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Static and Dynamic Mechanical Properties of Cotton/Epoxy Green Composites

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Abstract
A study on the effect of alkaline treatment on the mechanical properties of cotton fabric reinforced epoxy composites is presented in this paper. One hour treatment of cotton fabric was performed using three different concentrations of sodium hydroxide (NaOH) solution. 1% NaOH treated fabric reinforced composites exhibited maximum improvement in tensile strength. It was concluded that the said NaOH concentration improves interfacial adhesion between the cotton fabric and epoxy resin. Moreover the morphology of the fracture surface, evaluated by scanning electron microscopy (SEM), indicated that surface treatment can yield better adhesion between the fabric and matrix, demonstrating the effectiveness of the treatment. The dynamic mechanical analysis (DMA) results revealed that alkali treated (1% and 3% NaOH) fabric composites exhibit higher storage moduli and glass transition temperature (Tg) values as compared to the untreated fabric composites. However, for all the composite specimens, the storage modulus decreased with increasing temperature (25 - 100 °C). Tg values of 50.9, 56.7, 52.8 and 37.7 °C were recorded for the untreated and (1%, 3% and 5%) treated composites, respectively. The tan δ values decreased for all the composites with increasing temperature, indicating enhanced interactions between the polymer matrix and fabric reinforcement.

Key words: *natural fibres, adhesion, alkaline, composites, wettability, glass transition temperature.*

Introduction

Composite material is a useful combination of at least two materials, usually termed the matrix and reinforcement, with the aim of obtaining properties that cannot be achieved using the constituent materials separately. Research and development of natural fibre composites and their possible application areas have recently increased. Natural fibre-reinforced polymer composites are attracting the attention of many industrial applications because of their environmental and economic advantages. These benefits have also led to increased developments in the production of bio-composites [1, 2]. Bio-composites refer to composites that combine natural fibres, such as kenaf, jute, hemp and sisal, with either biodegradable or non-biodegradable polymers [3 - 5].

Cotton is the most popular natural fibre in the world, having a global trade of US$ 67.9 Billion (cotton fibre, yarn and woven fabrics, excluding apparel). In addition to clothing, cotton is used in manufacturing particularly for engineering and industrial uses of textiles, such as geotextiles, filters and composites. Among these technical textile applications, cotton fibre reinforced composites have recently attracted increasing attention because they offer advantages such as biodegradability, good physical properties and low cost as compared with manmade or inorganic fibres used in composites [6].

Unfortunately natural fibres are not suitable for certain applications due to some drawbacks, such as thermal and mechanical degradation during processing. Natural fibre reinforced composites also have several drawbacks such as poor wettability, incompatibility with some polymeric matrices and high moisture absorption. The main problem often encountered in their use is fibre - matrix adhesion [7]. Several methods have been adopted to address the issues related to natural fibre reinforced composites [8]. One is to modify the natural fibre surface such as the: graft copolymerisation of monomers onto the fibre surface or use maleic anhydride copolymers, alkyl succinic anhydride or stearic acid. It is also known that the use of coupling agents such as silanes, titanates, zirconates, and triazine compounds, improves fibre – matrix adhesion.

The use of fluorocarbons, hydrocarbons and hybrid fluorocarbons for the treatment of jute fibres also helps to increase the fibre – matrix interface by decreasing the moisture regain [4]. Alkali treatment is a common method to clean and modify a fibre surface to reduce surface tension and enhance the interfacial adhesion between a natural fibre and polymeric matrix [9]. It is also termed the mercerisation of fabric, and leads to fibrillation, which causes the breaking down of the composite fibre bundle into smaller fibres [10]. Mercerization reduces the fibre diameter, thereby increasing the aspect ratio, which leads to the development of

Table 1. Properties of the composite plates

Sample code	C1	C2	C3	C4
Reinforcement treatment	Untreated	1% NaOH	3% NaOH	5% NaOH
Fabric ply number	4			
Stacking direction	0°/90°			
Plate thicknesses, mm	3			
Fibre volume fraction, %	45			

a rough surface topography that results in better fibre matrix interface adhesion and an increase in mechanical properties [11].

Mercerization causes the removal of lignin and hemicellulose from plant fibres, and affects the chemical composition of plant fibres, the molecular orientation of cellulose crystallites and the degree of polymerisation. The cellulose–sodium hydroxide reaction is thought to be as follows [12]:

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

Extreme care must be taken when bleaching and/or mercerising cellulose with sodium hydroxide as when it is used in large quantities it tends to texturise the cellulose structure, thus affecting its mechanical properties [13]. Several publications have discussed the effects of alkali treatment on the structure and properties of other natural fibres such as jute fabrics [14], flax [15], sisal [16], sisal jute, and kapok fibres [17].

