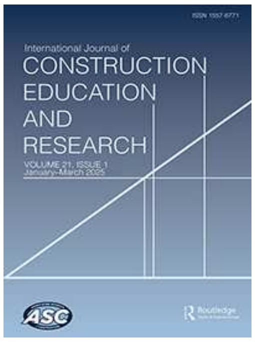

International Journal of Construction Education and Research

ISSN: (Print) (Online) Journal homepage: www.tandfonline.com/journals/uice20

Case Study of Bio-Based Coagulation and Flocculation Systems to Treat Contaminants in Concrete Washout Water

Philip S. McMichael, Maria C. Iglesias, Jacob Johnston, Paul Holley & Maria S. Peresin

INTERNATIONAL JOURNAL OF CONSTRUCTION EDUCATION AND RESEARCH
https://doi.org/10.1080/15578771.2024.2389832

Case Study of Bio-Based Coagulation and Flocculation Systems to Treat Contaminants in Concrete Washout Water

Philip S. McMichael[a], Maria C. Iglesias[a], Jacob Johnston[b], Paul Holley[b], and Maria S. Peresin[a]

[a]Sustainable Bio-Based Materials Laboratory, Forest Products Development Center, College of Forestry, Wildlife and Environment, Auburn University, Auburn, Alabama, USA; [b]McWhorter School of Building Science, Auburn University, Auburn, Alabama, USA

ABSTRACT

Concrete washout water is regularly generated in large volumes at construction sites across the world, often by the clean-up process for concrete pumps. This washout water is highly alkaline and contains high levels of heavy metals and solids. To treat suspended solids, it is often flocculated using polymeric flocculants or bentonite clay. In this pilot study, the potential use of a bio-based polysaccharide, citrus pectin, was investigated as an alternative flocculant and neutralizing agent. Pectin derived from orange peels was coupled with citric acid to test for efficacy in neutralization and flocculation. A variety of acid and flocculant combinations were analyzed through the measurement of suspended solids, dissolved solids, pH, and turbidity. Compared to bentonite, citrus pectin was found to have comparable flocculating capabilities, though citric acid was found to inhibit flocculation for both materials. The results demonstrate that pectin could serve as a bio-based alternative to existing commercial concrete washout water flocculants, an important distinction and addition to previous studies in the body of knowledge that did not utilize the much more turbid and solid-heavy concrete washout water. Such potential solutions could have a meaningful impact on improving jobsite logistics, time, and expense with respect to compliance and sustainability.

KEYWORDS

Cement; concrete washout water; flocculant; alkalinity; pectin

Introduction

Worldwide, concrete is the most widely used construction material (Mitchell Crow, 2008). In 2022, Portland cement production in the United States achieved 95 million tons, indicating an increase from 2021 (93 million tons), and above pre-pandemic levels (87.6 million tons in 2019) (U.S. Geological Survey, 2023). Concrete for traditional construction purposes is composed of coarse aggregate, slag, cement, water, and additives. These elements are mixed together at concrete batching plants, or on-site. Concrete is then stored and transported to construction sites via concrete-mixing trucks (colloquially called cement trucks). The United States Environmental Protection Agency (EPA) prohibits the discharge of wastewater from washout of concrete, unless managed by appropriate controls (EPA MS4 Permit Improvement Guide, 2010). Such regulations came into existence with

CONTACT Paul Holley hollepw@auburn.edu McWhorter School of Building Science, Auburn University, 118 M. Miller Gorrie Center, Auburn, AL 36849, USA

Supplemental data for this article can be accessed online at https://doi.org/10.1080/15578771.2024.2389832.

the United States Clean Water Act of 1972. Concrete washout water is highly alkaline, due largely to the partial dissolution and suspension of calcium hydroxide in the water mix, from limestone used in cement production (Street & Holley, 2019). Concrete washout water contains high levels of chlorides and heavy metals, derived from its cement, slag, and coarse aggregate components, as well as its additives, which can include plasticizers, chloride-based accelerators, water reducers, and air entrainers. Due to the presence of these components, untreated concrete washout water has very high Total Suspended Solids (TSS) and Total Dissolved Solids (TDS) contents. Proper on-site handling of this wastewater presents challenges and costs, with impact on site utilization, pour cycle timing, and in many cases – expensive 3rd party services. Evaporation ponds take up valuable space on the project site, come with maintenance issues and expense, and perhaps most importantly their efficacy is weather-dependent.

