Original article



Fabrication of bio-fiber based Eichhornia crassipes/Al₂O₃ particles hybrid biocomposites and investigation of important properties

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Nasmi Herlina Sari¹, Suteja¹, Sujita¹, Rushdan Ahmad Ilyas², Eka Sari³, Mavinkere Rangappa Sanjay⁴ and Suchart Siengchin⁴

Abstract

The need for environmentally friendly composite materials that meet the necessary criteria is growing. Making novel environmentally acceptable composites with hybrid *Eichhornia crassipes* (EGs)/Al₂O₃ particles is an option. This paper aims to investigate the thermal, tensile strength, and flexural strength properties of a polyester composite filled with hybrid EG/Al₂O₃ particles. The tensile strength, flexural strength, and morphology of EGs/Al₂O₃ hybrid composite will be investigated in this work. The hot pressed for 60 minutes at 170°C procedure was used to shape the hybrid composite. EGs/Al₂O₃ particles content was altered from 0:25 to 5:20, 10:15 to 15:10, 20:5, and 25:0 (vol.%). The results reveal that when the EGs content of the composite is in the range of 10–15% (vol.%), the tensile and bending strength of the composite increases. The EGI (15% EGs:10% Al₂O₃) and EGH (10% EGs:10% Al₂O₃) composites had the highest tensile and bending strengths, with 38.697 and 77.786 MPa, respectively. The boost in strength is thought to be due to Al₂O₃ acting as a cursor in the composite, preventing fracture progression. The scanning electron microscope (SEM) fracture morphology reveals a strong and tight interfacial connection between the EGs, Al₂O₃, and polyester layers. In addition, when the EGs grew, the composites' thermal characteristics improved. According to the findings, the EGs/Al₂O₃ particle hybrid composite can be used in automotive applications.

Keywords

Hybrid composite, water hyacinth particles, Al₂O₃, tensile and bending properties, morphological properties

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Introduction

Today, almost every country in the world strictly adheres to the guidelines and standards established by environmental regulatory agencies. These environmental rules and regulations direct researchers to develop environmentally friendly materials to replace non-biodegradable materials.^{1,2} Because of their simple fabrication procedures, low cost, light weight, comparable strength, and durability, synthetic fiber-reinforced composites are one of the non-biodegradable materials used in a variety of fields such as automotive, construction, and packaging. According to recent environmental policies, alternative materials for synthetic fiber-reinforced composites are required. Plant fiber-reinforced composites are an environmentally friendly alternative to synthetic fiber-reinforced polymers, with comparable mechanical, thermal, and morphological properties. The ability of plant fibers to bond with polymer resins is an important factor in determining the mechanical performance of fiber-reinforced plastics.⁴ Untreated plant fibers have a smooth hydrophilic surface, which reduces their ability to bond with polymers. Plant fibers are typically chemically treated to improve their bonding ability. This type of chemical treatment also alters the fiber's chemical composition, thermal stability,

Corresponding authors:

Nasmi Herlina Sari, Mechanical Engineering Department, Faculty of Engineering, University of Mataram, Mataram, West Nusa Tenggara, Indonesia. Email: n.herlinasari@unram.ac.id

Mavinkere Rangappa Sanjay, Natural Composites Research Group Lab, Department of Materials and Production Engineering, The Sirindhorn International Thai-German Graduate School of Engineering (TGGS), King Mongkut's University of Technology North Bangkok (KMUTNB), Bangkok, Thailand. Email: mcemrs@gmail.com

¹Mechanical Engineering Department, Faculty of Engineering, University of Mataram, Mataram, West Nusa Tenggara, Indonesia

²School of Chemical and Energy Engineering, Faculty of Engineering, Universiti Teknologi Malaysia, UTM Johor Bahru, Johor, Malaysia ³Chemical Engineering, Faculty of Engineering, Universitas Sultan Ageng Tirtayasa, Banten, Indonesia

⁴Natural Composites Research Group Lab, Department of Materials and Production Engineering, The Sirindhorn International Thai-German Graduate School of Engineering (TGGS), King Mongkut's University of Technology North Bangkok (KMUTNB), Bangkok, Thailand

surface roughness, and tensile performance. For surface modification, researchers use aqueous sodium hydroxide (NaOH) solution, potassium permanganate in acetone, and silanes in ethyl alcohol. NaOH treatment is the most cost-effective and efficient of these.⁵

