

Effect of Drying Methods on the Physical and Structural Changes in Oil-Seed Flax Fiber

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ABSTRACT

With the growing environmental concerns, biodegradable materials are gaining more importance. Biocomposites which are made from a combination of biological fiber such as flax and hemp together with plastics are finding a good number of applications in day to day life. Flax has good physical and mechanical properties that can be utilized in areas like construction, biomedical & bioproducts and electronics applications. The quality of fiber depends upon various unit operations used in the processing. Drying is one of the most important unit operations which significantly affect the quality of the fiber. The method of drying for removal of moisture from the fiber significantly affects the drying time and quality. In the present study the raw flax fiber was subjected to drying before and after chemical treatment. The physical properties such as; tensile strength, color and structural changes were measured for raw and chemically treated flax fibers. The diameter range was in-between 30-300 μm the tensile strength recorded for the entire range and were found to vary between 16 to 667 MPa and the elastic modulus values in the range of 2 GPa up to 63 GPa. The tensile strength and elastic modulus of untreated and treated fiber did not show any significant change. High power levels for longer period indicated some black spots due to localized heating. Differential scanning calorimetric data indicated that the degradation temperature of cellulose was found 350($\pm$ 10) $^{\circ}\text{C}$ for the treated and dried flax fiber. Among the drying methods microwave-vacuum took more time compared to microwave but could remove the moisture to less than 1 %. The maximum moisture removal using microwave-vacuum was achievable because fibers were dried for a longer period of time at comparatively low temperature.

Key words

Flax fiber, physical properties, morphological properties, biocomposites

INTRODUCTION

Natural fibers like hemp and flax have been used ever since prehistoric times but were mainly used for clothing purposes. With advances in science and technology, the natural agricultural fibers (NAF) are finding potential applications in the areas like biomedical, construction and electronics. Huang et al. (2007) has reported about the use of natural fibers in automotive parts and aerospace applications. One important reason why these natural fibers are gaining importance is due to degradable nature over time. Use of plastic and synthetic fiber is a big concern today as they do not degrade over time, costly and are not environmentally friendly. A typical example is most of the interior parts of an automobile are petrochemical based and these parts are disposed of either in landfills or by burning where both contribute to environmental pollution (Chen et al., 2005). However natural fibers are bio-degradable, environmentally friendly, renewable, non-abrasive, low in cost, and abundantly available in western Canada (Ghazanfari et al., 2006a; 2006b; 2006c). The physical properties of a natural fiber depend on various conditions like type of flax, environmental conditions, processing type, and its chemical treatment. The use of natural fiber along with plastics can help to reduce the plastic wastes generation. The important features of these biocomposites are they are non-corrosive, light weight, economical, and have comparable physical and mechanical properties and in-addition to that they are cheap and environmentally friendly. Natural fibers are used in the development of new materials used for construction, furniture, packaging, ropes, bags for grain storage, and even in automotive industries. All these advantages enable natural fibers viable composite materials. Presently auto manufacturers are trying to use parts made of materials that are fully recyclable or can be disintegrated easily. According to European Commission guidelines, car makers should use up to 95% recyclable parts by the year 2015 (Kemper and Hobi, 2003). They can either use interior parts made of single polymer or material which can easily be disintegrated like



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biocomposites. Biocomposites are composite materials that contain plastics and natural fiber. The application of biocomposites in automotive manufacturing will improve mechanical properties, lower the product weight, speed up processing time, enhance passenger safety, withstand extreme temperature, improve the insulation, and improve biodegradation ([Kemper and Hobi, 2003](#)).

