

Article

Fenton-Based Treatment of Flax Biomass for Modification of Its Fiber Structure and Physicochemical Properties

Nasrin Aliasgharlou ¹, Duncan E. Cree ^{2,*}  and Lee D. Wilson ^{1,*} 

¹ Department of Chemistry, University of Saskatchewan, 110 Science Place, Thorvaldson Building, Saskatoon, SK S7N 5C9, Canada

² Department of Mechanical Engineering, Mc Master University, 1280 Main Street West, Hamilton, ON L8S 4L7, Canada

* Correspondence: creed1@mcmaster.ca (D.E.C.); lee.wilson@usask.ca (L.D.W.)

Abstract: The availability of a sustainable technique for degumming lignocellulose fibers is a challenge for the fiber processing industry. Removal of non-cellulosic content from lignocellulose fibers is essential for improving their mechanical and chemical properties, which makes the fibers more suitable for various applications. Herein, a catalytic Fenton-based oxidation process was employed to isolate microcellulose fibers from raw flax fibers. Various complementary methods such as FT-IR/NMR spectroscopy and TGA were used to obtain insight into the thermal behavior of the treated fibers. The morphology of the fibers was studied using Scanning Electron Microscopy (SEM), whereas the surface chemical properties of the fibers was evaluated by a dye-based adsorption method, along with a potentiometric point-of-zero-charge method. To obtain fibers with suitable properties, such as uniform fiber diameter, several Fenton reaction parameters were optimized: pH (7), reaction time (15 h), iron sulfate (2 wt.%), and hydrogen peroxide (10 wt.%). The results indicate that, under the specified conditions, the average diameter of the raw fibers ($12.3 \pm 0.5 \mu\text{m}$) was reduced by 58%, resulting in an average diameter of $5.2 \pm 0.3 \mu\text{m}$ for the treated fibers. We demonstrate that the treated fibers had a lower dye adsorption capacity for methylene blue, consistent with the smoother surface features of the treated fibers over the raw flax fibers. Overall, this study contributes to utilization of the Fenton reaction an efficient oxidation technique for the production of lignocellulose fibers with improved physicochemical properties, such as reduced fiber diameter distribution, in contrast with traditional alkali-based chemical treatment.

Keywords: lignocellulosic fiber; degumming; Fenton oxidation; flax fiber; microcellulose fibers



Citation: Aliasgharlou, N.; Cree, D.E.; Wilson, L.D. Fenton-Based Treatment of Flax Biomass for Modification of Its Fiber Structure and Physicochemical Properties. *Appl. Sci.* **2024**, *14*, 6133. <https://doi.org/10.3390/app14146133>

Academic Editor: Cataldo De Blasio

Received: 19 June 2024

Revised: 10 July 2024

Accepted: 12 July 2024

Published: 15 July 2024



Copyright: © 2024 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

1. Introduction

Growing concern over the negative impact of the buildup of microplastics in the environment is related to the disposal of single-use plastics (e.g., air filters derived from synthetic polymer fibers). More recently, the development of sustainable air filters has been based on biomaterial platforms including hemicellulose, chitin, and chitosan, which represent a more sustainable approach due to the lower environmental impact upon disposal [1,2]. Cellulose is a natural biopolymer that is derived from lignocellulosic biomass sources such as flax, hemp, wood, and cotton [3].

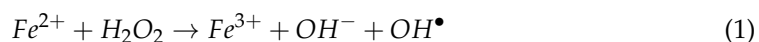
Flax (*Linum usitatissimum* L.) fiber belongs to the bast fiber family, which is sourced from the inner bark of the flax plant stem. These fibers are known for their high strength and rigidity, and, unlike synthetic fibers, they show little stretching under load [4]. Flax fibers (FFs) exhibit a composite-like hierarchical structure, deriving their properties from micro- and nano-level configurations, making them excellent candidates for creating biodegradable composites. The anatomy of a flax stem includes layers like bark, phloem, xylem, and a central void, with fiber bundles located along the outer surface. FFs can range in length from 80 to 150 cm, where the primary components are cellulose, hemicellulose, and lignin, along with the presence of residual oils [5,6].

Studies have shown that air filters made from biodegradable fibers have comparable filtration efficiency to traditional synthetic filters but may require some pretreatment to achieve the desired fiber properties, such as durability and filtration effectiveness [7]. In this regard, a wide range of biological, physical, and chemical oxidation processes have been studied [8–10]. Comparison of these methods indicates that chemical oxidation processes are potentially more efficient and cost-effective. One drawback accompanying chemical treatment with alkali agents such as sodium hydroxide (NaOH) relates to the generation of large quantities of wastewater that represent hazardous caustic waste to the environment and human health [8,11].

Several chemical treatments have been applied to natural fibers. For example, the use of NaOH involves immersion of the fibers in an alkali solution to remove fiber components such as lignin or impurities to yield cellulose-enriched products with greater surface area [12]. Alkali treatment can improve the wettability and adhesion of fibers, as well as enhance their mechanical properties [13]. However, excessive alkali treatment of fibers can lead to degradation and reduced tensile strength. By comparison, the acid treatment process involves exposure of fibers to acid media, such as aqueous sulfuric or hydrochloric acids. In turn, acid treatment can improve fiber flexibility and fiber strength, and alter the surface area and fiber porosity [14]. However, chemical treatment with acids can also damage fibers and release toxic byproducts into the environment. Additional types of chemical treatments include bleaching and dyeing [15–18]. Bleaching can improve fiber whiteness and cleanliness, while dyeing can add color and aesthetic appeal.

The choice of chemical treatment for the processing of natural fiber filter media should be based on a balance between the desired properties and sustainable processing to minimize potential negative environmental and health impacts. Optimization of treatment conditions (reagent concentration, temperature, and processing time) can reduce adverse effects while maximizing desired outcomes to obtain natural fibers with desired properties for tailored applications. In this regard, researchers have directed research toward the development of sustainable fiber treatment methodologies to address the limitations of conventional chemical processes [8].

The Fenton process is an advanced oxidation reaction (AOR) that has attracted attention due to its efficiency, cost-effectiveness, and potential for green chemical processing. Fenton reaction conditions do not involve high dosages of chemicals, extreme temperature, and pressure. According to Equation (1), ferrous ions catalyze the decomposition of hydrogen peroxide (H_2O_2) in aqueous media to produce hydroxyl radicals for the degradation of organic materials [19].



It follows that the Fenton process approaches outstanding performance in decomposition of the lignin content of natural fibers [20], which can generate microfibril flax fibers with reduced diameters that possess desirable properties for filter-based applications. Reducing fiber diameter is critical for enhancing performance in textile applications, since it leads to finer, smoother fabrics with improved softness and texture [21]. For the case of filter media, fibers with reduced diameter have a greater surface area, which yields improved entrapment of particles and contaminants [22]. For the case of polymer composites, the increased surface area of the fibers improves the fiber–matrix interaction, which leads to better stress transfer and mechanical performance of the composite [23]. Whereas the conventional Fenton process offers an effective chemical treatment method for natural fibers, there are sparse studies on the effect of Fenton treatment on the diameter of FFs for variable conditions [24,25].

This research aims to develop a sustainable treatment process for natural fibers that is based on the Fenton AOR process to isolate micron-sized flax fibers to improve their physiochemical properties (e.g., fiber size and chemical functionality). Structural modification of fibers by the Fenton AOR process is proposed as an alternative modification method to improve separation efficiency of the filtration products based on alteration of surface

chemistry and fiber diameter. In contrast to the conventional Fenton oxidation process, the modified Fenton treatment AOR is conducted on the surface of solid fibers under heterogeneous conditions. Consequently, the Fenton AOR process at solid surfaces may result in a reduced rate of chemical consumption and sludge generation, thereby improving the sustainability of the process [24]. To demonstrate the effectiveness of the modified Fenton AOR, treated flax fibers were characterized by several complementary methods: thermogravimetric analysis (TGA), spectroscopy (FT-IR and NMR), and microscopy (SEM). The results for the treated flax fibers were compared with a conventional alkali treatment process, where SEM was used to examine the surface of the raw and treated flax fibers. In turn, the Fenton AOR process reported herein is anticipated to contribute to a more sustainable route for the treatment of flax biomass to yield fiber products with improved properties for filter-based applications.

2. Results and Discussion

It can be inferred that the Fenton AOR reaction efficiency is dependent on the amount of iron catalyst, hydrogen peroxide, pH, and reaction time. Therefore, the impact of each parameter on the efficiency of the Fenton AOR process on the diameter of the FFs after the treatment process will be discussed. The introduced modified Fenton reaction was conducted in two stages: (i) pre-adsorption of Fe (II) onto the FF surface, and (ii) subsequent addition of hydrogen peroxide. Thus, it is important to determine the amount of iron adsorbed onto the fiber surface to evaluate the role of the reaction conditions for the Fenton AOR process on the structural modification of FFs.

