Cellulose in natural flocculant applications: A review

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Abstract. Natural polymeric flocculants have gained popularity in water and wastewater treatment in recent years due to their non-toxicity and biodegradability. Because of its broad availability, renewability, sustainability, and surface modification potential, cellulose is regarded as one of the foundation polymers for flocculant production and modification. The following literature review includes an overview of coagulation-flocculation, which is the process mechanism consisting of colloid destabilization for coagulation, followed by bridging, charge neutralization, and electrostatic patch for flocculation; aspects affecting the coagulation-flocculation performance; as well as the types of coagulants and flocculants that are commonly used. Furthermore, we will go over the physical and chemical properties of flocculants, as well as their usage as a coagulant-aid in the flocculation process following coagulation and as a flocculant in direct flocculation. There is also a discussion of the most recent advances in biopolymers, which are natural materials used to alter biopolymers as flocculants such as chitosan, tannins, starch, and cellulose. Whereas there is a review of the cellulose modifications that have been performed in past research to make it a natural flocculant, the use of ramie cellulose as flocculants has never been carried out to be used as a coagulant-aid and/or flocculant in drinking water and wastewater treatment. Ramie cellulose as backbone of biomaterial composites are expected to be applied as flocculants, have good flocculation performance, and can facilitate sludge handling in water treatment plants and/or wastewater treatment plants.

1. Introduction
Flocculants are typically polymers with long chains that are water soluble. Solids that do not settle are separated from suspended solutions using flocculants [1]. At present, the use of polymer flocculants is more widely used than inorganic metal coagulants to enable and assist the process separation because of its high efficacy, so that synthetic and natural polymer flocculants are becoming prominent nowadays in their application in water and waste treatment due to their inert nature in changing pH, high processing efficiency at low doses, and easy handling [2-4]. Both synthetic polymer flocculants and natural flocculants, on the other hand, have significant drawbacks. The former is deemed non-biodegradable and unfriendly to the environment, whilst the latter is less efficient than synthetic polymers [5-7]. As a result, current research on natural flocculant modification, also known as bio-based flocculants, has been intensively conducted in order to combine the characteristics of synthetic and natural polymers [3, 8], which are presented in this paper. Another factor to consider is that the sludge produced during the clarifying process following coagulation-flocculation is anticipated to be biodegradable and suitable for disposal after simple processing. This might lead to significant savings on maintenance [3, 9].
Cellulose is another viable choice for water and waste treatment due to its vast availability, renewability, durability, and surface modification capabilities. The usage of unmodified cellulose is limited, however, due to cellulose's inability to dissolve in water. The creation of nano-cellulose has been the focus of most of the research on using cellulose as a flocculant by changing its surface. Because nano-cellulose has a large surface area, surface modification is easier [1].

Synthesis of flocculants using synthetic cellulose has been widely carried out [8, 10-11]. Kono (2017) also succeeded in developing the flocculant into a flocculant with 48% degradability within 48 hours using cellulase enzymes. The synthesis of semi-natural cationic flocculants from synthetic cellulose as a backbone which is then grafted with polyacrylamide into copolymers has been carried out by Zhang et al. (2016). Polyacrylamide grafting succeeded in increasing flocculation efficiency compared to cationic flocculants without grafting [12], but polyacrylamide has acrylamide monomer which is known to be a carcinogenic compound. Meanwhile, there have been several studies that synthesize flocculants using natural cellulose [13-15]. Liimatainen et al. (2012) and Suopajärvi (2013) used cellulose extracted from bleached birch (Betula verrucosa and Betula pendula) to produce anionic cellulose. Zhu et al. (2015) modified bamboo pulp cellulose from Phyllostachys heterocycle into bamboo pulp cellulose-g-polyacrylamide which was applied as a coagulant-aid [15].

From previous studies, the cellulose used is synthetic cellulose derived from industry and there has been no research on modifying ramie plant cellulose into natural flocculants. The use of ramie-based biomaterial composites has never been carried out and is expected to be used as a coagulant-aid and/or flocculant in drinking water and wastewater treatment, especially for the formation of flocs from raw water and high-turbidity waste containing organic contaminants, dyes, heavy metals and minerals [2, 3, 16]. Ramie is a textile raw material that may be used to replace cotton. It has an 8-year productive period and can be harvested in 90 days for the first crop, 60 days for the second crop, and so on [17-18]. Ramie fiber is a bast fiber produced from plant bark that is used as a base material for textiles. Meanwhile, cellulose is still present in unused ramie stalks, so they may be reused. As a result, cellulose produced from ramie stem waste has the potential to be used as a foundation material for coagulant-aid and ecologically friendly biodegradable flocculants.

