

Industrial mineral suspension treatment with alkali dissolved cellulose-based flocculants

Tomi Eilamo

`tomi.eilamo@aalto.fi`

Aalto University

Olli Dahl

Aalto University

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Abstract

Fine mineral particles can limit water recycling in mining industry and cause environmental issues if not treated properly. Flocculation process can be used to increase the particle size of mineral particles and facilitate separation, but conventional flocculant polymers have sustainability issues. We studied alkali solutions of cellulose and phosphorylated cellulose as green flocculants for talc suspension, thickener overflow, and flotation tailing. The effectiveness of treatment was assessed by measuring the turbidity of the suspensions during settling. Cellulose-based flocculants significantly improved the turbidity reduction for all studied suspensions. Turbidity of talc suspensions could be consistently reduced by over 99.8%. Thickener overflow could be treated to 97.4% turbidity reduction with 10-minute settling using only 2 ppm dose of phosphorylated cellulose solution. For flotation tailing a turbidity reduction of 99.9% could be reached with a high flocculant dose of 84 ppm. Phosphorylation improved flocculant performance. Cellulose isolated from polyester cotton textiles had a comparable performance as a flocculant to commercial microcrystalline cellulose. Scanning electron microscopy was used to investigate the interaction between flocculated mineral particles and cellulose flocculant. Cellulose solution forms solid cellulose bridging between the suspended particles as it regenerates during the flocculation process.

Introduction

Suspended mineral suspensions contain fine particles that are difficult to control in process and discharge flows (Mierczynska-Vasilev et al. 2013). They accumulate in process flows and can limit water recycling in mining and mineral processing (Zago et al. 2023). If discharged, suspended solids can cloud and contaminate ground and surface waters, reduce visibility, and obscure sunlight causing harm to aquatic life and vegetation (Shaikh et al. 2017; Anju and Jaya 2022; Pavolová et al. 2026). An efficient way to improve the control over the fine particles is to increase their particle size by introducing a flocculant: a molecule with high aspect ratio capable of physically bridging gaps between suspended particles and bringing them in contact with one another.

Conventional flocculation chemicals are synthetic water-soluble polymers, most commonly polyacrylamides (PAM). Although they can be used in an environmentally beneficial way to remove solid pollutants from water to enable more water recycling and safer water discharge, they themselves are slowly biodegrading microplastics that are manufactured with toxic and carcinogenic monomers, and their emission to the environment should be prevented (Raza et al. 2023; Zago et al. 2023).

Talc, a slow settling hydrophobic layered silicate, was used as a model mineral in the present study. Talc flocculation has been done with various ways, such as polymer flocculation with PAM (Ozkan 2003), polyethylene oxide (Yeap et al. 2010), modified and unmodified starches (Mierczynska-Vasilev et al. 2013), and hydrophobic flocculation using kerosene (Yi et al. 2018). Carboxymethyl cellulose (CMC) is used as a depressant in flotation processes to reduce talc floatability (Bacchin et al. 2006; Manono et al. 2019).

We have recently discovered new green alternatives to synthetic flocculants: alkali solutions of cellulose and phosphorylated cellulose (Eilamo and Dahl 2026; Eilamo et al. 2026). Cellulose is abundant, renewable, and biodegradable polymer, and our method allows using it as a flocculant without toxic chemicals or expensive processes, whereas most other cellulose-based flocculants rely on more complicated, non-green chemistry to functionalize cellulose (Song et al. 2011; Cai et al. 2015; Wang et al. 2019; Koshani et al. 2020; Fauzani et al. 2021; Ribau Teixeira et al. 2024; Magalhães et al. 2024). One potential drawback with the alkali dissolved flocculants is that they inherently raise the pH of the treated water. However, the stability of talc suspensions is highly dependent on the pH of the surrounding water (Ozkan 2003; Ersoy 2011). This presents an opportunity to use both components of the flocculant solution, the cellulose and the alkali, as an aid for talc flocculation.

The purpose of this work was to show that alkali solutions of cellulose can be used as flocculants for waters that contain suspended minerals. We show that different types of cellulose solutions have different flocculation efficiency on talc suspensions, and that these flocculants are not limited to flocculation of synthetic mineral suspensions but also have potential for treating real process water flows from the mining industry.

Experimental

Flocculant preparation

Materials

Flocculant solutions were prepared from two cellulose materials. Dry cotton linter microcrystalline cellulose (MCC) powder was purchased from Sigma-Aldrich. Cotton linter MCC was used as it was commercially easily available. The intrinsic viscosity was determined previously as 130 ml/g (Eilamo and Dahl 2026). Cellulose isolated from post-industrial polyester cotton textile blends (PC-MCC), was received from Nordic Bioproducts Group as >99% pure cellulose and intrinsic viscosity of 170 ml/g.