The review of literature indicates that researchers have focussed on the investigation of tensile, flexural and impact properties of treated natural fibres and their subsequent composites. Cotton is the most abundantly produced natural fibre, and is widely used across the globe. Generally it is preferred for apparel, finding limited use as reinforcement in composite materials. No significant work has been reported on the use of cotton fibre as reinforcement nor on its surface treatments or performance in composite material. The aim of this research was to effectively use this natural resource as a reinforcement for composite materials, and address the related issues. This study focussed on studying the effect of surface (alkaline) treatment on the static and dynamic mechanical propertıes of cotton/epoxy green composıtes. Using sodium hydroxide (NaOH) for fabric treatment is expected to improve the surface characteristics of cotton fabric for use with epoxy matrix. The effect of NaOH treatment on cotton fabric morphology was investigated using scanning electron microscopy (SEM). The mechanical properties of treated and untreated cotton fibre reinforced composites was investigated by tensile testing and dynamic mechanical analysis (DMA) techniques.

Material and methods

Materials

The fabric used was 100% cotton with an areal density of 196 g/m^2, 22 ends/cm and 25 picks/cm. The cotton fabric was supplied by Arsan Textile Co., Ltd (Turkey). Purpox® epoxy resin EFLR-0190 provided by Polikor Inc (Turkey). was used as matrix material, which is a solvent free resin with a transparent coating. The density of this resin was 1.00 – 1.10 g/cm³, the while viscosity was 300,500 mPa·s. The epoxy resin and EFLR-0190 hardener were mixed at a weight ratio of 100:50 to produce the composite materials.

Methods

Alkali treatment

Alkali treatment was conducted using three concentrations (1%, 3% and 5%) of NaOH solution. The cotton fabrics were placed in NaOH solution at various concentrations for one hour at room temperature. The fabrics were rinsed with distilled water until the NaOH was removed and the rinsed solution reached a neutral condition (pH 7). The fabrics were then oven-dried at 60 °C for 45 min. The alkali treated fabrics were used as reinforcement for the fabrication of composites.

Composite preparation

The fabrics used in the production of the composite materials were cut into 30 × 30 cm pieces. The end (ends/cm) and pick count (picks/cm) of the fabrics could differ from one another. Thus the fabrics were slightly unbalanced with respect to the total fibre volume content in the warp and weft directions. To balance the warp- and weft-direction fibres in the composite laminate, the fabric layer orientation was alternated when laying the half-ply of samples.

Table 1 shows the main production parameters of the composite samples, such as the ply arrangement, number of fabric plies and thickness. The hand lay-up technique was used to produce the composite plates. All samples were produced on a glass plate. The samples were held for a minimum of 12 hours after resin infusion and post-cured at 80 ºC for 4 hours in an oven.

The thicknesses of the finished samples were measured using a calliper. The fibre volume fraction (V_f) was obtained based on the fabric weight and plate thickness as follows:

$$V_f = \frac{n \times m}{r \times h} \quad (1)$$

where, n is the number of fabric plies, m the areal fabric weight, ρ the fibre density and h is the plate thickness.

Measurements

Tensile testing

Tensile tests were performed on composite sheets according to ASTM standard D-3039. The samples, which were 197 mm long by 25 mm wide by 3 mm thick, were tested using an Instron 4411 (UK) (5 kN) universal tensile testing machine. The tensile strength testing was repeated five times for each composite and the average value of results reported.

Dynamic mechanical analysis

Dynamic mechanical analysis tests of the composites were performed using an Acoem Metravib-DMA1000+. The Young’s modulus was determined experimentally from the slope of a stress - strain curve created during tensile tests conducted on a sample of the composite material.

SEM observation

To observe the fractured surface morphology of the composites, scanning electron microscopy was performed. The fractured surfaces of the composite samples were coated with gold using plasma sputtering apparatus. These were then observed under SEM using a ZEISS EVO 40 (ZEISS Co., Oberkochen, Germany) operated at 10 - 20 kV.