2.1. Adsorption of Iron Species on the Flax Fiber Surface

To gain insight into the role of iron species in the Fenton process, XPS profiles were obtained to investigate the impact of various iron sulfate levels on the amount of iron adsorbed by the FFs (Figure 1). In this step, the fibers were soaked in FeSO_4 (aq) solutions (0.1, 1, 2.5, and 5 wt.%) for 1 h [24]. The results revealed that, by increasing the concentration of iron from 0.1 to 2.5 wt.%, there was an increase in the amount of iron adsorbed on the fiber surface [24]. In Figure 1, the XPS results indicate that there was no significant presence of iron on the surface of the flax fiber at either 0.1% or 1% FeSO_4 (aq). Therefore, the concentration of iron sulfate is considered as one of the key factors in the modified Fenton reaction for the treatment of flax fibers. Based on the findings of the XPS experiment, it was observed that there was no increase in the amount of iron adsorbed on the surface of the fiber as the iron sulfate concentration increased from 2.5 wt.% to 5 wt.% (Table 1).

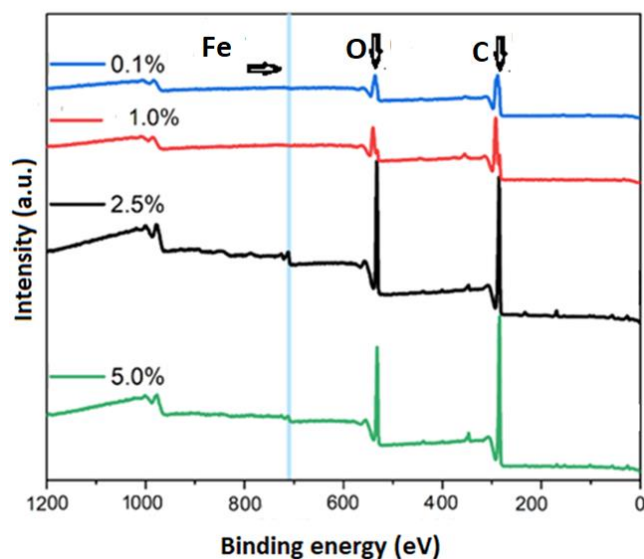


Figure 1. Wide-scan XPS spectra of flax fibers after exposure to an aqueous solution of iron sulfate at variable concentrations (wt.%).

Table 1. Atomic content weight (%) of iron adsorbed according to a binding energy of 710 eV on the surface of the flax fibers, as obtained by XPS analysis.

Iron Concentration (wt.%)	Iron Content (wt.%)
0.1%	Not detected
1.0%	Not detected
2.5%	1.64%
5.0%	4.50%

2.2. Investigating the Effect of Operational Parameters on Modified Fenton Reaction Efficiency

As noted in Section 1, a key goal of the oxidation process was to determine the optimal ranges of parameters for Fenton oxidation of FFs. The efficiency of the treatment process was demonstrated based on fiber diameter reduction. As shown in Figure 2, the average diameter of raw fibers (fitted with the Gaussian model) was estimated as $12.3 \pm 0.5 \mu\text{m}$.

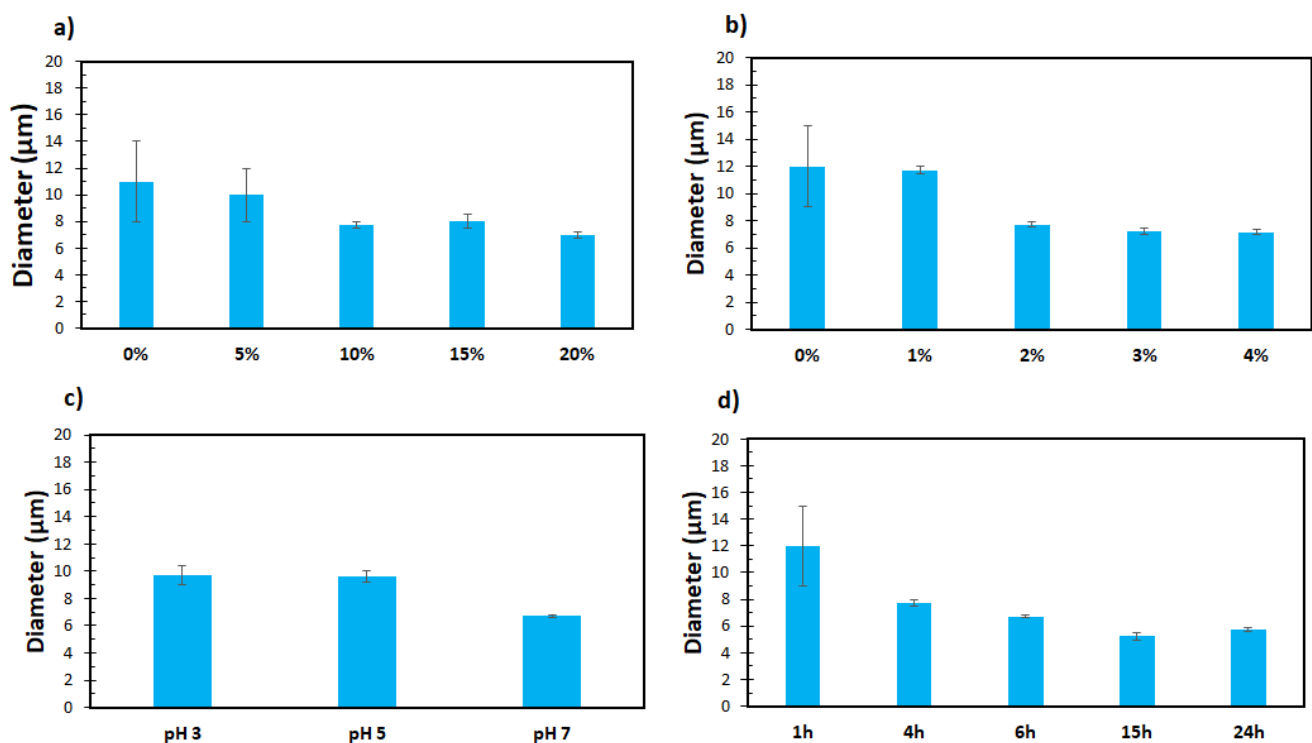


Figure 2. Effect of the reaction parameters on the average flax fiber diameter upon treatment with the modified Fenton reaction: (a) effect of hydrogen peroxide on the fiber diameter, (b) effect of iron sulfate on the fiber diameter, (c) effect of pH on the fiber diameter, and (d) effect of reaction time on the fiber diameter.

In the Fenton reaction, hydrogen peroxide and iron species can play significant roles that affect chemical consumption costs and reaction efficiency. To amplify reaction efficiency, it is important to find the optimal ranges of external parameters such as hydrogen peroxide, Fe (II), pH, and time, according to the reduction of fiber diameter (cf. Figure 2).

Figure 2 reveals the effect of the evaluated parameters on fiber diameter reduction. As shown in Figure 2a, for 10 wt.% H_2O_2 , the average diameter was $10.7 \pm 0.67 \mu\text{m}$. Compared to raw FFs, the reduction in average fiber diameter can be related to the washing step, which may remove some impurities and non-cellulosic components from the fiber surface. The results reveal that incremental concentrations of hydrogen peroxide resulted in a greater reduction of fiber diameter. At a 10 wt.% H_2O_2 concentration, a slight reduction in the average diameter was observed, while the maximum H_2O_2 concentration that yielded the greatest reduction in flax fiber diameter was found to be 15 wt.%. According to the results,

it can be concluded that there was no significant change in the average fiber diameter induced by increasing the H_2O_2 concentration from 10 to 15 wt.%.

In Figure 2b, the role of iron concentration on the diameter of FFs in the Fenton process is evidenced by augmentation of the iron dosage onto the surface of FFs, where the efficiency of the reaction increased. On the other hand, with a further increment of iron content of 2 wt.%, there was negligible decrease in the average diameter of the treated fibers. According to the operational costs and the efficiency of the process, the desired iron salt concentration was 2 wt.%, in agreement with the XPS results in Figure 1 [26].

The pH of the reaction medium is also an important operational parameter in the Fenton oxidation process, since it controls the generation of hydroxyl radicals, as noted in Figure 2c [27]. The results reveal that maximum Fenton reaction efficiency occurred at neutral pH, whereas reduced Fenton efficiency occurred in acidic media. This trend relates to the competition of H^+ and Fe^{2+} ions for adsorption sites onto the surface of FFs.

