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Impact of fiber properties on floc formation and turbidity removal

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ABSTRACT

The water treatment industry is interested in sustainable approaches to minimize chemical demand in the coagulation and flocculation process. In conventional physicochemical treatment, lower water temperatures act to slow down particle collisions, chemical reactions, floc formation, and floc settling rates. This study explored the use of fiber-based super-bridging agents to compensate for the effect of temperature on the coagulation-flocculation process. The efficacy of recycled cellulose fibers was evaluated at the lab scale (250 mL) at various temperatures, demonstrating high turbidity removal with both settling and screening as floc separation methods. For the fiber-based treatment, turbidity differences at temperatures near 5 °C, 10 °C, and 20 °C were minimal indicating that this technology is more effective than the conventional approach (coagulant and flocculant) which showed significant variations between temperatures. Furthermore, regardless of the fiber source and properties, different cellulose fibers were efficient in turbidity removal acting as a super-bridging agent. Additional experiments were conducted to understand how fiber length and diameter distributions influenced the performance of the fiber-based treatment. Fibers of length > 2000 μm and diameter < 100 μm were more efficient in reducing turbidity and translated to lower chemical demand (i.e., 20 % reduction in alum demand for a target water quality of 20 NTU) in the coagulation and flocculation process. This sustainable fiber-based water treatment approach has the potential to lower the operational cost of water treatment plants operating at different water temperatures as a function of the season and geographical location, though techno-economic analysis is required to validate this hypothesis.

1. Introduction

The coagulation-flocculation process is one of the most important and widely used processes for water treatment, mainly due to its low cost, simplicity, and effectiveness [1]. Coagulation and flocculation are known to alter the physical state of dissolved and suspended solids to facilitate their removal [2]. Although this process has been widely used since the early 1900s, it has certain limitations; for example, the use of the non-sustainable additive, alum, produces high Al-loaded flocs in the sludge, which can contaminate soils following sludge application [3,4]. Moreover, the flocs obtained by such conventional treatment have limited settling velocity requiring the use of a large settling tank with a non-negligible footprint [5].

We recently showed that fiber-based water treatment significantly increases the floc size compared to ballasted flocculation with the added advantage of having a more active surface area than sand ballast medium [5] and also being more efficient in promoting floc growth than bio-based polysaccharides, which are used as alternatives to synthetic coagulants/flocculants [6,7]. It has been demonstrated that in the developed fiber technology, fibers serve to bridge the flocs formed during coagulation and flocculation, leading to the development of super-sized flocs that are up to ten times larger than those produced by conventional treatment, which increase the flocs' settling velocity and enable the adoption of a screen for floc separation (no settling tank) or a smaller settling tank [5]. This approach has been proposed as a more sustainable water treatment technology, particularly by using fewer metal-based coagulants and synthetic flocculants and a sustainable and renewable cellulose-based fibrous material [5]. The presence of fiber allows for reducing the use of non-sustainable chemicals, which is a major concern in some countries for drinking water applications due to the toxicity associated with flocculant usage [8,9]. Also, a previous study shows that the recovered fibers from the sludge can potentially be

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reused in subsequent treatment cycles [10]. This reuse-maintained process performance while reducing coagulant demand. Besides, through surface functionalization of the cellulosic fibers, this technology has potential applications in the removal of classical and emerging contaminants such as total suspended solids, phosphorus, natural organic matter, hydrocarbons, microplastics, and nanoplastics [10,11]. Although the addition of fiber as a super-bridging agent in water treatment is a promising approach, little is known about how the water characteristics and fiber properties can affect the treatment efficiency. Low water temperatures (< 10°C) are known to slow down coagulation kinetics, which leads to the formation of more irregular and less compact flocs [12–14]. The effectiveness of the coagulation and flocculation process is determined by floc size and density, which allows for easy floc removal using settling or screening methods. At lower temperatures, longer coagulation time and higher coagulant concentration are required to maintain the water treatment efficiency achievable at >10 °C [15,16]. Therefore, the primary objective of this study is to evaluate the effectiveness of fiber technology in mitigating temperature effects and to investigate how fiber properties influence performance, using turbidity removal as an indicator.

Researchers have been working for decades to develop cutting-edge and sustainable processes that use less coagulant and flocculant for effective water treatment at low temperatures. Surface water in northern regions experiences significant temperature fluctuations of nearly 20 °C between summer (18 to 24°C) and winter (1 to 2°C) [17] and during extreme weather events, resulting in a significant decline in suspended matter removal [18]. This necessitates the development of novel technologies capable of mitigating the effects of temperature on water treatment – especially physicochemical treatment – while not increasing the demand for coagulants or flocculants. This will have positive implications by producing sludge containing a lesser amount of these unsustainable chemicals [19].

When combined with the coagulation-flocculation process, fibers were shown to greatly increase floc size and improve the performance of physicochemical treatment [5]. However, at low temperatures, fiber-based water treatment may have lower efficiency. This aspect of the fiber-based water treatment process has not been explored. Another factor that may impact the performance of fiber-based water treatment is the fiber properties. Cellulosic fiber is a sustainable resource that can come from various sources (e.g., recycled paper, banana fiber, coconut coir, hemp fiber) with variations in fiber mean length, diameter, rugosity, and flexibility. To date, only virgin or recycled cellulose fibers were tested in fiber-based water treatment; thus, there is a need to better understand how fiber sources and properties impact the treatment.

In this study, we investigate the efficacy of cellulose fibers from diverse sources in treating synthetic wastewater and surface water at different temperatures by conducting jar tests at ~5 °C, 10 °C, and 20 °C. Moreover, the impact of the source, length, and diameter of fibers on turbidity reduction was examined to understand how the fiber properties can be optimized for contaminant removal.