Phosphorylation

MCC and PC-MCC were phosphorylated with sodium dihydrogen phosphate (NaH_2PO_4) and heat curing (Naderi et al. 2016; Eilamo et al. 2026). With 400 RPM mixing NaH_2PO_4 was dissolved in 200 ml of water, cellulose was added, and pH was adjusted to 5 using 1 M NaOH. The consistency of cellulose was 10 % and the amount of NaH_2PO_4 was 44.3 g, corresponding to 2.3 phosphates per anhydroglucose. The mixture was dried at 105 °C overnight and cured at 150 °C for 1 h. It was then soaked overnight and washed until washing water conductivity was under 20 $\mu\text{S}/\text{cm}$. Degree of substitution (DS) for phosphorylated MCC (PMCC) and phosphorylated MCC from polycotton (PMCC) was determined by conductometric and potentiometric titration with 0.2 M NaOH (Reid et al. 1949; Eilamo et al. 2026).

Dissolution

MCC and PMCCs were dissolved in NaOH based on the freeze-thaw method (Isogai and Atalla 1998) with slight modifications (Eilamo and Dahl 2026; Eilamo et al. 2026). 2 g of cellulose was dispersed in 53.8 g of water followed by addition of 5 g of NaOH as pellets (VWR, GPR RECTAPUR). The mixture was then frozen overnight. 40.2 g of water was added, and the mixture was shaken at room temperature until thawed. It was then stored in refrigerator.

Flocculation

Talc suspensions

Magnesium chloride (MgCl_2) hexahydrate (VWR, AnalaR NORMAPUR, ACS/Reag. Ph.Eur.) was used as an electrolyte to enhance coagulation of talc. (Shaikh et al. 2018; Eilamo and Dahl 2026). Sodium chloride (NaCl) (Sigma Aldrich) was used as background conductivity. 350 mesh talc from Sigma-Aldrich (CAS: 14807-96-6) was used to mimic a turbid industrial mineral processing effluent.

Flocculation tests were done with Kemira Flocculator 2000 in 1 L containers. 5 g of talc was mixed with 1 L of 1 mM NaCl for of 100 ± 10 minutes at 400 RPM to assure dispersion and constant initial turbidity of 2400 NTU across all test points.

Flocculation procedure was (1) MgCl_2 addition as a 0.2 g/ml solution., (2) 30 s of rapid mixing at 400 RPM, (3) flocculant addition, (4) 10 minutes of slow agitation at 50 RPM followed by immediate removal of mixer from the container, (5) 10-minute settling with continuous turbidity measurement using YSI ProDSS at 7 cm below the surface and pH measurement with Thermo Scientific Orion 2 Star. Turbidity values were logged once a minute during settling and pH was measured once after 5 minutes of agitation at 50 RPM. 30 ml of water was collected from 2 cm below the surface after settling. Sedimented solids were separated by decanting.

Preliminary tests with MgCl_2 and MCC alkali solution were used to determine suitable doses for comparing flocculants.

Industrial process waters

Two types of water were received from an industrial mineral mine in Finland. Both waters were part of the mine's internal process loop and contained suspended minerals including talc. The process waters were a thickener overflow with dry matter content of 0.46% and initial turbidity of 320 NTU, and a low solid flotation tailing stream pumped to tailing pond for settling (later "flotation tailing") with dry matter content of 4.2% and initial turbidity of 7200 NTU.

Flocculation procedure was as with talc suspensions apart from omitting the NaCl and MgCl_2 addition. The process waters were mixed overnight before flocculation and continuously during sampling.

Scanning electron microscopy (SEM)

Additional talc flocculation samples were prepared for SEM at talc concentrations of 0.05%, 0.10%, and 0.25%. This was done to find more cellulose surfaces among the talc. SEM samples were also prepared from both process waters after flocculation.

Sedimented solids were dried with a Labconco Freezone 2.5 freeze dryer. Dried sediments were attached to SEM stubs on graphite tape and coated with a Quorum Technologies Q 150 R sputter coater with 80/20 gold/palladium at 20 mA for 30 seconds. Sediments were imaged using Zeiss Sigma VP at 2 kV EHT and SE2 detector.

Results

Flocculant preparation

Flocculant solutions were prepared from MCC, PC-PMCC, and PMCC. Two batches of PMCC flocculant solutions are denoted as PMCC A and PMCC B. The DS of phosphorylation was 0.07 for PC-PMCC and 0.09 for both PMCC A and PMCC B. This suggests that cellulose isolated from polycotton blend had lower reactivity for phosphorylation with the chosen method.

Flocculation

Talc suspensions

The prepared alkali flocculant solutions were used to flocculate 0.5% talc suspensions. As seen in Figure 1 and Figure 2, phosphorylation improved the turbidity reduction both after 1 minute and after 10 minutes of settling time, although after 10 minutes the difference was less significant. All PMCCs were effective flocculants reaching turbidity reduction of over 99.7% in 1 minute and over 99.8% in 10 minutes of settling compared to the initial turbidity of 2400 NTU. The difference between PC-PMCC and PMCC was smaller than the difference between the two PMCC batches A and B. These results are an average of three replicates with standard deviation depicted as a bar, apart from PC-PMCC which is an average of two replicates.

