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Article

Comparative Evaluation of Physical and Structural Properties of Water Retted and Non-retted Flax Fibers

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Abstract: Flax stems of Modran variety were subjected to water retting under laboratory conditions and its physical properties were compared with non-retted fibers. Physical properties including percentage of impurities, weighted average length, linear density, tenacity and elongation were analyzed and the results were compared. The analysis of retted and non-retted flax fibers showed that retting is the most important step in the processing of flax fibers and it directly affects quality attributes like strength, fineness, and homogeneity. Scanning Electron microscope images of fibers were also analyzed and the retted fibers showed much cleaner surface when compared to decorticated non-retted fibers.

Keywords: flax; water retting; linear density; tenacity

1. Introduction

Flax (*Linum usitatissimum*) is a fibrous plant and an important commercial crop grown all over the world. Flax plant is mainly grown for its seeds, which are used for various applications from cosmetic production to paint mixing [1]. Flax stem contains natural fibers, which could be used in various applications like production of biocomposites, apparels, high quality papers *etc.* [2,3]. Flax fibers are comparatively expensive and of high quality when compared to other fibers, which is due to the difficulty of it's processing. Natural fibers are low in density, cheap, and biodegradable. However, the big disadvantage of natural fibers is that they do not have same consistency in quality as compared to synthetic fibers. This inconsistency is due to variety of reasons such as climate, crop variety, retting process, and processing equipment used for fibers [4].

The processing of flax fiber from the stem is usually conducted by passing the straws through series of rollers under pressure exerted by the rollers. This action helps the outer skin, shives and other non-fibrous parts to detach from the fiber and the resultant product is a clean long fiber; the process is called decortication [3]. In decortication of non-retted flax stems, the resultant fibers are of inferior quality with impurities on it, because flax fibers are attached to itself and to the stem with chemical bonds such as pectin bond, which cannot be broken completely by applying mechanical pressure. In order to release the fiber completely from the stem, these stems have to undergo a process called retting. The retting process employs the action of micro- organisms and moisture on plants to destroy the cellular tissues and pectin surrounding blast-fibers bundles, and hence facilitating separation of the fiber from the stem [3]. Two primary methods for retting, namely water and dew retting have been used traditionally to extract fiber for textile and other commercial applications [5]. According to available literature, dew retting is considered to be the oldest method of retting. The quality of dew-retted fiber is inferior than water retted fiber, but due to lower labor costs and high fiber yield, it remains as an attractive practice for the farmers. In dew retting, the stems are pulled and spread uniformly on the ground in a thin layer for 3–4 weeks. Changes in moisture and temperature result in the colonization and partial degradation of flax stems, mainly by saprophytic fungi. The main disadvantage of dew retted flax fiber is its poor and inconsistent qualities. The field use of 3–4 weeks is also another drawback of the process [6]. In water retting, straw is submerged in water tanks, where a pectinolytic bacterial community develops [7]. Bacillus species are dominant for 10 to 40 h after the start of the water retting process, and are followed by spore-forming anaerobic Clostridium spp. when oxygen concentration in water tanks becomes lower. Clostridia, the major group of bacteria, which shows pectin-degrading activity, are responsible for water retting [8]. The sources of retting bacteria are suspected to be adhering soil particles, stem dust, air, and water. In water retting, consistent conditions for bacterial growth and activity exist, which results in the evenness of inoculum and colonization by bacteria and in turn the quality of the retted fiber [8]. Though water retting produces the highest quality flax fiber, the environmental pollution caused by it forced the industries to regulate the procedure and find other alternatives like enzyme assisted retting, steam explosion and electromagnetic energy assisted retting [5].

The objective of this study was to understand the effect of water retting at controlled temperature on physical properties of Polish flax variety Modran.

2. Experimental Section

2.1. Flax Straw

Non-retted flax straws were collected from the Institute of Natural Fibers and Medicinal Plants, Poznan, Poland. The average technical length and thickness was measured by taking average of 100 samples. The technical length is the distance between the beginning of root and first branch. Thickness of the straw was measured at the middle of the straw samples.

The average technical length of the flax straw was 60.42 ± 1.02 cm and the average thickness of the straw was 1.54 ± 0.13 mm. The color of the flax straw bundle was 60% yellowish and 40% greenish, which threw light on the maturity of the flax stems, because yellow stem indicates maturity and the green stem relatively less maturity. The color analysis was carried out by visual analysis.

2.2. Non-retted Flax Fiber

Non-retted flax fibers were processed by decorticating the non-retted flax stems. The decorticated flax fiber contained high percentage of shives, dust and other impurities.