Natural fiber in biocomposites works as a reinforcement material as they have high aspect ratio, high strength-to-weight ratio, and good insulation properties. These properties give natural fibers advantage over other synthetic fibers. However, the main disadvantage of not being popular in composite yet is that natural fibers are not compatible with matrix and also they are hydrophilic in nature. Being hydrophilic in nature they tend to absorb moisture from the atmosphere and create cavities. To reduce these cavity formations the fibers need to be dried before the compounding process for biocomposites production or extrusion. Chemical pre-treatment is normally carried to improve the adhesion between matrix and natural fiber. Studies shows that the adhesion between polymer matrix and natural fiber will be enhanced if it is treated by chemical reagents such as sodium hydroxide, trimethoxy silane, acetic acid, acrylic acid, maleated coupling agents, isocyanates, potassium permanganate, or peroxide. ([Ghazanfari et al., 2006](#); [Li et al., 2007](#)). After the chemical treatment, the natural fiber is cleaned with water. During all these operations fiber absorbs a large amount of water which has to be removed for further processing. A strong need for effective drying processes is therefore necessary to remove excess water and at the same time, retain the physico-chemical and mechanical properties of natural fiber. Some researchers use thin layer drying as a conventional process but there is very little literature available on the drying of natural fibers using microwave, microwave convective, and microwave-vacuum technology. [Panigrahi et al. \(2006\)](#) found that the efficiency of conventional drying method is very low due to improper utilization of energy. This method also takes a lot of time and consumes lot of energy for heating the air. [Panigrahi et al. \(2006\)](#) also concluded that microwave drying is very fast, but due to uneven distribution of heat, hot and cold spots may develop but the use of a rotating table may help maintain a uniform moisture distribution within the material to some extent. They have also concluded that discoloration was noticed at drying temperatures less than 190°C and moisture content fell below 1% at 200 and 300 W power levels, respectively. [Farmer and Brusewitz \(1980\)](#) investigated the use of a household microwave for alfalfa drying and for moisture determination. In general during microwave drying high temperatures are encountered which may result in degradation of quality of the materials.

Microwave and microwave-vacuum technologies have been used for a long time in the processing of agricultural products such as fruits and vegetables. This type of drying is now very

widely used in food industry for energy conservation and drying. For heat sensitive materials microwave-vacuum technology is recently being used to avoid quality loss ([Meda and Raghavan, 2004](#)). The addition of vacuum pressure to the microwave environment offers great advantages of dielectric heating but at a reduced processing temperature, which is the function of the operating pressure. In addition, a volumetric heat transfer mechanism coupled with drying in vacuum provides an ideal low-temperature drying technique resulting in better quality of dry fiber.

The studies on physical and morphological characterization of untreated and treated flax fiber after drying are important to understand their suitability in making of biocomposites. In general the overall quality of fiber is expressed based on fineness, length, strength, density, lustre, color, handle, alignment of strands, and absence of naps / knots. The four basic quality indicators of fiber which are important in terms of making biocomposites are a) fiber strength, b) color, c) morphology, and d) thermal degradation temperature. Very little or no information is available on physical characterization of natural fiber dried using microwave, microwave-convective, and microwave-vacuum technology.

The objective of the present study was to investigate the changes in the physical properties of flax fiber such as color, strength and structural changes in the natural fiber flax using microwave, microwave-convective and microwave-vacuum after treatment with alkaline and silane (trimethoxy vinyl silane) and without any treatment.

MATERIALS AND METHODS

PHYSICAL CHARACTERIZATION OF FIBER

The raw flax fiber which is procured is subjected to both silane and alkali treatment before drying using the microwave, microwave-convective and microwave-vacuum. The power levels of the microwave used in the present study are P₄ (371 W) and P₆ (614 W). The physical properties measured include diameter, tensile strength, color, morphology and thermal degradation temperature. The physical and chemical characterization of untreated and treated flax fiber is important to study the quality of fiber after chemical treatment and drying. In this research, two different chemical treatments of flax had been used with three different drying methods. There were twelve different treatments depending on the combination of chemical treatment, drying method, and power level. Raw fiber (untreated and undried) was also taken as a control to compare with treated and dried fibers. In the present research, two different chemical treatments of flax had been used with three different drying methods. There were twelve different treatments depending on the combination of chemical treatment, drying method, and power level. Also raw fiber