In order to make novel and innovative flocculants, a thorough and comprehensive characterization, also an assessment of flocculation performance are needed. Flocculation performance is evaluated by settling rate, supernatant turbidity, and other methods [1]. Therefore, in this review paper, the explanation is divided into five parts. Those are the overview of flocculation, recent developments of natural flocculants, cellulose as natural flocculants including synthetic and natural cellulose as the base and modification of cationic and anionic cellulose, characterization of flocculants, and lastly the assessment of flocculants performance.

2. Overview of Coagulation-Flocculation
One of the most commonly used processes for solid-liquid separation in water and wastewater treatment is coagulation-flocculation [6]. The addition of coagulants and/or flocculants facilitates the destabilization of colloid, then enabling the agglomeration of suspended and dispersed particles into flocs. Agglomeration is a contact and adhesion process in which dispersed particles are united together by weak physical interaction, which eventually leads to solid-liquid separation and particles formation that are larger than colloid size [19]. This is assisted by the help of polymers with high molecular weight or flocculants, through bridging mechanism [1]. These large particles or clumps will settle and produce clarified supernatants in secondary treatments such as sedimentation [3, 20-21].

The conventional coagulation-flocculation treatment method will be replaced by direct flocculation due to cost and time effectiveness factors. In addition, residues from inorganic coagulants are thought to cause high toxicity factors for some serious illness, such as enclopathy, Alzheimer’s, and neurodegenerative diseases, while some of them are also considered carcinogenic. Meanwhile, the polymer employed in direct flocculation is predicted to function in a wider pH range and with less sludge volume than coagulation-flocculation treatment. This necessitates the use of natural flocculants to substitute inorganic coagulants. A comparison between coagulation-flocculation and direct flocculation
is shown in Table I. Because the floc produced by the bridging process is robust, thick, and dense, direct flocculation yields less sludge volume [2, 3].

**Table 1.** Comparison between coagulation-flocculation and direct flocculation [2, 3].

| Criteria               | Coagulation-Flocculation                                                                 | Direct Flocculation                                                                 |
|------------------------|----------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------|
| Types of chemicals     | Coagulants (inorganic metals) and flocculants (polymers)                                | Polymeric flocculants, usually polyelectrolytes                                     |
| Purpose of application | Water with organic and inorganic contaminants                                           | Water with organic contaminants                                                    |
| Treatment ability      | Suspended and dissolved solids                                                          | Suspended solids and colloidal particles                                            |
| Treatment process      | With pH adjustment                                                                      | Without pH adjustment                                                               |
| Treatment cost         | More expensive: chemicals cost and large sludge treatment                               | Less expensive: one chemical is used and less sludge treatment                       |
| Flocculation Mechanism | Coagulation: charge neutralization                                                      | Charge neutralisation and bridging mechanism occur simultaneously                   |
| Sludge generation      | More sludge is produced, may contain metals and monomer                                 | Less sludge is produced, may contain monomers                                        |

The two primary processes in the flocculation process are the bridging mechanism and charge neutralization. They are typically determined by the size of the polymer and the surface characteristics of the particles in the solution [1, 3]. Electrostatic patches, destabilization processes due to increased Van der Waals tensile forces, decreased electrostatic repulsion, depletion flocculation, displacement flocculation, and other mechanisms in flocculation process are also proposed in several studies [3, 22].

As seen in Figure 1, when the chain of polymer is long enough to expand up to twice the size of its particle’s double layer, the bridging mechanism occurs. This mechanism requires high molecular weight polymers whose length exceeds the Debye length (1/κ) of the particle. Whereas the short polymer chain could not support the bridging mechanism. Likewise, polymer chains that stick evenly to the surface of the particles before they come into contact with other particles will lose their bridging capacity (as can happen with some electrolytes) [1].

