BI-LAYER HYBRID BIOCOMPOSITES: CHEMICAL RESISTANT AND PHYSICAL PROPERTIES

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Bi-layer hybrid biocomposites were fabricated by hand lay-up technique by reinforcing oil palm empty fruit bunch (EFB) and jute fibre mats with epoxy matrix. Hybrid composites were prepared by varying the relative weight fraction of the two fibres. The physical (void content, density, dimensional stability), and chemical resistant properties of hybrid composites were evaluated. When the jute fibre loading increased in hybrid composites, physical and chemical resistant properties of hybrid composites were enhanced. Void content of hybrid composites decreased with an increase in jute fibre loading because jute fibres showed better fibre/matrix interface bonding, which leads to a reduction in voids. The density of hybrid composite increased as the quantity of jute fibre loading increased. The hybridization of the jute fibres with EFB composite improved the dimensional stability of the hybrid composites. The performance of hybrid composites towards chemical reagents improved with an increase in jute fibre loading as compared to the EFB composite. The combination of oil palm EFB/jute fibres with epoxy matrix produced hybrid biocomposites material that is competitive to synthetic composites.

Keywords: Fibres; Oil palm; Jute; Hybrid composites; Physical properties; Chemical resistant

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INTRODUCTION

Natural fibres such as jute, kenaf, sisal, hemp, banana, flax, oil palm, *etc.*, have been in considerable demand in recent years due to their eco-friendly and renewable nature (Hassan *et al.* 2010; Jawaid and Abdul Khalil 2011; Akil *et al.* 2011). Natural fibres from renewable natural resources offer the potential to act as a biodegradable reinforcing material alternative for the use of synthetic fibres. The resurrection of interest in using natural fibers as reinforcement in polymer composites is mainly due to their techno-ecological advantages. They offer various advantages over glass and carbon fibres such as lower cost, light weight, lower specific weight, renewability, acceptable specific properties, lower density, less wear and tear in processing, lower energy requirements for processing, biodegradability, wide availability, and relative non-abrasiveness (Jawaid and Abdul Khalil 2011).The leading driver for substituting natural fibres for glass is that they can be grown with lower cost than glass (Satyanarayana *et al.* 2009).

Despite the advantages, use of natural fibre reinforced composites has been restricted due to its high moisture absorption tendency, poor wettability, and low thermal stability during processing, as well as poor adhesion with the synthetic counterparts. Most of the drawbacks that have been identified can be overcome by effective hybridization of natural fibre with synthetic or natural fibres (Abdul Khalil *et al.* 2011; Jawaid and Abdul Khalil 2011; Öztürk 2010). Extensive studies have been done on natural fibre reinforced composites such as sisal (Dwivedi *et al.* 2010), jute (Alves *et al.* 2010), flax (Cherif *et al.* 2010), hemp (Islam *et al.* 2011), kenaf (Abu Bakar *et al.* 2010), banana (Sapuan *et al.* 2007), oil palm EFB (Khalil *et al.* 2010), and pineapple (Wan Nadirah *et al.* 2011). These studies have shown that natural fibres have the potential to act as an effective reinforcement in a thermoset or a thermoplastic matrix.

In the case of polymer composites, hybrid composites are systems in which one kind of reinforcing material is incorporated into a mixture of different matrices (blends) (Thwe and Liao 2003), or two or more reinforcing and filling materials are present in a single matrix (Karger-Kocsis 2000; Fu *et al.* 2002), or both approaches are combined. The incorporation of two or more natural fibres into a single matrix has led to the development of hybrid composites. The behaviour of hybrid composites is a weighted sum of the individual components in which there may be a more favourable balance between the inherent advantages and disadvantages. The strength of the hybrid composites is dependent on the properties of fibres, aspect ratio, fibre content, length of an individual fibre, orientation of fibre, extent of intermingling of fibres, fibre to matrix interface bonding, arrangement of both of the fibres, and also on the failure strain of individual fibres (Sreekala *et al.* 2002).

