Effects of Silica Addition and Alkaline Surface Treatment of Kenaf Fibre on Mechanical Properties of Hybrid Epoxy/Silica/Kenaf Composites using Hand Lay-up Method

Che Nor Aiza Jaafar[1,*] and Ismail Zainol[2]

[1]Department of Mechanical and Manufacturing Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43000 Serdang Selangor, Malaysia.
[2]Chemistry Department, Faculty of Science and Mathematics, Universiti Pendidikan Sultan Idris, 35900 Tanjong Malim, Perak, Malaysia.

Abstract

Epoxy/silica/kenaf composites were fabricated using a simple technique of hand lay-up method. The effects of silica addition on mechanical properties of epoxy/kenaf composites were studied by varying silica contents, ranging from 10 to 50 wt% while kenaf fibre was fixed at 24.5 wt%. Non-woven kenaf fibre used in this research was treated with 3 wt% NaOH to improve surface interaction between epoxy matrices. The composites were fabricated using hand lay-up method in metal mould and then they were pre-cured at 80 ^oC for 1 hour and post-cured at 110 ^oC for 1 hour. The mechanical properties of composites were examined by means of flexural and impact tests. It is found that silica content of 30 wt% has produced epoxy/silica composites with highest impact strength of 6.5 kJ/m^2. However, addition of treated kenaf fibre has given the strongest composite with the values of 54 MPa for flexural strength and 10.6 kJ/m^2 for impact strength. Field emission scanning electron microscope (FESEM) was applied to investigate the morphology of the fractured surface and it revealed the treated kenaf fibre improved the surface interaction within epoxy matrix, thus lead to the higher mechanical properties of composites. The composite produced from this research has potential to be used in household and domestic product applications.

Keywords: kenaf fibre, silica, epoxy composites

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*Corresponding author: Che Nor Aiza; e-mail: cnaiza@upm.edu.my
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Introduction

Green composite made of renewable agricultural and forestry feedstock is the best alternatives to synthetic fibre reinforced composites. There are many advantages of using natural fibres such as abundance, biodegradability, non-corrosive, minimal health hazard, fracture resistance, acceptable specific properties and renewable [1]. In recent decade, application of conventional fibre has decreased as it is replaced with natural fibre which has similar strength and characteristics. Research from previous studied has proved the addition of fillers, catalyst and treated fibre from natural fibre can be used to substitute conventional synthetic fibre [2-3]. Sanjay and Yogesha [2] in their research reported that the application of natural fibre in hybrid composite was used mainly for lightweight applications. The application of natural fibre composite in various industries such as manufacturing and automotive can give benefits in term of economic, environmental and social aspects as this type of fibre has better properties in electrical resistance and possesses good thermal and mechanical properties as well as high resistance to fracture [3].

The major drawback of natural fibre as filler in polymer composites is their difference in surface properties where natural fibre is more hydrophilic as compared to hydrophobic nature of polymer matrix. As a results, poor interaction between fibre and matrix has reduced the mechanical properties of the composite materials [4]. These circumstances can be overcome by doing surface modification of natural fibres or modification of polymer matrix [5].

Alkali surface treatment has become an effective methods to modify the surface of natural fibre as its improved mechanical properties of natural fibre composites [6]. Research done by Ming et al. (2016) have proved that alkaline treatment of abaca fibres with 6% NaOH successfully improved crystallinity, tensile strength, Young's modulus of the treated fibre and interfacial shear strength of the epoxy/abaca composites [7]. Generally, treatment with alkaline loosen the fibre due to dissolution of component such as hemicellulose, lignin, wax and other impurities [8]. The fibres become shrunk, more parallel and rough after treatment [9]. Thus, it improved mechanical interlocking between natural fibre and resin. The schematic diagram of alkaline treatment of natural fibre is shown in Figure 1.