In addition to the above-mentioned parameters, reaction time is another significant parameter that influences the efficiency of the Fenton reaction. It is obvious that, by increasing the time, greater efficiency of the Fenton oxidation process occurred. To determine the desirable reaction time for modified Fenton treatment of flax fibers, the reaction was monitored for various periods up to 24 h. Thereafter, there was a minor change in the average diameter of the fibers. In Figure 2d, incremental time led to flax fibers with a more reduced average diameter, where the average diameter of the fibers after 15 h reached $5.2 \pm 0.3 \mu\text{m}$. It was shown that there was negligible improvement in the process efficiency by increasing the reaction time from 15 h to 24 h, according to the fiber diameter size profiles.

Based on the obtained results in Figure 2, it is evident that the optimum condition was determined as 10 wt.% H_2O_2 and 2 wt.% FeSO_4 for a 15 h reaction under neutral pH conditions. For the most favorable conditions, there was a significant reduction in fiber diameter distribution, including the average diameter of the FFs. Cellulose has a lower binding affinity to iron, as compared with other biomass fractions such as lignin, hemicellulose, and pectin that contain $-\text{COOH}$ groups. Lignins and pectins can facilitate favorable binding with metal ions such as $\text{Fe}(\text{II})$, especially in the ionized state [28,29]. As well, these components are more susceptible to Fenton oxidation, which results in the ability to produce micron-sized cellulose fibers [30].

Figure 3 represents the obtained diameter of the raw flax fiber (a; no treatment) and modified flax fiber (b; after Fenton AOR treatment). It was found that the modified Fenton AOR process removed non-cellulosic components such as lignin that comprise the fiber structure, resulting in the isolation of thinner fibers, as compared to alkali treatment (cf. Section 2.3). Also, Figure 4 shows that there was no significant change in the diameter of the samples upon treatment with alkali media (step 2, an additional treatment step) after the Fenton AOR process (step 1). Degumming of FFs with a modified Fenton oxidation AOR process affords isolation of cellulosic fibers, since oxidation treatment can effectively remove lignin, which serves as a binding agent for cellulosic composites in such lignocellulose materials [31].

Table 2. Experimental conditions and sample ID codes.

Sample	ID Code	Experimental Conditions
Raw flax fibers	Raw	
Treated flax fiber with Fenton AOR	F-T15 (Fenton AOR)	Flax fiber with Fenton AOR at described conditions ¹
Treated flax fiber with Fenton AOR + alkali treatment	F-T15-Alk (Fenton AOR + Alkali)	Flax fiber with Fenton AOR at described conditions ¹ , followed by NaOH 5% for 1 h
Treated flax fiber with alkali treatment	Alk (no Fenton AOR)	NaOH 5% for 1 h

¹ pH = 7, reaction time = 15 h, [iron sulfate] = 2% wt.%, [hydrogen peroxide] = 10% wt.%.

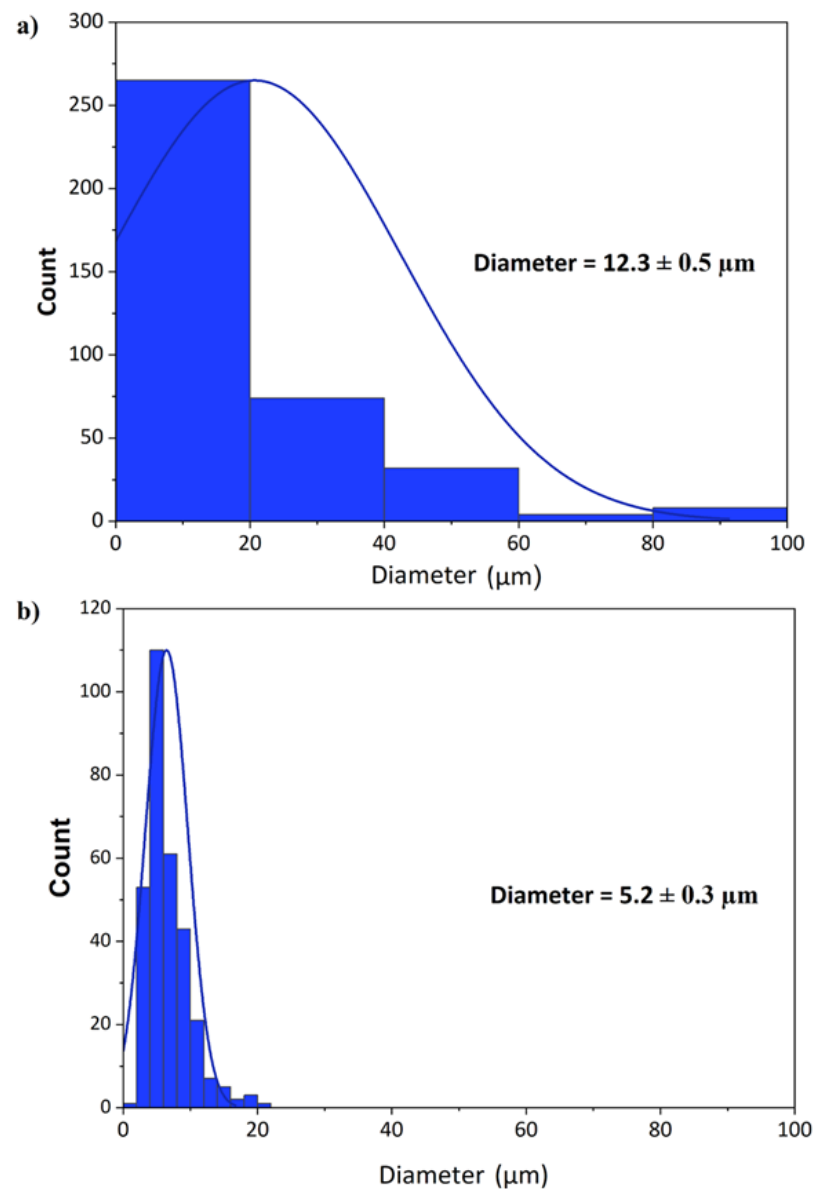


Figure 3. Optical microscopy analysis of flax fiber samples. (a) Raw flax fiber with its respective diameter distribution, and (b) treated flax fiber with its respective fiber diameter after treatment by the Fenton AOR process at optimal conditions.

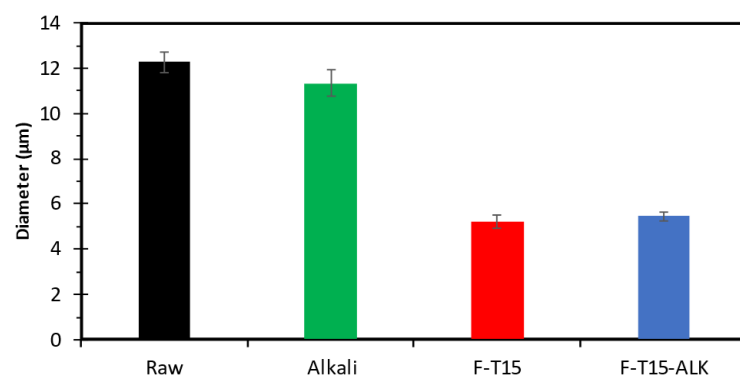


Figure 4. Impact of various degumming techniques on the surface of flax fibers, with their respective standard deviations. The reader is referred to Table 2 for definitions of the sample ID codes used in this figure.

2.3. FT-IR Spectroscopy

IR spectroscopy was utilized to characterize the functional groups of the lignocellulose fibers (cf. Figure 5). The broad peaks at 3387 cm^{-1} were ascribed to the vibrational bands of the hydroxyl functional groups. According to the literature, the IR bands at 1624 and 1734 cm^{-1} are associated with stretching of the C=C bonds of lignin and carbonyl vibrations of the acetyl/COOH groups of hemicellulose and pectin, respectively [32]. Also, asymmetric deformation of CH_3 and the C-H vibrations of lignin were observed at 1356 and 1430 cm^{-1} , respectively. A significant reduction in the intensity of the related vibrational bands near 1624 and 1734 cm^{-1} was evident, which suggests decomposition of lignin and hemicellulose due to oxidation of FFs by the modified Fenton (F-T15) and alkali-modified Fenton (F-T15-ALK) processes. The intensity of the band at 1734 cm^{-1} was lower compared to the intensity of the band near 1624 cm^{-1} in the modified Fenton process. This can be related to oxidation treatments that are less efficient for the degradation of hemicellulose [33]. On the other hand, alkali-treated samples showed a significant reduction in the intensity of the band at 1734 cm^{-1} because of the alkali treatment efficiency for the decomposition of hemicellulose content versus lignins.

