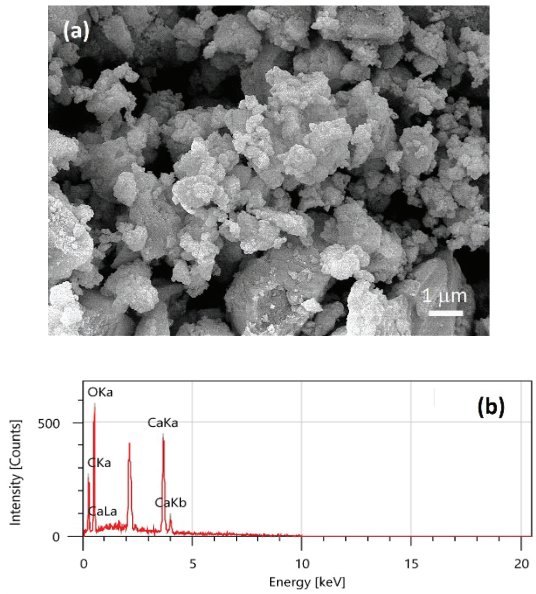



**Fig. 7:** Optical micrographs of all laminated composite specimens after the Charpy impact test were obtained from side views. The white and brown layers are glass and bamboo, respectively.

### 3.3 Water absorption

Water absorption in the composite materials becomes an important factor that must be considered, particularly the natural fiber-reinforced composites. In this study, woven glass fiber reduce the water absorption of the composites due to its hydrophobicity. Furthermore, as previously mentioned, the addition of EMPs to the present composites was expected to suppress water absorption. Fig. 8 depicts the morphology and chemical elements of the commercial EMPs used in this research. EMPs show agglomerated particles with uneven particle sizes (less than 5 µm) (Fig. 8a). According to EDS analysis, there are almost no impurities contained in EMPs, which are different from the eggshell by self-preparation, as previously reported: i.e., Si, Mg, Cu, Zn, and S [17]. Ca content in EMPs was higher (~ 50 mass%) than the previous eggshells: i.e., 28.49 mass% for chicken eggshells and 33.56 mass% for duck eggshells.




| Element | Mass% | Atom% |
|---|---|---|
| C | 11.16±0.16 | 20.20±0.28 |
| O | 38.68±0.65 | 52.58±0.88 |
| Ca | 50.17±0.94 | 27.22±0.51 |
| Total | 100.00 | 100.00 |

**Fig. 8:** SEM image of eggshell microparticles (a) and, EDS spectrum and chemical composition of EMPs (b).

Figure 9 shows water absorption curves for all composites, in which each laminated composite has a different weight gain. The composite with the stacking sequence GUbG reveals the highest weight gain (0.82%), higher than that of GBG (0.71%). The alkali treatment on the woven bamboo makes the interfacial adhesion with the matrix polymer stronger, which stops water molecules from entering. Previous studies have reported the influence of alkali-treated fiber on reducing water absorption[22]. However, the composite with the stacking sequence GBBG has a higher weight gain (0.77%) compared to GBG (0.71) and GBGBG (0.64%). It is known that G is hydrophobic, and B is hydrophilic. Thus, the G/B ratio in GBBG (1/1) is lower than that in GBG (2/1) and GBGBG (3/2), leading to higher water absorption in GBG than in others. Usually, an increase in natural fiber content increases water absorption in the composite [23], which is consistent with the present results (Fig. 9).

The lowest water absorption achieved in this study was lower than in a previous study on laminated composites of glass/bamboo/epoxy, with the lowest water absorption of 1.3% reached with the stacking sequence GBBGBBG [12]. Besides, it was also lower than that of the laminated composite of 4 perlon/3 sisal/2 carbon/PMMA (1.77%) [24]. This indicates that this is not only due to the excellent bonding between the laminates but also to the presence of EMPs. Therefore, using synthetic fiber, natural fiber surface treatment, and EMPs together have become important ways to reduce the moisture content in polymer composites.

On the other hand, aside from reducing water uptake, eggshells seem to play a role in functions related to absorption. The raw eggshells and hybridized eggshell-pandan leaf were used as an absorbent of metallic ions as a pollutant that was highly dissolved in water. The hybridized eggshell-pandan leaf results in a higher removal percentage of Cr (IV) ions in wastewater than the raw eggshell [25].

