Effect of Thermohydraulic Conditions on Fouling of Calcium Oxalate and Silica

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Calcium oxalate (CaOx) and amorphous silica (SiO_2) have been identified as the most intractable components of scale formed in sugar mill evaporators. Effects of thermohydraulic conditions such as surface superheat (5–15 K), fluid velocity (0.8–1.8 m/s), and bulk subcooling (20–30 K) on the composite fouling process of CaOx and SiO_2 and deposit characteristics were investigated in a dynamic system that contained a fouling loop under subcooled nucleate flow boiling. The rate of composite fouling was found to increase with increasing superheat as a result of enhanced bubble activities at the boiling surface and increase in deposit strength. For a constant surface superheat, the fouling rate reached a maximum value at a subcooling of 25 K, but remained constant over a range of fluid velocity ($\geq$1.1 m/s). This was attributed to the counterbalancing effects of deposit consolidation and hydrodynamic lift forces at the fouled tube wall. Scanning electron microscopy (SEM) analysis of composite deposits demonstrated the presence of two layers arising from deposit consolidation by CaOx in SiO_2 matrix through microlayer evaporation during nucleate boiling. Chemical microanalysis by energy dispersive X-ray spectroscopy (EDS) also indicated that the degree of deposit consolidation increased with surface superheat, fluid velocity, and bulk subcooling because of an increase in heat flux. © 2005 American Institute of Chemical Engineers *AIChE J*, 51: 641–648, 2005
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Introduction

The concentration of juice in multiple-effect evaporators of sugar factories is known to cause extensive scale formation on the surface of calandria tubes. This poses a major operational constraint on the heat-transfer efficiency. In addition, the removal of these deposits by chemical means is time-consuming and generates large quantities of toxic wastes requiring treatment. The composition of deposit varies depending on the stage of evaporation cycle in which it is formed and may include phosphates, silica, and organic acid salts.[1,2] Phosphate scales are more commonly observed in the earlier effects, whereas silica and calcium salts of organic acids such as oxalate usually deposit in the latter vessels because of temperature and concentration effects. Unlike most other scalants, CaOx [as calcium oxalate monohydrate (COM) and calcium oxalate dihydrate (COD), with COM being the most stable phase under high temperatures] and SiO_2 form a hard composite scale that is intractable to conventional cleaning methods. Ethylenediaminetet-
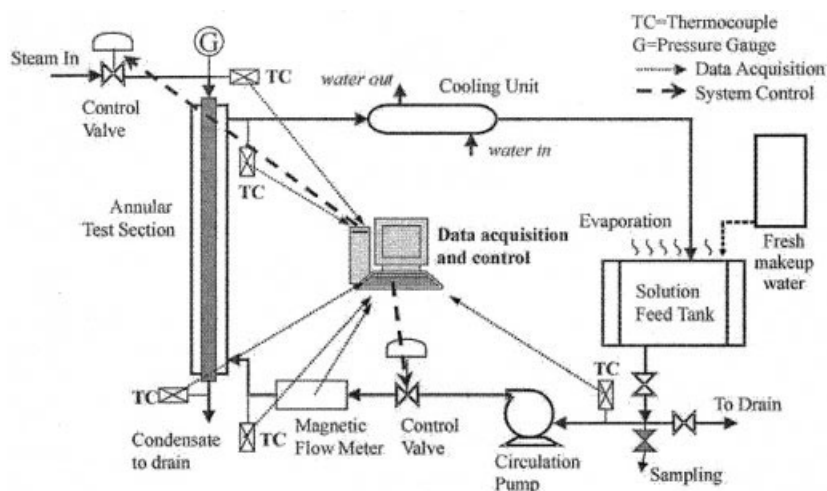
Figure 1. Test rig.

raacetic acid (EDTA), a chelating agent, is often required for its effective removal from the evaporators.[3]

Composite fouling is a form of fouling process involving more than one type of foulant or fouling mechanisms, which may interact with each other to produce the final deposit. The current knowledge gap on fouling has partly been associated with a lack of understanding of the mechanisms underlying the composite fouling process, despite its practical significance. Conventional study to date on fouling mechanisms has been restricted to single-component systems and only in a limited number of cases have the effects of interfering species on the fouling process of interest been reported.[4-8] It is known that the coexisting compounds often interact to give rise to effects that may be synergistic or antagonistic, the results of which are not predicted by single-component studies.[7,9] Also, industrial process streams such as those in sugar mill evaporators very often contain different types of fouling species, which may exert an impact on the removal of scales by altering their strength and structure in addition to the influences on the deposition rate. These facts have necessitated the need to study the interactive effects in multi-component fouling systems to obtain a better understanding of composite fouling mechanisms.

