

EVAPORATOR FOULING TENDENCIES OF THIN STILLAGE AND CONCENTRATES FROM THE DRY GRIND PROCESS

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ABSTRACT

In the US, more than 90% of fuel ethanol production is from the dry grind process. More than 200 maize processing plants use multiple effect evaporators to remove water from thin stillage and steepwater during dry grind and wet milling processes, respectively. During the dry grind process, unfermentables are centrifuged and the liquid fraction, thin stillage, is concentrated in multiple effect evaporators. Evaporator fouling occurs during thin stillage concentration and may be from deposition of proteins, fat, fiber and/or carbohydrates on evaporator surfaces. The consequences of fouling include increased capital costs, operating costs and environmental footprint of cleaning chemical disposal. Studies on evaporator fouling from maize processing streams are limited and fundamental causes are not well understood. Therefore, the overall objective was to investigate effects of compositional variation on evaporator fouling during thin stillage concentration.

Total solids effects were studied using thin stillage and evaporator concentrates. Many ethanol plants recover post fermentation corn oil from evaporator concentrates; therefore, the effects of oil recovery on evaporator fouling also was studied. The effects of oil content on thin stillage fouling was studied using thin stillage and post fermentation oil added to thin stillage as testing fluids. Glycerol accumulation in processing streams from thin stillage recycling was observed in earlier literature; therefore, experiments were conducted to compare the fouling rates when glycerol was added to thin stillage. Thin stillage (7% solids) had lower fouling rates compared to evaporator concentrates (8 to 11% solids). Simultaneous plant shutdown and evaporator cleaning decreased fouling rates. Addition of post fermentation corn oil (0.5 to 1.0%) increased thin stillage fouling rates but at higher oil concentration (1.5% added), fouling rates decreased. At 10% solids content in evaporator concentrates, oil recovery had no influence on fouling rates. Glycerol addition (1%) to thin stillage increased fouling rates.

INTRODUCTION

Fouling is defined as deposit formation on heat transfer surfaces, which increases costs from energy consumption, maintenance, labor and cleaning chemicals (Epstein 1981). Fouling increases pressure losses inside heat transfer equipment and decreases heat transfer rates (Taborek et al 1972). Following ethanol distillation, the remaining material is called whole stillage. Centrifugation of whole stillage produces thin stillage as the supernatant and wet grains. Thin stillage is concentrated in evaporators and mixed with wet grains to produce distillers dried grains with solubles (DDGS). Evaporator fouling is caused by materials that are present in thin stillage, i.e., carbohydrates, protein, fiber, fat and minerals.

Falling film evaporators are used in the food industry due to higher heat transfer coefficients at low evaporating temperatures and short residence times. They are economical, reliable, operate continuously and can handle large volumes of process streams (Wiegand 1971). In processing plants, instead of a single falling film evaporator, multiple effect evaporators are used to increase evaporator capacity and to reduce steam consumption. An operating parameter that affects heat transfer in a falling film evaporator is solids concentration (Chen and Jebson 1997). Total solids contents of thin stillage increase from 5 to 30% as material passes through each evaporator effect (Rausch and Belyea 2006; Singh et al 1999).

Despite longstanding problems of evaporator fouling in the corn ethanol industry, there have been relatively few studies to understand thin stillage fouling (Arora et al 2010; Challa et al 2015; Rausch et al 2013; Singh et al 1999; Wilkins et al 2006a; Wilkins et al 2006b). The pH effects on thin stillage fouling with dry matter (dissolved and suspended) ranging between 7.3 and 9.5% were investigated (Wilkins et al 2006b). Among replicates, there was variability in fouling characteristics, perhaps due to total solids content. Arora et al (2010) tested the effects of thin

stillage solids content on fouling by using microfiltration to lower total solids, protein and fat contents. Arora et al (2010) reported total solids (6.5% db), dissolved solids (3.0% db) and suspended solids (3.6% db) in thin stillage. They also used diluted thin stillage for fouling tests; diluted thin stillage was prepared by mixing commercial thin stillage with distilled water to solids levels similar to microfiltered thin stillage. Microfiltration reduced the fouling resistance by 50% when solids level decreased from 7.2 to 3.5%. When compared to commercial thin stillage, induction periods were longer and increased by 9.5 and 4.3 times due to microfiltration and dilution, respectively. However, ethanol plants do not have a filtration step prior to thin stillage evaporation and effects of solids concentration in evaporator effects is yet to be investigated.