As concerns have grown over the environmental effects of wastewater discharge from a variety of sources, construction site discharges have come under increasing scrutiny. In recent years, state and municipal governments have begun more intensive efforts to identify and fine construction sites that fail to safely dispose of their concrete washout water. The Alabama Department of Environmental Management (2019) lists specific standards for discharge of effluents, with a specific focus on pH (between 6 and 8.5), TSS (no greater than 50 mg/L), total residual chlorine (below 0.019 mg/L), and chlorides (below 860 mg/L). Concrete washout water may be removed from construction sites to be filtrated and safely disposed of in designated facilities, but such processes are expensive and time consuming (Street & Holley, 2019). Materials like bentonite and polymeric flocculants like polyaluminum chloride (PAC) and polyacrylamide (PAM) are used to flocculate undesirable particulates from the washout water mix, allowing for easier separation and disposal. While bentonite can be an effective flocculant, its production via mining operations is energy intensive and exists primarily as large surface-mining pits, which generate various waste streams that must be dealt with (Alladustov & Imamnazarov, 2021). PAM is derived from polyethylene, a synthetic fossil fuel, and PAC is processed by the reaction of aluminum with chlorine, both energy intensive and inherently unsustainable processes. Both, residual unbound PAC and PAM can pose potential toxicity risks to surrounding aquatic environments (Council, 2004), and both PAC and PAM have been shown to have detrimental effects on human health, and can be readily transported from wastewater streams into groundwater supplies (Krupińska, 2020; Tepe & Çebi, 2019).

It can be generally stated that the effectiveness of a given flocculating material is highly dependent on the composition of the wastewater or washout water that is being treated. The more unique components and variables that make up the washout water, the greater the amount of unique chemical and morphological interactions that will occur between the flocculating material and the suspension. The temperature and pH of wastewater can greatly alter the effectiveness of flocculation, and can inhibit settling, depending on suspension composition. When concrete washout water is properly treated, carbon dioxide dissolution is the most popular method for effectively neutralizing solutions, as the process produces a relatively weak acid (carbonic acid) that can be accurately modulated in dosage to prevent overdosing and acidification, which is a concern with some stronger acids.

An environmentally friendly, sustainable, and bio-based flocculant may serve as a replacement for existing commercial flocculants, if it rapidly and sufficiently separates suspended solid particulates, which are the source of the majority of the washout water contamination. Previous studies have explored the potential of various biobased flocculants for wastewater flocculation applications, including nanocelluloses and pectins. Chen et al. (2016) found that 2,3,6-tricarboxylate cellulose effectively flocculated kaolin clay suspensions, and effectively reduced the turbidity of the solutions. Morantes et al. (2019) utilized cationically charged cellulose nanocrystals (CNC) to effectively reduce the turbidity of a wastewater-mimicking silica suspension by 99.7% but found that unmodified CNC had no effect on turbidity. Wang et al. (2019) grafted methacryloxyethyltrimethyl ammonium chloride and acrylic acid onto microcrystalline cellulose and was able to reduce the turbidity of the kaolin clay suspension by 99.82%. Ibarra-Rodríguez et al. (2017) utilized pectins extracted from nopal (prickly pear cactus) to flocculate a variety of metallic ions in synthetic wastewater and found that a low dose of 0.019 mg/L could effectively remove 99% of the metal ions. Ho et al. (2010) tested pectin's ability to flocculate synthetic wastewater and found that it was most effective at a pH of 3, and compared with PAM, it was deemed an effective flocculator, even at low concentrations. Buenaño et al. (2019) tested pectin derived from plantain peels, orange peels, and tamarind seeds for its ability to purify raw natural water with an initial low turbidity. Only when combined with aluminum salts did the pectins demonstrate any flocculating activity. While these studies all looked at use of bio-derived materials for flocculation of inorganic particulates in water systems, many studies utilized chemically modified biomaterials that are presently unscalable for industrial applications, or treated solutions that were comprised of one or few components, differing significantly from the complex chemical and material composition of concrete washout water. While Buenaño's work showed promise with the use of fruit pectins, their study used only raw natural water, as opposed to concrete washout water. Due to the unique nature of washout water composition, a study designed with potential for scalability in mind was designed based on insights gathered from this prior literature.

In this study, we sought to analyze the potential for biobased materials, with a focus on pectins, to serve as flocculants for concrete washout water as a potential option to improve jobsite logistics and compliance by addressing both contamination by solids as well as alkalinity. Doing so with the use of naturally occurring materials presented the potential for a desirable option that could not only favorably impact the use of project site space and expense, but also in a sustainable manner.

