Sponge Gourd (*Luffa Cylindrica*) Reinforced Polyester Composites: Preparation and Properties

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ABSTRACT

Increasing environmental concern along with the drive to find substitutes for synthetic fibers and value added applications for low cost and renewable plant fibers have led to the development of composites based on biomaterials. One of the drawbacks encountered in such exercise is the lack of adhesion between the incorporated plant fibers and synthetic polymeric matrices. Such drawback can be reduced by appropriate treatment of fibers. This paper describes the chemical treatments used on sponge gourd (*Luffa cylindrica*) fibers of Brazil to prepare their composites with polyester resin. Production of short fiber-polymer composite as well as mat-polyester composites is presented here. Characterization of the composites in respect of evaluation of density, water absorption, thermal stability, tensile properties and impact strength were made and the results are discussed. Observed impact strength and tensile properties are discussed based on the fractographic studies of the composites.

Keywords: Fibers, polymer composites, surface treatment, mechanical properties, scanning electron microscopy, environment, green technology

1. INTRODUCTION

In recent years, there is increasing environmental concerns, greater attention are being paid towards the use of plant fibers taking the advantages of their abundance and availability as renewable resource and their biodegradability in the environment and economical for their cost effectiveness. Accordingly, a number of review papers have been published on plant fiber-polymer composites including biodegradable composites[1-7]. However, the use of plant fibers as reinforcements in composite materials requires extra care regarding fiber / matrix adhesion particularly when the matrix is hydrophobic in nature[6]. This is a consequence of the fact that strongly polarized cellulose fibers (hydrophilic) are inherently incompatible with hydrophobic polymers. The main chemical bond theory alone is not sufficient to elucidate the complex adhesion issue in composite materials and considerations of acid-base reactions at the interface. As a matter of fact, interface morphology and surface energy and wetting characteristics are all part of the interfacial phenomena[2]. Several authors have investigated fiber-matrix adhesion in natural fiber-polyester composites including *Luffa* fibers through different types of surface treatments. The modification of surface includes both physical treatment (e.g., corona discharge) and chemical (e.g., alkali treatment,

benzyl alcohol dewax treatment, acetylation, silane coupling agents, etc.) or thermal treatment. All these treatments as per requirements have usually resulted in changes in the fiber surface structure, their surface energy, crystallinity, chemical composition to influence the mechanical and other properties like water absorption characteristics[8-25]. It is well known now that due to the surface treatments to the lignocellulosic fibers, good compatibility between fibers and non-polar polymer matrices would be achieved through polymeric chains, which will favour entanglements and interdiffusion with the matrix. Besides, these surface treatments of the fibers remove some of the materials on the surface of the fibers, probably some times leading to defibrilation of individual elementary fibers. Such fibers increase the fiber-matrix interfacial area and also increase the area of load transfer from the fibers to the matrix. It is also reported that some of the chemical treatments such as mercerization removes the lignin and hemicellulose from the lignocellulosic fibers, which affects the chemical composition of the fibers, molecular orientation of the cellulose crystallites in the fibers and degree of polymerization[25]. Accordingly, various techniques have been used to monitor the treatment carried out by both chemical and physical methods, with the former providing information about the extent of reaction

and the nature of chemical groups and products got extracted from the fiber surfaces, the latter reveals mainly the structural changes undergone by the fiber. All the above aspects have been documented in some of the published reviews[26-30].

Sponge gourd, or vegetable sponge, belongs to the 'Cucurbitaceae' family and is mostly grown in sub-tropical regions in countries such as Brazil, China, Korea, Japan and a few from the areas of Central America. Despite a few studies on Brazilian fibers particularly on sponge gourd (*Luffa cylindrica*), hereafter referred to as '*Luffa*' fibers[11,15,16,21-24], not much attention is drawn to this fiber which could lead to better commercial exploration or value addition because of the lack of research, unavailability of technical data about its properties and behavior besides lack of technological information on the processing and production parameters for such composite materials.

Two studies have been undertaken in authors' university on this fiber. In the first, the authors' have characterized the '*Luffa*' fibers for various properties with and without chemical treatments using NaOH and methacrylamide on them[23] with a purpose to find increase the utilization for these fibers particularly in polymeric biocomposites. It was found that these surface modifications on the fiber was found to be dependent on the chemical agent, its concentration and treatment time with NaOH solution in the mercerization process. Such methacrylamide treatment could adequately modify the fiber surface without causing much damage to the fiber as the methacrylamide treatment. It was opined that these results could help in the use of these fibers as reinforcements in the preparation of composite materials, which is the main aim of the present study.

