



## **The Effects of Chemical Treatments of Flax Fiber on Some Engineering Properties of Biocomposite**

**Ahmad Ghazanfari**

Assistant Professor

Department of Agricultural Machinery

Shahid Bahobar University

Kerman, Iran

**Satya Panigrahi**

Research Chair

Department Agricultural and Bioresource Engineering

University of Saskatchewan

Saskatoon, Canada

**Lope. Tabil, Jr.**

Associate Professor

**Written for presentation at the  
CSBE/SCGAB 2006 Annual Conference  
Edmonton Alberta  
July 16 - 19, 2006**

**Abstract:** Flax fiber, produced through a conventional scotching mill, was washed using a commercial detergent and then it was chemically treated using silane, benzoyl and peroxide. The chemically treated fibers were dried by an air-cabinet drier at 70 °C. The dried fiber were ground and truly mixed with HDPE at a ratio of 10% flax fiber and 90% HDPE. After extruding and pelleting, the mixture was fed through a rotational molding machine and composite plates were produced. The resulting composites were tested for their various mechanical properties using standard ASTM procedures. The test results indicated that the mechanical strength of the composites was higher than the plates made from HDPE, however there was no significant difference between the mechanical strength of composites produced from various chemical treatments. The optical properties of the composites were investigated using NIR spectroscopy. The % of reflectance of the NIR at a wide range of wavelength indicated that HDPE plates were easily distinguishable, however the chemically treated composites and untreated composites were not distinguishable from each other using this technique.

# The Effects of Chemical Treatments of Flax Fiber on Some Engineering Properties of Biocomposite

Ahmad Ghazanfari<sup>1</sup>, Satya Panigrahi<sup>2</sup>, Lope. Tabil, Jr.<sup>3</sup>

1. Assistant Professor, Department of Agricultural Machinery, Shahid Bahobar University, Kerman, Iran

2. Research Chair , 3. Associate Professor

Department Agricultural and Bioresource Engineering, University of Saskatchewan, Saskatoon, Canada

## Abstract

Flax fiber, produced through a conventional scotching mill, was washed using a commercial detergent and then it was chemically treated using silane, benzoyl and peroxide. The chemically treated fiber were dried by an air-cabinet drier at 70 °C. The dried fiber were ground and truly mixed with HDPE at a ratio of 10% flax fiber and 90% HDPE. After extruding and pelleting, mixture was fed through a rotational molding machine and composite plates were produced. The resulting composites were tested for their various mechanical properties using standard ASTM procedures. The test results indicated that the mechanical strength of the composites was higher than the plates made from HDPE, however there was no significant difference between the mechanical strength of composites produced from various chemical treatments. The optical properties of the composites were investigated using NIR spectroscopy. The % of reflectance of the NIR at a wide range of wavelength indicated that HDPE plates were easily distinguishable, however the chemically treated composites and untreated composites were not distinguishable from each other using this technique.

**Key words:** flax fiber, biocomposite, chemical treatment, engineering properties

## Introduction

Engineering composites are traditionally manufactured using a polymer matrix and synthetic fibers such as glass or carbon fibers for reinforcement. The increasing ecological and environmental concerns, together have caused the manufacturers and researchers seek alternatives for synthetic fibers. In the past decade, the low cost and abundance of the natural

fibers, created a new interest in utilization of these fibers as potential replacement for synthetic fibers in production of composite materials. Natural fiber reinforced composites, known as “biocomposites”, are already used in automotive industry and there is an increasing demand for their usage in construction industries.

There have been many studies on utilization of flax (*Linum usitatissimum* L.) fibers as a reinforcing agent for bicomposite production in thermoplastic industries. Flax fiber, besides being environmentally friendly, has also proved to enhance mechanical properties of composites. The main disadvantage of flax fiber as well as other natural fibers is their hydrophilic nature that causes a weak bonding with hydrophobic polymers. This limitation is reduced through chemical modification of surface of the fibers.