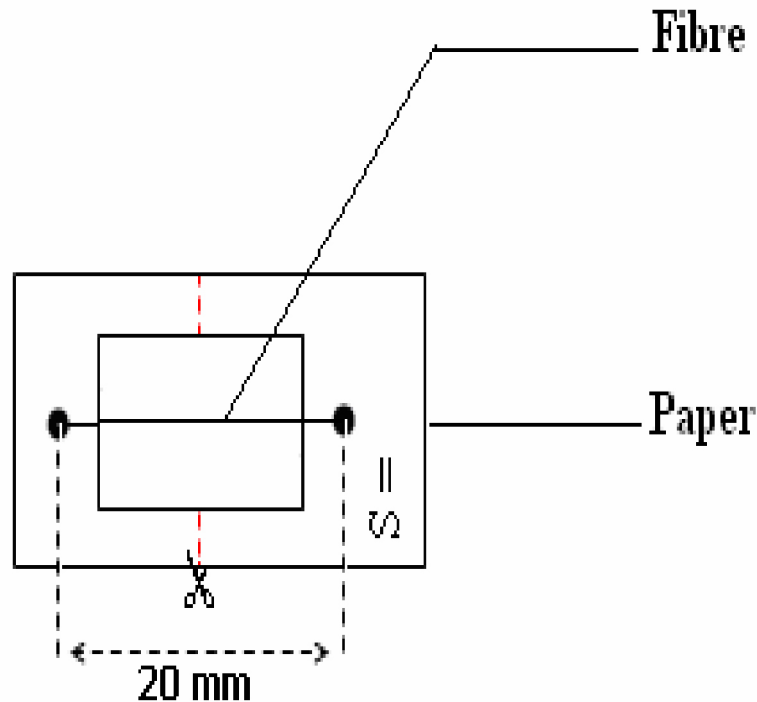


Figure 1. Schematic diagram of sample prepared for tensile test.

(untreated and undried) was also taken as a control to compare with treated and dried fibers.

DIAMETER MEASUREMENT OF FLAX FIBER

Length and diameter measurements of the flax fiber are required to measure the tensile strength. In this research, a bundle of flax fiber was selected. It was observed that the diameter of the fibers were not uniform along its length. It is wider at one end and shorter at the other, however it was assumed that the fiber is cylindrical shape and the diameter is circular for the convenience of conducting the experiments.

A single bundled fiber of uniform size was selected manually from bunch of fiber treated with alkali and silane. Simultaneously, samples from untreated and undried fiber were also selected. The schematic diagram of the sample was shown in [Figure 1](#). The prepared sample for the tensile measurement is shown in [Figure 2](#).

The fiber was selected manually by hand to obtain minimum number of fibers in a bundle and then glued onto a prepared paper sample holder that had a square hole in the middle. It was then glued with epoxy carefully by gluing two ends of the fiber. It was then left for 4-6 h for curing at ambient temperature. After the glue has been cured, the fiber diameter was measured using Nikon microscope (Model OPTIPHOT, Nikon Corporation, Japan) of 100X magnification fitted with PAX-IT digital camera. Nine replicates of single bundled

fibers were chosen for each of sample. The image was then magnified 100 times using a microscope. Each of the fiber samples was placed on a slide with a white background and eight readings were taken for each sample along the length of fiber and focusing on different parts of the fiber. As the fiber is not in a uniform cylindrical shape the diameter of the fiber varied from place to place. To overcome this problem the fibers were chosen in such a way that there would not be a big difference in the readings and a more uniform diameter of fiber would be selected. A canon microscope, PAX-IT camera and PAX-IT image acquiring and processing software. The experimental setup consisted of a Canon microscope with a 100× lens (10×10), and a PAX-IT digital camera fitted on the top interfaced with a PC with PAX-IT image capturing software. Once the image was focused and acquired it was then analyzed using PAX-IT imaging software. The obtained diameter was used for further calculation of tensile strength and elastic modulus of the treated and dried fiber for characterization. [Figure 3](#) shows the measured diameter of a single bundled fiber with PAX-IT camera and software.

