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# A Silicone Treatment Compared to Traditional Natural Fiber Treatments: Effect on the Mechanical and Viscoelastic Properties of Jute–Vinylester Laminates

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**ABSTRACT:** In this work vinylester–matrix composites reinforced with woven jute fabrics were investigated. The effect of a simple fiber treatment with silicone on the mechanical and viscoelastic response in comparison to traditional treatments was analyzed. Unless the composite with acetylated fibers, all composites exhibited higher values of rubbery storage modulus than the matrix. All treated fiber composites have similar damping capacities. Furthermore, silicone had a plasticizing effect on the vinylester matrix. The composites with washed fibers and with fibers treated with silicone exhibited slightly higher stiffness and strength values than other treated fiber composites. In addition to its experimental simplicity, the treatment with silicone also allowed us to obtain the composites with best combination of stiffness, strength, and toughness.

**KEY WORDS:** natural fiber, composite, mechanical properties.

## INTRODUCTION

THE INCREASING TREND of using composite materials in many structural and semi-structural applications, as a good alternative to conventional materials, makes necessary the knowledge of their load-bearing capabilities. Usually during service, composite materials not only are subjected to static loading but also undergo dynamic stressing in a wide range of temperatures. Therefore, in addition to mechanical studies, viscoelastic analysis are also required. Both viscoelastic and mechanical properties of

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composite materials depend on several factors, such as fiber content, fiber orientation, fiber/matrix interaction and mode of testing [1].

On the other hand, the low specific gravity, the workable specific modulus, the low cost, and the non-abrasive, renewable and biodegradable nature of cellulosic fibers, make them attractive to use as reinforcements in composite materials. Therefore, novel applications involving furniture as well as engineering end uses, such as building materials and structural parts for motor vehicles, were developed [2–4]. However, poor properties of natural fibers such as their low stiffness and poor environmental resistance still limit some uses.

Mechanical properties of the composites depend on the properties of the fibers and the interphase fiber/matrix, but in the case of natural fibers another effect can influence them. Each natural fiber possesses different contents of cellulose, hemicellulose, and lignin as main compounds, as well as other compounds such as pectins and waxes [3]. The technical natural fiber is composed of several elementary fibers jointed by cementitious material, such as pectin and lignin, and the fibers have a hole along the fiber formed by the lumen. Such interphase between elementary fibers is important because in this region a longitudinal splitting can occur.

Several treatments have been proposed in the literature to improve fiber–matrix properties [5–7] and hence, to induce better mechanical or water resistance properties [8]. When a chemical treatment is applied on synthetic fibers like glass fibers only the interface of the fiber is modified. On the contrary, on natural or vegetable fibers, different changes on the interphase between elementary fibers, roughness, and density of the technical fibers could be also modified by the chemical treatment [9,10]. In addition other factors such as the orientation of microfibrils of cellulose within each elementary fiber play an important role because it changes the crystallinity of the natural fiber [11]. The development of stress in fiber bundles is governed by the binder that separates the elementary fiber. Hence, two kinds of interface should be taken into account in this case: one between fiber bundles and the matrix and the other between elementary fibers. All these factors complicate the dependence of mechanical properties on treatment methods [12,13].

The polar groups present in natural fibers are responsible for their good adhesion with thermosetting matrices like phenolics, epoxies, and polyester. There exist several fiber treatments that are conducted on natural fibers (i.e., alkali treatment, silane, acetylation, acrylonitrile, etc.). Most of the treatment modified not only the interphase but also produced morphological changes on the fibers, as was explained before.

One of most economically viable is alkaline treatment [14–18]. This process removes impurities such as waxes, pectins, mineral salts, and some hemicellulose and lignin. Extensive structural changes can be caused by the alkaline treatment of natural fibers, which, in turn, depend strongly on the parameters of the alkaline treatment (i.e., Na(OH) concentration, temperature, and time of the treatment). This condition and the fact that each natural fiber has a different morphology and composition, complicates the evaluation of the effect of alkaline treatment on the properties of the composite [19] and this is the cause of the differences found in each paper with different fiber, matrix, and alkaline conditions.

One effect of alkaline treatment is the modification of the crystallographic cell from cellulose I to cellulose II. The alkalization process also affects the surface morphology of natural fibers resulting remarkably in a more distinct exposition of the surface fibrils and a

more pronounced surface relief. Due to the intra and interfibrillar swelling, the accessibility of fibers changes drastically [20,21]. Depending on the treatment condition, the alkaline treatment produces a rough surface, so the number of anchorage points increases, offering a good mechanical interlocking fiber–matrix [3].

Acetylation is one of the most studied reactions of lignocellulosic materials. Acetylation is a rather attractive method of modifying the surface of natural fibers and making it more hydrophobic. This is an important aspect because the higher water absorption of natural fibers produces major problems in many applications [22].

In spite of this a lot of research was done on chemical treatment on natural or vegetable fibers, are a few studies were done on woven textile fibers like jute woven fibers. The aim of the study was to attempt to find a treatment without any water or solvent in the procedure and compare it with traditional fiber treatments (i.e., alkaline and acetylation).

## EXPERIMENTAL

### Materials

Commercially available woven jute fabrics (Casthanal, Textil CIA, Brazil) were used as reinforcement. The matrix material was prepared from general purpose vinylester resin (Derakane Momentum 411–350 from Dow, kindly provided by Poliresinas San Luis, Buenos Aires, Argentina) and accelerator in a weight ratio of $1:0.05$, respectively.

### Fiber Treatment

#### WASHED
Jute fabrics were washed with distilled water and detergent solution, and dried until constant weight in an oven at $80°C$ under vacuum before use. These simply washed fabrics will be referred to here as washed fibers.

#### ALKALI TREATMENT
Jute fabrics were treated with Na(OH) aqueous solution (5% w/v) for 24 h at $25°C$ with continuous shaking [23]. Then, they were washed with distilled water until all the sodium hydroxide was eliminated, that is the pH was neutral. Finally, fibers were dried at $80°C$ until constant weight was found.

#### ACETYLATION: TREATMENT WITH ACETIC ANHYDRIDE/ACID
Fabrics were immersed in glacial acetic acid at room temperature for 30 h [10,13]. After that, they were decanted and soaked in acetic anhydride containing two drops of concentrated sulfuric acid for a period of 5 min. Then the fabrics were filtered and washed with distilled water until free from acid. Subsequently, fibers were dried at $80°C$ under vacuum until constant weight was found.

#### TREATMENT WITH SILICONE
Jute fabrics were sprayed with a silicone release coating (Prosil D-010) based on polydimethylsiloxane before molding.