The present study focuses on the fabrication of bi-layer hybrid composites of oil palm EFB/jute reinforced epoxy composites. Physical and chemical resistance properties of oil palm EFB composites as a function of jute fibre loading and fibre composition were studied. The properties obtained in different parameters were compared in order to analyze the effectiveness of hybrid composites. The study explores the potential utilization of jute fibres as reinforcement with the combination of locally available oil palm EFB fibres reinforcement in epoxy matrix, with the goal to enhance utilization of oil palm fibres in different applications. Challenges still exist in the development of more suitable cost-effective fabrication techniques, but the progress so far obtained in this field has allowed the application of natural-fibre polymer composites in many sectors such as in consumer items and more importantly, in the automotive industry. The automotive market sector shows an increase in consumption of natural-fibre usage. The insertion of natural fibers in the different sectors has experienced a growth rate of 13% compounded over the last 10 years to an annual use of approximately 275 million kilograms (Report 2004).

EXPERIMENTAL

Materials

Oil palm EFB fibre mat was supplied by Ecofibre Technology Sdn. Bhd., Malaysia. Jute fibre mat was procured from Indarsen Shamlal Pvt. Ltd. (Jute House Since 1948), Kolkata, India. Both oil palm EFB and jute fibre mats consist of chopped and unidirectional fibres. The physical and mechanical properties of oil palm EFB and jute fibres are tabulated in our previous work (Jawaid and Abdul Khalil 2011).The epoxy A331 (diglycidyl ether of Bisphenol A) and epoxy hardener A062 (reactive polyamide) were used in this study. Both the epoxy resin and commercial curing agent were obtained from Zarm Scientific & Supplies Sdn. Bhd., Malaysia. Benzyl alcohol was used as diluents, silicone oil was used as a releasing agent, and chemical reagents were supplied by Aldrich Company.

Preparation of Hybrid Composites

For the preparation of composites, a stainless steel mould with dimensions of 304 mm x 203 mm was used. The mould cavity was coated with a thin layer of silicone oil solution, which acts as a releasing agent. In order to make the bi-layer hybrid composites, epoxy resin and polyamide with 100:60 ratios were mixed. Benzyl alcohol was added as a diluent, and the mixture was mixed thoroughly by mechanical stirrer for 15 min (Table 1). Bi-layer hybrid composites were developed by using the hand lay-up technique for making the test sample. Keeping the different weight ratio of oil palm EFB and jute 4:1, 1:1, and 1:4, and total fibre loading at 40% by weight, bi-layer hybrid composites were prepared. Mats of fibres were prepared and were impregnated in epoxy resin in a mould. Then, the mould was closed for curing and was left to cure at 105^{o}C for 1 hour in a hot press. An open mould method was used in this research. The mould was compressed at a constant pressure of 275 bars while squeezing out the excess resin. Once the composite was cured, it was removed from the mould and followed by post curing in an oven at 105^{o}C for 30 minutes. Finally, bi-layered biocomposites were cooled in a cold press under constant pressure of 250 bars for 15 minutes in order to prevent warping of the hybrid composites.

Table 1. Resin Formulation Used For Impregnation Process

Types	Quantity
Epoxy Resin	100 phr*
Epoxy Hardener(Reactive Polyamide)	60 phr
Benzyl alcohol	10 wt% of total Epoxy-polyamide mixture

*phr=Parts per hundred by weight

CHARACTERIZATION

Void Content

For determination of void contents in composites, ASTM D2734 method was used. The void content was determined from the theoretical and experimental density of the composites through Equation (1),

$$Void\ Contents\,(\%) = \frac{\rho_{theoretical} - \rho_{experimental}}{\rho_{theoretical}} \quad (1)$$

where $\rho_{theoretical} = 1 \Big/ \left[\left(W_f \big/ \rho_f \right) + \left(W_m \big/ \rho_m \right) \right]$,

and W_f is the fibre weight fraction, W_m is the matrix weight fraction, ρ_f is the fibre density, and ρ_m is the matrix density.

Density

Density was measured by using the ASTM D1895 standard. The density of the samples was calculated by using Equation (2) below. Five samples of each different type of bi-layer hybrid composites were used, and the average value was reported. Density is given by,

$$Density\left(g \big/ cm^3 \right) = \frac{m}{v} \qquad (2)$$

where m is the mass of the composites, and v is the volume of composites.