Additional test points were done without flocculant to assess the effect of flocculant. With 2 mM of MgCl_2 turbidity after 10 minutes of settling was 146 NTU. With 2 mM of MgCl_2 and NaOH dose equivalent to 28 ppm of flocculant solution the turbidity reached 42.1 NTU after 10 minutes of settling. This would suggest that the effect of NaOH is significant in turbidity reduction. After 1 minute of settling this treatment resulted in turbidity of 1610 NTU leaving it far from even the least effective MCC flocculant that reached 33.5 NTU. Based on these comparisons it was clear that all flocculant polymers had a positive impact on turbidity reduction, and that phosphorylation had the most significant impact during mixing and at the start of settling.

Industrial process waters

PMCC B was used to flocculate both industrial process waters as shown in Figure 3. The effect of flocculant dose on turbidity and pH is shown for thickener overflow in Figure 4 and for flotation tailing in Figure 5. As expected, the pH increases with the flocculant dose in both waters due to the high amount of NaOH present as a solvent.

Interestingly, even the smallest investigated dose of 2 ppm had a significant effect on the turbidity of thickener overflow. The turbidity could be lowered from the initial 320 NTU to 84 NTU with just 1 minute of settling and all the way down to 8.25 with 10 minutes of settling. This was a major improvement over no flocculant addition which resulted in 178 NTU in 1 minute and 127 NTU in 10 minutes of settling. The final pH with this dose was 8.38, which is a minor increase from the initial pH of 8.02.

Increasing the flocculant dose in the treatment of thickener overflow improved the turbidity reduction after 1 minute of settling significantly. With the highest investigated dose of 56 ppm a turbidity of 3.39 NTU could be reached. The turbidity had further decreased to 2.0 NTU by two minutes of settling and remained there over the rest of the settling time. However, the pH also increased to 10.17 with this dose.

Flotation tailing was treated with higher flocculant doses ranging from 28 ppm to 112 ppm. This range was chosen due to the high initial turbidity of the suspension. Without flocculant the turbidity was 6800 NTU after 1 minute and 1500 NTU after 10 minutes of settling. Even with the lowest tested flocculant dose of 28 ppm this could be lowered to 42.5 NTU after 1 minute and 34.3 NTU after 10 minutes of settling. High flocculant dose of 112 ppm could reduce the turbidity all the way down to 6.53 NTU by 10 minutes of settling time but also resulted in the highest pH of 10.21.

Exhaustive table of flocculation data can be found in supplementary materials S1 Flocculation data.

Scanning electron microscopy (SEM)

SEM images were used to show the interaction between cellulosic flocculants and clay minerals. A selection of SEM images is presented in Figure 6. While the surfaces of the flocs are dominated by clay-like, smooth flakes, some porous cellulosic material can also be seen connecting them and interweaving in between.

Discussion

The motivation for choosing these mineral processing waters was that they contained suspended solid particles that should be settled more effectively to better control them in the industrial site. In case of thickener overflow the particles are fine minerals that would have ideally been already recovered in thickener and now need to be contained. In flotation tailing pond the slow settling may cause issues as the volume of the pond – and subsequently the available settling time – decreases over the lifetime of the pond as it fills with sediment. Based on the results of turbidity measurements, PMCC flocculant would be effective in improving the thickener efficiency or in an additional settling process after the

thickener. It also aided in settling the flotation tailing, making it a viable additive for enabling the settling pond to function at a shorter settling time.

The mechanisms of flocculation with cellulose alkali solutions have not been studied. Based on the SEM images, particularly Fig. 6a and d, it looks like the regenerated cellulose is physically connecting talc particles in the scale of several microns and aiding the formation of flocs of at least 100 μm in diameter. This suggests that the regeneration of cellulose to physically surround and connect the mineral particles could be one of the flocculation mechanisms in play.

The effect of phosphorylation is clearly beneficial on the flocculation ability of cellulose for talc suspensions. This leads us to believe that either the phosphate groups interact directly with talc particles or that they affect the dissolution and regeneration of cellulose in a way that enables higher flocculation efficiency. Even though PC-PMCC had a lower DS than PMCC, they had comparable flocculation efficiency. However, flocculation by bridging is also generally improved as the length of the flocculant polymer is increased. The higher intrinsic viscosity – that correlates with cellulose chain length – that PC-MCC had could therefore be compensating for the lack of phosphate groups. Overall, the phosphorylation DS was at a level expected with this method (Naderi et al. 2016; Eilamo et al. 2026).

Because commercial MCC is primarily made for high purity requirement applications such as food, pharmaceuticals, and cosmetics, it is often expensive. However, we could show that PC-PMCC could equal the performance of PMCC in flocculation. This means that more affordable MCC made from side streams and recycled cellulose can also be used in this application without forfeiting performance. However, it should be noted that polycotton-based MCC may still contain traces of polyester that should be filtered out after the dissolution to avoid releasing polyester microplastics to environment or contaminating process flows. The filtration of cellulose solutions is a well-known process in man-made cellulose fiber industry and should not require major investigation or investment.