## Composite Preparation

Each layer of jute fabric was pre-impregnated with matrix material and placed one over the other in the mold by a hand lay-up technique, taking care to keep practically achievable tolerances on fabric alignment. Four layers were compression molded in a hydraulic press for 1 h at 80°C. Then, the plaques were post-cured 2 h at 80°C followed by 2 h at 140°C in an oven. Different composites with simply washed jute fabrics or with treated fibers were prepared. For all composites, jute fiber content was 40 wt.%.

## Thermogravimetric Analysis

Dynamic thermogravimetric measurements were performed by using a Seiko Instruments Exstar 6000 TGA-DTGA analyzer. Temperature programs for dynamic tests were run from 30°C to 700°C at a heating rate of 10°C/min. TG/DTG tests were carried out under nitrogen atmosphere in order to prevent any thermoxidative degradation.

## Dynamic Mechanical Analysis

Viscoelastic properties were measured using a dynamic mechanical analyzer Perkin Elmer DMA 7e with three-point-bending geometry. Tests were performed in temperature scan mode from 25°C to 180°C at a fixed frequency of 1 Hz. The used span was 20 mm. For all experiments, the strain amplitude was 0.1%.

Rectangular specimens having a size of 25 mm × 5 mm × 3 mm were used for the evaluation of storage modulus ($E'$), loss modulus ($E''$) and damping parameter (tan $\delta$). The dynamic to static stress was 800 MPa and dynamic stress 500 MPa.

## Mechanical Tests

Tensile and flexural specimens were machined from compression-molded plaques of 3 mm thickness in accordance with ASTM D3039M–95 [24] and ASTM D790M [25] standard recommendations, respectively. Uniaxial tensile tests were performed in an Instron dynamometer 4467 at 2 mm/min by using an incremental mechanical extensometer to measure actual elongation during the tests. True stress–strain curves were obtained from these tests and Young's modulus and tensile strength values were determined from these curves. In addition, toughness values were also obtained from the area under the load–displacement tensile records divided by the area of the test specimens.

Flexural tests were done in an Instron dynamometer 4467 at 1.2 mm/min crosshead speed, and the span used was 48 mm.

Short beam tests were carried out by following ASTM D2344-84 [26] standard recommendations to determine the composites interlaminar shear strength (ILSS).

**Fracture Surface Analysis**

The fracture surfaces of broken specimens in uniaxial tension were analyzed by scanning electron microscopy (SEM) in a JEOL JSM-6460LV equipment after they had been coated with a thin layer of gold.

**Water Absorption**

Samples measuring $10\,m \times 10\,mm \times 3\,mm$ were immersed in distillated water at 25°C. Three specimens for each sample were weighted at different times. The water uptake was evaluated at $M(t) - M_0/M_0$, where $M_0$ was the weight of the specimen after drying at 80°C under vacuum until constant weight was found, and $M(t)$ was the weighted specimen at different immersion times. The equilibrium value ($M$) was obtained when a constant weight after immersion was found. The average equilibrium value was determined.

## RESULTS AND DISCUSSION

**Thermogravimetric Analysis**

Figure 1 presents the derivative results of mass percent of the temperature function for all composites with different fiber treatments. Except for the composite with alkali-treated fibers, the curves for all the samples showed three decomposition peaks. The first decomposition peak in the range of 250°C and 300°C corresponds to the thermal
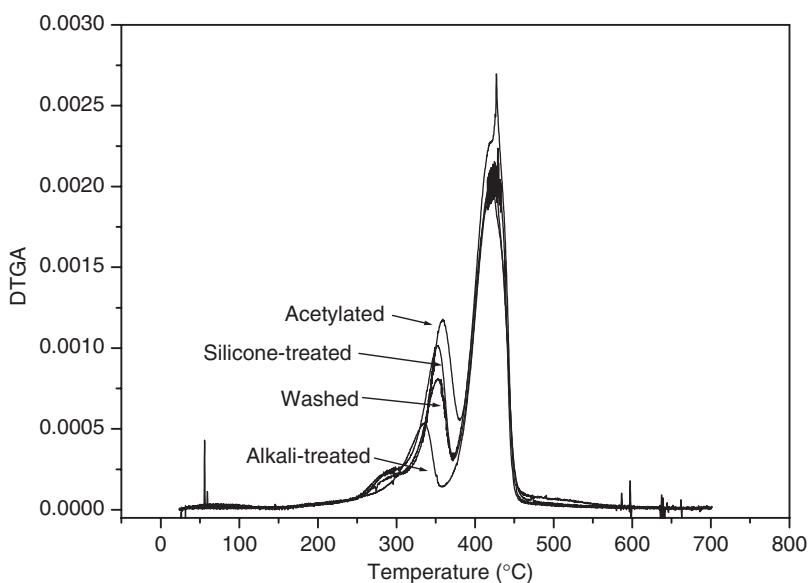


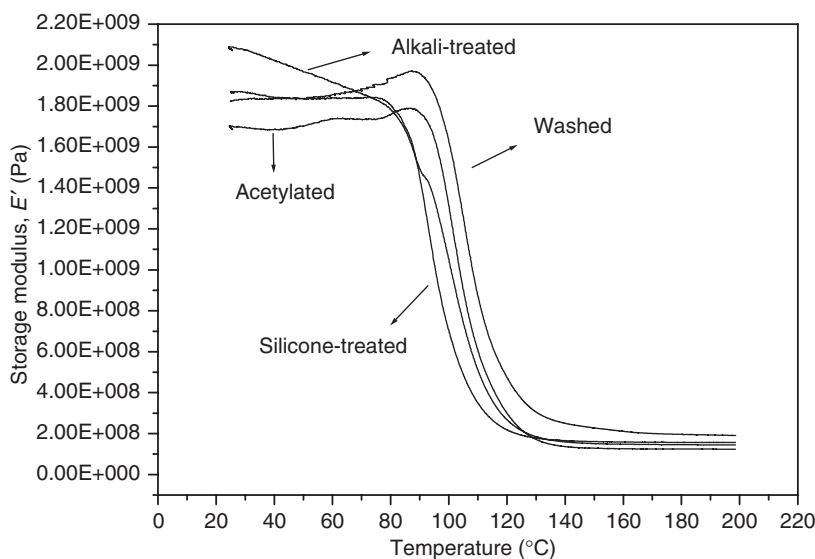**Figure 1.** *DTGA curves for the composites with different fiber treatments.*

**Figure 2.** *Storage modulus E' values as a function of temperature for the composites with different fiber treatments.*

depolymerization of hemicellulose, lignin, and the glycosidic linkages of cellulose [10]. The second peak at about 350°C for most of the samples represents the decomposition of $\alpha$-cellulose. Finally, the third peak at about 430°C is related to the decomposition of the vinylester matrix [22]. Furthermore, degradation of lignin is known to occur first and at a slower rate than the other components [10] having a broader peak between 200°C and 500°C. Therefore, this peak probably overlaps the other peaks.

