

EFFECTS OF TEMPERATURE CONDITIONS AND HEAT TREATMENT WITHIN A MULTIPLE EFFECT EVAPORATOR ON THIN STILLAGE FOULING CHARACTERISTICS

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ABSTRACT

In the fuel ethanol industry, evaporator fouling occurs when thin stillage is concentrated into a coproduct called condensed distillers solubles. Fouling affects the efficiency and environmental footprint of more than 200 biorefineries in the US. This study investigated: 1) effects of bulk temperature and initial probe temperature of the test apparatus on thin stillage fouling characteristics, 2) effects of exposure to evaporator heat treatment and 3) effects of facility shut down and cleaning on fouling characteristics. Experiments were conducted using model thin stillage (1% starch solution) and commercial thin stillage with varied temperature conditions. Increased initial probe temperatures increased fouling rates and maximum fouling resistances for commercial thin stillage and model thin stillage. At an initial probe temperature of 120°C, higher bulk temperature (80°C) increased fouling rates and reduced induction periods. Effects of exposure to evaporator heat treatment were studied by examining fouling behavior among samples from different locations within an evaporator. Effects of heat treatment were not detected. Samples before and after facility cleaning were collected to study effects of facility cleaning. Fouling tendencies were reduced after facility cleaning.

INTRODUCTION

The US Clean Air Act (1990) was established for reformulated gasoline to reduce air pollution. Ethanol and methyl tertiary butyl ether (MTBE) were approved as oxygenates and fuel additives. However, as the US Environmental Protection Agency phased out MTBE because of environmental and human health issues, ethanol became the only suitable fuel additive in the market. As a result, ethanol demand increased and ethanol production increased more than 10 times during the past 10 years, from 1.3 billion gal in 1994 to 14.3 billion gal in 2014 (RFA 2015). In 2015, there were more than 200 ethanol facilities in the US.

Fuel ethanol is made from corn by either dry grind or wet milling. In 2015, 90% of ethanol was produced from the dry

grind process (RFA 2015). In the dry grind process, nonfermentables (whole stillage) are centrifuged to separate soluble solids from insoluble solids. Thin stillage, the overflow from the centrifuge, is concentrated using multiple effect evaporators from 6 to 30% total solids to form a process stream known as syrup (condensed distillers solubles) which is mixed with wet grains and dried further to produce a coproduct called distillers dried grains with solubles (DDGS).

Heat transfer fouling is the accumulation and formation of unwanted materials on heat transfer surfaces. This impedes heat transfer and increases resistance to fluid flow. Fouling affects energy consumption of industrial processes and also results in frequent shut down and cleaning. There are five types of fouling mechanisms (Bott 1995), which makes fouling a complicated phenomenon. Awad (2011) estimated heat exchanger fouling costs consumed 0.25% of the US gross national product (US\$14.2 billion).

Heat transfer fouling increases capital investment to compensate for the reduced rate of heat transfer as well as increased operating costs to maintain desired temperatures and fluid conditions. Maintenance costs are increased to remedy effects of fouling (Bott 2007). Fouling of heat exchangers may cause environmental hazards and emissions (Muller-Steinhagen et. al. 2009).

In the corn dry grind process, fouling in evaporators provides resistance to heat transfer and restricts the flow of thin stillage. Fouling deposits must be removed periodically from the heat transfer surface. Periodical cleaning and maintenance results in increased capital, operation and maintenance costs. Understanding fouling tendencies would result in reduced labor costs, downtime and cleaning chemical costs (Agbisit et. al. 2003; Arora et. al. 2010; Wilkins et. al. 2006b).

Previous studies on thin stillage fouling included effects of Re, pH and membrane filtration as well as corn oil, carbohydrate and total solids contents (Agbisit et. al. 2003; Arora et. al. 2010; Challa et. al. 2015; Rausch et. al. 2013; Singh et. al. 1999; Wilkins et. al. 2006a; Wilkins et. al. 2006b). Among these studies, bulk temperatures varied from

40 to 75°C and initial probe temperatures varied from 100 to 120°C.

Because of the complex composition and variability of commercial thin stillage, Rausch et. al. (2013) developed model fluids to study fouling. Use of model fluids allowed controlled manipulation of thin stillage composition. Work by Challa and coworkers (2015, 2017) examined fouling characteristics of thin stillage and concentrates from different locations of a multiple effect evaporator in a dry grind facility. Total solids of samples varied from 7 to 11%. Fouling rates increased with increased solids concentration.

Objectives were to: 1) determine effects of bulk temperature and initial probe temperature of the test apparatus on fouling characteristics, 2) investigate effects of exposure to evaporator heat treatment and 3) observe effects of facility shut down and cleaning on fouling characteristics of samples from the thin stillage evaporator.