Pectin is a complex polysaccharide that can be found in the cell walls of plants, and commercially, is often derived from citrus plants like oranges. Citric acid is a weak acid that can be found naturally occurring in citrus fruits, especially lemons and lime, and also in oranges and grapefruits. The potential to use an agricultural waste source like orange peel residues for both its pectin and citric acid contents was a particularly intriguing aspect of this research. Pectin has not been tested as a flocculant for concrete wash water in any form. Unlike existing commercial flocculants (i.e. bentonite, PAC, and PAM), pectin can be directly extracted from sustainable agricultural operations, as a waste product in the form of citrus fruit peels, rather than competing with existing end product streams.

Materials and methods

Batch production

A crucial aspect of this research was mimicking the construction job-site environment on a laboratory scale. A custom-built concrete hopper was used, along with an electric concrete mixer, and a pressurized water sprayer was used to wash down the hopper. Concrete ingredients were mixed in the following order: coarse aggregate, then sand, then slag, cement, and additives. The material used was based on a standard concrete mix used in Birmingham, Alabama. Specific quantities and ratios of materials were based off of typical standards used in central Alabama. The standard batch totaled 33.171 kg, including 3.79 kg of Type I/II cement with a specific gravity of 3.15, 10.03 kg of natural sand with a specific gravity of 2.63, 16.09 kg of ¾”-1” limestone aggregate with a specific gravity of 2.78, .95 kg of GGBFS slag with a specific gravity of 2.8, and 2.31 kg of potable water from the local municipal supply. The four additives used in each batch included 1.643 ml of air entrainer (MBA-690), 15.334 ml of super plasticizer (Glenium 7920), 9.31 ml of water reducing agent (Pozzolith 322 N), and 43.813 ml of chloride-based accelerator (HE 122). Air entrainers are used in concrete to ensure a certain amount of entrapped air remains in the mixture, which improves structural integrity during freeze/thaw conditions. Plasticizers are used to assist in flow efficiency during concrete application and processing. Water reducers are used to decrease the amount of water needed to achieve the necessary concrete consistency. Chloride-based accelerators assist in reducing concrete set times by quickening chemical reactions. Quantities of admix components used in laboratory batches are shown in Table 1. These volumes remained consistent over the course of testing.

Concrete ingredients were mixed outdoors with water and chemical additives in a concrete mixer and then poured into the concrete hopper once the mix was visually homogenous. The concrete was released from the hopper into a circular plastic container, which was then rinsed down using a pressurized water sprayer (Figure 1). Once the entirety of the 4.206 l of rinsing water was used, the concrete and washout water was left to sit for 10 min. After 10 min, 3.5 l of washout water was extracted using a vacuum pump, ensuring that the extracted hose was never submerged deeper than 1.5 cm below the washout water level. The sample was collected into a 4-l Erlenmeyer flask and transferred into a HDPE bucket for storage in a cold room when not used for testing purposes. All testing was carried out within 7 days of batch production, and certain batch testing that was carried out within 24 h of batch production has been noted.

Table 1. Volumes of admix components used in batch production

Material	Amount	Unit
Cement I/II	3.788	kg
Slag/ash	0.949	kg
Sand	10.029	kg
Stone (coarse aggregate)	16.094	kg
Air entrainer	1.643	mL
Water reducer	9.310	mL
Super plasticizer	15.334	mL
Accelerator	43.813	mL
Mixing water	2.310	L
Rinsing water	4.206	L

Figure 1. Procedure of concrete batch production – ingredient mixing, followed by pour into hopper, followed by release into circular plastic container and rinsing.

Characterization

For the purposes of this research, pH, total suspended solids (TSS), total dissolved solids (TDS), and turbidity were analyzed. pH measurements were carried out in accordance with EPA NPDES Method 150.1, "pH (Electrometric)" (1982). Measurements were performed 15 times for each unique sample, at room temperature, and averaged. A SympHony Multiparameter B30PCI (VWR) pH meter was used. Total dissolved solids was measured in accordance with standard 2540 C, "Total Dissolved Solids Dried at 180 C," from Standard Methods for the Examination of Water and Wastewater, 23rd edition, co-published by the American Public Health Association (2017a), American Water Works Association, and Water Environment Federation 15. Total suspended solids was measured in accordance with standard 2540D, "Total Suspended Solids Dried at 103–105 C," from the same standard book (American Public Health Association, 2017b). Both TDS and TSS were measured in triplicate for each sample. Turbidity was measured based on standard 2130B, "Nephelometric Method," from the same standard book. A VWR portable turbidity meter, model 76152030, was used. For each sample, turbidity was measured in one-minute intervals for 20 min, in duplicate.

