

Fiber-based super-bridging agents improve flotation and settling during water treatment

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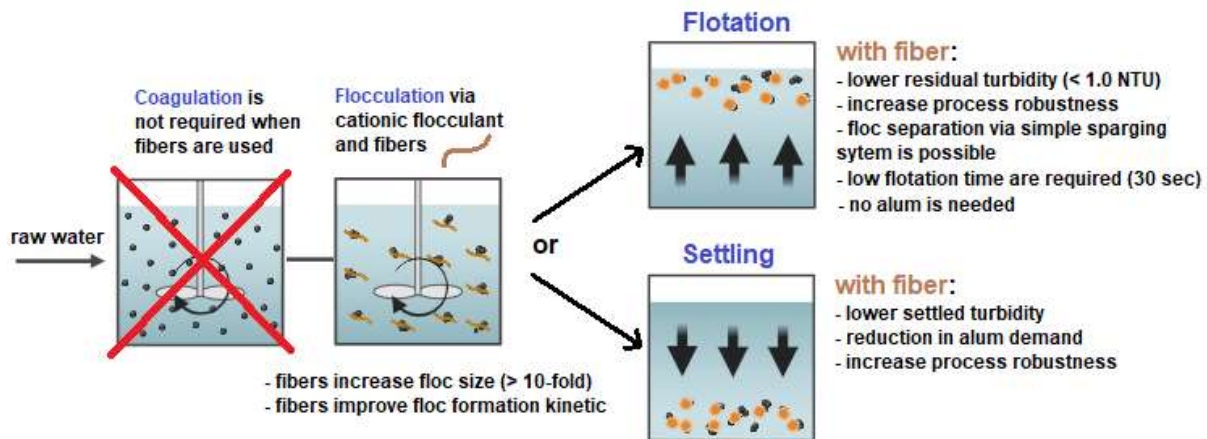
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Graphical abstract



Highlights

- Fibers generated flocs that are 10–100 times larger.
- Larger fibrous flocs incorporated more bubbles during air-sparging.
- The use of fibers resulted in a 50 % reduction in coagulant demand.
- Fibers enhanced the removal of flocs during settling and flotation.
- The use of fibers resulted in a reduction in flocculant demand.

37 **Abstract**
38

39 Increasing demand for water poses a major challenge to the water treatment industry. To maintain their floc
40 removal efficiency, water treatment plants are expected to require larger separation units and use more
41 chemicals, namely, coagulants and flocculants. Conventional physicochemical treatments produce flocs
42 that are limited in size, which limits floc removal efficiency via gravitation-based processes such as settling
43 and flotation. Introducing fiber-based super-bridging agents has improved the floc size, which is 10–100
44 times larger than conventional flocs. Such improvements could lead to important gains in floc separation
45 and ultimately increase the capacity of water treatment plants. This study analyzed the behavior and
46 interaction of fibers under various coagulation/flocculation conditions to improve flotation and settling.
47 Residual turbidity < 1.5 NTU was systematically achieved when the fibers were combined with
48 conventional physicochemical treatments (alum and polyacrylamide). The results also showed that fiber-
49 based super-bridging agents can allow a ~50 % reduction in coagulant. Three types of renewable fibers
50 originating from the residues were selected for jar tests: softwood cellulose, brown fibers extracted from
51 recycled cardboard boxes, and hemp fibers. The floc settling rate increased considerably when fibers were
52 previously incorporated into the floc structure during aggregation, thus acting as a super-bridging agent.
53 The benefits of fibers on floc settling velocity were particularly pronounced during suboptimal coagulation,
54 as the injection of fibers compensated for poor coagulation conditions. Air bubbles during air flotation were
55 also better incorporated into the larger and more porous floc structure obtained via the fibers, which
56 drastically improved floc removal during flotation. Ultimately, such fiber-based super-bridging agents can
57 be introduced in existing water treatment plants for wastewater and drinking water applications to increase
58 plant capacity, reduce the demand for coagulants/flocculants, and improve contaminant removal.