2.3. Water Retting of Flax Straw

Water retting chamber was a stainless rectangular chamber of 2 m × 1 m dimension. The chamber was connected to a temperature control system, which maintains the temperature of the water in the chamber during retting. The samples were kept in the retting chamber and concrete bricks were placed on the top of the straw bundles to avoid the straw from floating. After keeping the samples inside the chamber, the chamber was filled with water until all the samples were submerged in water. The temperature of the treatment was controlled at 32 °C for better bacterial activity and the duration of treatment was 72 h. The chamber after 72 h is as shown in Figure 1.

Figure 1. (**a**) Water retting of flax stems; (**b**) water retting chamber after 72 h of retting.

(**a**) (**b**)

After the treatment duration of 72 h the water was drained out and retted samples were rinsed. The samples were then passed through a roller press twice before subjecting them to drying. The samples were transferred to a hot air oven for 24 h at 80 °C. Once the drying process was complete the samples were taken out of the oven and kept at room temperature for 72 h before further processing. This is needed to acclimatize the fiber to the processing temperature and reduce any impact of processing on the physio-chemical properties of the fiber.

After three days, the dried samples were passed through a turbine-scutching machine, which separates long and short fibers without impurities.

2.4. Measurement of Impurity

Impurities were separated from a known quantity of flax fiber samples and the impurities were weighed. Impurities included shives, dust, wax, *etc.* If the total mass of sample is represented as M and the weight of impurities was m_i, the percentage of impurity is calculated using the following equation.

$$\text{Percentage of impurities (\%)} = \frac{m_i}{M} \times 100 \tag{1}$$

2.5. Scanning Electron Microscope (SEM) Analysis of Fiber Structure

The cross sectional and longitudinal analysis of flax fibers were determined by using a SEM (Hitachi S- 3400 N). The samples were prepared and attached on a metal plate, which was later transferred to a chamber for gold coating. The samples were then inserted in the SEM chamber and magnified to study the structural properties.

2.6. Weighted Average Length of Fiber

The length of the fiber was analyzed manually by placing them on a black sheet of paper and separating them into various ranges or classes starting from 0–2 cm, 2–4 cm to 68–70 cm Figure 2. After the process of separation, the flax fibers in each class were weighed to nearest 0.001 g and the percentage by weight in each section was documented [9]. The climatic condition for the experiment was set at a temperature of 20 ± 2 °C and 65 ± 2% relative humidity. The length of the average weight was calculated by using the following equation:

$$\textit{Weighted average length} = \frac{Sum\ of\ (average\ length\ of\ the\ class\ x\ mass\ of\ the\ fibre)}{total\ mass\ of\ the\ fibre} \tag{2}$$

Figure 2. Flax fiber separated in different classes of length 0–2 cm (extreme right) to 68–70 cm (extreme left).

2.7. Linear Density (Thinness)

The linear density or thinness of the fiber determines its fineness, and it is expressed as "Tex". Prior to linear density analysis, the samples were acclimatized under ambient conditions of temperature of 20 ± 2 °C and relative humidity of 65 ± 2%. Samples were prepared by cutting sections of 10 mm length from the middle portion of the flax fibers to ensure the evenness in analysis. From the samples, 100 fibers were separated in such a way that if a given fiber splits into two up to half of the length of the fiber, it should be considered as two separate fibers and should be counted as two fibers. Sometimes from one-fiber sample, we could get three or more separate fibers. After counting 100 fibers, the samples were weighed to nearest 0.1 mg and linear density was estimated using the following equation.

$$\text{Tex} = \frac{m}{n} \tag{3}$$

Where, m is the mass of the samples and *n* is the number of samples. The experiment was repeated five times for a better statistical validation.

2.8. Tenacity and Elongation

Breaking tenacity and elongation was measured by using STATIMAT ME testing machine (Textechno Herbert Stein GmbH & Co. KG, Germany). The acclimatized flax fiber samples without any impurities on the basis of mass (20 Tex) were taken and the length of the samples were fixed at 7 cm. Each sample was then mounted on the testing machine with a gauge length of 3 mm. 100 N load cell and the test speed of 10 mm/min was used to estimate the breaking tenacity, elongation and coefficient of variation of breaking force.

3. Results and Discussion

The fibers obtained from decortication without retting and long and short fibers after retting were tested for their physical properties and are reported here.