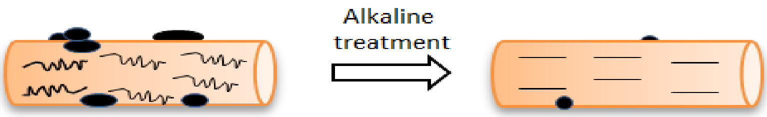

Figure 1. Principle of chemical treatment on natural fibre [10].

Hand lay-up is the simple technique to fabricate various composite products from small to large components. In this process, the non-woven fibres are cut into required size and placed in the mould followed by laminating resin on top of fibres. This technique is known as simple and economical as it does not use complicated machine. It is important technique in composite industries as their adaptability and this technique can produce a large quantity of composites [6].

Improvement of mechanical properties of epoxy resin by addition of silica as fillers have been studied by many researchers [11-12]. Mitja and Branka [13] have studied the effect epoxy/silica composites using different types of silica fillers and they found that the type of fillers have affected the flowability of epoxy. Fused silica produced lowest mechanical properties of epoxy composites as compared to glass beads, foundry sand and crystalline silica type.

The objectives of this research are to examine the effects of alkaline surface treatment of kenaf fibre in epoxy/kenaf composites on mechanical properties and addition of silica as fillers in epoxy/kenaf composites.

Materials and Methods

Materials

Epoxy resin, DER 331 was supplied by Dow Chemical Pacific Singapore and cycloaliphatic amine was used as curing agent manufactured by Epochemie. Non-woven kenaf fibre and silica powders (44 μm) were purchased from Innovative Pultrusion Sdn. Bhd. (IPSB), Seremban, Malaysia and Maju Saintifik Sdn. Bhd., Shah Alam, respectively.

Composite preparation

Non-woven kenaf fibre mat was cut into 180 mm x 180 mm to fit the metal mould. The kenaf fibre mat was treated with 3 wt% NaOH for 24 hours at room temperature and washed with distilled water before dried in oven at 60 ^{o}C. Silica powders were dried in oven at 80 ^{o}C for overnight. For epoxy/silica composites preparation, epoxy resin was mixed with 10 phr silica powder using mechanical stirrer for 30 min at 600 rpm. The curing agent was mixed to the mixture with the proportion 60:40 of resin: hardener. The mixtures were stirred and pour into the mould uniformly and undergone curing process. For epoxy/silica/kenaf composites fabrication, the kenaf fibre mat was placed on top of the thin layer of epoxy/silica resin (uncured) in the mould and the second epoxy/silica resin was applied on top of the fibre. Another layer of kenaf fibre was placed on top of the fibre in the mould and the resin was placed on top of the fibre. The process was repeated for four times to obtain four layers. Finally, the metal plate was placed on top of the composite and clamp securely before undergone curing process. The metal mould was placed in an oven and heat for 2 hours at 80 °C. The sample was then post cured for 2 hours at 110 °C.

After curing process and cooling at room temperature, the composites were detached from the mould and labelled. Epoxy composite formulations is summarised in Table 1. Sample EP refers to pure epoxy and sample EK is the epoxy/kenaf composite with 24.5 vol% of untreated non-woven kenaf fibre mat. Sample ES is epoxy/silica composite with 10 phr of silica content. Sample EKS20 to EKS50 corresponds to the epoxy/silica composites (20 to 50 phr of silica) added with 24.5 vol% untreated non-woven kenaf fibre mat. Meanwhile ETKS30 refers to epoxy/silica composite (30 phr silica) added with 24.5% alkaline treated non-woven kenaf fibre mat.

Table 1. Epoxy composites formulation

Sample Composite	Volume Ratio		Silica (phr)
	Epoxy Resin (vol%)	Kenaf (vol%)	
EP	100	0	0
EK	100	24.5	0
ES	100	0	10
EKS20	75.5	24.5	20
EKS30	75.5	24.5	30
EKS40	75.5	24.5	40
EKS50	75.5	24.5	50
ETKS30	75.5	24.5	30

Mechanical properties

Impact test was carried out using GUNT WP400 Charpy impact tester according to ASTM D6110 standard. Ten specimens with dimensions of 60 mm x 12 mm were prepared for further analysis. The energy required to break the samples were recorded and average of the measurements was taken as impact strength (kJ/m^2). Instron universal testing machine was used to conduct the flexural test according to ASTM D790. The specimens were cut into 120 mm x 12 mm dimension and the cross head speed was set at 2.0 mm/min. In this test the ability of the composite materials withstand applied bending forces was measured and the average data from five specimens were taken as the flexural strength.