For the composites with alkali-treated fibers the first peak was absent and the second peak shifted to a lower temperature due to the partial removal of lignin and hemicellulose promoted by the alkali treatment [10,27,28].

On the other hand, it is also possible to see in Figure 1 that the second peak is slightly shifted to a higher temperature for the acetylated fiber composite, suggesting higher thermal stability in this material.


### Dynamic Mechanical Analysis

*STORAGE MODULUS E'*

Figure 2 shows the storage modulus $E'$ values as a function of temperature for the composites with treated and untreatead fibers. As can be observed in this figure, a sharp drop of $E'$ in a small range of temperature was seen indicating the presence of the material glass transition temperature, $T_g$. The composite with silicone-treated fibers presented a lower value of $T_g$ probably due to a plasticizing effect of silicone on the matrix.

Figure 3(a,b) shows experimental values of storage modulus for the glassy and the rubbery state, respectively. No significant differences between the glassy storage modulus for the composites with different fiber treatments as well as for the matrix were observed (Figure 3a). In contrast, unless the composite with acetylated fibers, all composites
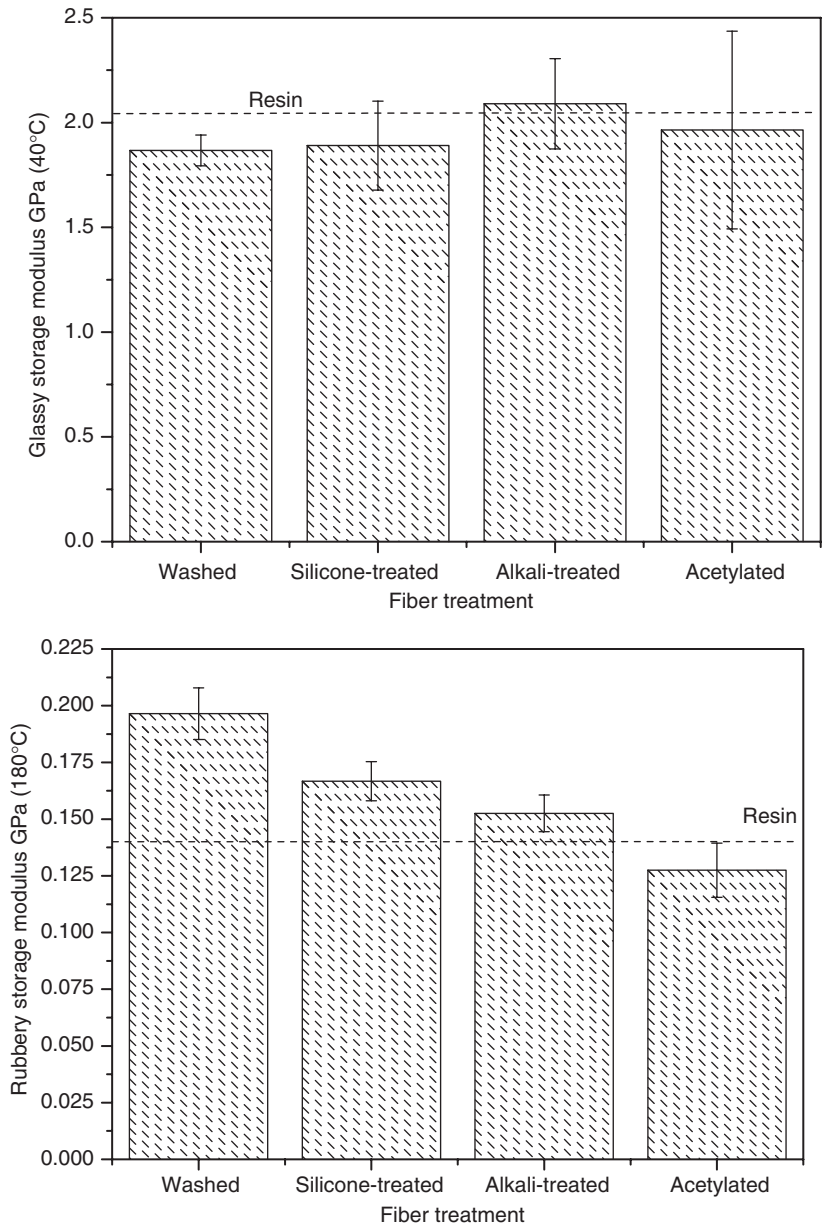
**Figure 3.** *Storage modulus E′ values for the composites with different fiber treatments. (a) Glassy state and (b) Rubbery state.*

exhibited higher values of storage modulus than the vinylester matrix in the rubbery state (Figure 3b) as the drop of the matrix modulus was compensated by the fiber stiffness [29]. In addition, all treated fiber composites exhibited slightly lower rubbery storage modulus than the composite with simply washed fibers having the composite with silicone-treated fibers the highest value among the treated-fiber composites.
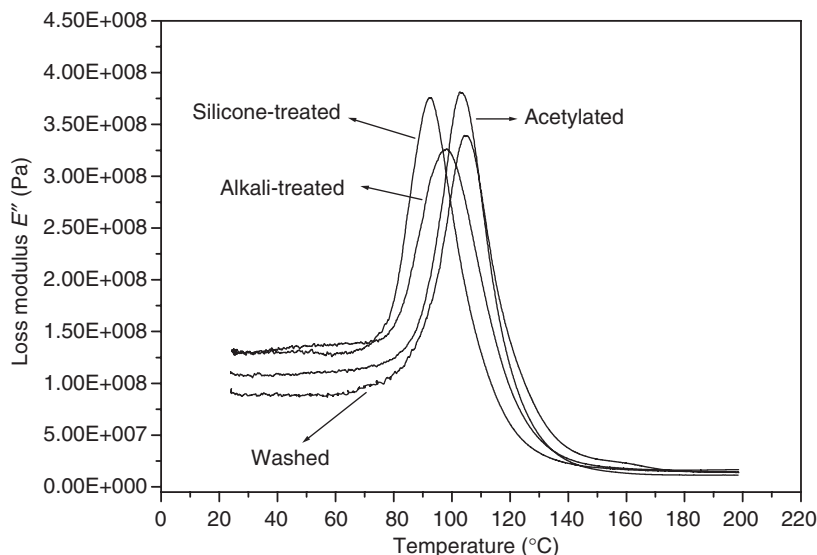
**Figure 4.** *Loss modulus E″ values as a function of temperature for the composites with different fiber treatments.*

## LOSS MODULUS E″

Figure 4 presents loss modulus $E''$ values as a function of temperature for all composites. The composite with silicone-treated fibers exhibited the lowest value of $T_g$ in agreement with storage modulus results while no remarkable differences in $T_g$ values were observed among the other composites.

