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# Fracture Toughness Properties and FTIR Analysis of Corn Fiber Green Composites

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***Abstract*—**The present work introduced a green composite consisting of corn natural fiber of constant concentration of 10% by weight incorporation with poly methyl methacrylate matrix biomaterial prepared by hand lay-up technique. Corn natural fibers were treated with two concentrations of sodium hydroxide solution (3% and 5%) with different immersed time (1.5 and 3 hours) at room temperature. The fracture toughness test of untreated and alkali treated corn fiber composites were performed. The effect of chemically treated on fracture properties of composites has been analyzed using Fourier transform infrared (FTIR) spectroscopy. The experimental results showed that the alkali treatment improved the fracture properties in terms of plane strain fracture toughness $K_{IC}$. It was found that the plane strain fracture toughness $K_{IC}$ increased by up to 62% compared to untreated fiber composites. On the other hand, increases in both concentrations of alkali solution and time of soaking to 5% NaOH and 3 hours, respectively reduced the values of $K_{IC}$ lower than the value of the unfilled material.

***Keywords*—**Green composites, fracture toughness, corn natural fiber, Bio-PMMA.

## I. Introduction

THE green composites made from natural fibers as a reinforcement material and biopolymer as a matrix material constitute a current area of interest in research. It has been noticed that these composites are not only biodegradable and renewable materials but also possess better electrical resistance, chemical resistance, and good thermal insulating than for composites reinforced with synthetic fibers such as glass fiber [1]. Also, they have other advantages such as low price, lightweight, non-abrasiveness, high specific strength, and save fabrication process as well as lower pollutant emissions, lower greenhouse gas emissions, and enhanced energy recovery [2]-[5]. Numerous applications of natural fiber green composites can be found in construction, furniture, automotive industries, and packaging fields. An important development has been observed in packaging.

In contrast to metals, the interest in natural fiber green composites is still in the beginning in terms of fracture mechanics application. The study of how material fracture is known as fracture mechanics [6]. Fracture mechanics provides a means of quantifying the toughness of a material by considering the conditions under which a pre-existing crack began to propagate unstably [7]. In field of composites, the resistance to fracture is determined in terms of stress intensity factor $K_{IC}$ and J- integral [8], [9]. In general, the mechanical properties of composite materials reinforced with short fibers can be considered as an isotropic material. Thus the behavior of this material is similar to that for a homogeneous material and the manufacturing process techniques for those can be applied without large difficulties [10].

The major problem of natural fibers when used for reinforcement is poor compatibility with thermoplastic materials due to their hydrophilic nature of plant fiber surface and contain amorphous substances in their composition such as lignin, hemicellulose, pectin, and waxes. For toughening, crack bridging can be considered the most prominent mechanism, by utilizing fibers with weak fiber/matrix interface.

When the matrix material fails, the fibers are left intact spanning the crack wake and can act as bridges to inhibit crack opening [11]. Therefore, to eliminate this problem it is necessary to apply various chemical treatments to alter their chemical composition such as mercerization, salinization, acetylation, and others thereby enhance the bonding strength between the natural fiber surface and thermoplastic matrix. Mercerization or alkali treatment Alkali treatment is one of the most used chemical method to produce high quality plant fibers when used as reinforcement in thermoplastic polymer matrix to fabrication composites [12]-[14]. The advantages done by alkaline treatment are the distribution of hydrogen bonding in the network structure thereby increasing fiber surface roughness [15], increasing the cellulose content due to removal of undesired components such as lignin, wax, and oil covering the external surfaces of the fiber cell wall, lowering fiber diameter thereby increasing aspect ratio via forming shorter fibers with increased effective surface area, and reducing the ability of natural fiber to water absorption [16], [17].

The aim of current work was to develop a new green composites based on biopolymethylmethacrylate resin reinforced with a constant weight fraction of randomly short corn natural fibers. Fracture toughness results and FTIR analysis were determined at two different concentrations of surface fiber treatment (3 and 5% NaOH) with various time of soaking (1.5 and 3 hours).