2. Materials and methods

2.1. Preparation and characterization of the fibers

Recycled cardboard from 3 different sources (packaging materials) was used as primary material to prepare fiber suspensions of “brown” fibers (named A, B, C). Corrugated cardboard was cut into pieces (5 × 5 cm) and blended and rinsed in deionized (DI, 500 mL) water using a Ninja blender (40 g/L) and then collected in a 160 μm sieve to retrieve the fibers from DI water. The same process was repeated with sheets of Kraft pulp (Sigma-Aldrich) to obtain “white” fibers. The process was repeated thrice, then the rinsed/blended fibers were dried at 60 °C for 48 h. To prepare a 5 g/L fiber stock suspension, 0.625 g of dried fiber was blended for 7 s with 125 mL of DI water. Different concentrations of fiber suspension (0.05 – 0.15 g/L) were prepared from the stock suspension based on the requirement for jar tests.

Recycled cardboard (brown fibers) and kraft pulp (white fibers) offer a broad fiber length distribution, making them suitable for studying the effect of fiber length on turbidity removal. The fiber suspension prepared from brown fiber A was fractionated to investigate the influence of different fiber length fractions on the fiber-based wastewater treatment efficiency, as this fiber has the widest fiber length distribution (Fig. 1b). The lengthwise fractionation of fibers was achieved using a sequential sieving method [20]. Prior to its sieving, brown fiber A stock suspension of 40 g fiber/L was prepared as previously described. The mixture was sequentially sieved in order to separate the fibers by size. First, the fibers were sieved three times through a 1000 μm metal sieve. The retained fibers were collected and oven-dried at 60 °C for 24 h. The fibers, passing through the sieve, were then collected and sieved again three times with a smaller sieve. Namely, this procedure was done with 500, 250 and 125 μm sieves. The fibers recovered by the 1000 μm, 500 μm, 250 μm, and 125 μm sieves are referred to by their average length, namely 2280 μm, 1370 μm, 770 μm, and 470 μm, respectively. For jar tests, the stock suspensions of various length ranges of fibers were prepared from dried fibers.

To study the influence of short and long fibers in the fiber-based water treatment, fiber suspensions were prepared with different mass ratios of the shortest (470 μm) and longest (2280 μm) brown fibers, namely 50:50, 65:35, and 80:20. Separately, different natural fibers were used to concomitantly study the influence of fiber length and diameter on the fiber-based water treatment. For this purpose, coconut (coconut husk fiber, Home Depot), banana (raw untreated banana fiber, Natural fiber company), and hemp fibers (degummed hemp silver, Hemptrader) were used. Before using them for jar tests, the raw fibers were cut manually using a metallic cutter into different length fractions having an average length ~700 μm, 2500 μm, and 8500 μm. Each fraction was characterized using Fiji [21] to determine the length distribution of each fraction of fibers. Finally, the fibers were weighed and mixed with DI to reach the desired fiber concentration.

Images of fibers were obtained with a stereomicroscope (Olympus, SZX16, magnification of 10x) and an optical microscope (Olympus IX71, magnification of 40x) to characterize the length and diameter distribution of the fibers. All fibers prepared as mentioned above were then measured from microscope images using Fiji [21]. Specifically, the average fiber length of white fibers (Fig. 1a), and brown fibers A, B, and C (Fig. S2) were determined based on 190, 180, 145, and 195 observations, respectively. The average diameter of brown fiber A, hemp, coconut, and banana fiber was calculated based on 220, 200, 210, and 205 observations, respectively (Fig. S9). The average length of brown fiber A retained in 1000 μm, 500 μm, 250 μm, and 125 μm sieves were calculated based on 120, 150, 165, and 130 observations, respectively (Fig. S6). Finally, different length fractions of hemp, coconut, and banana having an average length of ~700 μm were calculated based on 130, 170, and 160 observations, ~2500 μm based on 146, 145, and 149 observations, and ~8500 μm based on 105, 102, and 98 observations, respectively (Fig. S10, S11, and S12).

An electrokinetic analyzer (EKA, SurPASS 3, Anton Paar GmbH) equipped with a cylindrical cell was used to evaluate the surface charge (zeta potential) of the fibers (brown fiber A, hemp, coconut, and banana). The streaming potential was measured at room temperature as a function of pressure difference in a cylindrical cell, when an electrolyte solution was forced through the system. Two Ag/AgCl electrodes were used to record the streaming potential. The relationship between the measured streaming potential and the zeta potential is given by the Helmholtz–Smoluchowski equation [22]. The electrolyte was a synthetic wastewater (SWW) solution, prepared according to the recipe outlined in Section 2.2, using only SWW salts without the addition of urea, peptone, and meat extract. The pH of the electrolyte was between 7.1 and 7.3. For each data point, at least six measurements were taken.

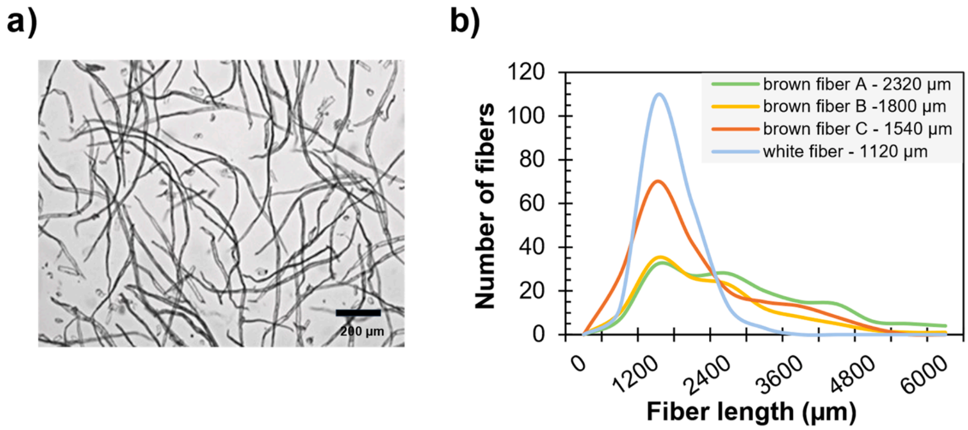

Fig. 1. Characterization of recycled cellulose fibers. a) Optical image of white fibers. b) Length distribution of white fiber, brown fiber A, brown fiber B, and brown fiber C, respectively.

