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Compositional and structural changes of sugarcane evaporator deposits after concentrated sodium hydroxide treatment

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ABSTRACT

Fouling of industrial heat exchangers and evaporators is not only associated with energy management but also reductions in heat transfer coefficients causing loss in productivity. This paper describes the effect of NaOH solution on the removal of sugarcane factory heat exchanger deposits. Apatite, calcium oxalate, calcite, quartz, tricalcium aconitate and non-diffracting materials including amorphous silica are present in the deposits. Fourier Transform Infra-red (FTIR) spectroscopy revealed that monomeric silica species were preferential removed over oligomeric and polymeric species, while ²⁹Si nuclear magnetic resonance (NMR) analysis not only confirmed this, but identified the types of silica units Q⁰ [Si(OH)₄], Q¹ [(OH)₃*Si(OSi)], Q² [(OH)₂*Si(OSi)₂], Q³ [(OH)*Si(OSi)₃] and Q⁴ [*Si(OSi)₄] present, rearranged, and removed. After chemical cleaning, the scale components became truncated with indentation and contain spherical and hairy-like micro-sized particles. Also, the morphology of tetragonal CaCO₃·2H₂O was found to be distorted and the proportion of crystalline phases decreased through fragmentation of the scale matrix.

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1. Introduction

Industrialized countries around the world spend US\$26 billion per year or 0.2% of the gross national product for the removal of deposit in heat exchangers including evaporators (Müller-Steinhagen, 2000). More than 80% of industrial heat exchangers (present in oil, gas, chemical, petrochemical, mineral processing, power generation, food, water purification and pulp and paper industries) suffer from fouling problems despite regular cleaning routine both during shut down periods and also during plant operation (Müller-Steinhagen, 2000). Apart from reductions in heat transfer that occurs in heat exchangers, fouling can also reduce mass transfer (e.g., membrane filtration) (Sheikholeslami, 2003) causing inefficiencies, plant shutdowns and degradation of product and plant equipment (Yu et al., 2002a). Deposit formation is not a simple process because a number of fouling mechanisms (e.g., particulate and crystallization) can occur simultaneously and is further complicated by the variable conditions present in industrial

systems.

Thailand is the fourth largest sugar producer in the world, contributing ~104 million tonnes of sugar annually (FAOSTAT, 2014). Although sugar production in Thailand has been steadily increasing since 2010, fouling is a major issue hindering production in Thailand and globally (FAOSTAT, 2014). Thailand is also the second largest sugar exporter in the world with 4.5 million tonnes in 2014 (Sornpoon et al., 2014). The Thailand sugar industry also sustains the core of the Thai ethanol industry as it supplies both molasses and cane juice. In Thailand, many sugarcane factories have additional evaporators to reduce the impact on production; however this is offset by higher capital cost. In 2015, a Thai sugar factory spent about US\$211,243 per year for scale removal process that consists of chemical, manpower and instrumental replacement cost (Personal communication, Thanachai Khuwattasuchart, head of evaporator section Mitr Phol Sugar, Thailand, July 14, 2016). In order to minimize fouling in multiple effect evaporator (MEE) vessels of sugarcane factory, it is necessary to know the nature, type

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and amount of each scale component in each effect of the MEE system so that optimal scale removal and control programs can be incorporated (Srivastava et al., 2016).

The sugarcane juice quality (suspended solids and other soluble impurities) and evaporation operating conditions (rate, pH, temperature and sucrose concentration) impact and influence the formation of scale in sugar factories. The juice quality is dependent on the cane quality, harvesting and crushing practices, and the clarification process. In the clarification process, inorganic and organic non-sugar compounds undergo physical and chemical transformations. The transformations are continued in the evaporators where, as the concentration of juice increases, compounds are precipitated as their solubilities are exceeded. These compounds and suspended solids are deposited as scale on the calandria tubes of the evaporators resulting in the fouling of the heated surface. The scale that is deposited on the tubes restricts the flow of juice and, as a result of its thermal conductivity, reduces the rate of heat that is transferred across the tube surfaces. Consequently, evaporator vessels require cleaning on a periodic basis which causes significant disruptions and cost to the sugar factories.