Eichhornia crassipes (EG) is a plant that grows in water and is often considered a weed. The EG plants grow very fast, spread rapidly throughout rivers, and float on the surface of the water, causing damage to waterways and water pollution.⁶ Like other fibers, water hyacinth has a high percentage of holocellulose so it becomes an advantage in its application as a reinforcing material for polymer materials.7 They also contain important compounds such as cellulose of 64.51%, pentose of 15.61%, lignin of 7.69%, silica of 5.56%, and ash of 12%.7 The potential of environmentally friendly EG fiber as a composite reinforcement is an opportunity to reduce the use of synthetic fibers such as glass fibers because they are not environmentally friendly, and harmful to health.8 Several researchers have investigated the physical and mechanical properties of EG fiber composites. Ramirez et al.⁹ have investigated the mechanical and thermal characteristics of an EG particle-filled polyester composite. They reported that polyester composites with EG concentrations in the range of 5-10 (weight%) gave the best results in terms of dynamic modulus of elasticity of 0.33-0.45 kg/cm². Static modulus elasticity and density of the composites were found in the range of 40,000–10,000 kg/cm² and 1.6–1.53 g/cm³ obtained from composites with a volume fraction of EG particles of 0-20 (wt.%). They also revealed that there was no evidence of a negative effect on the mechanical and thermal properties of the composites with the addition of EG to the polyester resin.¹⁰ Furthermore, water hyacinth fiber can provide competitive reinforcing qualities when compared to other natural fibers, such as hemp, abaca, and rice straw. Optimal mechanical properties of high-density polyethylene composites were obtained when the ratio of water hyacinth and HDP fiber content was 30%:70%. The density of the composite is known to tend to decrease when EG particles increase.¹¹ The maximum tensile strength of the composite containing 10% EG fiber (% volume) is known to be 27.27 MPa, and the maximum impact strength of 0.0161 J/mm³ was obtained from the composite with a fiber volume fraction of 40%.¹² From several perspectives, EG particle filler can reduce production costs and improve the mechanical properties of composites for application in various industries. Unfortunately, the mechanical properties of this water hyacinth fiber-reinforced composite are known to be low compared to synthetic fiberreinforced composites. This weakness can be solved by hybridizing EG with Al₂O₃ in the composite. This hybrid composite offers characteristics that cannot be obtained with a single filler. Al₂O₃ particles can increase the hardness and impact energy of epoxy composites compared to modifications using SiO₂ and TiO₂ particles.¹³ Heyi et al.¹⁴ have investigated the addition of Al2O3/SiC filler in polyester-bagasse fiber composites. They reported that the tensile and bending strength of the composite after the volume fraction of Al_2O_3 (wt.%) was increased. Then there was a decrease after adding more than 8% of SiC by weight and more than 10% of bagasse fiber by weight fraction. Tominaga et al.¹⁵ have combined Al_2O_3 and hexagonal boron nitride (h-BN) particles. They reported that the relative density of the composite film increased with increasing wt.% Al_2O_3 , which was greater than wt.% cellulose nanofiber (CNF) and nano-diamond particles (ND). The thermal conductivity of the composite film may vary depending on the shape of the ceramic filler. Previous studies have shown that hybrid Al_2O_3 with EG fiber needs to be developed and investigated to obtain the best properties and fulfill the desired application requirements such as in automotive field.

Therefore, this study aims to provide an understanding, and detailed information related to the thermal properties, tensile strength, and flexural properties of EG fibers (EGs)/Al₂O₃ particle composites. The ratio of EGs and Al₂O₃ particles has been varied to quantify the tensile strength and flexural strength properties of the composite.

Materials and methods

Materials

 Al_2O_3 particles were obtained from PT. Estrachemical Indonesia with a purity of 98% and a particle size of 325 mesh. EG plants were collected from the Batu Jai area, Central Lombok, Indonesia. Furthermore, the unsaturated polyester resin was used as a matrix, and methyl ethyl ketone peroxide (MEKPOSE) as a curing catalyst with a ratio of 1% of the resin volume fraction. Specifications of polyester resins are presented in Table 1.

Extraction of water hyacinth particles (EGs)

The process of extracting EGs is shown in Figure 1. The stems from the EG plant (Figure 1(a)) were cleaned of adhering impurities (Figure 1(b)) and cut to a length of 10 mm (Figure 1(c)). Next, they were dried in an oven at 105 °C for 1 h, then mashed using a ball mill to produce particles. The particles were then sieved using a 200-mesh sieve (Figure 1(d)), then oven-dried at 105 °C for 1 h, and finally, the EG particles (EGs) were ready to be used for the manufacture of composite samples. The density of EGs has known as 0.5 g/cm³. The chemical composition of the EGs is shown in Table 2.