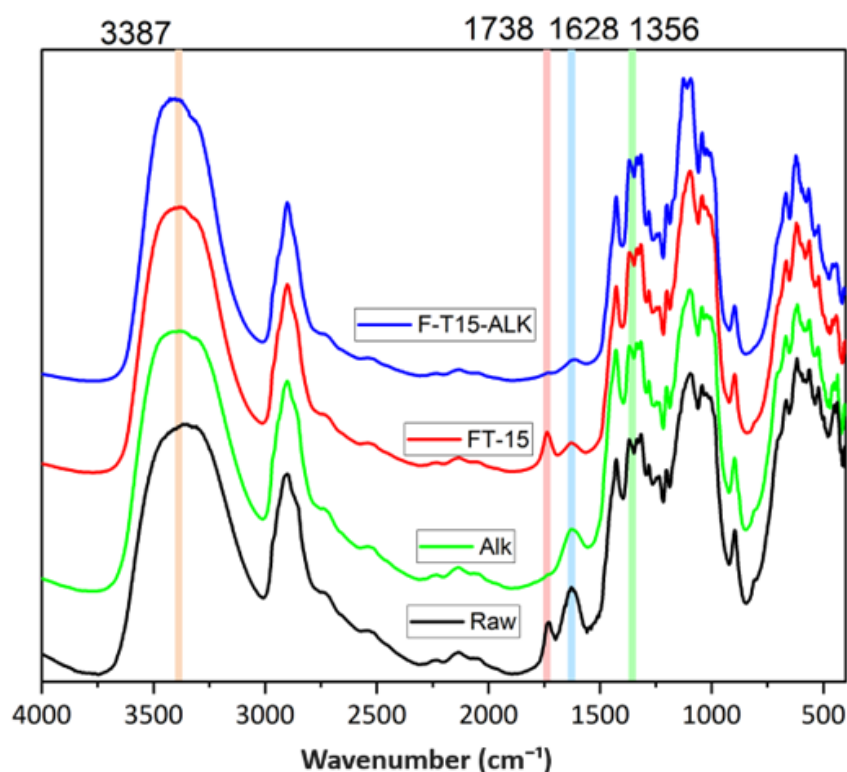


Figure 5. IR spectra of raw and treated fibers. The reader is referred to Table 2 for definitions of the sample ID codes used in this figure.

2.4. Thermal Gravimetry Analysis

Thermal gravimetry analysis (TGA) and differential thermal gravimetry (DTG) were employed to assess the thermal properties of pristine (untreated) and treated flax fiber samples. Figure 6 shows the weight loss and maximum decomposition temperature of raw and treated samples. The first phase close to $100\text{ }^{\circ}\text{C}$ is associated with the drying phase of flax fibers and water removal from the surface of flax fibers. The next weight loss event was ascribed to decomposition of the hemicellulose and cellulose fractions near $200\text{--}350\text{ }^{\circ}\text{C}$ [34]. In the last step, steady degradation of lignins was observed from $250\text{ }^{\circ}\text{C}$ [35]. Compared to the pristine sample, the treated FFs were shifted slightly to lower temperatures in the modified Fenton (FT-15), and alkali-modified Fenton treated samples (F-T15-ALK) due to the partial oxidation (and/or removal of lignins and pectins) from the flax fiber surface. In

turn, such modification led to enhanced electrostatic repulsion and a reduction in linkage between the components. Moreover, it should be noted that thermal decomposition of lignin occurs at a higher temperature (ca. 350 °C) than the cellulosic biopolymer fraction, near 300 °C, which was reported by Mohamed et al. [28]. A comparison of the DTG profile of the Fenton AOR oxidized fiber samples versus the pristine and alkali-treated fibers revealed a lower lignin content, in accordance with expectations for such oxidation processes [36].

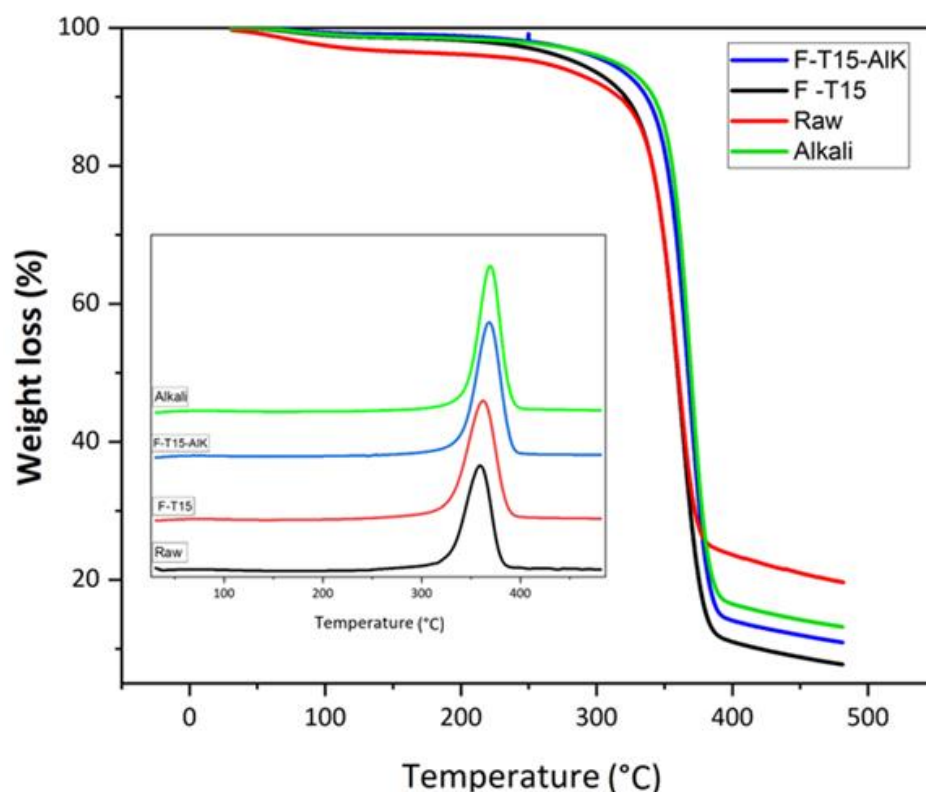


Figure 6. TGA and DTG (see inset) profiles for the thermal degradation of raw and treated fibers. The reader is referred to Table 2 for a description of the sample ID codes used in this figure.

2.5. NMR Spectroscopy

In order to acquire additional information about the Fenton AOR process on the flax fiber surface, ^{13}C NMR spectroscopy in the solid state was employed (Figure 7). The signals at 62.5 and 62.9 ppm are associated with C6 of cellulose. There were peaks at 71.6 associated with C5, and the shifting around 74.0 ppm related to C3 and C2. The peaks at 83.0 and 84.0 ppm are the ^{13}C signatures for C4 [20,24]. In the structure of the FFs, there was a sharp line at 104.5 ppm, attributed to C1. Overall, the NMR spectra suggest that all three samples contained both crystalline and non-crystalline cellulose. The variation in peak intensities among the samples may be attributed to relative variations in the nature of the supramolecular fibril structures. The presence of carboxyl group signatures near 171 ppm in the raw sample suggests that the hemicellulose in flax fibers may have been partially degraded or oxidized by the Fenton and alkali-Fenton treatments [37,38], according to the reduced intensity of the ^{13}C signature for $-\text{COOH}$ in FT-15 and FT-15-Alk, as shown in Figure 7. The weak spectral intensity in the upfield (10–40 ppm) and downfield (120–160 ppm) region for the treated composites supports a negligible non-cellulosic content (e.g., lignins, pectins) in the modified fiber materials, in agreement with the IR results (cf. Figure 5).

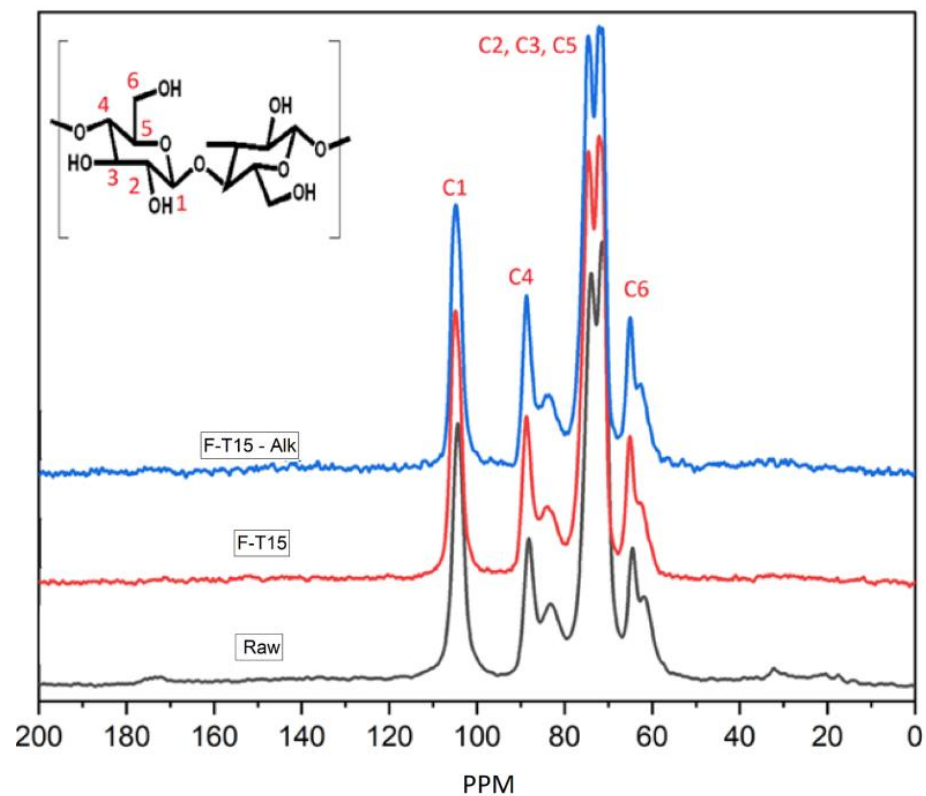


Figure 7. Solid-state ^{13}C spectra of fiber samples: raw flax fiber before treatment, modified-Fenton-treated sample (F-T15), and modified Fenton and alkali-treated sample (F-T15-ALK).