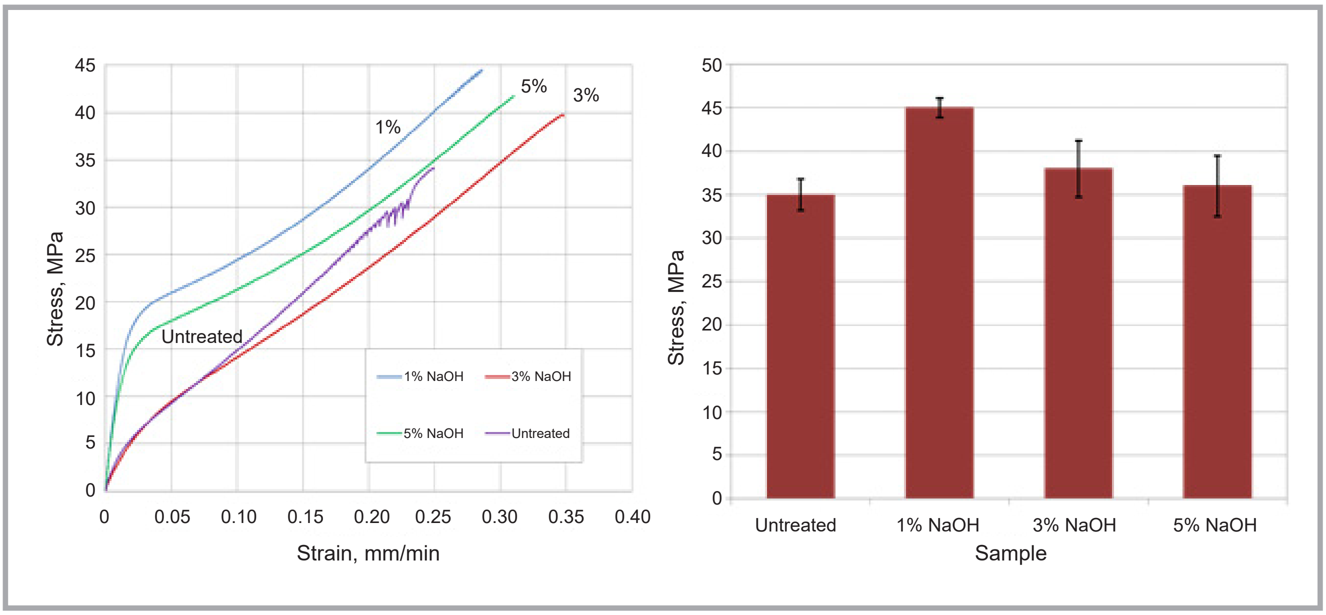

Figure 1. *Stress-Strain behaviour (a) and average tensile strength of untreated and alkali treated cotton fabric reinforced epoxy composites (b).*

