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Effect of alkaline treatment on mechanical properties of voile fabric reinforced epoxy composites

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Effect of alkali treatment on the mechanical properties of epoxy composites reinforced with alkali-treated voile fabric has been studied. The voile fabric is treated with different concentrations of NaOH solution (1% and 3%) for 1 h at 20 ± 2 °C. The epoxy-based composite obtained by reinforcing the alkali-treated fabric is evaluated for its tensile strength, and dynamic mechanical properties. Composite reinforced with 3% NaOH solution treated fabric shows significant improvement in tensile strength (~32.72%). This implies that the concentration of NaOH treatment greatly influences the interfacial adhesion between the voile fabric and the epoxy. Results of dynamic mechanical analysis show an increase in the storage moduli of the composites reinforced with 1% and 3% NaOH treated fabric as compared to untreated composites at 20 -100 °C. However, for all the composites, the storage modulus decreases with the increase in temperature with a significant fall in the temperature range 25 - 100 °C, indicating that the incorporation of voile fabric in epoxy matrix unconcluded reinforcing effects appreciably at higher temperatures. The loss moduli of untreated and treated composites decrease with the increase in temperature. The glass transition temperatures of the untreated and treated (1% and 3% NaOH) composites are found to be 35.36 °C, 35.81 °C, and 30.55 °C respectively. For all the composites, the value of tan δ decreases with the increase in temperature which indicates the level of interactions between the polymer matrix and the reinforced fabric. The fractured surface of composites is observed using SEM, which indicates the surface modification of the voile fabrics with alkali treatment and subsequent improvement in fibre-matrix adhesion.

Keywords: Epoxy composite, Fabric reinforced composites, Mechanical properties, Surface treatments, Voile fabric

1 Introduction

Naturally occurring cellulosic fibre, such as henequen, sisal, coconut, jute, palm, bamboo, wood, and paper as well as cellulosic waste products (shell flour, wood flour and pulp) have been used as reinforcement agents in various thermosetting and thermoplastic resins[1]. In the recent years, significant research efforts have been devoted towards the development of natural fibres composites and their potential application in diverse fields. The versatile applications of the fibres reinforced composite materials have made them an integral part of the textile world[2,3]. In general, natural fibres offer several advantages over traditional reinforcing materials including acceptable specific strength low cost, low density, good thermal properties, enhanced energy recovery and biodegradability[4].

However, natural fibres are associated with certain shortcomings, such as thermal and mechanical degradation during processing making them undesirable for certain applications. Besides, natural fibre reinforced composites also have several drawbacks such as poor wettability, incompatibility with certain polymeric matrices and high moisture absorption by the fibres. One of the main issues associated with the use of these composites is the fibre-matrix compatibility.

However, this problem could be circumvented via chemical treatment of the fibre surfaces. In principle, the performance of fibre-reinforced composite is greatly restricted by the properties of its constituents. Besides, it is essential to ensure good compatibility and bonding between the fibre and the matrix[5]. To this end, several studies have focused on alkali treatment of natural fibres to improve the bonding between the fibre and the resin matrix with subsequent improvement in their properties[6]. Among the various surface treatments, recently coupling methods, wherein chemical groups of coupling agents react with both the fibre and the matrix, have attracted significant interest[7]. Alkali treatment is a common method to clean and modify the fibre surface in order to lower the surface tension and enhance the interfacial adhesion between the natural fibre and the

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polymeric matrix[5]. So far, several studies have discussed the effects of alkali treatment on the structure and properties of natural fibres, such as jute fabrics[8] flax[9], natural fibre[10], and sisal[11].

Epoxies are the most commonly studied thermoset materials that offer a very wide range of industrial applications. Owing to their high specific stiffness and strength, epoxies are extensively used as matrix materials in glass reinforced epoxy composite materials and as adhesives in a multitude of applications, including aerospace, defence, automotive, marine and sporting goods. The unique advantages of these materials, such as high durability, design flexibility and lightweight make them attractive materials in these applications[12].