Coprecipitation of COM and SiO_2 has been demonstrated to promote COM precipitation and SiO_2 polymerization in sugar solutions.[10] The rate of composite fouling and the tenacity of composite scale were shown to depend on solution composition. The primary mechanism of composite fouling was found to be particulate deposition of colloidal COM–SiO_2 complexes by overcoming the interfacial energy barrier between the heat exchange tube surface and that of COM–SiO_2 particles, both of which were negatively charged.[7,11] Thus, the extent of fouling was surface-charge dependent, with maximum fouling attained as the surface charges of the heat exchange tube and/or colloidal particle were minimized. This indicated that the composite fouling of COM and SiO_2 was likely to be a surface-controlled process.[7,11]

To successfully develop a predictive model simulating the composite fouling of COM and SiO_2 in sugar mill evaporators, and to provide further insight into the composite fouling mechanisms, the effect of operating parameters on the fouling process needs to be known. This paper, therefore, investigates

composite fouling characteristics of COM and SiO_2 in synthetic juices over a range of thermohydraulic conditions including surface superheat, fluid velocity, and bulk subcooling typically found in the Australian sugar mill evaporators. The physicochemical properties of composite scales are also evaluated using instrumental analysis.

Experimental

Test rig

The experimental apparatus used in this work was a closed-loop circulation system, consisting of: a feed tank; a circulation pump; an annular test section; a cooling heat exchanger; a fresh makeup water tank; series of pressure, temperature, and flow measuring and control devices; and a PC for data acquisition and control. Figure 1 shows a schematic diagram of the major components of the test rig. It was designed to operate under either constant composition mode, to investigate the effects of operating conditions on the formation of composite scales, or continuous evaporation to simulate the multieffect evaporation cycle in a sugar mill. A detailed description of this novel test loop and experimental procedure has been reported elsewhere.[7,12]

The annular test section is composed of a removable stainless steel tube (Sandvik Australia P/L), concentric with an external glass shell (Pegasus, Canada), to allow visual observation of the fouling process during the experiment. The data acquisition and control system (Genie, American Advantech Co.) was used to collect inlet and outlet temperatures of both steam and water systems from embedded thermocouples and water flow rate from a magnetic flowmeter (Promag 50P, Endress & Hauser) and to control the water flow rate and heat flux with PID regulators (Spirax Sarco P/L). The data collected were automatically monitored to ensure that the operating conditions were carefully controlled. The water loss resulting from evaporation in the main tank was compensated by a feed of fresh makeup water regulated by a level control system, which includes a proportioning solenoid valve and pressure transmitter (Burkert Contromatic Australia P/L).

Condition	This work	Sugar mill evaporator (1st–5th effect)
Heat flux (kW/m^2)	56.4–104.9*	16.7–23.3
Superheat (K)	5.0–15.0	12.0–26.0
Velocity (m/s)	0.8–1.8	0.8–2.8
Reynolds number (liquid phase)	7875–18,121	n/a
Subcooling (K)	20.0–30.0*	0–17.0
Bulk temperature (°C)	70.0–80.0	54.0–100.0

*Different ranges from those used in the sugar evaporators, also see Yu et al.[12]

Test conditions and procedure

All experiments were performed under a constant solution composition and subcooled flow boiling heat transfer to evaluate the effects of thermohydraulic parameters on composite fouling. Table 1 shows the summary of operating conditions used in this work compared to those in the sugar mill evaporators. A fixed sugar concentration of 25% was used in all experiments. The initial concentrations of COM and SiO$_2$ were determined based on the results of previous runs of evaporative concentration, which suggested that composite fouling at an initial COM and SiO$_2$ concentration of 50 and 500 ppm, respectively, could start as early as when the concentration factor reached about 1.7.[7] Typical duration of each run was about 150–160 h, beyond which significant sucrose degradation occurs, as was evidenced by color formation,[13] and was adequate for the present purposes.

