

Composition, structure, and mechanical properties of hemp fiber reinforced composite with recycled high-density polyethylene matrix

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Abstract

Hemp fiber composites with recycled high-density polyethylene matrix were prepared in various compositions ranging from 20 to 40% of fiber volume fraction. The fiber–matrix interface was improved using 5% by weight NaOH-treated hemp fiber in each composite system. The surface morphology and chemical compound of hemp fiber after chemical treatment were analyzed by Scanning Electron Microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR). This study indicated that hemp fiber-recycled high-density polyethylene (rHDPE) composites could achieve maximum tensile strengths on the order of 60 MPa. Among the tested samples, the composites with 40% of fiber volume fraction demonstrated the best mechanical properties with regard to tensile strength, elastic modulus, and flexural strength and modulus.

Keywords

Natural fiber composite, hemp fiber composite, recycled thermoplastic

Introduction

Natural fiber composites (NFC), fundamentally cellulose fiber, have renewed interests in engineering community due to their unique material properties including fast growth, low density ($\sim 1/2$ of E-glass fiber), high specific strength and stiffness, excellent sound-absorbing efficiency, and high shatter resistance.^{1–8} Currently, in the automobile industry, hemp fiber composites have begun to replace fiberglass composite and steel alloy as interior and exterior systems for lightweight and fuel-efficient vehicles.⁹ In the civil and building construction industries, NFCs have recently been used as non-load bearing members, such as decking, in order to help mitigate the environmental and health issues caused by using heavy metal-treated wood.¹⁰

The chemical composition of natural fiber consists of cellulose (microfiber of the cell wall), hemicelluloses, and lignin (biopolymer components of the cell wall). The outer surfaces of plant fiber contain waxes, fats, and pectin. The cellulose group is a highly crystalline structure with theoretical Young's modulus of ~ 130 GPa¹¹; therefore, many natural fibers exhibit

good mechanical properties. In particular, hemp, flax, and kenaf have remarkable mechanical properties, with a comparable specific strength but higher specific modulus than E-glass fiber, as presented in Table 1.

Thermoplastic materials were chosen as the polymeric matrix in this study due to its recyclability. Every year, a large amount of postconsumer thermoplastic materials are generated worldwide. In 2005

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Table 1. Typical mechanical properties of cellulose fiber vs. E-glass fiber

Material	Density (g/cc)	Tensile strength (MPa)	Elastic modulus (GPa)	Specific strength (σ/γ)	Specific modulus (E/γ)	Elongation at failure (%)	Moisture absorption (%)	Cost (\$/lb)
E-glass ¹¹	2.62	3400	73	1297	28	4.8	N/A	1.10
Hemp ¹²	1.4	550–900	70	393–643	50	1.6	6–12	0.30
Flax ¹³	1.4	800–1500	60–80	571–1071	43–57	2.7–3.2	8–12	0.33
Ramie ¹⁴	1.5	500	44	333	29	3.6–3.8	8–17	0.34
Kenaf ¹⁴	1.45	930	53	641	36	1.6	10–12	0.24
Coir ¹³	1.25	220	6	176	5	1.5–4	8	0.20
Sisal ¹³	1.33	600–700	38	451–526	29	3–7	10–22	0.36
Jute ¹³	1.46	400–800	10–30	281–548	7–21	1.5	12–14	0.20

alone, around 30 million tons of waste plastics were generated in the United States while only 5.7% of this waste material was recycled.¹⁵ Previous studies have shown that the properties of recycled high density polyethylene (rHDPE) were similar to those of virgin HDPE (vHDPE) and the price was 31–34% cheaper.¹⁶

In this study, an investigation was performed to determine the chemical, physical, and mechanical properties of hemp fiber composites with rHDPE matrix. A compression molding technique was used to synthesize hemp fiber composites with fiber volume fractions of 20%, 30%, and 40%, respectively, for both treated and untreated fibers. Scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR) were used to investigate the surface morphology of the fiber and the resultant composite. The tensile strength, secant modulus, flexural strength, and flexural modulus of the composites having different volume fraction compounds were analyzed.

Experimental

Materials

Industrial hemp fibers were obtained from Hempline Inc (Delaware, Ontario, Canada). The average density of hemp fiber was 0.86 g/cm³ with a typical diameter of 22.5 μ m and length of 25 mm. The moisture content of the raw industrial hemp fiber was approximately 6%. The rHDPE pellets used in this study were obtained from Customer Polymer Inc (Charlotte, North Carolina, United States), which were recovered from detergent bottle applications, having an average bulk specific density of 0.98 g/cm³, a melt index (MI) of 0.45 g/10 min at 190°C, and a melting temperature range from 130 to 190°C.