Flocculants

Citric acid monohydrate was purchased from VWR, and bentonite and citrus pectin, in dry powder form, were purchased from Alfa Aesar. All flocculants were added at concentrations of 200 mg/L. When citric acid was used, a small portion of the sample was initially set aside to perform a titration with citric acid monohydrate at incremental concentrations, to determine what amount of citric acid would yield pH values within the acceptable range between 6.0 and 8.5. A variety of combinations involving wait times, stirring times, and flocculant/acid sequences of addition were performed, in order to gain a better understanding of which procedure would yield preferable and jobsite-reproducible results. Figure 2 gives a detailed visual representation of each flocculation and acid addition method used, with each sample name shown in bold.

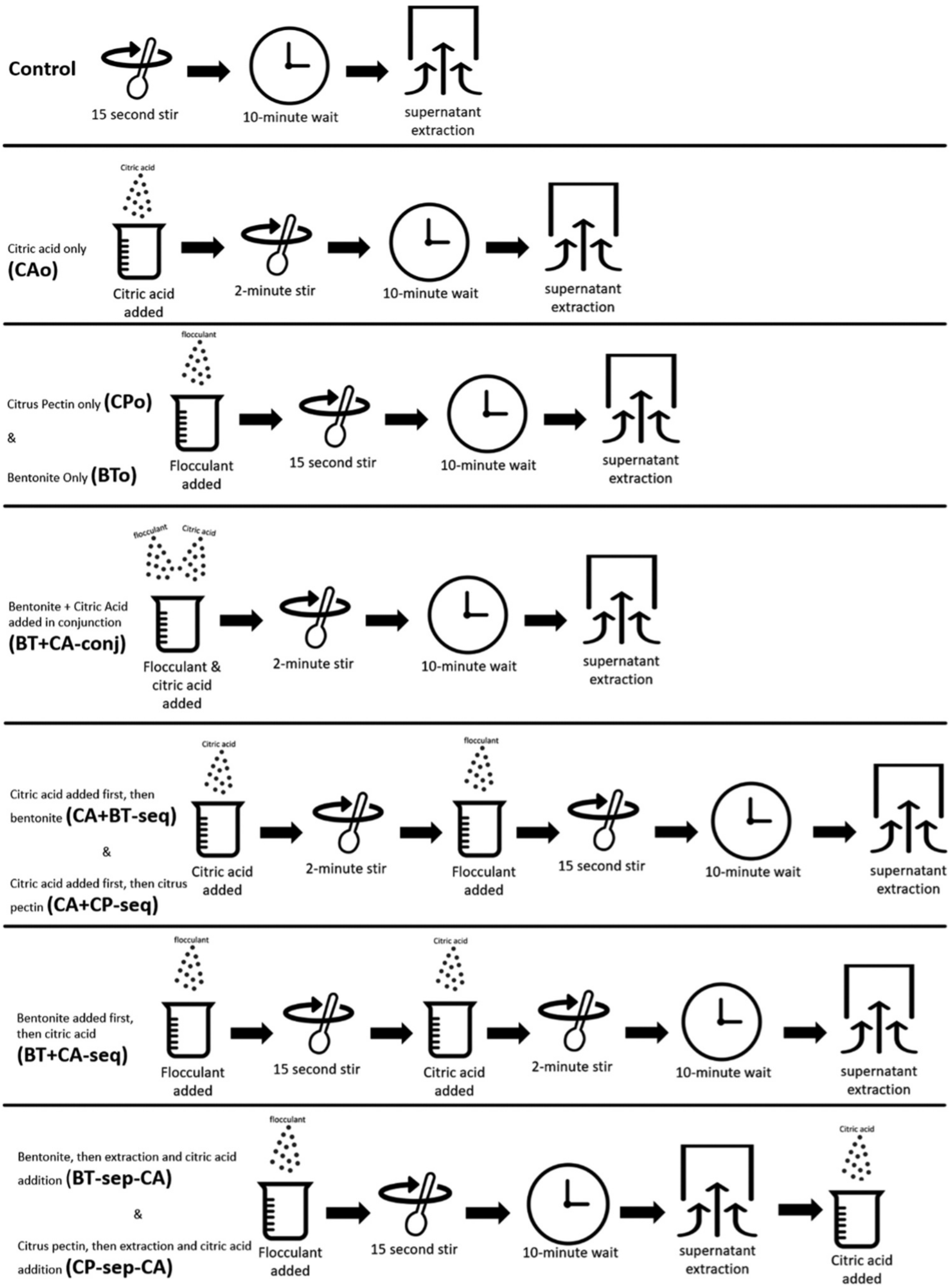

Figure 2. Procedures of flocculant and/or acid addition.