In the second study, some preliminary results on its biodegradable composites of '*Luffa*' fiber are obtained. With a view to further consolidate research efforts on Brazilian fibers and their utilization through composite technology, this paper presents results of chemical treatments using NaOH and methacrylamide aqueous solutions in an attempt to improve adhesion between *Luffa* fiber and polyester resin and their effect on physical, thermal and mechanical properties of the referred composites. Also, this is the first attempt to use both short *Luffa* fibers and their mats in the preparation of composites, which may provide an insight into the complexity of their preparation and the comparison of their characteristics. Despite knowing the fact that the short *Luffa* fibers were not effective as reinforcement in previous works[16,21], two types of composites were prepared, one with ground *Luffa* fibers and the other with their original mats keeping in view some degree of fiber orientation shown by mats. The purpose of preparing the composite with fibers alone was to study the effect of chemical treatments attempted on the fibers on the adhesion with the matrix used in randomly distributed short fibers in composites (first type) with a view to avoid the influence of fiber orientation on the results and to ensure an isotropic composite structure and to have a higher homogeneity in the chemical treatment of the fibre surface. On the other hand, composites using *Luffa* mats were prepared with a view to achieve higher tensile and impact properties in the composites.

2. EXPERIMENTAL METHODS

2.1 Materials

Luffa fibers from the Southeast region of Brazil were used as reinforcement in this work. The as received fiber (Fig. 1(a) untreated fiber was ground using a Willy cutter, which showed different fractions with ~ 10% of 40 mesh size, ~ 55% of 60 mesh size, ~18% of 100 mesh size and the remianing more than this. Accordingly, 60 mesh size fibers were used to make mats by compression of either central core, external or internal mat layers. Figures 1(b) and 1(c) show the natural *Luffa* mat with the outer and the inner structure, respectively, revealing distinct fiber distribution patterns.

Unsaturated polyester (ortophthalic) of Araashland, Brazil, was used as the matrix with PMEK peroxide (Araashland), 3% p/p in relation to the resin. NaOH (analytical grade) and methacrylamide (Aldrich) were used for chemical treatments.

2.2 Methods

2.2.1 Chemical Treatment to Fibers

The chemical treatments of the *Luffa* fibers were carried out as follows: First the ground fibers were washed thoroughly in distilled water followed by drying the fibers in air at room temperature for 24 h. Then the dried fibers were immersed in 2% (w/w) NaOH for different periods of time - 10, 60 and
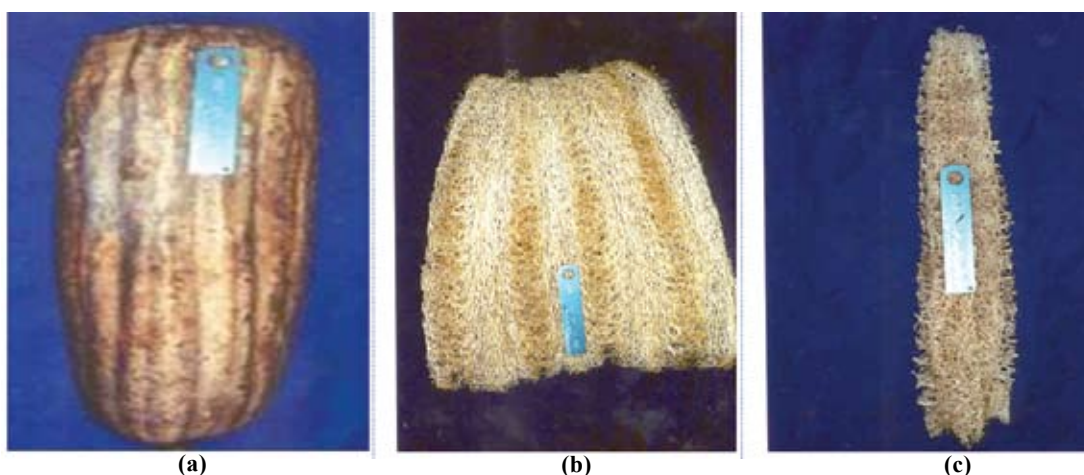

| (a) | (b) | (c) |

Figure 1. Photograph of (a) *Luffa cylindrica* as-received and natural *Luffa* mat-(b) the outer structure and (c) the inner structure.