Composite manufacturing

For this study, the NFCs were prepared using both treated and untreated hemp fibers. The treated hemp fibers were prepared using an Alkali solution, which contained a 5% concentration of sodium hydroxide (NaOH), prior to the fabrication of the polymeric composites. The hemp fibers were immersed in the NaOH solution for 24 h at 60°C to allow complete saturation. After immersion, the hemp fibers were washed with running distilled (DI) water with 1% of acetic acid to neutralize any remaining NaOH molecules. The hemp fibers were then removed from the DI water when their pH level ranged from 6.8 to 7.2 using an Orion 2 Star PH meter. The hemp fibers were then placed in a drying oven at 60°C for 24 h. The oven-dried hemp fibers were then stored in desiccators prior to being used to manufacture the polymeric composites.

The polymeric composite materials were fabricated using both a C.W. Brabender 19.05 mm single-screw extruder and Carver hydraulic press. Initially, the pellets of the rHDPE were ground using a laboratory miller manufactured by Arthur Thomas Co, Swedesboro, New Jersey. The grounded rHDPE powder was then processed into rHDPE films using the single-screw extruder. The extruder was operated at a temperature of 180°C with an extruder rotational speed of 60 rpm. The films that were extruded had a typical thickness of 0.3 mm and were then cut into a 254 mm $\times$ 254 mm sheets for use in the composite manufacturing process.

A compression molding technique using the Carver hydraulic press was used to manufacture the hemp fiber composites with the rHDPE films using a fabricated mold having the dimensions of 254 mm $\times$ 254 mm. Each composite sample was manufactured by sandwiching a layer of manually distributed treated or untreated hemp fiber in between two layers of rHDPE

Table 2. Description of the various composite tensile test samples

Composite designation	Hemp fiber fraction (%)	Polymer matrix	Polymer fraction (%)
rHDPE	0% of treated hemp	rHDPE	100
20 uHemp/80 rHDPE	20% of untreated hemp	rHDPE	80
20 Hemp/80 rHDPE	20% of treated hemp	rHDPE	80
30 uHemp/70 rHDPE	30% of untreated hemp	rHDPE	70
30 Hemp/70 rHDPE	30% of treated hemp	rHDPE	70
40 uHemp/60 rHDPE	40% of untreated hemp	rHDPE	60
40 Hemp/60 rHDPE	40% of treated hemp	rHDPE	60

rHDPE: recycled high-density polyethylene.

Table 3. Summary of tensile test results for hemp fiber composites

Composite	Density (g/cc)	Maximum strength (MPa)	SD (MPa)	Strain (%)	Secant modulus at 2% strain (MPa)	SD (MPa)
rHDPE	0.98	19.1	0.6	17.9	441	18.5
20 uHemp/80 rHDPE	1.00	15.7	2.4	4.5	546	34.8
20 Hemp/80 rHDPE	0.95	18.6	2.1	7.0	556	56.3
30 uHemp/70 rHDPE	1.03	27.4	4.9	5.4	682	98.4
30 Hemp/70 rHDPE	0.93	45.7	5.7	3.7	1670	163.5
40 uHemp/60 rHDPE	1.06	26.0	5.2	3.3	986	157.9
40 Hemp/60 rHDPE	0.89	60.2	7.3	3.0	2574	257.2

rHDPE: recycled high-density polyethylene.

films at a temperature of 180°C under a constant pressure of 1.5 MPa for duration of 15 min. The fibers were placed using a disoriented (random) distribution. These various built up sandwiches were used to fabricate the desired final composites. The weights of hemp fiber and rHDPE layers were controlled to maintain a 20%, 30%, or 40% fiber volume fraction. A summary of the composite materials that were manufactured is presented in Table 2. The fiber volume fraction V_f was determined using the following Equations (1) and (2):

$$V_f = (W_f/\rho_f)/(W_m/\rho_m) + (W_f/\rho_f) \quad (1)$$

$$V_m = 1 - V_f \quad (2)$$

where V_f denotes the volume fraction of hemp fiber, W_f is the weight of hemp fiber sandwiched in the composite, and ρ_f is the density of hemp fiber. V_m , W_m , and ρ_m represent the volume fraction, weight, and the density of rHDPE matrix, respectively. Before manufacturing the composite, the weight of fiber and rHDPE for each layer was measured using a Denver Instrument bench-top scale. The density of composite of each composition was measured by displacement methods conforming to

American Society for Testing and Materials (ASTM) D 792-08.¹⁷ The measured density of each fabricated composite is presented in Table 3.

