INFLUENCE OF ALKALI FIBER TREATMENT ON FRACTURE TOUGHNESS OF ALFA FIBERS REINFORCED POLYESTER RESIN

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Abstract
The aim of this paper is to describe the fracture behavior and failure mechanisms of Alfa fibers and its composites. The used reinforcement consists of Alfa fibers, extracted from the plant Stippa tenacissima from Hodna Region (Algeria). The Alfa fibers are subjected to alkali treatments with NaOH at 1, 5 and 10% for a period of 0, 24, and 48 h at 28°C. The composites reinforced with layers of Alfa random constituent a rate of 40% by weight. We studied the development of the different damage phases using the digital image correlation under static loading of compact tension test (CT). The plane-strain fracture toughness, K_R, of different random short Alfa fibers reinforced polyester resin was investigated with the effect of fibers treatment.

1 Introduction

The uses of natural fibers as reinforcement in polymer matrices are attracting more attention of researchers. The attractive features of natural fibers have been their low cost, light weight, high specific modulus and health hazards of composites reinforced with synthetic fibers such as glass and carbon fibers. These advantages place the natural fiber composites among the high performance composites having economical and environmental advantages [1–3]. However, the adhesion between the fibers and polymer matrices is generally insufficient [4]. Lack of good interfacial adhesion and poor resistance to moisture absorption made the use of natural fiber reinforced composites less attractive [5]. Several authors [1–6], have focussed the studies on the treatment of fibers to improve the compatibility of natural fibers with the matrix. A modification of the fibers can either increase or decrease the strength of the fibers, and thus an understanding of what occurs structurally is of great importance [3,6].

In the present investigation, we have chemically modified the surface of fibers in order to improve the fiber/matrix interface properties. Fibers are subjected to alkali treatments with NaOH at 1, 5 and 10% for a period of 0, 24, 48 h, and, five plates of composites are manufactured. We studied the development of the different damage phases using the digital image correlation under static loading of compact tension test. The stress intensity factors, K_R, in different composites were calculated and their values are compared.

2 Materials and testing methods

1.1 Materials

Alfa fibers were collected from Hodna region (Algeria). Alfa grass (Stipa tenacissima L.) is a tussock grass; it is constituted of stems with a cylindrical shape which have a maximum height of about 1m. Alfa fiber had a specific surface (in a dry state) of $3m^2/g$ [7]. According to Paiva et al. [8], the chemical composition of Alfa grass is composed of 45% of cellulose, 25% pentozane, 23% lignin, 5% wax and 2% of ash. Alfa fiber bundles are characterized by a mean diameter of 113 µm (ranging from 90 to 120 µm) and a density of ρ=0.89 $g.cm^{-3}$ [9].

The polar groups present in natural fibers are responsible for their good adhesion with thermosetting matrices like polyesters, epoxies and phenolics [10]. In this work, commercial unsatured polyester (UP) ISO for stratification is used as a resin.

1.2 Pretreatment

Once the Alfa fibers were harvested, they were washed with water (2% detergent solution) to remove the contaminants and adhering dirt. Thereafter, they were air dried for 72h at room temperature, and then Alfa stems were placed in packets, preserved in polyethylene bags and stored away from light for 60 days at ambient temperature. After the storage period, Alfa stems were cut into 6 cm lengths. These cut Alfa stems were milled using a vertical axis wheat mill. Its principle is to crush the chopped Alfa stems without destroying the fibrils. This is achieved by adjusting the distance between the grain grinders. The fibers obtained were then sieved to remove volatile compounds. Finally, the Alfa fibers are carded to make then soft and separated. After this Alfa pretreatment, the lengths of fiber varied from 0.4 to 6 cm. The resulting fibers were denoted as untreated Alfa fibers.

1.3 Alkali treatment of Alfa fiber

The Alfa fibers were soaked in a 1, 5 and 10% NaOH solution at 28°C. The fibers were kept immersed in the alkali solution for both periods of 24 and 48 h. The treated Alfa fibers were then washed several times with distilled water. Any traces of NaOH, remaining on the fiber surface, were neutralized with 2 % sulfuric acid during 10 min. The fibers were washed again with distilled water until obtaining a pH = 7. Subsequently, the fibers were dried at 60°C for 6 hours. The weight loss after treatment was measured accordingly.