MATERIALS AND METHODS

Test Apparatus and Procedures

The fouling test apparatus was similar to that used in previous research (Agbisit et. al. 2003; Arora et. al. 2010; Challa et. al. 2015; Rausch et. al. 2013; Wilkins et. al. 2006a; Wilkins et. al. 2006b). The system consisted of an annular probe, 20 L batch tank, centrifugal pump and heat exchanger (Fig. 1). The system was used to detect fouling under accelerated conditions, which were achieved by recycling test fluid under temperature conditions (bulk temperature, T_b , of 60 to 80°C) similar to the temperature (75°C) typical of thin stillage from a dry grind facility; initial inner wall probe temperature, T_i , was 100 and 120°C. To amplify potential differences in fouling characteristics, surface temperature conditions were more severe than in multiple effect evaporators in a dry grind facility. Test fluids were circulated from a batch tank using a centrifugal pump (345 W, S-115 RZ, Iwaki Walchem, Iwaki, Japan). A water bath (20 L) and a cooling coil were used to maintain bulk temperature. The annular fouling probe (FIREROD 1025, Watlow, St. Louis, MO) consisted of a stainless steel (SS 316) outer tube containing a resistance heater (208 V, 2000 W).

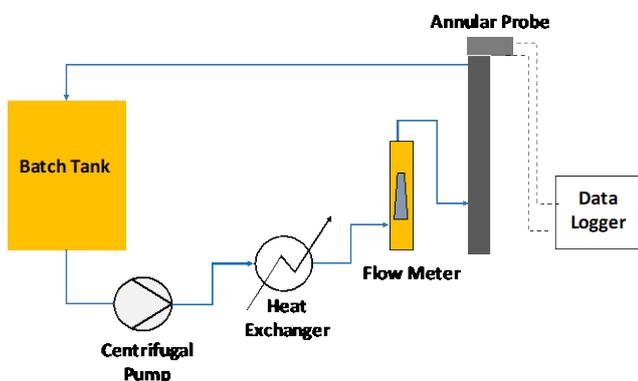


Fig. 1 Schematic of fouling test apparatus with annular fouling probe.

Fluid flow was through the annular space between the rod and outer housing tube. The rod contained an electrical

resistance heater and five type K thermocouples embedded in the inner wall of the rod. Four thermocouples were used to measure inner wall temperature (T_w) at four locations on the inner probe surface. The fifth thermocouple was used to shut off power supply to the heater rod when T_w reached 200°C. The annular geometry test section, has been used in several fouling studies (Agbisit et. al. 2003; Arora et. al. 2010; Challa et. al. 2015; Panchal and Watkinson 1993; Rausch et. al. 2013; Wilkins et. al. 2006a; Wilkins et. al. 2006b; Wilson and Watkinson 1996).

Each fouling test was operated at constant heat flux, velocity and bulk temperature. Using temperatures measured by the thermocouples, the overall heat transfer coefficient (U) was determined by:

$$U = \frac{q/A}{T_s - T_b} \quad (1)$$

Where q/A is the amount of heat transferred (power input) per unit area (W/m^2); T_s is probe surface temperature and T_b is fluid bulk temperature. T_s is calculated from the inner wall temperature T_w using Eq (2):

$$T_s = T_w - \left(\frac{x}{k}\right) \left(\frac{q}{A}\right) \quad (2)$$

Where x is the distance from the thermocouple to the probe surface and k is thermal conductivity of the probe metal. The x/k values were calculated for each thermocouple on the probe using the method of Wilson (1915), where a linear plot of $1/U$ vs V^{-n} (V is fluid velocity) is drawn using experimental data. Fouling resistance at time t (R_f) can be determined by heat transfer coefficients using Eq (3):

$$R_f = \frac{1}{U_t} - \frac{1}{U_0} \quad (3)$$

Where U_t (kW/m^2K) is the overall heat transfer coefficient at time t , U_0 is the initial ($t = 0$) overall heat transfer coefficient for a clean probe. By monitoring T_b and T_w , fouling resistance R_f at each time point can be calculated. During each test, fouling data (T_b , T_w and power input) were recorded every 1 min using a data logger.

Fouling resistance (R_f) was calculated as a moving average of three replicates for each data point and R_f vs time data were plotted to demonstrate overall fouling tendencies. Fouling rates for 1, 2 and 5 hr, were defined as R_f vs time linear regression lines for FR_1 , FR_2 and FR_5 , respectively. Induction period (IP) was defined as the period of time during which the 3 min moving average of R_f was less than $0.05 m^2 \cdot K/kW$ (Challa et. al. 2015). Maximum fouling resistance (R_{max}) was defined as the largest value of the 3 min moving average of fouling resistance during the 5 hr test period which also was used by Challa et. al. (2015, 2017). When deposits that have been formed break free from the probe, resistance to heat flow is decreased and R_f will decrease suddenly, a process called sloughing. This can be due to deposits having low adhesion to the deposit layers or probe surface. A sloughing point (SP) was defined as the point in time when R_f decreased abruptly by more than 30%. The fouling rate at the time of the first sloughing point (FR_s)

was defined as the slope of the regression line up to the first *SP*.

After each experiment, the fouling probe was removed from the outer tube and fouling deposits were removed partially using a plastic spatula. The probe was soaked in 5% (w/v) NaOH solution overnight to loosen residual deposits. Remaining deposits were removed using a wet sponge after soaking.

Means of fouling rates (FR_1 , FR_2 , FR_3), maximum fouling resistances (R_{max}) and induction periods (*IP*) were calculated. One-way and two-way ANOVA were used to compare means. Statistical analyses were performed using statistical software (RStudio 0.99.447, RStudio, Boston, MA) with a significance level of $p < 0.05$.