59 **Keywords:** Floc separation, Coagulation and flocculation, Water quality, Super-bridging agents, Settling, Flotation, Water
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Introduction

The water industry is exploring more sustainable and alternative treatment methods to improve conventional physicochemical and gravitational treatments¹ that currently treat over 70 % of the municipal water in North America.^{2,3,4} Coagulation and flocculation are used in synergy to maximize floc size and resistance to shearing, process robustness, settling rate, and floc removal.^{5,6,7} Physicochemical process is a key element in water treatment plants to simultaneously reach higher capacity and better remove both regulated and emerging contaminants.^{8,9} Nevertheless, more water treatment plants will reach capacity in 30 years. Moreover, some contaminants such as nanoplastics (NPs), per- and polyfluoroalkyl substances (PFAS), specific NOM (natural organic matter) fractions, and some metals are known to be refractory to conventional metal-based coagulants (e.g., alum and ferric sulfate) and organics flocculants (e.g., polyacrylamide (PAM)).^{10,11,12} Conventional coagulation and flocculation also represent an operational cost for water treatment plants as such chemicals cannot be reused, i.e., they are lost and accumulated in sludge.^{13,14,15} This sludge is costly to manage, treat, and transport. At the same time, the accumulation of metal-based coagulants also increases sludge toxicity, significantly reducing its potential for reuse in agricultural applications as fertilizer.^{16,17,18} Some metal-based coagulants are systematically used in the water treatment industry as they provide relatively high removal of several regulated contaminants for drinking (turbidity and NOM) and wastewater applications (phosphorus and TSS) – at low cost.¹⁹ However, using coagulants alone requires long settling times to ensure the removal of the small flocs formed. High molecular weight polyacrylamide-based flocculants, either anionic or cationic, usually increase the size of flocs and minimize the required settling time to meet turbidity and TSS removal objectives.²⁰ This synergy between coagulants and flocculants has been deployed in several physicochemical treatments globally to improve floc removal and/or increase plant capacity.

Fiber-based materials, used as super-bridging agents, have recently been proposed to increase the floc size and settling rates.^{21,22,9} These studies specifically focused on the impact of fibers during settling and screening on removing NOM, TSS, turbidity, and plastic debris (improved removal of nanoplastics and 15 μm microplastics, up to 80 %).^{23,24} In terms of cost, performance, and sustainability, these fibers offer promising solutions for eliminating various regulated and emerging contaminants. However, in previous studies, only a few types of fibers have been tested, and the impact of fibrous materials on the flotation performance has never been explored. Hence, combined with conventional coagulants and flocculants, this study assessed the impact of three different fibrous materials (softwood fibers, fibers extracted from recycled cardboard boxes, and hemp fibers) on floc removal after settling and flotation. To reduce the process complexity and operational cost, fibers were combined with cationic polyacrylamide (simultaneously acting as a coagulant and flocculant) to eliminate metal-based coagulants without impacting floc removal.

Materials and methods

Jar tests

5–60 mg alum/L (ALS, Kemira) and 0.025–0.400 mg PAM/L (Hydrex 3511, molecular weight $> 10^6$ g/mol, anionic charge density < 5 %) were injected as coagulant and flocculant, respectively. The alum and PAM concentrations were optimized to reach < 1.5 NTU during conventional treatment (i.e., without fibers). Coagulation with alum (110 rpm) was performed for 2 minutes, followed by flocculation (110 rpm) for 2 minutes. To maximize the floc size, the PAM was introduced dually: 50 % of the dose was introduced at the beginning of flocculation, while the remaining 50 % was added at mid-flocculation to reduce reconfiguration of the polymer chain (coiled vs. extended chain configuration).^{24,25} Different flocculation times were also tested (30–240 s) to reach an optimal floc maturation time for flotation and settling. The fibers were injected 20 s before flocculation (i.e., 20 s before PAM injection). Three types of fibers were

142 tested: i) cellulose-based brown fibers obtained from recycled cardboard boxes (fiber a), ii) softwood-based
143 fibers obtained from renewable sources, and iii) hemp-based fibers (fiber c) derived from plant cellulose.
144 Four fibrous treatments were tested: 1) 100 % fiber a, 100 % fiber b, 100 % fiber c, and fiber blends (50 %
145 of fibers a or b and 50 % fiber c). All the jar tests were conducted in duplicate.