1.4 Composite manufacturing methods

The chopped fibers were randomly spread in a mould cavity. Extreme care was taken to get uniform distribution. A mixture of diluent with 3wt% UP resin was sprayed onto the random mat so that it can handle during the molding. Mould was then closed and pressure was applied to get it as a single mat (Fig.1). Composites were made using a wood mould measuring: 200 x 150 x 10 mm3 length, width and depth, respectively. The resin selected for this study was an isophthalic polyester resin which was cured using 1% of methyl ethyl ketone peroxide catalyst and 0.5% of cobalt–naphthanate accelerator. The manufacturing of the structural composite consists in soaking two random mats with polyester resin in the mould impression, then to press them with the mould cover. To clarify effect of chemical treatment on the flexure properties of Alfa fibers reinforced polyester composite; five plates of composites are manufactured (Table1) using hand lay-up technique [11]. The mat was then impregnated in the resin and the post curing was done at 50° C for 24 h. The filler content was fixed at 40wt% for all the composite materials. A minimum of five compact tension specimens (CT) were tested for each composite. The methods of specimen preparation are outlined in [12].

Figure 1. The random mats of Alfa fibers.

Composites	Fibers treatment	Designation
Composite 1	Untreated Alfa fibers	T0000
Composite 2	01% NaOH at 24h	T0124
Composite 3	05% NaOH at 24h	T0524
Composite 4	10% NaOH at 24h	T1024
Composite 5	05% NaOH at 48h	T0548

Table 1. Different manufacturing composites.

1.5 Fracture toughness testing

The tests were conducted on an Instron machine using a 10 KN capacity servo-hydraulic testing machine. The monotonic loading during the tests was applied at constant displacement rate 0.3 mm/min, and a minimum of five (CT) specimens were tested for each composite. Load-displacement curves are recorded for all tests. According to ASTM standard E399, the fracture toughness, K_R, is given by the following analytical form [13]:

$$K_R = \frac{P}{B.\sqrt{W}}.f(\alpha) \qquad (1)$$

Where B is the thickness of the specimen, α is the relative notch depth (= a/W) and $f(\alpha)$, given by Equation (2), is the well-known polynomial function formulated by Srawley [14]:

$$Y(\alpha) = \frac{(2+\alpha)}{(1-\alpha)^{3/2}}.(0.886 + 4.64\alpha - 13.32\alpha^2 + 14.72\alpha^3 - 5.6\alpha^4) \qquad (2)$$

1.6 Measurement of crack growth

In the present study, the digital image camera (DIC) technique was monitored in situ during the fracture process. The goals of adopting the DIC method are (i) to distinguish the different failure sequences by direct observation, (ii) to detect the crack initiation and follow its growth along the CT specimen and (iii) to evaluate the crack increase at different times (t). Before starting the fracture test, the dimensional characteristics of the CT specimen are introduced into the software. In our case, the unbroken length (ligament) is (W–a). It is important to note that the system Image ProLavison, adopted in this study, is designed for the treatment of 2D problems where the vertical and/or the horizontal deformation (crack propagation) are easily obtained (Fig.2).

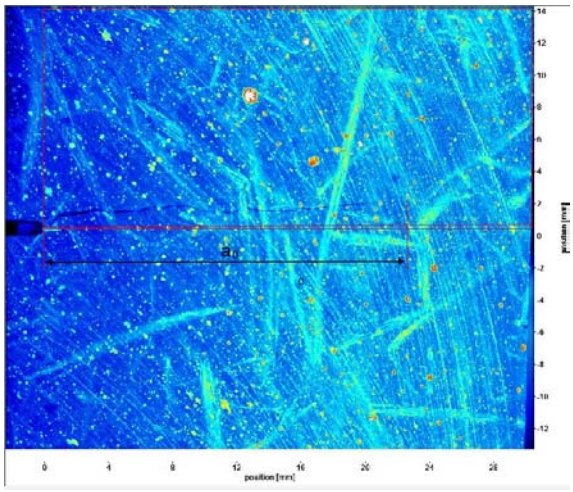

Figure 2. The determining of the increase of crack length in CT specimen by the DIC method.

