

Analysis of Heat Transfer Fouling by Dry-Grind Maize Thin Stillage Using an Annular Fouling Apparatus

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ABSTRACT

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In dry-grind processing to produce ethanol from corn, unfermented solids are removed from ethanol by distillation and dried to produce distillers dried grains with solubles (DDGS), an animal food. Fouling of thin stillage evaporators has been identified as an important energy consumption issue in dry-grind facilities. Using an annular fouling apparatus, four batches of thin stillage were analyzed to determine repeatability of fouling rate and induction period measurements. Dry solids, protein and ash concentrations, and pH were correlated to fouling rate and induction period to determine how variation in thin stillage from the same dry-grind facility affects these fouling parameters. Effects of increasing Reynolds

number (Re) in the laminar region on fouling rate, induction period, and fouling deposit protein and ash concentrations were also determined. Repeatability of fouling rate measurements was similar to other studies (CV < 7.0%) but repeatability of induction period measurements was high relative to other studies (CV < 88.7%). Fouling rate increased with increasing dry solids concentration. Thin stillage at Re = 440 had shorter induction periods and greater fouling rates than at Re = 880. Fouling deposits collected from Re = 440 tests had similar protein concentrations and lower ash concentrations compared with deposits from Re = 880 tests.

The U.S. Clean Air Act mandates that certain areas of the country with air pollution problems use reformulated gasoline containing 2% oxygen (Lyons 2003). Two additives are used to increase oxygen levels in gasoline: methyl-tertiary butyl ether (MTBE), a petroleum derivative, and ethanol. In recent years, MTBE has been phased out due to its possible carcinogenic properties and its discovery in ground water. As a result, U.S. ethanol demand has increased from ≈670 million liters (200 million gallons) in 1980 to 13.6 billion liters (3.6 billion gallons) in 2004, most of which was produced from maize (Shapouri and Gallagher 2005). Fuel ethanol is produced from maize using two methods: wet-milling and dry-grind (DG) maize processing. In 2004, DG facilities accounted for 70% of current U.S. ethanol production.

The DG process consists of grinding whole maize, using heat and enzymes to convert starch to glucose, fermenting glucose to ethanol with *Saccharomyces cerevisiae* yeast, separating ethanol from unfermented material, and drying the unfermented material to produce distillers dried grains with solubles (DDGS). Evaporation of water from the soluble nonfermentable fraction, known as thin stillage, consumes much of the energy in the DG process (Meredith 2003). Thin stillage is composed of proteins, ash, lipids, and other kernel constituents that were not fermented. Thin stillage (4–6% dry solids, w/w) is concentrated (25–30% dry solids, w/w) in multi-effect evaporators (Singh et al 1999) before being mixed with wet grains and dried to produce DDGS. Thin stillage evaporators foul rapidly, requiring plant shutdowns and cleaning every few weeks. Evaporator fouling increases heat transfer resistance, energy use, cleaning costs, downtime to restore evaporators to optimal performance, and capital costs to install extra evaporation capacity to compensate for cleaning. One strategy to improve long-term stability of the DG industry is to increase efficiency of unit operations in the process, particularly energy use. Understanding the fouling of heat transfer surfaces and the tendencies

of process materials to foul these surfaces would provide information to increase energy efficiency (i.e., less energy to produce a given volume of ethanol) of the DG industry.

Heat transfer fouling, the deposition of material on a heated surface, is found in heat transfer operations and is prevalent in processing of foods and biological materials (Changani et al 1997). Molecules found in foods, such as proteins, carbohydrates, and lipids, are heat sensitive and attach to heated surfaces in heat transfer equipment. A limited number of fouling studies have been published for maize processing (Singh et al 1999; Agbisit et al 2003). Instruments with annular flow configurations have been used to study fouling in other applications, including petrochemicals (Asomaning and Watkinson 1992; Panchal and Watkinson 1993; Wilson and Watkinson 1996). In maize processing, Singh et al (1999) observed that thin stillage from maize wet-milling fouled an annular fouling apparatus at a slower rate than thin stillage from a DG facility. They attributed this to higher oil content in DG thin stillage than in thin stillage from maize wet-milling. Additionally, Agbisit et al (2003) subjected steepwater from maize wet-milling to microfiltration. Steepwater permeates from microfiltration fouled the heated surface of the apparatus at a rate 80% slower than unfiltered steepwater.