Dimensional Stability Test

The dimensional stability tests involved were thickness swelling and water absorption. Thickness swelling and water absorption were conducted as per ASTM D 5229. Before testing, the weight and thickness of each sample were measured. Five samples of each type of composite were immersed in distilled water at room temperature. After 24 hours, the sample was taken out and dried before its weight and thickness were measured. The weight and thickness values of the samples were taken. The dimensional stability test was continued for several hours until the constant weight of samples was obtained.

Chemical Resistance Test

The chemical resistance of samples was studied using the ASTM D543-87. The effect of three solvents, *i.e.*, toluene, benzene, and carbon tetrachloride, the effect of three acids, *i.e.*, nitric acid, hydrochloric acid, acetic acid, and the effect of three alkalis, *i.e.*, NaOH, Na_2CO_3, and NH_4OH were studied on the EFB, jute, and hybrid composites respectively. In each case, five pre-weighed samples were dipped in the respective chemical reagents for 24 hours. They were then removed and immediately washed in distilled water and dried by pressing them on both sides with filter paper at room temperature. The samples were then weighed, and the percentage weight loss/gain was determined. The percentage of weight loss/gain was determined using the following equation.

$$Weight\ loss(\%) = \frac{Final\ Weight - Original\ Weight}{Original\ Weight} \qquad (3)$$

RESULTS AND DISCUSSION

Void Content

Table 2 shows the void content of the EFB, jute, and hybrid composites. Oil palm EFB composite showed higher voids, which was attributed to poor fibre/matrix bonding and ultimately to incomplete wetting of oil palm EFB fibre by epoxy matrix (Abdul Khalil *et al.* 2007). Void content of hybrid composites decreased with an increase in jute fibre loading in oil palm EFB composite. In other words, jute composite showed better fibre/matrix interface bonding, as evidenced by a reduction in voids. Voids formation in polymer composites can be attributed to the processing effect, which arises from several sources, such as air bubbles entrapped within the epoxy matrix, residual solvents, and volatiles arising during curing of the resin (Öztürk 2010). Voids may occur in the matrix at the fibre/matrix interface or within the fibre lumens, which will affect the composite properties and decrease the density of the composites (Karina *et al.* 2008). Jute fibre has better wettability with epoxy resin; because of that, jute composite contains few voids compared to EFB composite (Jawaid *et al*. 2011). High voids in the composite leads to lower fatigue resistance, greater susceptibility to water absorption, and increased variation in mechanical properties.

Table 2. Void Content, and Density of EFB, Jute, and Bi-layer Hybrid Composites

Composites	Void Content (%)	Density (g/cm^3)
EFB	8.67	1.04
EFB: Jute (4:1)	6.03	1.09
EFB: Jute (1:1)	4.27	1.12
EFB: Jute (1:4)	3.38	1.14
Jute	2.67	1.20

Density

Table 2 shows the density of EFB, hybrid, and jute reinforced epoxy composites. The density of the hybrid composite increased as the quantity of jute fibre loading increased. The density of oil palm EFB composite increased with an increase in addition of the jute fibre, which may be due to the higher density of jute fibre compared to the oil palm EFB fibres. It was observed that the density of jute composite was the highest as compared to the other composites. Similar work done on oil palm EFB/glass fibre hybrid composites indicated that the density of hybrid composites increases with an increase in glass fibre loading to oil palm EFB composite (Sreekala *et al.* 2002; Abdul Khalil *et al.* 2007). This result is mainly attributed to a higher density of glass fibres compared to oil palm fibres.

Thickness Swelling

Figure 1 shows the thickness swelling behaviors of EFB, jute, and EFB/jute hybrid composites after being immersed in water for several hours. Thickness swelling of hybrid composites significantly decreased with an increase in jute fibre loading, while a reverse trend was observed as the oil palm EFB fibre content increased in hybrid composites. It can be clearly seen that thickness swelling increased with immersion time and reached a certain value where no more thickness swelling occurred. When the immersion time of composites is increased, a significant amount of water is absorbed, resulting in the swelling of the fibre until the cell walls are saturated with water. Beyond that, water exists as free water in the voids, which leads to composite delamination or void formation (Gassan and Bledzki 1997; Das *et al.* 2000).