## II. Experimental

### *A. Resin and Fibers*

Bio-polymethylmethacrylate (Bio-PMMA) matrix materials was investigated in this work. The thermoplastic resin consists of two components, 617H55 resin and 617P37 hardener were

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supplied by Otto Bock Healthcare plc, Egham, Surrey, UK. Corn cobs were used as reinforcement. These natural fibers were obtained from local shops (Glasgow-Scotland), the fibers were extracted from the cobs and then dried at 37 ˚C for 240 hours in an incubator (Sanyo MIR262, Japan) before surface treatment. All the corn natural fiber types were cut in length of 10 mm.

### *B. Mercerization Treatment*

The corn natural fibers were soaked in NaOH solution with different concentrations (3 and 5 % wt) at room temperature for 90 minutes. After washing with distilled water, the fiber was dried at 37 °C for 240 hours and then collected in the form of a braid and cut to 3-5 mm lengths in an incubator.

### *C. Preparation of Composites*

A natural fiber green composite samples are prepared by hand lay-up fabrication technique. Calculated amounts of resin and hardener (100:3 by weight) were thoroughly mixed, using gentle stirring to minimize air entrapment. After the mixing process, a thin layer (≈2 mm thickness) of the doughy mixture was placed into the thermoplastic mold. The using mold made from polytetrafluoroethylene materials with dimensions 220 × 110 × 3 mm was fabricated. First, short corn natural fibers of length 5 mm were distributed uniformly over the resin/hardner mixture, then the remainder of the mixture was poured directly into the mold and the pressure of 27.5 MPa for 30 minutes were appiled. After the pressure is applied, the material was allowed to cure at room temperature for 1 day. Finally, the casting samples were taken out of the mold and machined both holes and notch into the required sizes for ASTM E647-13a standard test. The prepared green composites were manufactured with constant weight fraction of 10% wt natural corn fibers with or without alkali treatment. Non-filled PMMA was manufactured in the same manner to provide 0% filler samples.

### *D. Materials Characterization*

FTIR spectra of the prepared corn fiber green composites were recorded with Bruker Tensor 27 Fourier Transform Infrared Spectrophotometer. In this analysis, transmission measurements require a short path length, and this can be obtained by pressing the sample into the device. The measuring polymer spectra is attenuated total reflectance (ATR). No test sample preparation is needed with this technique, providing high quality assurance spectra data.

For compacting tension test, samples of natural fiber green composites were machined to typical shape and diemensions by CNC technique according to ASTM E647-13a as shown in Fig. 1. The green composite samples were tested at a cross head speed of 1 mm min$^{-1}$ at room tempreature. Five samples of each type of composites were tested and the resulting data of load – displacement curves were recorded after the test is performed. The fracture toughness $K_{IC}$ values of green composite samples was calculated using the following formula [18]:

$$\Delta K = \frac{P}{B\sqrt{W}}\frac{(2+\alpha)}{(1-\alpha)^{3.2}}(0.866 + 4.64\alpha + 13.32\alpha^2 + 14.72\alpha^3 + 5.6\alpha^4) \quad (1)$$

where:

*P*: the applied load, *a*: length of the crack, *B*: the sample thickness, and *W*: the width of sample, and $\alpha = a/W$; expression valid for $a/W \geq 0.2$.

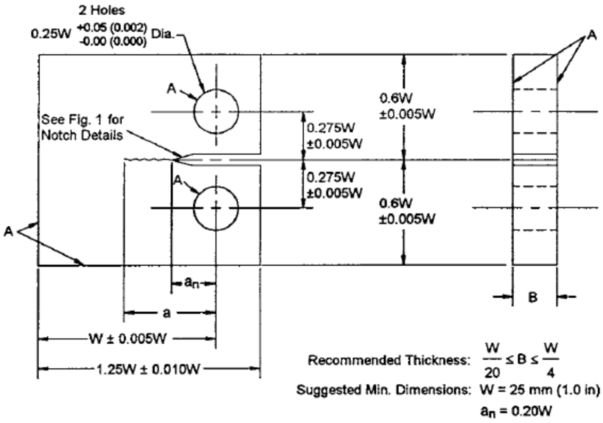


Fig. 1 A geometry of compact tension (CT) specimen according to ASTM E647 -13a [18]

## III. Results and Discussion

Load versus displacement curve of unfilled bio-PMMA resin is shown in Fig. 2. For a compact tension test, this thermoplastic matrix exhibited linear behavior with no plastic yielding prior to fracture as seen in Fig. 3. The average value of plane strain fracture toughness $K_{IC}$ was 0.81 MN (m)$^{-3/2}$.