Earlier fouling studies on thin stillage fouling had bulk temperatures of 60°C and were lower than starch gelatinization temperatures (63 to $70^{\circ}\text{C}</math>). These lower bulk temperatures were less representative of commercial temperatures of about 70 to $80^{\circ}\text{C}</math>; temperatures of 40 to $60^{\circ}\text{C}</math> increases the temperature difference between fouling probe surface temperature and bulk temperature as well as affecting thin stillage viscosity. Therefore, conditions during testing and resulting fouling rates of thin stillage determined in earlier studies may not represent accurately industrial fouling rates.$$$

Thin stillage characteristics may be influenced from evaporator and other processing equipment cleaning because solids accumulation was related to recycling of thin stillage. Inconsistency in test results or the lack of repeatability in fouling rates and induction periods were observed in an earlier study; the causes were not understood (Wilkins et al 2006a). We found thin stillage collected at three times within a month from a dry grind facility did not foul during testing (unpublished data). Prior to that time, the plant had been completely shut down for maintenance and cleaning.

In a dry grind plant, it is typical to recover post fermentation corn oil from concentrated thin stillage. This corn oil can be recovered using centrifugation, heating and condensation of thin stillage (Moreau et al 2010). Recovered oil can be used for biodiesel production.

Singh et al (1999) investigated effects of oil in thin stillage on fouling characteristics. Refined corn oil addition to dry grind ethanol thin stillage decreased the fouling rates. Commercial thin stillage had fouling rates higher than thin stillage with 1.0 and 1.5% refined oil added. This was attributed to thin stillage having 16.5% db of unrefined corn oil and addition of refined oil may have altered adhesion of lipids to metal surfaces. Copolymerization between lipids and proteins may foul the heat transfer surfaces (Lund and Sandu 1981). Heat transfer surface characteristics were altered by lipid adsorption onto metal surface by transforming high energy metal surfaces into unwettable or autophobic surfaces (Lund and Sandu 1981).

It is unknown whether oil separation at the final stages of an evaporator affects thin stillage fouling. Post fermentation corn oil has 11 to 16% free fatty acids; whereas in refined corn oil free fatty acids are removed completely (Moreau et al 2010). Wax esters and steryl esters in post fermentation corn oil ranged between 3.6 and 4.3%. Therefore, the objective of this study were to investigate the effects of post fermentation corn oil to commercial thin stillage and also the effect of oil separation from concentrates on evaporator fouling.

There are other factors, such as thin stillage recycling, which may influence fouling. Fermentation byproducts such as glycerol accumulate in dry grind process streams from thin stillage recycling (St. Julian et al 1990). Glycerol content increased in fermentation mash with successive recycling of thin stillage but decreased after recycle six (St. Julian et al 1990).

The objectives were to (1) determine fouling rates of commercial thin stillage and concentrates collected from stages of a multiple effect evaporator, (2) observe the added effects of post fermentation corn oil and glycerol on thin stillage fouling and (3) determine the effects of corn oil recovery.

MATERIALS AND METHODS

Samples from a dry grind ethanol plant

Total solids content in samples varied between 7.33 and 11.26% db (Table 1). Solids concentrations varied by $\pm 1\%$ db during 24 h; therefore, test samples were diluted with tap water such that the increment in solids content between stages was constant at 1% (Table 1).

Table 1. Treatments in thin stillage fouling tests.

Treatment	Total solids as sampled (% db) [†]	Adjusted total solids (% db) [†]	Viscosity (cP) [†]
TS	7.33 \pm 0.16	7	15.65 \pm 0.32
S1	8.33 \pm 0.27	8	17.13 \pm 0.71
S2	9.12 \pm 0.15	9	18.78 \pm 1.04
S3	11.63 \pm 0.15	10	22.04 \pm 0.25
S4	11.26 \pm 0.47	11	27.92 \pm 2.95

TS = thin stillage. S1, S2, S3, S4 = concentrates from stages 1, 2, 3 and 4, respectively. [†]Mean of two samples.