"CA" stands for citric acid, in a concentration selected based on the unique pH/acid concentration curve calculated for each batch. "CP" stands for citrus pectin, added in a concentration of 200 mg/L, and "BT" stands for bentonite, added in a concentration of 200 mg/L. For each unique sample, 500 mL of washout water was initially used, with 350 mL of supernatant being extracted for testing. pH, TSS, TDS, and turbidity measurements were performed on the extracted supernatant. For samples BT-sep-CA and CP-sep-CA, citric acid was added in increments of 100 mg/L, until a pH below 8.5 was reached, as a pH/acid concentration curve could not be produced due to limited samples. For the control sample data, an average of three control samples from three batches was taken to ensure accurate reference data.

Results

Figure 3 shows the results of total suspended solids and total dissolved solids, measured in milligrams of solids per liter of washout water, for all ten unique samples. While TDS was generally variable among all samples, when citric acid was added to the washout water before supernatant extraction, TSS content increased dramatically. This was noted to be the case regardless of the order of flocculant and acid addition or wait times preceding supernatant extraction.

As the TSS content of concrete washout water is a measured metric by the Alabama Department of Environmental management, special focus should be placed on samples that yielded the lowest TSS contents. Figure 4 highlights the six samples that demonstrated the lowest TSS contents. Citrus pectin alone (CPo) and bentonite alone (BTo) both demonstrated clear flocculating abilities. However, reduction of pH is a critical aspect of preparing washout water for safe disposal, and citric acid demonstrated an undesirable propensity to prevent settling of the suspended solids in the washout water solution. A two-step process, beginning with the addition of flocculant, followed by supernatant extraction, concluded by pH neutralization, yields a washout water with a TSS value significantly below the regulated maximum limit. Citrus pectin demonstrated superior flocculating abilities to bentonite without supernatant extraction or acid

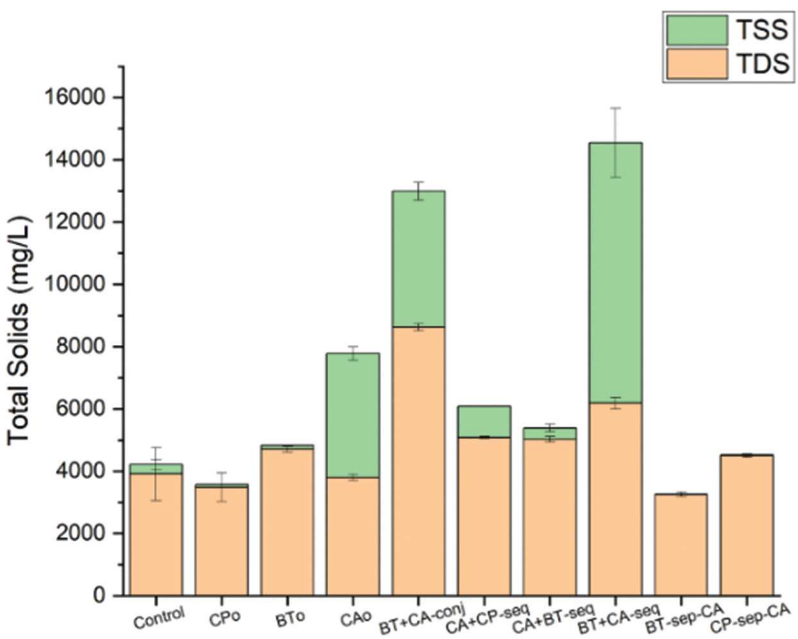

Figure 3. Total suspended and total dissolved solids for all samples.