## TAN δ (DAMPING PARAMETER)

Figure 5 shows the results for the damping parameter $\tan \delta$ as a function of temperature for the different composites investigated. All composites exhibited lower values of the $\tan \delta$ peak height than the matrix value reported in the literature [29,30], indicating that the incorporation of stiff fibers in the matrix restricted the movement of polymer molecules as expected.

On the other hand, Figure 6 shows the $T_g$ values obtained from the $\tan \delta$ peak. Unless the composite with fibers treated with silicone, no significant differences between the $T_g$ values for the different composites assayed were found in agreement with $E'$ and $E''$ results (Figures 2 and 4, respectively).

The peak shift in the loss modulus and $\tan \delta$ diagrams to lower temperature for the silicone-treated composite can be mostly attributed to the plasticizing effect of the silicone on the matrix. This effect counteracts the immobilization of the polymer molecules near the surface of the fiber, which otherwise would shift the peak to a higher temperature [16].

## Mechanical Tests

Unlike the composite with acetylated fibers, most composites exhibited stress–strain curves with some degree of non-linearity before maximum load (Figure 7) as a result of the development of incipient damage such as matrix cracking, fiber failure, or fiber pull-out.
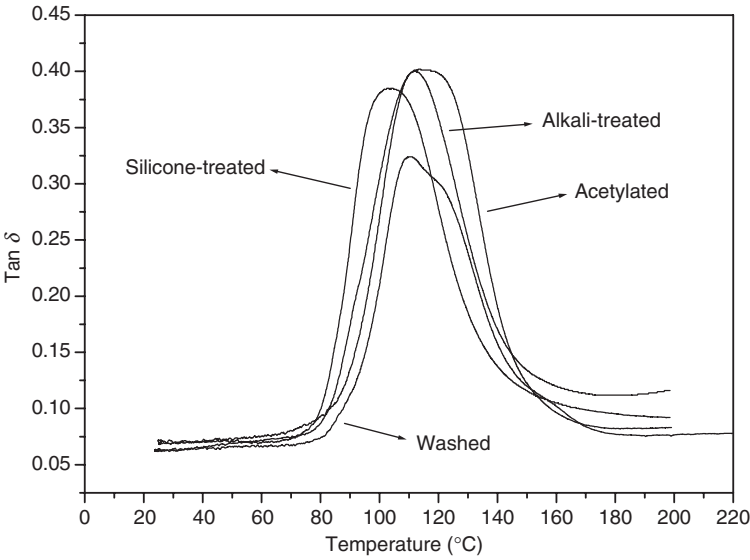
**Figure 5.** *Damping parameter tan δ values as a function of temperature for the composites with different fiber treatments.*
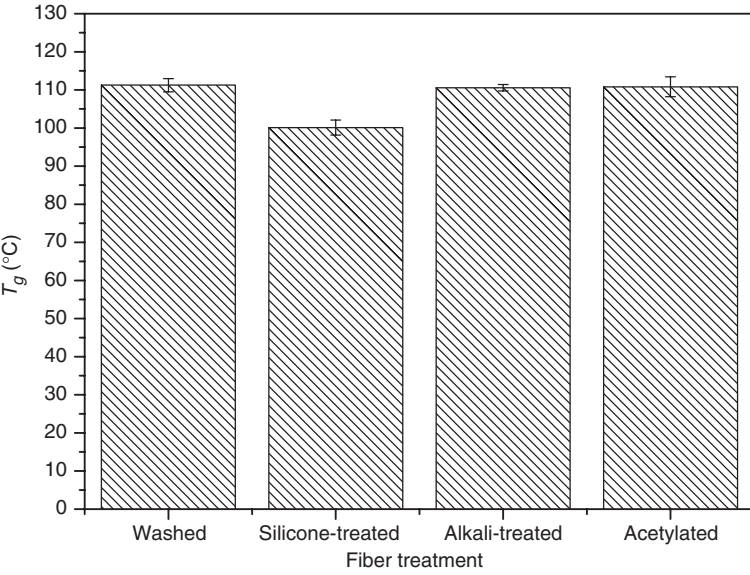


**Figure 6.** *Glass transition temperature $T_g$ values obtained from the tan δ peak for the composites with different fiber treatments.*

A marked drop of load after fracture was also observed in all samples suggesting simultaneous fracture of fibers and matrix.

In the composite with acetylated fibers, the poor interfacial adhesion led to a premature pull out of the whole yarns which hindered the development of the damage mentioned above.
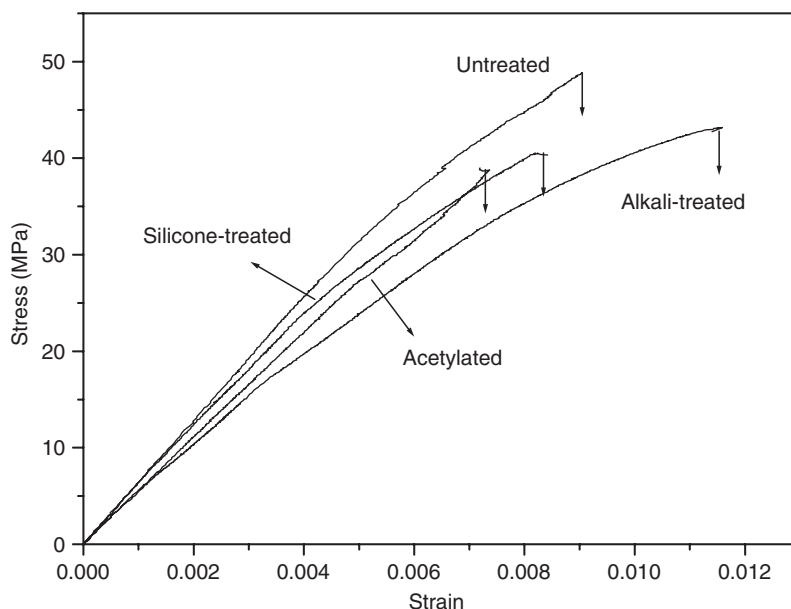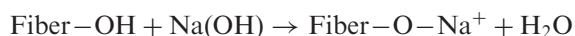
**Figure 7.** *Stress–strain curves for the composites with different fiber treatments.*

Figure 8(a,b) presents Young's modulus and strength values, respectively, for the composites with different fiber treatments. The composites with washed fibers and with fibers treated with silicone exhibited slightly higher stiffness and strength values than alkali-treated and acetylated fiber composites.