The present study investigates the effects of alkaline treatment on the mechanical properties of voile fabric reinforced epoxy composites. Furthermore, the effects of surface treatment of the fibres on the interfacial properties of the composite have been analysed by using scanning electron microscopy.

2 Materials and Methods

2.1 Materials

Cotton fabric, having the specifications: plain weave structure, 12 ends/cm, and 27 picks/cm, and fabric weight ratio 34 g/m^2, was used for the study. Epoxy resin was obtained from Epoxy Center Co., Ltd, Istanbul, Turkey and cotton fabrics were purchased from Arifoglu Textile Co., Ltd and Arsan Textile Co., Ltd, Adana and K.Maraş, Turkey. All chemicals used in this study were of laboratory grade, and deionized water was used in all experiments.

2.2 Methods

2.2.1 Alkali Treatments

In this study, alkali treatment of the voile fabric was performed in two different concentrations (1%, and 3%) of NaOH solution for 1 h at 20 ± 2 °C. Following the treatment, the cotton fabrics were rinsed with distilled water to ensure the removal of NaOH. The rinsing process was performed till the *p*H of the rinsed solution became neutral (*p*H 7). Subsequently, the cotton fabrics were oven-dried at 60 °C for 45 min. Alkali-treated fabrics thus obtained were used for the preparation of composite.

2.2.2 Composite Preparation

The composite was manufactured by using hand lay-up technique. The mould was made of wood and teflon by using three layers of double-sided tape to form a square shaped mould. Initially, appropriate quantities of epoxy and hardener were mixed together based on the weight percentage to form a matrix. Following that the matrix was poured over the fabric, compressed and distributed evenly to obtain the desired dimension (3.0 mm thick, 197 mm long and 25 mm wide) in accordance to the ASTM D 3039 standard[8,5]. Subsequently, the laminates were pressed in the mould at 20 ± 2 °C for 24 h. Finally, the cotton fabric/epoxy composites were cured at room temperature.

2.2.3 Mechanical Properties of Composite Specimens

The dimensions of the specimens used for tensile testing were adapted from ASTM D 3039 standards. Tensile strength tests were performed on both treated and untreated cotton fabric/epoxy composites. Three specimens were tested for each type of composite. Tensile tests were performed at room temperature (25± 2 °C) and 65±5% humidity using an Instron [4411 (kN)] model testing machine.

2.2.4 Dynamic Mechanical Analysis

Rectangular specimens having a size of 35mm × 15.11mm × 4.7 mm were used for the dynamic mechanical analysis (DMA). The tests were performed using a three-point bending system in the temperature range 20 – 100 °C at a heating rate of 1K/min and at frequency range 0.1-200 Hz.

DMA is a widely accepted method for measuring temperature dependence properties such as storage modulus (E'), loss modulus (E'') and damping factor ($\tan \delta$). Dynamic mechanical properties of the treated and untreated composites were measured by using Acoem Metravib - DMA1000+ dynamic analyser in the tension-torsion mode. At least three samples were tested and the results were averaged.

2.2.5 Scanning Electron Microscopic Study

Analysis of the fractured surfaces of the samples can be used to illustrate the effect of different fibre surface treatments on the effective properties of the composite. To this end, the fractured surfaces of the composites were examined by using scanning electron microscope (ZEISS EVO, ZEISS Co., Oberkochen, Germany), operated at 10 - 20kV.

3 Results and Discussion

3.1 Tensile Strength Testing

It is observed that the average tensile strengths of the untreated and treated (1% and 3% NaOH) composites is observed as 17.56 MPa, 22.17 MPa, and

23.31 MPa respectively. The results indicate that the tensile strength increases for the exposure time of 1h with alkali treatment. However, with increase in alkali concentration, the tensile strength increases slightly from 22.17 MPa to 23.31 MPa. The observed enhancement in tensile strength of the alkali-treated fabric composites can be attributed to the improved wetting of alkali, treated fabric with the matrix. It can be noted that the alkali treatment at NaOH concentration of 3% for 1 h greatly improves the resin pick-up or wettability of fibres, which results in 32.72% rise in the tensile strength of composites. It is believed that this alkali treatment results in an improvement in the interfacial bonding by offering additional sites of mechanical interlocking, thereby promoting more resin/fibre interpenetration at the interface[1, 13, 16].