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147 For some jar tests, to avoid the use of metal-based coagulation (alum), the coagulation phase was
148 eliminated, and the anionic PAM used in the flocculation phase was replaced with a cationic high molecular
149 PAM (C-498, Kemira; 0–3 mg PAM/L; molecular weight > 10⁶ g/mol; cationic charge density > 40 %).²⁶
150 In that case, such cationic PAM simultaneously acted as the coagulant and the flocculant to initiate
151 aggregation (i.e., coagulation) and complete floc maturation via bridging mechanism (i.e.,
152 flocculation).^{27,28,5} Eliminating the use of alum and using only one chemical would also simplify the
153 operation and reduce the operational and capital expenditures (storage tank and injection system), a key
154 element for smaller treatment plants in municipalities and marginalized communities. Avoiding alum also
155 reduces the need for water treatment to readjust the pH after physicochemical treatment because metal-
156 based coagulants consume alkalinity and reduce the pH during coagulant reaction/hydrolysis.⁶

157
158 Turbidity and pH were measured on raw and treated water (Turbidimeter, Hach 2100N, SM 2130 B; pH
159 meter, OAKTON-510).²⁹ The impact of fibers (200 mg/L injected in each jar test; sampled from a
160 suspension of 5 g fibers/L in deionized water) on aggregation and floc size was examined after settling (30
161 and 180 s) and flotation (30 and 60 s) (objective: < 1.5 NTU after treatment).

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164 **Preparation of fibers**

165 Brown cellulose, softwood, and hemp fibers were selected to increase floc size during the jar test. The
166 preparation method was reported previously by Kurusu et al. (2022).²² Briefly, 5 g of fibers were blended
167 with 1 L of DI water to ensure a homogeneous suspension (Ninja Blender, model BL450C, ≈ 1200 rpm for
168 7 s).

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171 **Water preparation**

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173 Synthetic surface water (6.2 ± 0.7 NTU) was prepared by using 250 mL of tap water (city of Montréal,
174 Canada) in which 100 µL of polysaccharides-based organic matter (from a solution of 500 mg/L; Corn
175 Starch 2395-113-12-00, Selection) and 250 µL of kaolin (from a suspension of 10 g/L; Kaolin 7567750,
176 Cattier) were added. All jar tests were performed at room temperature. The synthetic water was pre-
177 stabilized in a jar at room temperature and agitated with a magnetic stirrer at 110 rpm for 5 min in a 500
178 mL glass beaker.