2.6. Fiber Surface Charge Estimation via Determination of PZC

Point-of-zero-charge (PZC) analysis reflects the surface charge of the raw and treated samples and the differences between them. The results in Figure 8 revealed that the samples had a low PZC value (~ 5.0 – 5.5). The PZC values for the raw (5.5), modified Fenton (5.0), and alkali-modified Fenton (5.5) were estimated. Flax fiber generally carries a negative surface charge in water because it contains ionizable acidic substances like lignins. As expected, oxidation of flax fibers resulted in a reduction of fiber surface charge, according to the shift in PZC values [39,40]. This phenomenon can be related to enhancement of the fiber's surface area and increased exposure of oxygen-rich groups, which leads to a more negative surface charge versus the untreated flax fibers that possess a more intact fibril structure. However, the slight positive shift in the surface charge in the F-T15-ALK sample can be attributed to the presence of adsorbed sodium ions on the surface of the fibers.

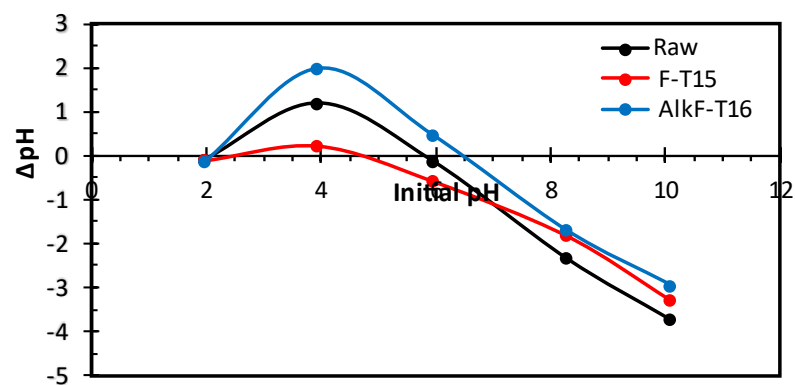


Figure 8. PZC of raw and treated flax fiber samples, via the ΔpH of the starting and final solutions after 48 h. The reader is referred to Table 2 for definitions of the sample ID codes used in this figure.

2.7. Dye Adsorption

Methylene blue (MB) dye adsorption was carried out in aqueous media to gain insight on the role of the textural properties (surface area and porosity) of the treated and untreated flax fibers and the relative MB uptake for the various fiber systems. As shown in Figure 9, analysis of dye uptake by the raw FFs revealed enhanced MB dye adsorption, as compared with the treated samples. MB adsorption by the F-T15-ALK and FT-15 samples was reduced by 75% and 50%, respectively. The greater MB uptake by raw samples can be related to the higher abundance of lignin, pectins, hemicellulose, and ionizable groups ($-\text{COOH}$, $-\text{OH}$, etc.) that favor electrostatic adsorption of the MB cationic dye [41,42]. In the case of treated FFs, removal of non-cellulosic fiber components such as lignin, pectin, and hemicellulose occurred, which resulted in a reduced level of ionizable groups that have lower affinity to MB dye due to enrichment of cellulose content at the expense of the other components with ionizable groups. The results concur with the removal of non-cellulose biomass fractions by the treatment techniques, resulting in fibers with reduced polar and/or ionizable functional groups (cf. Figures 5 and 7). Parallel conclusions were drawn by Mir & Wilson [40] for flax fiber composites that contained incremental levels of chitosan. The decreased uptake of MB was related to greater formation of complexes between the lignin components of FFs as the chitosan loading increased.

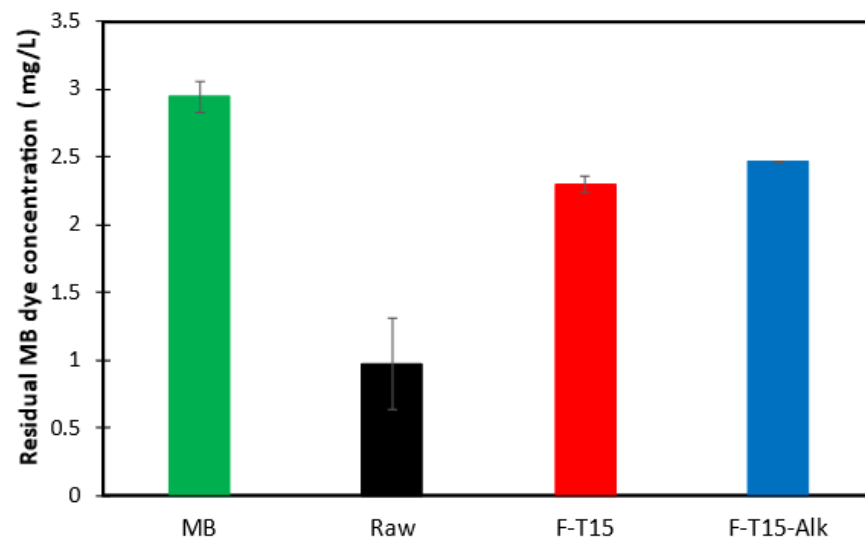


Figure 9. Residual MB dye in aqueous solution after exposure of a fixed concentration of MB dye with raw and treated fibers for 48 h, along with their respective standard deviations. The reader is referred to Table 2 for definitions of the sample ID codes used in this figure.

2.8. Scanning Electron Microscopy

Figure 10 displays SEM images of both untreated and treated FFs. The untreated fibers had uneven surfaces with larger overall fiber diameters, while the alkaline-treated fibers revealed a smoother fiber surface. This trend indicates that alkaline treatment contributes to the removal of non-cellulosic components (e.g., hemicellulose, lignins, and pectins) and impurities such as wax, which contribute to smoother surface features [43]. Fenton treatment resulted in improved surface characteristics of the fibers by reducing the average diameter of FFs and the non-cellulose biomass fractions. The F-T15 and F-T15-ALK samples showed the most notable reduction in fiber diameter, with a smoother surface appearance. These results concur with those obtained from optical microscopy, NMR, and FT-IR analysis, revealing that Fenton treatment effectively removed the non-cellulosic content of the fibers and defibrillated the fiber bundles into fibers with reduced diameter.

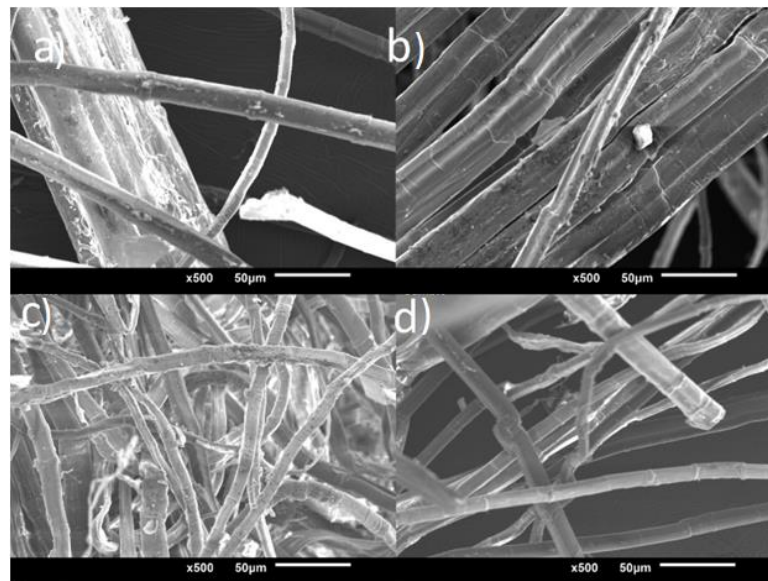


Figure 10. SEM micrographs of (a) Raw, (b) Alk, (c) F-T15, (d) F-T15-ALK. The reader is referred to Table 2 for definitions of the sample ID codes.