Samples were collected before and after scheduled evaporator cleaning and plant shutdown. All samples were stored at $4^{\circ}\text{C}</math> prior to testing. Processing conditions caused variability in total solids content in thin stillage and concentrate from each stage (Fig. 1). Thin stillage and concentrate from evaporator effects were collected during a 2 month period with the first batch of samples collected one week before schedule evaporator cleaning. The second batch of samples was collected one month after evaporation system cleaning.$

For studies related to oil recovery effects on evaporator concentrate fouling, samples were collected from stage 7, before oil recovery (Fig. 2), and were called unskimmed condensed distillers solubles (UCDS). Samples from stage 8 after oil recovery were called skimmed condensed distillers solubles (SCDS). Thin stillage and concentrates total solids were determined by standard oven method (AACC 2000). A 50 mL sample was oven dried at 49°C for 24 h, then dried further at 135°C for 2 h. Samples were filtered using a 0.2 μ filter to separate particulate matter. Suspended solids in thin stillage was found to be 50% of total solids. Thin stillage, UCDS and SCDS compositions were determined using HPLC methods (Table 2).

Table 2. Composition of thin stillage samples (% w/v) before and after plant cleaning cycles.†

Component	Thin Stillage (before)	Thin Stillage (after)	UCDS	SCDS
DP4+	0.22 ±0.003	0.21 ±0.004	1.01 ±0.152	1.23 ±0.199
DP3	0.20 ±0.020	0.21 ±0.001	0.68 ±0.075	0.81 ±0.088
Maltose	0.45 ±0.070	0.55 ±0.017	1.50 ±0.055	1.79 ±0.060
Glucose	0.03 ±0.002	0.12 ±0.005	0.30 ±0.090	0.36 ±0.110
Glycerol	1.28 ±0.054	1.35 ±0.003	4.52 ±0.662	5.40 ±0.760
Lactic acid	0.10 ±0.010	0.29 ±0.014	0.61 ±0.149	0.74 ±0.181
Acetic acid	0.06 ±0.003	0.07 ±0.005	0.20 ±0.003	0.26 ±0.011

†Mean of two samples ± 1 standard deviation. DP = degree of polymerization. UCDS = unskimmed condensed distillers solubles. SCDS = skimmed condensed distillers solubles.

Due to pump limitations on the test apparatus, total solids of UCDS and SCDS were adjusted to 10% solids concentration using tap water. Post fermentation corn oil recovered from evaporator concentrate was sent to a commercial lab for compositional analysis (Table 3). During fouling test experimentation, oil was added to a batch of thin stillage (0.50, 1.0 or 1.5% db) as the material recirculated in the test loop, prior to activating the probe heater.

Table 3. Composition of skimmed crude corn oil.

Moisture (%)	1.63
Unsaponifiable matter (%)	1.61
Total fatty acid (%)	96.7
Free fatty acid (%)	9.83
Iodine value	117
Sulfur (ppm)	8.4
Phosphorus (ppm)	8.2
Acid value (mg KOH/g fat)	19.05

The evaporation system at this facility had two effects. The first effect consisted of four falling film evaporators

connected in series to form four stages (Fig. 1). Initial thin stillage samples were collected prior to evaporation.

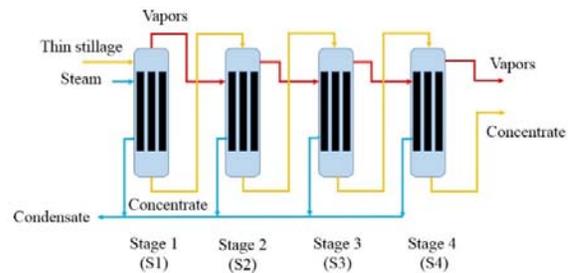


Fig. 1. First effect of a multiple effect evaporator in a dry grind ethanol plant.

The second effect also consisted of four stages; four evaporators in the second effect were connected in series but a post fermentation corn oil separation step was included in ethanol plants where they recover oil from evaporator concentrate by centrifugation (Fig. 2).

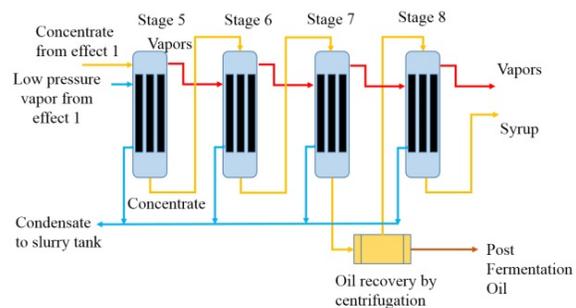


Fig. 2. Second effect of a multiple effect evaporator in a dry grind ethanol plant. Post fermentation corn oil recovery from evaporator concentrate by centrifugation.