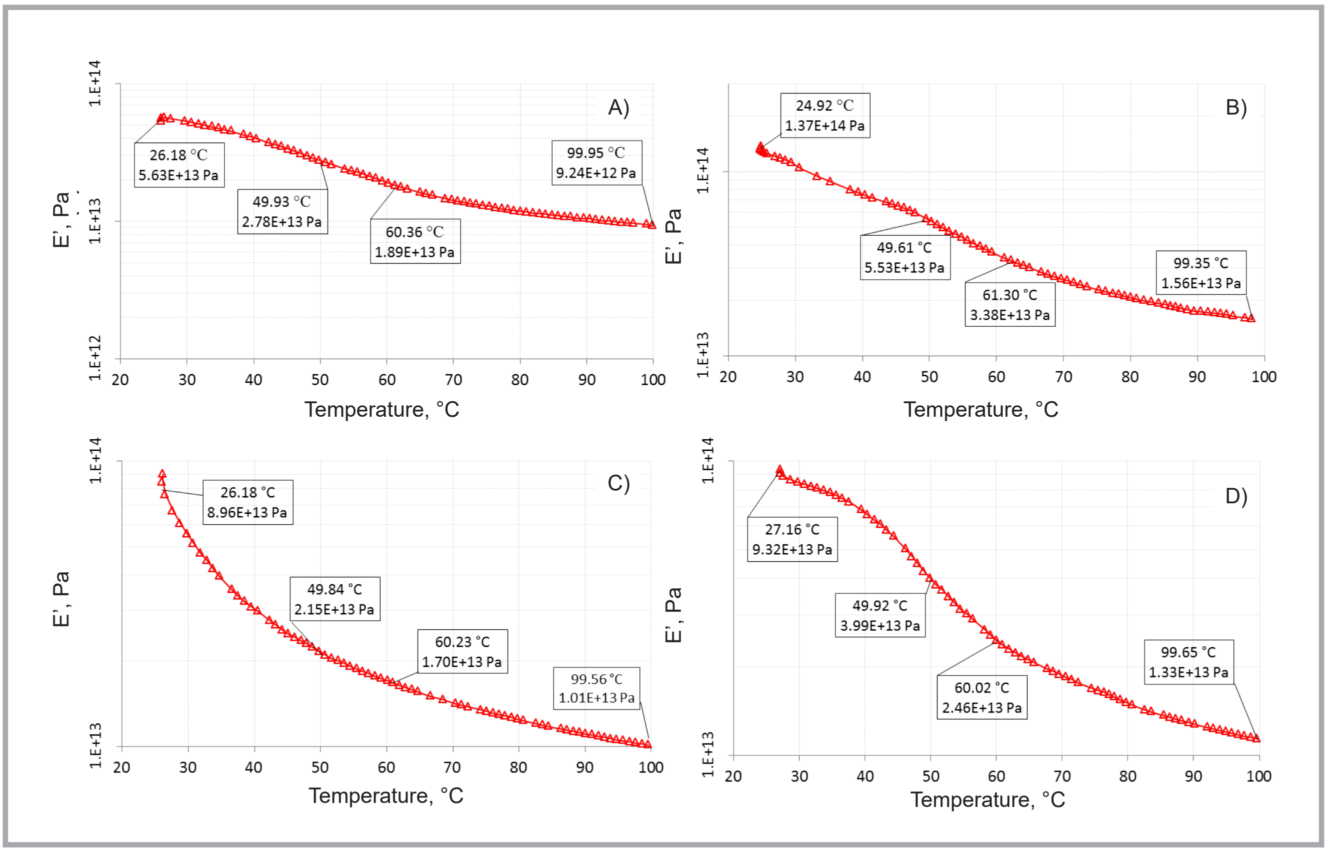

Figure 2. *Variation in storage modulus of untreated and treated cotton fabric-epoxy composites as a function of temperature. (A) Untreated, (B) 1% NaOH treated, (C) 3% NaOH treated and (D) 5%NaOH treated.*

Results and Discussion

Tensile testing results

The tensile strength of the untreated and alkali treated fabric reinforced composites was measured, the results of which are presented in ***Figure 1***. The 1% NaOH treated fabrics exhibited higher tensile strength of 45.94 MPa as compared to the 3% & 5% NaOH treated and untreated fabrics. In addition, the tensile strength of the untreated fabric (34.55 MPa) was lower than that of the alkali treated fabrics, as shown in ***Figure 1***. An increase in the alkali concentration was expected to have a positive effect on the tensile properties of the composite; however, a very high concentration of alkali solution would certainly damage the fabric and consequently reduce the tensile strength of the fabric and its composites [7, 14].

