Fiber extraction efficiency, quality and characterization of cattail fibres for textile applications

Koushik Chakma, Nazim Cicek and Mashiur Rahman
Department of Biosystems Engineering, University of Manitoba, Winnipeg, MB, R3T 5V6

Abstract
An investigation has been carried out to characterize the textile properties of extracted cattail fiber. It was found that unlike bast fibers, no retting was necessary to extract the fiber from cattail plants as whole cattail plants (stem and leaves) could be transformed into fibers under controlled experimental conditions in aqueous alkaline solution. Since the whole cattail plant was utilized, the fiber yield (%) was found to be between 40 to 60% at 80°C. It was found that the crimp-adjusted fiber length and plant length is similar. The diameter of the fiber is similar to cotton, however, statistically different than wool. The cattail fiber has burning behavior similar to cotton and decomposition temperature higher than cotton. The moisture regain (%) of cattail fiber is found to be ≈10% at 60% RH and 25°C.

SEM microphotographs revealed two distinct layers: epidermis and probable fiber layer as well as individual elliptical cell, lumen and canal between the cells. Further, SEM micrographs showed a unique submicroscopic 'crenelated' (rectangular indentation) structure.

The textile and submicroscopic characteristics of cattail fiber suggests that the fiber may be used for apparel and non-apparel applications (insulation and biomedical).

Keywords: Cattail fiber, alkaline treatment, Textile properties, Combustion behavior, Crenelated structure.

1. Introduction
Cotton and polyester fibres dominate both apparel and non-apparel textile markets. The production of both natural and synthetic fibers has significant impacts on the environment. Cotton requires enormous amounts of pesticides and water. To produce 1kg of cotton it can take more than 20,000 liters of water (WWF, ND). Polyester production also requires large quantities of expensive chemicals (petrochemicals) which is energy, and non-disposable fibers that do not degrade quickly in landfills. Both contribute same accountability for environment pollution. Other bast fibres (flax, hemp, jute and canola) are produced from plant stems, stiff and have very limited applications.
Hundreds of thousands of neglected wetland plants exist all over the world. Since many years ago some natural wetland plants have been considered as potential crops because of their high productivity, interesting chemical composition, and natural growth on a substantial portion (Anderson et al., 1984). One of them is *Typha Latifolia*.

Recent studies found harvesting of cattail wetland plant provides only in economic benefits due to their enormous growth rate and yield but also in environmental as well (Krus et al., 2014). Moreover, cattails help with reducing excess nutrients to capture and remove nutrients thereby reducing nutrient loading (i.e. phosphorus) to aquatic systems (Grosshans, 2014). Researchers have established that cattails can be used as a source of “biofuel,” fermented to produce ethanol and gas tanks. The phosphorus from cattail ash could be used as a crop fertilizer. Cattail can be incorporated with thermoset or thermoplastic or other biodegradable polymers and new functional forms such as interior partition, in packaging, electronic display, charcoal bars, and use for waste-water treatment (Luamkanchanaphan et al., 2014, Sundari and Ramesh, 2012).

Cattail (*T. Latifolia*) cellulose are considered as a new vegetable fiber source (Sana et al., 2014). Further, the chemical constitutions of cattail are composites of predominantly 63% cellulose, 8.7% hemicellulose, 40% fiber, 8.9% moisture content, 9.6% lignin and pectin and other water soluble matters such as 1.4% wax and 2% ash (Sopit, 2007). The cellulose content in various bast fibres lie between 60 to 80% and in cotton, the cellulose content is about 90%. Considering the total cellulose and hemicellulose content (%), it is reasonable to assume that cattail fiber, if it can be extracted, might possess textile characteristics.

Therefore, the objective of this current study is to develop a method to extract fiber from the cattail plant and characterize the extracted cattail fiber for textile apparel and non-apparel applications.

### 2. Methods

#### 2.1. Materials

The main materials were collected from the cattail on December 2, 2016 in a constructed wetland Niverville, Manitoba (49°35'42.7"N, 97°02'50.3"W), Canada and it was simply cut by using knife. The plants were collected during fall term and identified as *Typha Latifolia* the green plant with the flower dark brown in color with cylindrical shape. The stem is light green in color, 6-12 mm in diameter. The branches have 4-10 leaves or blades which measures up to 2.5 m long and 10-27 mm wide.