Mierczynska-Vasilev et al. (2013) found that different types of starch can be used to improve talc settling and water clarification. Although different talc concentrations, settling setup, and units of clarity/turbidity make comparisons non-trivial, it is fair to say based on their results that starches are limited to 80–95% reductions in suspended solids compared to no flocculant, whereas PMCC used in this study can consistently reach turbidity reduction of > 99.5% (from 1290 NTU to < 5 NTU) with comparable dose of 28 mg/L. Ozkan (2003) reached flocculation power of 90–95% with neutral PAM at pH > 10 with a low dose of only 0.1 mg/L and only 1 minute settling time. Although the talc concentration was only 0.1% this result showcases the effectiveness PAM has in low doses compared to many biobased alternatives.

The combined effect of MgCl_2 and pH adjustment with NaOH seemed to work as a coagulation process for talc. There are many variables that could influence this phenomenon. Firstly, it is known that talc suspension stability is affected by pH and presence of cations. Secondly, magnesium acts as a coagulant as pH is raised, aiding in particle size growth. This also makes magnesium more broadly a synergistic pairing with alkali dissolved cellulosic flocculants in high pH coagulation-flocculation systems, at least whenever pH is raised above 9.5 where magnesium hydroxide precipitation

approximately begins (Semerjian and Ayoub 2003). Magnesium has also been considered an attractive coagulation chemical compared to more broadly used iron and aluminum coagulants as it is recyclable which can reduce the chemical costs and problems with sludge disposal (Li et al. 2016). In treatment of thickener overflow and flotation tailing from an industrial mineral mine magnesium addition did not aid in flocculation in preliminary tests and was therefore not studied further. It is likely that the soluble cations that are readily present in these waters can produce similar effect when flocculant is added.

When doing separation processes such as the flocculant assisted solid-liquid separation done in this study, one should always consider the resulting fractions. In this case we produce liquid fractions with significantly less suspended solids compared to no flocculant addition in all waters investigated. However, we are introducing NaOH in the water with our flocculant polymer. This results in an optimization problem for the flocculant dose: increasing the dose improves turbidity reduction and settling rate but also increases the pH and therefore likely increases the consumption of neutralizing chemical later in the process. With a 28-ppm flocculant dose about 350 μ L of 30% HCl is needed per liter of treated water to counteract the pH change, or more broadly, for each liter of flocculant approximately 250 ml of 30% HCl is needed.

By using a cellulosic flocculant, we are only introducing a safe, biodegradable polymer to the sedimented fraction. This means that potential applications for the floc are largely determined by the content of the process water and not limited by the flocculant addition. Many white clays and minerals such as talc, kaolinite, and calcium carbonate are used as filler material in papermaking, and hybrid fillers with a polymer as a binder have been shown to have good properties in paper applications (Kuusisto and Maloney 2017; Kuusisto 2018; Pedrosa et al. 2022). The flocs seen in this study from talc and mine thickener overflow could have potential in this type of application in paper products due to their white color, and flocs from flotation tailing could be potentially used with unbleached pulp products such as cardboard. Of course, a more thorough investigation into the chemical content and process suitability of the flocs as well as the logistics of using side streams from mining industry in a paper mill is needed to assess the viability of this concept.

Conclusion

MCC and PMCC dissolved in alkali were used to improve the separation of talc from synthetic wastewater and clay from industrial mine process waters. This is a step towards showing that flocculants based on alkali dissolved cellulose are not limited to laboratory work on synthetic wastewaters with carefully selected contaminants. The effectiveness of a PMCC flocculant was similar whether it was manufactured from textile waste or commercial MCC. Manufacturing flocculants from cheaper cellulose sources can make them more economically viable. The flocs produced with a cellulosic flocculant are free of synthetic polymers and aluminum hydroxide sludge enabling more degrees of freedom in their disposal or utilization.

Declarations

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Competing interests

Tomi Eilamo has no relevant financial or non-financial interests to disclose.

Olli Dahl is a shareholder in Nordic Bioproducts Group.

Author contributions

Tomi Eilamo: Writing – review & editing, Writing – original draft, Visualization, Validation, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. Olli Dahl: Supervision, Funding acquisition, Conceptualization.

