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Mechanical and Thermal Properties of Jute Fibre Reinforced Composites

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KEYWORDS	ABSTRACT			
Natural fibre Jute Alkali treatment Mechanical Thermal properties	Composite materials based on natural fibres, especially jute, have the potential to be used in engineering load bearing applications. Inherent flaws within natural fibres in terms of the presence of hydrophilic hydroxyl groups in their structure absorb atmospheric moisture which reduces the compatibility of these fibres with polymer matrices. Fibre surface modifications using chemical treatments have the potential to improve fibre-matrix compatibility. In this study, jute fibres were treated with different concentration of alkali (NaOH) solutions. The changes in fibre structure after treatment were investigated by Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA). Treated fibres were reinforced in polyester matrix to prepare composites by using hand lay-up method. The compatibility between fibre and matrix were analysed through mechanical property testing of the composites. Mechanical properties such as flexural and compressive strength, modulus and strain at break of composites were studied and compared. Best results were observed on compressive and flexural properties respectively for 5% and 7% NaOH treated samples. Thermal stability was also enhanced for the treated fibres. In general, thermal and mechanical properties of the treated fibres.			
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1. INTRODUCTION

Natural fibres have recently attracted the attention to be used as reinforcement in plastics. The attractive features of these fibres are in the ecological advantages, commercially available and higher specific mechanical properties. These fibres are environmentally friendly, biodegradable, renewable and provides the facility to be used as a green sources of reinforcing materials. Low density, high specific strength and modulus properties of these fibres have the potential to be used in engineering fibre composites for load bearing application with partial replacement of synthetic fibres [1].

Natural fibres such as jute, flax, coir, sisal etc are widely being used as reinforcing fibres in thermoset and thermoplastic matrix. Among these fibres, jute (*Corchorus capsularis*) is commercially available and inexpensive source of lignocellulosic fibre. The approximate chemical composition of this fibre is cellulose (51-84%), hemicellulose (12-20%), lignin (5-13%), pectin (0.2%) and wax (0.5%) [2]. Cellulose is considered as the major framework component of the fibre structure. It provides strength, stiffness and structural stability of the fibre. The cellulose structure of the fibres is distinguished through crystalline and amorphous regions, whereas hemicellulose and lignin are completely amorphous. In cellulose crystalline region, large numbers of strongly linked hydroxyl groups are present. These hydroxyl groups are inaccessible and so other chemicals have difficulty penetrating the crystalline region. However, in the amorphous region, the hydroxyl groups are loosely linked with the fibre structure and are relatively free to react with other chemicals [3]. Due to this freedom, hydroxyl groups present in the amorphous region can easily combine with water molecules from the atmosphere. The hydroxyl groups present in the amorphous hemicellulose and lignin initially give the access of water molecules to penetrate the fibre surface. Wa-

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ter molecules then combine with the hydroxyl groups that are present in cellulose (in the amorphous region) and stay in the fibre structure. This makes the fibre hydrophilic and polar in character. Figure 1 shows the schematic structure and the orientation of fibre constituents that absorb moisture. Hydrophilic fibres worsen the ability to develop adhesive characteristics with most hydrophobic binder materials during composite processing. As a result strong bonding between fibre and the binder materials are compromised which reduced the composites mechanical properties. This problem can be addressed by treating these fibres with suitable chemicals. Treatments can decrease the amorphous hydroxyl groups, remove the lignin and hemicellulose coverings from the fibre surface and expose the cellulose structure to react with binder materials. Different types of fibre surface treatments were used by several researchers by using diverse type of chemicals and methods. Among which alkali treatment is economically viable and more effective fibre surface treatment to improve composite properties. Cellulose, hemicellulose and lignin constituents of fibre are sensitive to the action of NaOH [4]. Treatment reduces hydroxyl groups that present in the amorphous region of fibre (Fibre-OH + NaOH \rightarrow Fibre-ONa + H₂O). Due to this hydrophilic nature of the fibre reduced. Alkali treatment also takes out a certain portion of hemicellulose and lignin covering materials. As a result, larger number of possible reaction sites of cellulose is available for matrix (binder) adhesion [5–6].

Yan et al. [7] and Dipa & Sarkar [8] reported that alkali treatment breaking down the fibre bundle by removing hemicellulose, lignin and waxy substances from the cellulose surface. This increases effective surface area of fibre and facilitate the cellulose hydroxyl groups to react with binder materials. Prasad et al. [9] used 5% NaOH for 76 hours and showed 40% higher flexural strength properties of treated coir-polyester composite compared to the untreated sample. Leonard et al. [10] used 0.16% NaOH treatment on hemp fibre for 48 hours and found treated fibre composites had 30% and 50% higher tensile and shear properties. Dipa et al. [11] used 5% NaOH treatment on jute fibres for 2 to 8 hours and showed 35% and 20% improved flexural strength and modulus respectively for the treatment of 4 hours. Viviana et al. [12] reported hemicellulose and lignin constituents were sensitive to higher temperature and removal of these constituents increased 4% higher thermal stability of hemp fibre after 8% NaOH treatment.