2.2. Jar test procedure

Jar tests were carried out using a SWW recipe from [23] and surface water (Saint Lawrence River: turbidity 9 ± 2 NTU and pH 7.8 ± 0.2). To assess the effectiveness of super bridging fibers in mitigating water temperature effects, this study used synthetic wastewater as a simplified and controlled test matrix. The following compounds were used to make a 10-fold SWW: 35 mg sodium chloride (NaCl, Fisher Chemicals, #FW58.44), 20 mg calcium chloride (VWR, #1B1110), 0.15 g urea (Alpha Chemicals), 10 mg heptahydrate magnesium sulfate ($MgSO_4.7H_2O$, #M5921), 0.14 g dipotassium phosphate (K_2HPO_4, #P3786), 0.55 g of meat extract (#70164) and 0.8 g of peptone (#70951). Unless otherwise noted, all compounds were purchased from Sigma-Aldrich. The 10-fold concentrated SWW compound mixture was added to 500 mL of tap water and was agitated for 30 min. With 1 M NaOH solution, the pH of the SWW was adjusted to 8.

The SWW was prepared for 250 mL jar tests by combining 20 mL of the 10-fold concentrated SWW, 230 mL of tap water, and 350 μL of a SiO_2 suspension (40 g/L, 1 – 5 μm, #55631). The average initial turbidity of prepared SWW was 57.0 ± 1.5 NTU and pH was 7.7 ± 0.4. Jar tests were conducted by agitating the 250 mL SWW with a magnetic stirrer in a 500 mL beaker. Alum (ALS, Kemira: 0 – 65 mg of dry alum L^{-1}) was used as a coagulant to aggregate and precipitate the suspended particles [13,24]. Polyacrylamide (PAM, Hydrex 3511, Veolia Water; 0 – 2.0 mg PAM L^{-1}) is a polymer-based material used as a flocculant-aid to improve suspended matter removal [25,26]. It was prepared daily by dissolving 50 mg of PAM in 100 mL of DI water and vigorously agitated for 45 min. Fibers from different sources were added to the jar as a super-bridging agent at different concentrations (0 – 0.2 g/L; 0 – 15 mL of the 5 g/L fiber suspension) [5].

The protocol followed for conventional treatment and the fiber-based treatment involves two stages. The treatment process starts with the coagulation by adding alum to the jar (250 rpm for 2 min), followed by adding PAM at two equal time intervals (50 % at the beginning of flocculation and 50 % at mid-flocculation for a total flocculation time of 2 min) to reduce the floc breakage [27]. The fiber suspensions previously described were added to the jar 15 s before the first PAM dose, allowing it to mix homogeneously in the sample before being in contact with the flocculant.

The effectiveness of fiber-based treatment was studied using two floc-removal techniques: settling and screening (Fig. S1). For the settling method, 10 mL of the treated water was collected after 30 and 180 s of settling at a 2 cm depth for turbidity analysis, whereas in the screening method, a 2000 μm screen basket was inserted in the jar during flocculation (10 s before the end of flocculation) and a 10 mL sample was collected through the screen at a depth of 2 cm for turbidity analysis. Turbidity was measured with a TB 300 IR turbidimeter (Clear Tech). Each condition was tested in triplicate, except where noted.

The alum concentration at which the recycled cellulose fibers showed the best removal efficiency in fiber treatment was found by testing at different alum concentrations. Specific jar tests were conducted to study the impact of temperature on conventional water treatment, as well as the effectiveness of fiber-based technology in mitigating this impact. Specifically, this was done by conducting jar tests using water at different temperatures (5 ± 2 °C, 10 ± 2 °C, and 20 ± 2 °C) with constant coagulation (30 s) and flocculation time (120 s). Moreover, the effect on coagulation kinetics was studied by conducting jar tests with different coagulation times while keeping the flocculation time constant and maintaining the sample temperature ∼5 ± 2°C. Furthermore, various jar tests were performed with fibers from different sources to understand the impact of fiber length and diameter on fiber-based water treatment.

3. Results and discussion

3.1. Impact of fibers on water treatment

3.1.1. Length distribution of fibers

The length distribution of white fiber, brown fiber A, B, and C were analyzed and are shown in Fig. 1b. The average length of white fiber, brown fiber A, B, and C, is 1120 ± 430 μm, 2320 ± 1340 μm, 1800 ± 1070 μm, and 1540 ± 1020 μm, respectively. The white fiber has the shortest average length, 1120 μm, followed by brown fibers C, B, and A when listed in increasing order. The length distributions of fibers, shown in Fig. 1b, highlight differences between the brown fibers and the white fiber. In general, two distinct fiber length distributions are observed, one with a tighter distribution represented by the white fiber, and the other with a wider distribution represented by the brown fibers (Fig. 1b). Compared to the white fiber, the three types of brown fibers have more longer fibers > 2000 μm resulting in a broader size distribution. Among the three brown fibers, brown fiber C has more fibers < 2000 μm than the other two. Interestingly, all fibers are dominantly composed of fibers with a length centered around 1200 μm. For all fiber types, the fiber population length between 1200 and 2400 μm represents more than 60 % of the fibers. These recycled fiber materials were used to study the impact of temperature on fiber treatment and to observe differences in water treatment efficiency using recycled fibers from various sources.

3.1.2. Optimization of coagulation with alum

A first series of experiments was done to identify the working alum concentration at which various fibers perform well in terms of turbidity removal (Fig. S3). For all the tested fibers, when the alum concentration was 50 mg/L, the 180 s settled turbidity reached a minimum of 4 ± 2 NTU irrespective of differences in fiber length distribution (Fig. S3). The temperature impact study on conventional and fiber-based water treatment utilizing different recycled cellulose fibers was performed with an alum concentration of 50 mg/L, where a uniform behavior for all fibers was observed.