Experiment 1. Surface and bulk temperature effects on fouling characteristics

Commercial thin stillage. Thin stillage samples were collected from a commercial dry grind facility. Thin stillage samples were stored for a period of 1 to 2 week as in previous studies (Arora et. al. 2010; Singh et. al. 1999; Wilkins et. al. 2006a; Wilkins et. al. 2006b). Zheng (2013) stored commercial thin stillage samples at room temperature to and found no differences in fouling characteristics up to 20 days of storage. For this study, thin stillage samples were stored at room temperature ($15 \pm 5^\circ\text{C}$) and tested within 7 days. Five batches (50 L) were collected separately during a 2 month period with four tests conducted per batch. A 10 L subsample was used for each fouling test. Total solids were measured using a standard method (AACCI 2000).

Each fouling test was started after the system was cleaned. A commercial thin stillage (10 L) sample was added to the tank and was mixed by circulating at maximum flow rate (14 to 19 L/min) for 5 min. Sample volume was reduced to 7 L by draining. Water bath heated the sample to desired bulk temperature. Density and viscosity were measured after fluid reached T_b for each treatment. As Re was found to affect thin stillage fouling (Wilkins et. al. 2006a), Re was kept in a range of 460 to 520 for each treatment. Tap water was introduced in the heat exchange system to maintain T_b .

Treatments were arranged in a randomized complete block design with three replications for each treatment. T_b was adjusted to the desired treatment conditions ($60 \pm 2^\circ\text{C}$ and $80 \pm 2^\circ\text{C}$). After reaching desired T_b , flow rate was adjusted and probe power supply was turned on. Test was initiated when T_i reached desired conditions ($100 \pm 2^\circ\text{C}$ and $120 \pm 2^\circ\text{C}$). Each test lasted for a period of 5 hr. T_b of 80°C and T_i of 120°C were similar to temperature conditions Challa et. al. (2015) used ($T_b = 75^\circ\text{C}$, $T_i = 120^\circ\text{C}$). A stable T_i was difficult to maintain when T_b was lower than 60°C . The largest temperature difference between T_b and T_i for the system to be stable was 60°C .

Model thin stillage. Model fluids were used in previous work (Challa et. al. 2015; Rausch et. al. 2013) and found to be a repeatable experimental material. Model thin stillage using corn starch had rapid fouling compared with other carbohydrate mixtures. Regular yellow dent maize starch (obtained from Tate & Lyle, Decatur, IL, US) slurry (1%

w/v) was used as model thin stillage to study effects of T_b and T_i on fouling.

Tap water (7 L) was circulated and preheated to desired T_b (60 or 80°C) in the system. Starch (70 g) was added slowly into the tank to form 1% starch slurry. Slurry was circulated by the pump at maximum flow rate (15 L/min) for 30 min. Each experiment was started when the probe power was turned on and reached the desired T_i (100 or 120°C).

Experiment 2. Evaporator heat treatment effects on fouling characteristics of thin stillage

Samples from various locations within a multiple effect evaporator from a dry grind facility were collected and diluted to the same solids content ($7\% \pm 0.5$) to reduce influence of solids content. Samples were collected from the facility during a period of 90 days. Two batches of samples were collected before a scheduled complete facility cleaning and three batches were collected following the facility cleaning. The evaporation system had two effects; each effect contained four stages (Fig. 2). An oil skimming process took place between stages 7 and 8.

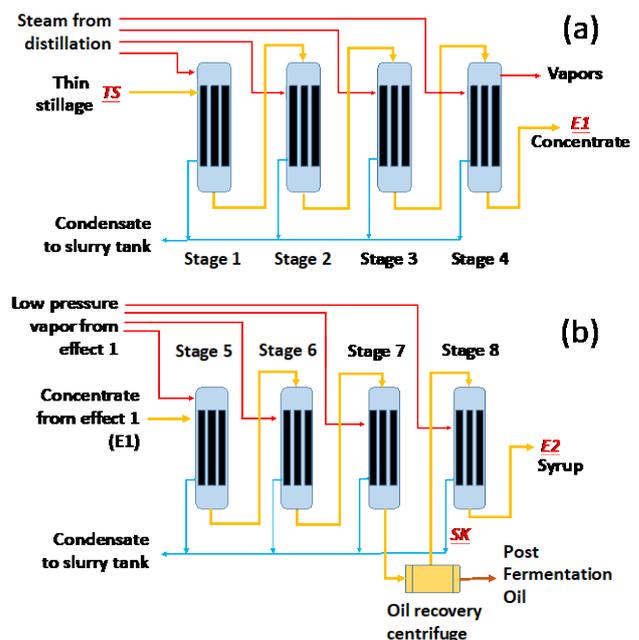


Fig. 2 Sample locations (TS, E1, SK, E2) within a multiple effect evaporator in a dry grind facility. (a) First effect, (b) Second effect (adapted from Challa 2014).

Samples (10 L) were collected at four locations (Fig. 2): thin stillage (TS) prior to the evaporator, concentrate from the end of effect 1 after stage 4 (E1), concentrate after skimming before entering stage 8 (SK) and concentrate from the end of effect 2 after stage 8 (E2), also known as condensed distillers solubles or syrup. Total solids contents were determined using a standard oven method (AACCI 2000). Samples (20 ml) were dried in a 49°C oven overnight (12 hr) and further dried in a 135°C oven for 2 hr. Three determinations were made. Before each fouling test, samples were diluted using tap water to $7 \pm 0.5\%$ solids content,

similar to thin stillage samples, were stored at room temperature ($15 \pm 5^\circ\text{C}$) and tested within 7 days.