Composite characterization and testing

SEM analysis. Surface morphology of the treated and untreated hemp fiber, fiber distribution, and the fiber–matrix interface were analyzed using A JSM-6764 SEM. The SEM specimens were selected from bulk samples of the treated and untreated fibers and then coated with a thin layer of gold using a Denton Desk IV sputtering instrument. The SEM instrument was operated at room temperature with 10 kV. The surface morphology of the treated and untreated hemp fiber and the hemp fiber–matrix interface of the rHDPE composites were observed.

FTIR measurement. Chemical compound of untreated and 5% NaOH treated hemp fiber were analyzed using a Perkin-Elmer 100 Spectrometer (Boston, Massachusetts, United States). A total of eight scans were taken for each sample between 650 cm⁻¹ and

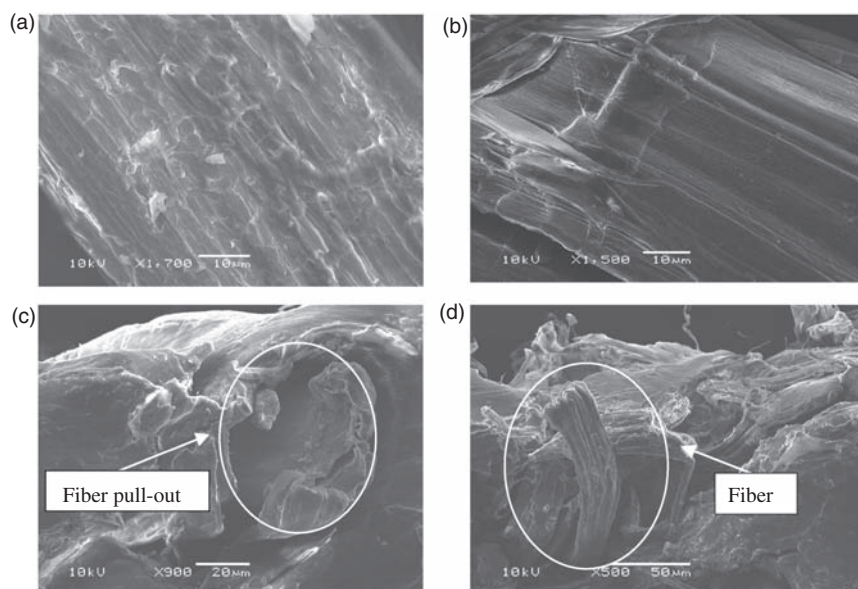


Figure 1. SEM image of (a) untreated hemp, (b) 5% NaOH treated hemp, (c) untreated hemp fiber being completely pull out from matrix, (d) 5% NaOH treated hemp fiber–matrix interface. SEM: Scanning electron microscopy.

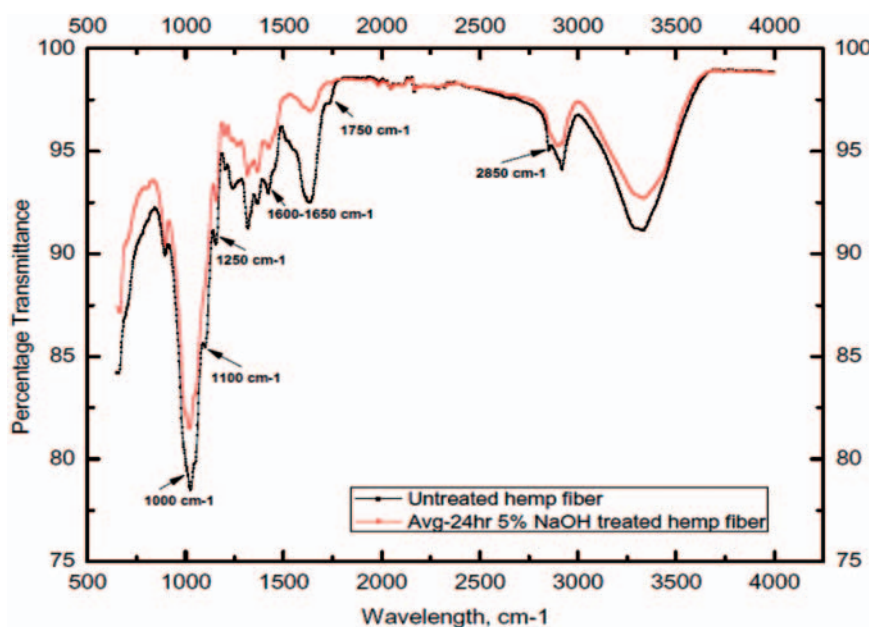


Figure 2. FTIR spectra of untreated hemp and 24 hour 5% NaOH treated hemp fiber. FTIR: Fourier transform infrared spectroscopy.

