Chemical Modification of Urena lobata (Caezer Weed) Fibers for Reinforcement Applications

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Abstract
The reinforcements of composites with natural fibers have attracted more concerns than their synthetic counterparts due to the biodegradability, less-expensiveness, CO₂ neutrality, ready availability, non-abrasiveness, low weight, renewability and high specific mechanical properties of these fibers. However, the hydrophilicity, reduced interfacial bond, and reduced thermal strength of fibers of natural origin deter their attraction for use in composites reinforced with natural fibers. The treatment of naturally-occurring fibers chemically helps to clean up the fiber surface making them rougher; reduces moisture absorption; and increases fiber-matrix adhesion. In this study, Urena lobata fibers produced by natural water retting for use as composite reinforcements were subjected to alkaline-treatment with sodium hydroxide solutions at varying concentrations. The transformations that occurred in the structures and morphologies of the fibers were checked using scanning electron microscopy (SEM). Tests were done on several bundles of the Urena lobata fibers so as to understand how the chemical treatments had effects on the tensile behaviours of the fibers. The results obtained reveal enhanced mechanical behaviours of the treated Urena lobata fibers in comparison to the untreated ones. Interestingly, the chemical treatment with 6 wt% NaOH solution yielded the best mechanical behaviours (2.91 ± 0.01%, 53.26 ± 0.01 GPa and 2611.34 ± 0.045 MPa for ductility, elastic modulus and tensile strength, respectively) amongst other alkaline-treated fibers.

Keywords: Urena lobata, natural fibers, alkaline-treated

1. Introduction
Urena lobata, whose alternative names are; caeser weed, congo jute and aramina, is a member of the Malvaceae family and belongs to the class of bast fibers. The plant wildly grows in hot and sub-tropical areas of the universe, but more especially in tropical Africa, such as Nigeria, Congo, Ghana, Madagascar, amongst others [1]. Urena lobata is a perennial herbaceous plant that grows up to 2-5 meters high, with leaves varying in size and shape. The fiber resembles jute, roselle and kenaf in strength and appearance. Historically, the fibers are used in making fishing nets and lines, sacks, cordage, carpets, twines, ropes, strings; while the stems are used as tying materials [1]. Urena lobata fibers can serve as reinforcements in polymeric matrices for composite production in place of glass fibers [2,3]. This is because being a natural fiber; it has more advantages than the synthetic fibers such as glass, in being, readily available, less expensive as compared to synthetic fibers, non-toxic, biodegradable, non-carcinogenic, good mechanical properties, and can also be recycled [4,5]. The superiority of the fiber-matrix adhesion is
essential in transferring load in fiber reinforced composites [6]. However, the main challenges encountered in using naturally-occurring fibers in polymeric matrices are always reduced adhesion to the polymer matrices, high moisture absorption due to their hydrophilicity, and their inability to withstand high temperatures [3,7]. All these reduce the mechanical behaviours of the produced composite materials [8]. For these limitations to be overcome there have been several reports from some researchers on the chemical treatments of naturally-occurring fibers consisting of banana, jute, kenaf and sisal [9-11]. Currently there are no reports on chemical treatment of *Urena lobata* fibers in open literature. Against this background, this study reports on using the solution of sodium hydroxide for the treatment of the *Urena lobata* fibers because of the effectiveness and simplicity of the method. Various authors have written concerning the alkaline treatment of naturally-occurring fibers with varying concentrations of NaOH; kenaf treated with 3 wt.%, 6 wt.%, and 9 wt.% concentration of NaOH [10], coir treated with (2 wt.%, 4 wt.%, 6 wt.%, 8 wt.%, and 10 wt.% concentration of NaOH [12], and alfa treated with 1 wt.%, 3 wt.%, 5 wt.% and 7 wt.% concentration of NaOH [13]. In this study, 3 wt. %, 6 wt. % and 9 wt. % concentration of NaOH were used for the chemical treatment of fibers from *Urena lobata* plants and the optimal NaOH concentration determined. In addition, this study investigated the response of *Urena lobata* fibers to the solution of NaOH; and how the concentration affects the mechanical strength and morphology of *Urena lobata* fibers.

2. Methodology

2.1 Extraction of *Urena lobata* fibers

The *Urena lobata* plants were obtained from Obinze, Imo State Nigeria, (geographical coordinates as 5° 25’ 0” North, 6° 58’ 0” East). The plants were identified in Crop Science department, Federal University of Technology Owerri, Imo State, Nigeria. The freshly cut stems of young plants, that are not up to one year, were placed in a running stream for about 14 to 16 days to ret. After which, the retted fibers were rinsed in water and open-dried.

2.2 Alkaline treatment of *Urena lobata* fibers

NaOH solution having concentration of 3 wt%, 6 wt% and 9 wt% were used in the treatment of the *Urena lobata* fibers at ambient temperature for about 2 hours. Once the treatment was done, the *Urena lobata* fibers were cleansed with distilled water so as to take out all the NaOH solution, and then dilute acetic acid was used neutralization and then later washed with water to ensure the pH of 7. Then the alkaline-treated *Urena lobata* fibers were dehydrated at 70°C for 72 hours [14], [15].