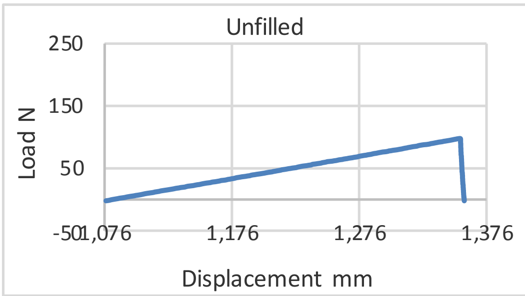


Fig. 2 Load-displacement curve of unfilled bio-PMMA material

Fig. 3 A pure bio - PMMA compact tension test specimen during the test

The load-displacement curves of all untreated and treated corn fiber composites are shown in Figs. 4-7. The fracture toughness test results of treated corn fiber composites compared to untreated fiber composite are shown in Fig. 9. From these results, it can be noticed that each of the four mercerization treatment variables had effect on the fracture behavior of the short corn fiber green composites. The mercerization treatment of 3% NaOH for 1.5 hrs. at room temperature have a significant effect on fracture properties. The composite displayed the highest plane strain fracture toughness $K_{IC}$ of 1.314 MN $(m)^{-3/2}$ compared to the untreated fiber composite of plane strain fracture toughness $K_{IC}$ of 1.183 MN $(m)^{-3/2}$. With further increased of concentration of NaOH solution and time of immersion, the plane strain fracture toughness $K_{IC}$ were dropped to 0.663 MN $(m)^{-3/2}$ of 3% NaOH and 0.68 MN $(m)^{-3/2}$ of 5% NaOH for 3 hrs. The high concentration of the NaOH solution, the damage done to the fiber resulting in a weak corn fiber/bio-PMMA adhesion. In general, all the treated corn fiber composites showed extensive stable crack growth. The standard fracture toughness test corn fiber composite sample is seen in Fig. 10.

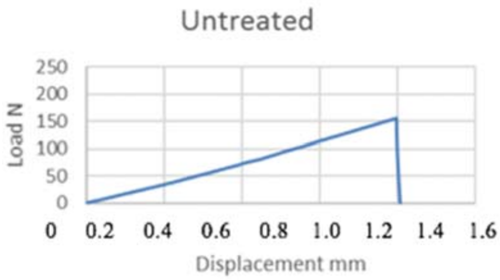


Fig. 4 Load-displacement curve of untreated corn fiber composite material

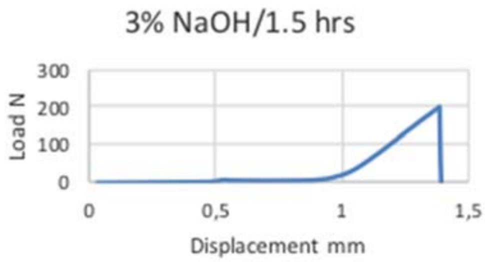


Fig. 5 Load-displacement curve of 3% NaOH corn fiber composite material for 1.5 hours

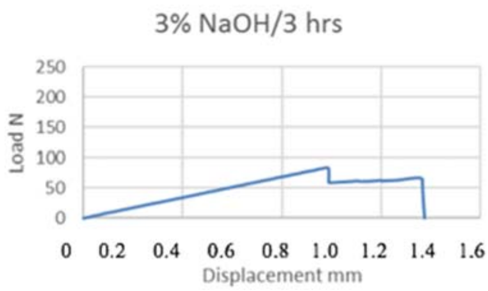


Fig. 6 Load-displacement curve of 3% NaOH corn fiber composite material for 3 hours