#### 2.2. Bast fiber retting

In order to make the filament bundles appropriate for spinning, non-cellulose substances must be removed through fiber extracting or degumming process. The traditional retting methods were applied at cattail fibers were extracted from epidermis (outer surface) layer on the tested plant by most widely practiced method called water retting. Cattail plants were cut in 5cm and placed into several beakers performed by submerging in cold water, hot water, acid, alkali, and enzyme treatment at room temperature (21°C). In order to establish better fiber extraction chemical methods applied and Launder-o-meter (SDL-Atlas) was used with a machine speed of 40 ± 2 rpm and pre-determined set of time and temperature. Following completion of the treatment, fibres were manually separated from the plants and washed in distilled cold water.

#### 2.3. Determination of yield (%) by weight loss

The yield of fibers (R %) is measured by the percentage of the ratio between the final mass of the fibers after chemical extraction process (Mf) and that of the *Typha* plant before chemical extraction process (Mi), which is given in Equation 1.

\[
R (%) = \left( \frac{Mf}{Mi} \right) \times 100 \quad \text{(Equation 1)}
\]
2.4. Diameter measurement
The diameter of the fiber was determined using Bioquant Analyzer which is connected with a projection microscope and camera. Diameter of 100 cattail fiber samples was measured alongside cotton and wool fibres. Statistical analysis was done to compare the fiber diameter between cattail, cotton and wool.

2.5. Moisture regain (%) measurements
Moisture regain of cattail fiber was measured according to the ASTM D 1776/D1776M -16. The samples were kept in a humidity chamber at 60%RH and 25°C for a set period of time and by oven drying the fibers at 105°C for ten hours. The moisture regain was calculated as the ratio of the amount of water absorbed to the dry weight of the sample.

\[ \text{Moisture Regain(%) } = \frac{W}{O} \times 100\% \]  

(Equation 2)

Where:
- \( W \) = Weight of water in the fibers (g)
- \( O \) = Oven dry fiber weight (g)

2.6. Scanning electron microscopy (SEM)
SEM method was used to investigate the cattail fiber surface appearance and cross-section. The sample molds were prepared from cattail plant and fibers using gold coating and without coating. Morphologic details signal was collected with ETD (Everhart-Thornley detector) and LFD (large Detector Field). Different detectors such as secondary electron commonly known as (ETD) in HiVac at low pressure for Coated cattail samples, LFD in LoVac mode at high pressure for uncoated cattail samples, Concentric Backscattered Electron (BSE) Detector (CBS) in Hi and LoVac mode at high pressure for cattail samples.

2.7. Thermal analysis

2.7.1. Burning behavior
The most common test for textile materials is burn-test-response method used to study burning characteristics of cattail fiber compare to cotton and polyester fiber. In this experiment tweezers had been used to hold cattail, cotton and polyester fiber clusters. The fibers were slowly brought toward the flame at 45° angle to bunsen burner and observed its reaction as the flame is approached. Further, fiber burning behavior while in the flame and its reaction following the removal from the flame was also observed.

2.7.2. Decomposition temperature
The decomposing point analyzer, used for analyzing thermal decomposition temperature, was comprised of a heating stage controller with link pad (Model: T95 HS, Make: January, 2011), an imaging station (Linkam Scientific Instrument, UK) and a monitor (Brand: Dynax, Model: DX-22L 150A11, Make: August, 2011). The final decomposition temperature was determined by the visual analysis of the color change on the sample displayed on the monitor.

3.0. Results and discussion

3.1. Water retting
In general, the bast fibers (flax, canola, hemp and jute) are extracted from epidermis (outer surface) layer of the plant by most widely practiced method called water retting. However, water retting method did not produce any fiber either at 20°C for 16 days, (Sample: S1-W; Figure 2) and at 80°C for 6 hours (Sample: S2-W; Figure 8) as shown in Table 1.
3.2. Chemicals retting