INDIVIDUAL FIBER STRENGTH MEASUREMENT (TENSILE)

Fiber is used as a reinforcement material in biocomposites, so the tensile strength of the fiber must be measured before it is put into the composite product ([Morrison III et al., 2000](#)). In the literature reviewed above, it was found that the single fiber fragmentation test is a better method than the bundled



Figure 2. Prepared samples for measuring tensile strength of flax fiber

fiber fragmentation test. Because a single fiber is very fine, it is very difficult to measure tensile strength using Instron 1011 machine (Instron Corporation, Canton, MA, USA). Therefore, the tensile test was conducted using the Texture Analyser (Texture Technologies Corp., TX2, NY, USA). Manual clamps were used to hold the sample. Ten replicates were done for each sample. After each step of the chemical treatment and drying process, a sample was analyzed. After the diameter had been measured, the tensile strength of flax fiber was measured. The data was acquired through Texture Expert Exceed software (for Windows, version 2.64- English, Stable Micro Systems, Surrey, England) and saved in a text file. The test was done each time until the fiber breaks. The crosshead speed of 0.05 mm/s, the gauge length of 20 mm, and the pre-test speed of 0.02 mm/s was used in this experiment. The maximum force ($F_{\max}$) was recorded from the text file through Texture Expert Exceed software and the tensile strength was calculated using the equation a below:

$$\sigma_t = F_{\max} / A \quad (1)$$

where $F_{\max}$ was maximum load value (N) and A was the area of cross section (mm²).

The elastic modulus (E) was also calculated by following equation:

$$E = \Delta \sigma / \Delta \epsilon \quad (2)$$

where $\Delta \sigma$ was the change in tensile stress and $\Delta \epsilon$ was the change in tensile strain.

COLOR PROFILE

Color is one of the important product properties which play an important role in acceptability. Measurement of color is one of the vital parameters for a product development process. The measurement of the color of fiber after drying is important to understand the effect of fiber processing parameters on the appearance. Color measurements were done using the HunterLab spectrophotometer model LABSCAN2 (Hunter Associates Laboratory Inc, Reston, VA, USA). The color change of the dried flax was determined by measuring the L, a, and b coordinates. The scales used were L (0=black to white=100), a (-a=green to +a=red) and b (-b=blue to +b=yellow).

After initial calibration against a standard white ceramic surface plate, three measurements for each sample were taken. The viewing area of 12.8 mm and a port size of 25.4 mm were used in this experiment. After the calibration was done, the sample was prepared by weighing fiber of 2.46 g each time and put inside a transparent plastic petri dish. The petri dish was stuffed fully to avoid airspace and transmission of light through it. After each measurement was taken, the petri dish was cleaned thoroughly with alcohol. Each time three measurements were taken in different locations of sample and repeated on both sides.

L is the lightness indicator and a and b are the chromacity coordinates. The untreated fiber sample taken was standard and change were measured after fiber being treated and dried. Color difference values ΔL , Δa and Δb were calculated