3.1. Impurities

The impurities in non-retted flax fibers and retted flax fibers were measured and tabulated in Table 1. From the table, it can be observed that the percentage of impurities was maximum in non-retted decorticated flax fibers and minimum in retted turbine scutched long fiber [10]. Short fibers were separated with the impurities in the scutching machine because they contained more shives (small wooden particles). In non-retted flax fiber, the stem parts are attached closely to the fiber bundles by strong pectin bonds [2]. Hence, it was concluded that decortication process without retting is not enough to obtain pure fibers.

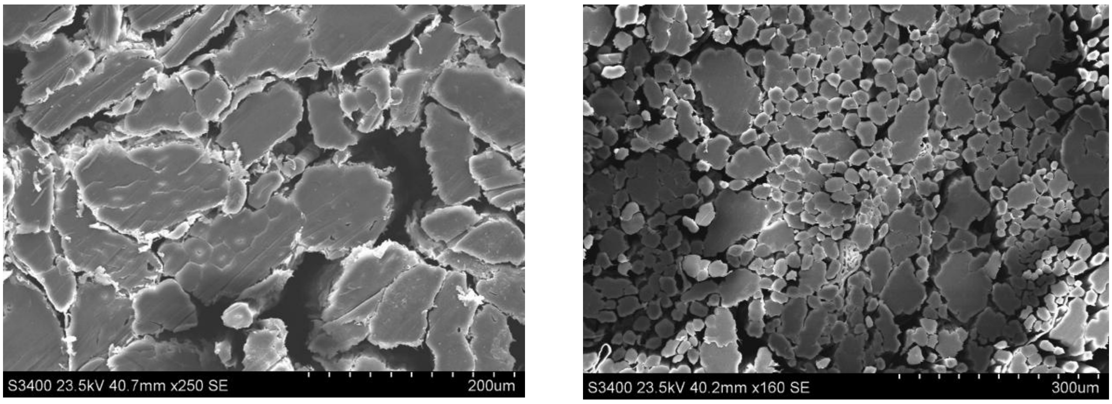
Table 1. Percentage Impurities in fibers.

Fibers	Impurity (%)	SD
Non retted	38	0.05
Retted (long)	0.3	0.01
Retted (short)	16.5	0.03

3.2. SEM Analysis

The SEM cross-sectional views of non-retted and retted fibers are shown in Figure 3.

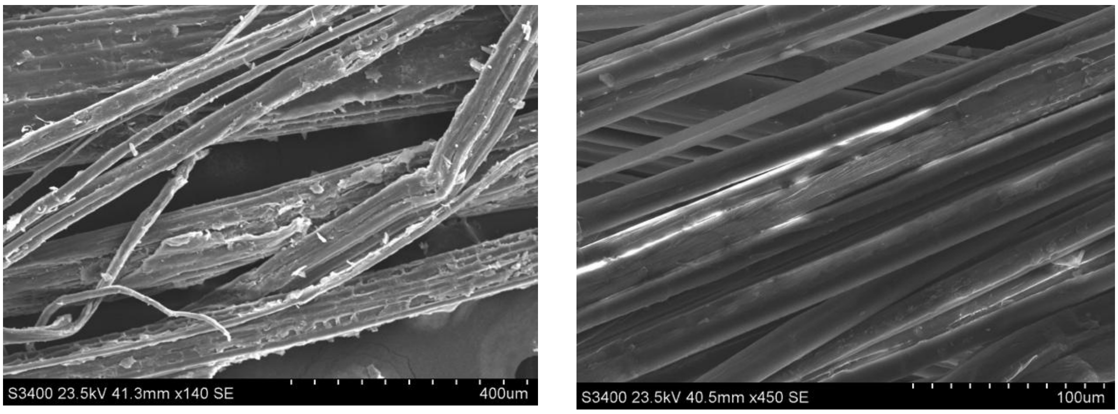
Figure 3. Cross-sectional view of (**a**) non retted and (**b**) retted flax fibre.



(**a**) (**b**)

It can be observed that in non-retted flax fiber (Figure 3a), the fiber's were bundled because waxes, shives and other impurities were attached to them [11,12], but the retted fibers (Figure 3b) were more separated and of smaller cross-section because of retting. The effect of retting in processing of flax fiber is illustrated in Figure 4. The non-retted flax fiber's (Figure 4a) were rough and its surface contained lots of foreign particles like wood parts, dust *etc.* However, retted flax fiber's (Figure 4b) were clean and of high visual quality.

Figure 4. Longitudinal SEM view of (**a**) non-retted and (**b**) retted fiber.



(**a**) (**b**)

3.3. Weighted Average Length

Figures 5–7 represent the percentage weighted average length of non-retted and retted short and long fibers.

Figure 5. Percentage of lengths by weight–non-retted flax fiber.