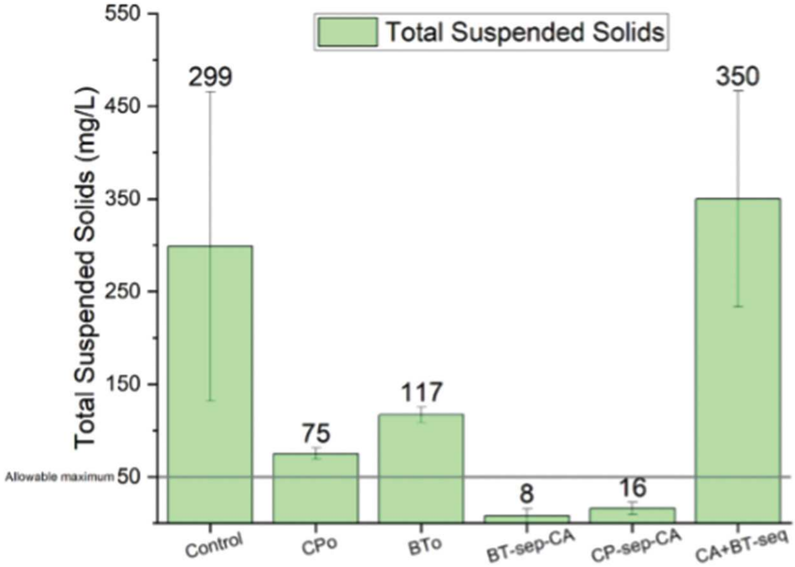

Figure 4. Total suspended solids for six samples with lowest TSS values.

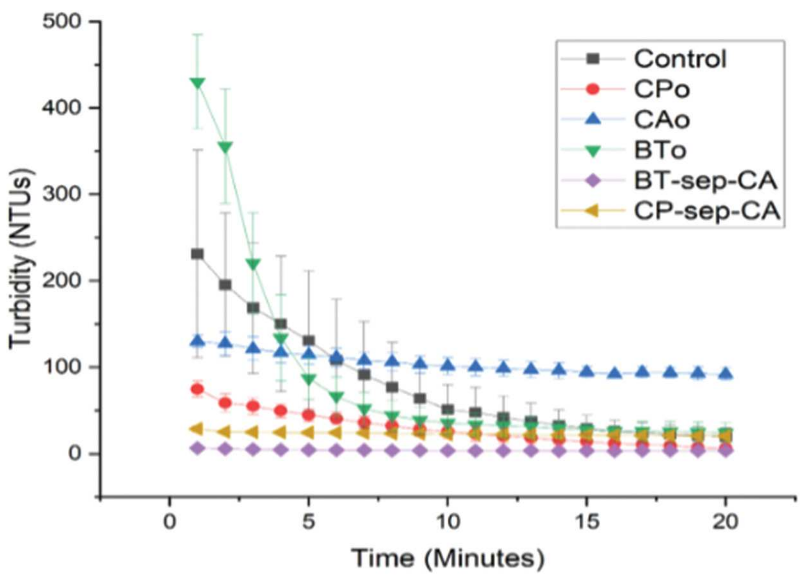

Figure 5. Turbidity readings for best-performing six samples.

addition, but interestingly, bentonite performed better with the two-step process. Such an occurrence may be attributed to the slight variability inherent in the batching process.

Turbidity measurements served as an effective method for understanding the flocculating ability of the materials and methods utilized, as well as the sample settling behavior over time. A more rapidly settling suspension and rapidly flocculating material

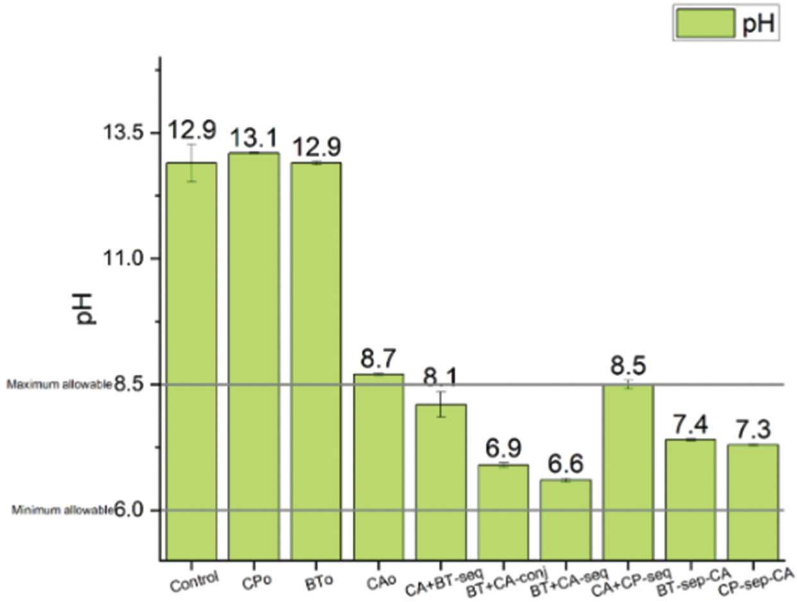

Figure 6. pH readings for all samples.