Synthetic juice solutions were prepared from a commercial sugar (purity 99.5%) kindly supplied by Sugar Australia, Pty, Ltd., NSW. The initial pH of the tank solutions was adjusted to 6.5 using HCl and NaOH. All other salts and chemicals used (for preparation of silica and COM stock solutions) were AR-grade reagents. Data collection was initiated after all the predissolved chemicals were mixed in the tank. The changes in COM and SiO$_2$ concentrations during the run were monitored through periodic sample withdrawal. A portion of each withdrawn sample was immediately filtered through a 0.22-μm filter (Millipore Co.) and acidified with concentrated HCl to stabilize the cation (for COM analysis). The remaining sample solutions were diluted with distilled water to stabilize the SiO$_2$ in solution (for SiO$_2$ analysis). Calcium, oxalate, and total (that is, reactive and colloidal) SiO$_2$ contents were determined using ICP-AES (inductively coupled plasma atomic emission spectrometer; Varian Vista AX) and UV–visible spectroscopy (Varian Cary 1E spectrophotometer). Fresh chemicals were added to the main tank at an average rate of 1.9 ppm COM/h and 2.7 ppm SiO$_2$/h to maintain the concentrations of COM and SiO$_2$ in solution, which tended to decrease with time because of deposit buildup on the heat exchange surface and bulk precipitation.

After each experiment, the scale was removed from the tube, washed, and dried under room temperature before being characterized by SEM (Hitachi S4500) and X-ray powder diffraction (XRD; Siemens D5000) analyses. Sample scale was also treated with resin setting (to give polished cross sections) and its chemical composition examined by EDS analysis (Oxford Link ISIS 200) (see Yu et al.[15] for more details). New heat exchanger tubes were used for each experiment. The tube surfaces were cleaned with 2% (w/v) hot caustic soda followed by dilute acid (pH = 1) to remove dirt and grease before being installed in the loop.

Data analysis

The instantaneous fouling resistance, $R_{f(t)}$ (m^2 K/kW), was calculated from the overall heat transfer coefficient, U_o (kW/m^2 K) at the beginning of the each run and at any given time t, when fouling has taken place (Eq. 1). The overall heat transfer coefficient (U_o) is determined from the heat duty q (kW), the heat transfer surface area A (m^2), and the log-mean-temperature difference ΔT_m (K), for a countercurrent arrangement (Eq. 2)

$$R_{f(t)} = \frac{1}{U_{o(t)}} - \frac{1}{U_{o(t=0)}} \qquad (1)$$

$$U_o = \frac{q}{A \Delta T_m} \qquad (2)$$

The uncertainties in the overall heat transfer coefficients were found to be less than ±9%.[7,12] To compare the effects of operating conditions, the instantaneous net fouling rate, ϕ_f (m^2 K/kW·s), was calculated by

$$\phi_f = \phi_d - \phi_r = \frac{dR_{f(t)}}{dt} \qquad (3)$$

where ϕ_d (m^2 K/kW·s) and ϕ_r (m^2 K/kW·s) are the deposition and removal rates, respectively. Depending on the relative extents of deposition and removal rates, the R_f vs. t fouling curve can show a linear, falling rate or asymptotic behavior. For particulate fouling, the deposition term is usually considered to be constant, whereas the removal term increases with the deposit growth, resulting in an asymptotic-type fouling curve.[8,16] The linear fouling kinetics generally indicates hard and adherent deposits attributed to crystallization fouling, whereas the falling rate may result from a falling deposition rate and/or increasing removal rate. It has to be noted, however, that the linear or falling rate fouling kinetics of a species may actually be the early stage of asymptotic behavior, and a sufficiently long period of fouling experiment would be required to be confident of its real fouling behavior.[17]