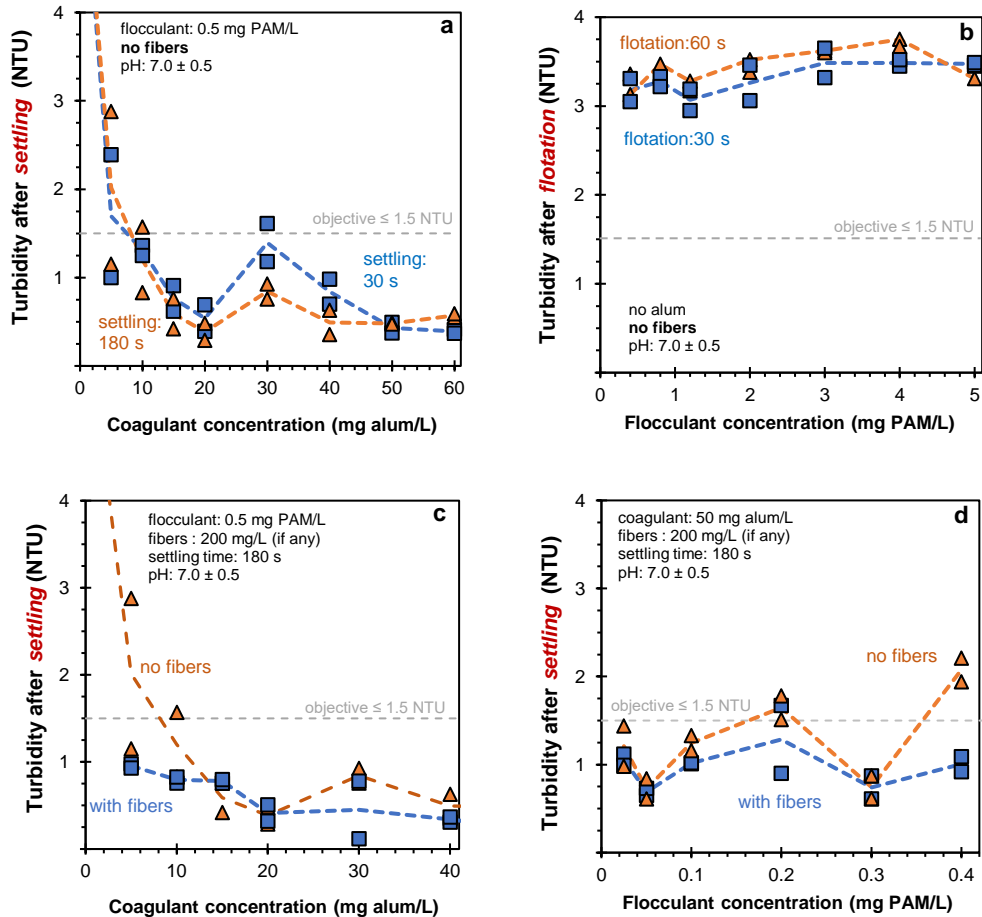
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181 **Results and Discussion**

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183 **Impact of fibers on turbidity removal and coagulant/flocculant demand**

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185 Settling experiments investigated various alum (5–60 mg of dry alum/L) and PAM concentrations (0.25 –
186 5 mg PAM/L). These tests determined the optimal values for conventional treatment processes without
187 fibers. The concentration was optimal when the residual turbidity lower than 1.5 NTU was achieved.
188 Turbidity was still high under certain treatment conditions for which the floc size was too small, for
189 example, for coagulant concentrations < 10 mg alum/L (>1.5 NTU) (Figures 1a and 1c). A mixture of alum
190 and PAM was used along with 200 mg/L of fibers to evaluate the effect of the fibers on the conventional

191 treatment process. When fibers were introduced, all the tested alum and anionic PAM concentrations
 192 achieved settled turbidity of < 1.5 NTU (Figures 1c and 1d; blue curves). All removal performances were
 193 mainly attributed to the formation of larger flocs via the tested fiber-based super-bridging agent.
 194

195 For the flotation process (Figure 1b), c-PAM without fibers was used as a control. Despite the relatively
 196 high cationic PAM concentration injected during the jar test (up to 5 mg cationic PAM/L), the residual
 197 turbidity after flotation was still considerably higher than that of the 1.5 NTU target (> 3 NTU). A longer
 198 flotation time or smaller air bubbles would probably have been required for experiments without fibers to
 199 allow separation and better turbidity removal.
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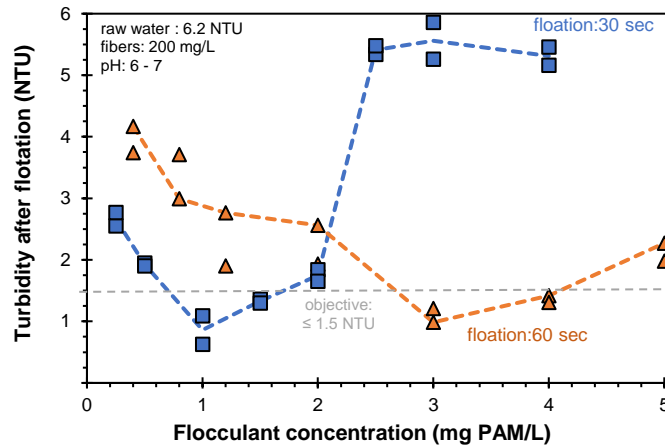