Increasing the alkali concentration is expected to have a positive influence on the tensile strength of the voile/epoxy composite. However, in the present study, an increase in alkali concentration does not show a significant improvement in tensile strength. This could be due to the damage of fibre structure at higher alkali concentration, as reported earlier[5, 4, 17,18].

As mentioned earlier for 3% NaOH treatment, the tensile strength increases for the exposure time of 1h, as compared to others. This could be attributed to the synergistic effect of the improvement in interfacial adhesion and the damage of fibre as a result of alkali treatment.

3.2 Dynamic Mechanical Analysis

Dynamic mechanical analysis can be used to determine the response of a given material to cyclic deformation as a function of temperature. Typically. DMA results are expressed by the following three main parameters, viz (i) storage modulus (E'), corresponding to the elastic response of the deformation, (ii) the loss modulus (E''), corresponding to the plastic response of the deformation, and (iii) tan δ which is E''/E', that can be used for determining the occurrence of molecular mobility transitions such as the glass transition temperature[19]. The ratio of these parameters is denoted as loss factor (tan δ), which is used to determine the glass transition temperature[20]. Figure 1 shows the variations in storage modulus of untreated and treated voile fabric-epoxy composites as a function of temperature. The storage moduli (E') of the untreated and treated (1% and 3% NaOH) composites are found to be 1.63e8Pa. 1.9e8Pa and 1.75e8Pa respectively at 60 °C. All the samples show high storage modulus at room temperature followed by significant drops in the temperature range 25 ° - 100 °C. The higher E' values of the treated composites is due to greater interfacial adhesion and bond strength between matrix resin and fibre as reported in previous studies[21, 22]. Upon heating from 20 °C to 100 °C there is a decrease in storage modulus (E'). Similarly, Fig. 2 shows the variation in loss modulus of the untreated and treated composites with temperature. The untreated and treated composites show the loss modulus peak at 25 - 26 °C, which could be attributed to the mobility of resin molecules[23]. The ratio of these parameters is denoted as loss factor (tan δ), which is used to determine the glass transition temperature. Figure 3 shows the variation in tan δ with temperature for untreated and treated composites in the frequency range 0.1-200 Hz. The tan δ value of 3% NaOH treated composite (0.5403 at 35.81 °C) is higher than that of the untreated (0.5656 at 35.36 °C) and 1% NaOH treated

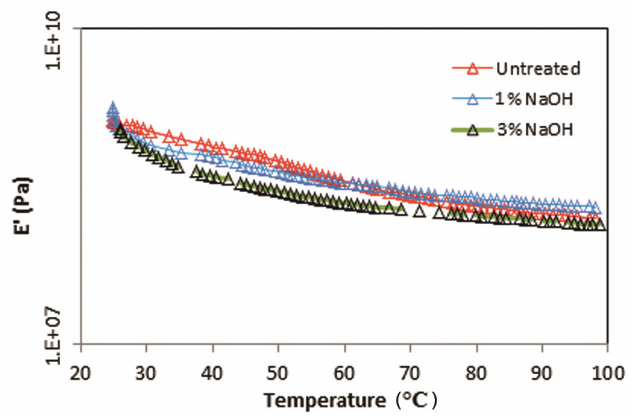

Fig. 1— Variation in storage modulus of untreated and treated voil fabric-epoxy composites as a function of temperature

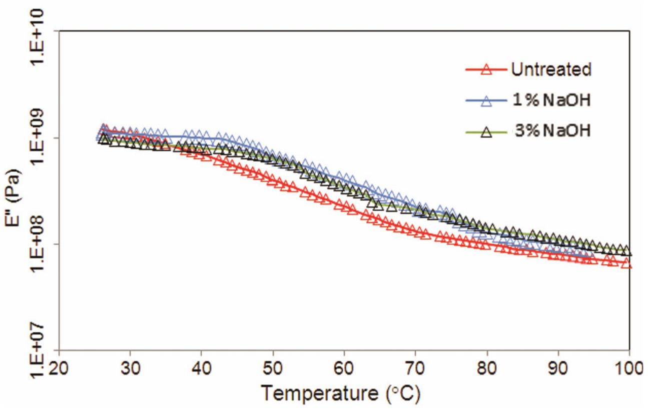

Fig. 2— Variation in loss modulus of untreated and treated voile fabric-epoxy composites as a function of temperature