The change in the tensile strength (increase and decrease) of the treated fabric

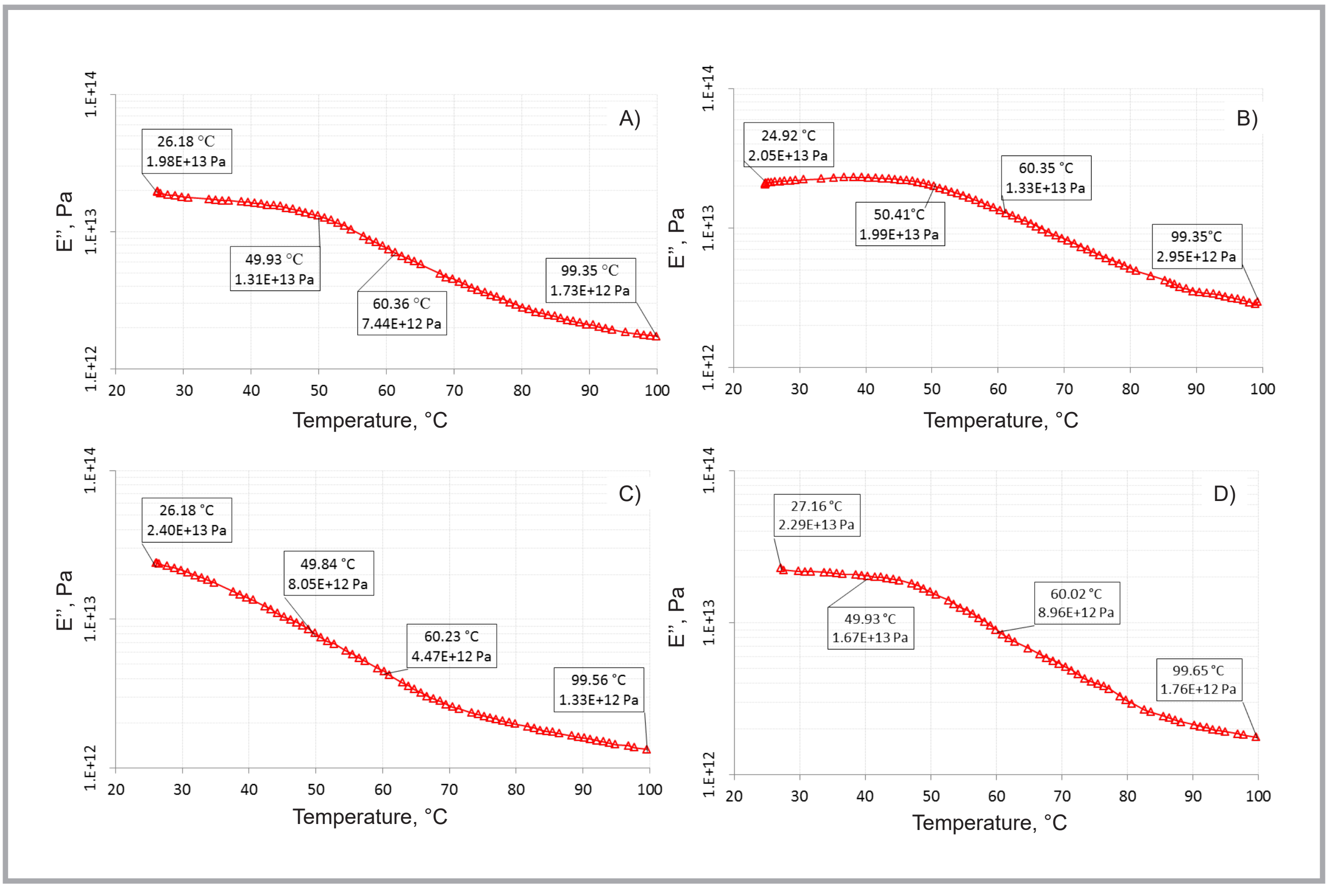

***Figure 3.** Variation in loss modulus of untreated and treated cotton fabric-epoxy composites as a function of temperature. (A) untreated, (B) 1% NaOH treated, (C) 3% NaOH treated and (D) 5%NaOH treated.*