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203 **Figure 1:** Impact of coagulant concentration on turbidity removal after 30 and 180 sec of settling (a). Impact of flocculant (cationic
 204 PAM) concentration on turbidity removal (no fiber) after 30 and 60 sec of flotation (b). Impact of fibers on coagulant demand (180 sec
 205 of settling) (c). Impact of fibers on flocculant (anionic PAM) demand (180 sec of settling) (d). Coagulation time: 2 min. Flocculation
 206 time: 2 min (50 % flocculant injected at the beginning and 50 % injected at mid-flocculation). Blue and orange dashed lines are included
 207 as eye guides connecting average values obtained from duplicate experiments. The grey dashed line shows the turbidity target after
 208 treatment (< 1.5 NTU). Raw water: 6.2 ± 0.7 NTU.
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212 To reach the 1.5 NTU target during flotation, fiber-based super-bridging agents were used in synergy with
 213 cationic PAM (Figure 2). The impact of flotation time on turbidity removal (30 and 60 s of flotation) was
 214 evaluated. Different cationic PAM concentrations (0.25 – 5 mg/L) were also evaluated for the tested
 215 flotation times (Figure 2; no alum). A shorter flotation time (30 s) reduced the consumption of cationic
 216 PAM (< 1.5 NTU) to 1 mg PAM/L. However, for a longer flotation time (60 s), the PAM concentration

217 required to reach the 1.5 NTU target was 3 mg PAM/L. For the tested jar test conditions, it was hypothesized
 218 that a longer flotation time during air sparging could induce inadequate shear stress on the floc (leading to
 219 floc fragmentation), affecting the floc separation efficiency. These experiments revealed the importance of
 220 adjusting the PAM concentration based on the flotation time, especially when fibers are used. However,
 221 with no fiber treatment combined with flotation, the 1.5 NTU was never reached for all conditions tested
 222 (Figure 1b).
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 226 **Figure 2:** Impact of flocculant concentrations on turbidity removal after 30 and 60 s of flotation. Conditions: 200 mg/L of brown
 227 cellulose fibers. Flocculation time: 2 min. Flocculant: cationic high molecular cationic PAM (C-498). Dashed lines are included as eye
 228 guides connecting average values obtained from duplicate experiments. The grey dashed line shows the turbidity target after treatment
 229 (< 1.5 NTU).
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231 **Effect of flocculation and flotation time on fiber-based floc formation**

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 233 Different flocculation (15 – 240 s) and flotation times (30 – 150 s) were tested using the optimal cationic
 234 PAM concentration established in Figure 2 (3 mg PAM/L; 60 s of flotation). The primary objective is to
 235 minimize the size of an eventual full-scale system by determining the most effective flocculation time to
 236 form large flocs and achieve efficient flotation. Evaluating the optimal flocculation time is important in
 237 aggregation systems combined with gravitational separation. Multiple factors, such as pH, temperature,
 238 polymer, and water characteristics, influence the floc formation kinetics, size, and resistance to
 239 shearing.^{5,30,31} An optimal flocculation time of 150 s was determined in Figure 3a, as the lowest turbidity
 240 (0.85 NTU) was measured, which met the turbidity target of 1.5 NTU. For such experiments, the optimal
 241 flocculation time was determined to be a compromise between inducing sufficient flocculation time,
 242 generating enough PAM-fiber-particle interactions, and inducing an inadequately long flocculation time,
 243 leading to floc fragmentation and breakage.^{32,33,34}
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245 The optimal flocculation time (150 s) shown in Figure 3a was used to determine the optimal flotation time.
 246 Figure 3b shows that the optimal flotation time was 120 s. It is hypothesized that such a flotation time
 247 allows more air bubbles into the fiber-based floc structure while limiting the shearing stress occurring at
 248 longer flotation times. Floc fragmentation was observed at longer flotation times (150 s), which limited the
 249 incorporation of air bubbles during sparging.
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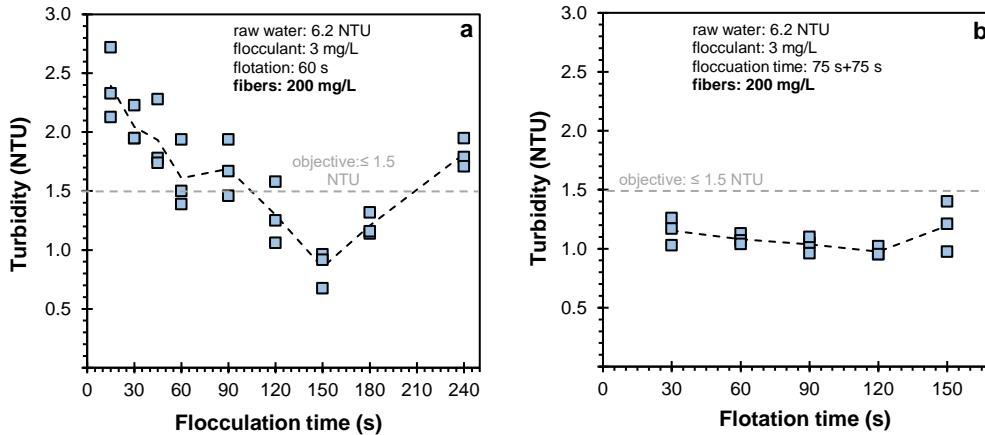