Cellulose fibers are hygroscopic in nature; thus absorb water to some extent. Moisture absorption causes swelling of the fibers which leads to micro-cracking of the composite and degrades mechanical properties. Moisture absorption problem can be greatly reduced by treating these fibers with a suitable chemical reagent. Some chemical treatments also activate hydroxyl groups and introduce new moieties that can effectively interlock with the matrix.

The ultimate mechanical properties of biocomposites depend to a great extent on the adhesion between the reinforcing fibers and surrounding matrix. The adhesion between the two materials is a function of several factors among which are surface roughness and surface coating and both can be enhanced by special chemical treatments. Measurement of engineering properties of the composites is a good method for evaluating the effectiveness of adhesion between fiber and polymer matrix due the applied chemical treatments.

In this research flax fiber was treated with different chemicals including silane, benzoyl chloride and dicumyl peroxide. The treated fibers are processed in similar manufacturing steps to produce rotational molded biocomposites. The strength and optical properties of biocomposites were measured and compared to evaluate the effectiveness of the chemical treatments.

## **Material and Methods**

### **Fiber preparation**

Flax fibers, derived from linseed flax grown in Saskatchewan and decorticated on a standard scotching mill at Durafiber in Canora, SK, Canada, were used for these experiments. The fibers were first washed thoroughly with commercial detergent and dried in an air oven at 70°C for 24 h. Then the fibers were pre-treated with 5% NaOH for about half an hour in order to activate the OH groups of the cellulose and lignin in the fiber. Alkaline treatment or mercerization is one of the most used chemical treatment of natural fibers when used to reinforce thermoplastics and thermosets. The important modification done by alkaline treatment is the disruption of hydrogen bonding in the network structure, thereby increasing surface roughness. This treatment removes a certain amount of lignin, wax and oils covering the external surface of the fiber cell wall, depolymerizes cellulose and exposes the short length crystallites. Fibers were soaked in 5% NaOH for about half an hour in order to activate the OH groups of the cellulose and lignin in the fiber. The fibers treated in this way were used as chemically untreated fiber.

The pre-treated fibers were dipped in an alcohol water mixture (60:40) containing triethoxyvinylsilane coupling agent. The pH of the solution was maintained between 3.5 and 4, using the METREPAK Phydron buffers and pH indicator strips. Fibers were washed in double distilled water and dried in the oven at 80°C for 24 h.

For the next treatment the pre-treated fibers were suspended in 10% NaOH solution and agitated with benzoyl chloride. The mixture was kept for 15 min, filtered, washed thoroughly with water and dried between filter papers. The isolated fibers were then soaked in ethanol for 1 h to remove the benzoyl chloride and finally was washed with water and dried in the oven at 80°C for 24 h.

For the peroxide treatment fibers were coated with dicumyl peroxide from acetone solution after alkali pre-treatments. Saturated solution of the peroxide in acetone was used. Soaking of the fibers in the solution was conducted at a temperature of 70°C for 30 min. High temperatures were favored for decomposition with the peroxide. The chemically treated fibers were washed with distilled water and placed in an oven at 80°C for 24 h.

## **Making composites**

The treated and untreated fibers were ground by the grinding mill (Falling Number, Huddinge, Sweden) Mixtures of thermoplastic powder and 10% by weight of flax fibers were thoroughly mixed by using a tumbler. This was performed to aid in homogeneous mixing of fibers and thermoplastics during extrusion process. The blend was fed to the laboratory mixing extruder (Dynisco, Franklin, MA) using a barrel to die temperature profile of 175°C with a screw speed of 100 rpm. Extruded strands were then palletized and ground using a grinding mill (Retsch GmbH 5657 HAAN, West Germany). The ground product was fed into rotational molding machine to make 30 × 30 cm plates.