Oil palm EFB/jute (1:4) hybrid composite showed the lowest percentage in thickness swelling compared to other hybrid composites (Fig. 1). The decrease in thickness swelling is mainly attributable to the less hydrophilic nature of jute fibre compared to oil palm EFB fibres and less void content in hybrid composites compared to EFB composite. Therefore, as the EFB fibre content decreases, thickness swelling of the hybrid composites decreases due to the decrease in water absorption into the composites.

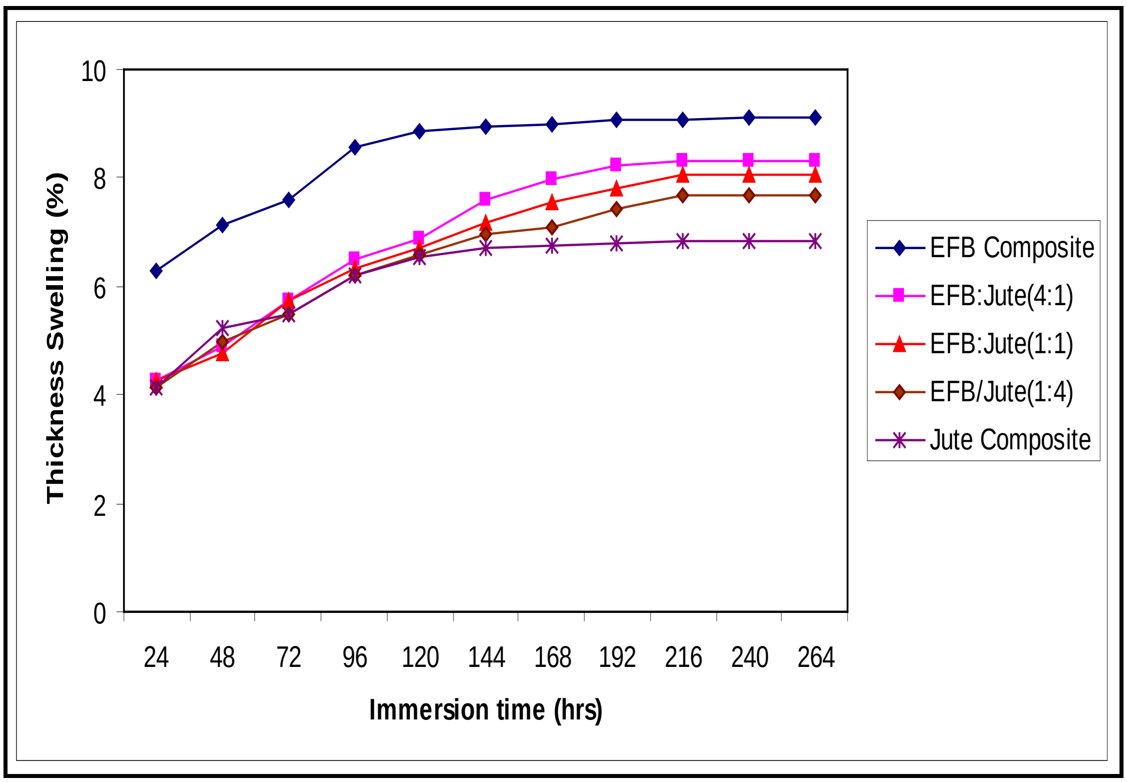

Fig. 1. Thickness swelling (%) of bi-layer hybrid composites, EFB, and jute composites

