Understanding flocculation properties of soil impurities present in the factory sugarcane supply

Hakan Bakir [a], Zhanying Zhang [a], Marek S. Zbik [b], Mark D. Harrison [a], William O.S. Doherty [a, *]

[a] Centre for Tropical Crops and Biocommodities, Queensland University of Technology, GPO Box 2434, Brisbane, QLD, 4001, Australia
[b] Geotechnical Engineering Centre, The University of Queensland, Australia

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ABSTRACT

Soil impurities in sugarcane supplies affect clarification and filtration unit operations in the raw sugar manufacturing process. Juices expressed from the sugarcane plant grown in certain soils display poor clarification and dewatering behaviours. In this study, the salient features of these soils were determined in order to understand why they influence flocculation and settling of juice particles (flocs). The soils were characterized by X-ray powder diffraction, transmission-, transmission x-ray-, and scanning-electron microscopy, and their solution chemistries determined in electrolytes. In general, clay mineral composition, cation exchange capacity, and particle size contributed towards the poor settling characteristics of the soils. Fine particles (i.e., <200 nm) were found in difficult to clarify soil samples and were identified as nano-clays. The structure-building phenomenon of these clays if present in sugar juice are likely to impact on clarification, hindering the juice particles ability to settle despite changing environmental chemistry.

1. Introduction

Settling, dewatering, and filtration are important stages of solid/liquid separation processes in many industries including the mining, water treatment, coal, and sugar industries. In many of these industries, settling is a low energy input process that can operate on a large scale. However, settling is not without its' challenges. For example, Fedorak et al., 2002 reported a settling time for fine soil particles in tailing ponds of oil sand extraction of up to 150 years. In the sugar industry, many factories can experience very slow settling rates (<20 cm/min instead of >30 cm/min) of the flocced particles, low underflow mud solids content (<1–5 wt%), and high clarified juice turbidity (>50 turbidity @ 900 nm) caused by certain soils that come in with the cane supply (Crees et al., 1978; Steindl, 1998; Cronje and Sahadeo, 2014). In some seasons, a 500 t/h Australian sugarcane factory can incur losses in excess of AU$0.5 M/y due to sugar loss to the mud cake, lost time, and increased mud cake transport costs as a consequence of a 5% increase in moisture (Qureshi et al., 2001).

Kaolinite, illite, and smectite, are the most common clay minerals in soils and their presence in aqueous systems is the primary cause that prevents particle aggregation and increased settling speeds. They are sheet silicates, are platy and have a high platelet diameter to thickness ratio (i.e., aspect ratio). For kaolinite, this aspect ratio is usually ~10 but can vary widely (Zbik and Smart, 1998). The aspect ratios for smectite minerals, such as montmorillonite, are generally much larger (e.g., 80–500). Hence, the area of the crystal basal planes is dominant over the edge area in expandable clay minerals like smectite in comparison to kaolinite.

Because of their platelet-like shape their equivalent diameters are 200–1000 nm for kaolinite and 5–200 nm for smectite particles. As a result of the electric charges present on the mineral surfaces (basal and edge sites), clay particles have the ability to form complex aggregates and network assemblies in aqueous solution. This behaviour is dependent on water and clay chemistry, as well as clay particle packing density (Bowles, 1968). The electric charge is compensated for by the adsorption of cations from solution (van Olphen, 1977). In the presence of water, the adsorbed cations in the clay sorption complex can be exchanged with cations of another species in the aqueous environment, and they are therefore known as the exchangeable cations.

Soil enters the raw sugar manufacturing process with the cane

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supply. Although, the cane plant has some soil particles adhered to it due to the effects of wind and rain most of the soil enters as a result of mechanical harvesting. The height of the harvester's base cutters are adjusted and operated so that the cane stalk is cut just above ground level. Due to variations in the field topography, at times, the base cutters penetrate into the ground and pick up soil with the cut cane. Harvesting during wet weather and crops that are affected by cyclones during the growing season can significantly increase the level of soil in the cane supply. In Australia, the soil levels in the cane supply of around 2 wt% are typical and in extreme cases it can be above 10 wt% (Sandell and Agnew, 2002).

Kampen (1974) reported that the soil entering the sugar factory reduced the overall plant efficiency — including poor settling rates of the particles in the clarifier and higher mud volumes. In the work Kampen (1974) conducted on three different types of field soils (sandy, silt and clayey) typically found in the Louisiana (USA) cane growing region, clayey soils increased the final mud volume, increased non-sugar impurities and lowered juice purity in comparison to the two other soils.

Although the present study did not use real sugarcane juice, it builds on the work undertaken by Kampen (1974) to obtain a greater understanding of the effect of soil types on clarification. Therefore, the identification of the mineral composition of the soils from sugarcane regions, their macroscopic and morphological attributes and their physical and chemical properties in electrolytes will provide useful information for subsequent studies on the clarification properties of real sugarcane juices.