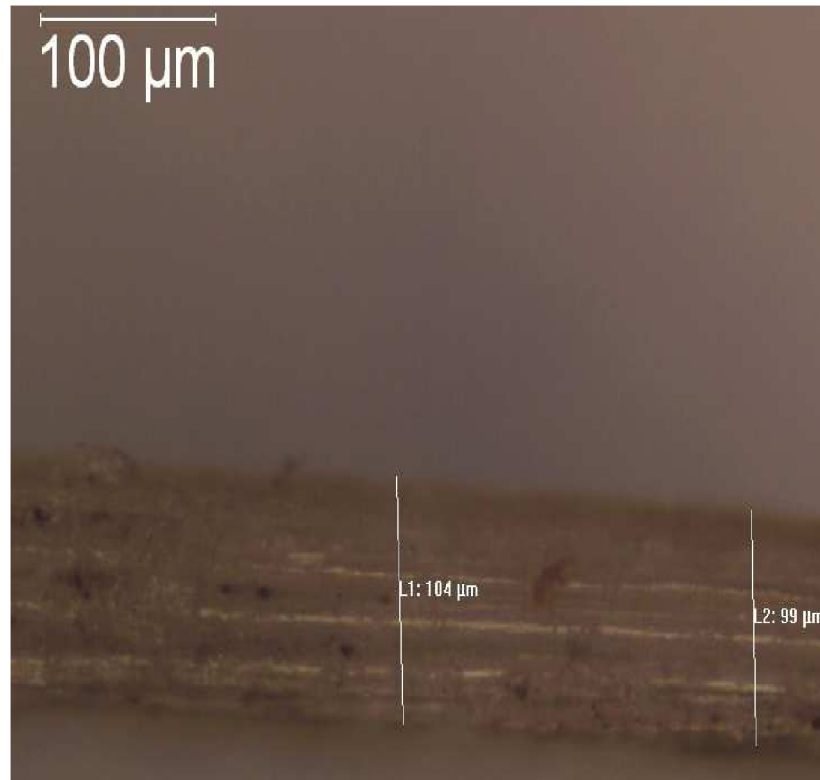


Figure 3. Measured diameter of a single bundled fiber with PAX- microscope.

according to the following equations: center format the equations

$$\Delta L = L_0 - L_1, \quad \Delta a = a_0 - a_1, \quad \Delta b = b_0 - b_1 \quad (3)$$

where L_0 , a_0 and b_0 are the measured values of the specimen (treated and dried sample), L_1 , a_1 , and b_1 are values of the target color (untreated sample).

The target colors in this experiment are L , a , and b of the untreated flax fiber. The total color difference ΔE_{ab} is measured using the L , a , b color coordinates and as defined by Eq. 4 (Minolta, 1991):

$$\Delta E_{ab} = \text{SQRT} \left[\frac{(\Delta L)^2}{(4)} + \frac{(\Delta a)^2}{(4)} + \frac{(\Delta b)^2}{(4)} \right] \quad (4)$$

The analysis of the data was carried out with help of statistical package SPSS 14.0 for Windows.

MORPHOLOGY

The scanning electron microscopy (SEM) gives a clear idea about the morphology of the fiber internal structure. Therefore, samples of fibers treated using the different chemical treatment and drying processes were analysed using

SEM (JEOL 840A, JEOL Ltd, Tokyo, Japan) at an accelerating voltage of 15 kV to examine the change in morphological structure. The sample should be conductive before viewing by the SEM. A small sample of thin fiber bundle was cut with 2 mm in length and paste with double sided tape on the sample holder. The sample was then sputtered with thin layer of gold. After it is conductive, sample can be mounted and placed in the vacuum chamber of the scanning electron microscope. Through this analysis, the change in the internal structure of the material can be studied before and after drying with different drying methods. As we know microwave drying generates localized hot spots; SEM can find if any hot spots appear after drying with microwave, microwave convection and microwave-vacuum dryers.

THERMAL DEGRADATION TEMPERATURE

Differential scanning calorimetric (DSC) analysis was used for studying thermal stability, decomposition, dehydration, oxidation, determination of volatile material, binder burnout, and high temperature gas corrosion. The decomposition or the degradation temperature is very important for the processing of natural fiber based biocomposite material. In this study, the degradation characteristics of flax fiber were analyzed using the DSC (DSC model Q2000, TA Instruments, New Castle, USA). This is necessary to determine how much temperature the flax fiber can withstand