Water Absorption

The water absorption curves of EFB, jute, and hybrid composites are shown in Fig. 2. Water absorption in a natural fibre reinforced composite is dependent on temperature, fibre loading, orientation of fibres, permeability of fibres, surface protection, area of the exposed surfaces, diffusivity, void content, hydrophilicity of the individual components, *etc.* (Sreekumar *et al.* 2007). Hybridization of jute fibre with oil palm EFB fibre in epoxy resin decreases water absorption of the EFB composite. As the loading of jute fibre increases, the amount of water absorption of hybrid composites decreases. This is mainly attributable to the packed arrangement of hybrid composites and the less hydrophilic nature of jute fibre as compared to oil palm EFB fibres. This could be due to fewer voids in hybrid composites compared to EFB composite. It is consistent with the fact that water molecules are able to accumulate in the voids, leading to an increase in the water absorption of composites. Oil palm EFB/jute (1:4) hybrid composite showed the lowest percentage in water absorption compared to other hybrid composites. Furthermore, the increased water absorption of the hybrid composites at a high weight fraction of oil palm EFB fibre can be attributed to poor compatibility between the EFB and epoxy matrix. The oil palm EFB/jute (4:1) bi-layer composite show higher water absorption behaviour as compared to tri-layer hybrid composites at the same ratio of oil palm and jute fibres (Jawaid *et al.* 2011)

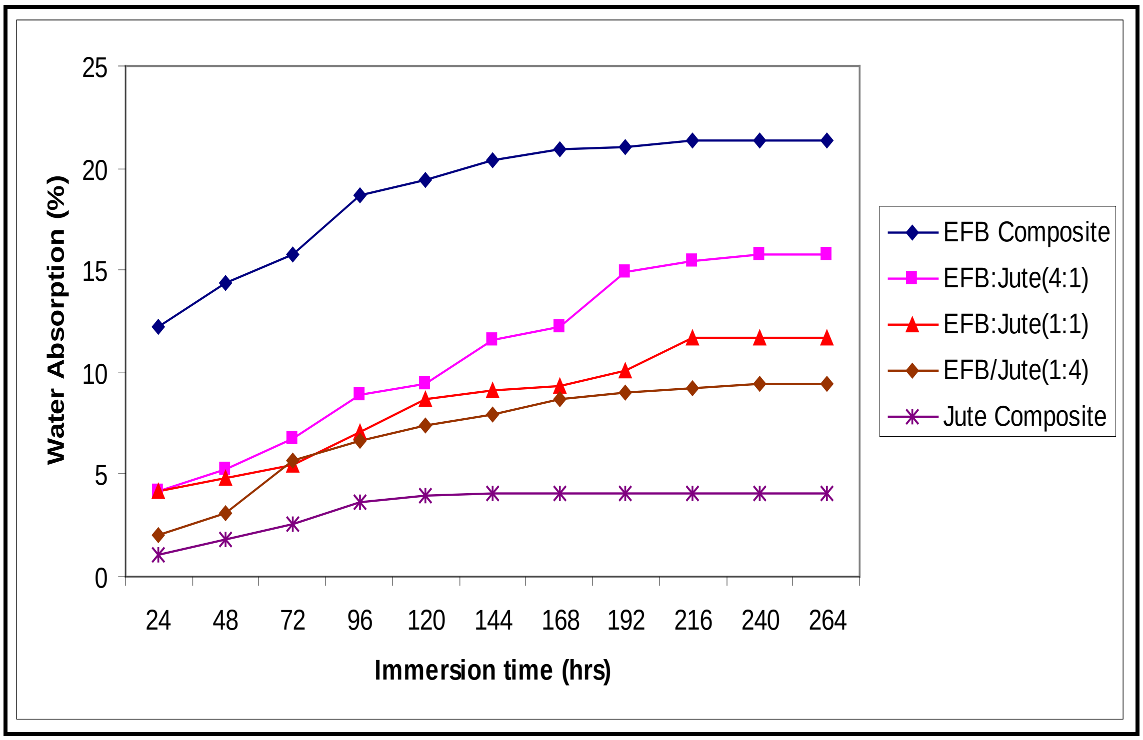

Fig. 2. Water absorption (%) of bi-layer hybrid composites, EFB, and jute composites

At higher volume fraction of oil palm EFB fibres, the micro-level processing of the composites becomes difficult and leads to fibre layering, which creates micro-voids and cracks within composites (Abdul Khalil *et al.* 2007). Micro-void and crack formation in the composites creates pathways for the water molecules to diffuse easily in composite materials, thereby enhancing water absorption. Absorbed water causes weakening of fibre/matrix interfacial adhesion (Pavlidou and Papaspyrides 2003). Cracking and blistering of fibres cause high water absorption, while degradation causes leaching of small molecules (Bao and Yee 2002). Hybridization of oil palm EFB composite with jute fibre caused a reduction in voids and cracks in the hybrid composites and reduced water absorption of the EFB composite.