Viscosity and density of the samples after diluting were tested when bulk temperature reached 75°C . Flow rates were adjusted (11 to 15 L/min) to maintain Re of 450 ± 50 . Fouling tests were started when T_b reached 80°C and T_i reached 120°C . Fouling tests lasted for 5 hr or when T_w reached 200°C . T_b was maintained constant during tests ($80 \pm 2^\circ\text{C}$).

Experiment 3. Comparison of fouling characteristics before and after facility shut down and cleaning

The entire fuel ethanol facility was shut down completely for 25 days for extensive cleaning and scheduled maintenance in addition to the routine evaporator cleaning which occurred every week. As described above, two batches were collected prior to cleaning and maintenance and three batches were collected following. Samples were collected from TS, E1, SK and E2 from within the evaporator (Fig. 2). Fouling characteristics before and after facility cleaning were compared. As a method to assess differences in fouling profiles (i.e., R_f vs time plots), linear regression was used to assess linearity of the profiles using a calculated R^2 value for each 5 hr fouling test. Batches 3, 4 and 5 were collected at 1, 2 and 4 weeks, respectively, after the facility resumed operation and presumed to be operating under steady conditions.

RESULTS AND DISCUSSION

Experiment 1. Surface and bulk temperature effects on fouling characteristics

Effects on commercial thin stillage. At higher T_i and T_b , fouling characteristics tended to increase (Fig. 3). When $T_i = 120^\circ\text{C}$ and $T_b = 80^\circ\text{C}$, fouling resistance increased rapidly during the first 2 hr. FR_1 and FR_2 were higher for $T_i = 120^\circ\text{C}$ and $T_b = 80^\circ\text{C}$ than for other treatments (Table 1); R_f did not increase after reaching an R_{max} of $0.47 \text{ m}^2\cdot\text{K}/\text{kW}$. A sudden decrease of more than 60% of fouling resistance was observed and attributed to deposit sloughing which also was observed by Challa et. al. (2017).

Rather than reaching a maximum during an intermittent time point, R_{max} ($0.36 \text{ m}^2\cdot\text{K}/\text{kW}$) was reached at the end of the 5 hr fouling tests for $T_i = 100^\circ\text{C}$ and $T_b = 60^\circ\text{C}$. For most replicates tested under these conditions, sloughing did not occur during the 5 hr test period.

IP ranged from 0.14 to 5 hr and generally increased as T_i or T_b were reduced. IP of 1.8 hr were observed for treatments at $T_i = 120^\circ\text{C}$ and $T_b = 60^\circ\text{C}$ (Fig. 3, Table 1).

Test conditions for the largest FR_1 , FR_2 , FR_5 , FR_S and R_{max} and shortest IP occurred at $T_i = 120^\circ\text{C}$ and $T_b = 80^\circ\text{C}$ (Table 1). Variation of means for FR , indicated by the coefficient of variation (CV, Table 2), were observed generally to increase as T_b and T_i conditions became less severe. This provided insight on temperature settings to use for future fouling tests; higher temperatures speeded data collection protocols as well as reduced variability in characteristics derived from the fouling data.

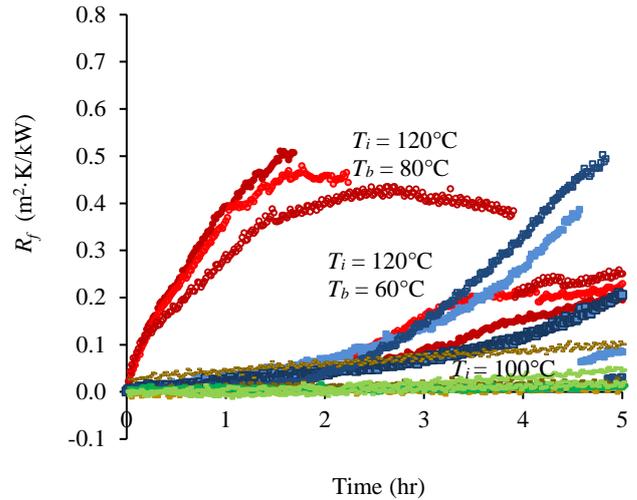


Fig. 3 Fouling resistance of commercial samples of three replicate samples. $T_i = 100$ or 120°C ; $T_b = 60$ or 80°C .