2.3 Mechanical analysis of *Urena lobata* fibers

Monsanto Tensometer Tensile Tester (model A104) was used to check the mechanical behaviours (ductility, elastic modulus and tensile strength) of the non-treated (untreated) *Urena lobata* fibers and the untreated ones. The average diameter (D) of the fibers was obtained, and the cross sectional area (A) was determined as follows:

\[
\text{Cross sectional Area, } A = \pi r^2; \tag{1}
\]

where *A* is the cross sectional area; *r* is \(\frac{D}{2}\); *D* is the diameter, \(\pi = 3.142\)

The tests were carried out in triplicates for accuracy of data.
2.4 Morphological characterization
SEM analysis was carried out on the treated and untreated Urena lobata fibers to understand the influence of the varying concentration of NaOH on the treated fibers. Aluminum stubs were used in mounting the treated and non-treated Urena lobata fibers with the help of carbon tapes and gold-palladium (AuPd) was used for double coating. A Carl Zeiss Sigma Field Emission Scanning Electron Microscope (FESEM) (Oxford X-ACT) operated at a voltage of 5.00 kV was used for the analysis.

3. Results and discussions

3.1 Mechanical properties of the fibers
The results of the mechanical tests (ductility, elastic modulus and tensile strength) of the differently treated Urena lobata fibers and the non-treated Urena lobata fibers are presented Table 1. From the results, the mechanical behaviours (ductility, elastic modulus and tensile strength) of the non-treated Urena lobata fibers are more than those of the treated Urena lobata fibers at 3% NaOH. This implies that instead of treating the fibers with concentration as low as 3% of NaOH, it would be better to use the fibers as untreated for composite production to ensure better mechanical properties. The treated fibers at 6% NaOH gave the best mechanical behaviours (ductility, elastic modulus and tensile strength) having the highest values amongst all the mechanical properties (behaviours). Thus the cleansing of the impurities from the fiber surface gave rise to more exposure of cellulose and enhanced the mechanical behaviours of the fibers that were treated. The optimum NaOH concentration established in this work is in agreement with reports from published works [10, 16-19]. However, the treated Urena lobata fibers at 9% NaOH showed a reduction in the mechanical behaviours in the mechanical behaviours (ductility, elastic modulus and tensile strength) as compared to the fibers treated at 6%. Although, the ductility (or elongation at break) and tensile strength for the Urena lobata fibers treated at 9% are more than those of the non-treated fibers, but the cell walls were much more degraded resulting in the reduction in stiffness (reduced elastic/Young’s modulus) as compared to that of the untreated and Urena lobata fibers treated at 3% NaOH.

Table 1: Mechanical behaviour of non-treated and differently treated Urena lobata fibers

| Sample      | Diameter (mm) | Ductility (%) | Elastic modulus (GPa) | Tensile strength (MPa) |
|-------------|---------------|---------------|-----------------------|------------------------|
| Non-treated | 0.17 ± 0.006  | 2.45 ± 0.010  | 48.31 ± 0.01          | 2380.12 ± 0.015        |
| 3% treated  | 0.15 ± 0.006  | 2.41 ± 0.015  | 42.05 ± 0.01          | 2014.16 ± 0.015        |
| 6% treated  | 0.22 ± 0.006  | 2.91 ± 0.010  | 53.26 ± 0.01          | 2611.34 ± 0.045        |
| 9% treated  | 0.21 ± 0.010  | 2.59 ± 0.006  | 44.61 ± 0.01          | 2530.24 ± 0.030        |

3.2 Surface morphology of the fibers
The surface of the untreated Urena lobata fibers (Figure 1(a)) is relatively smooth and clear, indicating that the constituent materials and other impurities on the surface are still intact [19,20]. As can be seen
from Figure 1(b), there is evidence showing the unevenness of the outer layer of the fiber; which indicates the partial cleansing of the outer materials at 3% NaOH concentration. Furthermore, from Figure 1(c), a rougher surface can be seen by treating the fibers with 6% sodium hydroxide concentration. The rough surface would thereby enhance the interfacial bond between the fibers of *Urena lobata* fibers and the polymer matrices when the fibers are used in making composite materials. Also the roughness of the surface indicates enhancement of mechanical intertwine ment involving the *Urena lobata* fibers and the matrix [21]. However, the treatment with 9% NaOH concentration resulted in the degradation of the *Urena lobata* fibers as shown in Figure 1(d). This severe degradation on the *Urena lobata* fibers could result in the reduction of the tensile behaviours in comparison with that of the 6% treated fibers, as presented in Table 1.
4. Conclusions
The alkaline treatment of *Urena lobata* fibers was carried out using varied concentration of NaOH solution. Mechanical tests (ductility, elastic modulus and tensile strength) were done on the non-treated and treated *Urena lobata* fibers. Results showed that fibers treated at 6% NaOH displayed better mechanical behaviours (ductility, elastic modulus and tensile strength) as compared to the non-treated *Urena lobata* fibers. Furthermore, the fibers treated at 6 wt% NaOH possess the best mechanical behaviours in comparison with the *Urena lobata* fibers that are treated at 3 wt% and 9 wt% NaOH. SEM micrograph of *Urena lobata* fibers treated at 6% NaOH was rougher than that of the 3% treated fibers. At 9% NaOH solution, degradation of the fibers, was observed which gave rise to poor performance of the mechanical behaviours of the treated *Urena lobata* fibers.

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