Fouling test apparatus

The same fouling apparatus and annular probe were used from previous study (Challa et al 2015) using 7 L batches. The annular test section consisted of an inner stainless steel rod within a concentric stainless steel tube. The rod contained an electrical heater and five thermocouples imbedded in the inner wall of the probe (Table 4). The test apparatus consisted of the annular probe, batch tank, heat exchanger (to control bulk temperature), and pump. Fouling tests were conducted at bulk fluid temperature $75 \pm 2^\circ\text{C}$, laminar flow rate ($Re = 360 \pm 10$) and initial probe surface temperature $120 \pm 2^\circ\text{C}$. These temperature and flow conditions approximate those of evaporators in the fuel ethanol industry. Heat transfer fouling resistance (R_f) was measured from the change in overall heat transfer coefficients. R_f was determined experimentally from the overall heat transfer coefficients of fouled and unfouled probe surfaces (Arora et al 2010; Challa et al 2015; Rausch et al 2013; Wilkins et al 2006a; Wilkins et al 2006b).

$$R_f = \frac{1}{U_{fouled}} - \frac{1}{U_{unfouled}} \quad (1)$$

where U_{fouled} was the overall heat transfer coefficient of fouled probe surface at time t and U_{unfouled} was the clean overall heat transfer coefficient of clean probe at time $t = 0$.

The overall heat transfer coefficient was calculated from

$$U = \frac{Q/A}{T_s - T_b} \quad (2)$$

where Q/A was the heat flux at the probe surface ($A = 0.004 \text{ m}^2$) calculated from the power input ($Q = 410 \pm 10 \text{ W}$) to resistance heater inside the annular probe. T_b was the bulk temperature of the testing fluid and T_s was the outer surface temperature of the probe. T_s was determined from the mean thermocouple temperature (T_w) inside the probe.

$$T_s = T_w - \left(\frac{x}{k}\right) \left(\frac{Q}{A}\right) \quad (3)$$

The ratio of the distance of thermocouple from the probe surface (x) and thermal conductivity (k) was determined using a graphical method (Wilson 1915). The x/k values for four thermocouples were 0.0749, 0.1095, 0.0971 and 0.0976 $\text{m}^2\text{K}/\text{kW}$. The annular probe (FIREROD 1025, product no. SJ24AX-2835) was purchased from Watlow, St. Louis, MO. Design specifications were obtained from the manufacturer (Table 4).

Table 4. Dimensions of the annular fouling probe.

Parameters	Annular probe
Material	SS 316
Diameter (mm)	12.6
Annulus outer diameter (mm)	25.4
Heated length (mm)	101.6
Length from thermocouple location to leading edge (mm)	222.0
Length from entrance to thermocouple location (mm)	286.0

Cleaning procedure

Fouling apparatus and annular probe were cleaned following each test. Fouling deposits were removed with a wet sponge and the annular probe was soaked overnight in 5% (w/v) NaOH solution. Remaining deposition on the probe was removed the next day. Fouling apparatus was cleaned by recirculating 1% (w/v) hot detergent solution (Alconox, New York, NY), followed by a hot water (150 L) rinse.

Statistical analyses

Statistical analyses were performed using statistical software (v.9.4, SAS Institute, Cary, NC, USA) with a significance level of $p < 0.05$. Fouling rate was determined as the slope of the regression line (R_f vs t) from $t = 0$ until the end of test period, 5 h. Induction period was the time from

the beginning of the test ($t = 0$) until the 1 min moving average of R_f was greater than $0.01 \text{ m}^2\cdot\text{K}/\text{kW}$. Mean fouling rates, clean overall heat transfer coefficients of thin stillage and oil added thin stillage were compared using one way ANOVA with the least significant difference method.

RESULTS

Thin stillage and concentrate fouling

Thin stillage had lower fouling resistance compared to the concentrate collected from stages 1, 2, 3 and 4 (Fig. 3). Sloughing of deposits occurred for concentrate collected from stages 3 and 4 (point \emptyset in Fig. 3). No induction periods were observed for commercial thin stillage and concentrate samples. The fouling rates increased with increasing solids concentration (Table 5).

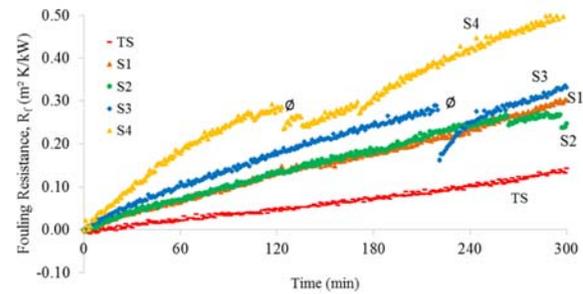


Fig. 3. Fouling resistance of thin stillage and concentrates from stage 1, 2, 3 and 4 before evaporator cleaning (1 observation). TS = thin stillage, S1, S2, S3 and S4 = concentrates from 1st, 2nd, 3rd and 4th stage in a multiple stage evaporator; \emptyset = apparent sloughing of deposits.