Morphology analysis of fractured surface

Field emission scanning electron microscopy (FESEM), model Hitachi SU8020 was used to observe the morphology of the fractured surface of impact samples and kenaf fibre. The sample was coated with Pt for 150 second prior to analysis.

Results and Discussion

Kenaf fibre treatment

Surface morphology of the alkaline treated and untreated kenaf was analysed using FESEM as it shown in Figure 2. The untreated kenaf shows rough surface with many powdery impurities (Figure 2a). The impurities in untreated kenaf might come from cellulose, hemicellulose and lignins. The powders attached to the kenaf surface were probably from the broken fibres. Kalia et al. [14] in their research have reported that rough fibre surface occurred was due to high concentrated hydroxyl groups on the surface of kenaf. Meanwhile the alkaline treated kenaf showed clean and rough surface as shown in Figure 2b. It is clearly observed that the fibre become shrunk and more parallel after treated with 3% NaOH.

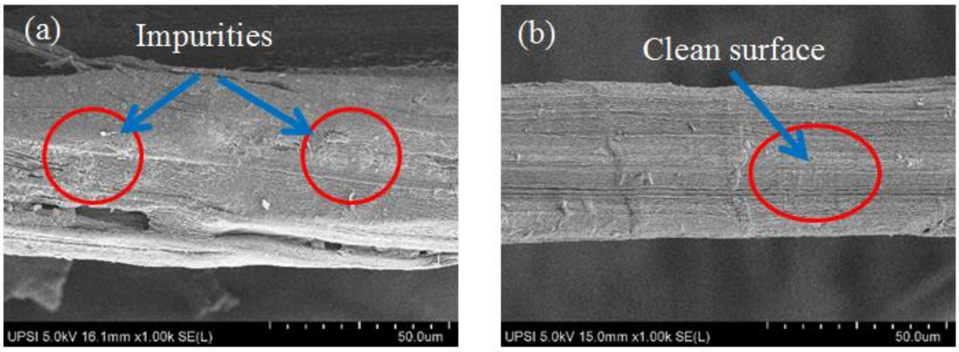

Figure 2: FESEM micrographs of (a) untreated kenaf and (b) 3 wt% NaOH treated kenaf fibre

The functions of alkaline treatment is to remove the hemicellulose, lignin and wax from the outer surface of kenaf fibers. Similar observation was also reported by Kasiviswanathan et al. [15] where they reported that the alkaline treatment washed away the impurities on the surface of kenaf, thus improved the surface interaction between kenaf fibre and polymer matrix. The alkaline surface treatment mechanism is shown in Figure 3. The surface of kenaf become more hydrophobic as most the hydroxyl groups and impurities was eliminated after alkaline treatment.

$$Cell\text{-}OH + NaOH \rightarrow Cell\text{-}O^{-}Na^{+} + H_2O + \text{surface impurities}$$

Figure 3. Chemical reaction during alkaline treatment

Impact Test

The results of impact strength of epoxy and its composites are summarised in Figure 4. It can be seen that the addition of 24.5 vol% of kenaf fibre in epoxy composite (EK) has increased the impact strength of pure epoxy (EP) from 1.5 kJ/m^2 to 2.0 kJ/m^2. However, addition of 10 phr silica as fillers in epoxy composite (ES) have improved the impact strength of epoxy/silica composite to 2.82 kJ/m^2. The addition of silica fillers of 20 phr in epoxy/silica/kenaf composite has slightly improved the impact strength to 3.2 kJ/m^2 as indicated by composite EKS20. The highest impact strength is shown by composite EKS30 as 30 phr silica content was added to the epoxy/silica/kenaf composites.