Ethics and Consent to Participate declarations: not applicable

Consent to Publish declaration: not applicable

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Figures

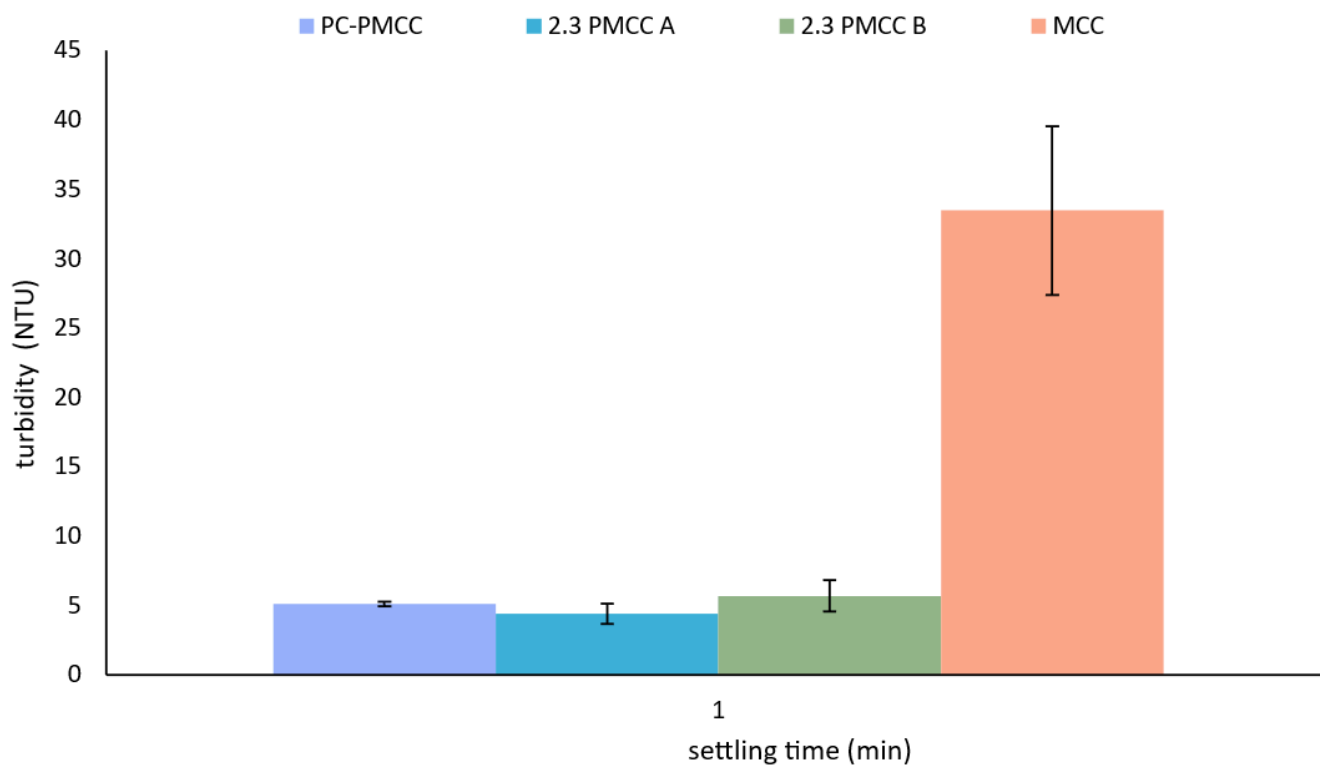


Figure 1

Turbidities of 0.5% talc suspensions treated with 2 mM of MgCl_2 and 28 ppm of flocculant after 1 minute of settling