Doherty and group (Yu et al., 2002b, 2005; East et al., 2013a, East et al., 2014) have studied the composition of sugarcane factory deposits as well the fouling mechanisms. Compounds such as calcium oxalate and hydroxyapatite are deposited by crystallization fouling, while amorphous silica and organic matter are deposited by particulate fouling. In many situations, there are preferential precipitation of hydroxyapatite on the tube side as opposed to the juice side of the calandria tube. Recently, the fine structural features and compositional profiles of layered deposits on heat exchange tubes from Australian sugar factories were identified by these authors (East et al., 2010). The presence of the mineral fouling, containing Si-Mg-Al-Fe-O was identified using elemental mapping, and found to be hard to clean which impacts on the poor chemical cleaning performances observed in some sugar factories. The severity of fouling that occurs in sugarcane heat exchangers warrants the need to develop cost-effective cleaning formulations. NaOH solution is the most common cleaning agents used in the removal of deposits in the chemical industry. Unlike acids, it is used in every cleaning cycle in sugarcane factories as it is less destructive on the mild steel and stainless steel components of the evaporators. Its effectiveness on deposit removal is dependent on a number of complex inter-relations, and includes such factors as (a) the amount and nature of the deposit composition, (b) working temperature (Fryer and Bird, 1994), (c) the fluid flow (Bird and Fryer, 1991), and (d) its concentration (Gallot-Lavallee et al., 1984). While all these information have been useful for the design of chemical cleaning formulations and scale inhibitors, knowing the composition and structural features of the components of residual scale after chemical cleaning will provide additional information for improved design of cleaning formulations. This is particularly so in Thailand, where there are no guidelines for chemical cleaning of evaporators. Besides, only the traditional cleaning chemicals such as NaOH are used in Thailand, which are not effective in the removal of many scale components. Therefore, the objective of the study is to determine the composition and structural changes of scale components collected before and after chemical cleaning with NaOH as a first step in the development of NaOH-based chemical cleaning formulations. The compositional analysis of the scales collected from a Thai sugar factory was carried using X-ray powder diffraction (XRD), scanning electron microscopy (energy dispersive spectroscopy, EDX) techniques, thermogravimetry/differential thermal analysis (TG/DTA), Fourier transform Infrared spectroscopy (FTIR) analysis, and ^{29}Si magic angle spinning nuclear magnetic resonance (NMR).

2. Materials and methods

2.1. Materials

Scale deposits were dislodged with high pressure water from the evaporator station of Mitr Phol Kalasin (MPKS) factory in the South North Eastern part of Thailand. In this factory, the juice is split into three parallel quintuple evaporator trains. Steam in a multi-effect arrangement is used to heat the vessels.

After the juice is drained from the vessels, the vessels are sprayed with water to remove residual juice, and then steam is introduced to the calandria at 120 °C. The vessels are then heated for 3 h. At the end of this period, the vessels are sprayed with caustic soda (40–50 w/v%) and heated at ~100 °C for 3 h. The caustic soda solution is then flushed out, and the vessels washed repeatedly with water until the pH of the solution is ~7.

The scales were collected after two weeks of processing before and after chemical cleaning at the early part of the season. This is to ensure that months of residual deposit accumulation did not obscure the results if samples were collected periodically during the season to make composite scales. Each of the scale samples (20–50 g) collected was washed repeatedly with distilled water (4 × 400 mL) and then dried to constant weight at 100 °C for 8 h.

2.2. Methods

The elemental composition of the bulk samples were analyzed using a PanAnalytical PW2400 WDXRF spectrometer. The powdered sample, glass bead and Type 22:12 flux were mixed and melted, and the prepared bead was analyzed by the XRF spectrometer.

X-ray powder diffraction (XRD) studies on the ground samples were conducted using a PANalytical X'Pert MPD, Cu K α (1.5418 Å) radiation. To each sample was added 10% corundum, as an internal standard in order to quantify the amount of the crystalline compounds. The X-ray powder diffraction spectra were indexed based on parameters obtained from the ICDD powder card XRD index. The analysis of the data were carried out using a HighScore (v2.2, PANalytical, Netherlands) using a Rietveld-based technique. The sum of the concentrations of the modelled crystalline phases is subtracted from 100% to give the non-diffracting phases. As an approximation, the non-diffracting phases have been grouped as the amorphous portion of the scale.

The elemental composition of the surface of the scales analyzed by electron dispersive spectroscopy using a JOEL, JXA-840 (20 kv, 1.0 nA, T3) and Analysis Program (v3.30, JOEL). The scales were carbon coated prior to analysis. For each sample, analysis was conducted on five different spots and the average value obtained for each element. The experimental error in the analysis is ±4%. Scanning electron images of the gold-coated scale flakes were obtained using the JOEL, JXA-840 instrument.

Decomposition studies of the scale samples were carried out in a TA instruments Q500 in a nitrogen-flowing atmosphere of 15 mL/min. Approximately, 20 mg of sample was heated at a rate of 10°C/min from ambient temperature to 1000 °C, and the mass loss was recorded to identify the various degradation stages.

For the determination of functional groups, Infrared spectra were collected using a Nicolet Diamond 5700 ATR FTIR spectrometer equipped with a Smart Endurance single bounce diamond ATR accessory (Nicolet Instrument Corp., US). The spectra were collected in the spectral range 4000–525 cm $^{-1}$, using 64 scans at 4 cm $^{-1}$ resolution with a mirror velocity of 0.6329 cm/s.

The Si profile of the samples was analyzed using Agilent direct drive spectrometer, operating at 79.41 MHz using the standard one-pulse sequence. Samples were spun at 5 kHz in a 5 mm silicon nitride rotor. Spectra were recorded with 313 data points

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