2 Results and discussion

2.1 Load displacement graphs

The compact tension (CT) experiments were monitored through the recording of the associated load–displacement curve (P–d), where the displacement was measured between the two loading pins. For different composites, the (P–d) curves evolution are not similar (Fig.3). Results show that mechanical proprieties are strongly changed with fibers treatment [3]. Two types of evolution in (P–d) curves are observed in figure 3. In (P–d) curves of composites with treated Alfa fibers for 24 hours, the load increases linearly with displacement. Upon reaching the peak load, the load drops drastically to 50% of maximum load, than the (P–d) curves seem evaluated again. Generally, composites reinforced with treated Alfa fibers for 24 hours fail in a brittle manner. The evolution of (P–d) curves of composites T0000 and T0548 show a controlled fracture.

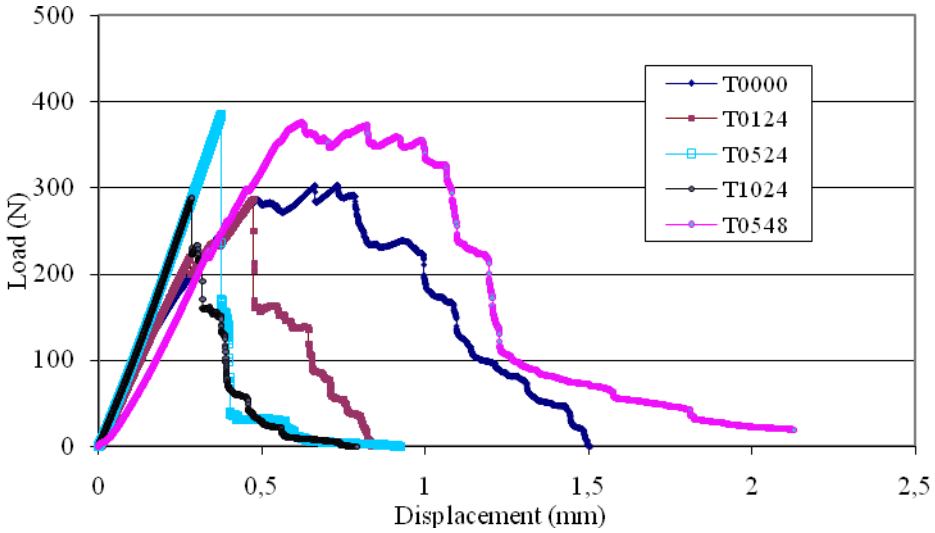

Figure 2. The determining of the increase of crack length in CT specimen by the DIC method.

The tests carried out on composites materials show that crack initiation and crack propagation for different tested materials are dissimilar. Critical load levels that are determined from a combination of values obtained by testing fracture (curve (P–d)) and photographs produced by CID technique, are so different (Table 2). These values showed that the alkali treatments of Alfa fibers have a significant effect on fracture behavior of composites.

Composites	Load (N)		Time(s)
	In first cracking	Maximum	In first cracking
T0524	356.07	384.67	70.00
T0548	272.87	375.67	83.50
T1024	215.34	287.60	61.50
T0124	207.16	279.97	56.00
T0000	186.55	302.45	52.00

Table 2. Remarkable levels of loads of the various studied composites.

According to Table 2, it appears that the composites reinforced with treated fibers at 05% of NaOH recorded the higher values in first cracking and maximum load. However, the first cracking in the materials T1024 and T0124 occurs at a very close load level of not-treated fibers composites. The remarkable reduction in the mechanical properties found in the composites T1024 and T0124 should be mainly the result of the lower or higher rate of NaOH of Alfa fibers. Mishra et al. [15] reported that 5% NaOH treated sisal fiber-reinforced polyester composite had better tensile strength than 10% NaOH treated composites. This is because at higher alkali concentration, excess delignification of natural fiber occurs resulting in a weaker or damaged fiber. The tensile strength of the composite decreased drastically after certain optimum NaOH concentration.