Table 1. Mean fouling rates, induction periods and maximum fouling resistance of commercial thin stillage.*

T_i/T_b^1	FR_1^2	FR_2	FR_5	FR_S^3	R_{max}^4	IP^5
120/80	0.380a	0.250a	0.0720a	0.259a	0.470a	0.14a
120/60	0.023b	0.022b	0.0470a	0.050b	0.360a	1.80b
100/80	0.021b	0.015b	0.0090b	0.0095b	0.045b	3.80c
100/60	0.0016b	0.0018b	0.0041b	0.0041b	0.028b	5.00c

*Means of three replicates, value with the same letter in the same column were not different ($p \leq 0.05$)

¹ T_i = initial probe and T_b = bulk temperatures ($^\circ\text{C}$) during tests

² FR_1 , FR_2 and FR_5 = fouling rate during 1, 2 and 5 hr testing, $\text{m}^2\cdot\text{K}/\text{kW}\cdot\text{hr}$

³ FR_S = fouling rate before deposit sloughing, $\text{m}^2\cdot\text{K}/\text{kW}\cdot\text{hr}$

⁴ R_{max} = maximum fouling resistance, $\text{m}^2\cdot\text{K}/\text{kW}$

⁵ IP = induction period, hr

Table 2. Coefficients of variation (CV, %) for fouling rates, induction periods and maximum fouling resistance of commercial thin stillage.

T_i/T_b^1	FR_1^2 CV	FR_2 CV	FR_5 CV	FR_S^3 CV	R_{max}^4 CV	IP^5 CV
120/80	15	16	35	40	0.08	110
120/60	5	45	37	36	0.40	30
100/80	150	130	130	129	1.1	54
100/60	240	530	73	73	0.67	--

¹ T_i = initial probe and T_b = bulk temperatures ($^\circ\text{C}$) during tests

² FR_1 , FR_2 and FR_5 = fouling rate during 1, 2 and 5 hr testing, $\text{m}^2\cdot\text{K}/\text{kW}\cdot\text{hr}$

³ FR_S = fouling rate before deposit sloughing, $\text{m}^2\cdot\text{K}/\text{kW}\cdot\text{hr}$

⁴ R_{max} = maximum fouling resistance, $\text{m}^2\cdot\text{K}/\text{kW}$

⁵ IP = induction period, hr

Effects on model thin stillage. At $T_i = 120^\circ\text{C}$ and $T_b = 80^\circ\text{C}$, fouling deposits accumulated rapidly indicated by the fouling rate ($FR_1 = 0.91 \text{ m}^2\cdot\text{K}/\text{kW}\cdot\text{hr}$). IP were less than 5 min (Fig. 4, Table 3). R_f decreased after R_{max} reached $0.71 \text{ m}^2\cdot\text{K}/\text{kW}$, indicating deposit removal from the probe surface was greater than the rate of deposition.

At $T_i = 120^\circ\text{C}$ and $T_b = 60^\circ\text{C}$, there were induction periods of approximately 1 hr (Table 3). Increases in R_f were small after reaching R_{max} of $0.36 \text{ m}^2\cdot\text{K}/\text{kW}$ (Fig. 4).

When T_i was 100°C , IP were longer than 5 hr for all tests. R_{max} was lower than $0.05 \text{ m}^2\cdot\text{K}/\text{kW}$. No observable deposits were found on the probe surface after the 5 hr tests.

T_i was a factor in fouling characteristics in the aspect of fouling rates (1, 2 and 5 hr), R_{max} and IP . T_b was significant in FR_1 and FR_2 fouling rates but not for FR_5 . This corresponded with previous analysis that fouling rates and R_{max} increased with the increase of initial probe temperature, while induction periods decreased. Decrease of T_b resulted in a decrease of R_{max} and increased IP (Table 3). R_{max} of $0.71 \text{ m}^2\cdot\text{K}/\text{kW}$ was higher than that reported by Challa et al. (2015) ($0.41 \text{ m}^2\cdot\text{K}/\text{kW}$) with the same T_i (120°C) and slightly lower T_b (75°C).

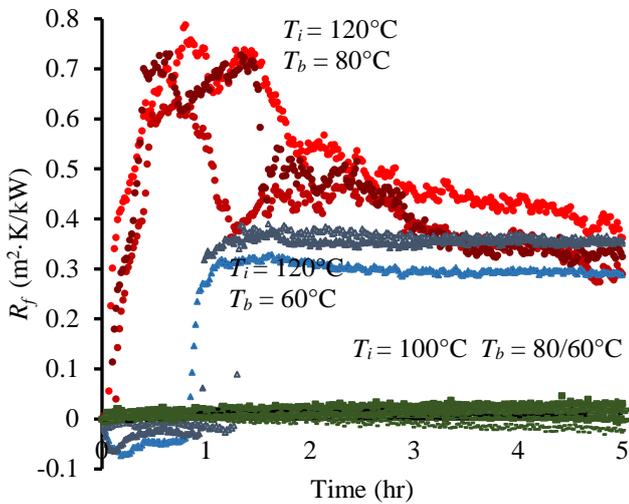


Fig. 4 Fouling test temperature effects (T_i and T_b) on fouling profiles of model thin stillage.