3.1.3. *Impact of temperature on water treatment*

Low water temperatures as encountered in regions such as northern USA, Canada, Norway, Sweden, and central Asia [28] are known to affect water treatment [18]. Various studies on suspended colloids and adsorption processes reported that temperature change can alter the surface charge, coagulation (Al-species hydrolysis), flocculation, the extent of adsorption, floc settling, viscosity, and collision frequency [13,29]. Low temperature (4 °C to 17 °C) exerts adverse effects on the hydrolysis of metals and aggregation of particles [12,30]. Furthermore, a decrease in temperature from 22 °C to 2 °C results in the formation of more irregular and less compact flocs [12].

As previously mentioned, fiber-based water treatment relies on coagulation and flocculation mechanisms [5,10,31]. Therefore, it is expected that temperatures may impact fiber-based treatment. Fig. 2a and S4a present wastewater turbidities obtained at various water temperatures for both treatment approaches. After 180 s of settling, the conventional treatment (no fiber) shows a significant difference in turbidity between temperatures (paired *t*-test, p-value < 0.05, Fig. 2a). A decrease in temperature (5 °C) in conventional treatment could reduce the proportion of particles able to overcome repulsive forces, thereby lowering the overall aggregation rate. This results in the formation of more irregular, less compact flocs that are less effective in settling and turbidity removal [12]. The settled turbidity also increased at higher temperature (20 °C), which may be because of temperature-influenced conformational changes in PAM bridging ability resulting in weaker or less stable floc formation [32,33]. Additionally the turbidity removal at 20 °C could be influenced by temperature-dependent changes in the charge characteristics of dissolved and suspended species, which can affect the optimal coagulant dosage and the efficiency of floc formation [34]. However, for fiber-based treatment, a temperature effect is not observed. Moreover, for the conventional treatment, no turbidity removal was observed at 30 s of settling (Fig. S4a). Such observations demonstrate that the wastewater temperature clearly impacts turbidity removal when using the conventional method. Interestingly, the fiber-based treatment demonstrated comparable turbidity removal at different temperatures, when either 30 s or 180 s of settling was applied (Fig. 2a and S4a). This demonstrates the efficacy of this new sustainable water treatment process at the tested temperatures. The effectiveness of fiber-based treatment is highlighted by the significant differences in turbidity removal compared to the conventional method at 5 °C and 20 °C (paired *t*-test, p-value < 0.05, Fig. 2a). From the results presented in Fig. 2a, fiber-based technology seems to be less impacted by temperature than conventional treatment. The fiber-based treatment also shows similar efficiency when tested with surface water at different temperatures (∼ 4 °C and 20 °C) with different coagulant concentrations (Fig. S4c). Therefore, regardless of the region's climate, fibers can be used to improve suspended matter removal over the traditional approach.

In a second experiment, the impact of fibers on turbidity removal at 5 °C was studied for different durations of coagulation. Results are presented in Fig. 2b and S4b. For the conventional treatment and

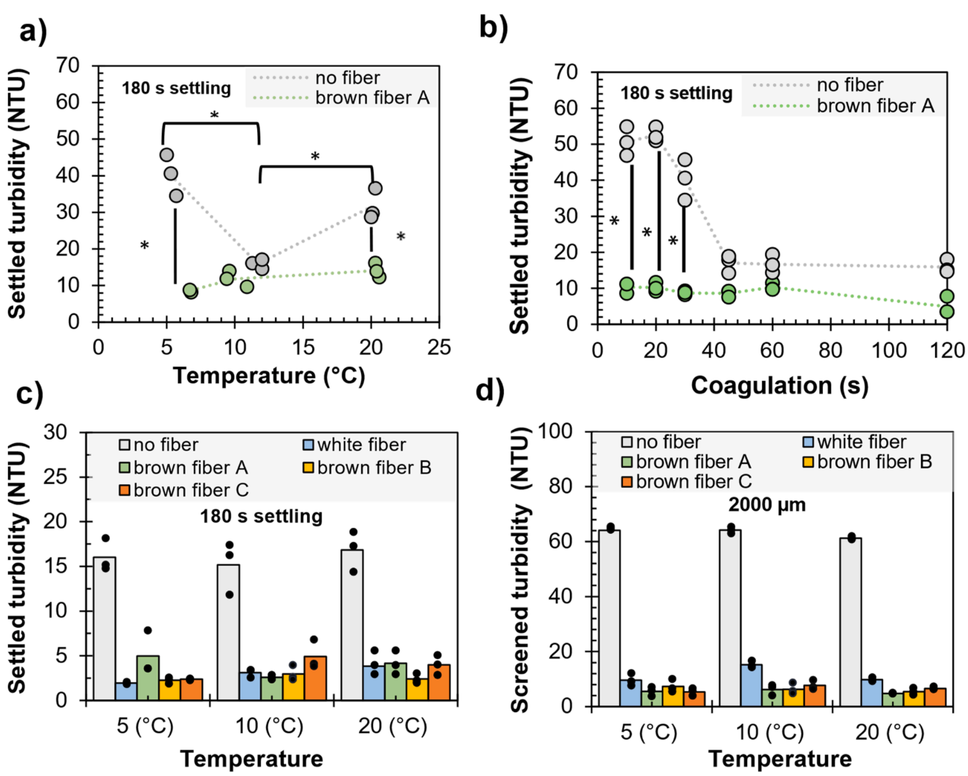

Fig. 2. Impact of temperature on turbidity removal a) Settled turbidity after 180 s for increasing temperature with coagulation and flocculation times of 30 and 120 s respectively, constant alum and PAM concentrations (50 mg/L and 0.5 mg/L. respectively), without and with fibers (0.15 g/L). b) Settled turbidity after 180 s for tests at temperature near 5°C (±2°C) for different coagulation times (0-120 s) and constant flocculation time (120 s), constant alum and PAM concentrations (50 mg/L and 0.5 mg/L, respectively), without and with fibers (0.15 g/L). a and b: Dashed lines are included as eye guides connecting average values obtained from triplicate experiments. c-d) Settled turbidity and screened turbidity (2000 µm screen) for increasing temperature with constant coagulation (120 s) and flocculation (120 s) time using white fiber, brown fiber A, brown fiber B, and brown fiber C, respectively, constant alum, PAM, and fiber concentrations (50 mg/L, 0.5 mg/L, and 0.15 g/L, respectively). Bars indicate the average value from triplicate experiments while symbols represent individual data points. * Paired *t*-test < 0.05 between indicated average turbidity values (n=3).

