



The effect of cleaning agents on the structural features of heat exchanger deposits from sugar factories

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ABSTRACT

The removal of deposits from heat exchangers is one of the biggest challenges facing many food and chemical industries. Sodium hydroxide and acid cleaning are generally used on a regular basis to remove composite scale deposits from sugarcane factories. This paper evaluates the use of Fourier Transform Infrared (FTIR) spectroscopy, X-ray fluorescence, X-ray powder diffraction and ²⁹Si nuclear magnetic resonance (NMR) to study the composition and the structural features of residual solid after NaOH treatment and use a second treatment of ethylene diamine tetracetic acid tetra sodium salt (Na₄EDTA) or dilute HCl to unravel the transformations of silica. There is no evidence of strong association between the silica matrix and the oxalates, or calcium carbonate components, but the use of dilute HCl resulted in the formation of complex and intractable Q³ [(OH)*Si(OSi)₃] and Q⁴ [*Si(OSi)₄] species as a consequence of re-organization/repolymerization processes. On the contrary, the use Na₄EDTA resulted in the formation of the simpler Q⁰ [Si(OH)₄] and Q¹ [(OH)₃*Si(OSi)] species. However, when the deposit contains a high proportion of hydroxyapatite (Ca₅(PO₄)₃OH), HCl is more effective than Na₄EDTA. When a cleaning regime involves a two-step process the order of addition is important for effective removal of deposits.

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1. Introduction

Fouling of heat exchangers is a nuisance to the sugarcane and other food industries as well the chemical manufacturing industries. These industries spend considerable amount of money and man-hours to clean the heat exchangers/evaporators because of the presence of deposits that consist of compounds with different solubility characteristics. Unlike the sugarbeet factories, the use of scale inhibitors such as polyacrylic acid and polymaleic acid or a combination of both, have limited or have no noticeable effect in preventing or reducing the amount of deposits formed in sugarcane factories (Doherty et al., 1995). This is mainly due to higher proportions of inorganic and organic impurities present in sugarcane juice compared to sugarbeet juice (Honig, 1953). The traditional cleaning agents, NaOH, sulfamic acid, and inhibited HCl are not effective for the removal of composite scale that consists of combinations of amorphous silica, hydroxyapatite, calcium sulfate, iron oxide, calcium oxalate and organic matter, as residues of these

compounds still remain at the end of a chemical cleaning exercise (Doherty, 2000). These residual solids provide nucleation sites that accelerate scale deposition in subsequent juice processing cycle reducing the heat exchangers' efficiency. Also, the fouling resistance of the deposit is high and so even with a few microns in thickness remaining, after chemical cleaning; there is a significant lowering of the heat transfer coefficient.

Like other food and chemical industries, the sugarcane industry wants to reduce the amount of energy used for the production of the primarily product (i.e., marketable sugar) in order to use it elsewhere for value-adding. For instance, many sugarcane factories around the world want to increase their profit base by increasing the amount of available bagasse that can be used in value-adding. This is achieved by reducing the amount of steam/energy consumed by the factory through increases in the bleeding of vapors from the evaporating effects (Phakam et al., 2017). To maintain constant vapor bleeding, from an evaporator station, the heat transfer coefficient across the station should be at a high and constant value so that the temperature of the bleeding vapor available for heating is adequate. For this to be achieved, the evaporators should not be significantly fouled. As such, cleaning of the evaporators during each cycle must be effective so as to not to allow residual solid to remain.

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Yu et al. (2002a) demonstrated the effect of the mutual interference between calcium oxalate and silica on their formation as composites in deposits. They proposed that the formation of calcium oxalate-silica species through complexation and bridging (surface neutralization) mechanisms influenced the amount and proportions of these compounds formed in a composite. The extent of silica polymerization rate depends on the amount of calcium oxalate particles and the calcium oxalate-silica complexes, both of which acted as additional nucleation sites for the polymerization process. In another study by Yu et al. (2002a), the type of the morphology of calcium oxalate formed was explained on the basis of electrostatic effects, geometrical matching and stereochemical compatibility between calcium oxalate crystallites and amorphous silica. None of these studies provided direct evidence as to whether silica and calcium oxalate existed as simple mixtures or are interconnected in any form, such as hydrogen bonding. Recent efforts by Phakam et al. (2017) were focused on identifying the types of silica substructures from different sugarcane factories in order to ascertain whether the differences in cleaning effectiveness were related to, in part, the structural complexity of the silica matrix and the conditions used in the cleaning process. In that study, there was no effort to examine interactions between silica and the crystalline phases in the composite either before or after the deposit were treated with acid or a base. The studies by Demadis and his group (Demadis et al., 2011a, 2011b and Demadis et al., 2012) specifically studied the effect of various additives (e.g., phosphonobutane-1,2,4-tricarboxylic acid, gallic acid and ammonium bifluoride) and their structural features on the dissolution of colloidal silica. However, none of these studies were relevant to that of composite deposits present in sugar factories.