Table 3. Mean fouling rates and maximum fouling resistances of model thin stillage from various temperature conditions.*

T_i/T_b^1	FR_1^2	FR_2	FR_5	R_{max}^3	IP^4
120/80	0.91a	0.42a	0.13a	0.71a	0.083a
120/60	0.0087b	0.18b	0.068a	0.36b	1.03b
100/80	0.012b	0.0077c	0.00043b	0.020c	5.0c
100/60	0.0089b	0.0044c	0.0043b	0.023c	5.0c

*Means of three replicates, values with the same letter in the same column were not different ($p \leq 0.05$)

- ¹ T_i = initial probe and T_b = bulk temperatures ($^\circ\text{C}$) during tests
- ² FR_1 , FR_2 and FR_5 = fouling rate during 1, 2 and 5 hr testing, $\text{m}^2\cdot\text{K}/\text{kW}\cdot\text{hr}$
- ³ FR_5 = fouling rate before deposit sloughing, $\text{m}^2\cdot\text{K}/\text{kW}\cdot\text{hr}$
- ⁴ R_{max} = maximum fouling resistance, $\text{m}^2\cdot\text{K}/\text{kW}$
- ⁵ IP = induction period, hr

Experiment 2. Evaporator heat treatment effects on fouling characteristics of commercial thin stillage (before and after facility shut down)

Before facility shut down and cleaning. For samples collected before cleaning, deposits began to accumulate

rapidly during fouling tests, indicated by a rapid increase of fouling resistance and with large fouling rates (before deposit sloughing) of 0.23 to $0.86 \text{ m}^2\cdot\text{K}/\text{kW}$ (Table 4, Fig. 5). IP were less than 0.083 hr (5 min). Due to high variability in replicate tests, no differences were detected among sample locations (TS, E1, SK, E2). The cause of the batch-to-batch variability is not known. Among fouling rates calculated (FR_1 , FR_2 , FR_5 and FR_S), FR_S would be the best to illustrate fouling behavior prior to deposit sloughing. When sloughing occurred, there was a decrease of fouling resistance (more than 30%), resulting in decreased fouling rates.

After reaching maximum fouling resistance, deposits often would slough off the probe surface as indicated by a sudden decrease of R_f in the fouling curve (Fig. 5). Deposit sloughing took place about 1 hr after tests started. A higher fouling rate was observed before the sloughing took place for SK and E2 samples compared with TS and E1 samples. Those samples (SK and E2) tended to have more complete sloughing, indicated by fouling resistance values that decreased to less than $0.05 \text{ m}^2\cdot\text{K}/\text{kW}$.

Table 4. Fouling parameters for samples with the evaporator before facility cleaning (see Fig. 2 for locations).**

Location	FR_1^1	FR_2	FR_5	FR_S^3	FSP^4	R_{max}^5	IP^6
TS	0.27	0.17	0.066	0.23	2.65	0.38	0.083
E1	0.47	0.14	0.067	0.46	1.04	0.43	0.008
SK	0.55	0.15	0.081	0.73	0.87	0.47	0.008
E2	0.43	0.14	0.091	0.86	0.80	0.52	0.008

- **Mean value of two tests.
- ¹ T_i = initial probe and T_b = bulk temperatures ($^\circ\text{C}$) during tests
- ² FR_1 , FR_2 and FR_5 = fouling rate during 1, 2 and 5 hr testing, $\text{m}^2\cdot\text{K}/\text{kW}\cdot\text{hr}$
- ³ FR_S = fouling rate before deposit sloughing, $\text{m}^2\cdot\text{K}/\text{kW}\cdot\text{hr}$
- ⁴ FSP = time of first sloughing point, hr
- ⁵ R_{max} = maximum fouling resistance, $\text{m}^2\cdot\text{K}/\text{kW}$
- ⁶ IP = induction period, hr

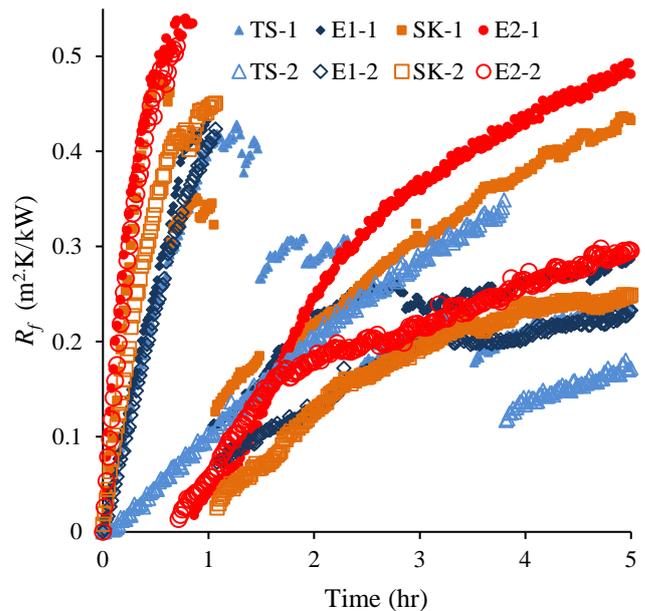


Fig. 5 Fouling resistance of thin stillage and evaporator concentrates before facility cleaning. TS, E1, E2, SK described in Fig. 2. “1” or “2” denote separate batch results. $T_i = 120^\circ\text{C}$ and $T_b = 80^\circ\text{C}$ during each test.

After facility shut down and cleaning. There were no differences among fouling parameters (FR_1 , FR_2 , FR_5 and FR_S) due to high variability among replicate batches. Although not conclusive from these data, FR_5 would be a useful parameter to characterize fouling. From the mean fouling curve (Fig. 6), we observed thin stillage had slower accumulation of fouling deposits compared with concentrates (E1, SK and E2). TS mean fouling rate was 0.018 ($m^2 \cdot K/kW \cdot hr$) and E2 had a mean fouling rate of 0.066 $m^2 \cdot K/kW \cdot hr$ (Table 5). Induction periods of TS were 0.65 hr (40 min), while induction periods of concentrates were 0.10 to 0.31 hr (15 to 17 min) (Table 5). Maximum fouling resistance was 0.19 $m^2 \cdot K/kW$ which was lower than R_{max} before cleaning (0.52 $m^2 \cdot K/kW$) (Table 4).