It has been previously reported in the literature that the effect of the alkali treatment on individual natural fibers [10] or yarns [8] is an increase in the elastic modulus and strength. It has been attributed to the change of cellulose I into cellulose II promoted by the alkali treatment that leads to a tighter packaging of the chains and an increase in the degree of molecular orientation by Cyras et al. [10] and to the interaction factors as well as to a highly reduced fiber diameter by Gassan and Bledzki [8]. Partial extraction of lignin and hemicellulose has also been reported. However, as it has been well established by Cyras et al. [10], individual mechanical properties of natural fibers are not the unique factor for determining the composite mechanical properties.

For the case of the composite with alkali-treated fibers, an increase in the OH concentration due to the changes in the spiral angle and higher exposition of OH, when cellulose I changes to cellulose II [11] is expected. The fiber with a higher amount of OH groups would become more compatible with the vinylester matrix [1]. The chemical reaction should be [31]:

$$\text{Fiber}-\text{OH} + \text{Na(OH)} \rightarrow \text{Fiber}-\text{O}-\text{Na}^+ + \text{H}_2\text{O}$$

Our results suggested that the effect of alkali treatment on the composite tensile modulus and strength was slightly detrimental due to the concentration and time which was used in the treatment, which can produce loss of cementitious material between the fiber cells.
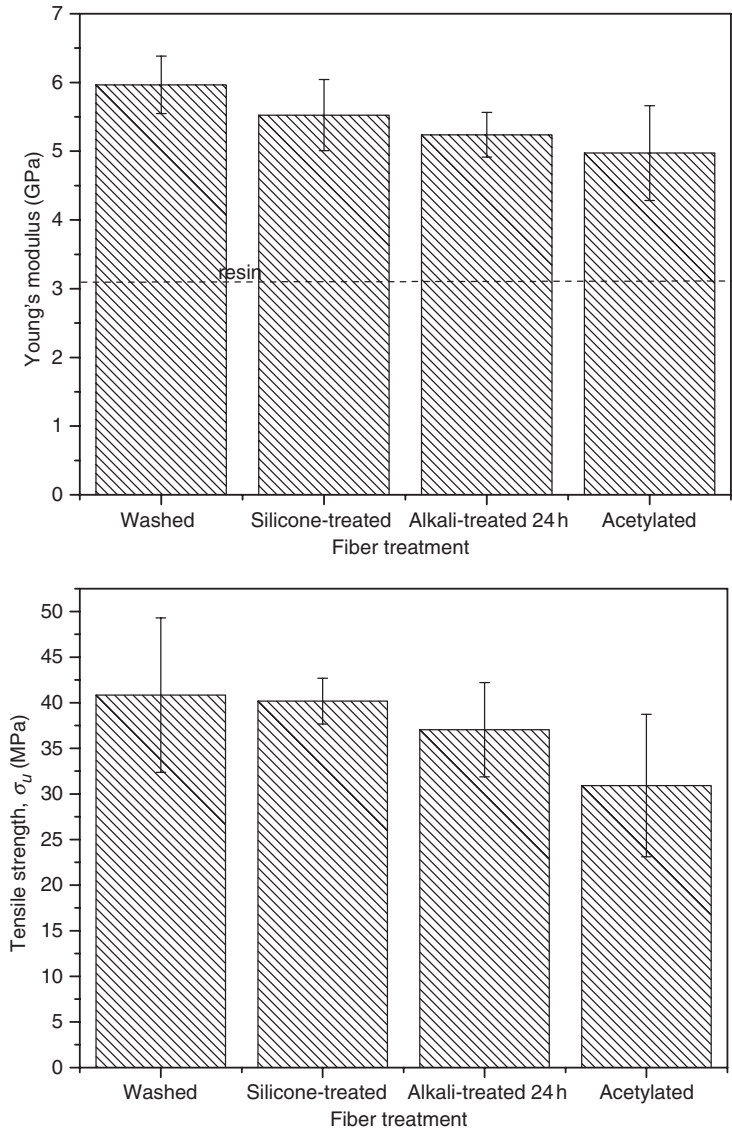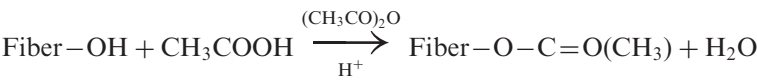
**Figure 8.** *Uniaxial tensile properties for the composites with different fiber treatments. (a) Young's modulus (dotted line indicates the matrix stiffness value) and (b) Tensile strength.*

On the other hand, during the acetylation treatment the replacement of hydroxyl groups by acetyl groups leads to the reduction of the OH concentration, segmental mobility is restricted by the presence of more voluminous groups in the final structure, the orientation of cellulose chains changes, and cellulose backbone stiffness increases. The chemical reaction is [31]:
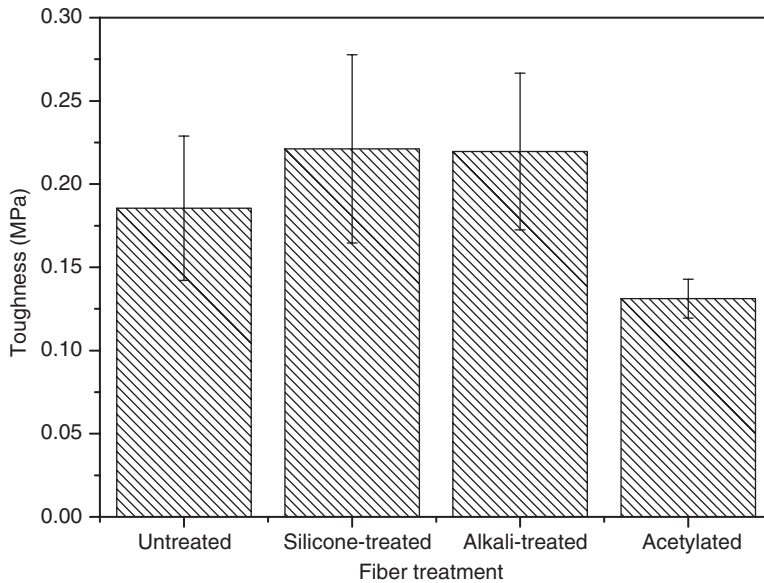
$$\text{Fiber}-\text{OH} + \text{CH}_3\text{COOH} \xrightarrow[\text{H}^+]{(\text{CH}_3\text{CO})_2\text{O}} \text{Fiber}-\text{O}-\text{C}=\text{O}(\text{CH}_3) + \text{H}_2\text{O}$$

**Figure 9.** *Toughness values obtained from the area under the stress–strain curves.*

**Table 1.** *Shear and equilibrium water absorption values for the different composites with different treated jute fibers.*

| Composite with jute fibers | ILLS (N/m$^2$) | %water absorption at the equilibrium (*M*%) |
|---|---|---|
| Washed | 6.6 ± 0.3 | 14.5 ± 1.11 |
| Alkaline treated | 7.1 ± 0.3 | 11.89 ± 0.13 |
| Acetylated | 7.1 ± 0.8 | 11.70 ± 0.13 |
| Silicone treated | 8.1 ± 0.5 | 11.09 ± 0.8 |

For the acetylated fiber composite, tensile properties were not improved. Moreover, slightly lower values of stiffness and strength were observed in this work for the acetylated fiber composite in comparison with the composite with washed fibers, as pointed out before.