90 min or in aqueous methacrylamide solutions of different concentrations - 1, 2 or 3% for different times - 60, 120 and 180 min. Then, both the chemically treated fibers were washed with water until a neutral pH was reached followed by drying them at 60 °C for 24 h. The treatment with methacrylamide may be termed as 'copolymerization' or 'grafting'.

2.2.2 Preparation of Composites

Composites were compression molded in a steel mould with an inner cavity of 150 × 150 × 5 (± 1.5 mm). Alternate layers of dried fibers (70 °C for 2 h) and resin (with the initiator) were placed in the mold until the intended amount of material had been used. The mold was closed, placed in a press and subjected to 3.6 MPa pressure for 2 h at 70 °C. The mold was allowed to cool for 1 h and the composite was retrieved. Fiber volume fraction (% V_f) in the composites was kept at 42.6 % and 24.5-35.2 % for the short-fiber and fiber mat composites, respectively. The amount of the fibers in the mats used in accordance with the convenience of molding for the fabrication of the composite.

The composites so prepared were cut to make the sample for the test 150 × 25 mm (length × width) for tensile testing, which was carried in an EMIC DL10.000 universal testing machine with 1000 kgf load-cell and at 50 mm/min strain rate. Charpy impact tests were carried out using unnotched specimens (9 × 2.5 × 1.5 mm) using a PANTEC-PW-4 impact tester with of 1 J hammer at an impact velocity of 2.90 m.s^{-1} following ASTM standard D256-84.

Fiber diameter was evaluated using an OLYMPUS BX-60 optical microscope based on more than 30 observations (with magnification of 2.5×). And the apparent density of the *Luffa* fiber and its composites was determined using a glass pycnometer.

A Phillips Scanning electron microscope (XL 30) was used to observe the fracture composite surfaces coated by gold sputtering.

Differential scanning calorimetric (DSC) analysis of the matrix to determine its curing time and curing temperature were carried out using a Netzsch DSC 209 equipment from 20 to 180 ºC at a heating rate of 10 ºC/min for the sample. The DSC of the matrix was carried out as per ASTM D3418 standard. Thermogravimetric analysis (TGA) of the composites was carried out as per ASTM E1131 from room temperature to 560 °C at a heating rate of 20 °C/min in a N_2 atmosphere.

Water absorption of the composite samples [90 × 25 × 5 mm (± 1.5)] was determined following ASTM D570-95 specification. They were immersed in distilled water at ambient temperature during 35 days (the water was changed every after 7 days). The experimental results obtained were analyzed for statistical significance.

3. RESULTS AND DISCUSSION
3.1 Dimensions and Density

A large variation of fiber diameters was observed in any single fiber and a mean value of 0.631 ± 0.217 mm (maximum = 1.000 mm and minimum = 0.200 mm) was obtained. Such size variation in plant fibers is expected, and such variation contributes to property fluctuations in the fibers as well as in

their composites[1-6].

The apparent density of *Luffa* fiber was determined as 920 (± 0.05) kg.m^{-3}, whereas the density of the treated fibers could not be determined with the described methodology because a large amount of bubbles were formed when they were in contact with the used fluids - water or ethanol. This would invalidate the measurements.

Table 1 shows relative density values of composites containing short-fiber after chemical treatments for various concentration and time of treatments. It can be seen that the short-fiber composites showed low density values though not very significant for composites having different treated fibers. This is understandable, since the fiber itself has a lower relative density than the polyester resin. Similarly, the very low apparent density of the *Luffa* mat structure is due to its sponge like structure.

Reduction in fiber density (up to 6 % weight reduction of fibers for 3 % chemical treatments for 60-180 min was observed on chemical treatments, but no significant reduction for 1-3 % of chemical treatments for 60 min[23]), and consequently of the respective composite, takes place with the chemical treatment due to the removal of lignin and hemicelluloses. Besides, the presence of voids is also playing a role in reducing the density of all the composites.

Table 1. Density values of composites with and without fiber treatment

Type of fiber	Density (kg.m^{-3})
Untreated	1160
NaOH 2% - 10 min	1110
NaOH 2% - 60 min	1100
NaOH 2% - 90 min	1110
Methacrylamide 1% - 60 min	1110
Methacrylamide 1% - 120 min	1100
Methacrylamide 1% - 180 min	1090
Methacrylamide 2% - 60 min	1090
Methacrylamide 2% - 120 min	1100
Methacrylamide 3% - 60 min	1070
Methacrylamide 3% - 120 min	1070
Methacrylamide 3% - 180 min	1070

3.2 Short-fiber Composites
3.2.1 Morphology

Morphology of chemically treated *Luffa* fibers for different conditions has already been reported[23]. The effect of the treatment on the fibers, especially with methacrylamide, was noticed on the appearance of the composites (not shown here), since the process of removing soluble material from the fiber yields a lighter colored fiber and its composite.