The water absorption of jute/glass and oil palm EFB/glass hybrid composites were found to be less than oil palm EFB/jute composite (Abdul Khalil *et al.* 2007, 2009; Koradiya *et al.* 2010). This is due to the fact that fibre in hybrid composites are arranged in a close packed manner in which the water-impermeable glass fibres act as a barrier, preventing the contact between water and the natural fibre and lowering water absorption properties of these composites (Mishra *et al.* 2003). The water uptake of hybrid composites was found to be less than that of unhybridized composites (Priya and Rai 2006). Researchers studied water absorption behaviour of different hybrid composites and observed that a sisal/cotton composite absorbs more water than ramie/cotton or jute/cotton hybrid composites (Alsina *et al.* 2007). These results were due to a higher hydrophilic characteristic of sisal fibre compared to jute and ramie fibres.

Chemical Resistance

The percentage of weight gain (+)/loss (-) of the EFB, jute, and hybrid composites, immersed in three acids, three alkalis, and three solvents for 24 hours are shown in Table 3.

Table 3. Chemical Resistance of Oil Palm EFB/Jute Fibre Reinforced Bi-Hybrid, EFB, and Jute Composites

Chemicals	EFB Composite	EFB: Jute (4:1) Composite	EFB: Jute (1:1) Composite	EFB: Jute (1:4) Composite	Jute Composite
Benzene	8.30	6.25	3.16	1.57	0.50
Toulene	4.35	4.43	3.67	2.52	0.45
CCL_4	5.11	4.35	2.68	1.09	0.39
H_2O	20.49	12.98	7.89.	5.34	3.00
Hcl	15.96	11.37	8.43	4.27	1.93
HNO_3 (40%)	13.76	9.83	6.67	3.79	2.14
CH_3COOH (5%)	20.46	18.93	11.32	7.87	3.97
NaOH (10%)	15.55	11.62	8.66	6.82	5.33
Na_2CO_3 (20%)	10.08	6.88	4.93	2.87	0.89
NH_4OH (10%)	21.25	13.06	10.85	7.49	4.06

From the table, it is evident that weight gain was observed in EFB, jute, and hybrid composites for all chemical reagents. The performance of hybrid composites toward chemical reagents improved with an increase in jute fibre loading compared to that of EFB composite. In this work, the lower void content at higher jute fibre loading caused lower chemical absorption by the hybrid composites because the composite materials were swollen with gel formation rather than dissolving EFB composite due to higher jute fibre content. The positive value indicates that the composite materials were swollen with gel formation rather than dissolving in the chemical reagents (Raghu *et al.* 2010). Thus, an increase in jute fibre loading to EFB composite contributes to good chemical resistance to acids, alkalis, and solvents due to close packing between jute fibre and epoxy matrix. Several researchers have studied chemical resistance of hybrid composites and found that the hybrid composites are strongly resistant to almost all chemicals except carbon tetrachloride (John and Naidu 2007; Subba Reddy *et al.* 2010; Venkata Reddy *et al.* 2007; Raghu *et al.* 2010). Chemical resistance test observation of hybrid composites suggests that these hybrid composites can be used for making storage tanks for water and chemicals.

CONCLUSIONS

1. Bi-layer hybrid composites are constituents of natural fibres and show good physical properties and environmental advantages. Hybridization of EFB composite with jute fibres significantly reduced void content and increased the density of hybrid composites.
2. As the loading of jute fibre increased, the amount of thickness swelling and water absorption of bi-layer hybrid composites decreased. This is mainly attributed to the packed arrangement of hybrid composites and less hydrophilic nature of jute fibre compared to oil palm EFB fibres.
3. The oil palm EFB/jute (1:4) bi-layer hybrid composite showed the lowest percentage in thickness swelling compared to other hybrid composites. In this study the chemical resistance of epoxy based bi-layer EFB/jute hybrid composites clearly indicates that these composites are strongly resistant to all chemicals.

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