It is also important to note that the composites toughness obtained from the area under the tensile stress–strain curves was observed to increase with silicone and alkali treatment (Figure 9) in comparison to the washed and acetylated fiber composites. Therefore, in addition to its experimental simplicity, the treatment with silicone allowed us to obtain the best combination of stiffness, tensile strength and toughness among the different composites investigated.

On the other hand, in order to characterize the interlaminar composites behavior, short beam tests were also carried out on the different composites. Nevertheless, the required interlaminar failure could not be attained [32]. The specimen failure mode appeared to be bending by simultaneous fiber breakage and partial fiber pull-out. However, in order to compare each sample, the apparent shear values measured at low span ratio are shown in Table 1 for each composite. The silicone composite showed the highest value. The terminal
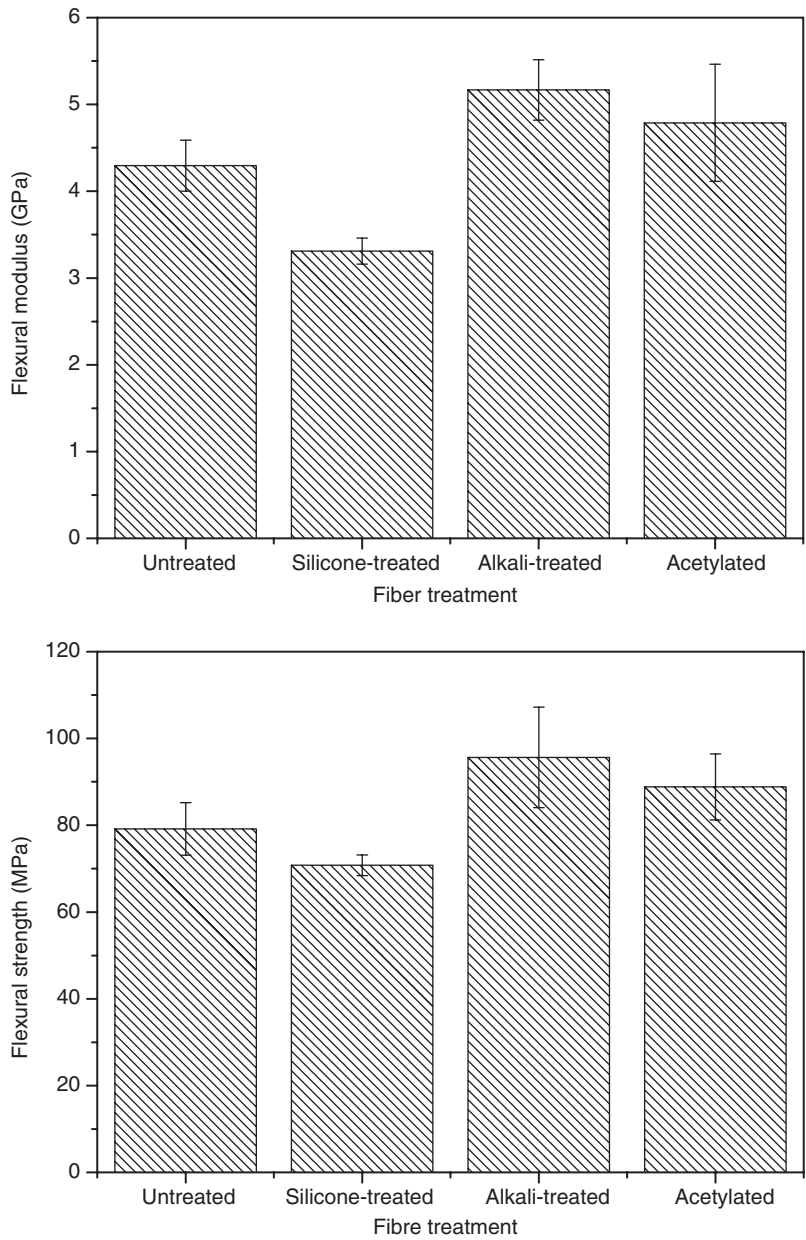
**Figure 10.** Flexural properties for the composites with different fiber treatments. (a) Flexural modulus and (b) Flexural strength.

groups of the polydimethylsiloxane $(HO(CH_3)_2SiO)_nH)$ would form hydrogen bonds with the OH groups of the natural fibers and therefore would lead to a better interfacial property.

The modulus and strength values obtained in flexural tests are presented in Figure 10. It is observed in Figure 10(b) that all experimental values of flexural strength were higher than those obtained under tension. In tensile tests, the whole cross-sectional area of a

material is subjected to homogeneous tensile stress while in flexural tests one half of the cross-sectional area is under tension and the other half is under compression. Therefore, the higher values of flexural strength can be attributed to the better behavior of the thermosetting matrix under compression. However, in contrast to the tensile results, the silicone-treated composite showed the lowest value of flexural strength in spite of the better interfacial properties exhibited by this composite. This is probably due to the plasticizing effect of the silicone on the matrix mentioned before which leads to a significant reduction of the compressive yield strength.

### Water Absorption Measurements

Water uptake was measured for each composite. Table 1 shows the results of the equilibrium value obtained at longer times. The composite which absorbs more water was the one based on washed jute fibers, because the jute fibers are cleaner. The composite with treated fiber has almost the same equilibrium value.

### Fracture Surface Analysis

Figure 11(a–e) shows SEM micrographs of the fracture surfaces of composites with different fiber treatments broken in uniaxial tensile tests. As clearly observed in Figure 11, properly oriented fibers were pulled out from the matrix. In addition pull-out length was greater for non-treated and silicone-treated fiber composites which also which exhibited improve tensile toughness in comparison with alkali-treated fiber composites where a greater number of fibers were pulled out through a short length. Pull-out of the elementary fibers from the hemicellulose/lignin matrix was also observed in alkali-treated fiber composites. On the other hand, in the acetylated fiber composite (Figure 11e), probably as a result of poor interfacial properties, the pull-out of jute yarns as a whole hindered individual fiber pull-out.