coagulation times > 45 s, the 180 s settled turbidity value dropped drastically to reach a plateau at 17 ± 2 NTU (Fig. 2b). In contrast, the fiber-based treatment yielded effective treatment regardless of coagulation time (Fig. 2b). Both results presented in Fig. 2a and 2b emphasize the ability of the fibrous material to overcome the temperature effect on the coagulation process. These results suggest that fiber can be used as a flocculant aid in mitigating the temperature effect on coagulation kinetics known to decrease water treatment efficiency [35]. At low temperatures, in traditional treatment, extra alum doses are added to counter temperature effects [17]. By adding fibers, as no temperature effect was observed on the coagulation kinetics, it is expected that further alum consumption reduction may be possible. At room temperature, such alum reduction was already reported at the lab and pilot scales for drinking water and wastewater treatment [5,31]. Moreover, the coagulation time may be reduced, which will increase the volume of water treated per hour.

Although this study evaluated the performance of both conventional and fiber-based technologies with synthetic wastewater and water from the St. Lawrence River at various temperatures (Fig. S4c), in reality, wastewater and surface water have a complex and variable composition, including natural organic matter, emerging and persistent organic pollutants, inorganic salts, and colloids that can significantly affect the coagulation and flocculation process. Therefore, it is essential to further investigate the effects of temperature and coagulation kinetics using diverse real wastewater matrices from different sources to better understand the turbidity removal efficiency of fiber-based treatments.

The water treatment efficiency of the different recycled cellulose fibers at various temperatures was studied, using two different floc separation methods (settling or screening) with the effective coagulation and flocculation time of 120 s as used in [31], and the results are presented in Fig. 2c and 2d, respectively. After 180 s of settling, for fiber-based treatment, no significant treatment efficiency differences were noted between the temperatures and all fibers studied (Fig. 2c). This may be explained by the fiber-length distribution of the recycled fibers studied. Most of them are composed of fibers having a length between 1200 to 2400 μm, attributed to 60 % of the fiber distribution (see section 3.1.1). This range of fiber lengths may be more effective in bridging the flocs formed and thereby enhancing the turbidity removal.

As there is no significant difference between the settled turbidity after 30 s and 180 s (Fig. 2c and S5a), this indicates that the settling velocity of the flocs was substantially improved due to the super-bridging property of fibers, allowing for rapid settling of flocs formed during the coagulation-flocculation process [5,10]. In contrast, without fibers, in the conventional treatment, 30 s settled turbidity values for all temperatures tested were significantly higher (> 50 NTU) than those obtained after 180 s of settling (< 20 NTU). The flocs being smaller than those obtained using fiber, they need more time to settle and to reduce turbidity. This difference in floc settling time is further observable when comparing turbidity values obtained for both methods with a settling time of 30 s (< 8 NTU, Fig. S5a). Fibers clearly accelerate the floc settling as already demonstrated [5].

The potential of fiber-based treatment on turbidity removal using the screening method shown in Fig. 2d is in agreement with our previous work [31]; the differences in screened turbidity achieved by the different recycled cellulose fibers are considerably low at all temperatures. However, the conventional treatment and fiber-based technology have a significant difference (> 50 NTU, Fig. 2d). This implies that the flocs formed in conventional treatment were smaller, limiting the removal ability of the 2000 μm screen used. In contrast, the super-bridging ability of fibers improved the floc size in fiber treatment, thereby preventing the transport of flocs through the screen. This shows that fiber-based technology is effective in improving water quality irrespective of the water temperature as well as the method employed for floc separation.

The turbidity removal efficacy of recycled cellulose fibers demonstrates that fiber-based technology can be more efficient than conventional water treatment, facilitating coagulation mechanisms through the addition of materials that increase the probability of collision and floc formation [5], notably at low temperatures as encountered in mid-latitude regions during the year [28]. This first study focusing on the effectiveness of fiber-based technology on water treatment at different temperature conditions is important to prove the relevance of its application in different climates. Synergistically combining fibers with coagulants could improve the water treatment plants' robustness and capacity globally, hence helping municipalities meet regulations despite increasing demand. Therefore, this technology will open a possibility for new as well as existing water treatment plants throughout the world to utilize this technology regardless of the floc separation methods that have been adopted and the local temperature conditions.

3.2. *Impact of fiber properties on wastewater treatment*

Although the efficiency of fiber-based water treatment has been studied, the influence of cellulose fiber dimensions (e.g., length and diameter) on turbidity removal was not specifically addressed. Preliminary results using recycled fibers from different sources with similar diameters seem to indicate that the 1200-2400 μm fiber fraction may be more effective in bridging flocs than the other fractions (Fig. 2c). In order to confirm this hypothesis and to optimize the effective length and diameter range of fibers used in fiber-based water treatment, further experiments were conducted.

3.2.1. *The contribution of different fiber fractions to turbidity removal*

Previous research has shown the super-bridging ability of recycled cellulose fibers which leads to the formation of super-sized flocs that are up to ten times larger than those formed in conventional treatment, as evidenced by floc characterization [5,31]. Expanding on this, the present study focuses on the impact of fiber length in turbidity removal by utilizing the recycled cellulose fibers having a broader fiber length distribution (Fig. 1b). A sequentially sieved recycled fiber was used to study the contribution of different length fractions of fibers to turbidity removal. As described in section 3.1.1, recycled brown fibers are composed of different fiber length populations, mainly between 450 and 5000 μm (Fig. 1b). Previous results suggested that the fraction between 1200 and 2400 μm may play a crucial role (Fig. 2c). To confirm this, brown fiber A was sequentially fractionated. Brown fiber A was selected here because it has the widest range of lengths (average length 2320 ± 1340 μm, Fig. 1b). The sieved length distributions of brown fiber A are shown in Fig. 3a and S6. The fibers recovered by sieving have a mean length of 470 ± 170, 770 ± 260, 1370 ± 650, and 2280 ± 980, for the 125, 250, 500, and 1000 μm sieve fractions, respectively. The impact of fiber length on settled turbidity was evaluated by conducting jar tests with a settling separation and using a lower alum concentration, namely 40 mg/L. This lower amount of alum allowed to challenge the coagulation [36] and therefore better understand the coagulant-aid provided by the fibers of different length fractions via bridging of the smaller flocs formed at lower alum concentrations.