## **Flexural Test**

The mechanical strength of the prepared plates was evaluated by flexural test and the results were compared with each other. The appropriate ASTM methods were followed, and six replicate specimens were tested for each property and the results were further evaluated using ANOVA. The flexural tests were conducted at standard laboratory atmosphere of 23 °C and 50% relative humidity. An Instron Universal testing machine (SATEC Systems, Inc., Grove City, PA) was used to perform the flexural strength test at a crosshead speed of 5 mm/min as described in ASTM procedure D790, and each test was performed until flexural failure occurred. The collected data included yield displacement, yield strain, yield load, yield stress and modulus of elasticity.

## **Optical Test**

Square pieces of biocomposites (3 × 3 cm) were prepared and placed in an NIR machine and the machine was set to generate wavelength ranging from 250 to 2500 nm. The percentage of the reflected rays were measured and recorded. The percentage of the reflectance versus the wavelength were plotted and visually inspected to see if there was a significant difference between the optical properties of the composites resulted from the treated and the untreated flax fiber.

## **Microstructure of Fiber-reinforced Composites**

As a supplementary tool, the microstructure of the modified fiber-polymer matrix interface was

examined using a scanning electron microscope (SEM505 Philips, Holland) at the accelerating voltage of 30 KV. The sample surfaces were vacuum coated with a thin layer of gold on the surface of interest using a Sputter Coater S150B (Edwards, USA) to provide electrical conductivity and did not significantly affect the resolution. Scanning electron micrograph of fiber-reinforced composites showed the interfacial bonding between flax fiber and polymer matrix to indicate the extent of fiber-matrix adhesion.

## **Results and Discussion**

The results of flexural test on the biocomposites and the composite from HDPE are presented in Tables 1-5. Comparing the average yield displacement for different composites indicates that the yield displacement for various composites occurred in a range of 16.19 to 16.33 mm which indicates that yield displacement was almost the same for all of them. Yield strain for the biocomposites had very close values, however this value for the HDPE was significantly different from the other treatments.

The value for load at the yield point for untreated fiber, silane and peroxide treatment were very close to each other but the benzoyl treated fiber resulted a lower yield point. The ANOVA test was performed on these data indicated that the mean of these treatments were significantly different from each other and Doncan test indicated that benzoyl treatment was different from the untreated, silane and peroxide treatment.

The modulus of elasticity calculated for the treatments are presented in the last column of tables 1-5. The highest module was obtained for was for composites from untreated fiber. Thus by adding flax fiber to the HDPE the resulting biocomposites had significantly higher modulus of elasticity. Among the biocomposites the benzoyl treated fiber resulted the lowest modulus of elasticity. The untreated fiber resulted higher modulus of elasticity, which indicates that chemical treatment does not necessarily increases the mechanical properties, however chemical treatment produces more uniform composites with more flexibility as shown by ESM pictures (Panigrahi et al, 2002). However, more similar tests and other related tests must be performed to draw a concrete conclusion in this regard. On the other hand, since no significant difference was noticed between treating fiber with different chemical, thus both economical and environmental aspects

of using chemical must be considered.

**Table 1. The data for biocomposite resulted from untreated fiber.**

Test No.	Yield Displacement (mm)	Yield Strain (%)	Yield Load (N)	Yield Stress (MPa)	Modulus of Elasticity (MPa)
1	15.65	6.79	58.8	20.47	474.3
2	16.66	6.17	53.8	18.73	418.7
3	16.11	5.97	57.5	20.04	466.0
4	16.10	5.96	56.3	19.6	468.1
5	17.61	6.52	63.8	22.22	495.8
6	15.87	5.88	58.8	20.47	482.1
Average	16.33	6.22	58.17	20.26	467.5

**Table 2. The data for biocomposite resulted from peroxide treated fiber.**

Test No.	Yield Displacement (mm)	Yield Strain (%)	Yield Load (N)	Yield Stress (Mpa)	Modulus of Elasticity (MPa)
1	16.12	5.96	50.0	17.42	399.5
2	15.48	5.78	55.1	19.17	446.7
3	15.93	5.89	50.4	17.42	401.9
4	17.17	6.36	67.5	23.52	549.4
5	16.17	5.99	45.8	15.68	320.5
6	17.07	6.32	67.5	23.52	547.6
Average	16.30	6.05	55.8	19.46	444.3