3. Materials and Methods

3.1. Materials

Flax fibers (short and retted fibers) were obtained from Biolin Research Inc. (Saskatoon, SK, Canada). All chemicals except hydrogen peroxide (laboratory grade) were of ACS-grade quality. Sodium hydroxide (99%) and hydrogen peroxide (30%) were purchased from Fisher Scientific (New York, NY, USA). Ferrous (iron) sulfate (99%) and potassium bromide (99.5%) were provided by Sigma Aldrich (Oakville, ON, USA). Deuterium oxide (D_2O ; 99.9%) was supplied by Cambridge Isotope Laboratories (St. Louis, MO, USA). All aqueous solutions were prepared using Millipore water (18.2 M Ω).

3.2. Flax Fiber Treatment Process

3.2.1. Washing Step

Before all experimental procedures, the flax fibers were washed with Millipore water at 100 °C for 1 h to remove impurities from the surface of the FFs. Then, the FFs were dried in an oven at 50 °C for 24 h.

3.2.2. Modified Fenton AOR Process

To address excess waste chemical consumption, wastewater, and sludge generation, it is important to reduce the volume of the reaction media. This can be done through the adsorption of iron sulfate on the fiber surface to prevent excess consumption of reagents. Therefore, the flax fibers were first pretreated via adsorption of iron sulfate media, where the resulting pH of the acidic aqueous media was ~4. Then, the excess Fe^{2+} (aq) was filtered, followed by initiation of the reaction onto the surface of the FFs by the introduction of a pre-determined hydrogen peroxide solution [44]. First, 250 mg of washed flax fibers were soaked in an iron sulfate solution for 1 h [44]. The mixture was placed in an SK-O330-pro shaker with a rotation speed of 200 rpm to allow Fe^{2+} to diffuse and adsorb onto the fibers. Then, the excess iron sulfate solution was filtered, and a fixed dosage of hydrogen peroxide solution was introduced to the filtered FFs. The reaction took place for fixed reaction times and at fixed pH values (adjusted by NaOH (aq) and HCl (aq)). The treated samples were washed and neutralized with Millipore water, followed by oven drying at 50 °C (Figure 11). Also, flax fibers were treated in the absence of iron sulfate to provide a reference (control) condition.

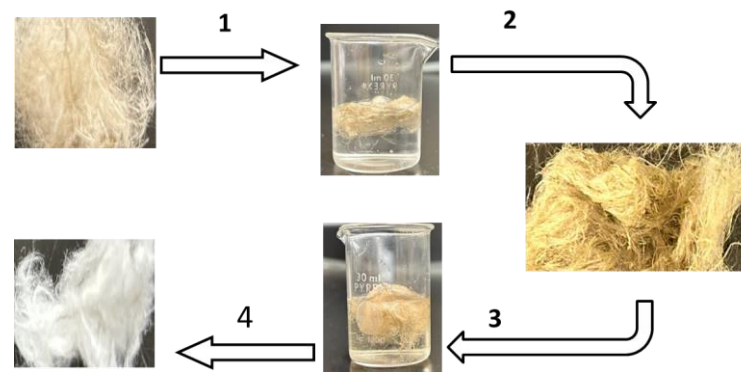


Figure 11. Schematic representation of the pretreatment process of flax fiber using the Fenton (AOR) process. *Step 1:* Fibers were treated with 0.5 wt.% FeSO_4 (aq); *Step 2:* the excess iron solution was filtered; *Step 3:* the reaction was initiated on the surface of the flax fiber with the introduction of H_2O_2 (aq); and *Step 4:* the treated fibers were washed with Millipore water.

3.2.3. Alkali Treatment

In the next step, the Fenton-treated flax fibers at optimum conditions were soaked in NaOH (5 wt.% aqueous media) for 1 h at 70 °C. Afterward, the treated samples were washed and neutralized with Millipore water and dried in an oven at 50 °C for 24 h. Also, an additional sample of raw FFs was treated and soaked in alkali medium with the same conditions, as described in a previous report [45].

The samples were powdered ($\leq 1000 \mu\text{m}$ in length) with liquid nitrogen by grinding in a metal mortar and pestle before characterization unless specified otherwise. The sample ID codes are outlined in Table 2 below.

3.3. Characterization Methods

3.3.1. X-ray Photoelectron Spectroscopy (XPS)

X-ray photoelectron spectroscopy (XPS) measurements were conducted using a Kratos AXIS Supra system from Manchester, UK. The system was equipped with a 500 mm Rowland circle monochromatic Al K- α (1486.6 eV) source, along with a hemispherical analyzer (HSA) and a spherical mirror analyzer (SMA). Data were collected using a spot size hybrid slot ($300 \mu\text{m} \times 700 \mu\text{m}$), and all survey spectra were obtained in the -5 – 1200 eV binding energy range with 1 eV steps and a pass energy of 160 eV. The analysis utilized an accelerating voltage of 15 keV and an emission current of 15 mA.

3.3.2. Fourier Transform Infrared (FT-IR) Spectroscopy

An FTS-40 spectrometer (Bio-Rad, Mississauga, ON, Canada) was utilized to obtain diffuse reflectance FT-IR spectra. The powdered samples were mixed with KBr with a 1:10 weight ratio. FT-IR spectra for samples employed reflectance mode at 295 K to acquire spectra with a resolution of 4 cm^{-1} over a fixed region of 400 – 4000 cm^{-1} . Spectral normalization employed the IR band at 1097 cm^{-1} to obtain a unitary intensity.

3.3.3. Thermal Gravimetry Analysis (TGA)

The thermal properties of raw and treated flax fibers were investigated by employing a TGA Q50 thermal analyzer (TA Instruments, New Castle, DE, USA). In aluminum pans, the powdered pristine and treated fibers ($20 \pm 0.2 \text{ mg}$) were analyzed under a nitrogen atmosphere from 30 to 500 °C with a heating rate of $10 \text{ }^\circ\text{C}/\text{min}$. TA Instruments Q50 TGA software was employed to integrate the TGA band profiles.

3.3.4. Optical Microscopy

Optical microscopy with $\times 10$ magnification was employed for obtaining micrographs of the fibers, where the fiber diameter distribution and the average diameter were deter-

mined by the ImageJ and the Origin 2021b software system. Each fiber was measured at three locations along its length and averaged.

3.3.5. ^{13}C NMR Spectroscopy

^{13}C solid-state NMR spectra were obtained with a 4 mm DOTY CP-MAS probe and a Bruker AVANCE III HD (BRUKER, Billerica, MA, USA) spectrometer operating at 125.77 MHz (^1H frequency at 500.13 MHz) for the ^{13}C nuclei. The ^{13}C CP/TOSS (Cross Polarization with Total Suppression of Spinning Sidebands) spectra were obtained at a spinning speed of 7.5 kHz, with a ^1H 90° pulse of 5 μs and a contact time of 2.0 ms with a ramp pulse on the ^1H channel. For all ^{13}C NMR spectra of powdered samples, ca. 2500 scans were accumulated with a recycle delay of 1 s, along with a 50 kHz SPINAL-64 decoupling sequence during acquisition. ^{13}C NMR chemical shifts were referenced to adamantane at 38.48 ppm (low field signal). To prepare samples for dissolution, the ^{13}C NMR treated samples in a solution state were dissolved in aqueous media containing a mixture of NaOH (7 wt.%), urea (12 wt.%), and several drops of D_2O to provide magnetic field lock during spectral acquisition.

3.3.6. Point-of-Zero-Charge (PZC)

Point-of-zero-charge (PZC) was determined using the pH shift method, where an approximate amount of 60 mg of sample was added to 25 mL of a 0.005 M CaCl_2 (aq) solution. After 48 h of equilibration at 22 $^\circ\text{C}$, the pH of each system was measured in aqueous media, where the pH_{PZC} was determined at the interaction's final pH and $\Delta\text{pH} = 0$. The pH was adjusted using NaOH (aq) and HCl (aq).

3.3.7. Dye Adsorption

The dye adsorption of flax fibers was performed using a ca. 20 mg sample of FFs in 20 mL of methylene blue (MB (aq), 50 mg/L). After 48 h of equilibration at 22 $^\circ\text{C}$, the amount of residual MB dye (aq) in the solution phase was measured using a Thermofisher Scientific SPECTRONICTM 200 UV-vis spectrophotometer (San Diego, CA, USA).

3.3.8. Scanning Electron Microscopy (SEM)

SEM images of untreated and treated flax fibers were obtained utilizing a JEOL JSM-6010 LV (Tokyo, Japan) with an operating voltage of 10 kV. Before imaging, the samples were coated with a thin layer of gold to prevent charging.