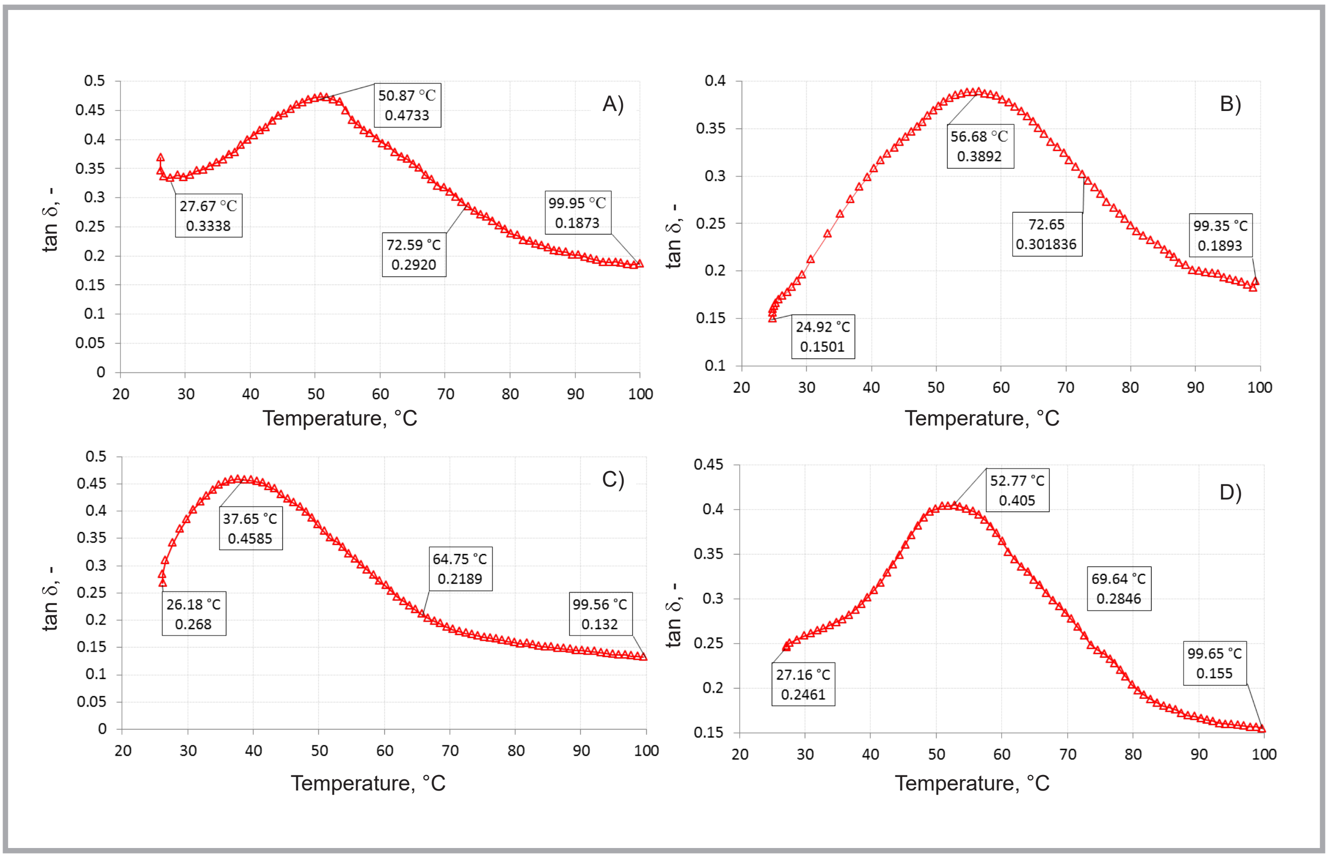

***Figure 4.** Variation in tan δ untreated and treated cotton fabric-epoxy composites as a function of temperature. A) untreated, B) 1% NaOH treated, C) 3% NaOH treated and D) 5%NaOH treated.*

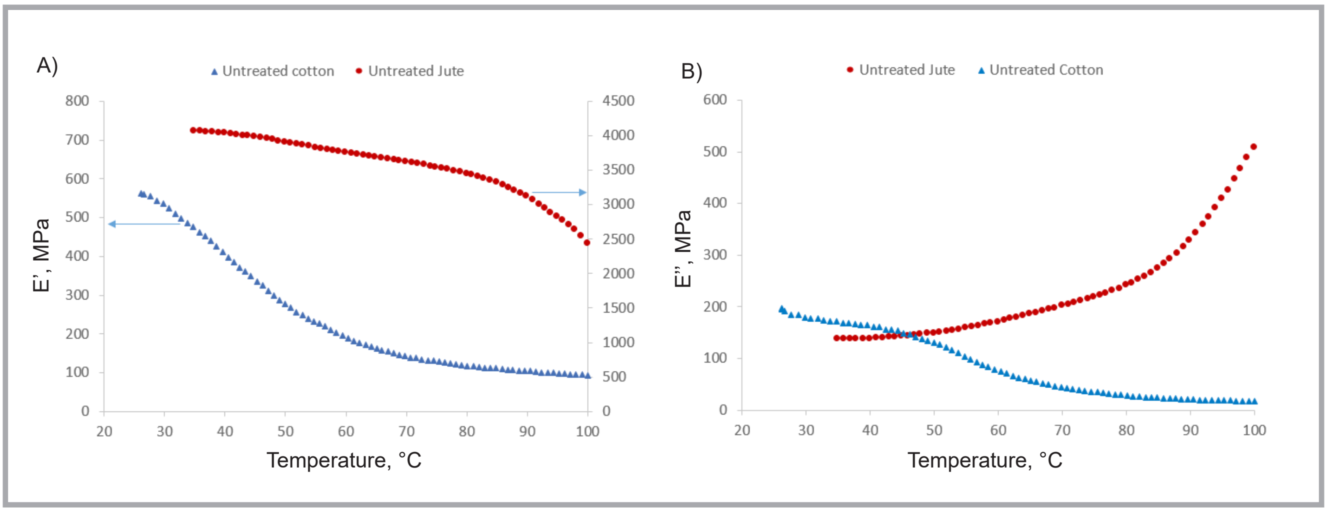

***Figure 5.** Comparison of the (a) storage modulus and (b) loss modulus of cotton and jute/epoxy [26] composites.*

composites may be due to the improved adhesion interface between the fabric and matrix or damage to the fabric caused by alkali treatment. The enhancement of the tensile strength of the alkali treated (1% NaOH concentration with 1 h soaking time) fabric composites is attributed to the improved wetting of the alkali treated fabric with the matrix [18]. The alkali treatment removes some of the hemicellulose, lignin and wax covering from the external surface of the fabric and a rougher topography is attained. Thus the mechanical interlocking of fabric in the matrix and interface quality are enhanced [19]. In addition, the hemicellulose and lignin contents decreased in this system after alkali treatment, thereby increasing the effectiveness of the orientated cellulose fibres. Alkali treatment improved the dispersion of fabric in the matrix, which resulted in an increase in the fabric aspect ratio. This increase resulted in an increase in the reinforcement effectiveness of the fabric, and hence increased the strength of the composites [7]. Because the size of the fibre in the matrix decreased, the extent of stress concentration transferred from the fibre also decreased, leading to an increase in strength. However, the contact area of the fibre with the matrix increases with increased interaction between the fibre and matrix [20].

Tensile curves of the samples are given in ***Figure 1.a***. The stress-strain curves are highly similar for treated samples. But for the untreated sample, there are some oscillations near the breaking stress on the curve, indicating abrupt changes in stress, since there is shear between the fibre and matrix due to wax on the external surface of the fibres for untreated samples.

The tensile strength of cotton/epoxy composites is significantly lower than for the flax/epoxy and jute/epoxy composites. Compared with those of the flax/epoxy [3] and jute/epoxy composites, the tensile strength of the cotton-epoxy composites was 34% and 59% lower, respectively, for similar fibre volume fractions.

