Journal of Food Engineering 181 (2016) 20–27

Contents lists available at ScienceDirect

Journal of Food Engineering

journal homepage: www.elsevier.com/locate/jfoodeng

The influence of impurities on calcium phosphate floc structure and size in sugar solutions

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A R T I C L E I N F O

Article history:
Received 6 November 2015
Received in revised form
8 February 2016
Accepted 27 February 2016
Available online 3 March 2016

Keywords:
Sugar cane
Calcium phosphate
Floc
Floc structure
Fractal dimension

A B S T R A C T

Settling, dewatering and filtration of flocs are important steps in industry to remove suspended solids and improve subsequent processing of the aqueous system. The influence of non-sucrose impurities (Ca^{2+}, Mg^{2+}, phosphate and aconitic acid) on the calcium phosphate floc structure (scattering exponent, S_f), size and shape were examined in synthetic and authentic sugar juices using X-ray diffraction techniques. In synthetic juices, S_f decreases with increasing phosphate concentration to values where loosely bound and branched flocs are formed. These types of flocs are effective for the removal of suspended colloidal particles. Ca^{2+} and Mg^{2+} ions, and aconitic acid did not affect S_f increasing concentration, although the floc size significantly decreased with increasing aconitic acid concentration, thereby reducing the ability of the flocs to remove particles. In authentic juices, the flocs structures were marginally affected by increasing proportions of non-sucrose impurities. However, optical microscopy indicated the formation of well-formed macro-floc network structures in sugar cane juices containing lower proportions of non-sucrose impurities. These structures are better placed to remove suspended particles in sugar solutions.

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1. Introduction

Extensive studies on the properties of flocs formed during solid/liquid separation of aqueous systems have been evaluated in applications such as water, wastewater, beverage and food industries (Bache and Gregory, 2007; Morton and Murray, 2001; Xiao et al., 2011). Flocs are irregular aggregate structures (*i.e.*, clusters) formed as a result of the destabilisation of suspended particles. Mandelbrot (1977) was the first to use the concept of aggregates possessing self-similar structures to describe flocs. This allows flocs to be recognised as fractal objects, and hence can be characterised by fractal analysis (Meakin, 1998). The technique of small angle laser light scattering (SALLS) was used to examine the internal structure of flocs by determining the fractal dimension (D_f) using the light scattering data obtained from the size distribution measurements. The D_f of aggregates has been widely employed in the literature as it provides useful information on the aggregate structure (East et al., 2014; Greenwood et al., 2007; Logan and Wilkinson, 1990; Selomulya et al., 2001). Loosely held or more open floc structures have low D_f values typically in the range of 1.6–2.4 (Witten and Cates, 1986), while more compact structures have fractal dimensions in the range of 2.5–3.0 (Schaefer, 1989). Loosely bound and highly branched flocs are able to effectively trap suspended particles and the adsorption of colloidal particles onto the surface of the floc network structure (Schaefer, 1989). Compact flocs are unable to effectively trap suspended particles because of reduced porosity. This gives rise to an aqueous solution with high turbidity.

In Australian and many overseas sugar factories, the typical clarification process is simple defecation. As such, the main reaction when lime is added is the formation of calcium phosphate micro-flocs. These micro-flocs are mainly amorphous, and a high molecular weight flocculant typically, poly(acrylamide-co-sodium acrylate) polymer is added to bridge the micro-flocs to form larger flocs. The larger flocs, as they settle, sweep and trap suspended particles thereby reducing the solution turbidity. The floc settling behaviour, and in effect solution shear, is influenced by the characteristics of the floc such as size, density, fractal dimension and porosity (Spicer et al., 1996).

Prior to the clarification process, the sugar cane is harvested with the tops and leaves (*viz.*, trash) extracted by either burning or, most commonly, removed using a mechanical harvester. The

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http://dx.doi.org/10.1016/j.jfoodeng.2016.02.019
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sucrose-rich stalk is then processed in the factory. Considerable amounts of residual fibrous material (*i.e.*, bagasse), left from crushing the stalk to extract the juice, during the milling process, is burnt in the boilers as a low energy source for the processing of sugar cane juice within the factory. The excess bagasse in some factories is used in electricity generation (*i.e.*, co-generation) and sold to the electricity grid. The juice that is expressed from the stalk is relatively easy to clarify, and the calcium phosphate flocs formed during the clarification process are easy to settle, dewater and filter. However, processing the whole sugar cane crop (WC), which includes the trash, increases the amount of available fibre for the production of higher value-added products such as cellulosic ethanol and chemicals. However, the juice expressed from WC is of poor quality, and results not only in poor sugar quality and low sugar yield, but impacts on the operation and performance of the sugar factory (Kent et al., 2010; Saska, 2008). Kent et al. (2010) observed that when WC juice is clarified, the settling rates of the flocs were typically between 10 and 30 cm/min, impacting on productivity. Higher settling rate values of the flocs between 40 and 50 cm/min were consistently obtained with juices that were obtained only from the sugar cane stalk. These settling rates allow good floc compaction and reduce floc carry over to the evaporators.