4000 cm^{-1} , with a resolution of 8 cm^{-1} . Each sample was prepared in filament form.

Composite mechanical strength. Tensile and flexural testing were conducted using an Instron 5582 constant rate of extension (CRT) universal testing machine in accordance with ASTM D638¹⁸ and

D790¹⁹, respectively, under the following test conditions of (i) a cross-head speed of 1.3 mm/min , (ii) air temperature 23°C , and (iii) 65% relative humidity. For the tensile tests on the various composites manufactured using treated and untreated hemp fibers, the typical tensile stress–strain behavior including analyses of the maximum tensile strength, strain at maximum

tensile strength, and the secant modulus at 2% strain are presented and reported. For the flexural tests on the various composites manufactured using treated hemp fibers, the typical flexural stress–strain behavior including analyses of the maximum flexural strength, strain at maximum strength, and the flexural modulus at 1% and 3% strain of are presented and reported.

Results and discussion

Surface morphology results

Figure 1(a) and (b) shows the surface morphology of untreated and 5% NaOH treated hemp fibers. Significant differences of surface morphology of treated/untreated hemp fibers are clearly observed. As can be seen in Figure 1(a), the as-received fiber exhibited smooth noncellulose structure boundary layers with wax/protein composition and surface impurities. Figure 1(b) indicated that the alkylation process removed the weak boundary layer of noncellulose structure, therefore, the surface roughness and surface area of the hemp fiber have been significantly increased, likely resulting in improved interfacial adhesion between fiber and rHDPE matrix. Figure 1(c) shows the SEM image of the fracture surface of a composite with 30% untreated hemp fiber volume fraction. Complete fiber pullout was often observed in the resin rich region. This could be attributed to the poor fiber–matrix interface due to the weak surface boundary observed in Figure 1(a), suggesting that the failure mechanism in the untreated/rHDPE composite could have resulted from debonding. On the other hand, Figure 1(d) presents a typical SEM image of the fracture surface of a composite with 30% volume fraction of NaOH treated hemp/rHDPE matrix. Fiber breakage without pullout from the matrix was often observed in many areas within the test specimen as shown in Figure 1(d). This may suggest that there is improved fiber–matrix interface adhesive strength after alkali treatment.

FTIR results of NaOH treated hemp fiber

Figure 2 presents the FTIR spectra for both untreated and 5% NaOH treated hemp fiber. The spectra show various transmission bands. After 24 h of NaOH treatment, the peak at 1000 cm^{-1} ($-\text{OH}$ group) is significantly increased with associated hydroxyl group available for fiber–matrix interface bonding. The reaction of hydroxyl bonds with the carboxyl group is given in the range $3200\text{--}3600\text{ cm}^{-1}$. The peak in this range has increased after the 24-h treatment. The similar increases in intensity for both 1000 and

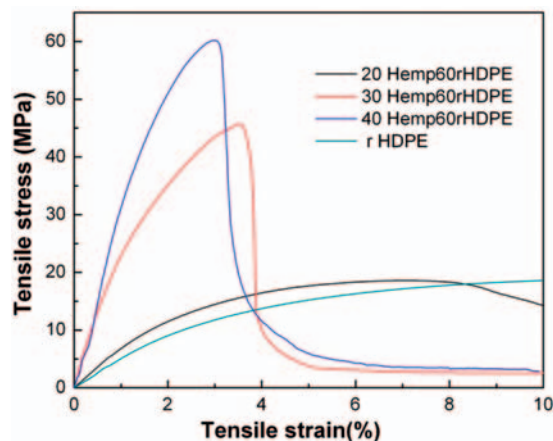


Figure 3. Typical tensile stress–strain behavior of treated hemp fiber composites.