These studies have been focused on the different concen-



Cellulose

Figure 1. Schematic presentation of the orientation of fibre constituents that absorb moisture.

tration of alkali treatment on jute fibres. The treatment effect changes the content of cellulose, hemicellulose and lignin constituents of the fibre. Theses constituent changes can be detected by thermal (TGA and DSC) analysis. TGA aids in demonstrating the reaction mechanisms of fibres which occur at the molecular level of the fibre constituents as a function of progressive temperature. Fibre constituents degrades at different temperatures and results in weight loss. DSC allows a measurement of the chemical changes among the fibre constituents when subjected to heat. Untreated and treated jute fabric was reinforced in polyester matrix to prepare composites. The treatment effects on fibre-matrix interface bonding were investigated through mechanical property analysis. Experimental three point bending and compression tests were carried out in order to compare the stiffness and load-carrying capacity of these composites containing untreated and treated fibres.

2. EXPERIMENTAL

2.1. Constituent Materials

The woven jute fabric (jute gunny bag) was collected from the local market in Australia. T102 regular (paraffin) wax was used as a mold releasing agent. General purpose unsaturated polyester resin was used as matrix and methyl ethyl keton peroxide was utilised as a curing catalyst.

2.2. Fibre Surface Treatment

The alkali treatments used different concentrations of NaOH solution. The solution was prepared by adding 4%, 5%, and 7% NaOH (by weight) in a water ethanol mixture (water: ethanol = 80:20). It was then stirred at room temperature for an hour. Jute fabrics were soaked with different concentrations of NaOH solutions for 3 hours at room temperature. The fibres were then washed several times with distilled water to allow absorbed alkali to leach from the fibre. The washed fibres were dried at room temperature for 8 hours, and then oven dried at 100° C for another 6 hours. The dried fibres were stored in a sealed plastic bag to avoid atmospheric moisture contamination prior to thermal analysis and composite processing.

2.3. Composite Processing

Jute fabric reinforced polyester composite was prepared by hand lay-up process. In this process, the fibre-matrix mixing occurred in an open mould (a flat carbon steel sheet was used as a mould). The mould was waxed several times with a mould releasing agent. The first layer of jute fabric was laid over the mould and the polyester matrix (with 1.5% MEKP) was spread over the fibre carefully to ensure maximum fibre matrix wetting. A roller brush was used in the fibre-matrix mixing process. Extreme care was taken to achieve a uniform matrix distribution throughout the fibre cross-sections in the sample and removed any possible entrapped air in the matrix. Then the second layer of jute fabric was placed on the top of the first layer and above process was repeated. Afterwards, a waxed flat glass plate was placed on the top of the mat with a weight to ensure smother surface of the fabricated composites. The fibre content was 25% of the weight of the produced samples. The samples were kept at room temperature overnight then post cured at 80°C for 4 hours. The sample was released from the mould and kept in an oven at 80°C for 4 hours to post-cure. The cured panel ($400 \times 400 \text{ mm}^2$) was trimmed and sectioned for mechanical testing.

3. TEST METHODS

3.1. Differential Scanning Calorimetry (DSC)

DSC analysis on treated fibres was performed by using a thermal analyser (DSC Instrument, Model No. Q100). Fibre samples (between 6 to 10mg) were heated between the temperature ranges of 10°C to 450°C at a rate of 10°C/min. The experiment was conducted in a nitrogen environment purged at 20 ml/min. Nitrogen was used for efficient heat transfer and removal of volatiles from the sample. Each fibre sample was analysed separately and overlapped for comparison.

3.2. Thermogravimetric Analysis (TGA)

TGA analysis on treated fibres was carried out by a thermal gravimetric analyser (TGA-Model No. Q500). Between 6 mg to 10 mg of fibres was taken for analysis. Fibre samples were heated between the temperature range of 25°C to 500°C at a rate of 10°C/min. Experiments were carried out in a helium medium (60 ml/min) and the weight was recorded as a function of increasing temperature. The heating conditions were constant for all treated fibre samples.

3.3. Flexural Test

Flexural testing of the composites was conducted following ISO 14125 (1998) by using a 10 kN MTS testing machine at a loading rate of 2 mm/min. The specimens were simply supported and tested under 3-point loading with the span set at approximately 16 times the thickness of the specimen. Six specimens were tested for each sample. The test setup and instrumentation are presented in Figure 2. The load was applied at mid-span of the specimens, perpendicular to the fibre direction. Before each test, the loading pin was set to almost touch at the top surface of the specimen. To determine the strength and elastic properties in bending the load and mid-span deflection were recorded up to the specimen failure.