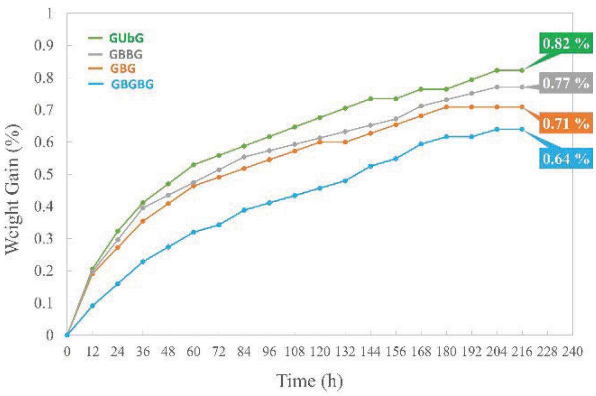



**Fig. 9:** Water absorption

### 3.4 Potential applications

All composites produced in this study were compared with some previously studied biomedical composite materials used in prosthetic socket applications to understand the potential application of the present composites in terms of mechanical strength and water absorption. The composite with a stacking sequence of GBGBG achieved the highest mechanical properties (flexural and impact toughness) and the lowest water absorption. Table 1 lists the comparison of their properties.

The results in Table 2 indicate that the flexural strengths, as well as the impact toughness, of the present composites, are higher than those of the previous composites, except for the composite using untreated bamboo (GUbG) due to weaker interfacial bonding between the fillers and the matrix than the treated ones. However, composites with the stacking sequences GUbG and GBG have impact toughness lower than PLF/P but higher than PLF/E. In addition, based on the water absorption results, the lowest water absorption reached by the composites with the stacking sequences of GBGBG of 0.64% is lower than that of Abdulrahman's studies [26]. Based on the results in Table 2, the composite with the stacking sequences of GBGBG was considered to have the potential for development as a prosthetic socket material.

Table 2. Comparison of the mechanical and physical properties of the present composites and the previous composites used in prosthetic sockets.

| Composite | Flexural strength (MPa) | Impact toughness ($kJ/m^2$) | Water absorption (%) | Ref. |
|---|---|---|---|---|
| GUbG | 82.60 | 48.23 | 0.82 | Present study |
| GBG | 163.07 | 63.30 | 0.77 | Present study |
| GBBG | 167.67 | 73.60 | 0.71 | Present study |
| GBGBG | 177.19 | 88.13 | 0.64 | Present study |
| 2G/4PF/3JF/P | 137 | 62 | - | [19] |
| 4PF/3SF/2CF/ PMMA | - | - | 1.77 | [24] |
| 25F/15S/60E | 145 | - | 3.66 | [27] |
| PLF/P | 70 | 65 | - | [28] |
| PLF/E | 85 | 45 | - | |
| 2Pr/4C/3J/2Pr/P | - | - | 0.688 | [26] |

PF: perlon fiber, JF: jute fiber, P: polyester, SF: sisal fiber, CF: carbon fiber, E: epoxy, PLF: pineapple leaf fiber

Related to prosthetics, the composite material has recently been studied for kneecap prosthetics[29]. In the past, people typically used alloys such as stainless steel, Co-Cr alloys, and Ti6A14V. During contact with human tissue for some time, metallic ions will diffuse in the body, even though metals have high strength and fatigue properties. However, composite materials have high properties such as chemical resistance, biocompatibility, and tribology. Therefore, composite materials are developed for a wide range of applications.

However, this study has some limitations. In the composite fabrication, EMPs and polyester did not mix in a vacuum. Without a vacuum, it can cause air trapping during mixing and possibly form voids. This void can reduce the composite's properties.

The mechanical testing of composite specimens in this research is limited. Thus, the data obtained to support the composite application of prosthetic socket materials would be less accurate. It will be better if compression testing is done rather than tensile testing, as carried out in previous studies on prosthetic socket composites [27, 28], 30]. Therefore, further research is required to improve data acquisition on hybrid composites with bamboo and glass fiber as reinforcement materials.

## 4. Conclusions

This study successfully fabricated, mechanically, and physically characterized laminated composites of woven glass, woven bamboo, and polyester filled with EMPs in various stacking sequences. The flexural strength and impact toughness of the composites have similar trends in reaching the properties at the following stacking sequences: i.e., GBGBG > GBG > GBBG. The composite with the GBGBG stacking sequence had the highest flexural strength of 177.19 MPa, an impact toughness of 88.13 kJ/m$^2$, and the lowest water absorption of 0.64%. These results provide useful contributions to the composites studied for prosthetic socket applications. Further research is required to confirm whether this composite has any potential use in other biomedical materials.

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