is preferable on a construction jobsite, reducing wait times and improving efficiency. Figure 5 shows turbidity readings for six samples, over a period of 20 min, measured at 1-minute intervals.

Samples BT+CA-conj, CA+BT-seq, CA+CP-seq, and BT+CA-seq were excluded from Figure 5 due to turbidity readings consistently greater than 300 NTUs, and in the case of BT +CA conj and BT+CA-seq, readings greater than the upper limit measurable by our turbidimeter (>2000 NTUs). Turbidity measurements over time highlighted how settling alone can have a significant effect on turbidity, as at 20 min, our control sample had a similarly low turbidity as bentonite (BTo) and citrus pectin (CPo) alone. However, the rapid flocculating capabilities of citrus pectin were clearly demonstrated by its initial low turbidity measurements. Interestingly, BTo had an initially high turbidity, but decreased rapidly and surpassed the control sample within 4 min. As expected, BT-sep-CA and CP-sep-CA, samples that underwent supernatant extraction before neutralization, also demonstrated low initial turbidity readings. Citric acid alone (CAo) further demonstrated that water acidification works to prevent suspension settling, as only a minor decrease in turbidity was observed over the 20-min period.

pH measurements for all samples are shown in Figure 6. Since each batch required a varying amount of citric acid (ranging from 4.5 g/L to nearly 10 g/L) to reach a pH below 8.5, it can be surmised that the pH of concrete washout water can be highly variable.

This may be an effect of variations within the concrete batching process, specifically with regard to chemical additive quantities. It was also observed that as citric acid concentration increased, pH decreased, and pH rate of change increased. Such an effect is to be expected, but makes acid over-dosing a concern, and is a clear indicator as to why existing commercial operations utilize finely tunable carbon dioxide dissolution methods. pH was especially sensitive for samples BT-sep-CA and CP-sep-CA, and both samples required less citric acid to reach a pH below 8.5 (1.10 g/L and 1.30 g/L, respectively).

Conclusions

As improper concrete washout water dumping is a finable offense at construction jobsites in many states and municipalities across the United States, a cheap and fast method for separating harmful compounds from safely-disposable water is highly desirable. Current compliant options on construction jobsites are generally limited to either space/logistics-heavy evaporation ponds, or time consuming and expensive treatment processes. Unfortunately, these limited options often result in contractors ignoring the regulation requirements altogether. Identifying an alternative by which they could easily treat concrete washout water on the jobsite to allow for immediate discharge in a compliant manner could improve efficiency in clean-up on pour days, and could also significantly reduce or eliminate the need for evaporation ponds that usurp valuable space on the project site.

The concrete batching process can be relatively variable, which was an inherent limitation of our study, and a variety of flocculant addition and washout water extraction methods were tested to better understand what effects sit time and stirring time had on flocculation capabilities. While both materials demonstrated clear flocculating abilities, neither had the capacity to affect pH of the highly alkaline washout water, as such, a biomass-derived acid, citric acid, was utilized. It was further shown that washout water suspension stability and settling capabilities may be pH-dependent, and a two-step process may be necessary to both remove suspended solids and reduce pH to an acceptable level. Whether such a multi-step solution requiring assessment and quantification of additives would be practical to implement by tradesmen and equipment operators remains to be seen, and represents an opportunity for additional research.

Future research

Future research on pectin-based flocculation systems may benefit from a better understanding of the composition of the TDS and TSS components of the washout water, and how these components may act as stabilizers and pH buffers depending on their dissolved or precipitated states. There is also potential for studying the efficacy of this system on a range of mixtures, with particular emphasis on mixture where high levels of TSS may be a greater concern. Nevertheless, the research conducted in this study demonstrated the potential of citrus-derived pectin to act as a flocculant for concrete washout water, with potential for industrial scalability due to pectin's status as a sustainable agricultural waste by-product.

Disclosure statement

No potential conflict of interest was reported by the author(s).