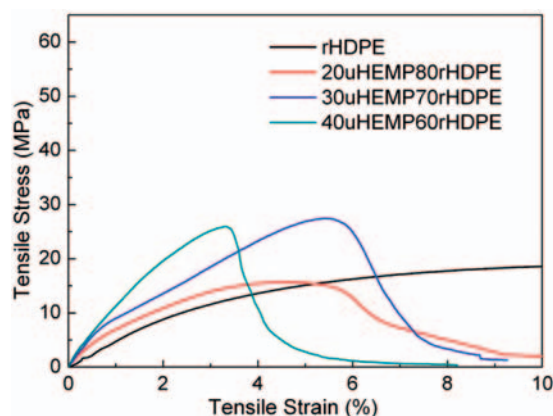


Figure 4. Typical tensile stress–strain behavior of untreated hemp fiber composites.

$3200\text{--}3600\text{ cm}^{-1}$ band in hemp fibers with NaOH treatment have also been reported in previous literature.²⁰

Compared to untreated fiber, the peak at 1250 cm^{-1} of treated hemp fiber is clearly removed. This peak belongs to the C–O stretching of acetyl groups of lignin. It appears that the lignin is completely removed from the hemp fiber surface after NaOH treatment. Also, the hemicelluloses group is partially removed from the fiber surface after the NaOH treatment as is evident by the decreased carbonyl peak at $1600\text{--}1650\text{ cm}^{-1}$ in treated hemp fibers.

The peak at $1740\text{--}1750\text{ cm}^{-1}$ in untreated hemp has also been removed after the NaOH treatment. This is due to the removal of pectin and wax that is present on the hemp fibers. The peaks observed at 1100 cm^{-1} and 2850 cm^{-1} in untreated fibers also disappeared after treatment. The disappearance of 1100 cm^{-1} peak could be explained by the reaction of NaOH with a secondary alcoholic group, and the peak at 2850 cm^{-1}

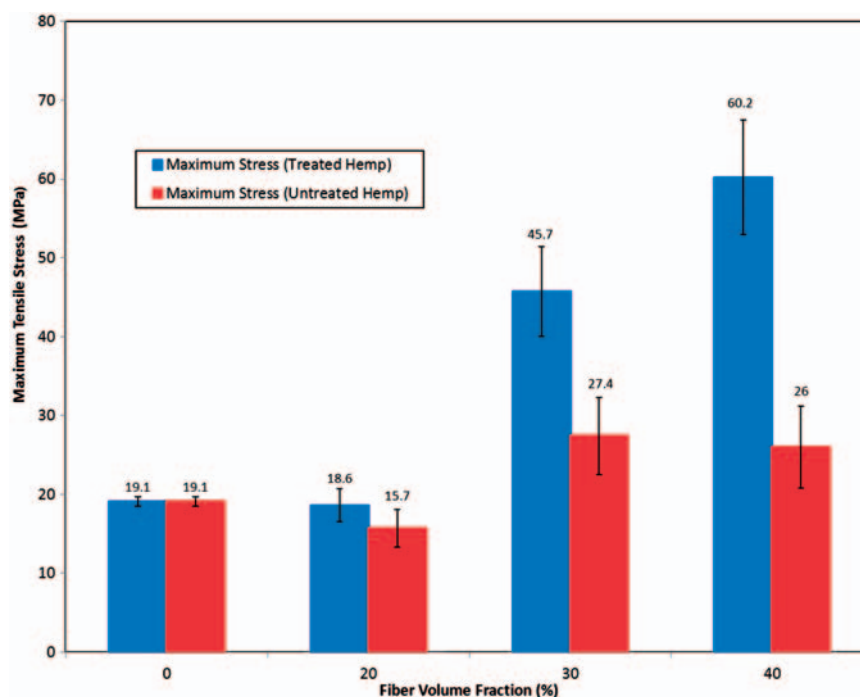


Figure 5. Maximum tensile stress of hemp fiber composites.

disappeared after NaOH treatment probably due to the removal of a methane group.

Tensile strength

The tensile strength of the hemp fiber composites with rHDPE were determined from data obtained in accordance with ASTM D638. The tensile tests were conducted using the standard dog bone-shaped test coupon having manufactured dimensions of 12.7 mm in width, 63.5 mm in length, and a thickness of 2.5 mm. Five coupons were made from each test sample composite. Table 2 presents a summary of the composite materials that were evaluated, which included composites manufactured with treated and untreated fibers.