Drying of the fibers just prior to composite manufacturing was found to be crucial in improving mechanical properties since moisture increases void content to adversely, interfere with the fiber/matrix interface[2]. Even though the void volume fraction was found to be still high ca. 5.30 % accentuated by the use of untreated polyester resin without even applying

degassing operation, yet it is still within the range mentioned by others[31].

3.2.2 Tensile Properties

Stress-strain curves for the short- *Luffa* fiber composites (with untreated and NaOH treated fibers) showed some amount of non-linearity before reaching the maximum load probably due to incipient damage such as matrix cracking, fiber failure or fiber pull out. These curves showed a similar profile to those reported in the literature for plant fiber composites including biodegradable ones[32]. Besides, almost similar Young's modulus value (~3400 MPa) was observed for the three composites.

Figure 2 shows the mean tensile strength for the different composites as a function of the chemical used. Strength for all composites varied within 16-19 MPa, which is in the same range reported in the literature for polyester composites based on untreated *Luffa* mats (%V_f = 30%) and polyester[11]. The composites exhibited a slight trend towards higher strength for longer times for all treatments, however, with those treated with NaOH showed highest tensile strength.
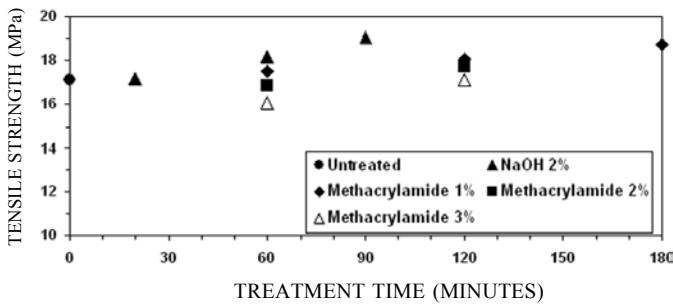

Figure 2. Plots of tensile strength of the short-*Luffa* fiber/ polymer composites showing the influence of chemical treatments on the fiber.

An increase in the concentration of the methacrylamide solution, however, seems to have an inverse effect, which may be a consequence of severe fiber damage as observed earlier[23]. Unfortunately, it was not possible to measure individual *Luffa* tensile strength due to the difficulty in isolating single fibers from the source for testing and therefore this fiber damage (strength reduction) could not be quantified. Although there is evidence of fiber surface modification with methacrylamide[22,23], the use of this chemical is not justifiable since, apart from its relatively high cost, the improved adhesion does not yield enough strengthening in the composite. Perhaps, if one were to use methacrylamide treatment, one should opt for a low concentration for longer periods to reach an optimum tensile strength.

NaOH treatment or methylacramalide treatment of *Luffa* fibers also alters composition and morphology of the fiber[23]. This is the result of extraction of waxy layer/amorphous regions, besides the partial dissolution of lignin and/or development of crystalline cellulose and probable increase in molecular orientation during the process of stretching the fiber causing realignment of the molecular configuration to effect improved mechanical properties in the fibers[12,25]. Nevertheless, NaOH treated fibre appeared to be more effective in increasing tensile strength than the corresponding methacrylamide treated

fibre. Similar results have been reported for different fiber composites including those with *Luffa* fibers[10,15,16,22,24-30], while change of crystallinity of the lignocellulosic fibers with surface treatments with consequent change in mechanical properties of their composites have also been reported[33-35].

However, it is important to keep in mind that the observed increase in tensile strength is only a trend, since statistical analysis did not show significant differences between any of the conditions studied here. Therefore, considering only tensile strength (TS), it can be stated that the *Luffa* short fibers (length mainly in 0.25-0.42 mm range) do not appear appropriate to reinforce the polyester matrix in conformity with the earlier reports[15,21], which is stronger by itself (TS = 19.4 MPa) than most of the composites produced. Therefore, it may be worthwhile to use this fiber in combination with other stronger fibers such as glass fibers, in order to obtain reasonable mechanical properties.