A number of studies have been carried out to examine the floc structure in sugar solutions. Greenwood et al. (2007) reported the average size and D_f of calcium phosphate flocs formed in synthetic sugar juice solutions. They found that the floc structures were more compact in water than in sugar solutions and similar result was obtained when the pH of sugar solutions were neutralised to pH 7.6 ($D_f = 2.66$) rather than pH 7.8 ($D_f = 2.61$). In a recent study, the authors examined the effect of the non-sucrose impurities starch, Na^+ ions, SiO_2 and phosphate ions on flocs formed in synthetic sugar solutions (Thai et al., 2015). The results from the study showed that the presence of Na^+ ions or SiO_2 and their mixture affected the floc size and structure. As the size of the primary particles is not directly proportional to the wavelength of the incident light, Thai et al. (2015), used the term, “scattering exponent”, S_f instead of D_f to characterise the floc structure as the magnitude of D_f may not provide the true D_f value. There was a linear decrease in S_f, from 2.50 to 2.25 with increasing Na^+ ion content, while the floc size generally increased with increasing Na^+ ions. The drop in S_f was not as significant with the presence of SiO_2 as the result obtained with Na^+ ions. Surprisingly, the floc size was not influenced by phosphate ions, though S_f was highest when 350 mg/kg as P_2O_5 of phosphate ions were added. Increasing the proportion of starch affected the floc structure. The results obtained on the floc size with increasing starch addition showed no discernible trend. As an extension of the study of Thai et al. (2015), an investigation of the effects of the inorganic ions (Ca^{2+}, Mg^{2+} and phosphate ions), and aconitic acid on floc structure and size was carried out in both synthetic and authentic sugar juice solutions using SALLS, X-ray powder diffraction and optical microscopy. These non-sucrose impurities are present at higher concentrations in juices expressed from the sugarcane trash than the juices expressed from only the sugarcane stalk. The information derived from the study was then used to determine the clarification performance of sugar cane juices containing different levels of non-sucrose impurities.

2. Experimental

2.1. Preparation of calcium phosphate flocs formed in synthetic juice

Synthetic sugar cane juice samples were prepared by mixing commercial white sugar (produced from sugar cane) and Milli-Q water (18.2 MΩ cm, Millipore Purification System, Sydney, Australia). Lime saccharate was prepared with 725 g/L of sucrose (Sigma–Aldrich, Castle Hill, NSW, Australia) and 50 g/L of $Ca(OH)_2$ (Chem-Supply, Gillman, SA, Australia). The appropriate amounts of $Ca(OH)_2$, KH_2PO_4 (anhydrous, Chem-Supply), $MgSO_4 \cdot 7H_2O$ (anhydrous, Merck, Kilsyth, VIC, Australia) and aconitic acid (Sigma–Aldrich, Castle Hill, NSW, Australia) were accurately weighed to obtain 500 mL of synthetic juice solutions containing 0–400 ppm of CaO, 0–333 mg/kg of P_2O_5, 0–1000 mg/kg as MgO and 0–1500 mg/kg of aconitic acid. The concentrations of these components were around the standard set of values as shown in Table 1. These components (and their concentrations) are typically found in sugar cane juice streams. Different amounts of phosphate, Ca^{2+}, Mg^{2+}, and aconitic acid were added to the synthetic juice solution prior to liming in order to examine the influence of each component on floc structure and size.

The formation of calcium phosphate flocs was obtained by drop wise addition of freshly prepared lime saccharate to heated synthetic juice solutions (76 ± 2 °C) to pH 7.8. The pH of the juices was measured using a portable pH metre (Hach H 160, Cleveland, CO, USA) with a glass probe (model 93 × 218814, Eutech Instruments, Singapore). The conditions used were to simulate the clarification process in Australian sugar mills. The limed juices were then boiled for 1 min before transferring the solution to a temperature controlled oil bath maintained at 98 ± 2 °C. The treated synthetic juices were subsequently analysed in random order.

2.2. Preparation of calcium phosphate flocs formed in sugar juice solution

Sugar cane juices that were obtained from hand-cut cane located in the fields of Rocky Point, Queensland, Australia during August 2012. One hundred and fifty (150) randomly chosen whole-stalks with green leaves still attached were collected and separated to obtain the stalk and trash components. The cane components were crushed through a two-roll laboratory mill (430 × 220 mm roll, 12.8 mm groove pitch, 12.0 mm groove depth, 4 rpm operating speed and a 7.5 kW motor, Sugar Research Institute, Brisbane, Australia) and the juices were collected after pressing through a 1 mm mesh sieve. All juices obtained were stored at −20 °C and thawed prior to chemical analyses and clarification experiments.

Jar tests were conducted to obtain calcium phosphate flocs formed in several juice samples prepared from stalk with varying amounts of juice expressed from trash. For each of the jars tests, the juice samples (50 mL) were maintained at 76 °C and stirred rapidly at 200 rpm after lime saccharate was added drop-wise until the juice pH reached 7.8. The coagulation process after the addition of lime was conducted under the following conditions: rapid mixing at 200 rpm for 1 min, followed by slow mixing at 40 rpm for 15 min, boiling to remove entrained air and then 5 mg/kg of flocculant (Magnafloc LT27, acrylamide/acrylate co-polymer, $M_W = 23 \times 10^6$ Da, TD Chemicals, Robina, QLD, Australia) was added before 30 min of quiescent settling.

Table 1
Composition of synthetic sugar cane juice samples.

Component	Concentration (mg/kg)	Substance
White sugar	120,000	Sucrose
$Ca(OH)_2$	300	CaO
KH_2PO_4 (dry)	265	P_2O_5
$MgSO_4.7H_2O$	400	MgO
trans-acontic acid	1000	Organic acid

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