On the other hand, closer views of Figure 9 are presented in Figure 12(a–d). The best fiber–matrix adhesion was obtained for the composite with silicone-treated fibers (Figure 12b), whereas the worst interfacial adhesion was found for the composites with alkali-treated and acetylated fibers (Figure 12c and d, respectively). However, irrespective of the fiber treatment applied, all jute fibers appeared clean of the matrix material suggesting a relatively poor fiber–matrix interaction in agreement with the slight differences observed in the mechanical properties within the experimental scatter. Furthermore, alkali-treated and acetylated fibers appeared more defibrillated due to the lignin and pectin extraction in the former as shown above in the thermogravimetric analysis section. Evident collapse of the cellular structure of natural fibers after alkali treatment has already been reported by other authors [10]. On the contrary, silicon-treated fibers kept the original structure of the technical fiber probably promoted by the existence of the silicone coating mentioned above.

Finally, the mechanical behavior exhibited by the different composites was in agreement with fracture surface observations. The higher tensile strength value displayed by the silicone-treated fiber composite could be attributed mainly to the higher interfacial adhesion between fiber and matrix and also among elementary fibers within the technical fiber exhibited by this composite in comparison to the other fiber-treated composites. It is
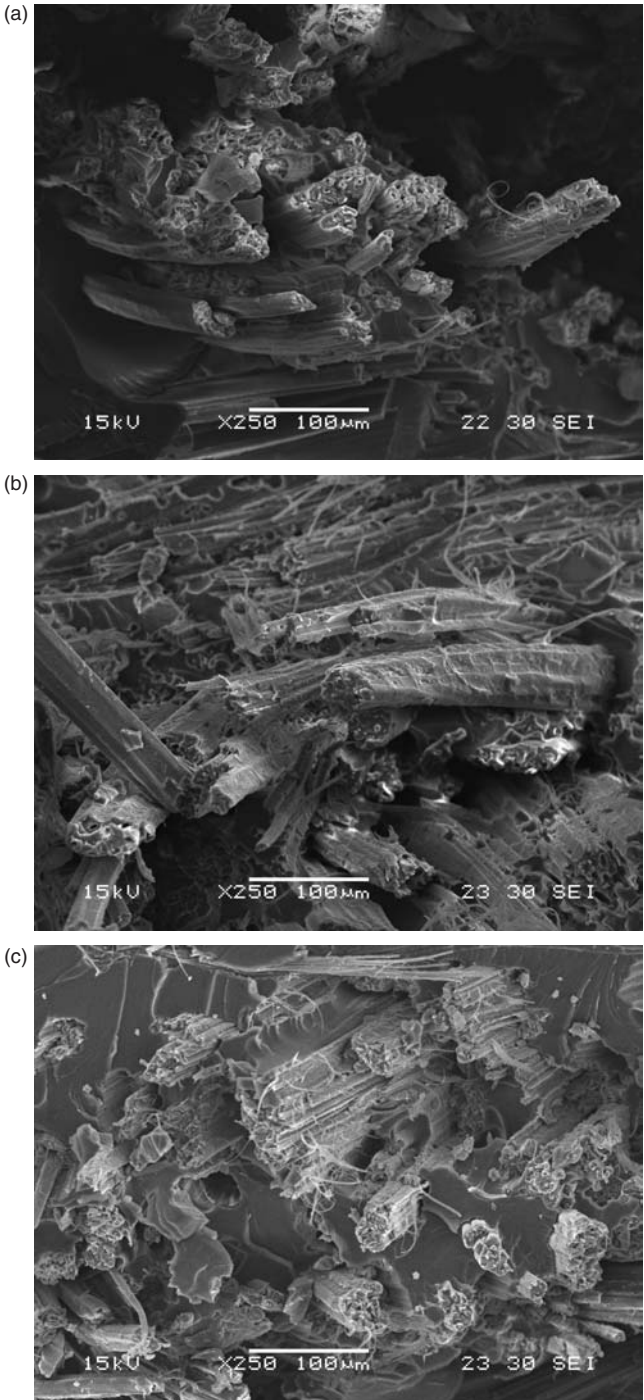
**Figure 11.** SEM micrographs of fracture surfaces of the composites with different fiber treatments broken in tension. (a) Composite with non-treated fibers. (b) Composite with silicone-treated fibers. (c) Composite with alkali treated fibers. (d) Composite with acetylated fibers (internal view of a pulled-out yarn). (e) Global view of the composite with acetylated fibers.
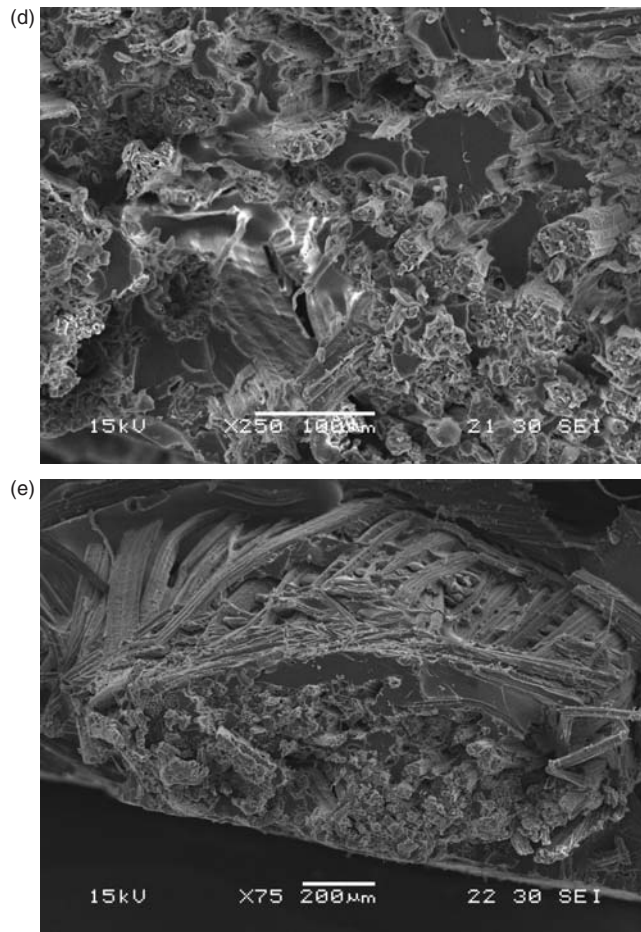
(d)

(e)

**Figure 11.** *Continued.*

also clear that when a chemical treatment with solvent was performed on the jute fibers, a change on the morphology was also obtained. In addition, the improvement in toughness found for this composite could be a result of the combination of long pull out lengths and relatively strong fiber–matrix interface. The combined effect of the pull-out of many single fibers through a short length and the pull-out of elementary fibers from the hemicellulose/lignin matrix, would be responsible for the toughness increase found in the alkali-treated fiber composite. On the other hand, acetylated fiber composites showed the lowest toughness value (Figure 9) probably due to the pull-out of the yarns as a whole which restricted single fiber pull-out with subsequent lower energy consumption.