Fouling behavior of the samples collected at same locations varied from batch to batch. An average coefficient of variation (CV) of more than 50% was observed among batches. E1 samples had the largest level of variation (more than 90% CV for fouling rate and maximum fouling resistance). E2 samples had the smallest level of variation (30%) among three batches.

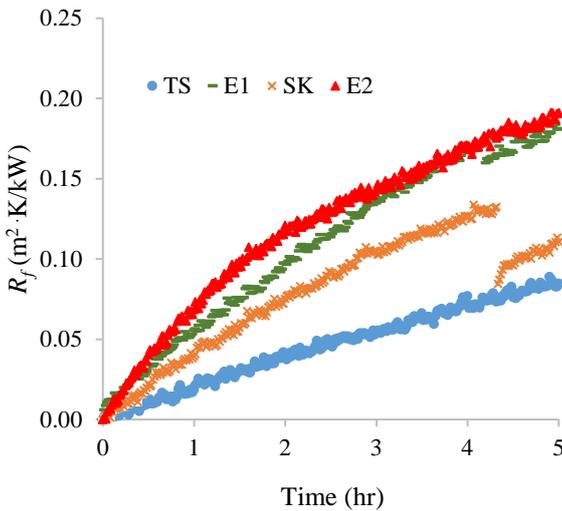


Fig. 6 Mean fouling resistance vs time for samples after cleaning. Means are from three observations. Sample locations described in Fig. 2. $T_i = 120^\circ C$ and $T_b = 80^\circ C$ during each test.

Table 5. Fouling parameters of thin stillage and concentrates after cleaning. **

Location	FR_1^1	FR_2	FR_5	FR_S	FSP^2	R_{max}^3	IP^4
TS	0.019	0.020	0.018	0.018	>5	0.089	0.65
E1	0.06	0.051	0.042	0.042	>5	0.19	0.31
SK	0.042	0.039	0.030	0.033	>5	0.14	0.27
E2	0.073	0.064	0.045	0.045	>5	0.19	0.096

**Mean value of three tests. $T_i = 120^\circ C$ and $T_b = 80^\circ C$ during each test. No differences were detected among all treatment means ($p \leq 0.05$)
¹ FR_1 , FR_2 , FR_5 = fouling rates after 1, 2, and 5 hr of testing, respectively,
 FR_S = fouling rate before first sloughing time point, $m^2 \cdot K/kW \cdot hr$
² FSP = first sloughing point, hr
³ R_{max} = maximum fouling resistance, $m^2 \cdot K/kW \cdot hr$
⁴ IP = induction period, hr

Experiment 3. Comparison of fouling characteristics before and after facility shut down and cleaning

R_f were generally lower following the plant shut down and cleaning period (Fig. 6). Prior to cleaning, R_{max} ranged from 0.38 to 0.52 $m^2 \cdot K/kW \cdot hr$ (Table 4), whereas following cleaning, R_{max} means for the four locations ranged from 0.089 to 0.19 $m^2 \cdot K/kW \cdot hr$ (Table 5). Fouling rates and maximum fouling resistances of all four samples decreased after the facility shut down and cleaning (Fig. 7). Fouling rates before deposit sloughing for each sample at the same location were different before and after facility shut down and cleaning. R_{max} of TS, SK and E2 samples were different before and after facility shut down and cleaning; for E1, R_{max} means were not different. Both fouling rates and maximum fouling resistances decreased after facility cleaning. FR_S decreased by more than 90% after facility shut down and cleaning. R_{max} decreased by more than 50%. FR_1 , FR_2 and FR_5 decreased after facility cleaning at each sample location, while IP increased (Table 4 and 5).