Figure 2. Flexural test setup of composite specimen.

3.4. Compression Test

Compression testing was carried out on the composites following ISO 14126 (1999) by using a 100 kN MTS machine with a loading rate of 1 mm/min. Six specimens were tested for each sample. The specimens were cut in to the dimension of $L \times W = 140$ mm × 12 mm and were clamped in a Wyoming Modified Celanese Compression test fixture. The load was applied in parallel to the fibre direction and the resulting deformation was recorded until failure.

4. RESULTS AND DISCUSSION

The thermal and mechanical properties of treated fibre and their composites were compared with the untreated samples.

4.1. DSC Analysis of Fibres

DSC analysis is used to determine the thermal energy released or absorbed via chemical reactions of the fibre constituents during heating. A series of exothermic and endothermic reactions take place during sample decomposition at different temperatures [13]. The magnitude and location of the exothermic and endothermic peaks indicate the thermal phase transformation of the fibre. In the case of an endothermic event, heat is absorbed by the sample, whereas heat is released in an exothermic event. Endothermic reactions provide information on sample melting, phase transitions, evaporation, dehydration and pyrolysis. Exothermic reactions provide information on crystallisation, oxidation, combustion, decomposition and chemical reactions [14].

From Figure 3 it can be observed that both untreated and treated fibres shows one broad endothermic peak between the temperatures of 10–150°C. This peak was corresponded to the evaporation of moisture absorbed by the fibre. From Table 1 it can be observed that, for untreated fibre moisture evaporation temperature (which was 77.07°C) was higher than that for the alkali treated fibres (79.18–85°C). This is

might be the case of treated fibres has less moisture content compared to the untreated fibres [15].

The region between 150-250°C shows no exothermic or endothermic changes which reflects fibres are thermally stable between these temperatures. It was reported that, lignin starts degrading at a temperature around 200°C. Therefore, hemicellulose and cellulose degrades at higher temperatures [16]. Hence the exothermic peak higher than 200°C is associated with the thermal degradation of lignin, hemicellulose and cellulose constituents. The first exothermic peak in Figure 3 for untreated fibre was observed at a temperature around 300°C, which was associated to the degradation of hemicellulose. For treated fibres, the heat required to generate these peaks were higher than the untreated sample indicated treatment removed hemicellulose from the fibre. Afterwards, there was a very strong endothermic peak for untreated fibre at about 368°C, however, treated fibres showed exothermic peak at this region (340-400°C). This indicated the degradation of lignin and cellulosic matters form the treated fibres. From Table 1 it can be observed that less temperature (350-360°C) were required for the treated fibres exothermic peak compared to the untreated fibre (368°C). This was due to the removal of lignin and cellulosic matters from the treated fibre with the effect of alkali treat-



		DSC Analysis (Thermal Degradation)			TGA Analysis (Thermal Degradation)			
Fibre Treatment		1st Stage (Moisture Evaporation) Tempt Range 10–150°C	2nd Stage (Hemicelluloses and Lignin) Tempt Range 280–320°C	3rd Stage (Cellulosic Components) Tempt Range 340–400°C	1st Stage (Moisture Evaporation) Weight (%)	2nd Stage (Hemicelluloses and Lignin) Weight (%)	3rd Stage (Cellulosic Components) Weight (%)	
								Untreated
NaOH treated	4%	84.07	307.99	361.04	5.55	18.32	75.59	
	5%	84.90	312.21	354.95	7.98	21.07	63.40	
	7%	79.17	290.80	350.40	6.75	28.91	61.02	

Table 1. Thermal Analysis of Jute Fibre.

ment. For untreated fibres these constituents were present and absorbed higher temperature to degrade. From this analysis it can be stated that, alkali treatment removed hemicellulose, lignin and cellulosic matters from the fibre surface. As a result fibres become more thermally stable, improved hydrophobic nature and facilitate better adhesion possibilities with the matrix.

4.2. TGA Analysis of Fibres

Thermo gravimetric analysis (TGA) was used to measure the weight loss of fibres as a function of rising temperature. The weight loss in fibres occurs due to the decomposition of cellulose, hemicellulose and lignin constituents during heating. Higher decomposition temperatures give greater thermal stability [17].

From Figure 4 it can be observed that, there are three stages of weight loss occurred as temperature increased. The first stage of weight loss was observed between $25-150^{\circ}$ C, which was correspondence to the release of moisture content by the fibre. The second stage of weight loss occurred within the temperature between $200-380^{\circ}$ C, which was related to the degradation of lignin and hemicellulose. The last stage of weight loss occurred between the temperature of $310-380^{\circ}$ C, indicated the degradation of cellulose and other cellulosic matters from the fibre [15].