Impact properties of the short *Luffa* fiber composites showed more scattered results (Fig. 3). It should be noted that the error bars are not included in the figure as is normally done with this kind of plots. Instead a more complex statistical analysis (ANOVA) was carried out. From Fig. 3, no significant differences are found when statistical treatment was applied. Thus, no clear trend could be established for neither NaOH nor methacrylamide treatments.
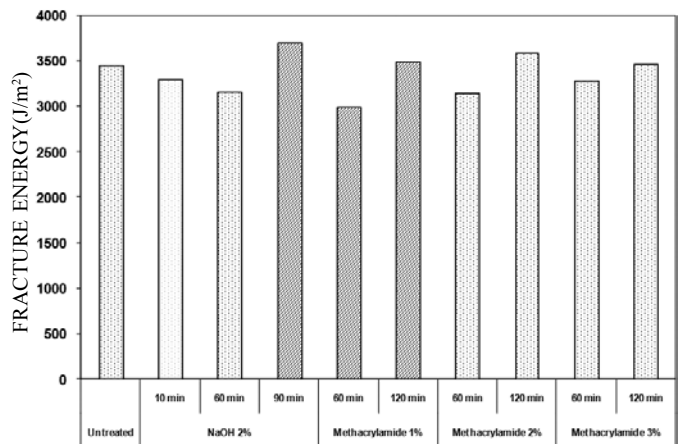

Figure 3. Energy of fracture for the NaOH and methacrylamide treated *Luffa* fiber Composites.

Nevertheless, the NaOH treatment for 90 min showed the highest average fracture energy (3710 J/m²), slightly higher than that for the untreated fiber composite (~3500 J/m²).

The above can be discussed in the light of the fractographic studies carried out. Figure 4 shows the fracture surface of composites with (a) untreated *Luffa* fiber, (b) *Luffa* fiber treated with 2% NaOH solution for 90 min and (c) fiber treated with 1% methacrylamide solution for 60 min. Many fiber pull-outs (e.g. shown by the solid arrow), i.e. cavities due to fiber debonding, can be seen in the untreated fiber composites (Fig. 4(a)) indicating poor fiber/matrix adhesion. Also, analysis of the fracture area reveals the presence of voids with sizes in the range of 40-50 μm (darker areas in SEM micrographs - shown by the dotted arrow). Both contribute to the low mechanical strength of the material and its brittleness. Even
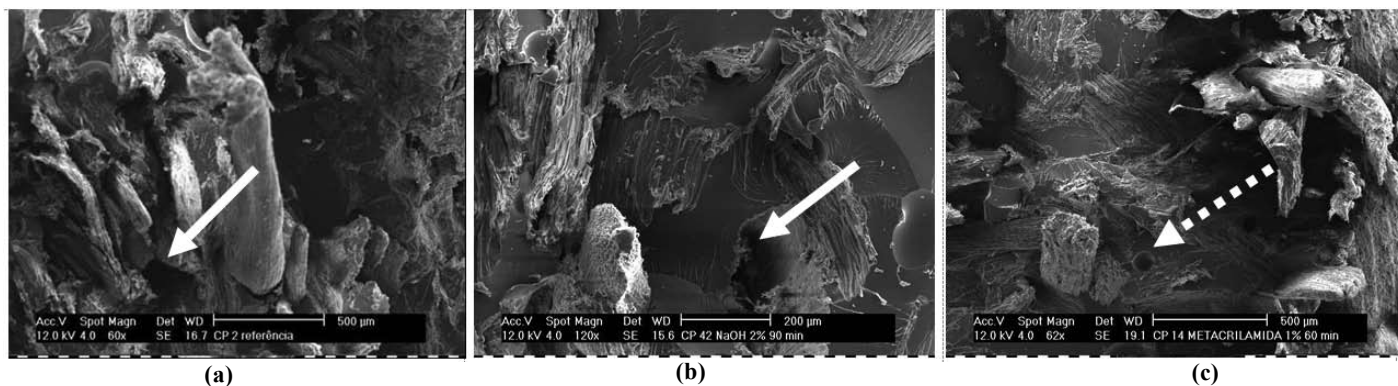
Figure 4. SEM fractographs of the fractured surface of polyester short-*Luffa* fiber composites: (a) untreatead *Luffa* fiber; (b) *Luffa* fiber treated with NaOH (2 % - 90 min) and (c) *Luffa* fiber treated with metacrylamide (1% - 60 min).

composites prepared with methacrylamide-treated fibers (1% for 60 min) seem to exhibit better adhesion with less pull-out sites (Fig. 4(c)) compared to those with NaOH-treated *Luffa* fiber (2% up to 90 min) (Fig. 4(b)) as evident from the gap between the fiber and the matrix and presence of cavities due to fiber debonding.