Table 5. Fouling rates of thin stillage and concentrates.

Sample	Total solids (% db)	Mean fouling rate ($\text{m}^2\text{K}/\text{kWh}$)	Clean U_o ($\text{kW}/\text{m}^2\text{K}$) [†]
TS	7	0.027±0.001a	2.81±0.140a
S1	8	0.059±0.001ab	2.61±0.065a
S2	9	0.059±0.001ab	2.59±0.075a
S3	10	0.068±0.010bc	2.55±0.065a
S4	11	0.096±0.020c	2.41±0.090a

TS = thin stillage. S1, S2, S3, S4 = concentrates from stages 1, 2, 3 and 4, respectively. [†]Mean of two samples.

Values with the same letter were not different ($p < 0.05$).

Fouling rates obtained for concentrates were similar to thin stillage fouling rates observed by Wilkins et al (2006a). However, thin stillage fouling rate ($0.024 \text{ m}^2\cdot\text{K}/\text{kW}\cdot\text{h}$) was lower than that observed by Wilkins et al (2006a) at similar solids content ($8.01 \pm 0.3\%$ db).

Clean overall heat transfer coefficients calculated for thin stillage and concentrates from stages 1, 2, 3 and 4 ranged from 2.41 to 2.81 $\text{kW}/\text{m}^2\cdot\text{K}$ (Table 5). As the commercial thin stillage was concentrated from one stage to another, concentrate viscosity increased causing an increase in fouling

rates (Fig. 4a). Fouling rates were greater for concentrates collected from stages 2, 3 and 4 (Fig. 4a).

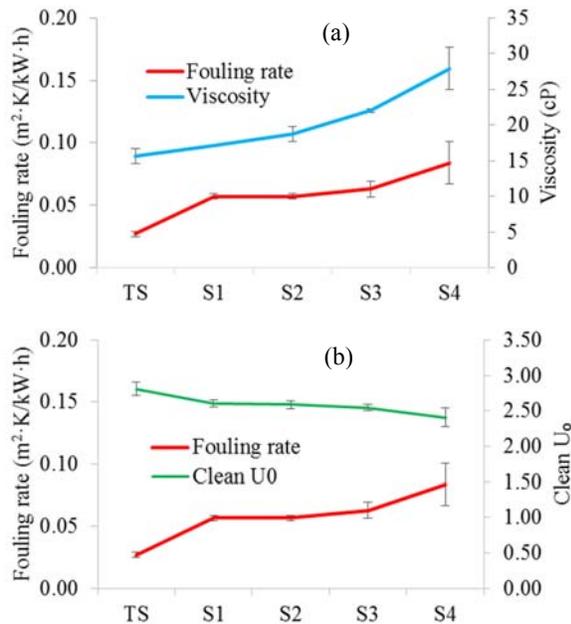


Fig. 4. (a) Comparison of viscosity and fouling rates of thin stillage and concentrates from stages 1, 2, 3 and 4. (b) Comparison of clean overall heat transfer coefficient (U_o) and fouling rates.

Even though the viscosities of commercial thin stillage and concentrate from stage 1 (S1) were similar, fouling rates of commercial thin stillage were lower than the concentrate from stage 1 (S1) (Fig. 4a). Viscosity of concentrates increased with stages and therefore the fouling rates also were increased. Fouling rate coefficient of variation (CV) was between 5.2 and 11.1% for thin stillage and concentrates from S1 and S2. Fouling rate CV for S3 and S4 ranged between 14.3 and 28.6% and CVs were higher. As thin stillage was concentrated from stage 1 to 4, clean overall heat transfer coefficient (U_o) decreased from thin stillage to concentrate from stage 4 while fouling rates simultaneously increased (Fig. 4b).