Different chemical retting such as alkali, enzymes, acid and alcohol were used for fiber extraction from cattail plant and the results are shown in Table 1. Only alkali retting at 60°C (Sample: S3-NaOH, Figure 9) and at 80°C (Sample: S5-NaOH, Figure 11) produced fiber, while acid at 21°C/16 days (Sample: S1-CH₃COOH, Figure 3), 60°C/6 hours (Sample: S4-CH₃COOH, Figure 10) and at 80°C/8 hours (Sample: S6-CH₃COOH, Figure 12); enzyme at 21°C/16 days (Sample: S1-Enzyme, Figure 6), and at 40°C/30 minute (Sample: S-Enzyme, Figure 7); and alcohol at 21°C/16 days (S1-Alcohol, Figure 5) retting failed to produce any fiber. Although no fiber can be seen at 21°C in NaOH for 16 days (Sample: S1-NaOH, Figure 4), after 45 days of retting, fibres were visible when pulling by fingers (data not shown here). The fiber yield (%) for NaOH retting depends on the retting temperature that at 60°C the yield (%) is 78.0, while at 80°C the value is 56.0 (Table 1). It is worth mentioning here that unlike other bast fiber retting where the fiber is extracted from the outer layer of the plant, for cattail plant – whole fiber could be transformed into fiber and no fiber extraction is needed. Further, the yield (%) for cattail plant is much higher than other plant fiber.

Table 1: Effect of different chemicals treatment in fiber extraction

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Time, temp.(°C) &amp; conc.</th>
<th>Plant specimens after retting</th>
<th>Yield (R %)</th>
<th>Sample ID</th>
<th>Time &amp; temp.(°C)</th>
<th>Plant specimens after retting</th>
<th>Yield (R%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>N/A</td>
<td>Figure 1</td>
<td>N/A</td>
<td>S7-Enzyme</td>
<td>30 minute</td>
<td>Figure 7</td>
<td>Neg.</td>
</tr>
<tr>
<td>*S1-W</td>
<td>16 days 21°C, 3%</td>
<td>Figure 2</td>
<td>**Neg</td>
<td>S2-W</td>
<td>6 hours 80°C, 3%</td>
<td>Figure 8</td>
<td>Neg.</td>
</tr>
<tr>
<td>S1-CH₃COOH</td>
<td>16 days 21°C, 3%</td>
<td>Figure 3</td>
<td>Neg</td>
<td>S3-NaOH</td>
<td>6 hours 60°C, 1%</td>
<td>Figure 9</td>
<td>78.0</td>
</tr>
<tr>
<td>S1-NaOH</td>
<td>16 days 21°C, 3%</td>
<td>Figure 4</td>
<td>Neg</td>
<td>S4-CH₃COOH</td>
<td>6 hours 60°C, 1%</td>
<td>Figure 10</td>
<td>Neg.</td>
</tr>
<tr>
<td>S1-CH₃CH₂OH</td>
<td>16 days 21°C, 3%</td>
<td>Figure 5</td>
<td>Neg</td>
<td>S5-NaOH</td>
<td>8 hours 80°C, 3%</td>
<td>Figure 11</td>
<td>56.0</td>
</tr>
<tr>
<td>S1-Enzyme</td>
<td>16 days 21°C, 3%</td>
<td>Figure 6</td>
<td>Neg</td>
<td>S6-CH₃COOH</td>
<td>8 hours 80°C, 3%</td>
<td>Figure 12</td>
<td>Neg.</td>
</tr>
</tbody>
</table>

* S: sample; W: water; Enzyme: Pectinase from Aspergillus Aculeatus; Neg: Negligible; M:L-1:50;

3.3. Effect of various alkali on fiber yield

Since cattail fiber was obtained using aqueous NaOH solution, two other alkalis (KOH and LiOH) were used fiber extraction. The fiber yield (%) with experimental conditions for these two alkalis is given in Table 2. Similar to NaOH treatment, fiber was obtained for both KOH and LiOH in all experimental conditions (Figures 13-23). The fiber yield (%) decreases with the increase in treatment time. For example, at 80°C in KOH, the fiber yield (%) is 50 (S8-K1), 55 (S8-K2), 35 (S8-K3) and 15 (S8-K4) after 2, 4, 6 and 8 hours respectively. Similar trend in decreasing yield (%) with treatment time can also be seen at 95°C in KOH (S9-K5, S9-K6 & S9-K7) and at 80°C in LiOH (S10-L1, S10-L2, S10-L3 & S10-L4). However, for the similar treatment conditions, the yield (%) at 95°C is much lower than the yield (%) at 80°C (Table 2). It is worth noting here that for the equivalent treatment conditions, the yield (%) in LiOH is much higher than the KOH – particularly after 6 and 8 hours’ treatment. For example, after 6 hours’ treatment at 80°C, KOH treatment produced 35% fiber (S8-K4), whereas the LiOH produced 56% fiber (S10-L3), and after 8 hours’ treatment, KOH treatment produced 15% fiber (S8-K4) compared to 53% fiber (S10-L4) produced in LiOH.
between 12.2±1.1 mm and 12.8±2.1 mm respectively. Statistical analysis showed that the difference in diameter was significant at 95% confidence level. For comparison, cotton and wool fibers have diameters of 12.7±2.1 mm.