Typical strain–stress curves of hemp fiber composites with different fiber–matrix volume fraction are presented in Figure 3 for treated fiber composites and Figure 4 for untreated fiber composites. It should be noted that the axial strains were calculated based on the displacement of the CRT's cross-head movement and the initial clamp spacing for each test specimen. A continual improvement in maximum tensile strength and a reduction in strain at maximum strength were observed with the increase in hemp fiber volume fraction for the treated fiber composites. There is a significant improvement in the tensile stress–strain behavior of the treated fiber composites (Figure 3) compared to the untreated fiber composites (Figure 4), which may support the

findings from the SEM and FTIR, which suggests there is improved interfacial adhesion due to the fiber treatment. Overall, the hemp/rHDPE composites were well behaved with regard to their initial stiffness and each had a distinct rupture failure ranging from 3% to 7% strain, as can be seen in Figure 5, the maximum tensile strength for the treated hemp/rHDPE composite with 40% of fiber volume demonstrated an approximate three time improvement from the treated hemp composite with 20% of fiber volume fraction, yielding an maximum strength of 60.2 MPa and a strain at maximum strength of 3.0. The tensile testing results of the treated hemp fiber with recycled HDPE matrix exceeded the previous reported data regarding hemp fiber composites manufactured with virgin Polylactic Acid (PLA) matrix.²¹

Since these manufactured NFCs are being considered for their potential use in the civil and building construction sector as possible structural elements, there is an interest at understanding their low strain behavior. Due to the nonlinear behavior of the NFCs, a secant modulus at 2% strain was selected to evaluate the low strain behavior and stiffness. Figure 6 presents the secant modulus at 2% strain as a function of hemp fiber volume fraction from 20% to 40% for both the treated and the untreated composites. There is an observed continuous improvement in the composite stiffness with the increase in fiber volume fraction for the treated fiber composites. The greatest increase in composite stiffness was observed for the 30% hemp

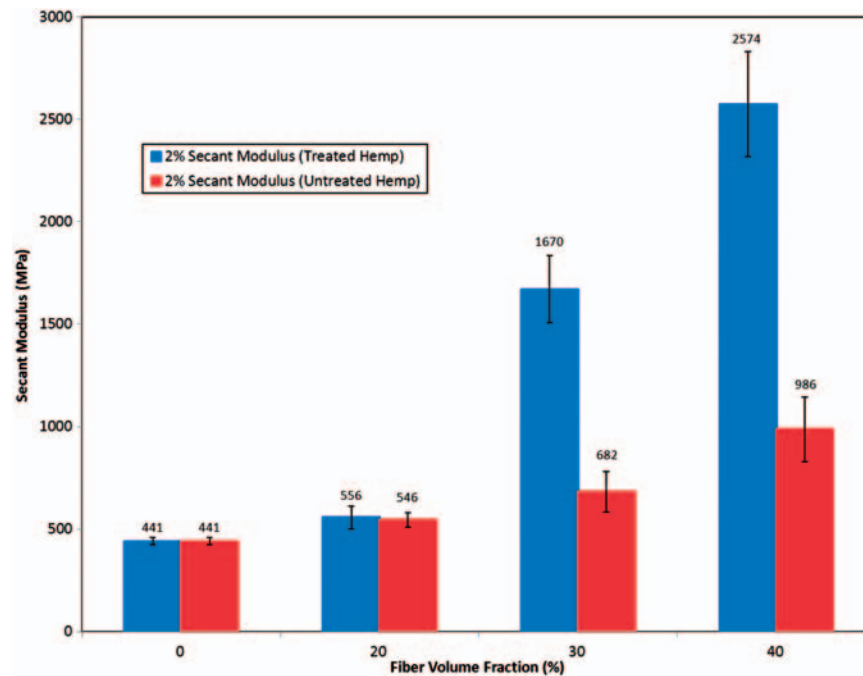


Figure 6. Secant modulus at 2% strain of hemp fiber composites.

fiber volume fraction having an elastic modulus of 1670 MPa as compared to the secant modulus of 556 MPa for the 20% hemp fiber volume fraction. The secant modulus at 2% strain for the treated hemp fiber composite with rHDPE matrix with a 40% fiber volume fraction was 2574 MPa. A summary of the tensile properties of maximum strength, strain at maximum strength, and secant modulus at 2% strain, which were measured during this study with their corresponding results for the hemp fiber composites are presented in Table 3.