The above results indicate that the chemical treatments given to Luffa fibers did not produce the intended characteristics in a homogeneous way, that is, uniform increase of roughness, cleaning of the fiber surface and activation of surface functional groups, which otherwise would have contributed to enhanced mechanical strength.

3.2.3 Thermal Studies

DSC analysis of the resin and that of composite with both treated and untreated *Luffa* fibers indicated that the time and the temperature used in the present study to prepare the composites were within the reported parameters for curing of the matrix. For example, DSC analysis of the resin kept at 70 °C revealed that the cure process began immediately after mixing as an exothermic process, lasting for about 10 min and completed at 120 °C. These results are in agreement with the data provided by the manufacturer concerning the gel time of the resin, which is between 9-11 min. However, the completion of the resin cure at 170 °C, as indicated by the resin manufacturer could enhance a degradation process of the *Luffa* fibers and so, processing by compression molding of the composites was made at 70 °C. Therefore, DSC analysis of composites with untreated *Luffa* fibers suggested that the composites were not completely cured around 80 °C, what might have contributed to the mechanical properties results, although as shown by TGA, there is no significant degradation of fibers until 250 °C.

Figure 5. shows the TGA curves for the three types of composites. It can be seen that the variations were not significant up to 230 °C. However, above this temperature, the composites prepared with treated *Luffa* fibers showed slightly lower mass loss than the untreated fiber composite. All the composites showed a similar profile indicating homogeneous behavior during thermal decomposition, with severe degradation starting around 270 °C.

Regardless of the treatment, all thermograms are typical of lignocellulosic materials, and the mass loss at each temperature range can be associated with the characteristic constituents of
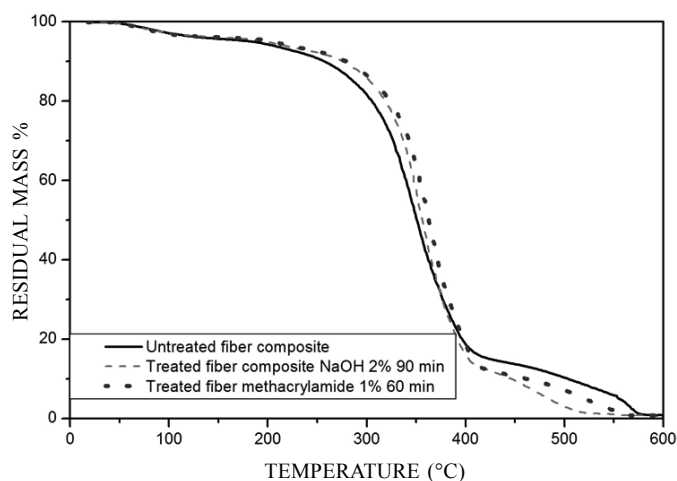

Figure 5. TGA curves of the composites, (i) untreated *Luffa* fibers, and (ii) NaOH treated and (iii) methacrylamide treated fibers.

the fibers. Up to 100 °C, mass reduction is related to the loss of moisture and volatile substances. Mass loss values observed at this stage were higher, for untreated fiber composites and those with methacrylate treated fibers (~8 to 8.7%) compared to those with NaOH treated fibers (~7 %). On the other hand, similar mass loss values of ~70-71 % was observed between 250-360 °C for the composites containing chemically treated fibers compared to ~64 % for the composites with untreated fibers. Similarly, almost similar mass loss values of ~21 % was observed between 360-580 °C for the composites containing chemically treated fibers compared to ~27.5 % for those containing untreated fibers. These two mass losses are related to the decomposition of cellulose and lignin, respectively[36]. These temperature ranges are slightly different from 240–350 °C and lignin degradation at 280–500 °C reported for *Luffa* fibers and other plant fibers[23,36].

In addition, there was mass loss between 200-260 °C due to hemicelluloses in the case of fibers. Besides, the polyester also decomposes and part of the observed weight loss is due to its contribution.

3.2.4 Water Absorption Studies

One of the important properties to be evaluated for composites reinforced with plant fibers such as *Luffa* is water

absorption, since it can limit their use. The fact is that the plant fibers can easily absorb water and that is actually one of the reasons for the requirement of treatment of fiber. The treated fibers may absorb less moisture thus favoring adhesion with the polymer matrix and can provide better performance in humid environments.