The turbidity removal achieved by different lengths of brown fiber A after the 30 and 180 s settling times are shown in Fig. 3b and S7a. After 180 s of settling, a significant difference in turbidity was observed between the 125 and 1000 μm sieved fibers (paired *t*-test, $p\text{-value} < 0.05$, Fig. 3b) as well as between fibers sieved through 500 and 1000 μm sieves (paired *t*-test, $p\text{-value} < 0.05$, Fig. 3b). However, there is no statistically significant difference in removal between the 1000 μm sieved fibers, having an average length of 2280 ± 980 μm, and the unsieved fibers having an average length of 2320 ± 1340 μm ($p\text{-value} > 0.05$, Fig. 3b). This can be explained by the almost uniform fiber length distribution of both fiber fractions, which have the non-negligible presence of fibers > 2000 μm (Fig. 3a). The results suggest that longer fibers play an important role in the formation of larger flocs, namely those fibers having a length greater than ~1000 μm. Therefore, the longer fibers act as a super-bridging agent by effectively bridging the smaller flocs

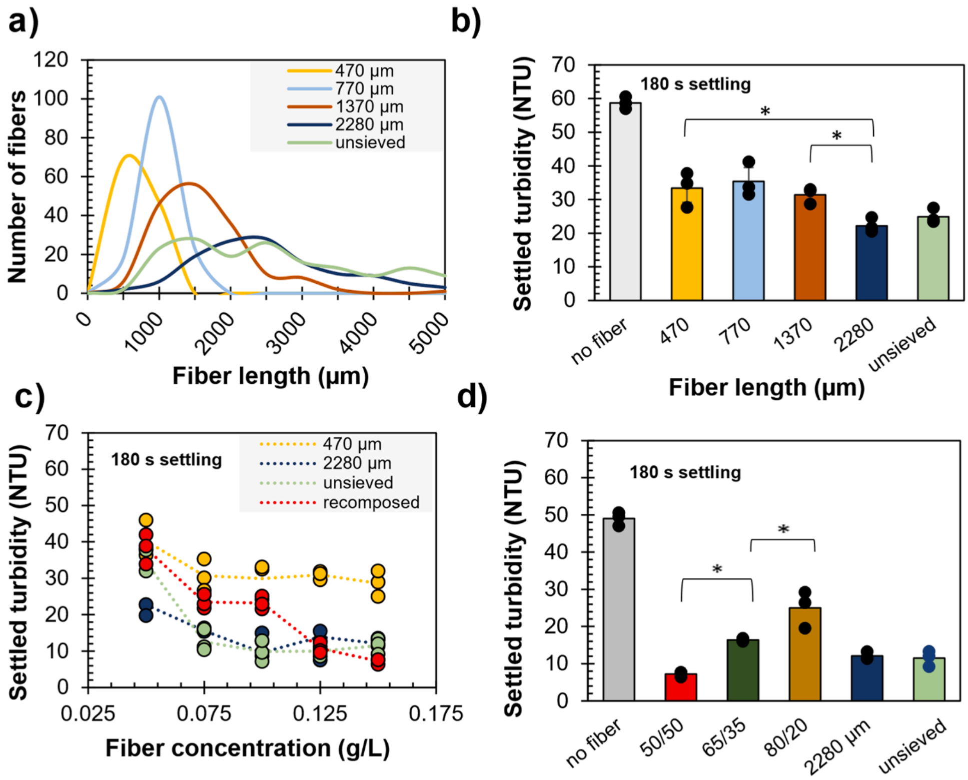

Fig. 3. Impact of fiber length on turbidity removal. a) Length distribution of fibers retained in 125 μm (n= 130), 250 μm (n= 165), 500 μm (n= 150), and 1000 μm (n= 120) sieves, respectively. b) Settled turbidity after 180 s using the sieved brown fiber A (0.15 g/L), constant alum, and PAM concentrations (40 mg/L and 0.5 mg/L, respectively). Note that the fiber length reported is the fiber average length. c) Settled turbidity after 180 s with different lengths of brown fiber A (470 μm, 2280 μm, unsieved, and recomposed (50:50 of 470 μm and 2280 μm based on mass, respectively)) of 0.05 to 0.15 g/L concentration, constant alum and PAM concentrations (45 mg/L and 0.5 mg/L, respectively): Dashed lines are included as eye guides connecting average values obtained from triplicate experiments. d) Settled turbidity after 180 s with controlled mixture of brown fiber A having average lengths of 470 μm and 2280 μm in the ratio of 50:50, 65:35, and 80:20, respectively with fiber concentration of 0.15 g/L, constant alum and PAM concentrations (45 mg/L and 0.5 mg/L, respectively): Bar in the graph indicates the average value from triplicate experiments while symbols represent individual data points. * Paired *t*-test < 0.05 between indicated average turbidity values (n=3).

formed at lower alum concentrations. Although direct microscopic floc characterization was not performed in this study, macroscopic observations such as turbidity removal and the rapid settling of agglomerates resulting from the formation of larger flocs strongly support the super-bridging mechanism of the fibers.