From the impact test results, it inferred that silica powders has acted as particulate fillers which reinforced the matrix by providing stress transfer between fillers and matrix. The presence of silica fillers in the composite was believed to improve interfacial interaction between fillers and polymer matrix that helped to transfer stress between fillers and matrix [6]. The impact strength however reduced to 5.2 kJ/m^2 after 50 phr silica was added to the composite. It is interesting to note that the impact strength of composite has achieved to the maximum value of 10.6 kJ/m^2 after kenaf fibre was alkaline treated. Overall impact analysis concluded that the composite with treated kenaf fibre and silica addition of 30 phr exhibited the highest impact strength at about 187.73 % which was higher than pure epoxy. This was probably due to strong surface interaction between kenaf fibre and matrix gave additional energy absorption during impact [16].

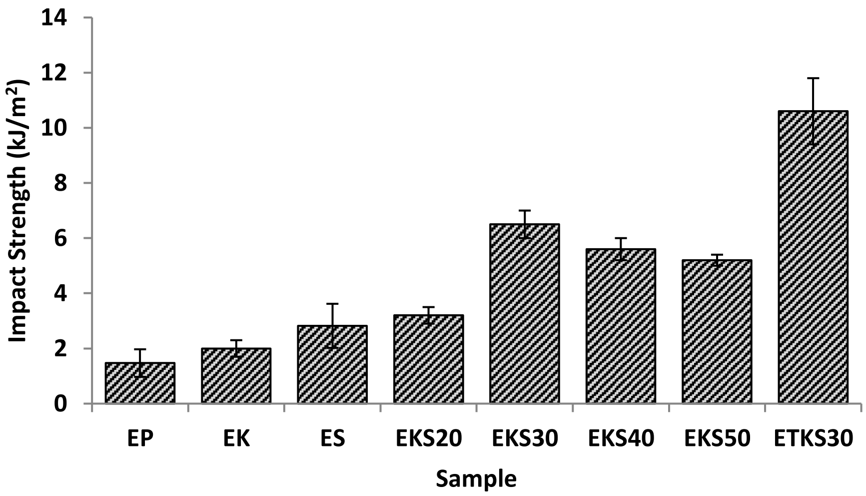

Figure 4: The effect of epoxy composites formulation on impact strength

Flexural Test

Flexural strength of pure epoxy (EP) and selected composites (EKS30 and ETKS30) are shown in Figure 5. It can be noticed that the flexural strength of epoxy/treated kenaf composites with addition of 30 phr silica filler (EKS30) was higher than that of epoxy/silica/untreated kenaf composites (ETKS30). The maximum flexural strength of epoxy/silica/treated kenaf composites is 54.0 MPa. These results indicated that the epoxy composites with surface treated kenaf fibre have significantly improved the flexural strength. This can be explained by the cleaning the kenaf's surface can improve the ability of fibre to interact with the matrix, thus it increased the stress transfer between fibre and matrix. Similar observation was also reported by Yousif et al. [17] in their work on epoxy/kenaf composite with alkaline treated kenaf fibre.

The presence of silica powders in the polymer matrix permitted plastic void growth around the debonded particles, thus increased the difficulty of crack propagation during fracture [18]. The results in Figure 3 also show that the composite with addition of 30 phr of silica with or without surface kenaf treated exhibited higher impact strength for epoxy/silica composites. The flexural strength of composite was more improved after being alkaline treated as seen in ETKS30 composite. Higher filler loading is required to achieve high toughening effect of epoxy composite, but this increases the viscosity of modified epoxy resin [19].