**Figure 1.** Bridging mechanism in flocculation [1].

The bulk of suspended particles in surface water and wastewater are hydrophobic and negatively charged. As a result, adding polyelectrolyte cations reduces particle surface charge, therefore lowering electrostatic repulsion. Following that, particle aggregation might occur. Flocculation occurs as a result of a decreasing zeta potential on the surface of particles, and suspended particles form tiny flocs, as illustrated in Figure 2. Optimal flocculation happens when the flocculant dose necessary to neutralize
particles or get the zeta potential near to the isoelectric point is reached. The particles will agglomerate near the isoelectric point owing to the Van de Waals effect, destabilizing the colloidal solution. Overuse of polymers, on the other hand, has the potential to return particle charge and re-disperse colloidal particles [3].

3. Recent Developments of Natural Flocculants

Natural polymers are polymers derived from biomaterials such as plants and animals. Natural polymers have lately piqued the curiosity of numerous researchers working on water and waste treatment. Among the most common natural polymers are cellulose, chitosan, starch, amylpectin, and alginate, which are non-toxic, biodegradable, and inexpensive [1, 23]. This section discusses the development of natural polymer syntheses, including chitosan, starch, and cellulose which function as bio-based flocculants.

Direct flocculation (that is, without the addition of a coagulant) using polyelectrolytes, both cationic and anionic, could be possible to replace inorganic coagulant. Direct flocculation may be divided into two types: single polymer systems and multiple polymer systems. The use of cationic polymers with medium charge densities and high molecular weight in a single polymer system. The cationic polymer is intended to neutralize the negative charge of the particles and then produce floc via a bridging process. In a multiple polymer system, cationic polymers are introduced initially for charge neutralization, followed by anionic polymers with long chains for the bridging mechanism. This results in bigger flocs that settle faster [9].

Chitosan is a derivative of chitin, the second most common polysaccharide on the planet after cellulose, and may be found in the cell walls of invertebrates and some fungi. Chitosan comes from organic materials, can decompose well in the environment, and is cationic polyelectrolyte so that in the water treatment process it is very potential to be used as a natural coagulant [24]. Chitosan has an amine group (NH₂) which is a strong nucleophile which causes chitosan to be used as a multi-functional polyelectrolyte and plays a role in floc formation [12]. Based on the research that has been done, it shows that chitosan can be used as a coagulant that is more effective and efficient than alum, this can be seen from the reduction in water turbidity even with low chitosan concentrations [25]. The coagulation-flocculation process using chitosan can also reduce suspended inorganic and organic particles and dissolved organic matter [26]. The advantages of chitosan as a coagulant are that it is non-toxic, easily biodegradable, and can easily interact with other organic substances such as protein [20].

Because of its renewability, biodegradability, and low cost, starch has garnered interest in water and waste treatment and has been used in industrial wastewater treatment. Starch also one of the most abundant polymers in nature, consisting hydroglucose, amylase, and amylpectin [27]. Application of starch in wastewater treatment mostly uses modified starch with formaldehyde and caustic soda, hence delivering higher efficiency in wastewater treatment. However, both of formaldehyde and caustic soda are hazardous and corrosive. Teh et al. (2014) investigated the efficacy of starch as an unmodified natural coagulant in the palm oil mill wastewater treatment [28]. Therefore, unmodified starch or natural starch is considered the better alternative because it reduces the use of harmful inorganic coagulants [3, 29-30].
4. Cellulose as Natural Flocculant

Cellulose is a biodegradable homopolymer composed of linear polysaccharide chains formed by repeated connections of D-glucose. It has applications in a variety of industries as a raw material for chemical processing. Cellulose may be derived from wood, plants, microorganisms, and animals, as well as seed fibers (cotton), wood fibers, tree bark fibers (flax, hemp, jute, and ramie), grass, bamboo, algae, and bacteria. Because of the abundance of free-OH groups on the chain, cellulose has a good water purification effect, allowing for the effective removal of metal ions and organic debris from water with a strong chelating impact [3].

Because of its low water solubility and chemical reactivity, cellulose has traditionally had limited use as a flocculant. Cellulose modification was performed to address cellulose’s deficiencies of weak water solubility and low chemical reactivity, and carboxymethylation is a traditional and effective chemical modification technique. Related studies regarding the modification of cellulose for use as a flocculant can be seen in Table 2. Water-soluble modified cellulose serves an essential role as a potential replacement for oil-based flocculants. Several cellulose derivatives have been studied and shown to be effective at eliminating suspended particles. There is also growing interest in the development of low-cost biomass (cellulose) absorbers for the treatment of dyestuff-contaminated (decolorized) waste water from various waste water sources. Anionic sodium carboxymethyl-cellulose (CMCNa) produced from agricultural palm oil waste, for example, has been investigated as an ecologically friendly flocculant in combination with aluminum sulphate as a coagulant to lower turbidity in drinking water treatment [30].