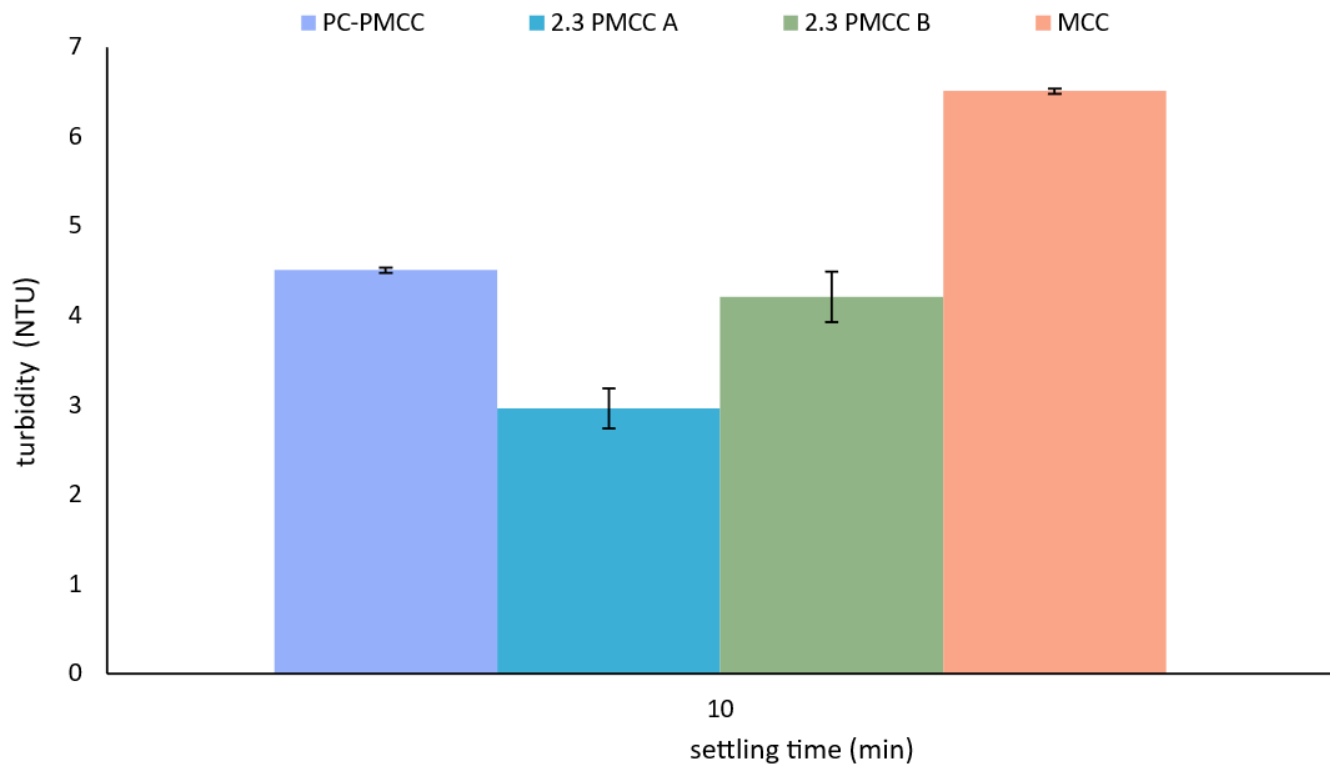


Figure 2

Turbidities of 0.5% talc suspensions treated with 2 mM of MgCl_2 and 28 ppm of flocculant after 10 minutes of settling

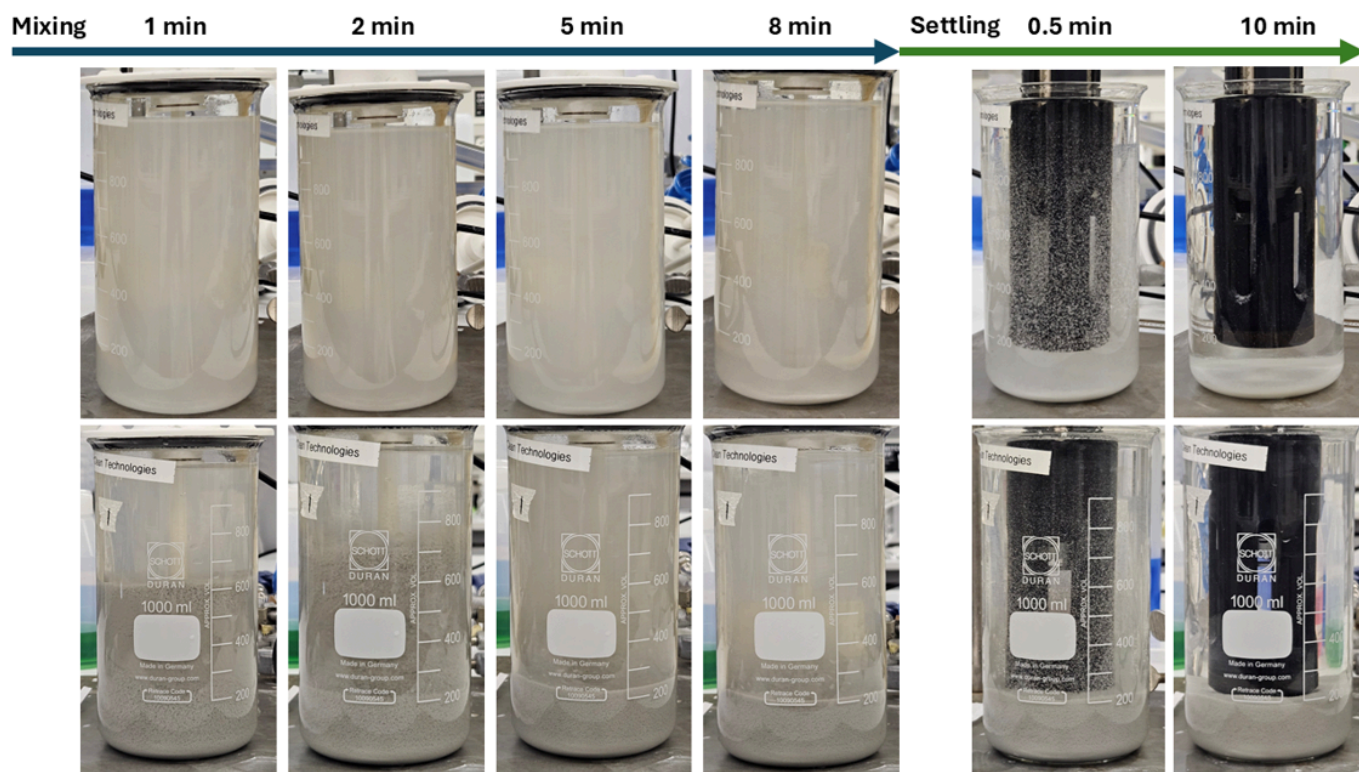


Figure 3

Thickener overflow (top) and tailings pond flow (bottom) from an industrial mineral mine treated with 28 ppm and 84 ppm of PMCC flocculant, respectively