Results and Discussion

Effect of superheat/heat flux

Under the subcooled flow boiling conditions used in this work, the fouling rate ϕ_f was found to increase with heat flux, especially at high superheat levels (>10 K) (Figures 2a and b and Table 2). The dependency of ϕ_f on superheat was approximately to the power of 3.3 (determined by fitting a power equation to data), which was comparable to those found for the deposition of hematite particles in boiling water systems.[18] The increased composite fouling rate at higher heat flux may be related to enhanced bubble activities during nucleate boiling. Bubble–particle interactions have been investigated for oxides and silica by Leenaars,[19] mainly to characterize an approach of removing submicron particles by passage of an air/liquid mixture. The author observed that colloidal SiO$_2$ particles could be
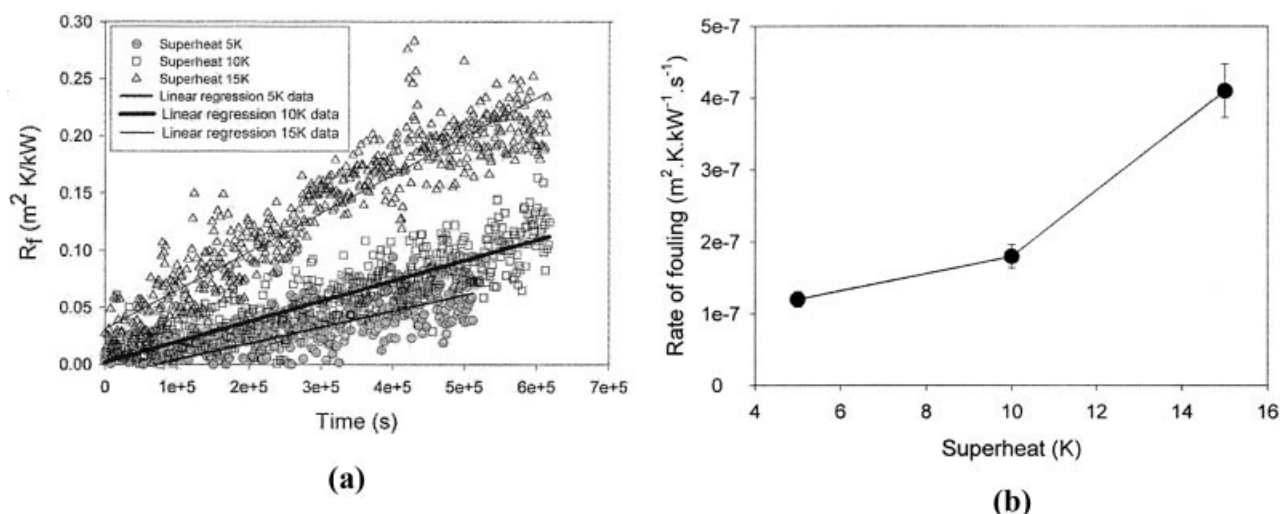
Figure 2. (a) Effect of superheat on the rate of composite fouling in sugar solution, velocity 1.1 m/s, subcooling 20 K, sugar concentration 25%; (b) the composite fouling rate in sugar solutions as a function of superheat at velocity 1.1 m/s, subcooling 20 K, and sugar concentration 25%.

preferentially accumulated at the gas–liquid interface, provided that the wetting characteristics of the particles were brought into appropriate range. Thus it was possible that, under certain conditions, the bubble nucleation sites act as particle binder, thereby promoting deposition. Furthermore, the evaporation of microlayer water at the vapor–liquid–solid triple interface of bubble nucleation sites (Figure 3), which was proposed for the deposition of silica and corrosion products in boiling water systems,[20-22] may also be a significant factor here. Microlayer evaporation would not only cause a local increase in the concentration of COM and SiO_2 at bubble nucleation sites, but also induce a convective flow of water, which carried the colloidal COM/SiO_2 particles to the heating surface (Figure 3).[21,23]

The fouling curve generally exhibited linear increase (Figure 2a) within the duration of the experiment, implying a constant rather than increased removal rate with the growing deposit. This behavior was not surprising based on the tenacious nature of the composite deposit.[7] Assuming an adhesion-controlled deposition process for the range of Reynolds number used in this work (Table 1)[7] and 100% particle attachment efficiency, the rate of particle deposition could be expressed as

$$\phi_d \approx \frac{K_a C_b}{\rho_f k_f} \tag{4}$$

where K_a is the adhesion rate coefficient (m/s) and C_b is the bulk concentration of particles (kg/m^3). ρ_f and k_f are the density (kg/m^3) and thermal conductivity (W/m K) of deposit, respectively.[8,24] The adhesion rate coefficient is related to surface temperature T_s (K) by an Arrhenius-type dependency analogous to a chemical reaction

$$K_a = K_o \exp(-E/RT_s) \tag{5}$$

where K_o is the preexponential factor, R is the gas constant (J/K mol), and E is the activation energy (kJ/g mol), which is a measure of bonding strength of deposit.[25] The activation energy was estimated (see Sheikholeslami et al.,[4] Sudmalis and Sheikholeslami,[5] Yu et al.,[6,7] and Oliveira et al.[8]) to be about 128 kJ/g mol, considerably higher than the literature values typical of silica scales (~40–85 kJ/g mol, surface temperature-based).[26] The higher bonding strength of the composite deposit

Table 2. Summary of Composite Fouling Rate Data for Various Thermal Hydraulic Conditions, Sugar Concentration 25%

Run No.	Superheat (K)	Heat Flux (kW/m²)	Velocity (m/s)	Subcooling (K)	$\phi_f^* \times 10^{-7}$ (m² K/kW · s)
1	5	56.4	1.1	20	1.2
2	10	67.9	1.1	20	1.8
3	15	80.4	1.1	20	4.1
4	10	48.0	0.8	20	4.2
5	10	79.4	1.4	20	1.7
6**	10	79.4	1.1	25	3.2
7**	10	87.7	1.1	30	2.4
8	10	104.9	1.8	20	1.7