Table 1. Specification of polyester resin	. 10,1	1
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Specification	Value		
Tensile strength	40 MPa		
Flexural strength	45 MPa		
Maximum elongation	1%		
Modulus of elasticity, (E)	3.3 GPa		
Density	$1.09 (g \text{ cm}^{-3})$		
Viscosity	6–8 P (at 25 °C)		



Figure 1. EGs extraction process: (a) EG plant, (b) EG rod, (c) EG pieces with a length of 10 mm, (d) EG rod refining process, and (e) particles of EG (EGs) were ready to be used as composite filler.

 Table 2. The chemical composition of the Eichhornia crassipes

 particles.¹⁸⁻²²

Chemical composition	Content (%)		
Cellulose	18–23.86		
Hemicellulose	50		
Lignin	3.8–5.8		
Ash	19–20.1		
Moisture	4.9		

Hybrid composite fabrication

Hybrid composites were fabricated with a predetermined ratio of EGs versus Al_2O_3 particle composition (Table 3). The polyester resin and catalyst were mixed and stirred at 600 rpm for 10 minutes, then poured into a steel plate mold filled with EGs and Al_2O_3 , then the mold was closed with an emphasis of 5 MPa at a temperature of 105 °C. The composite is removed from the mold for further characterization. Figure 2 shows the samples of tensile and flexural tests of composites, respectively. There are six different types of samples have been made with three repetitions for each different composite.

Characterization

Density. The density of composites can be obtained experimentally by the use of equation $1.^{23}$

$$\rho_{\rm c} = \frac{m}{\nu' - \nu} \tag{1}$$

Where, ρ is density (g/cm³); *m*, *v'*, and *v* are the mass of the composite sample (g), and the volume of water after and before the sample is immersed (cm³), respectively.

Table 3. The composition ratio of EGs, AI_2O_3 , polyester in composites.

No.	Codes	Volume fraction (vol.%)		
		Polyester	EGs	Al ₂ O ₃
I	EGA	75	0	25
2	EGB	75	5	20
3	EGH	75	10	15
4	EGI	75	15	10
5	EGX	75	20	5
6	EGY	75	25	0

Tensile strength test. Tensile strength testing on composites is intended to measure the force required to stretch the composites, and the amount of elongation until fracture of the composite. Tensile testing was carried out using a universal tensile machine (UTM) RTG-1310 at a room temperature of 28 °C with a relative humidity of 40%. The UTM machine is operated with a loading speed of 5 mm/min and a maximum load of 5 kN. The composite was formed according to the ASTM D3039 standard with dimensions of 250 mm × 25.4 mm × 6 mm.

Bending strength test. Flexural testing of the composite has been carried out concerning the ASTM D790 standard. The three-point bending method was adopted to investigate the bending characteristics of the hybrid composite and carried out with the same test equipment for the tensile test with a crosshead speed of 5 mm/min. The composite sample measures $127 \text{ mm} \times 13 \text{ mm} \times 6 \text{ mm}$. In each group, five specimens were tested, and mean values were computed.



Figure 2. Samples of composites: (a) tensile test and (b) flexural test.

Scanning electron microscopy. Morphological analysis of the fracture surface in the composite was carried out on the sample fracture after a tensile test was carried out using an Inspect S50 scanning electron microscope (SEM). The SEM is run with an acceleration voltage of 20 kV and an emission current of 18 mA. The sample was coated with a 10 nm thin gold layer for taking morphology photos. Enlargement of the image is done to get a clear image.

Thermogravimetric analysis. Composites were tested using a TA Instrument thermogravimetric analysis (TGA) Q500 V20.13 Build 39. All the samples were heated from 25 to 600 °C with a heating rate of 10 °C/min.

Results and discussions

Density analysis

Figure 3(a) shows the densities of the different EGs/Al_2O_3 hybrid composites. It was found that more voids were found in the composites with the addition of EGs in composites. The density of a composite material depends on the relative proportion of reinforcing and matrix material, and it is one of the key factors in determining the properties of the composites. The voids significantly affect some of the mechanical properties and even the performance of composites in the workplace. Understandably, a good composite should have fewer voids.