Table 2. Recent research on the modification of cellulose as natural flocculants.

| Base Polymer                      | Modification                                                                 | Result                                                                                                                                                                                                 | Ref. |
|----------------------------------|-----------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------|
| Eucalyptus kraft bleached pulp   | Synthesis of cationic cellulose using CHPTAC reagent and synthesis of anionic cellulose using sodium metabisulfite as reagent. | The results showed that the percentage of color removal by CC (cationic cellulose) and ADAC (anionic dialdehyde cellulose) gave a processing efficiency of more than 90%. | 3    |
| Powdered α-Cellulose             | A novel biodegradable flocculant: 2,3,6-tricarboxylate cellulose (TCC)       | For kaolin suspension, TCC shows outstanding flocculation performance.                                                                                                                                 | 8    |
| Hydrophilic natural cotton        | Synthesis of biodegradable natural-based flocculants by sulfonating cotton to produce cellulose sulfate (CS). | Direct flocculation using CS is possible and can reduce the use of chemicals (alum or alum) for the coagulation process.                                                                                 | 10   |
| Powdered Cellulose               | Synthesis of water-soluble quaternized cellulosues.                        | With a maximum degradation of 48 percent within 48 hours using cellulase, quaternized cellulose shows good biodegradability and enabling a biopolymer recovery and reuse. | 11   |
| Microcrystalline cellulose       | Cationic cellulose (CC) is synthesized by a CHPTAC cellulose reaction. Polyacrylamide is then grafted into cationic cellulose-grafted-polycrylamide (CC-g-PAM). | Under acidic and neutral conditions, CC-g-PAM is efficient as a flocculant for the suspension of kaolin, while CC demonstrates improved results under alkaline or alkaline conditions. | 12   |
Bleached birch (Betula pendula) chemical kraft wood pulp

Synthesis of bioflocculants: cationic nanocelluloses, dicarboxylic acid nanocellulose (DCC), sulphonated nanocellulose from kraft pulp (ADAC) and cationic nanocellulose (CDAC).

In the coagulation-flocculation process with ferric as a coagulant, anionic nanocelluloses (DCC and ADAC) demonstrated good efficiency in urban wastewater treatment. Meanwhile, CDAC is successful over a large range of pH and temperature in flocculating kaolin suspension.

Bleached birch (Betula verrucosa and Betula pendula) Combined alum coagulation-flocculation treatment and soluble/nanoparticle anionic dialdehyde cellulose derivatives (ADAC).

The coagulation-flocculation treatment combination is only effective in acidic conditions (pH <5) due to the presence of alum in the coagulation process. ADAC is stable at pH 3-9 and temperature 30-60 oC.

Bamboo pulp cellulose from Phyllostachys heterocyclica

Synthesis of cellulose-g-polyacrylamide bamboo pulp

The best coagulation-flocculation efficiency is given by the combination of Fe$^{3+}$ with BPC-g-PAM.

Date palm rachis

Anionic polyelectrolyte: sodium carboxymethylcellulose (CMCNa).

Comparison of modified flocculants with PAM shows that CMCNa produces 10% better flocculation performance than PAM.

In another research, the anionization of flocculant dicarboxylic acid nanocellulose (DCC) in municipal wastewater was developed and analyzed for its iron sulfate coagulant flocculation properties [13]. Zhang et al. (2015) and Pellizzer (2015) studied the production and homogenous characterisation of polyacrylamide-grafted cationic cellulose flocculants with CHPTAC or 3-chloro-2-hydroxypropyltrimethylammonium chloride as a reagent (2016). Meanwhile, Khiari et al. (2010) and Chen et al. (2016) produced anionic biodegradable flocculants with a carboxymethylation process. Graph copolymerization has been shown to be an effective approach and can improve flocculation ability [6, 8, 31-32].

5. Characterization of Flocculants

In order to determine the physicochemical properties of cellulose and the resulting bio-based flocculants, characterization was carried out. The zeta potential desired in each of the bio-based flocculants or polyelectrolytes is positive for Cationic Cellulose and negative for Anionic Cellulose [8, 12]. In addition, the following analysis need to be carried out.