From Figure 4 it can be observed that, untreated fibre degraded faster than the treated fibres in the second stage of fibre degradation process. From this result, it can be stated that hemicellulose and lignin constituents were partially removed from the treated fibres which enhanced thermal stability of fibre. This was evidenced by the degradation rate of the treated fibre with higher temperature and by lowering the weight loss compared to the untreated fibre. From Table 1 it can be observed that in every stages of weight loss (%) for treated fibre was less than the untreated fibre. These results also indicated that treatment reduced the moisture and constituent (hemicellulose and lignin) contents of fibre and exhibited greater thermal properties.

4.3. Flexural Properties of the Composites

Flexural properties of untreated and treated fibre composites are shown in Figure 5 and Table 2. During testing various load distribution mechanisms, such as tension, compression and shear between fibre and matrix were taken place simultaneously [18]. As a result, different failure modes were observed such as bending, shear and matrix cracking on the tested samples. From Table 2 it is observed that treated fibre composites has higher flexural strength properties compared to the untreated fibre composites. The flexural strength of the untreated fibre composites was 39.63 MPa, whereas for 4%, 5% and 7% NaOH treated fibre composites showed 18%, 32% and 31% higher strength properties respectively. Figure 5 presents stress-strain behaviour of the untreated and treated samples. Treated samples exhibited higher failure strain (2.66-3.81%) properties compared to the untreated samples (2.52%). The difference in flexural properties of

		Flexural Properties			Compressive Properties		
Type of Reinforcing Fibre		Flexural Strength (MPa)	Flexural Modulus (GPa)	Strain at Break (%)	Compressive Strength (MPa)	Compressive Modulus (GPa)	Strain at Break (%)
Untreated		39.63	1.56	2.52	56.09	0.75	7.42
NaOH treated	%	47.91	1.77	2.70	57.42	0.44	12.97
	%	57.16	1.49	3.81	69.01	0.88	7.77
	%	56.75	2.13	2.66	55.63	0.65	8.59

Table 2. Mechanical Properties of the Composites.



the composites supported well as the untreated fibre holds moisture due to the presence of hemicellulose, lignin and cellulosic matters in its structure. As a result, ineffective interfacial bonding between the fibre and matrix occurred and showed poor mechanical properties. On the other hand, due to the partial removal of hemicellulose, lignin and cellulosic matters from the fibre by the action of alkali treatment better bonding with the matrix achieved and showed improved mechanical properties. Several authors reported similar observations on improved flexural properties after alkali treatment of reinforcing fibres [19].

4.4. Compressive Properties of the Composites

Compressive properties of untreated and treated fibre composites are presented in Figure 6 and Table 2. From Table 2 it is observed that, 5% treated sample showed 19% and 15% higher compressive strength and modulus than the untreated samples. The stress-strain behavior in Figure 6 also exhibited higher failure strain properties for the treated composites compared to the untreated samples. 4%, 5% and 7% treated fibre composites showed 48%, 45% and 22%







Figure 6. Compressive properties of the composites.

higher strain compared to the untreated samples. These results indicate that better fibre matrix interface bonding was achieved in case of treated fibre composites. This was due to the removal of fibre surface coverings (hemicellulose and lignin) by NaOH treatments. The removal of the fibre surface coverings facilitated strong interfacial bonding with matrix materials. Strong interfaces prevented fibre buckling into the matrix, which ultimately delayed the formation of matrix cracks. Greater stress transfer from matrix to fibres took place and thus composite exhibited higher compression properties.

5. CONCLUSIONS

Following conclusions can be drawn from the above results and discussion:

- 1. Jute fibre surface was modified by the different concentration of alkali treatment. This treatment removed hemicellulose, lignin and cellulosic constituents from the fibre. As a result, hydrophilic tendency of fibres were reduced and also exhibited higher thermal stability. This provides better interfacial adhesion between the fibre and matrix.
- 2. Alkali treated fibre composites had higher flexural properties compared to the untreated samples and the properties became greater at higher treatment concentrations (5-7%NaOH). These results indicate treatment reduced moisture from the fibre by removing hemicellulose, lignin and cellulosic constituents and thus improved compatibility with the matrix which resulted superior mechanical properties.
- Compressive properties of treated composite samples showed higher failure strain properties. The higher properties indicate greater adhesion between the treated fibre and matrix were achieved which allowed the sample to take larger deflection before failure.

From the above discussion, it can be stated that alkali treatment on jute fibres achieved some degree of success in

improving thermal and mechanical properties of the fibre and composites. This method of fibre modification will help to develop natural fibre composites with better performance under load bearing conditions.

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