The influence of the length of fibers on fiber-based wastewater treatment was also studied by changing the fiber concentration (0.05 to 0.15 g/L) while maintaining alum and PAM concentrations constant at 45 mg alum/L and 0.5 mg PAM/L, respectively (Fig. 3c and S7b). For small fibers, 470 ± 170 μm in average length, turbidity values after both 30 and 180 s settling are significantly higher compared to other length fractions at different fiber concentrations (Fig. 3c and S7b). This result highlights the inability of shorter fibers to act as super-bridging agents to produce larger flocs with higher settling velocities. This emphasizes that the flocculant-aid properties of fiber materials demonstrated in the work of [5] are principally due to the long fiber length population (> 1000 μm). By contrast, the unsieved fibers and those having an average length of 2280 ± 980 μm showed essentially identical removal rates except at very low fiber concentration, down to 0.05 g/L (Fig. 3c and S7b). This high turbidity removal at low fiber concentration was also observed by [31], while studying the scalability of this technology for wastewater treatment. Those results confirm that fiber-based water treatment may work well at low concentrations, namely ~ 0.075 g/L, making the process even more sustainable and cheaper by reducing operational costs. However, it is also important to acknowledge that the addition of fibers leads to increased sludge volume, and the higher organic content may affect dewatering efficiency and disposal costs. Preliminary unpublished data from our laboratory shows that the addition of functionalized cellulose fibers can enhance sludge dewatering. Nevertheless, the potential increase in disposal costs associated with increased sludge volume should be further explored in future research.

The results in Fig. 3b revealed the lower turbidity removal efficiency of fibers < 500 μm. However, as shown in Fig. 3b, raw recycled fibers (unsieved) including both shorter and longer fibers (see Fig. 1b) achieved comparable water treatment efficiency to that of the sieved longer fibers, having an average length of 2280 μm. This indicates that fiber-based water treatment is tolerant to shorter fibers. However, the treatment may be impacted due to the greater presence of shorter fibers as indicated in Fig. 3d. As recycled fibers are not a controlled material, it is expected that shorter fibers may be present at various proportions depending on the recycled cardboard sources and production batches. In this view, it is highly relevant to study the maximal proportion of shorter fibers for which no negative impact is observed on the fiber-based water treatment efficiency.

To this end, further experiments were conducted to investigate if there is a substantial change in turbidity removal when there is a greater presence of shorter fibers in the mix. First, a controlled mixture of fibers (50 % of 470 μm and 50 % of 2280 μm) was tested. This "recomposed" fiber sample attained better outcomes when the fiber concentration was above 0.1 g/L (Fig. 3c and S7b). In contrast, the 2280 μm fraction mainly composed of longer fibers was still effective in reducing turbidity down to 20 NTU at 0.75 g/L. Interestingly, the 470 μm fraction, for all concentrations tested, was not effective in reducing turbidity down to 20 NTU. Thus, it is clear that small fibers have a negative impact on fiber-based treatment. To identify the maximum proportion of shorter fibers that can be present with minimal impact on turbidity removal, the fibers of 470 μm and 2280 μm were mixed in the ratios of 50-50 %, 65-35 %, and 80-20 %, based on mass, respectively. Fig. 3d and S7c, clearly show that the turbidity removal of the controlled mixture improves when the fraction of short fibers reaches 50 %. When the ratio of the fraction of short fibers increased from 50 % to 65 % and 80 %, the final turbidity significantly increased. This is because shorter fibers are more likely to remain suspended rather than settle, emphasizing the negative impact of small fibers on the fiber-based treatment (p-value < 0.05, Fig. 3d and S7c). This highlights the importance of the minimum proportion of longer fibers needed for the effective removal of shorter fibers as well as bridging of the flocs formed in the treatment. This also explains the comparable behavior of unsieved fibers and fibers having an average length of 2280 ± 980 μm (p-value > 0.05, Fig. 3b and S7a).

Therefore, it is clear that the fibers of length > 2000 μm are certainly more effective in removing the shorter fibers and in bridging the flocs formed during the coagulation and flocculation processes, thus enhancing the removal mechanism regardless of the settling time. According to this, when using recycled fibers, it may be important to characterize the proportion of the fibers of length < 1000 μm as the presence of too many shorter fibers may decrease the resulting water treatment efficiency.

3.2.2. *Role of fiber diameter in turbidity removal*

Cellulosic material from numerous sources can be used for fiber-based water treatment. Although these fibers are mainly composed of cellulose, they show differences in terms of diameter and length (see Table S1). In general, their diameters range between 10 and 300 μm. Such differences in fiber diameter may impact the fiber behavior and thus its performance during fiber-based treatment.

In order to investigate the effect of fiber diameter and length on turbidity removal as well as its role in the super-bridging character of the fibers, a few different types of cellulose fiber were used; namely hemp, coconut, and banana. As expected, these fibers showed differences in diameter. The hemp, the coconut, and the banana have an average diameter of 30 ± 12 μm, 180 ± 68 μm, and 198 ± 76 μm, respectively (Fig. S9). The diameters of brown fiber A, hemp, coconut, and banana fiber show distinct distributions (Fig. 4a). The diameter distributions of brown fiber A and hemp are narrower, whereas coconut and banana have a wide range of diameter values between 100 to 350 μm.

Jar tests were performed to assess the combined impact of the fiber diameter and length on suspended matter removal. For this purpose, the fibers were cut to different lengths as characterized in Fig.s S10, S11, and S12, and the average length of each fraction of fibers obtained is described in Table S2. Both the 30 s and 180 s settled turbidity values indicate that fibers with larger diameters, such as coconut and banana, are less efficient in turbidity removal for all three length ranges tested (Fig. 4b and S13). This could be attributed to the low density of these fibers, which causes them to float instead of settle, as well as their surface characteristics, which might influence the super-bridging nature of the fibers [37,38].