Flexural strength

Based on improved tensile strengths of the treated hemp fiber NFCs, flexural strength testing was conducted only on composites manufactured from treated hemp fibers. The flexural strength, strain at maximum strength, and flexural modulus at 1% and 3% strain for these NFCs materials were tested on the CRT testing machine in accordance with ASTM D790. Each three-point flexural bending test was conducted using a rectangular test coupon having typical dimension of 25.4 mm in width, 6.35 mm in thickness, and 127 mm in length. Five coupons were made from each test sample composite. The same treated hemp fiber composites that were manufactured for the tensile tests were used for the flexural tests as described in Table 2.

Figure 7 presents the flexural stress of the treated hemp/rHDPE composites as a function of the flexural

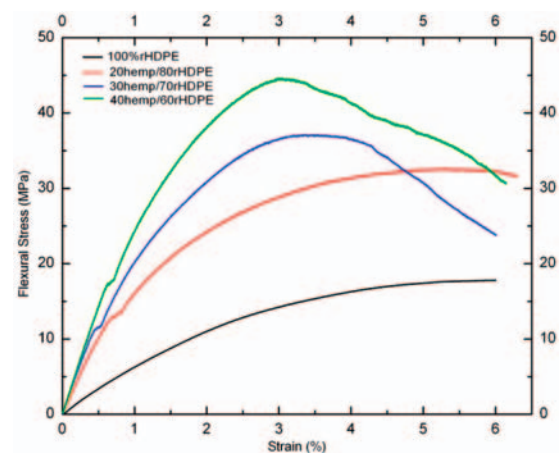


Figure 7. Typical flexural stress–strain behavior of treated hemp fiber composites.

strain. The flexural strains were calculated based on the procedure outlined in ASTM D790 using the displacement of the CRT's cross-head movement. It is interesting to observe that as the fiber fraction increased, there was a proportional increase in bending strength and stiffness. The most significant improvement bending strength and stiffness was observed in the higher 40% fiber fraction composite. It can be clearly seen that with an increase in fiber volume fraction, there is an increase in the maximum flexural strength as is presented in Figure 8.

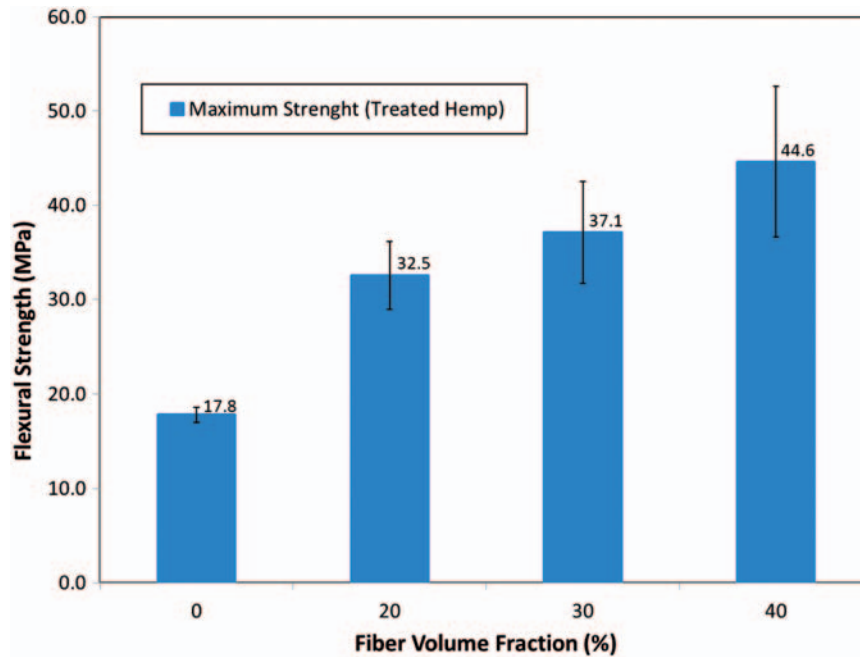


Figure 8. Maximum flexural strength of treated hemp fiber composites.