Tensile strength analysis

Figure 3(b) shows the tensile strength of the different EGs/Al_2O_3 hybrid composites. From Figure 3(b) it is known that the tensile strength of the composite increased significantly in the range of 10.25–32.75% of the EGA composite. The composite (EGI sample) with the highest tensile strength had a denser interface between $EGs-Al_2O_3$ -polyester and the particle distribution was more even than the other composites evaluated. The

addition of EGs in the range of 5-10% in the composite is supposed to help the polyester resin effectively transfer the produced stress to the particles, hence enhancing the tensile strength. On the other hand, a decrease in the tensile strength of the composite indicates that improper adhesion inhibits the increase in tensile strength. When the Al₂O₃ content increases (15-20%), there is an accumulation of agglomeration of particles so that the polyester cannot wet the particles because the resin does not enter between the two adjacent filters,²⁴ and the cavities become trapped in the composite, which in turn reduces the tensile strength. However, the tensile strength value of the Al₂O₃/EG hybrid composite (sample EGB. EGH, EGI, EGX, EGY) was higher than that of the flax fiberreinforced composite with Al2O3 filler with a tensile strength value of 25.01–32,8 MPa.²⁵ This shows that the EGs/Al₂O₃ hybrid composite considered in terms of its tensile properties can replace the flax fiber/Al2O3 composite in the automotive industry.

Figure 3(c) shows the elongation values of different EGs/Al₂O₃ hybrid composites. The elongation of the hybrid composite increased significantly as the volume fraction of EGs increased, or in other words, the elongation value of the composite decreased with the increase in the volume fraction of Al_2O_3 in the composite. The highest elongation is owned by the EGY sample (1.5%) and the lowest elongation is owned by the EGA sample (0.8%). This shows that the Al₂O₃ particles are harder and brittle, causing the hybrid composite to be stiffer, which in turn reduces the % elongation of the composite. Maitra et al.²⁶ revealed the fact that Al₂O₃ filler in composites makes it difficult for the chain of movement of the polyester resin molecules which in turn reduces the composite strain. Meanwhile, the hydrophilic nature of natural materials (EG) causes the composite to be softer and unable to sustain the force deformation so that when a tensile load is applied, the composite will shift before fracture.

Figure 3(d) shows the modulus of elasticity of the hybrid composite EGs/Al_2O_3 . It was found that the



Figure 3. Mechanical properties of the EGs/Al₂O₃ hybrid composite.

modulus of elasticity of the hybrid composite increased significantly when the content of EGs 5 and 10 (vol.%) was 10.166% and 22.776%, respectively, of the EGA sample, then decreased with increasing volume fraction of Al_2O_3 particles (\geq 15 vol.%). The EGH composite has the highest modulus of elasticity with a modulus of elasticity of 2554.302 MPa. The modulus of elasticity of the EGA composite is 2092.145 MPa. The value of this modulus of elasticity also shows the stiffness of the composite. The increase in modulus of elasticity is due to the even distribution of Al_2O_3 particles which increases the interface with EG and polyester particles. These Al_2O_3 particles have a higher surface area which allows

complete wetting by the resin and can adapt during failure leading to more effective stress transfer between Al_2O_3 , EG, and resin. Manaila et al.²⁷ stated that complete wetting of the resin in the filler can develop a cross-linked structure that limits the free mobility of the polymer chains, thereby causing the modulus of elasticity of the composite to increase. Meanwhile, for EGI, EGX, and EGY composites, the modulus of elasticity values tends to decrease with increasing volume fraction of EG or decreasing volume fraction of Al_2O_3 . In EGI composites, EGX and EGY modulus of elasticity decreased by 9.78%, 15.666%, and 21.631%, respectively, from EGI composites. This decrease was due to the reduced volume



Figure 4. Composite fracture morphology: (a) EGY, (b) EGX, (c) EGI, (d) EGH, (e) EGB, and (f) EGA.

fraction of Al_2O_3 which is the fact that a large amount of Al_2O_3 can increase the interfacial bond between EG particles and the resin which in turn causes an increase in the modulus of elasticity in the composite.

Flexural strength analysis

Figure 3(e) shows the flexural strength of the different Al_2O_3/EGs hybrid composites. Figure 3(d) it is found that the bending strength of the composite tends to increase significantly when the volume fraction of EGs is in the range of 5–10% and decreases when the Al_2O_3 content is above 10%. The highest bending strength was obtained from the EGH composite of 77.78 MPa. The increase in flexural strength of the composites (EGB and EGH samples) from the EGA samples suggested the intrinsic toughness of the EGs as indicated by the number of Al_2O_3 along the crack path. In addition, the

strong interfacial bond of the EGs–polyester particles with Al_2O_3 (which acts as a cursor) makes it an efficient inhibitor for immobilization and the development of crack propagation during bending testing, thereby increasing its strength.