5.1. FTIR Spectroscopy

FTIR is an instrumentation used to characterize organic compounds by looking at the constituent functional groups [33]. At a certain angle, infrared light is directed at an optical solid crystal with a high refractive index. By measuring the variations in the infrared light emitted internally from the optical solid crystal, attenuated absolute reflection (ATR) works. Every evanescent wave's attenuated energy is transferred back to the IR beam, which then exits from the opposite end of the crystal and is sent to the IR spectrometer detector [3]. The initial step of this analysis is the manufacture of cellulose fiber pellets by mixing it with KBr. The fibers are crushed with KBr until they are homogeneous and become a fine powder. After homogeneity, a number of powders are taken which are then put into a tool that functions to make pellets. Inside the device, the cellulose-KBr fibers are pressurized with a force of about ten tons. The pellets that have been formed are then inserted into the infrared spectrometer (FTIR) [33]. The system then generates an infrared spectrum.
The optimal characterization targets for cationic cellulose are peaks of 1378, 1316, 1157, 1061 and 892 cm\(^{-1}\) for functional cellulose groups; 1479 cm\(^{-1}\) for ammonium metal groups; and 1415 cm\(^{-1}\) for C-N, suggesting that CHPTAC has been successfully incorporated into the cellulose backbone [12]. Anionic cellulose, meanwhile, is at 1603 cm\(^{-1}\) peak to demonstrate the presence of a carboxyl group [8].

5.2. \textit{SEM and EDS Analysis}

For elemental analysis of cellulose, energy dispersive X-ray (EDS) diffraction with SEM (Scanning Electron Microscope) was used. EDS relies on the interaction of multiple X-ray excitation sources and samples. This ability to characterize is primarily due to the basic concept that each element has a unique atomic structure that causes the electromagnetic emission spectrum to have a unique set of peaks [34]. These techniques are widely used for material surface analysis to find out the distribution (mapping) of elements in the tested sample.

5.3. \textit{Molecular Analysis}

The molecular characteristics to be analyzed are molecular size and molecular weight. Dynamic Light Scattering (DLS) for molecule size analysis, better particle aggregate identification, and diluted sample molecular calculation. Meanwhile, molecular weight analysis is conducted using Static Light Scattering (SLS). The range of molecular weight measurements is up to 500 gr/mol for linear polymers and 20,000 gr/mol for spherical polymers and proteins. Proton NMR spectroscopy or also known as 1H NMR spectroscopy, can also be used to determine the chemical structure of molecules and to determine the organic framework of molecules [3].

6. \textit{Performance Assessment of Flocculants}

As described earlier, polyelectrolytes (cationic, anionic, or amphoteric) conduct this suspended particle aggregation process primarily through bridging or charge neutralization mechanisms. The characteristics of the appropriate floc often differ depending on the method of service of the industrial plant. For instance, the secondary process of filtration requires a small, less dense floc while a large, dense floc is required for the secondary process of deposition. The following are physical and chemical factors which are important parameters that must be controlled for the optimization of the flocculation process.

- Physical factors: conditions of particle dispersion, particle size, shear strength and form, duration of agitation, and polymer dosage.
- Chemical factors: polymer composition, polymer charge, charge density, molecular weight of polymer, solution material and pH.

These factors determine the flocculation kinetics, floc growth, and floc morphology. In the meantime, floc deposition rate, sediment volume in the Sludge Volume Index (SVI), the percentage of deposited solids, supernatant turbidity, and the percentage of pollutant removal in the form of processing efficiency or water recovery are general variables to monitor flocculation quality [3].

It is important to use a testing protocol to determine the optimum state for the overall operation, which sequentially preserves and parameter constant when determining the optimum value of a given parameter. A common testing procedure is using jar test as an apparatus, research shown that jar test produces information quickly, economically, applicable to plant design with excellent scale-up with operating plants. Apart from the chemical dosage and pH as the basis criteria, there are some wider criteria on the following list [35], based on three categories:

- colloidal charge reduction (colloid titration);
- settle-ability of flocs (jar test, floc formation time, floc size comparisons, electronic particle counting);
- filterability of flocs (membrane refiltration).
7. Conclusion

Synthesis and modification of novel polymer flocculant that are derived from natural resources become more popular because of the great demand for saving water resources and protecting the environments from pollutants [1]. This review reveals that there an increasing amount of study focusing on cellulose modification as natural flocculant due to its availability and biodegradability. There are also some polymer characterization techniques and performance assessment of flocculants that are needed to be carried out to recognize the quality and optimization of cellulose-based flocculants.

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