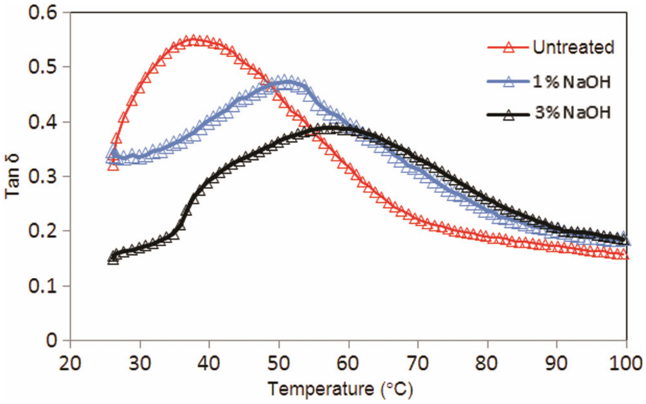

Fig. 3— Variation in tan δ untreated and treated voile fabric-epoxy composites as a function of temperature

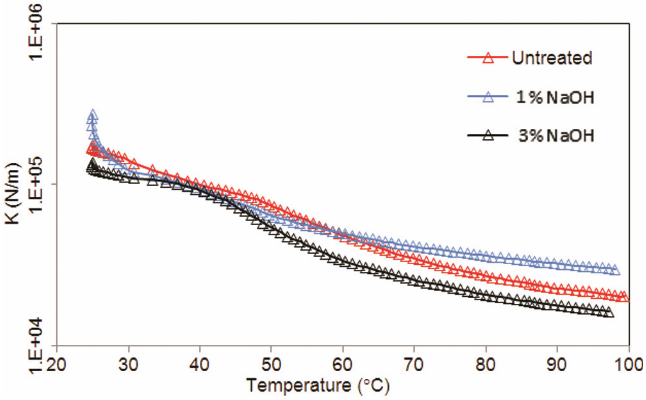

Fig. 4— Variation in stiffness untreated and treated voile fabric-epoxy composites as a function of temperature

composites (0.3952 at 30.55 °C). It is reasonable to expect a higher tan δ peak for the treated composite when compared to the other composites. This is due to the enhanced interfacial adhesion between untreated fabric and matrix. The lower value of tan δ for 1 % NaOH treated composite indicates that the composite has good load bearing capacity, high interfacial adhesion and improved stress transfer[24,25]. Figure 4 shows that the tensile stiffness decreases with increase in temperature. The stiffness (KN/m) values of untreated and treated (1 % and 3 % NaOH) composites are 9.35e4 N/m, 1.93e5 N/m, and 1.33e5 N/m at 25 - 26 °C respectively. At temperatures above 25 - 26 °C for instance at 100 °C, there is a decrease in stiffness. However, this does not significantly influence the drop in the modulus on passing through the glass transition temperature.

3.3 SEM Study

Figure 5 (a) shows the SEM image of the fractured surface of the untreated cotton fibres reinforced epoxy composites. The surface of the