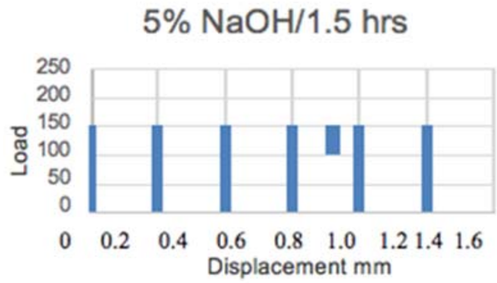


Fig. 7 Load-displacement curve of 5% NaOH corn fiber composite material for 1.5 hours

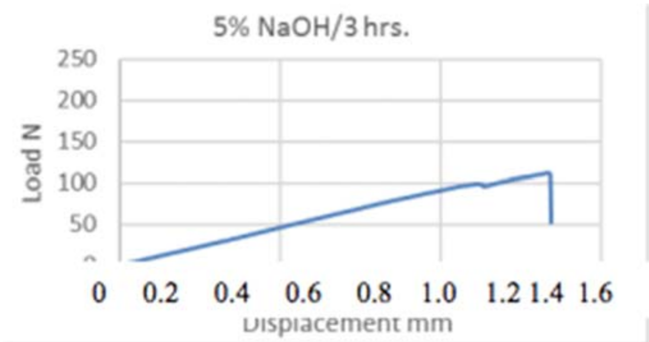


Fig. 8 Load-displacement curve of 5% NaOH corn fiber composite material for 3 hours

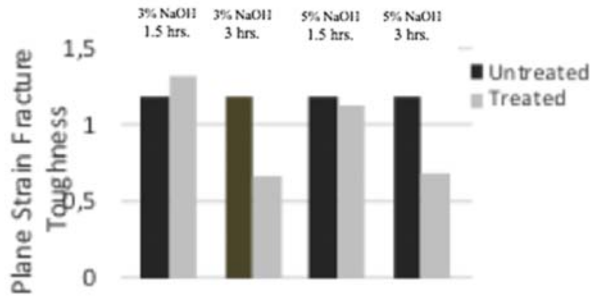


Fig. 9 Fracture toughness of untreated and treated corn fiber green composites tested

Fig. 10 A 3% NaOH corn fiber composite compact tension test specimen during the test

FTIR spectra were used to characterize the improvement of adhesion between the fiber surface and the polymer matrix. Fig. 11 shows the FTIR spectrum of unsaturated polyester. Many peaks were appeared in this figure such as aliphatic, aromatic and vinyl CH stretching at 2850.08 to 2955.28 $cm^{-1}$. FTIR spectra of short corn fiber composites with untreated and mercerization treated fibers are shown in Figs. 12-16. In this figure, we can see a peak at (1733.3) $cm^{-1}$. Also the peak at 1463.03 $cm^{-1}$ is attributed to the $CH_2$ bending of cellulose. While the peak of C=O stretch for acetyl group in lignin appears at 1253.19 $cm^{-1}$ which was reduced after chemical treatment. This was due to the partial removal of lignin from the fiber surface.