4. Conclusions

This study employed the Fenton oxidation reaction under heterogeneous conditions for the treatment of flax fibers (FFs). The results showed that treatment under the desired concentrations (10 wt.% H_2O_2 and 2 wt.% FeSO_4) in water at neutral conditions (pH $\sim$ 7) for 15 h resulted in fibers with a reduced size distribution with a lower average fiber diameter, as compared with untreated fibers. The results reveal that the Fenton AOR process under heterogeneous conditions offers a sustainable approach for the pretreatment of biomass FFs to yield biomass with enriched cellulose content. As well, modified FFs showed a 60% reduction in average fiber diameter from $12.3 \pm 0.5 \mu\text{m}$ to $5.2 \pm 0.3 \mu\text{m}$. Thus, the Fenton AOR process can selectively remove the non-cellulosic content (e.g., hemicellulose, lignin, etc.) of FFs in an effective manner. This trend was attributed to the higher affinity of the biomass fractions toward ferrous species, as compared with pure cellulose. The study found that the use of traditional techniques, such as alkali treatment, was less effective for the removal of non-cellulosic content versus the Fenton AOR method. The combined use of alkali treatment with the Fenton AOR process did not significantly decrease the average diameter of FFs, as compared to the Fenton AOR process without alkali. Based on these results, the Fenton AOR process is an effective technique for producing cellulosic microfibrils in FFs, as compared with harsh alkali treatment methods. The effective removal of the non-cellulose components for the treated fibers showed a measurable reduction in

MB dye adsorption versus the pristine flax fibers, which are known to contain a higher lignocellulose content. This is attributed to the role of structural alteration of FFs and/or chemical removal of non-cellulose components with the Fenton AOR method. Despite its known effectiveness in homogenous media, chemical treatment of natural fibers under heterogeneous conditions was demonstrated. The AOR process for the reduction of fiber diameter of flax reveals its potential utility in synthetic fiber modification. Various technological applications of modified fibers include advanced polymer–fiber composites, fiber carriers for drug delivery, biomedical devices, advanced filtration, and adsorbent media [40,46].

Author Contributions: Conceptualization, L.D.W.; methodology, N.A.; formal analysis, N.A.; writing—original draft preparation, N.A.; writing—review and editing, N.A., L.D.W. and D.E.C.; supervision, L.D.W. and D.E.C.; project administration, L.D.W.; funding acquisition, L.D.W. All authors have read and agreed to the published version of the manuscript.

Funding: L.D.W. acknowledges the support provided by the Government of Canada through the Government of Saskatchewan (Ministry of Agriculture) Agriculture Development Fund (Project #: 20200408) for support of this research.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The raw data supporting the conclusions of this article will be made available by the authors on request.

Acknowledgments: Bernd G. K. Steiger is acknowledged for technical support with the collection of X-ray photoelectron spectra of fiber samples. Alvin Ulrich of Biolin Research Ltd. is acknowledged for helpful discussions during this research project. The authors wish to make a land acknowledgment that this work was carried out in Treaty 6 Territory and the Homeland of the Métis. As such, we pay our respect to the First Nations and Métis ancestors of this place and reaffirm our relationship with one another.

Conflicts of Interest: The authors declare no conflicts of interest.

References

1. Suriaman, I.; Hendrarsakti, J.; Mardiyati, Y.; Pasek, A.D. Synthesis and characterization of air filter media made from cellulosic ramie fiber (*Boehmeria nivea*). *Carbohydr. Polym. Technol. Appl.* **2022**, *3*, 100216. [\[CrossRef\]](#)
2. Lv, D.; Zhu, M.; Jiang, Z.; Jiang, S.; Zhang, Q.; Xiong, R.; Huang, C. Green Electrospun Nanofibers and Their Application in Air Filtration. *Macromol. Mater. Eng.* **2018**, *303*, 1800336. [\[CrossRef\]](#)
3. Du, Y.; Pundienė, I.; Pranckevičienė, J.; Kligys, M.; Girskas, G.; Korjaks, A. A Review of Biomass Wood Ash in Alkali-Activated Materials: Treatment, Application, and Outlook. *J. Compos. Sci.* **2024**, *8*, 161. [\[CrossRef\]](#)
4. Wang, J.; Li, Y.; Li, Q.; Long, Y.; Yu, T.; Li, Z. Evolution of stiffness in flax yarn within flax fiber reinforced composites during moisture absorption. *Compos. Part B Eng.* **2024**, *268*, 111096. [\[CrossRef\]](#)
5. Hall, L.M.; Booker, H.; Siloto, R.M.P.; Jhala, A.J.; Weselake, R.J. Chapter 6—Flax (*Linum usitatissimum* L.). In *Industrial Oil Crops*; McKeon, T.A., Hayes, D.G., Hildebrand, D.F., Weselake, R.J., Eds.; AOCS Press: Urbana, IL, USA, 2016; pp. 157–194.
6. Yan, L.; Chouw, N.; Jayaraman, K. Flax fibre and its composites—A review. *Compos. Part B Eng.* **2014**, *56*, 296–317. [\[CrossRef\]](#)
7. More, A.P. Flax fiber-based polymer composites: A review. *Adv. Compos. Hybrid Mater.* **2021**, *5*, 1–20. [\[CrossRef\]](#)
8. Pankaj; Jawalkar, C.S.; Kant, S. Critical Review on Chemical Treatment of Natural Fibers to Enhance Mechanical Properties of Bio Composites. *Silicon* **2021**, *14*, 5103–5124.
9. Oushabi, A. The pull-out behavior of chemically treated lignocellulosic fibers/polymeric matrix interface (LF/PM): A review. *Compos. Part B Eng.* **2019**, *174*, 107059. [\[CrossRef\]](#)
10. Hasan, A.; Rabbi, M.S.; Maruf Billah, M. Making the lignocellulosic fibers chemically compatible for composite: A comprehensive review. *Clean. Mater.* **2022**, *4*, 100078. [\[CrossRef\]](#)
11. Wang, W.; Wang, X.; Zhang, Y.; Yu, Q.; Tan, X.; Zhuang, X.; Yuan, Z. Effect of sodium hydroxide pretreatment on physicochemical changes and enzymatic hydrolysis of herbaceous and woody lignocelluloses. *Ind. Crops Prod.* **2020**, *145*, 112145. [\[CrossRef\]](#)
12. Luchese, C.L.; Engel, J.B.; Tessaro, I.C. A Review on the Mercerization of Natural Fibers: Parameters and Effects. *Korean J. Chem. Eng.* **2024**, *41*, 571–587. [\[CrossRef\]](#)
13. Montreuil, A.; Mertz, G.; Bardon, J.; Guillot, J.; Grysan, P.; Addiego, F. Flax fiber treatment by an alkali solution and poly(dopamine) coating: Effects on the fiber physico-chemistry and flax/Elum® composite interfacial properties. *Compos. Part A Appl. Sci. Manuf.* **2024**, *177*, 107963. [\[CrossRef\]](#)