Evaporator cleaning and plant shutdown effects

In a separate series of tests, effect of evaporator cleaning on thin stillage fouling was studied (Fig. 5). Samples were collected a week after the plant had been completely shut down for cleaning. Thin stillage storage tanks were drained during this shut down. Fresh stillage was produced; samples were taken from this newly made material. Cleaning had an impact on evaporator fouling. Unlike the samples collected before plant shutdown (Fig. 3), the fouling rates of thin stillage and concentrates from individual stages were similar and had induction periods longer than the test period, 5 h (Fig. 5). Sloughing was not observed for samples collected a week after evaporator cleaning. Thin stillage collected after evaporator cleaning had higher concentrations of glucose, lactic acid, methanol and ethanol (Table 2). But the acetic

acid levels in thin stillage were similar to the samples collected prior to evaporator cleaning.

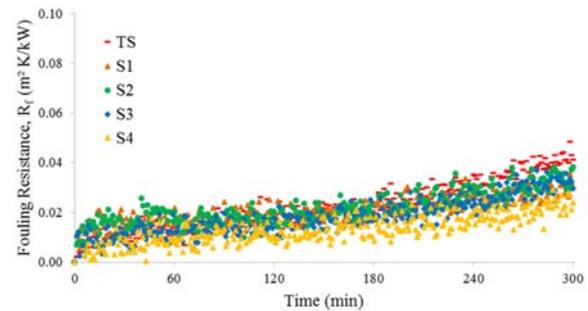


Fig. 5. Fouling resistance of dry grind thin stillage and concentrate from stage 1, 2, 3 and 4 after cleaning (1 observation). TS = thin stillage, S1, S2, S3 and S4 = concentrate from 1st, 2nd, 3rd and 4th stage in a multiple effect evaporator, respectively.

Fouling of corn oil added to thin stillage

Fouling resistance followed an increasing trend for thin stillage and oil added thin stillage until the end of the 5 h test period. Each test was replicated and no data scatter was observed. At the beginning of each test, no negative fouling resistances were noticed for thin stillage and oil added thin stillage. Oil addition to thin stillage increased fouling rates of commercial thin stillage but at higher oil concentration (1.5%), fouling rates decreased compared to thin stillage mixtures added with 0.5 and 1.0% post fermentation oil (Fig. 6).

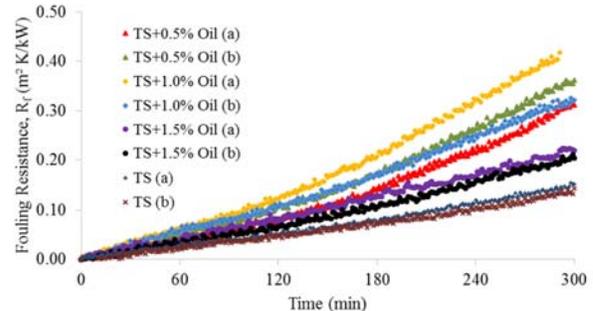


Fig. 6. Effect of post fermentation oil addition (0.5, 1.0 and 1.5% w/w) on thin stillage fouling. TS = thin stillage.

Mean fouling resistance of thin stillage was 0.027 m²·K/kWh (Table 6). Upon addition of 0.5% oil to thin stillage, average fouling resistance increased to 0.066 m²·K/kWh. Average fouling resistance of 1% oil added thin stillage was 0.077 m²·K/kWh and higher than 0.5% added thin stillage and commercial thin stillage. However, 1.5% oil added thin stillage had lower average fouling resistance (0.039 m²·K/kWh) compared to original thin stillage and 0.5 or 1.0% oil added thin stillage. Mean fouling rates of thin stillage were lower than post fermentation oil added thin stillage. Fouling rates CV were

between 7.7 and 9.0% for oil added thin stillage and 11.1% for commercial thin stillage.

Table 6. Mean fouling rates and clean overall heat transfer coefficients of thin stillage and post fermentation oil added thin stillage.

Sample	Mean fouling rate (m ² K/kWh)	Clean U _o (kW/m ² K)†
TS	0.027 ± 0.003a	2.81 ± 0.014a
TS + 0.5% oil	0.066 ± 0.006bc	2.57 ± 0.075a
TS + 1.0% oil	0.077 ± 0.007c	2.53 ± 0.007a
TS + 1.5% oil	0.039 ± 0.003ac	2.48 ± 0.031a

TS = thin stillage. †Mean of two tests. Values with the same letter are not significantly different (p < 0.05).

Increasing order of fouling rates: thin stillage, thin stillage + 1.5% oil, thin stillage + 1.0% oil, thin stillage + 0.5% oil. Viscosities of post fermentation oil added thin stillage were similar at all oil concentrations (0.5, 1.0 and 1.5%). Viscosity of thin stillage and post fermentation corn oil added thin stillage did not vary with oil concentration. Therefore, it is unclear whether viscosity had any impact on clean overall heat transfer coefficient. Clean overall heat transfer coefficients (U_o) were not different. No induction periods were observed for thin stillage and also for oil added thin stillage.