2.2 Comparison of fracture toughness

In figure 3, the values for the fracture toughness (K_R) were plotted as a function of crack length to produce a resistance R-curves which were estimated from the experimental data using Eqs. (1) and (2). In each R-curve, two stages are observed; the first stage is an increase in the toughness values with stable crack growth. The second one is propagation according to a plateau value; this level is characterized by crack instability. There are no distinguishing features between the stable and the unstable crack growth regions [12].

As may be seen, in all cases the treatment of Alfa fibers results in an improvement in the intrinsic fracture toughness ($K_{I\text{-}init}$) (first values in the curves and correspond to the onset of crack growth in each composite). However, the plateau toughness parameters ($K_{I\text{-}prop}$) were strongly infected by the NaOH effect (Table 3).

Composites	K_R(Mpa.m$^{1/2}$)	
	$K_{I\text{-}init}$	$K_{I\text{-}prop}$
T0524	1.22	1.16
T0548	0.94	2.06
T1024	0.87	0.99
T0124	0.72	1.28
T0000	0.62	1.53

Table 3. Summary of fracture toughness results.

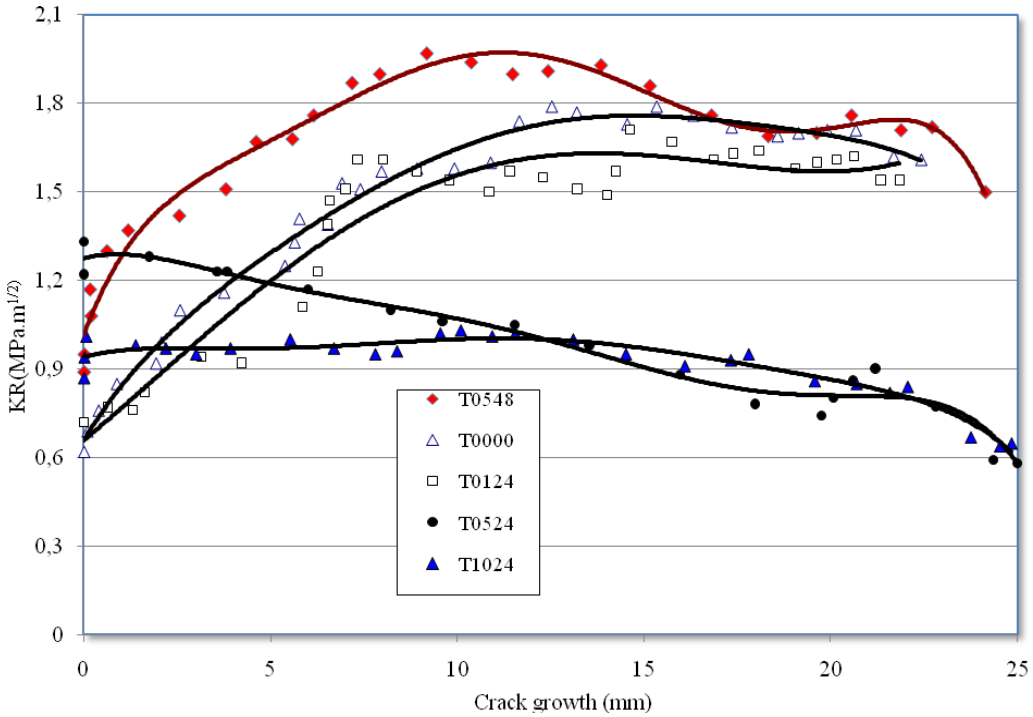

Figure 3. Fracture toughness (K_R) of materials composites.

As known, the cracks seek the path of least resistance provided by the most damaged micro-structural features ahead of it. It's the case of not-treated fibers composites (Fig.4a). However, In all tested CT specimens with treated Alfa fibers, the fracture paths are predominantly linear. This means that the damage zone evolution is accompanied by significant bundle bridging (Fig.4b).