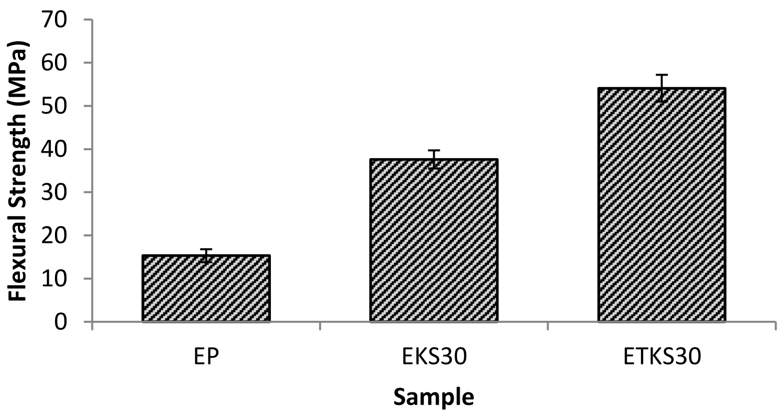

Figure 5: Flexural modulus of silica modified epoxy composites

Morphology analysis of fractured surface

Figure 6 shows the FESEM micrographs of fractured surface of ES, EKS30 and ETKS30 composites. As shown in Figure 6a, the clean fracture surface of epoxy/silica composites (ES) revealed a brittle failure mode of the composites. The smooth surface morphology of the composite showed homogeneous spreading of silica fillers in entire matrix. Similar observation was also reported by Xue et al. [20].

FESEM micrograph of epoxy/silica/untreated kenaf (EKS30) composites (Figure 6b) exhibited a rough fractured surface with fibre pull out was seen on the surface. The pull out of fibre (marked with white arrow) indicated the weak surface interaction between fibre and matrix. The pull out of silica particle can be seen as cloudy spherical in the matrix. The presence of dark spot (red circles) in the matrix was due to void formation as a results of fibre pull-out due to weak surface interaction between fibre and matrix. The dark spot observed on the surface of composites indicated the failure of fibres that contributed to the strength of the composites [17]. It can be seen that ETKS30 composite (Figure 6c) has less dark spot but more broken fibre (red circles) distributed in the matrix, meaning that good surface interaction between fibre and matrix, thus gave higher impact and flexural strength of the composites.

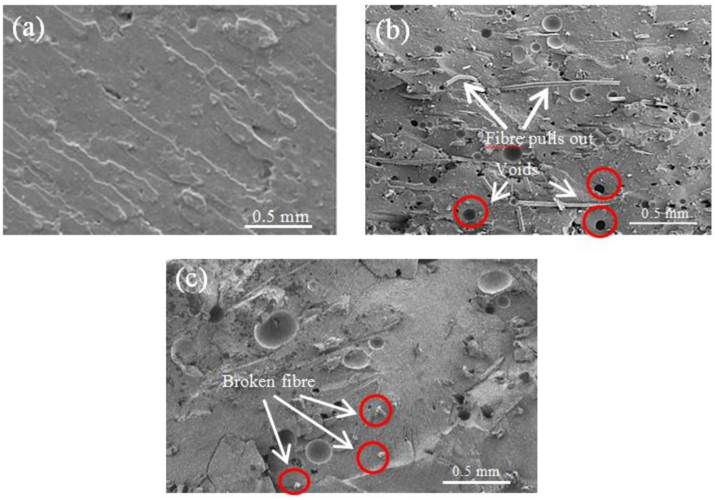

Figure 6: FESEM micrographs of fractured surfaces of (a) ES composite (b) EKS30 composite and (c) ETKS30 composite

Conclusions

Hand lay-up method has successfully produced reasonably good mechanical properties of epoxy/silica/kenaf composites. Epoxy composite modified with silica and alkaline treated kenaf exhibit the highest impact and flexural modulus which are 10.6 kJ/m^2 and 54.1 MPa, respectively. Analyses using FESEM proved the alkalinisation of kenaf with 3 wt% of NaOH improved interfacial interaction between fibre and matrix.

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Author Contributions

All authors contributed toward data analysis, drafting and critically revising the paper and agree to be accountable for all aspects of the work.

Disclosure of Conflict of Interest

The authors have no disclosures to declare.

Compliance with Ethical Standards

The work is compliant with ethical standards.

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