Figure 3: Impact of flocculation time on turbidity removal during flotation of fiber-based flocs (a). Conditions: 3 mg PAM/L, 200 mg fibers/L. Impact of flotation time on turbidity removal of fiber-based flocs (b). Conditions: 3 mg PAM/L, 200 mg fibers/L. No alum was used (a and b). Flocculant: cationic and high molecular weight PAM (C-498). Dashed lines are included as eye guides connecting average values obtained from triplicate experiments. The grey dashed line shows the turbidity target after treatment (< 1.5 NTU).

Impact of fiber type on floc formation and turbidity removal

Different fibers (brown cellulose, softwood, and hemp fibers) were introduced at a fixed concentration of 200 mg/L as a bridging structure to improve floc formation kinetics and size (Figure 4a). Three concentrations of cationic PAM were tested using three fibers: 1.0, 1.5 and 2.0 mg PAM/L. The results shown in Figure 4a were compared by evaluating the turbidity removal after flotation for each cationic PAM concentration and fiber type. Softwood fibers exhibited the lowest residual turbidity at higher cationic PAM concentrations (2 mg /L): 0.81 NTU after 30 s of flotation. The brown fibers exhibited the lowest residual turbidity at lower PAM concentrations (1 mg/L): 1.11 NTU after 30 s flotation. However, at 1 mg PAM/L, hemp fibers showed a higher residual turbidity of 2.23 NTU after 30 sec flotation: this lower performance was however compensated with higher PAM concentration as a lower residual turbidity of 1.53 NTU was measured with 2 mg PAM/L. This suggests that the hemp fibers may have less affinity for the tested polymer, resulting in a lower flocculation efficiency and higher post-flotation turbidity.

Combinations of different fiber types can improve the flocculation efficiency, but the synergy between them is variable. 1.5 mg PAM/L worked best for the hemp/brown fiber blend (200 mg fibers/L; Figure 4b), reducing turbidity after flotation. For the hemp/softwood blend, 2 mg PAM/L was optimal for turbidity reduction (figure 4b). However, the combination of fibers did not consistently improve the treatment, highlighting the need to find the right balance between the concentration of cationic PAM and fiber content/type. Further experiments synergistically combining two types of fibers – some of them coming from very cheap residues – could be completed to reduce the number of fibers injected, operating cost, and process footprint.