Figure 6(a) shows the fiber weight gain for untreated and 2 % NaOH treated fiber composites, while Figure 6(b) shows the same for methacrylamide treated fiber composites with similar % fibre volume, V_f in all composites. It can be seen that both untreated and NaOH treated fiber composites samples showed an initial rapid weight gain up to about 7 days of exposure. The elimination of residual styrene present in the resin was observed during the first 100 h. In addition, water turned hazy indicating loss of materials (e.g. some of the resin, of the residual styrene or soluble materials of the fibers). There could be 3–5% mass loss in the case of all composites.
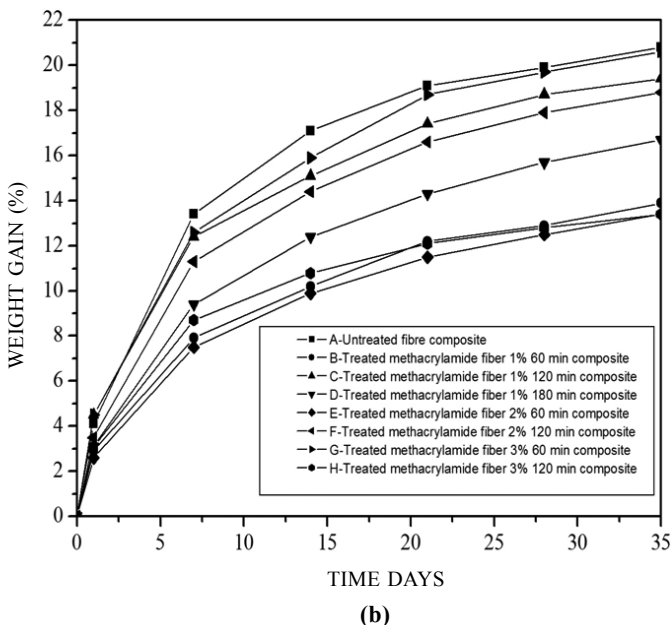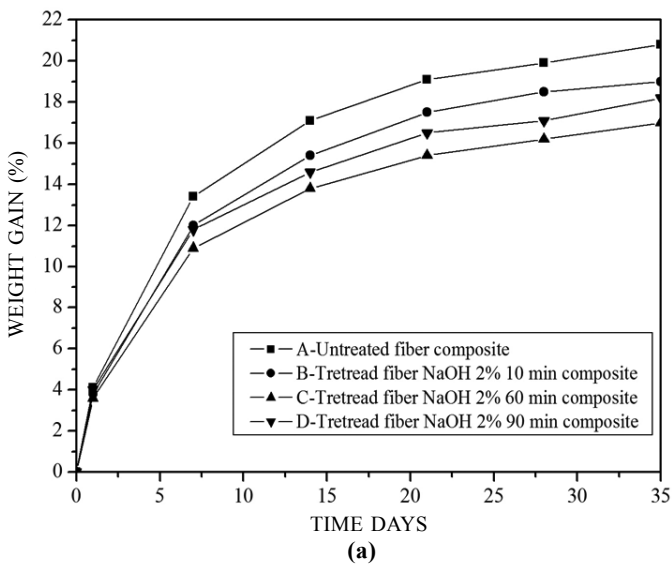

(a)

(b)

Figure 6. Weight gains for the composites containing (a) untreated and NaOH treated *Luffa* fibers and (b) methacrylamide treated *Luffa* fibers.

The weight gain of the composites varied with the type of treatment. For example, for NaOH treated fiber composites, it was 17.5 to 19.3 %. However, it was 13.0-14.5 % for shorter periods of treatment and 13.0-21.0 % for longer periods for methacrylamide treated fiber composite, the gain increasing with treatment time. Methacrylamide treated fiber composites showed comparatively slower weight gain in the beginning, reaching almost constant values after 7 days of exposure only.

The high water absorption uptake of the composites may be an indicative of difficulties during processing, such as incomplete curing of the thermoset matrix[31] or of the presence of voids or cracks or even poor matrix/fiber adhesion[10,24].

Reduction of hydrophilicity of the fibers indicates that methacrylamide treatment at distinct conditions (~60 min) was more effective in reducing the water absorption than NaOH treatment. These results are similar to those reported earlier for polyester composites containing *Luff* fibers[11,15,16] and other fibers, such as sisal, coconut, jute and ramie[8,10,18].

3.3 Fiber Mat Composites

By using the original *Luffa* mat, one may take advantage of the natural mat structure either planar random (inner mat) or directionally oriented (outer mat), the latter could result in composites with good directional mechanical properties[37]. Besides, the cutting procedure is not a necessary step to produce the fibrous reinforcement when using *Luffa* mats and it is easier to handle mats than short fibers.