## CONCLUSIONS

Vinylester–matrix composites reinforced with woven jute fabrics were investigated, focusing on the effect of a simple fiber treatment with silicone on the viscoelastic and mechanical response in comparison to traditional treatments.
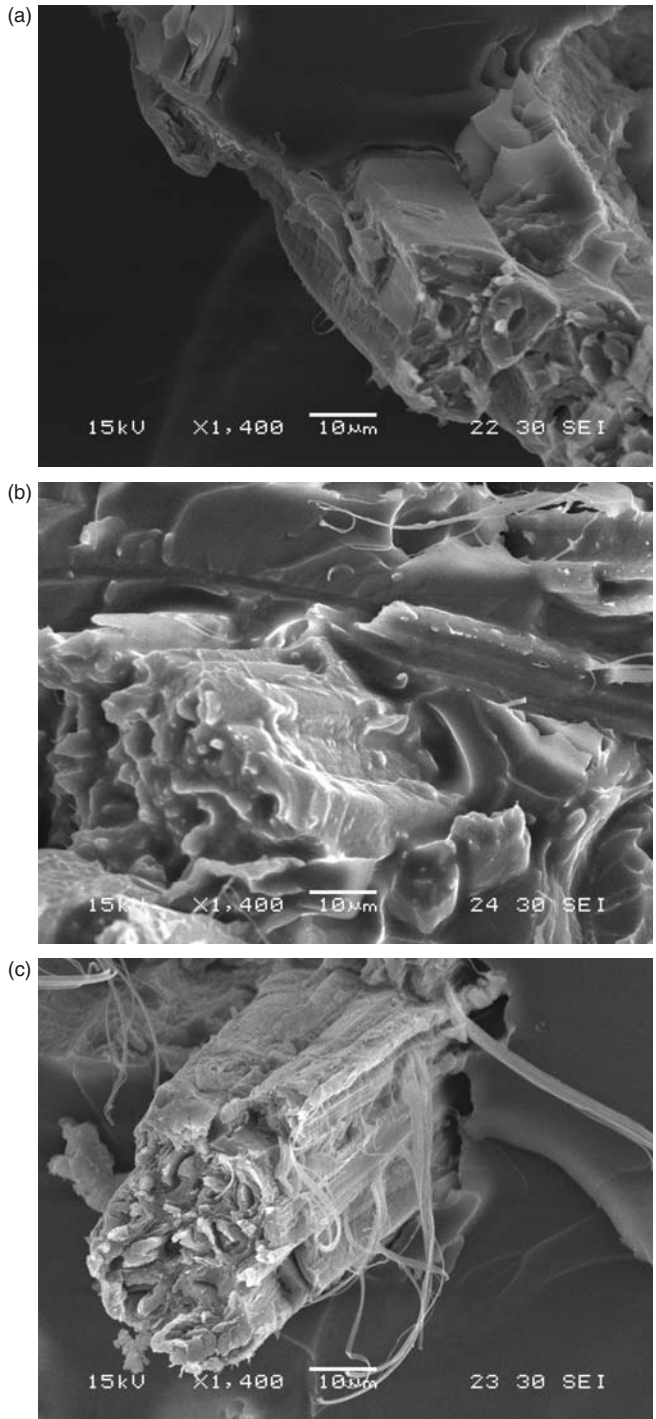
(a)

(b)

(c)

**Figure 12.** *Closer views of fracture surfaces of the composites with different fiber treatments broken in tension. (a) Composite with non-treated fibers. (b) Composite with silicone-treated fibers. (c) Composite with alkali treated fibers. (d) Composite with acetylated fibers.*
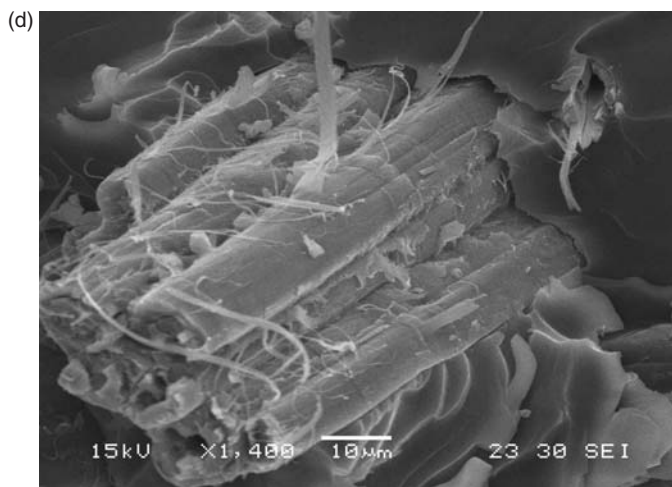
**Figure 12.** *Continued.*

From dynamic mechanical analysis, no significant differences between the composites with different chemical treatments on the fibers and the matrix for the glassy state were observed. In contrast, unless the composite with acetylated fibers, all composites exhibited higher values of rubbery storage modulus than the matrix since the drop of the matrix modulus was compensated by the fiber stiffness. On the other hand, all composites exhibited lower values of the $\tan \delta$ peak height than the matrix, indicating that the incorporation of stiff fibers in the matrix restricted the movement of polymer molecules. In addition, all treated fiber composites have similar damping capacities.

Furthermore, from the results of dynamic mechanical analysis it was evident that silicone had a plasticizing effect on the vinylester matrix since a lower $T_g$ value was observed for this composite.

Under uniaxial tensile loading, all composites exhibited significantly higher values of stiffness than the matrix, whereas quite lower values of tensile strength were observed for the composites. Furthermore, the composites with washed fibers and with fibers treated with silicone exhibited slightly higher stiffness and strength values than alkali-treated and acetylated fiber composites.

Water absorption experiments confirm that the silicone treatment has the same final value than the other treated fibers. The composites based on washed fiber had the highest water uptake value.

The results obtained from mechanical tests were confirmed from SEM analysis. Toughness results were also explained in terms of the different failure mechanisms observed by SEM. The best fiber–matrix adhesion was obtained for the composite with silicone-treated fibers which exhibited the highest tensile strength value.

Silicone treated fiber composites under tension showed the highest value of toughness and therefore, the best combination of stiffness, strength, and toughness. Also, water absorption did not increase making it the simplest treatment among those investigated here.

## ACKNOWLEDGMENTS

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