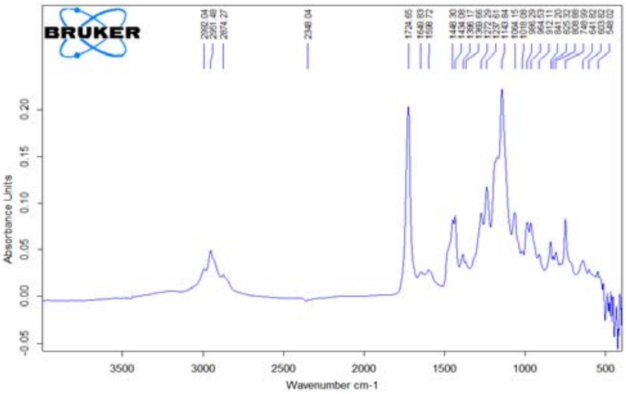


Fig. 11 FTIR spectrum of unfilled bio-PMMA material

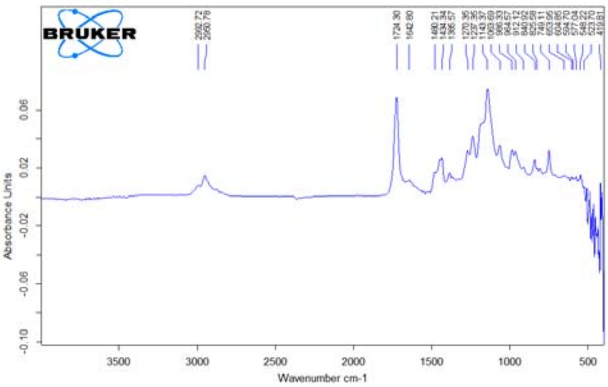


Fig. 12 FTIR spectrum of untreated corn fiber composite

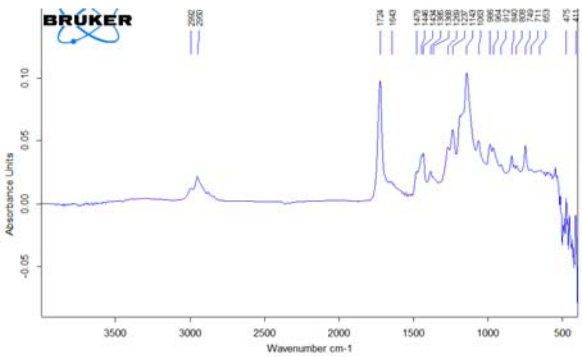


Fig. 13 FTIR spectrum of 3% NaOH corn fiber composite for 1.5 hours

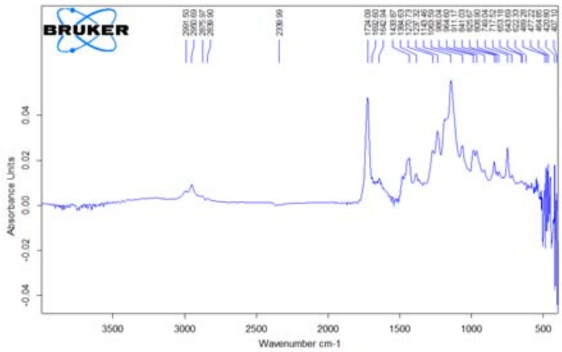


Fig. 14 FTIR spectrum of 3% NaOH corn fiber composite for 3 hours

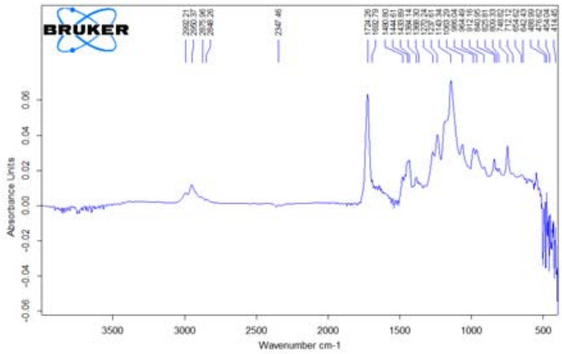


Fig. 15 FTIR spectrum of 5% NaOH corn fiber composite for 1.5 hours

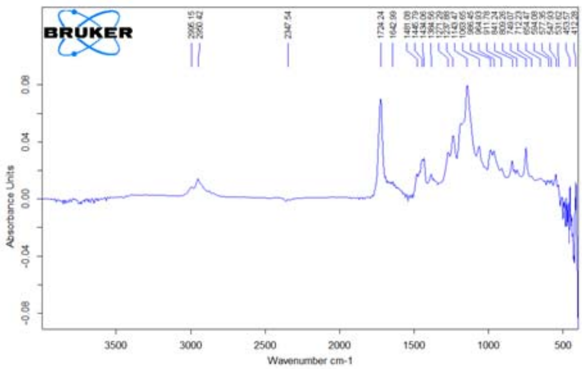


Fig. 16 FTIR spectrum of 3% NaOH corn fiber composite for 3 hours

## IV. Conclusion

The conclusions that can be derived from this work are as follows:

1. The corn natural fibers were treated with 3 and 5 % NaOH solution for 1.5 and 3 hours.
2. The fracture toughness of 3% corn fiber green composite register higher plane strain fracture toughness than other type of mercerization treatments.
3. The increase of concentration and immersion time of mercerization treatment reduced the toughness of corn fiber green composites.
4. Natural corn fiber can be extracted at a relatively low cost compared to other types of natural plant fiber, thus it can

be predicted that it will have a very promising future in green composite materials.

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