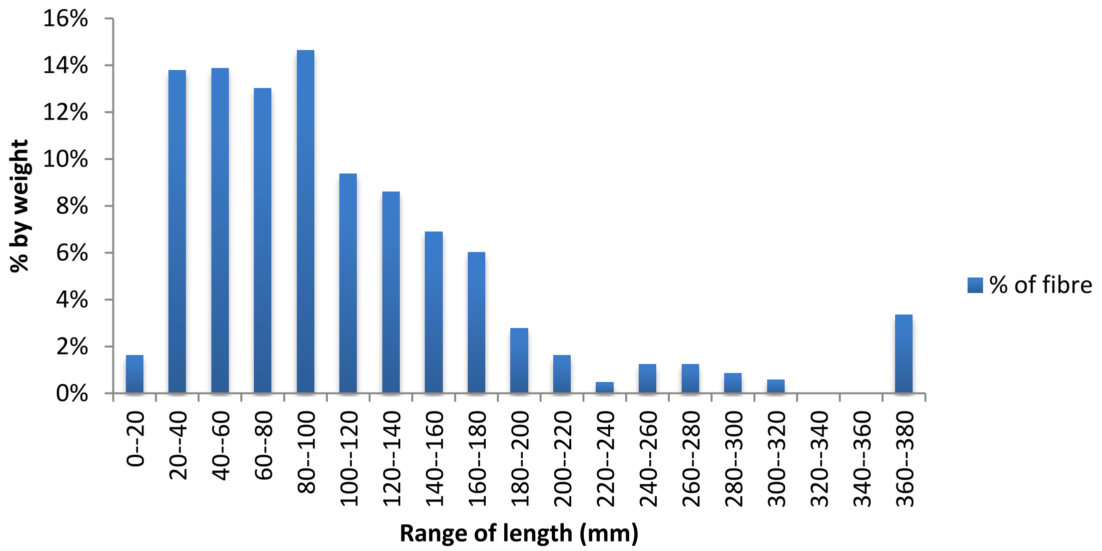


Figure 6. Percentage of lengths by weight–retted flax fiber (long).

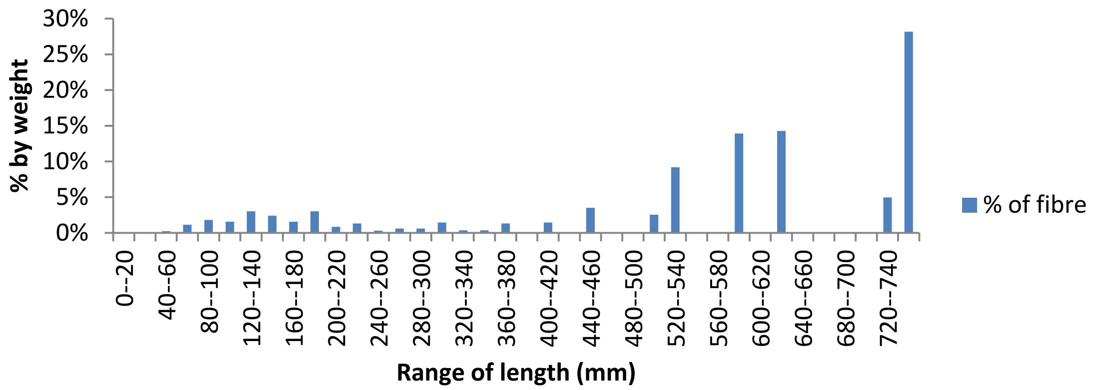
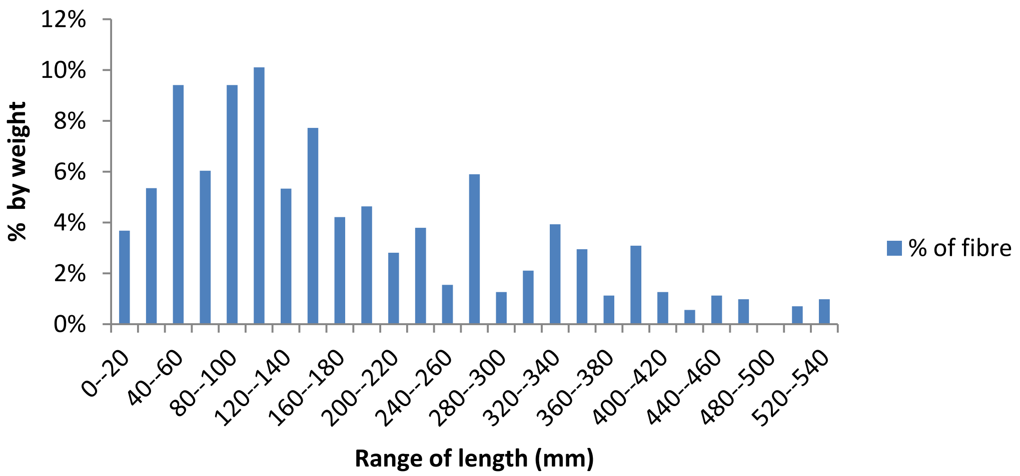


Figure 7. Percentage of lengths by weight–retted flax fiber (short).



