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Investigating the characteristic strength of flocs formed from crude and purified Hibiscus extracts in water treatment

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ABSTRACT

The growth, breakage and re-growth of flocs formed using crude and purified seed extracts of Okra (OK), Sabdariffa (SB) and Kenaf (KE) as coagulants and coagulant aids was assessed. The results showed floc size increased from 300 μm when aluminium sulphate (AS) was used as a coagulant to between 696 μm and 722 μm with the addition of 50 mg/l of OK, KE and SB crude samples as coagulant aids. Similarly, an increase in floc size was observed when each of the purified proteins was used as coagulant aid at doses of between 0.123 and 0.74 mg/l. The largest floc sizes of 741 μm, 460 μm and 571 μm were obtained with a 0.123 mg/l dose of purified Okra protein (POP), purified Sabdariffa (PSP) and purified Kenaf (PKP) respectively. Further coagulant aid addition from 0.123 to 0.74 mg/l resulted in a decrease in floc size and strength in POP and PSP. However, an increase in floc strength and reduced d_{50} size was observed in PKP at a dose of 0.74 mg/l. Flocs produced when using purified and crude extract samples as coagulant aids exhibited high recovery factors and strength. However, flocs exhibited greater recovery post-breakage when the extracts were used as a primary coagulant. It was observed that the combination of purified proteins and AS improved floc size, strength and recovery factors. Therefore, the applications of Hibiscus seeds in either crude or purified form increases floc growth, strength, recoverability and can also reduce the cost associated with the import of AS in developing countries.

1. Introduction

For decades, different chemicals have been applied in water treatment to aid the removal of contaminants and harmful substances. Chemical coagulants are added to destabilise the dispersed colloids, with charge neutralisation, adsorption and sweep flocculation being the major mechanisms of action (Duan and Gregory, 2003). Accelerated sedimentation is achieved by aggregating the flocs via slow mixing (flocculation) to form larger macro flocs facilitating removal in a sedimentation tank. However, to achieve satisfactory treatment, flocs must demonstrate sufficient strength so as not to be broken by the turbulent flow field found in the flocculator and clarifier. Thus, the merit of each coagulant is judged based on, *inter alia*, the strength, size and density of the flocs formed. Previous work has observed that smaller flocs are more likely to resist rupture than larger flocs but may pose some challenges during removal compared to bigger flocs (Boller and Blaser, 1998; Jarvis et al., 2005c), as the mechanism and general mode of floc transportation is hampered if the flocs are small in size and so cannot settle effectively. Conversely, it can be argued that smaller and more compact flocs with tighter bonds will resist breakage and settle faster than larger, weaker flocs (Jarvis et al., 2005c). However, it has been reported that the stronger the flocs, the larger they can grow under certain shear conditions (Mühle, 1993). However (Sharp et al., 2006a), revealed that larger flocs can easily break in high turbulent condition, because they are weaker. It can be deduced here that highly compact flocs are generally stronger and smaller in size. Thus, it is challenging to prevent floc breakage under normal plant conditions, particularly in highly turbulent areas; consequently, the regrowth potential of flocs post-rupture is of interest.

Many researchers have investigated floc properties, including floc strength, using different coagulants and under different plant operating conditions. Previous work has monitored floc growth, breakage and re-growth phases after the introduction of high shear rate (Jarvis et al., 2005b; Yu et al., 2012; Xu et al., 2014). Yukselen and Gregory (2004) and Li et al. (2007) observed in their separate studies that AS flocs exhibit irreversible breakage. Conversely, Yu

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et al. (2014) evaluated the property of kaolin-alum flocs at low pH and showed that 100% floc recovery is possible if AS or Kegging polymer Al13 $[AlO_4Al_{12}(OH)_{24}(H_2O)_{12}]^{7+}$ was used as coagulant at acidic pH. Several others workers have also reported the importance of low pH in improving floc strength and recoverability using different chemical coagulants (Cao et al., 2010; Sun et al., 2011). Sharp et al. (2006b) investigated the properties of ferric-NOM flocs and revealed that flocs generated by iron salts are larger and more resistant to breakage than AS flocs, resulting in accelerated settling. Beside AS producing irreversible, smaller and weaker flocs (Yukselen and Gregory, 2004; Sharp et al., 2006b; Li et al., 2007), research has also been undertaken to investigate the relationship between residual aluminium in water and Alzheimers disease (Gauthier et al., 2000; Flaten, 2001). Cost issues associated with the import of coagulants such as AS exacerbate these issues further for developing countries. It is, therefore, imperative to search for alternative natural coagulants and coagulant aids that will lower the cost of water treatment in developing countries and also improve water treatment efficiency. By so doing, the number of deaths resulting from drinking contaminated water supply could be lowered in rural areas and life expectancy increased.