Many variables affecting fouling behavior of maize processing streams are poorly understood. Relationships between thin stillage composition, fluid temperature, pH, flow rate, and the fouling of heated surfaces have not been quantified. For example, flow rate directly influences the shear stress at the evaporator heating surface and tends to disrupt formation of fouling deposits. Shear stress on evaporator surfaces has been increased by increasing flow velocities and creating flow geometries that promote turbulence, such as spiral heat exchangers and the rippled surfaces of plate heat exchangers (Young and Sloan 2003). Belmar-Beiny et al (1993) found the amount of whey protein fouling deposited in a tubular fouling apparatus decreased with increasing Reynolds number (Re), a dimensionless parameter indicating flow turbulence. Effect of Re on the amount of fouling deposit formed was asymptotic; a larger deposition decrease was observed when Re increased from 1,800 to 4,000 than when increased from 4,000 to 9,000. Karabelas et al (1997) used a CaCO₃ solution to observe resistance to heat transfer in plate heat exchangers. They observed an asymptotic effect of increased flow velocity on heat transfer resistance: as flow velocity increased the rate of change in the fouling rate, the rate of change of the overall heat transfer coefficient decreased. Effect of Re on fouling measurements observed with thin stillage has not been reported. The repeatability of the fouling measurement technique was unknown for maize processing

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streams from the DG process. This study was designed to 1) compare fouling rate repeatability and induction period measurements on DG thin stillage to other studies using an annular fouling probe, 2) determine how variation in the composition and pH of thin stillage affects fouling behavior, and 3) determine effect of Re in laminar flow on fouling measurements. Fouling behavior for this study was defined by the fouling rate and the induction period, the initial period where no fouling occurs (Belmar-Beiny et al 1993).

MATERIALS AND METHODS

Equipment

Fouling experiments used an annular fouling apparatus (Heat Transfer Research, College Station, TX) similar to a design outlined in Fischer et al (1975) and used by Singh et al (1999) and Agbisit et al (2003). The fouling apparatus consisted of a cylindrical rod inside a stainless steel housing that had a circular cross section to create an annular flow space (Fig. 1). The rod tubing was constructed of stainless steel (SS 316) and contained an electrical resistance heater and four thermocouples that monitored temperature of the inner rod surface. Power was supplied to the electrical resistance heater to heat a 102-mm section of the rod. Fouling deposits formed on the outer surface of the heated rod tubing; this increased heat transfer resistance, resulting in an increase in temperature inside the rod tubing. The increased temperature was used to calculate change in heat transfer coefficient as material was deposited on the outer surface of the rod tubing.

Three type E thermocouples were used to measure the temperature of the inner rod surface (T_{ic}). The fourth type E thermocouple was used to operate an electrical relay that turned off power at temperatures $>200^{\circ}\text{C}$ to protect the probe's wiring from overheating. Power supplied to the resistance heater (Q) was measured by a wattmeter (PC5-110D, Ohio Semitronics, Hilliard, OH). Temperatures and power readings were acquired every 10 sec by a datalogger (OM-3000, Omega Engineering, Stamford, CT) and later downloaded to a computer.

Resistance to heat transfer, or fouling resistance, was calculated by monitoring T_{ic} with thermocouples at three locations. Rod surface temperature (T_s) was determined by

$$T_s = T_{ic} - (x/k) Q/A \quad (1)$$

where T_{ic} is the temperature ($^{\circ}\text{C}$) measured at the inner surface of the rod; x/k is the radial distance (m) of the thermocouple from the surface divided by the thermal conductivity ($\text{kW/m} \cdot \text{K}$) of the rod metal; Q is power supplied to the resistance heater (kW); and A is the area (0.0034 m^2) of the heated section of the rod based on the outside diameter of the rod and assumed to be constant (Singh

et al 1999). Quantity x/k is determined for each thermocouple by a calibration procedure described in Fischer et al (1975) and based on a graphical technique by Wilson (1915). The ratio x/k was 0.061, 0.091, and $0.10 \text{ m}^2 \cdot \text{K/kW}$ for each of the three thermocouples. Because the thermal conductivity of stainless steel is constant, differences in x/k values indicate small variations in radial distance of each thermocouple from the rod surface. Individual rod surface temperatures associated with each thermocouple were calculated using T_{ic} and x/k values for each thermocouple and averaged for each time interval to calculate a mean T_s . The overall heat transfer coefficient for the probe (U) ($\text{kW/m}^2 \cdot \text{K}$) is determined by

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

where T_b is the thin stillage bulk temperature ($^{\circ}\text{C}$). Fouling resistance at time t (R_f) is calculated as

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

where U_0 is the heat transfer coefficient ($\text{kW/m}^2 \cdot \text{K}$) at the beginning of the test. The beginning of the test ($t = 0$) is defined as the time when T_{ic} reached 100°C . U_t is the overall heat transfer coefficient ($\text{kW/m}^2 \cdot \text{K}$) at time t .