* Fouling rate determined by linear regression.
**At comparable initial COM/SiO_2 supersaturations to those of the other runs because of different solubilities of COM and SiO_2 in solution resulting from changes in the bulk temperature.
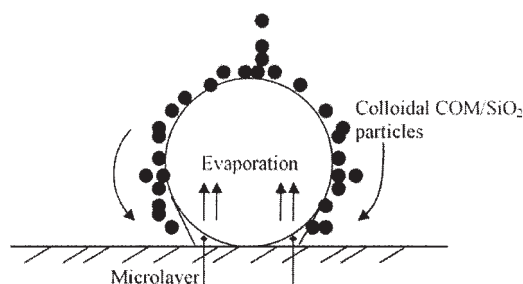
Figure 3. Deposition model of microlayer evaporation at bubble nucleation site.

was in good agreement with previous findings by the authors that COM–SiO$_2$ composite scale was more tenacious than silica scale.[7]

It was proposed by Nicholson and Sarbutt,[27] Hussain et al.,[28] and Turner and Klimas[29] that deposit consolidation experienced by scales formed under high temperatures on the boiling surface might result in an increase in the bond strength of the deposit, making it hard to remove with low shear forces. Consolidation is a process in which particles are attached to the heat transfer surface or preexisting deposit by chemical bonding. The driving force of consolidation varies according to the type of fouling species present and may include Ostwald ripening and boiling-induced precipitation of dissolved species.[29] For COM–SiO$_2$ composite fouling, consolidation might have been achieved by the cementing effect of calcium oxalate precipitation in the silica matrix through microlayer evaporation (see Figure 3).[7] Consequently, the rate and extent of consolidation will depend on the rate of evaporation or heat flux.

Effect of subcooling

Figure 4 shows the effect of bulk subcooling on composite fouling under a constant superheat (10 K) and fluid velocity (1.1 m/s). The rate of composite fouling was markedly higher at a bulk subcooling of about 25 K compared to those obtained
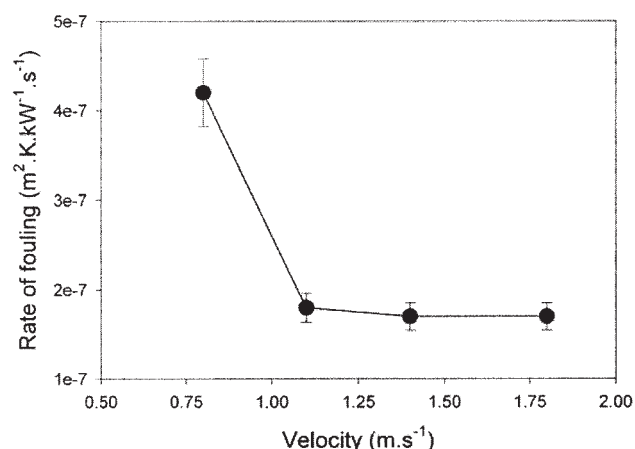
Figure 5. Changes in the fouling rate as a function of fluid velocity at surface superheat 10 K, bulk subcooling 20 K, and sugar concentration 25%.

at 20 and 30 K (Table 2). The initial increase in fouling rate between 20 and 25 K subcooling was consistent with previous studies of particle deposition in subcooled boiling water,[30] which may be attributable to enhanced deposit consolidation at high heat flux (Table 2). An increase in the bulk subcooling, however, would also lead to changes in the physical properties of liquid suspension such as viscosity. It was established by Williams et al.[31] that near-wall lift forces or drainage forces,[30,32] a component of the hydrodynamic lift forces governing particle reentrainment, have a linear dependency on the fluid viscosity. So the drops in the fouling rate at higher subcooling (30 K) may be the result of increased hydrodynamic lift forces, which counterbalanced the effects of deposit consolidation under increasing heat flux (Table 2).

Effect of fluid velocity

The effect of fluid velocity on the rate of composite fouling at a superheat of 10 K and a bulk subcooling of 20 K is shown in Figure 5. An increase in the fluid velocity reduced the rate of fouling, although only a slight decrease in ϕ_f could be observed for velocities > 1.1 (m/s) (Figure 5). The inverse dependency of fouling rate on velocity correlated well with the proposal that COM/SiO$_2$ composite fouling was a surface adhesion–controlled process[7]—the deposition rate was independent of fluid velocity (Eq. 4), whereas the removal rate increased with increasing shear and drag forces at the wall of fouled tube.[8,32] Taking the influences of hydrodynamic lift forces into account, recent studies of particle deposition from flowing water suspension have actually shown that the attachment efficiency of particle to the surface also tended to decrease with the wall shear stress.[33,34] Thus the effect of fluid velocity may involve a dual role of enhancing removal forces as well as inhibiting the attachment of particle.