Oil recovery effects

The fouling resistance curves obtained for skimmed and unskimmed concentrates were similar. Tests were conducted to observe the effect of evaporator cleaning on fouling resistances (Fig. 7).

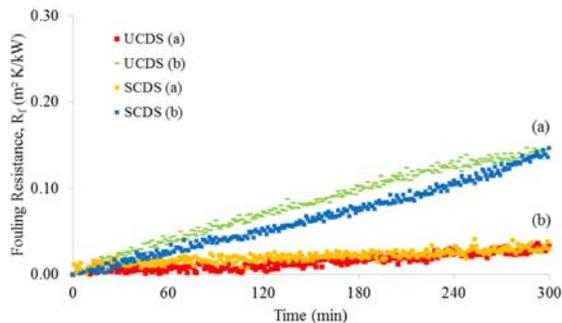


Fig. 7. Effect of cleaning on skimmed and unskimmed concentrate (1 observation). UCDS = unskimmed concentrate, SCDS = skimmed condensed distillers solubles; (a) = before plant and evaporator cleaning, (b) = after cleaning.

Fouling resistance of samples collected before evaporator cleaning showed no induction periods (a, Fig 7) but the samples collected a week after evaporator cleaning had induction periods longer than 5 h (b).

Glycerol effects

As thin stillage was concentrated in the multiple effect evaporator, glycerol concentration increased from thin stillage (1.28% w/v) to concentrate from stage 8 (5.4% w/v) (Table 2). Glycerol added thin stillage had higher fouling rates relative to thin stillage (Fig. 8). This is similar to the increase in fouling rates of post fermentation oil added thin stillage.

Mean fouling rate of glycerol added thin stillage was higher than thin stillage (Table 7). Clean overall heat transfer coefficients were not different and induction periods were not observed.

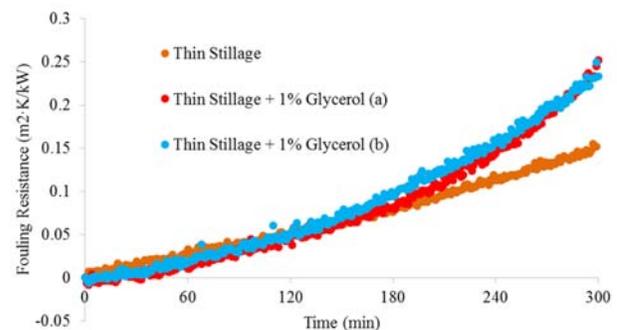


Fig. 8. Fouling resistance of thin stillage and glycerol added thin stillage. (a) Replicate 1. (b) Replicate 2.

Table 7. Mean fouling rates and clean overall heat transfer coefficients of thin stillage and glycerol added thin stillage.

Sample	Mean fouling rate (m ² K/kWh)	Clean U _o (kW/m ² K)
TS	0.027 ± 0.003a	2.81 ± 0.014a
TS + 1.0% glycerol	0.048 ± 0.006b	2.61 ± 0.053a

DISCUSSION

Thin stillage fouling rates observed were lower than earlier studies (Arora et al 2010; Singh et al 1999; Wilkins et al 2006a; Wilkins et al 2006b); it is possible the fouling rates decreased due to deposit sloughing from the probe surface. Using a higher bulk temperature (75°C) may have increased solubility of thin stillage components and decreased the particulate matter suspended in bulk fluid which reduced particulate fouling.

Thin stillage and concentrates from stages 1, 2, 3 and 4 did not show negative R_f values (Fig. 5). In earlier studies, negative R_f values were observed for thin stillage which indicated nucleate boiling from bubble formation on the probe surface (Crittenden and Alderman 1988; Singh et al 1999; Zuber 1963). There may be a large number of active nucleation sites formed on heating zone on probe surface at high bulk and wall temperatures which increased initial deposit rates. Wilkins et al (2006a) obtained fouling rates at lower bulk temperatures (40 ± 2°C) which may have resulted in fewer active nucleation sites and resulted in induction

periods. Also lower initial probe surface temperatures ($100 \pm 2^\circ\text{C}$) could have contributed to longer induction periods in previous studies (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) as heat transfer coefficients decrease with increase in temperature difference between bulk fluid and heat transfer surface. Previous bulk temperatures (40 to 60°C) were lower than industry (70 to 80°C) while surface temperatures during tests (100 to 170°C) were likely higher than industry. Exact temperature data from industry evaporators were not available. In the current study, bulk temperatures (75°C) were similar to those in industry and surface temperatures were likely higher, giving conditions overall that are more severe than industry evaporators.