In the present study, solid-state ^{29}Si magic angle spinning nuclear magnetic resonance (^{29}Si MASNMR) and Fourier Transform Infra-red (FTIR) were used to characterize intractable silica-based deposits remaining after NaOH treatment in the fourth and fifth effects of quintuple evaporators of sugarcane factories. These evaporators were selected as the deposits present in them are the most difficult to remove. Scanning electron microscopy (SEM) was used to examine the size and shape of the particles. Changes in the molecular structure of the silica matrix and scale composition as a result of a second treatment with ethylene diamine tetracetic acid tetra sodium salt (Na_4EDTA) or dilute HCl were examined, as well as the effectiveness of these cleaning agents to dissolve the residual solid obtained after NaOH treatment. X-ray powder diffraction (XRD) was also used to determine the crystalline phases remaining or formed during second treatment process. The interactions among the scale components were further examined by ^{29}Si NMR and the effect of the treatment conditions on the type of silica species formed were also evaluated. It is the intention of this study to provide information to that would add to the design of new chemical cleaning formulations for the removal intractable deposits.

2. Materials and methods

2.1. Materials

Scale deposits were collected at the start of the season to minimize the accumulation of residual solids left behind from previous chemical cleans. They were collected after cleaning of the evaporators at the end of two weeks processing time. High pressure water was used to dislodge the deposit from the evaporators. Scale samples were collected and washed with distilled water (4×400 mL) and then dried to constant weight at 100°C for 8 h. Rust particles were removed using a magnet. The amount of rust removed was up to ~ 10 – 12 wt%.

The deposits were collected from three sugar factories located at different geographical locations in Thailand. The factories are Mitr Phol Phu Khieo (MPPK) located in the Far North Eastern part of Thailand, Mitr Phol Kalasin (MPKS) in the South North Eastern part of Thailand, and Mitr Phol Singburi (MPSB) located in Central part of Thailand. In the MPPK factory, the juice is split into five parallel quintuple evaporator trains. In the MPKS factory, the juice is split into two parallel quintuple evaporator trains. In the MPSB factory, the juice is fed into the first effect. Steam in a multi-effect arrangement is used to heat the vessels.

There are slight variations in chemical cleaning of the heat exchanger/evaporator stations in the factories. In the MPKS factory, after the juice is drained from the evaporators, the evaporators are sprayed with water to remove residual juice, and then steam is introduced to the calandria at 120°C , and the vessels heated for 3 h. At the end of this period, the vessels are sprayed with caustic soda (40 – 50 w/v%) and heated at $\sim 100^\circ\text{C}$ for 3 h. The NaOH solution is then flushed out, and the vessels washed repeatedly with water until the pH of the solution is ~ 7 . The cleaning protocol of MPPK involves the use of NaOH solution (20 – 40 w/v%) for between 6 and 8 h at a temperature of $\sim 100^\circ\text{C}$.

In the MPSB factory after the juice is drained and sugars and soluble impurities are removed with hot water spray, NaOH solution (50 w/v%, 60 – 80°C) is then sprayed and the vessels maintained at 60 – 80°C for 6 h. Water is then used to rinse the vessels until the pH of the solution is neutral.

Fumed silica (Supplier, Country), $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ (Sigma Aldrich, Sydney Australia) and CaCO_3 (Chem Supply, Gilman, Australia) used in making synthetic silica-based scale were analytical grade materials.

2.2. Methods

The elements present in the scale samples were determined using a Bruker-AXS 54 Pioneer X-ray fluorescence spectrometer equipped with a AG Rh 22 (Rhodium anode) tube. The error in the analysis is $\pm 1\%$.

The Infrared spectra of the scale samples were obtained 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 .

X-ray powder diffraction (XRD) spectra of the samples were obtained using a PANalytical X'Pert MPD, Cu K_α (1.5418 Å) radiation. Corundum (10%) was used as the internal standard in order to determine the proportion of the crystalline phases. The X-ray peaks were indexed based on parameters obtained from the ICDD powder card XRD index. The data were analyzed using a HighScore (v2.2, PANalytical, Netherlands) based on the Rietveld technique. The sum of the concentrations of the modelled crystalline phases is subtracted from 100% to give the non-diffracting phases, which is an approximation of the amorphous content.

The ^{29}Si analysis of the samples was investigated using an Agilent direct drive spectrometer, operating at 79.41 MHz using the standard one-pulse sequence. The samples were spun at 5 kHz in a 5 mm silicon nitride rotor. The spectrum of each sample was recorded with 313 data points over a spectral width of 200 ppm using an acquisition time of 0.019 s, a relaxation delay of 15 s and 5000 transients.

For the scanning electron microscopy (SEM) images, the deposits were carbon coated and examined using a FEI Quanta 200 Environmental scanning electron microscope at an accelerating voltage of 15 kV.

In separate experiments, scales samples (1 g) were treated with

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