Dynamic mechanical analysis test results

DMA is an analytical technique in which an oscillating stress is applied to a sample, and the resultant strain is measured as a function of both the oscillatory frequency and temperature [21]. The technique separates the dynamic modulus *(E)* of materials into two distinct parts: an elastic (storage modulus) component *(E')* and viscous (loss modulus) component *(E")*. *E'* is the component of the dynamic modulus, where the strain is in phase with the applied stress, and *E"* is the component of the dynamic modulus, and where the strain is 90º out of phase with the applied stress. The ratio of *E"* to *E'* gives the tangent of the phase angle δ, and tan δ is known as the damping and is a measure of energy dissipation. These parameters provide quantitative and qualitative information about the material behaviour [22].

The variation in the storage modulus of the untreated and treated composites as a function of temperature is shown in ***Figure 2*** (see page107). It is evident from the ***Figure 2*** that the composites reinforced with surface treated fabrics exhibit higher storage moduli than those reinforced with untreated fabric. This finding indicates that the adhesion between the epoxy matrix and cotton fabric is improved with alkali treatment. The composite with alkali treatment exhibits the highest storage modulus, which suggests that alkali treatment is the best method to improve interfacial adhesion between the epoxy matrix and fabric. İn addition, the storage modulus of all the samples decreased with increasing temperature, and a significant decrease occurs in the regions between 25 and 100 °C. However, the softening temperature with surface treatment is higher than that of the epoxy, which might be due to the interactions resulting in a decrease in chain mobility and a regular reinforcing effect [23].

Loss moduli of the untreated and treated cotton fabric-epoxy composites are presented in ***Figure 3***. The *E"* value corresponding to T_g was lowest for the untreated composites but increased slightly for the treated composites (for 1%, 3% and 5% alkali treated composites, respectively). A similar increase in T_g and the loss modulus due to alkali treatment of the fibres has been reported by other researchers [24, 25]. The maximum heat dissipation occurred at a temperature where the loss modulus is maximum, indicating T_g of the system. The T_g values of composites treated by NaOH increased to higher temperatures. These results can be explained based on the retardation of the relaxation of amorphous regions due to physical interaction with the reinforcing phase and crystalline regions of the epoxy matrix [26].

The variation in tan δ as a function of the temperature is shown in ***Figure 4***. The tan δ values were higher in the (1%,

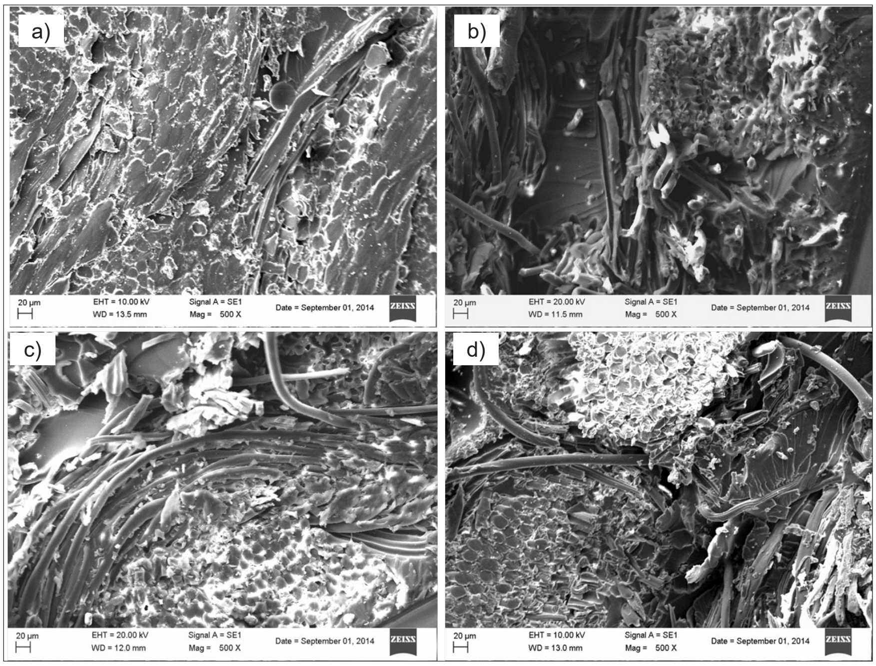

Figure 6. *SEM micrographs of cotton/epoxy composite: a) untreated composite, b) 1% NaOH, c) 3% NaOH, d) 5% NaOH treated composite.*

and 3%) treated composites compared with the untreated and 5% treated ones. The T_g values were 50.9 °C, 56.7 °C, 52.8 °C and 37.7 °C for the untreated and (1%, 3% and 5%) treated composites, respectively. The increase in T_g is associated with the decreased mobility of the matrix chains, which indicates enhanced interfacial adhesion between the fabric and epoxy matrix. However, T_g of the 5% NaOH treated composite decreased significantly. This result could be due to the mobilisation of polymer molecules near the surface of the cotton fibres resulting from various molecular interactions, which decreased T_g of the composites [27, 28]. The SD values were 1.07, 0.90, 0.85 and 0.87 for the untreated and treated composites. respectively.