The effects of simultaneous plant shutdown and evaporator cleaning on thin stillage and concentrates fouling in multiple effect evaporators were unclear (Fig. 5 and 7). No experiments were conducted to test thin stillage compositional effects on fouling rates but it appeared that plant shutdown removed residual constituents that were accumulated from backset recycling.

Due to many compounds in crude corn oil, it was hypothesized that addition of crude corn oil would increase fouling. To some degree, this was observed (Fig. 6), but at the highest level of addition, fouling decreased. This is indicative of other factors, such as lipids changing deposition rates, becoming more influential during the fouling process. Fouling tendencies of post fermentation corn oil added thin stillage differed from an earlier study that used refined corn oil (Singh et al 1999). In this study, thin stillage had lower fouling rates compared to post fermentation oil added thin stillage. High concentrations of wax esters in post fermentation oil may have contributed to higher fouling rates of oil added thin stillage. Paraffin wax fouling on heat exchanger surfaces was controlled by bulk temperature, flow rate and wax concentration in bulk fluid (Bott and Gudmundsson 1977). Fouling tests need to be conducted using model thin stillage mixtures using wax esters and free fatty acids to understand their effects on thin stillage fouling.

No induction periods were observed for thin stillage and refined oil added thin stillage from a dry grind ethanol plant and wet milling facility (Singh et al 1999). However, negative fouling rates were observed by Singh et al (1999). Wilson and Watkinson (1995) suggested that negative fouling resistances were from particles disturbing the thermal boundary layer. Higher bulk temperatures ($75 \pm 2^\circ\text{C}$) in the current study may have increased solubility of thin stillage components which decreased particulate matter. In earlier studies, temperature difference between the bulk temperature and probe surface temperature was high ($> 50^\circ\text{C}$) and in the current study it was less than 50°C . Heat transfer surface characteristics may have been altered by lipid adsorption onto metal surface by transforming high energy metal surfaces into unwettable or autophobic surfaces (Lund and Sandu 1981).

Fouling rates increased in thin stillage with added glycerol (Fig. 8) which suggest that accumulation of certain compounds will contribute to accelerated evaporator fouling. A certain percentage of thin stillage is recycled to the beginning of the production process (called backset), causing an increase in total solids and soluble compounds to increase relative to thin stillage during plant startups (Bayrock and Ingledew 2005). Glycerol concentrations were as high as 5.4% w/v in evaporator concentrates (Tab. 2), which is attributed to the high backset (56%) added to corn slurry and also to yeast stress during fermentation (Ingledew and Patterson 1999; Murthy et al 2012). Therefore, reduced backset addition to corn slurry in a dry grind ethanol plant is one possible method to mitigate evaporator fouling.

CONCLUSIONS

1. Total solids in evaporator concentrates increased from passing thin stillage through first effect stages in a multiple effect evaporator. Fouling rates increased with increased total solids content.
2. Post fermentation corn oil addition at 5% w/w increments to thin stillage increased fouling rates. At 1.5% oil addition to thin stillage, fouling rates decreased but were higher than thin stillage without addition.
3. Glycerol addition (1%) to thin stillage increased thin stillage fouling rates.
4. Commercial thin stillage fouling rates were lower than earlier studies.
5. Thin stillage and evaporator concentrates collected a week after plant shutdown and evaporator cleaning exhibited induction periods longer than 5 h. Factors that influenced these fouling tendencies were unknown.

NOMENCLATURE

A	probe surface area, m^2
k	thermal conductivity, W/mK
Q	power input to probe, W
R_f	fouling resistance, $\text{m}^2\text{K/kW}$
Re	Reynolds number, dimensionless
T_b	bulk temperature, K
T_s	surface temperature, K
T_w	mean wall temperature, K
TS	thin stillage
U_o	clean overall heat transfer coefficient, $\text{kW/m}^2\text{K}$
U	overall heat transfer coefficient, $\text{kW/m}^2\text{K}$
x	distance, m
x/k	calibration constant for the probe, $\text{m}^2\text{K/kW}$

Subscript

b	bulk
f	fouling
s	surface
w	wall

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