**Table 3. The data for biocomposite resulted from silane treated fiber.**

Test No.	Yield Displacement (mm)	Yield Strain (%)	Yield Load (N)	Yield Stress (Mpa)	Modulus of Elasticity (MPa)
1	17.13	6.34	47.5	16.55	377.3
2	16.13	5.97	57.5	20.04	460.2
3	16.92	6.27	58.8	20.47	444.3
4	15.22	5.64	57.5	20.04	482.6
5	16.68	6.18	72.5	14.81	340.1
6	15.04	5.57	53.8	18.73	447.2
Average	16.19	5.99	57.93	18.44	425.3

**Table 4. The data for biocomposite resulted from benzoyl treated fiber.**

Test No.	Yield Displacement (mm)	Yield Strain (%)	Yield Load (N)	Yield Stress (Mpa)	Modulus of Elasticity (MPa)
1	21.22	7.85	47.5	16.55	346.9
2	14.73	5.45	55.1	19.17	467.4
3	15.43	5.71	47.5	16.55	386.2
4	14.95	5.54	43.8	15.25	349.3
5	15.35	5.67	53.8	18.73	430.1
6	16.14	5.98	45.3	15.68	341.6
Average	16.3	6.04	48.8	19.99	386.9

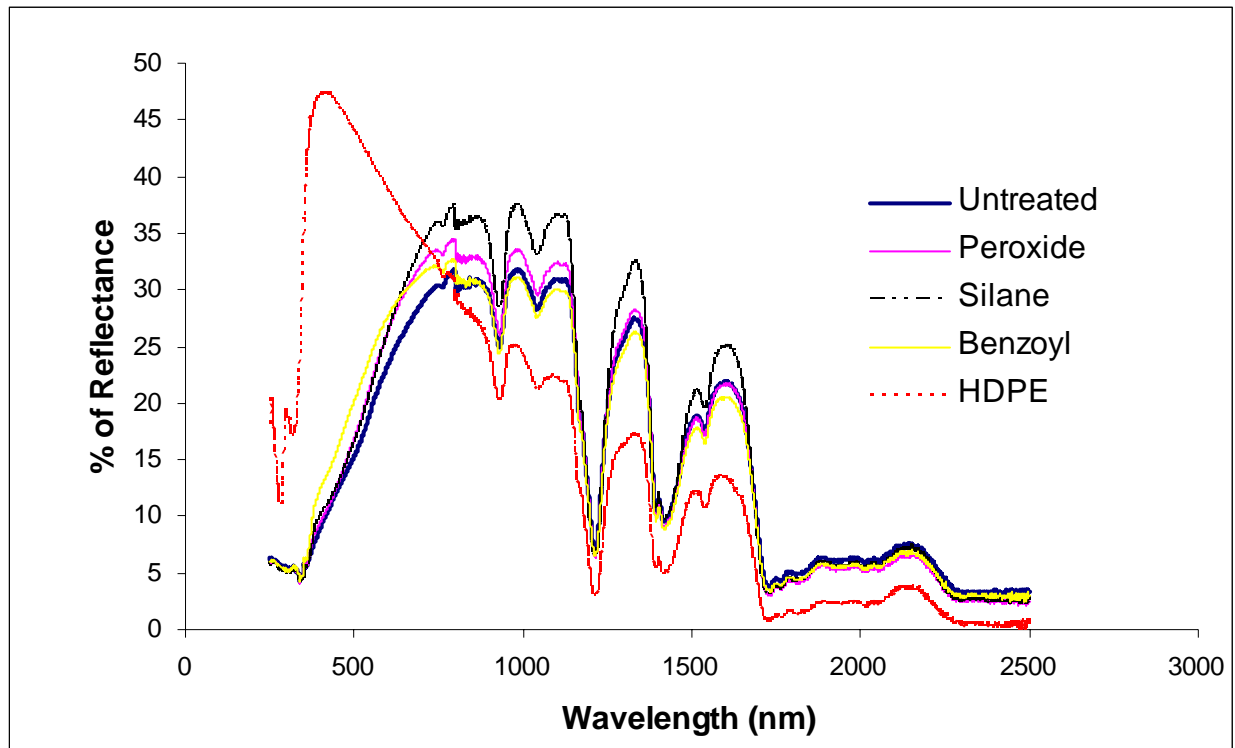


**Table 5. The data for composite resulted from HDPE.**

Test No.	Yield Displacement (mm)	Yield Strain (%)	Yield Load (N)	Yield Stress (MPa)	Modulus of Elasticity (MPa)
1	15.74	3.50	22.5	12.78	178.1
2	17.57	3.90	21.3	11.57	209.7
3	13.82	3.07	20.1	13.36	178.1
4	16.89	3.098	20.9	11.94	196.4
5	17.3	3.812	21.5	11.40	178.9
6	13.82	3.072	20	13.36	191.3
Average	16.21	3.48	21.35	12.40	188.75

The graphs of the percentage of reflectance NIR test on the biocomposites are presented in Figure 1. The trends of variation in the reflectance of the biocomposites are quite different from the HDPE composite. The HDPE composite had a white color which had the highest reflectance at the wavelength below 600  $\eta m$ . The reflectance values for the biocomposites had very similar trends. Taking into the account the huge variation in the percentage of reflectance, it can be concluded that there was no difference between the reflectance of the biocomposites, treated or untreated with different chemical agents.