Recently, several natural materials have been studied to assess their coagulation potential in water treatment. Preliminary investigation of some of these natural extracts has so far provided encouraging results for people in developing countries. Naturally-occurring plant extracts including *Moringa oleifera* (MO), *Cactus latifaria*, and *Mustard seeds*, have coagulation capability and can be used in water treatment (Jahn Samia, 1998; Diaz et al., 1999; Bodlund et al., 2014). Similarly, other natural plants, such as Hibiscus, are widely used in many tropical countries because of their nutritional values. Among the many Hibiscus plant species, only OK seed pod has been investigated as a flocculant in the treatment of water and wastewater (Agarwal et al., 2001; De Jesus et al., 2013). Recently, Jones and Bridgeman (2016) have demonstrated the capability of OK seed extract in removing turbidity and bacteria in river water. Additionally, it has been reported that activated carbon derived from KE fibre, another Hibiscus plant could be used to treat water and wastewater with high heavy metal contents (Chowdhury et al., 2012). Conversely, there is no known report on the use of SB seed in either water or wastewater treatment. However, SB extract was found as an effective inhibitor of microbial growth when it was applied on some isolated microbes (Nwaiwu et al., 2012). Most of the reported work has centred on the coagulation activities of the extracts, whereas problems related to floc strength and recovery have not been investigated, despite their importance in the treatment process. Therefore, the aim of this study was to investigate the potential of using Hibiscus plant as a primary coagulant and as a coagulant aid, and to assess the floc characteristics in terms of floc size, strength, and recovery ability.

2. Materials and methods

2.1. Collection and preparation of the seeds

All the seeds used in this study, OK, KE and SB, were obtained from a local market in Nigeria. The seeds were manually prepared by removing the seeds from the capsules and pods to access the seed kernels. The seeds were cleaned by washing with tap water to remove contaminants such as stones, plant debris and dust and then dried in an oven at 60 °C for six hours. The dried seeds were ground into a fine powder for 2 min using a Tema laboratory disc mill. The ground seed powders were then sieved and the powder retained in the 212 μm, and 300 μm sieve sizes was combined and subsequently used in the preparation of the coagulants.

2.2. Chemicals and reagents

Analytical grade sodium chloride, aluminium sulphate and hydrochloric acid (Fisher Scientific, UK), kaolin Fluka-60609, (Sigma-Aldrich, Germany), sodium phosphate monobasic monohydrate (Sigma-Aldrich, Germany), and sodium phosphate dibasic (Sigma-Aldrich, UK) were used in the study. Deionized (DI) water was used to prepare all suspensions and concentration solutions.

2.3. Preparation and extraction of the natural seed coagulants

1 M sodium chloride (NaCl) solution was prepared by dissolving 58.5 g NaCl in 1000 ml of DI water to obtain the required concentration. The crude seed extract (CSEs) were prepared from the ground seed powders by adding 1.0 M NaCl solutions to the seed powder to make 2% (w/v) suspension. The suspension was stirred vigorously using a magnetic stirrer for 15 min at room temperature (19 ± 2 °C). The suspension was then centrifuged at 4500 rpm for 10 min using a Heraeus Megafuge16 (Thermo Scientific, Germany). The suspension was decanted and the residual solids dried in an oven at 50 °C overnight. The weight of the dried solid material was measured to ascertain the amount of seed powder used in making the suspension. The decanted suspension was then filtered through a Whatman No. 42 filter paper. The filtrates were termed crude extracts and were then used as primary coagulant or coagulant aids in a series of jar test experiments.

2 g of AS powder was dissolved in 100 ml of DI water and the suspension rapidly mixed for 15 min, using a magnetic stirrer. This AS coagulant was applied in the jar test experiments to determine the optimum coagulant dose required in the strength test.

2.4. Protein purification and lipid extraction from the seed

The ground seed powders (212 μm–300 μm) were defatted using high-grade hexane in an electro-thermal Soxhlet extractor. 20 g of the seed powder was used during the extraction. For efficient extraction, 2 L of solvent volume (hexane) was used and heated to 60 °C. The process was run continually for 8 h with each complete cycle taking 2–3 min. The residues were dried overnight at room temperature (19 ± 2 °C) and the dried residue was ground into a fine powder using pestle and mortar and was applied in the subsequent purification processes.

2.4.1. Purification by ion exchange column chromatography

A HiTrap Q HP (1 ml) anion column, (GE Healthcare, Sweden) was used for the purification of the protein of interest of the hibiscus plants. The column connected to a pump (Watson-Marlow Breeder pump 323, UK), and the pump head adjusted to a flow rate of 1 ml per minute. The preservatives were washed with 10 ml of DI water, followed by ten column volumes (CV) of 1 M NaCl dissolved in the phosphate buffer. The column was then equilibrated with the phosphate buffer 10 C V before loading the protein. 5 g of the oil-free powder was dissolved in 0.1 M phosphate buffer and mixed thoroughly for one hour using a magnetic stirrer. The mixture was centrifuged at 20,000 rpm at 4 °C for 40 min before decanting the supernatant. The supernatant was injected using a peristaltic pump onto the ion exchange column to separate the protein of interest from the contaminants.

The sample was loaded at a flow rate of 1 ml per minute, where the protein of interest was bound to the Column matrix throughout the loading process. The weakly bound contaminants were washed away with the equilibrating (initial) buffer using 10 C V. The proteins of interest were eluted, beginning with, 0.3, 0.5 and 1.0 M of NaCl–phosphate buffers and the various fractions collected. The collected fractions were analysed for absorbance using a