Only composites with mats, treated with 2 % NaOH for 90 min showing the most promising results for short-fibers, were produced using various fiber mat volume fractions. The initial mat composites showed a porous appearance and this was minimized by pre-wetting the *Luffa* mats in resin for about 30 min under pressure of approximately 50 kg to ensure better wetting. The composites were tested for tensile properties only.

Figures 7 (a) and 7 (b) show the mean values of tensile strength and Young's modulus, respectively, obtained for composites prepared with fiber mats.

Considering the range of volume fractions studied, analysis of these figures reveal: (i) higher the volume fraction, higher is the tensile strength; (ii) fiber surface treatment with 2% NaOH aqueous solution seems to yield the most favourable material for easy impregnation of the resin during the preparation of the composites; (iii) chemically treated fiber mat composites show higher tensile strength (~22 MPa) and Young Modulus (~5200 MPa) compared to those of untreated fiber mat (TS: ~14 MPa ;YM:~3200 MPa); (iv) The elongation at break of the composites with the chemically treated mats (0.48 %) and that of the composites with untreated mats (0.56 %) are similar, both being slightly higher than that of matrix only (0.34 %). These results suggest that the orientation of the natural structure of *Luffa* reduces brittleness of the polyester matrix based composites material; and finally (v) although the scatter in the results has decreased for composites with treated *Luffa* fibers, it is still large due to the inherent variation of fiber properties, mat structure and also as a consequence of the composite preparation.

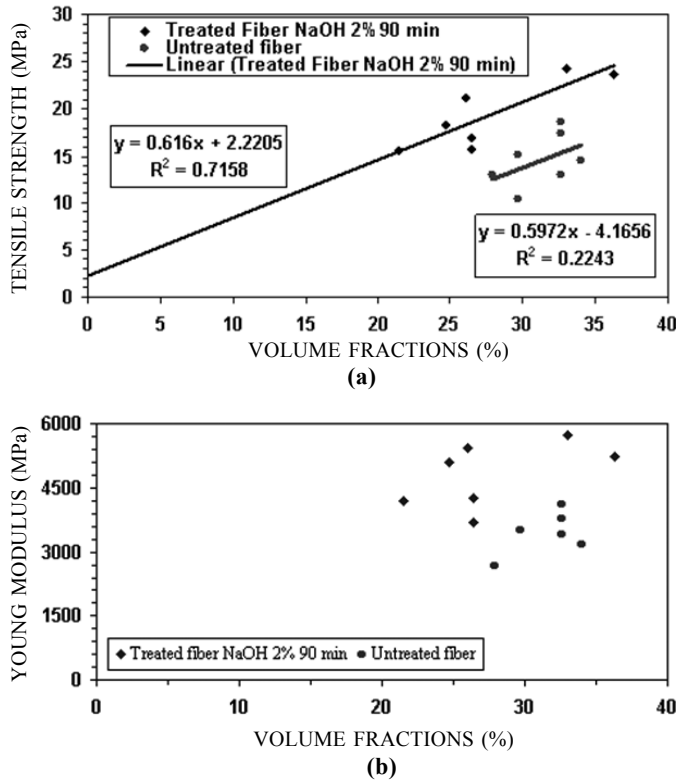Furthermore, a more controlled fracture mode (type III)

Figure 7. (a) Tensile strength and (b) Young modulus of polyester composites with *Luffa* fiber mats for different fiber volume fractions.

was observed during tensile testing of the mats, unlike the brittle and sudden fracture mode (type I) observed for the short-fiber composites.

4. CONCLUSIONS

It was possible to prepare composites with both short fibers and mats of *Luffa cylindrica* with polyester matrix by compression molding. Surface treatment of *Luffa* fibers with 2% NaOH for 90 min was found to be the best treatment with reference to highest fracture energy. However, no significant increase in tensile strength of the composites was observed compared to untreated fiber composites.

These composites presented water absorption similar to those of other plant fiber like sisal-, jute- or rami- polyester composites. Nevertheless, fiber treatment with methacrylamide was efficient in decreasing water absorption of the composites. The composites containing 35.3% volume fraction of *Luffa* mats treated for 90 min with 2% NaOH aqueous solution exhibited the best tensile properties although still lower than those of other plant fiber composites. In view of the good appearance of *Luffa*/polyester composites prepared with short fibers, their use as ornamental panels, linings and products without structural function may be envisaged.

ACKNOWLEDGEMENTS

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