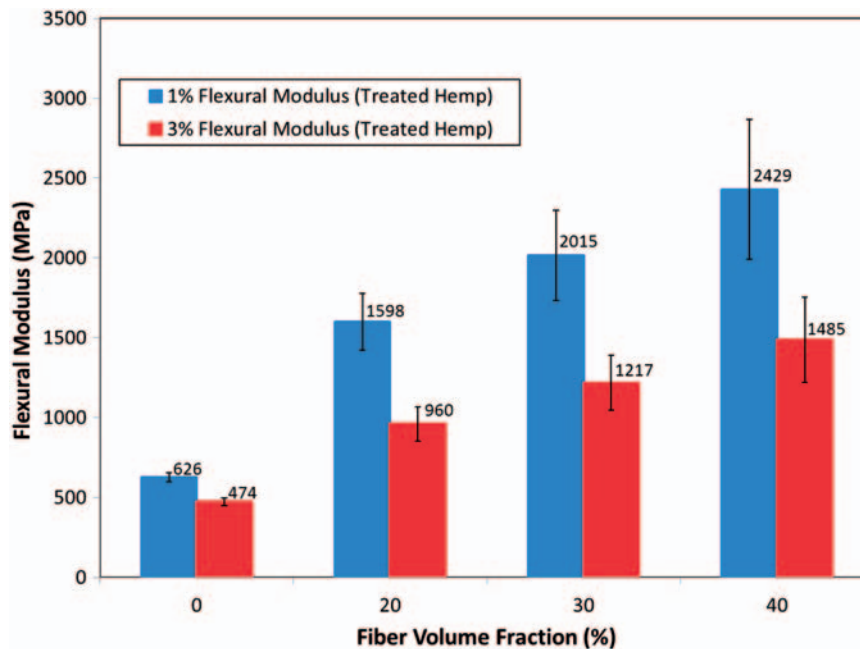


Figure 9. Flexural modulus at 1% and 3% flexural strain of treated hemp fiber composites.

Based on the potential use of these manufactured NFCs in the civil and building construction sector as possible structural elements, there is a need in understanding their low flexural strain behavior. Due to the more uniform behavior of the NFCs, the secant modulus at 1% and 3% strain were selected to evaluate the low flexural strain behavior and stiffness. The flexural

moduli of the composites with different fiber volume fraction are presented in Figure 9. The results indicate that there is an increase in composite flexural moduli with the increase in fiber volume fraction associated with a corresponding reduction in strain. However, the observed moduli reduce in stiffness as the flexural strain increases. This behavior may be beneficial with

Table 4. Summary of flexural test results for hemp fiber composites

Composite designation	Maximum flexural strength (MPa)	SD (MPa)	Strain at maximum strength (%)	Flexural modulus at 1% strain (MPa)	SD (MPa)	Flexural modulus at 3% strain (MPa)	SD (MPa)
rHDPE	17.8	0.8	3.1	628	27.6	474	23.7
20 Hemp/80 rHDPE	32.5	3.6	3.6	1598	177	960	105.6
30 Hemp/70 rHDPE	37.1	5.4	5.8	2015	282.1	1217	170.4
40 Hemp/60 rHDPE	44.6	8.0	6.0	2429	437.2	1485	265.8

rHDPE: fiber-recycled high-density polyethylene.

regard to absorbing impact loadings that a building structure may incur. A summary of the flexural strength properties including the maximum flexural strength, strain at maximum strength, and flexural modulus at 1% and 3% flexural strain, which were measured during this study with their corresponding results are presented in Table 4.

Conclusion

Hemp fiber composites with rHDPE were manufactured using extrusion and compression molding process techniques. Prior to composite fabrication, the natural hemp fibers were treated with a NaOH solution. The effect of alkali treatment was investigated by FTIR and SEM. FTIR results indicated that there is an increase in the percentage of $-OH$ groups, which may provide more reaction sites for fiber–matrix adhesion. Therefore, the interfacial adhesion between the fiber and matrix may possibly be increased. Pectin, wax, and lignin were completely removed from hemp fiber surface, which resulted in large surface area and improved surface roughness. The FTIR also indicated that the hemicelluloses group was partially removed. SEM images of treated hemp fiber support the conclusions from FTIR results. SEM images of fracture surface of hemp fiber composites showed clearly improved interfacial adhesion between the hemp fiber and the polymer matrix. The resultant composites have demonstrated promising mechanical properties with regard to their tensile strength, tensile modulus and strain at maximum strength, flexural strength, and modulus for each hemp fiber composite that has been studied. Based on these reported experimental results, the hemp fiber–rHDPE composites with 40% fiber volume fraction yielded very promising results of tensile strength and modulus and flexural strength and modulus of 60.2 MPa, 2575 MPa, 44.6 MPa, and 2429 MPa (at 1% strain), respectively. The resultant composite have a good potential for light load applications in civil

infrastructure industry, for instance, short-span bridges and hurricane proof panels.

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Conflict of interest

None declared.

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