A comparison of the DMA results of cotton epoxy composites to those of the Jute /epoxy composites [29] is shown in ***Figure 5*** (see page109). From the results it can be seen that the storage modulus of the cotton epoxy composites is less as compared to the jute epoxy composites. Further to this, the increase in temperature caused a reduction in the storage modulus of both cotton and jute composites. In cotton epoxy composites the decrease is significant in the temperature range of 30 - 60 °C, while the jute composite shows a decrease in the storage modulus at a higher temperature. İn the case of the loss modulus, both composites behave differently. The loss modulus of cotton epoxy composites decreases with the temperature, while that of the jute epoxy composite increases with the temperature. As the loss modulus measures the energy dissipated as heat, hence it is supposed to dissipate more energy as heat at elevated temperatures under dynamic loading.

SEM observation

SEM analysis was used for direct observation of the failure of the composite surface and the resin-fabric interface. SEM micrographs of the fracture surface of the treated and untreated cotton/epoxy composites are presented in ***Figure 6***. ***Figure 6.a*** shows the fracture of the untreated cotton fabric/epoxy composite with many pull-outs. The surface of the untreated fibre is smooth and there is no evidence of any matrix resin adhering to the cotton fabric. The fibre pull-out and clean fibre surface in the micrograph are indications of low fabric/matrix adhesion. The low fabric-matrix adhesion explains the poor mechanical properties in the composite reinforced with untreated fabrics. ***Figures 6.b, 6.c*** and ***6.d*** also reveal fairly clean fabric surfaces and that the fibre pull-out was relatively smaller. The 1% NaOH treated fabric composites exhibited the maximum improvement in tensile strength, compared with the 3% NaOH and 5% NaOH treated composites. Based on the SEM analysis, significantly good bonding occurred between the fabric and matrix. Apparently the surface of the fabric was not rougher than that of the untreated fabric composite sample, but it is thought that NaOH treatment removed the wax from the fibre external surface, which may be attributed to the enhancement of the bonding strength between the fabric and matrix.

The cotton fabric reinforced epoxy composite fractured after tensile loading for one hour mainly because of the dissolution of hemicellulose, which increased the interfacial bonding between the fabric and matrix. Consequently an increase in the surface area occurs, which results in increased adhesion at the fabric-matrix interface in the composites. Comparing the treated and untreated fabrics, the maximum improvement in the tensile strength was observed to be approximately 28.57%. This change in the tensile strength is attributed to interacting factors such as the rupture of alkali-sensitive bonds existing between the cellulose and hemicellulose (because of the removal of hemicellulose making the fibre more homogeneous) and the stress transfer between interfibrillar regions [30]. For the untreated fibres, hemicellulose remained dispersed in the interfibrillar region separating the cellulose chains from one another, and because of this barrier, these chains were in a state of strain. The fibres tended to be closely packed because of the large scale removal of hemicellulose by alkali treatment and the formation of new hydrogen bonds between the chains of cellulose fibrils. Consequently the fibrils rearrange themselves in a more compact manner, resulting in closer fibre packing [31].

Conclusion

This study focussed on the development of cotton/epoxy composites by treating fabric with 1%, 3% and 5% NaOH solutions for 1 hour. It was observed that the tensile strength of the composites increased after alkali treatment because of the improved fabric structure. The highest tensile strength was observed for 1% NaOH solution treatment and may be attributed to the large amount of hemicellulose dissolution. The hydrophilic nature of cotton fabric was reduced because alkali treatment increased interfacial bonding between the matrix and fabric. SEM micrographs of the fractured surface revealed that fibres became finer after alkali treatment due to the dissolution of hemicellulose and increased aspect ratio, which resulted in better fibre-matrix adhesion. The dynamic mechanical analyser (DMA) showed an increase in the T_g values after alkali treatment (1% NaOH) from 50.9 °C to 56.7 °C. The composites reinforced with surface treated fabrics exhibit higher storage moduli than those reinforced with untreated fabric. In addition, the storage modulus of all the samples decreased with increasing temperature. A similar trend was shown by the loss modulus of the untreated and treated composites. Consequently this study demonstrates that cotton fabrics can be favourably considered as a reinforcement in green composites.

Acknowledgements

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