2. Materials and methods

2.1. Materials

Field soil samples were obtained from three sugar cane growing regions in Queensland, Australia. The soil sample name, its origin, and typical factory clarification performance of the juices extracted from the different cane supply sources are shown in Table 1. The soil samples were collected from multiple locations and from the top ~10 cm of the soil and then mixed to produce composite samples. The larger soil aggregates were broken down to form fine loose soil using a Resch BB200 jaw crusher prior to mixing to form composites.

Chemicals, $CaCl_2 \cdot 2H_2O$ (Chem Supply, Gillman, Australia), $MgCl_2 \cdot 6H_2O$ (Merck, Kilsyth, Australia), NaCl and KCl (Sigma-Aldrich, Sydney, Australia), were analytical grade.

The stock synthetic juice solution was prepared by dissolving 1.91 g of NaCl, 47.67 g of KCl, 18.34 g of $CaCl_2 \cdot 2H_2O$ and 31.37 g of $MgCl_2 \cdot 6H_2O$ in 1000 mL of Milli-Q water.

2.2. Methods

2.2.1. X-ray powder diffraction

The mineral composition of the samples was established using X-ray powder diffraction (XRD). The samples were oven dried at 60 °C and thoroughly mixed using an agate mortar and pestle before being pressed onto stainless steel sample holders.

A sub-sample (~1.5 g) was ground for 10 min in a McCrone

micronizing mill with 2 mL of ethanol. The resulting slurry was oven dried at 60 °C then thoroughly mixed using an agate mortar and pestle before being pressed into a silicon sample holder prior to XRD analysis. The clay content was determined from a study of oriented samples, air dried on silicon wafer and mixed with ethylene glycol (Moore and Reynolds, 1997). XRD patterns were recorded using a PANalytical X'Pert Pro Multi-purpose diffractometer using Fe filtered Cu K_α radiation, auto-divergence slit, 2° anti-scatter slit and fast accelerator Si strip detector. Diffraction patterns were recorded in steps of 0.016° 2θ with a 0.4 s counting time per step, and logged to data files for analysis.

2.2.2. Structure characterisation by electron microscopes

Electron microscopy is the 'tool of choice' to study the micro-structure of clays (Smart and Tovey, 1982; Smart et al., 2004). Electron microscopy was undertaken using a JEOL-2100 Transmission Electron Microscope (TEM) with 200 kV accelerating potential. Scanning Electron Microscope (SEM) JEOL 6040 with a featured energy-dispersive X-ray (EDX) spectroscopy was used to investigate the sample coated with platinum with accelerating voltage 15–20 kV. A known weight (1.00 g) was dispersed in 9 mL of Milli-Q water, sonicated for 1 min, and then allowed to settle for 5 min. Using a Pasteur pipette, a droplet of the supernatant (i.e., the clay fraction) was placed on a sticky stub and dried at room temperature. For TEM analysis, a droplet of the supernatant was deposited directly onto a carbon film before drying the sample in an oven at 45 °C. Transmission X-ray microscopy (TXM) with a photon flux of 5 × 1012 photons/s and an accelerating voltage of 200 kV was used to investigate the micro-morphology of the clays in the soils at National Synchrotron Radiation Research Centre (Taiwan). TXM samples were prepared by depositing a droplet of supernatant onto a silicon wafer plate and allowed to dry at room temperature.

2.2.3. Measurement of cation exchange capacity and settling rate of the soils

The preparation and analysis for cation exchange capacity (CEC) were carried out based on methods 15A1 and 15A2 respectively as described by Rayment and Higginson (1992).

Batch settling tests were carried out to determine the settling rates of the soil particles. The settling tests were conducted in 100 mL measuring cylinders using 1.0 wt% soil solution in an electrolyte mixture of chloride salts (Na^+, 30 ppm; K^+ 1000 ppm; Ca^{2+} 200 ppm; and Mg^{2+} 150 ppm). The electrolyte composition was chosen to mimic the concentration of these ions present in a typical sugarcane juice solution. Prior to settling tests the soils were disintegrated in jars containing 1 g of the soil and 20 mL of Milli-Q water inside an ultrasonic water bath for 5 min. The soil solution was then transferred to a 150 mL beaker and stirred using a magnetic bar. A further 76 mL of Milli-Q water was added to the beaker followed by 4 mL of the stock synthetic juice solution to produce a 100 mL solution. The solution mixture was stirred for 1 min before being transferred to a 100 mL measuring cylinder and allowed to settle.

The mud interface height (boundary line between solid and liquid phases) was measured via visual assessment at 1 min intervals over a 30 min period. A plot of mud interface height versus

Table 1
Soil sample names, origins and historical factory clarification performances.

Sample name	Origin	Soil moisture (wt%)	Typical factory clarification; settling rate, cm/min
Mt Mackay	Tully	10.44	30–60 (Good)
Invicta 255B	Invicta	2.7	1–20 (Poor)
Gluepot 3522A	Mackay	7.89	1–20 (Poor)