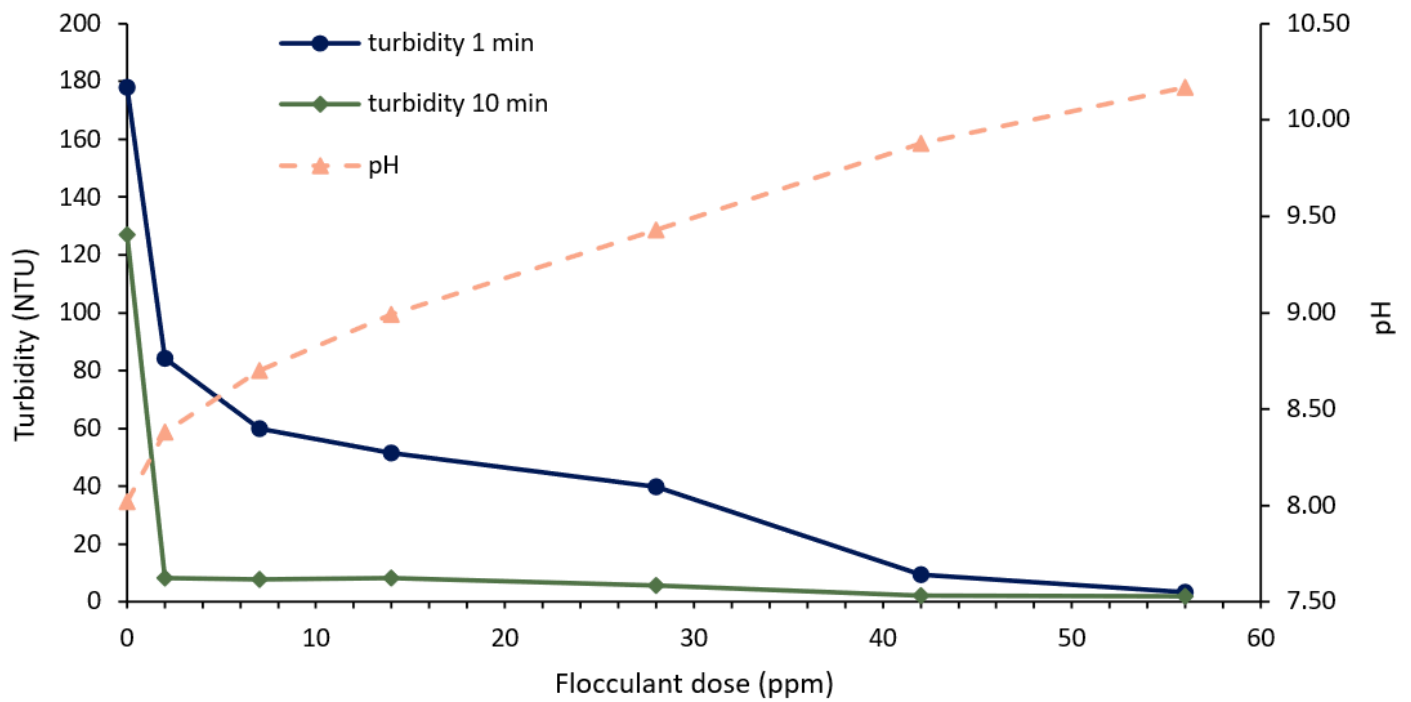


Figure 4

Turbidity after 1 and 10 minutes of settling and pH of thickener overflow after treatment with different PMCC flocculant doses