In summary, adding the flax fiber to the HDPE resulted in significantly different mechanical properties of resulting composites. However, chemical treatments did not improve the tested stiffness properties of the composites. The optical properties of the resulting composites were also altered by adding flax fiber. However, the optical properties of the biocomposites were very similar to each other. Thus, the NIR method cannot be used to distinguish biocomposites treated with different chemical agents.



### Acknowledgement

The authors would like to acknowledge the Department of Agricultural and Bioresource Engineering at the University of Saskatchewan for the use of their facilities and equipment. Financial support of this study was given by Saskatchewan Flax Development Commission and the Agriculture Development Fund of Saskatchewan Agriculture, Food and Rural Revitalization. The support of Parkland Plastics and the Shahid Bahonar University, Kerman, Iran is also acknowledged.

### References

ASTM standard, D 1042, Standard Test Method for Linear Dimensional Changes of Plastics. 1999 Annual Book of ASTM Standards,. 7(1):46-58

Joseph, K., L.H. Mattoso, R.D. Toledo, S.Thomas, L.H. de Carvalho, L.Plthen, S. Kala and B.

James. 2000. Natural fiber reinforced thermoplastic Composites. *Natural Polymers and Agrofibers Composites* 159:201

Mohanty, A.K., M. Misra and L.T. Drzal. 2001. Surface modification of natural fibers and performance of the resulting biocomposites: An overview. *Composite Interfaces* 8 (5): 313-343.

Munker, M., R. Holtmann and W. Michaeli. 1998. Improvement of the fiber/matrix-adhesion of natural fiber reinforced polymers. Proceeding of *the 43rd International SAMPE symposium*, May 31-June 4.

Panigrahi, S., L.G. Tabil, W.J. Crerar and S.Sokhansanj. 2002. Application of Saskatchewan grown flax fiber in rotational molding of polymer composites. Paper No. CSAE 02-302. Saskatoon, SK: Canadian Society of Agricultural Engineering.

Weyenberg V., J. Ivens, A. De Coster, B. Kino, E. Baetens and I. Verpest. Influence of processing and chemical treatment of flax fibers on their composites. *Composites Science and Technology*, 63, 1241 – 1246.