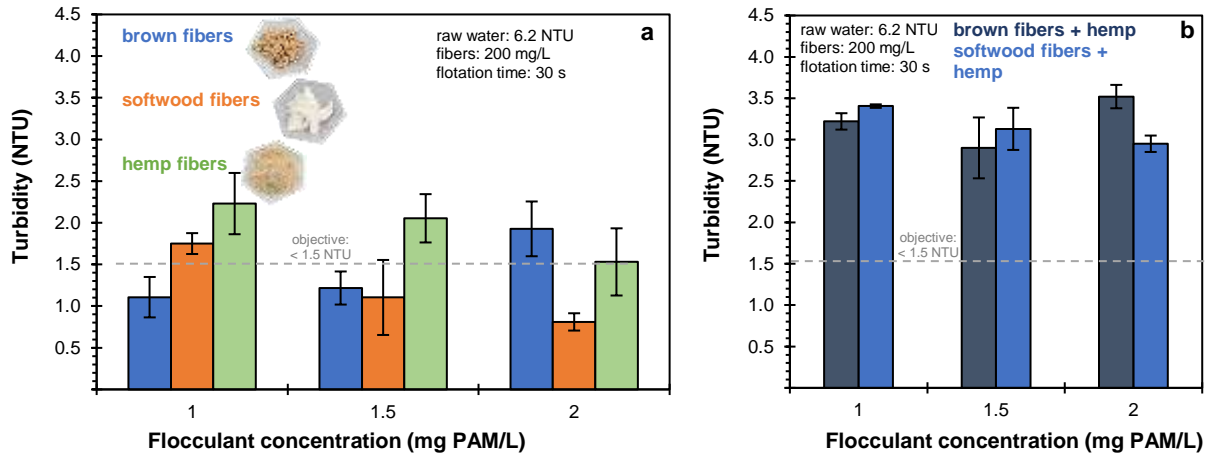


Figure 4: Impact of fiber types on turbidity removal: comparison between brown, softwood, and hemp fibers (a). Impact of fibers combination on turbidity removal (b). Flocculant: cationic high molecular PAM (C-498). Conditions: 200 mg fibers/L, flotation time: 30 seconds, c-PAM: 1–2 mg PAM/L. Means values are presented, and error bars represent the standard deviation obtained from triplicate experiments (a and b). The grey dashed line shows the turbidity target after treatment (< 1.5 NTU).

Benefits of fibers in the water treatment industry

Fibrous super-bridging agents are expected to improve the efficiency and sustainability of separation methods used in water treatment, especially in physicochemical treatments followed by settling, for which fibers have been shown to drastically improve floc size, stability, formation kinetics, and settleability.^{23,35,36} The present study shows that fibers can also improve floc removal during flotation. The potential for significant cost reduction (smaller separation units or reduction in chemical demand) is also one of the most notable benefits of incorporating fibers into water treatment processes. Conventional water treatment processes rely on expensive single-use metal-based coagulants and synthetic flocculants.²⁴ However, experiments have shown that fibers can be used as cost-effective alternatives. The tested cellulose-based fibers were also inexpensive, biodegradable, nontoxic, and readily available.^{37,38} Such fiber-based flocculant aids could also increase operational flexibility and process robustness.^{39,40,41}

Conclusion

Cellulose fibers and cationic polyacrylamide significantly improved the flotation performance during water treatment. Fibers drastically improve the floc formation kinetics and increase the floc size, increasing air incorporation into the floc structure during sparging. Flocculation time also played a critical role in floc formation when fibers were used, and the optimal time was shown to be a compromise between floc formation and fragmentation owing to shear overexposure.

In combination with cationic polyacrylamide, cellulose-based fibers achieved a 75% reduction in turbidity by flotation and drastically outperformed conventional physicochemical treatments (turbidity removal > 30 %). This improvement was attributed to the fact that the fibers increased the stability of the flocs and facilitated flotation by creating lighter flocs owing to air incorporation. The experiments also showed that metal-based coagulants were not required during flotation when fibers were synergistically used with cationic flocculants; consequently, a significant reduction in operational costs and sludge production is expected.

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Author Contributions Statement

B. Raissouni and L. Benkara conducted the experiments. B. Raissouni wrote the first draft of the manuscript. M. Lapointe conceived and designed the study and prepared the manuscript. M. Lapointe developed fiber-based superbridging agents for flotation applications.

Competing Interests Statement

M. Lapointe applied for a patent on using fiber-based materials in water treatment. The authors declare no conflicts of interest.

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