Data availability statement

All data, models, and code that support the findings of this study are available from the corresponding author upon reasonable request.

References

Alabama Department of Environmental Management. (2019). *Discharge and monitoring requirements – existing and temporary facilities*. https://www.adem.alabama.gov/programs/water/waterforms.cnt

Alladustov, U. B., & Imamnazarov, S. N. (2021). Ecological impact on the environment of industrial mining of bentonite clays. *IOP Conference Series: Earth and Environmental Science*, *868*(1), 012048. https://doi.org/10.1088/1755-1315/868/1/012048

American Public Health Association. (2017a). Total dissolved solids dried at 180 C. *Standard Methods for the Examination of Water and Wastewater*. https://doi.org/10.2105/SMWW.2882.030

American Public Health Association. (2017b). Total suspended solids dried at 103–105 C. *Standard Methods for the Examination of Water and Wastewater*. https://doi.org/10.2105/SMWW.2882.030

Buenaño, B., Vera, E., & Aldás, M. B. (2019). Study of coagulating/flocculating characteristics of organic polymers extracted from biowaste for water treatment. *Ingeniería e Investigación*, *39*(1), 24–35.

Chen, K., Lv, W., Chen, W., Wang, Y., Zhang, Y., Zhang, X., & Yao, J. (2016). 2,3,6-tricarboxylate cellulose as a fully biodegradable flocculant: Efficient synthesis and flocculation performance. *Bio Resources*, *11*(3), 5918–5930. https://doi.org/10.15376/biores.11.3.5918-5930

Council, A. R. (2004). *Overview of the effects of residual flocculants on aquatic receiving environments* (Vol. 31). TP226, Technical Publication.

Environmental Protection Agency. (1982). *PH (Electrometric), Method* 150.1. https://www.nemi.gov/methods/method_summary/4685/

Ho, Y. C., Norli, I., Alkarkhi, A. F. M., & Morad, N. (2010). Characterization of biopolymeric flocculant (pectin) and organic synthetic flocculant (PAM): A comparative study on treatment and optimization in kaolin suspension. *Bioresource Technology*, *101*(4), 1166–1174. https://doi.org/10.1016/j.biortech.2009.09.064

Ibarra-Rodríguez, D., Lizardi-Mendoza, J., López-Maldonado, E. A., & Oropeza-Guzmán, M. T. (2017). Capacity of 'nopal' pectin as a dual coagulant-flocculant agent for heavy metals removal. *Chemical Engineering Journal*, *323*, 19–28. https://doi.org/10.1016/j.cej.2017.04.087

Krupińska, I. (2020). Aluminum drinking water treatment residuals and their toxic impact on human health. *Molecules*, *25*(3), 641. https://doi.org/10.3390/molecules25030641

Mitchell Crow, J. (2008). *The concrete conundrum*. Chemistry World. https://www.chemistryworld.com/features/the-concrete-conundrum/3004823.article

Morantes, D., Muñoz, E., Kam, D., & Shoseyov, O. (2019). Highly charged cellulose nanocrystals applied as a water treatment flocculant. *Nanomaterials (Basel, Switzerland)*, *9*(2). https://doi.org/10.3390/nano9020272

Office of Water, Office of Wastewater Management, & Water Permits Divison. (2010, April). *MS4 permit improvement guide (EPA 833-R-10-001)*. U.S. Environmental Protection Agency. https://nepis.epa.gov/Exe/ZyPURL.cgi?Dockey=P100A9N3.txt

Street, P., & Holley, P. (2019). Alkalinity of concrete washout water: A Pilot to determine jobsite conditions for potential neutralization. Proceedings of the Creative Construction Conference, Budapest, Hungary (pp. 619–624). https://doi.org/10.3311/CCC2019-085

Tepe, Y., & Çebi, A. (2019). Acrylamide in environmental water: A review on sources, exposure, and public health risks. *Exposure and Health*, *11*(1), 3–12. https://doi.org/10.1007/s12403-017-0261-y

U.S. Geological Survey. (2023). *Mineral commodity summaries 2023* (Report 2023). National Minerals Information Center. https://doi.org/10.3133/mcs2023

Wang, Z., Huang, W., Yang, G., Liu, Y., & Liu, S. (2019). Preparation of cellulose-base amphoteric flocculant and its application in the treatment of wastewater. *Carbohydrate Polymers*, *215*, 179–188. https://doi.org/10.1016/j.carbpol.2019.03.097