14. Lorenci Woiciechowski, A.; Dalmás Neto, C.J.; Porto de Souza Vandenberghe, L.; de Carvalho Neto, D.P.; Novak Sydney, A.C.; Letti, L.A.J.; Karp, S.G.; Zevallos Torres, L.A.; Soccol, C.R. Lignocellulosic biomass: Acid and alkaline pretreatments and their effects on biomass recalcitrance—Conventional processing and recent advances. *Bioresour. Technol.* **2020**, *304*, 122848. [[CrossRef](#)] [[PubMed](#)]
15. Świątek, K.; Gaag, S.; Klier, A.; Kruse, A.; Sauer, J.; Steinbach, D. Acid Hydrolysis of Lignocellulosic Biomass: Sugars and Furfurals Formation. *Catalysts* **2020**, *10*, 437. [[CrossRef](#)]
16. Benali, M.; Oulmekki, A.; Toyir, J. The Impact of the Alkali-Bleaching Treatment on the Isolation of Natural Cellulosic Fibers from *Juncus Effesus* L Plant. *Fibers Polym.* **2024**, *25*, 525–533. [[CrossRef](#)]
17. Shehap, A.M. Thermal and Spectroscopic Studies of Polyvinyl Alcohol/Sodium Carboxy Methyl Cellulose Blends. *Egypt J. Solids* **2008**, *31*, 75–91.
18. Ferro, G.; Minciardi, R.; Podestà, E.; Robba, M. An optimization model for the sizing of the biomass plants' supply chain. *IFAC-PapersOnLine* **2018**, *51*, 114–119. [[CrossRef](#)]
19. Bautista, P.; Mohedano, A.F.; Casas, J.A.; Zazo, J.A.; Rodriguez, J.J. An overview of the application of Fenton oxidation to industrial wastewaters treatment. *J. Chem. Technol. Biotechnol.* **2008**, *83*, 1323–1338. [[CrossRef](#)]
20. Zhang, Y.; Chen, X.; Fang, L.; Zhang, C.; Xu, X. Fenton-reaction-aid selective delignification of lignocellulose by *Inonotus obliquus* to improve enzymatic saccharification. *Fuel* **2023**, *333*, 126355. [[CrossRef](#)]
21. Qadir, M.B.; Alsaiani, M.; Ali, Z.; Afzal, A.; Khaliq, Z.; Irfan, M.; Rahman, A.; Jalalah, M.; Harraz, F.A. Conversion of sugarcane biomass into sustainable fabrics: Softening of fibers using alkali and silicone softener treatment. *Mater. Res. Express* **2024**, *11*, 025103. [[CrossRef](#)]
22. Ge, J.; Lv, X.; Zhou, J.; Lv, Y.; Sun, J.; Guo, H.; Wang, C.; Hu, P.; Spitalsky, Z.; Liu, Y. Multi-level structured polylactic acid electrospun fiber membrane based on green solvents for high-performance air filtration. *Sep. Purif. Technol.* **2024**, *331*, 125659. [[CrossRef](#)]
23. Karthik, A.; Bhuvaneshwaran, M.; Senthil Kumar, M.S.; Palanisamy, S.; Palaniappan, M.; Ayrilmis, N. A Review on Surface Modification of Plant Fibers for Enhancing Properties of Biocomposites. *ChemistrySelect* **2024**, *9*, e202400650. [[CrossRef](#)]
24. Sun, Y.; Li, D.; Yu, Y.; Chen, J.; Fan, W. Separation and Characterization of Cellulose Fibers from Cannabis Bast Using Foamed Nickel by Cathodic Electro-Fenton Oxidation Strategy. *Polymers* **2022**, *14*, 380. [[CrossRef](#)] [[PubMed](#)]
25. Zhou, J.; Li, Z.; Yu, C. Property of ramie fiber degummed with Fenton reagent. *Fibers Polym.* **2017**, *18*, 1891–1897. [[CrossRef](#)]
26. Qin, R.-C.; Ma, Y.-Y.; Wang, D.; Bao, N.-Z.; Liu, C.-G. Preparation of Cellulose Nanofibers from Corn Stalks by Fenton Reaction: A New Insight into the Mechanism by an Experimental and Theoretical Study. *J. Agric. Food Chem.* **2023**, *71*, 1907–1920. [[CrossRef](#)] [[PubMed](#)]
27. Mazarji, M.; Minkina, T.; Sushkova, S.; Antonenko, E.; Mandzhieva, S.; Dudnikova, T. Impact of humic acid on degradation of benzo(a)pyrene polluted Haplic Chernozem triggered by modified Fenton-like process. *Environ. Res.* **2020**, *190*, 109948. [[CrossRef](#)] [[PubMed](#)]
28. Mohamed, M.H.; Udoetok, I.A.; Solgi, M.; Steiger, B.G.K.; Zhou, Z.; Wilson, L.D. Design of Sustainable Biomaterial Composite Adsorbents for Point-of-Use Removal of Lead Ions from Water. *Front. Water* **2022**, *4*, 739492. [[CrossRef](#)]
29. Kong, D.; Kusrini, E.; Wilson, L.D. Binary Pectin-Chitosan Composites for the Uptake of Lanthanum and Yttrium Species in Aqueous Media. *Micromachines* **2021**, *12*, 478. [[CrossRef](#)] [[PubMed](#)]
30. Fernandez, R.; Phillips, S.F. Components of fiber bind iron invitro. *Am. J. Clin. Nutr.* **1982**, *35*, 100–106. [[CrossRef](#)] [[PubMed](#)]
31. Akhtar, N.; Gupta, K.; Goyal, D.; Goyal, A. Recent advances in pretreatment technologies for efficient hydrolysis of lignocellulosic biomass. *Environ. Prog. Sustain. Energy* **2016**, *35*, 489–511. [[CrossRef](#)]
32. Lazić, B.D.; Pejić, B.M.; Kramar, A.D.; Vukčević, M.M.; Mihajlovski, K.R.; Rusmirović, J.D.; Kostić, M.M. Influence of hemicelluloses and lignin content on structure and sorption properties of flax fibers (*Linum usitatissimum* L.). *Cellulose* **2018**, *25*, 697–709. [[CrossRef](#)]
33. Fathi, B.; Harirforoush, M.; Foruzanmehr, M.; Elkoun, S.; Robert, M. Effect of TEMPO oxidation of flax fibers on the grafting efficiency of silane coupling agents. *J. Mater. Sci.* **2017**, *52*, 10624–10636. [[CrossRef](#)]
34. Dehabadi, L.; Karoyo, A.H.; Soleimani, M.; Alabi, W.O.; Simonson, C.J.; Wilson, L.D. Flax Biomass Conversion via Controlled Oxidation: Facile Tuning of Physicochemical Properties. *Bioengineering* **2020**, *7*, 38. [[CrossRef](#)] [[PubMed](#)]
35. Khiari, B.; Ibn Ferjani, A.; Azzaz, A.A.; Jellali, S.; Limousy, L.; Jeguirim, M. Thermal conversion of flax shives through slow pyrolysis process: In-depth biochar characterization and future potential use. *Biomass Convers. Biorefinery* **2021**, *11*, 325–337. [[CrossRef](#)]
36. Alabi, W.O.; Karoyo, A.H.; Krishnan, E.N.; Dehabadi, L.; Wilson, L.D.; Simonson, C.J. Comparison of the Moisture Adsorption Properties of Starch Particles and Flax Fiber Coatings for Energy Wheel Applications. *ACS Omega* **2020**, *5*, 9529–9539. [[CrossRef](#)] [[PubMed](#)]
37. Sun, J.X.; Sun, X.F.; Zhao, H.; Sun, R.C. Isolation and characterization of cellulose from sugarcane bagasse. *Polym. Degrad. Stab.* **2004**, *84*, 331–339. [[CrossRef](#)]
38. Kostyukov, S.G.; Petrov, P.S.; Kalyazin, V.A.; Masterova, Y.Y.; Tezikova, V.S.; Khluchina, N.A.; Labzina, L.Y.; Alalvan, D.K. Determination of Lignin Content in Plant Materials Using Solid-State ¹³C NMR Spectroscopy. *Polym. Sci. Ser. B* **2021**, *63*, 544–552. [[CrossRef](#)]

39. Puziy, A.M.; Poddubnaya, O.I.; Martínez-Alonso, A.; Castro-Muñiz, A.; Suárez-García, F.; Tascón, J.M.D. Oxygen and phosphorus enriched carbons from lignocellulosic material. *Carbon* **2007**, *45*, 1941–1950. [[CrossRef](#)]
40. Mir, M.; Wilson, L.D. Flax Fiber-Chitosan Biocomposites with Tailored Structure and Switchable Physicochemical Properties. *Carbohydr. Polym. Technol. Appl.* **2023**, *6*, 100397. [[CrossRef](#)]
41. Rodríguez-Ramírez, C.A.; Tasqué, J.E.; Garcia, N.L.; D'Accorso, N.B. Hemicelluloses hydrogel: Synthesis, characterization, and application in dye removal. *Int. J. Biol. Macromol.* **2023**, *253*, 127010. [[CrossRef](#)]
42. Feng, Q.; Cheng, H.; Chen, F.; Zhou, X.; Wang, P.; Xie, Y. Investigation of cationic dye adsorption from water onto acetic acid lignin. *J. Wood Chem. Technol.* **2016**, *36*, 173–181. [[CrossRef](#)]
43. Samaei, S.E.; Mahabadi, H.A.; Mousavi, S.M.; Khavanin, A.; Faridan, M.; Taban, E. The influence of alkaline treatment on acoustical, morphological, tensile and thermal properties of Kenaf natural fibers. *J. Ind. Text.* **2020**, *51*, 8601S–8625S. [[CrossRef](#)]
44. Li, Q.; Wang, A.; Long, K.; He, Z.; Cha, R. Modified Fenton Oxidation of Cellulose Fibers for Cellulose Nanofibrils Preparation. *ACS Sustain. Chem. Eng.* **2019**, *7*, 1129–1136. [[CrossRef](#)]
45. Aly, M.; Hashmi, M.S.J.; Olabi, A.G.; Benyounis, K.Y.; Messeiry, M.; Hussain, A.I.; Abadir, E.F. Optimization of Alkaline Treatment Conditions of Flax Fiber Using Box–Behnken Method. *J. Nat. Fibers* **2012**, *9*, 256–276. [[CrossRef](#)]
46. Anisimov, Y.A.; Evitts, R.W.; Cree, D.E.; Wilson, L.D. Polyaniline/Biopolymer Composite Systems for Humidity Sensor Applications: A Review. *Polymers* **2021**, *13*, 2722. [[CrossRef](#)] [[PubMed](#)]

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.