Fifty percent of non-retted flax fibers were of the length between 20 mm to 100 mm and 30% of retted long fibers were within the range of 740–760 mm. Fifty percent of retted short fibers were located within 20 mm to 160 mm range. The weighted average length of the flax fibers were, 108.5 mm, 543.9 mm and 175.1 mm for non-retted, retted long fiber and retted short fiber respectively. The weighted average length signifies the application of fibers; long fibers are used mainly in textile industries whereas short fibers are used in bio-composites and paper industries [5].

3.4. Linear Density (Thinness) of the Flax Fibers

Table 2 represents the linear density (thinness) of all the flax fibers. It can be observed that retted long and short fibers had the lower linear density in comparison to non-retted flax fibers. A similar result was observed in the SEM analysis of the fibers.

Table 2. Linear density of flax fibers.

Samples	Linear density (Tex)	Coeff. of variance (%)
Non retted	3.26	19
Retted (long)	2.54	9
Retted (short)	2.3	19

3.5. Tenacity and Elongation

Maximum load, elongation, work to rupture, bundle strength and time to rupture are tabulated in Table 3.

Table 3. Strength test result comparison of retted and non-retted fibers.

Samples	Force (N)	Elongation (%)	Work to rupture (N.cm)	Bundle strength (daN/mg)	Breaking force cN/Tex	Time to rupture (sec)
Non-retted	58.75	41.35	4.012	2.218	39.73	12.16
Retted (long)	72.76	44.67	11.09	2.73	39.97	29.78
Retted (short)	65.64	42.9	4.74	2.38	41.37	13.37

The breaking force was 39.73 cN/Tex for non-retted fiber, 39.97 cN/Tex for retted long fiber and 41.37 cN/Tex for retted short fiber. It was concluded that the strength of the retted fiber was more than that of non-retted flax fiber. There are not much difference among the elongation percentage of all the samples. The strength analysis revealed that, retting improved the bundle strength, breaking force, work to rupture and elongation.

3.6. Applications of Retted and Non-retted Fibers

Flax fibers have many applications depending on their physical qualities; some of the major applications are discussed below.

3.6.1. Specialty Paper and Pulp

Flax fibers are used to make high quality papers for currencies, cigarettes, filter media, condenser and battery separator pages. Hammer milled non-retted oilseed flax fibers are used to make specialty pulps for high quality papers [13].

3.6.2. Insulation

Non-retted flax fiber composites are used as both sound and thermal insulators. Glass and synthetic fiber reinforced mats are avoided mainly because of its low degradability which raises concerns regarding environmental pollutions [13].

3.6.3. Geotextiles

Geotextiles are used for making ground cover to suppress weeds, control erosion by keeping the top soil, rain water filtration and should be degradable as well. Non-retted flax fibers could be used as geotextiles while considering its strength, hydrophilic nature, and degradability [14].

3.6.4. Building Products

Flax fiber composites are used for making various types of building materials replacing concrete blocks to make houses, substitutes for wooden materials, and plastic materials [15].

3.6.5. Textiles

Retted long fibers are used in the textile industry. The processed flax fibers will be subjected to various processing steps like bleaching, coloring, weaving to obtain the final product [16].

3.6.6. Bio-plastic Composites

Retted long and short fibers are used in the plastic composite industry; Composite industry includes lots of possible end-use applications ranging from airplane bodies to pipes, to mining slurry containers to pocket calculator bodies. Bio- plastic composites are fiber-resin matrix, which is made by compression molding, vacuum molding, sheet compound molding, rotation molding, extrusion molding, injection molding and shoot and spray molding [17].

4. Conclusions

After the analysis of physical qualities of retted and non-retted flax fibers, it can be concluded that retting is the most important step in the processing of flax fiber in terms of its physical and structural qualities. The fibers from retted flax straws could be used for high end applications like in textile and automotive industries, whereas the fibers from non-retted flax fibers can be used for lower quality products including yarn, door mat, *etc.*

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Conflicts of Interest

The authors declare no conflict of interest.

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