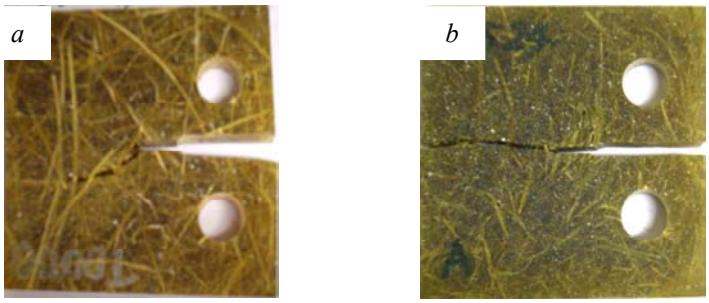

Figure 3. Fracture path observed in CT specimens (a) not-treated fibers composites and (b) treated fibers composites

2 Conclusions

From this investigation, a number of conclusions can be drawn.

1. The intrinsic fracture toughness in each treated composite improved compared to not-treated material. However, the plateau toughness parameters were strongly infected by the NaOH effect.
2. The treatment of Alfa fibers with a lower or higher rate of NaOH can increase or decrease materials properties.
3. LEFM approach is applied with notable success.

References

[1] Rout J., Misra M., Tripathy S.S., Nayak S.K., Mohanty A.K. The influence of fibre treatment on the performance of coir-polyester composites. *Compos. Sci. Technol.*, **61**, pp. 1303-10 (2001).

[2] Herrera-Franco P.J., Valadez-González A. A study of the mechanical properties of short natural-fiber reinforced composites. *Compos. Part B,* **36**, pp. 597–608 (2005).

[3] Rokbi M., Osmani H., Imad A., Benseddiq N. Effect of chemical treatment on flexure properties of natural fiber-reinforced polyester composite. *Procedia Engineering,* **10***,* pp. 2092-97 (2011).

[4] Rong M. Z., Zhang M.Q., Liu Y., Cheng Yang G., Zeng H.M. The effect of fiber treatment on the mechanical properties of unidirectional sisal-reinforced epoxy composites. *Compos. Sci. Technol.*, **61**, pp. 1437-47 (2001).

[5] Kalia S., Kaith B.S., Kaur I. Pretreatments of natural fibers and their application as reinforcing material in polymer composites-A Review. *Polym. Eng. Sci.,* **49**, pp. 1253-72 (2009).

[6] Abdelmouleh M., Boufi S., Belgacem M.N., Dufresne A. Short natural-fibre reinforced polyethylene and natural rubber composites: Effect of silane coupling agents and fibres loading. *Compos. Sci. Technol.,* **67**, pp. 1627–39 (2007).

[7] Manikandan Nair K.C., Diwan S.M., Thomas S. Tensile Properties of Short Sisal Fiber Reinforced Polystyrene Composites. *J. Appl. Polym.* Sci., **60**, pp.1483–97 (1996).

[8] Paiva M.C., Ammar I., Campos A.R., Ben Cheikh R., Cunha A.M. Alfa fibres: Mechanical, morphological and interfacial characterization. *Compos. Sci. Technol.,* **67**, pp.1132–38 (2007).

[9] Bessadok A., Roudesli S., Marais S., Follain N., Lebrun L. Alfa fibres for unsaturated polyester composites reinforcement: Effects of chemical treatments on mechanical and permeation properties. *Compos. Part A*, **40**, pp.184–95 (2009).

[10] Alvarez V.A., Vázquez A. Influence of fiber chemical modification procedure on themechanical properties and water absorption of MaterBi-Y/sisal fiber composites. *Compos. Part A*, **37**, pp.1672–80 (2006).

[11] Ben Brahim S., Ben Cheikh R. Influence of fibre orientation and volume fraction on the tensile properties of unidirectional Alfa-polyester composite. *Compos. Sci. Technol.,* **67**, pp.140–47 (2007).

[12] Rokbi M., Osmani H., Benseddiq N., Imad A. On experimental investigation of failure process of woven-fabric composites. *Compos. Sci. Technol.,* **71**, pp. 1375–84 (2011).

[13] ASTM standard E399-90. Test method for plane-strain fracture toughness of metallic materials. (1993).

[14] Srawley J.E. Wide range stress intensity factor expressions of ASTM E399 standard fracture toughness specimens. Int. J. Fract., **12**, pp.475–6 (1976).

[15] Mishra S., Mohanty A.K., Drzal L.T., Misra M., Parija S. Studies on mechanical performance of biofibre/glass reinforced polyester hybrid composites. *Compos. Sci. Technol.,* **63**, pp.1377–85 (2003).