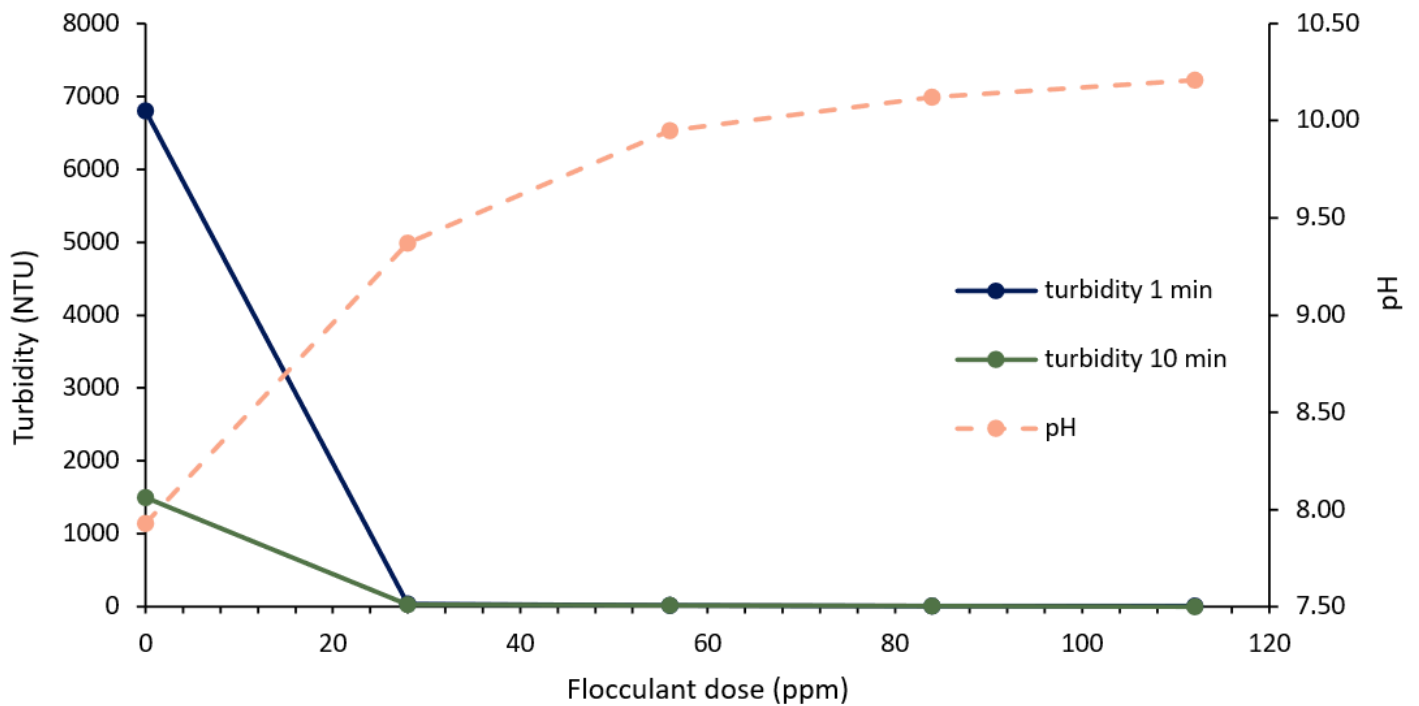


Figure 5

Turbidity after 1 and 10 minutes of settling and pH of flotation tailing after treatment with different PMCC flocculant doses

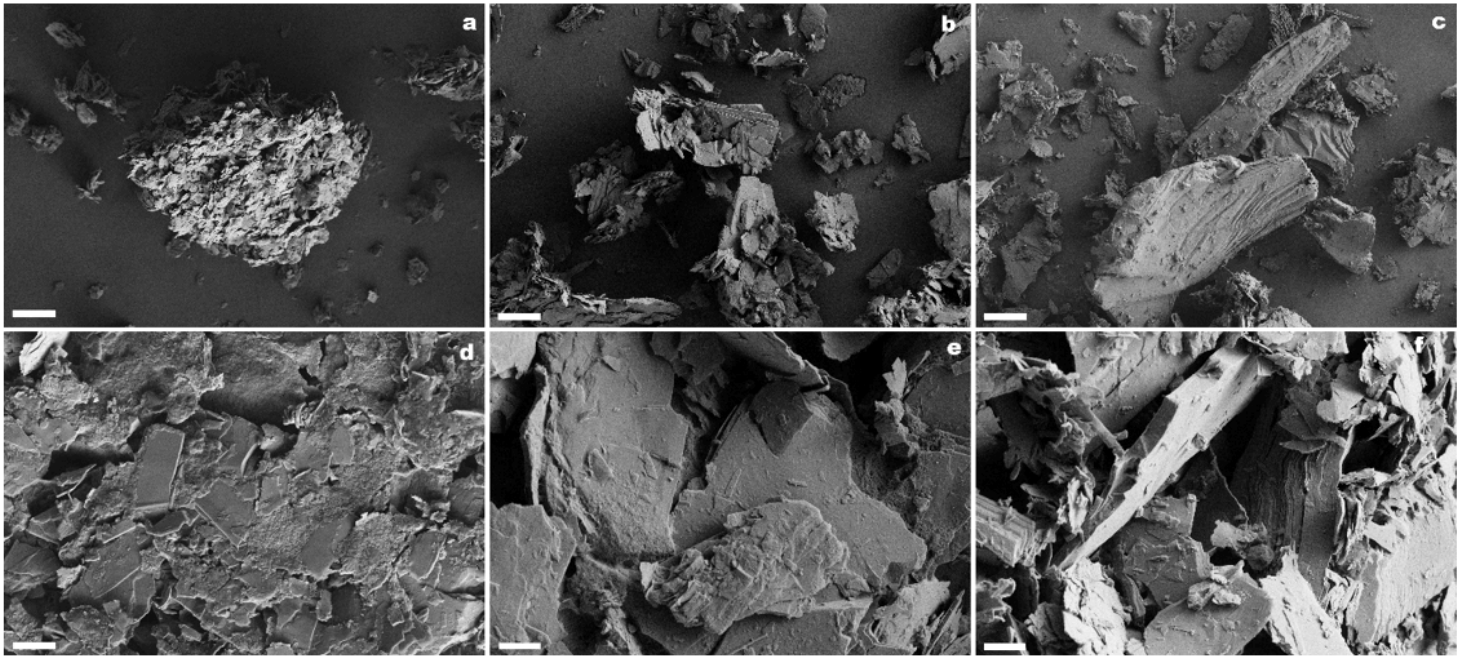


Figure 6

SEM images of sedimented talc (a,d), thickener overflow (b,e), and flotation tailing (c,f) after flocculation with PMCC. Smooth, flake-like structures are talc or other clays, whereas the porous surfaces are PMCC. Scale bars are 20 µm for the top row and 2 µm for the bottom row.

Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- [S1Flocculationdata.xlsx](#)