The fiber diameter is related to softness, pliability, and hand. The thinner fibers bend more easily and fabric becomes softer and drapery. The cattail fiber diameter [Sample: S8-K3(6/80)] is found to be 12.7±2.1 mm. For comparison, cotton and wool fiber diameters were also measured, which were 12.2±1.1 mm and 12.8±2.1 mm respectively. Statistical analysis showed that the difference in diameter between cattail and cotton (t_cal,198=2.11>t_0.01=1.96) is significant at 95% confidence level. However, the...
difference in diameter between cattail and wool is not significant ($t_{cal,198}=0.337<t_{0.05}=1.96$) [Booth, 1968, p40].

3.4.3. Microscopic evaluation
The scanning electron microscopy micrographs are shown in Figures 25-29 and major submicroscopic features are given in Table 4. It appears that the cross-section of cattail plant is divided into two distinct layers - outer layer (epidermis, diameter 38.5 mm) and inner layer (probable fiber layer, diameter 20.1 mm) [Figure 28] and the ratio of fiber to plant area is 52.0%. This ratio is almost similar to the fiber yield (%). Each fiber is comprised of numerous cells (=55 in 740 µm² area, 4.5 – 6.0 µm²/cell) which are elliptical in shape and the distance between each cell is about 0.70 µm (Figures 25-26). Each cell has lumen and the size of the lumen varies which was as high as 1.03 µm (Figure 27). It was also found that the cattail fiber is 'crenelated' (rectangular indentation) (Figure 29).

Table 4: Submicroscopic structural features of Cattail fiber

<table>
<thead>
<tr>
<th>Sub-microscopic features</th>
<th>Dimension</th>
<th>Figure no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Individual Cell Size and Distance of Cattail Fiber</td>
<td>4.5 – 6.0 µm²/cell</td>
<td>25</td>
</tr>
<tr>
<td>Single Cattail Fiber Diameter</td>
<td>30 µm</td>
<td>26</td>
</tr>
<tr>
<td>Small ridges that correspond to the fibers crenelated (rectangular indentation) shape</td>
<td>---</td>
<td>27</td>
</tr>
<tr>
<td>Lumen diameter</td>
<td>1.03 µm</td>
<td>28</td>
</tr>
<tr>
<td>Two distinct layers</td>
<td>38.5 mm (outer layer) and 20.1 mm (inner layer)</td>
<td>29</td>
</tr>
</tbody>
</table>

3.4.4. Moisture regain of textile materials
The moisture regain (%) of cattail plants and cattail fiber obtained from various treatments and conditions (relative humidity and temperature) is given in Table 5. For comparison purpose, wool fiber (raw), whose moisture regain has already been established, is also determined. The use of wool fiber helped to determine the accuracy of the moisture regain measurement process.
The moisture regain (%) for cattail plant ranges between 9.4 to 12.7 (data not shown here). For cattail fiber, the moisture regains (%) was found to be about 10% at 59% RH and 25°C, which is similar to wool (Table 5). In order to determine the hysteresis of cattail plants, samples were conditioned from atmospheric condition and dry state. The moisture regain (%) was 10.6 and 9.6 for cattail samples conditioned from atmospheric and dry states respectively (Table 5 and the difference in moisture regain was found to be 1.0. Similar experiment was conducted for wool fiber and the hysteresis was found to be 2.3. The hysteresis value for wool fiber varies depending on the wool fiber type (woolen, worsted), yarn type and other factors. However, a more reasonable value seems to be 2.1% as reported by Cookson and Slota (1993). It is worth mentioning here that the experimental moisture regain (%) obtained from the current study for wool fiber is similar to the published value (Booth, 1968).