Figure 3(e) also shows that the bending strength of the EGA sample (66.96 MPa) is lower than that of the EGB sample (72.822 MPa) presumably because a large number of Al_2O_3 particles are the beginning of the formation of micro-cracks for composite failure. On the other hand, the EGA composite was still more effective in resisting bending strength than the EGY composite sample (49.36 MPa). This phenomenon indicates that the EGs have a lower interaction with the polyester matrix compared to Al_2O_3 particles. Other factors that cause high flexural strength of composites, such as organic powder–polyester–inorganic powder interactions, voids, compatibility of particles with the matrix, and

uniformity of particle size. In addition, finer particles can provide mechanical properties. which is higher than the composite due to the strong interaction between the particles and the matrix.²⁸

Figure 3(f) shows the flexural modulus of the EGs/ Al₂O₃ hybrid particle composite. The bending modulus of the EGA composite was 12436.35 MPa, then increased by 11.41% and 16.88% (EGB and EGH composites), respectively. EGH composite has the highest bending modulus of 14535.62 MPa. This increase is a result of the increased interaction of EG-polyester-Al₂O₃ particles so that the particle-matrix interface bond also increases. However, for the volume fraction of EGs greater than 10%, the composite stiffness modulus decreased. For EGI, EGX, and EGY composites, the bending stiffness decreased by 7.38%, 13.44%, and 17.25%, respectively, from the EGH composite. This decrease may be due to the incompatibility of EGs with polyester so that the resulting bonding interface is low, and ultimately accelerates crack propagation and decreases the bending stiffness of the EG/Al₂O₃ hybrid composite.

Morphology analysis

Figure 4 shows the fracture surface of the tensile test of the EGs/Al₂O₃ particle hybrid composite. Figure 4a-d shows almost the same fracture surface where the interface bonds between the particles and the resin appear to be getting tighter and stronger. But compared to the other composites studied; strong and tight interfacial bonds are found in EGI composites, this supports the reason why the strength of EGH composites is the highest. After the addition of Al₂O₃ particles of about 5–15 (vol.%), crack deflection started to occur, and an increase in the hackle area could be attributed to the increase in the stiffness value of the composite. The fracture morphology of the EGX and EGY samples (Figure 4(e) and (f)) showed an increasing number of EGs pull-outs with increasing EGs; and the fracture surface was found to be smoother indicating the type of



Figure 5. TGA curves of EGs/Al₂O₃ hybrid composite.

brittle fracture, this is thought to be the cause of the tensile strength of the composite tends to decrease. Greenhalgh²⁹ stated that brittle fracture in hybrid composites is characterized by the presence of mirror, mist, and hackle regions on the fracture surface.

Thermogravimetric analysis

Figure 5 illustrates the TGA curve of the various EGs/ Al₂O₃ composite hybrid. The composites EGA exhibits a slight weight loss starting at approximately 240 °C, and the sudden drop at approximately 300 °C corresponds to the thermal decomposition of EGA, whereas the corresponding temperature of the single filler composites is significantly enhanced. A decomposition temperature of approximately 375 °C was acquired in the EGs-Al₂O₃ composite. Moreover, the residual weight of the EGY composite reached 81%, which is an evident enhancement compared to 78% for EGI, 59% for EGH, and 39% for EGA. The enhancement in thermal performance may be attributed to the combined effects of the hybrid fillers. The good dispersion of fillers in the hybrid composite boosts the interaction between the matrix and fillers, which can successfully restrict the kinetic motion of molecular chains from the matrix. Hence, this indirectly enhances the thermal stability of the composite materials. The outstanding thermal resistance of EGs composites can effectively guard the bump heat decomposition, which indicates considerable potential as an underfill encapsulation material for electronic packaging.

Conclusion

Bio-fiber based EG/Al_2O_3 particles hybrid biocomposites have been developed in this work and their properties have been investigated. The following conclusions can be drawn:

- The EGI sample and the EGH sample had the highest tensile strength and flexural strength of 38.697 and 77.786 MPa, respectively.
- The increasing EG concentration in the composite from 5% to 10% has increased the bond strength between EGs–Al₂O₃–polyester, enabling the mechanical strength of the hybrid composite to increase. Because it forms several voids in the composite, the presence of a considerable amount of Al₂O₃ in the composite diminishes the mechanical and thermal properties of the composite. As a result, the mechanical strength of the composite reduces.
- The total composite demonstrated strong thermal properties with the addition of EGs, with the best heat resistance properties obtained when the composite comprised solely EGs.
- According to the experimental results, the mechanical properties of the EGs/Al₂O₃ hybrid composite are suitable for replacing flax fiber composites with Al₂O₃ filler in the automotive industry.

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ORCID iD

Mavinkere Rangappa Sanjay D https://orcid.org/0000-0001-8745-9532

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