The slight variation in the fouling rate for velocities 1.1–1.8 (m/s) (Figure 5) may be attributable to an increase in the rate of consolidation and thus deposit strength with shear stress,[29,35,36] which probably compensated for the tendency of high fluid velocity to reduce the net rate of fouling. Melo and Pinheiro[35] investigated the particulate fouling of kaolin–water
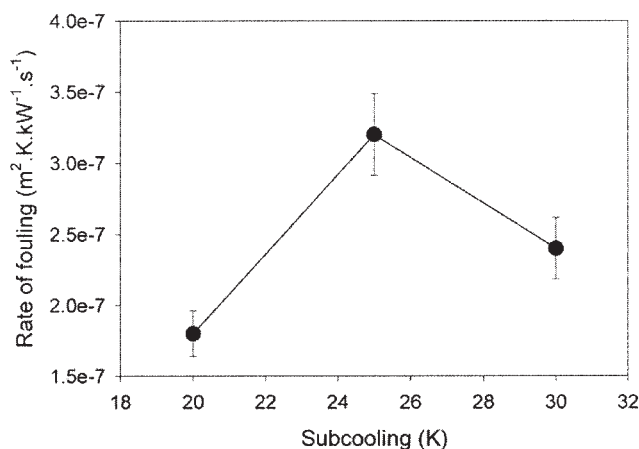

Figure 4. Effect of bulk subcooling on the rate of composite fouling in sugar solution, surface superheat 10 K, fluid velocity 1.1 m/s, sugar concentration 25%.
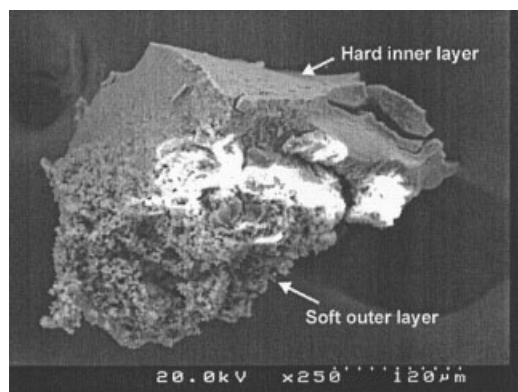
Figure 6. Two-layer structure of composite deposit (Run 6) formed in 25% sugar solution.

systems in an annular heat exchanger. They showed that the kaolin deposit consisted of a two-layer structure, with the inner layer being more cohesive than the outer layer. The deposit strength was found to increase with increasing Reynolds num-

ber because of a rising proportion of hard layer formed in the deposit. This phenomenon was supported in the present study by results of the instrumental analysis on the deposits as well as the visual and tactile study showing two layers in the deposit: a hard inner layer and a soft outer layer, as will be discussed in the next section.

Deposit characteristics

Visual and tactile inspection of the deposition process during and after the fouling experiments suggested the existence of two layers of different characteristics: a hard inner layer (~0.10 mm thickness) covered by a soft outer layer (~0.15 mm thickness). This deposit bilayer was later confirmed by SEM analysis (Figure 6), which showed that the outer layer had a porous structure containing aggregates of large particles, whereas the inner layer appeared to be much more compact and homogeneous.