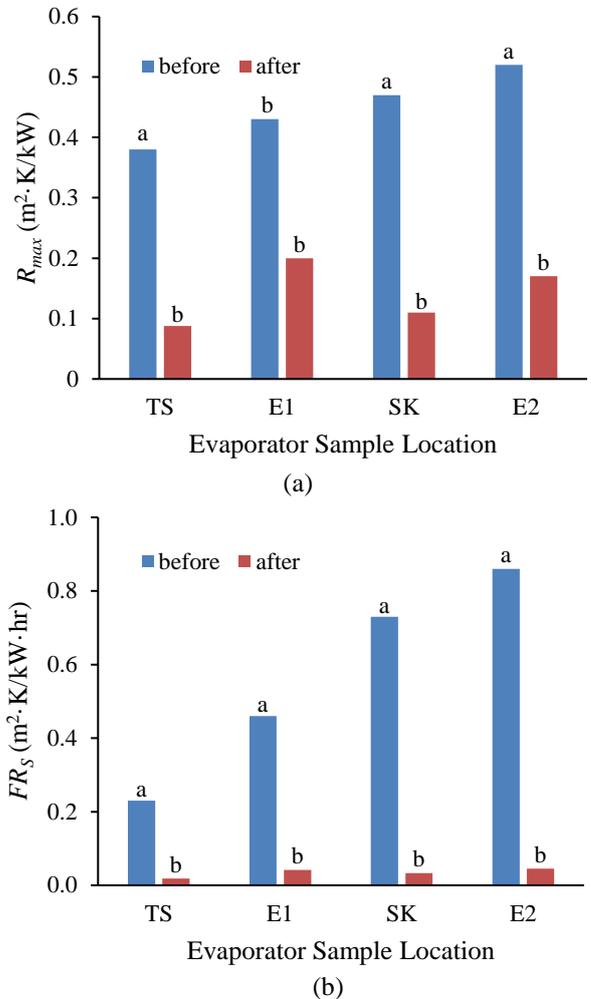


Fig. 7 (a) R_{max} and (b) FR_S for samples from various locations within an evaporator before and after facility cleaning. Letters distinguish means (before and after) within the same location ($p < 0.05$). Sample locations described in Fig. 2.

Fouling curves of samples tested after cleaning were linear ($R^2 = 0.98$) while the fouling curves of samples tested before cleaning were less linear ($R^2 = 0.73$). Fouling curves of samples were more linear ($0.96 \leq R^2 \leq 0.99$) after cleaning than before cleaning ($0.71 \leq R^2 \leq 0.76$).

Sloughing rarely was observed during these experiments. Only one sloughing point was seen during the fouling tests after cleaning. There were less sloughing points after facility cleaning (1 points) than before facility cleaning (10 points). This may indicate that deposits were thicker, or had lower adhesion to the probe surface or were softer in general, allowing more sloughing for fluids tested prior to facility cleaning.

Even though samples were collected 1 to 4 week after facility start up (and thought to be collected during steady conditions), shut down and cleaning were factors influencing fouling characteristics. It is theorized that some as yet unmeasured components take time, on the order of several weeks, to accumulate within the process and affect fouling of the evaporator surfaces.

CONCLUSIONS

- T_i and T_b had effects on commercial and model thin stillage fouling characteristics. Fouling rates and R_{max} increased with the increase of T_i for commercial and model thin stillage.
- For model thin stillage, increasing T_b increased R_{max} . T_b did not affect R_{max} for commercial thin stillage. Lower T_b increased IP in commercial and model thin stillage fouling.
- For $T_i = 120^\circ\text{C}$ and $T_b = 80^\circ\text{C}$, rapid fouling was observed for commercial and model thin stillage samples. For $T_i = 100^\circ\text{C}$ and $T_b = 80$ and 60°C , little or no fouling occurred for both test samples.
- For commercial thin stillage, $T_i = 120^\circ\text{C}$ and $T_b = 80^\circ\text{C}$ were recommended temperature conditions for future fouling test protocols because of rapid fouling and less variation in fouling parameters. For future fouling tests of model thin stillage, $T_i = 120^\circ\text{C}$ and $T_b = 60^\circ\text{C}$ would be recommended temperature conditions because of the rapid fouling and repeatable induction periods.
- Large variations of fouling parameters among batches were observed for samples collected both before and after facility cleaning. No differences among the different heat treatment samples were detected.
- Facility shut down and cleaning had effects on fouling characteristics of thin stillage and concentrates weeks following startup. Fouling rates and R_{max} decreased and induction period increased for samples collected after facility cleaning. Facility shut down and cleaning reduced fouling in general.
- Future work is needed to study changes of thin stillage composition after facility cleaning that affect fouling.

NOMENCLATURE

- A heated area of probe, m^2
 E1 sample location, concentrate from end of evaporator effect 1 after stage 4

- E2 sample location, concentrate from end of evaporator effect 2 after stage 8, also known as condensed distillers solubles
 FR fouling rate, $\text{m}^2\cdot\text{K}/\text{kW}\cdot\text{hr}$
 FSP time of first sloughing point, hr
 IP induction period, hr
 k thermal conductivity of probe metal
 q power input, W
 q/A heat flux to probe, W/m^2
 R_f fouling resistance, $\text{m}^2\cdot\text{K}/\text{kW}$
 R_{max} maximum fouling resistance, $\text{m}^2\cdot\text{K}/\text{kW}$
 Re Reynolds number
 SK sample location, concentrate after skimming before entering state 8 of evaporator
 SP time of sloughing, when deposits abruptly detach from probe surface, hr
 T temperature, $^\circ\text{C}$
 TS initial thin stillage from dry grind process
 T_s probe surface temperature, $^\circ\text{C}$
 T_b fluid bulk temperature, $^\circ\text{C}$
 T_w inner wall temperature of probe, $^\circ\text{C}$
 U overall heat transfer coefficient, $\text{kW}/\text{m}^2\cdot\text{K}$
 U_t heat transfer coefficient at time t , $\text{kW}/\text{m}^2\cdot\text{K}$
 U_0 heat transfer coefficient of clean probe, $\text{kW}/\text{m}^2\cdot\text{K}$
 x distance from the thermocouple to the probe surface
 x/k ratio determined experimentally by method of Wilson (1915)

Subscript

- $I, 2, 5, S$ rate at 1, 2 and 5 hr and time of sloughing, respectively
 b bulk
 i initial
 s surface
 w inner wall

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