In contrast, brown fiber A and hemp fiber having the same average diameter of 30 μm, are efficient in turbidity removal especially when the length of fibers is greater than 2000 μm (Fig. 4b and S13). This result supports the observation of the fiber length impact study described in the previous section: fibers with length > 1000 μm were shown to be the fraction having the most influence on turbidity removal. Interestingly, for the same short length fractions (< 1000 μm), the brown fiber A reached higher turbidity removal than the hemp fiber (Fig. 4b and S13). The difference in turbidity removal may be linked to the distinct fibers' surface characteristics [39,40], which are expected to play an important role in changing the super-bridging character of the fibers. In any case, hemp fiber can be used as an alternative to brown fiber A when there is a demand for using fibers of greater length in fiber-based water treatment as there is less variation in 30 s and 180 s turbidity removal between these fibers (< 5 NTU) when the length exceeds 1000 μm (Fig. 4b and S13). In another potential scenario, if the use of a particular recycled cardboard yields too many fibers < 1000 μm (i.e., > 50 %), hemp fiber of greater length may be added in order to reach a balance of short and long fibers.

This series of experiments demonstrates that fibers with lengths greater than 2000 μm and diameters less than 100 μm effectively remove suspended matter. While the variations in the surface characteristics and other properties of fibers might influence the super-bridging character of fibers [41], this factor appears to be less important for longer fibers. The zeta potential of brown fiber A (–3.5 mV) appears less negative than that of hemp (–5.9 mV), coconut (–5.6 mV), and banana (–4.3 mV), as shown in Fig. S14. This indicates that brown fiber A is generally less anionic than the other fibers, which may contribute to its better performance in turbidity removal. However, when comparing hemp to coconut and banana fibers, it becomes evident that the surface charge of fibers alone does not play a significant role in turbidity removal, as the zeta potentials of hemp and coconut are quite similar. Additionally, other surface properties—such as fiber morphology, porosity, roughness may influence the super-bridging ability of the fibers, which needs to be further investigated in future research so that fibers with optimal properties can be selected from a wide range of available fiber materials.

4. Conclusion

This study investigated the effectiveness of fibrous materials in treating synthetic wastewater and natural surface water (Saint Lawrence River) at various temperatures. The results indicate that fiber-based

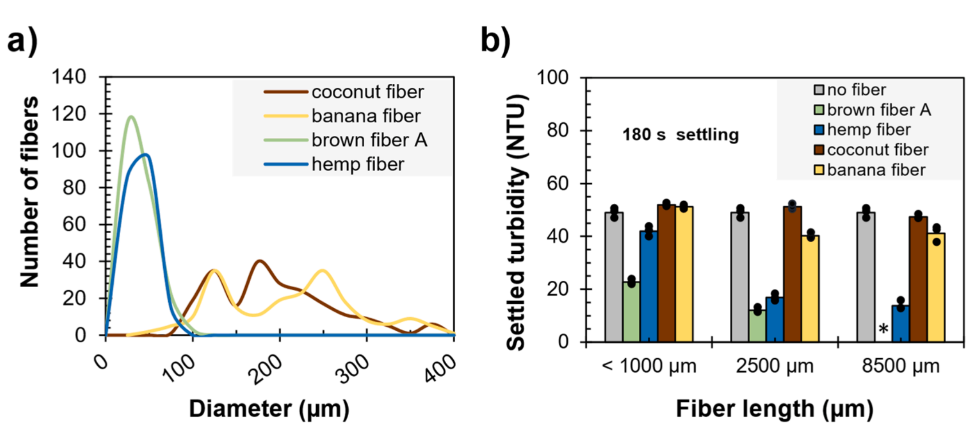

Fig. 4. a) Diameter distribution of brown fiber A, hemp, coconut, and banana fibers based on microscope image analysis. Impact of fiber diameter and length on turbidity removal. b) Settled turbidity after 180 s with and without fibers (0.15 g/L), constant alum, and PAM concentrations (45 mg alum/L and 0.5 mg PAM/L, respectively): Bars indicate the average value from triplicate experiments while symbols represent individual data points. * Condition not tested.

super-bridging agents are promising solutions for suspended matter removal regardless of temperature and methods employed for floc removal (i.e., settling vs screening). In contrast, the conventional treatment shows significant differences in turbidity removal between temperatures tested. Although the fiber-based treatment presents interesting advantages, the origin of the fibers may have an impact on fiber properties such as length and diameter, which in turn influence the treatment outcome. The results show that fibers with lengths > 2000 μm and diameters < 100 μm exhibit better treatment performance than shorter and thicker fibers. However, other fiber properties such as surface morphology, roughness, porosity as well as density might alter the behaviour of fibers in the coagulation-flocculation process. Moving forward, more research in this area is needed to better understand the role of these fiber properties, allowing the use of locally available recycled fibers and/or coproduct fibers in different geographical regions. It is important to note that this study used synthetic wastewater as a controlled matrix to systematically examine the impact of water temperature and fiber properties. Although previous studies shown the effectiveness of fiber technology in turbidity removal from real wastewater at ambient temperature, additional investigation is needed to evaluate its effectiveness at lower water temperature using real wastewater samples, which have a more complex composition. While a techno-economic analysis was beyond the scope of this study, but this and previous work shows that irrespective of the separation methods utilized or the climate of the region, fibers could help maintain the plant capacity and aid in coagulation at cold temperatures, also allowing to design and implement a smaller settling tank in existing and future water treatment plants and thus lower operating costs. Also, while not tested in the present study, there is potential to reuse the fibers recovered from the sludge in subsequent cycles; however, further research is needed to fully understand and effectively manage the sludge produced at the end of the treatment process.

CRediT authorship contribution statement

Krishnaveni Kannan: Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Florent Blancho:** Writing – review & editing, Validation, Methodology, Formal analysis, Conceptualization. **Haifa Rjab:** Writing – review & editing, Investigation, Formal analysis, Data curation. **Mathieu Lapointe:** Writing – review & editing, Validation, Supervision, Methodology, Conceptualization. **Nathalie Tufenkji:** Writing – review & editing, Supervision, Resources, Project administration, Methodology, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests

Nathalie Tufenkji and Mathieu Lapointe have patent pending on the use of fiber-based materials for water treatment.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.ceja.2025.100880.

Data availability

Data will be made available on request.

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