Generally, deposits obtained at low surface superheat (5–10 K), velocity (0.8–1.1 m/s), and bulk subcooling (20 K) (Figures 7A–C) appeared to be more porous than those formed in
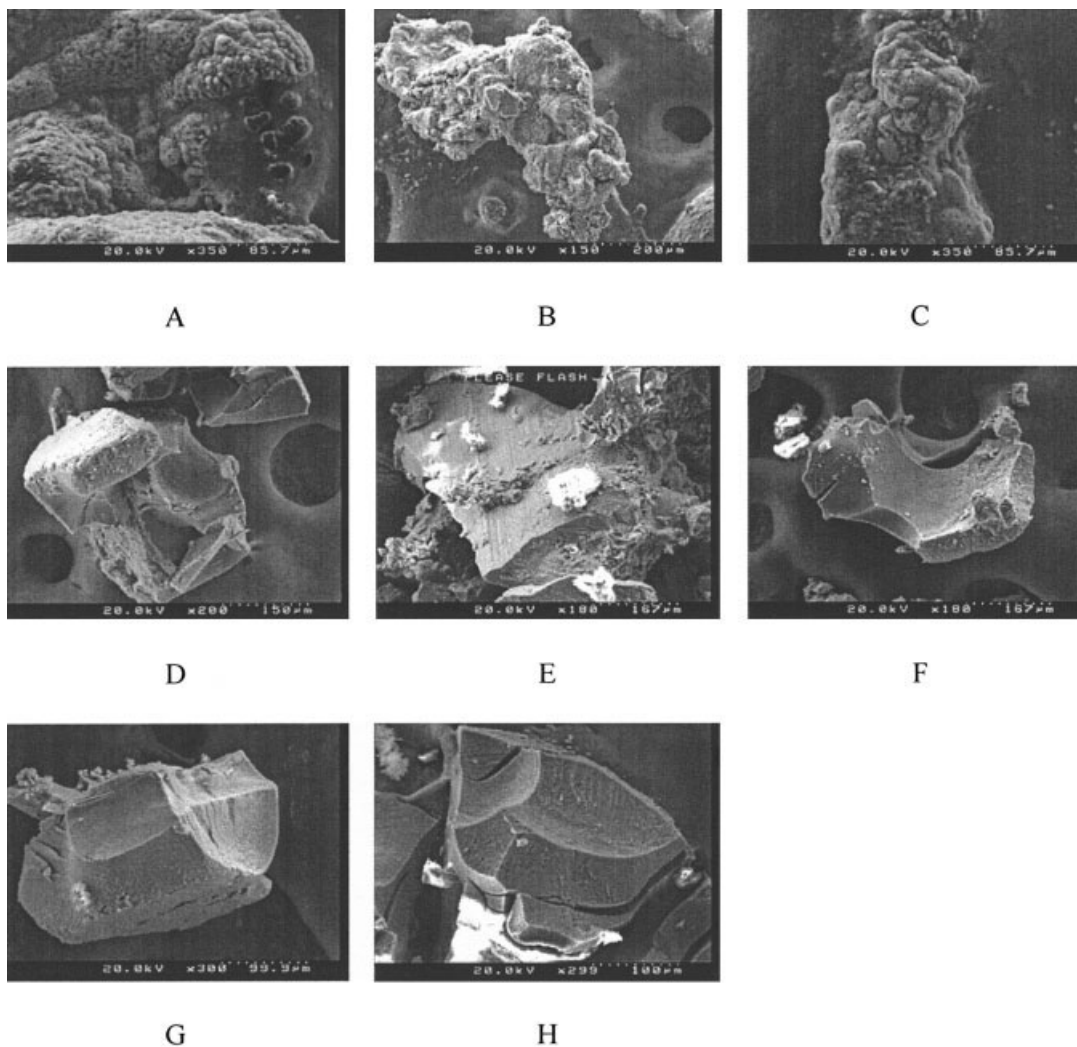

Figure 7. SEM micrographs of the composite deposits obtained under different thermohydraulic conditions, sugar concentration 25%: (A) Run 1, (B) Run 2, (C) Run 4, (D) Run 3, (E) Run 5, (F) Run 6, (G) Run 7, (H) Run 8.
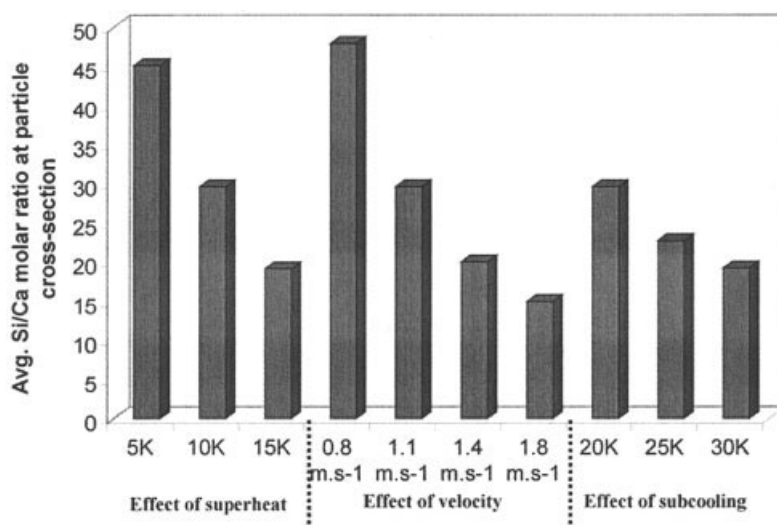
Figure 8. EDS analysis of Si/Ca molar ratios at particle cross sections of composite scales from various operating conditions at 25% sugar concentration.