### 3.4.5. Burning behavior of cattail fiber

This burning behavior was carried out to observe how cattail fibers respond to flame. Cotton and polyester fibres were used to compare the burning behavior of cattail fiber with these fibres in Table 6.

<table>
<thead>
<tr>
<th>Fiber</th>
<th>Approaching flame</th>
<th>In the flame</th>
<th>Removal from flame</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Shrink</td>
<td>F</td>
<td>Smoke /Smell</td>
</tr>
<tr>
<td>Cotton</td>
<td>No</td>
<td>No</td>
<td>White/ Paper or Wood</td>
</tr>
<tr>
<td>PET</td>
<td>Yes</td>
<td>Yes</td>
<td>Black/ Chemical, Sweet</td>
</tr>
<tr>
<td>Cattail</td>
<td>No</td>
<td>No</td>
<td>White/ Paper or Wood</td>
</tr>
</tbody>
</table>

F: Fuse; R: Rapidly; S: Slowly; SE: Self-extinguish; PET: Polyester;

Burning behavior of Cattail is almost similar to Cotton, for example, Cattail does not produce fume when approach to flame and in the flame do not melt, however, burn rapidly and not self-extinguished after remove from the flame like Cotton. Polyester produced hard black bead where Cotton and Cattail produced grey and feathery ash residue.

### 3.4.6. Thermal properties

The thermal behavior of cattail plant, cattail fiber and cotton fiber are presented in Table 7. In order to obtain the thermal decomposition point, the machine was set to run up to 300°C without any intermediate holding temperature and holding time. The decomposition temperature for cattail plants, cattail fiber and cotton fiber was found to be 234.9°C, 268.7°C and 248.3°C respectively (Table 7). The
decomposition point was recorded when the control materials [Figures 30(Left)-cattail plant, 31(Left) - cattail fibre & 32(Left) - cotton fibre] turned dark brown or yellow color as shown in Figures 30(Right), 31(Right) and 32(Right) for cattail plant, cattail fiber and cotton fiber respectively. The lower decomposition temperature for cattail plant than the cattail fiber may be due to the presence of lower amount of non-cellulosic materials in the cattail fiber. However, this value should be taken with caution as the decomposition is conducted by pyrolysis method not hot plate method.

Table 7: Thermal properties of cattail plant, cattail fiber and cotton fiber

<table>
<thead>
<tr>
<th>Material type</th>
<th>Decomposition temperature (°C)</th>
<th>Comments</th>
<th>Figure #</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cattail plant</td>
<td>234.9</td>
<td>Fibers turn to dark brown</td>
<td>30-left 30-Right</td>
</tr>
<tr>
<td>Cattail fiber</td>
<td>268.7</td>
<td>Fibers turn to dark brown</td>
<td>31-Left 31-Right</td>
</tr>
<tr>
<td>Cotton fiber</td>
<td>248.3</td>
<td>Fibers turn to dark brown</td>
<td>32-Left 32-Right</td>
</tr>
</tbody>
</table>

Figure 30: Cattail plant, before (left) & after heating
Figure 31: Cattail fibre–before (L) & after heating (R)

Figure 32: Cotton fiber – before (L) and after heating (R)

4.0. Conclusion
In the current study, it was found that only alkali treatment produced fiber from the cattail plants. It was further concluded that treating cattail plant with KOH at 80°C for 6 hours will produce better fiber considering visual assessment (Figure 15). Cattail fiber diameter, moisture regain, burning behavior and thermal properties are similar to commonly used textile fiber such as cotton, wool and polyester. The crimp adjusted fiber length is similar to plant cut length. However, cotton-crimp adjustment value (1:1.20) was used and the result should be taken with caution.

Submicroscopic structure revealed that the fiber cell has a lumen and cells are separated by canal. This feature as well as ‘crenelated’ structure might have significant implications in industrial and biomedical applications for trapping antibiotics and other chemicals.
Acknowledgements
This study supported by Natural Sciences and Engineering Research Council of Canada (NSERC) and grateful to Dr. Mashiur Rahman and Dr. Nazim Cicek for their intellectual guidance and valuable advice. The authors gratefully acknowledge to Nicholson and Joe Ackerman for tremendous support by providing cattail plant.

References