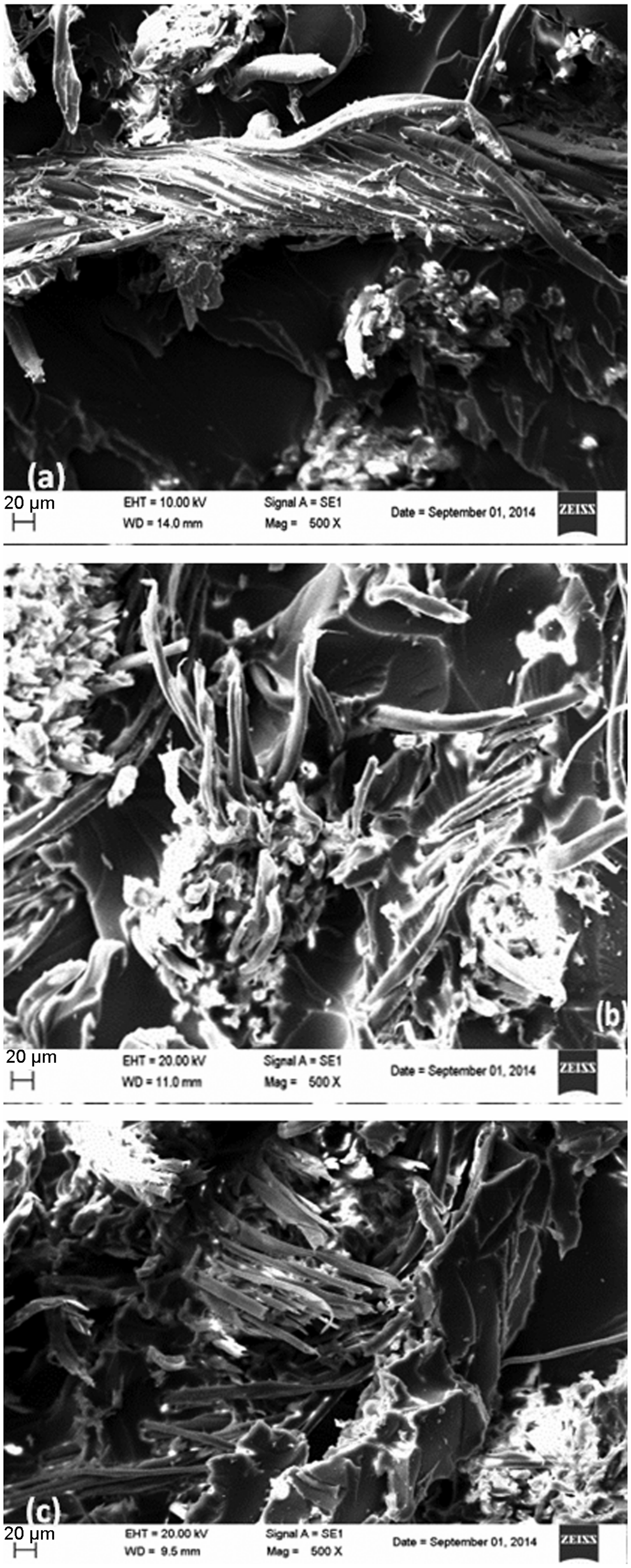

Fig. 5— SEM micrographs of (a) untreated, and alkali-treated [(b) 1% NaOH and (c) 3% NaOH] voile fabric reinforced epoxy composites

untreated fibre is smooth with no evidence or trace of any matrix resin adhering to the cotton fibere. This low fibre/matrix adhesion, explains the poor mechanical properties of the composite reinforced with untreated fibres[8]. Figures 5 (b)-(c) show the removal of cementing material from the multicellular matrix to some extent with the individual cells becoming more prominent. In particular, there is a significant improvement in interfacial adhesion. Consequently, the improved adhesion leads to a significant increase in mechanical properties. Similar results have also been reported by Ray *et al.*[26] and Weyenberg *et al.*[27].

4 Conclusion

This study reports the mechanical properties of voile fabrics treated with 1% and 3% NaOH for 1 h. Results indicate an increase in the tensile properties of the fabrics with alkali treatment due to the improvement in fabric structure. Best results are observed for fabrics treated with 3% NaOH solution for 1 h. which exhibit an increase in tensile strength by 32.72%. This could be attributed to the improvement in fibre- matrix adhesion as a result of alkali treatment. The surface morphology of the fabric becomes rough as a results of alkali treatment with the roughness increasing with the treatment due to the removal of hemicellulose. Results of DMA show that the treated composites have higher $E^{'}$ and lower tan δ indicating greater interfacial bond strength and adhesion between the matrix resin and the fabric when compared to the untreated composites. These results suggest the suitability of voile fabrics as a reinforcement agent for green composites.

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