the presence of high superheat (15 K), velocity (1.4–1.8 m/s), or subcooling (25–30 K), which exhibited disc- or ring-shaped morphologies (Figures 7D and F) and consisted primarily of cohesive structures with the loose layer on the edges and surfaces (Figures 7D–H). The observed structural transformation supported the general argument of deposit consolidation by COM by microlayer evaporation under high heat flux, which increased the fraction of the compact layer formed in the deposit. Furthermore, the results of the XRD analysis indicated the presence of both COM and COD together with SiO_2 as the main ingredient, consistent with data obtained previously from batch coprecipitation tests.[15]

EDS analysis was used to determine the elemental composition of the deposit as a function of thermohydraulic conditions under which the composite deposits formed, as shown in Figure 8. The data are presented as Si:Ca molar ratios at the cross sections of sample particles, which reflected the extent of deposit consolidation by CaOx and thus the deposit strength. It is evident (Figure 8) that the deposit strength increased with increasing surface superheat, velocity, and bulk subcooling, which confirmed the SEM results (Figure 7).

As indicated earlier, the influences of these operating parameters on deposit structure were *in essence* a consequence of the corresponding changes in the heat flux of the system, and potential models that could appropriately describe the deposit consolidation data needed at least to include such an effect. For this reason, a consolidation factor, β_{CaOx}, has been defined to quantify the effects of heat flux on the extent of deposit consolidation arising from the incorporation of CaOx

$$\beta_{CaOx} = \frac{1}{M_R} \quad (6)$$

where M_R is the Si/Ca molar ratio of deposit cross sections obtained under different superheat values. Figure 9 shows a linear relationship between deposit strength and heat flux.

It is of interest to note that at a superheat of 10 K, an increase in the velocity from 1.1 to 1.8 m/s was seen to increase the

deposit strength β_{CaOx} (Figure 8), whereas the fouling rate remained relatively constant (Figure 5 and Table 2). These results implied that velocity adjustment toward higher range (>1.1 m/s) during sugar processing would incur additional costs of energy consumption and cleaning arising from the higher tenacity of scales without having much impact on the rate of composite fouling in the evaporator. It should be recognized, however, that the actual structural transformations of the composite deposits could be much more complicated in sugar factories; if the presence of other nonsugar organic species and changes in the sugar concentration significantly altered the composite fouling behavior and deposit strength, for instance, then the measured threshold velocity of 1.1 m/s obtained in this work would simply have little practical value. Further work is currently under way to develop a composite fouling model incorporating key operating parameters and the mechanistic assumptions proposed herein.

Conclusions

This study has shown the effects of operating conditions on $CaOx/SiO_2$ composite fouling and the nature of the scales
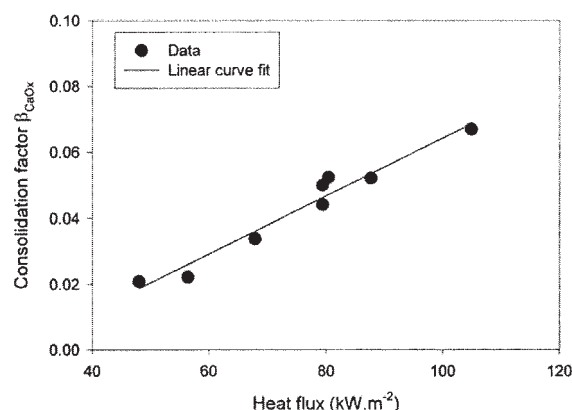

Figure 9. Effect of heat flux on deposit consolidation.

formed in synthetic sugar solutions. Three key operating parameters—surface superheat, fluid velocity, and bulk subcooling—were studied over ranges similar to those in the Australian sugar mill evaporators. The linear fouling rate was strongly dependent on the surface superheat, with the fastest fouling observed for the highest superheat. For a constant superheat of 10 K, the linear fouling rate was found to exhibit a maximum at a subcooling of about 25 K, but remained constant for fluid velocities 1.1 m/s or higher. These changes may be explained by bubble–particle interactions at the boiling metal surface, hydrodynamic lift forces at the wall of the fouled tube, and structural consolidation of the composite deposit. The composite scales formed under the current thermohydraulic conditions were shown to have a high bonding strength ($\sim$128 kJ/g mol), possibly arising from the structural consolidation by CaOx through microlayer evaporation. The extent of consolidation was found to increase with surface superheat, velocity, and bulk subcooling.

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