Thin stillage was obtained from a commercial DG plant, stored at 4°C , and used within one week of collection. For each test, 30 L of thin stillage was placed into a stainless steel tank and stirred continuously ($\approx 30 \text{ rpm}$) with a top-mounted impeller (Fig. 2). A diaphragm pump (Hydra-Cell, Wanner Engineering, Minneapolis, MN) with a 3.7 kW drive (SEW Eurodrive, Bruchsal, Germany) pumped thin stillage through the system at $13 \pm 0.5 \text{ L/min}$, which was measured using a rotameter. A shell and tube heat exchanger (1.5 m, Graver Technologies, Glasgow, DE) using water as the heating fluid was used to heat thin stillage to the desired T_b . Thin stillage pH was measured by a pH probe (Digi-sense, Cole-Parmer, Vernon Hills, IL). After the desired T_b was reached, power was supplied to the resistance heater and was adjusted until an average T_{ic} of 100°C was reached, after which constant power was maintained ($\pm 15 \text{ W}$). Each test was terminated when the relay thermocouple reached 200°C .

Following each test, a plastic spatula was used to scrape fouling deposits from the rod surface to avoid scratching the rod surface. After most of the deposit was removed using the spatula, the rod was soaked in 5% (w/v) NaOH solution at room temperature overnight. After soaking, any remaining deposit was removed using a wet sponge. The fouling analysis system (batch tank, tubing, heat exchanger, pump, and probe housing) was cleaned by recirculating 20 L of 1% (w/v) detergent solution (Alconox, New York, NY) for 20 min, followed by a rinse with cold water (250 L).

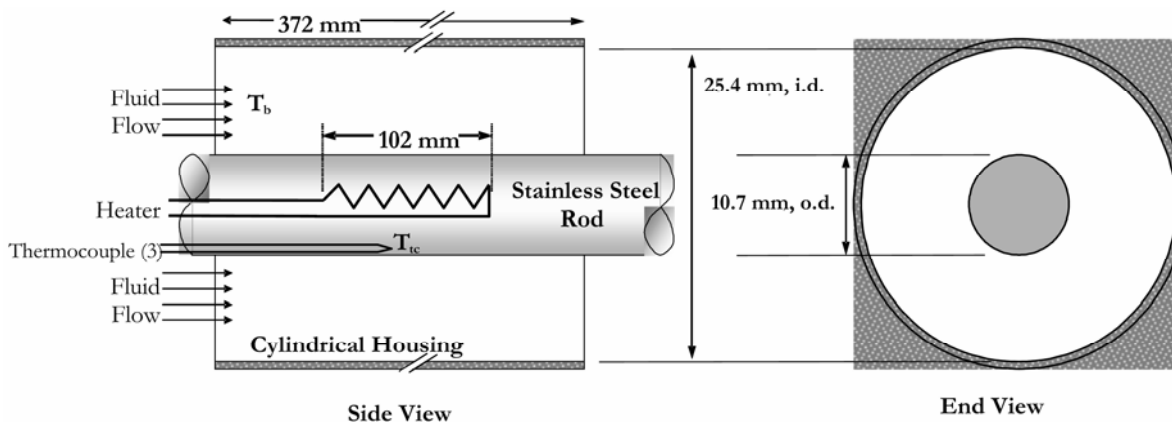


Fig. 1. Schematic of annular fouling apparatus (not to scale).

Experiment 1: Repeatability of Fouling Measurements and Effect of Variation in Thin Stillage Composition and pH

Four 120-L batches were collected over two months with three replicate tests conducted per batch. Each test was conducted as described above. The T_b was maintained at $40 \pm 2^\circ\text{C}$ by the heat exchanger throughout each test, which was the T_b used in previous studies (Singh et al 1999; Agbisit et al 2003). This allowed comparison of measurements from this study to other studies, particularly Agbisit et al (2003). Composition of each thin stillage batch and fouling deposits were determined using methods described below.

Experiment 2: Effect of Reynolds Number in Laminar Flow

Re for each flow rate is determined using

$$Re = \frac{\rho v D}{\mu} \quad (4)$$

where ρ is the density (kg/m^3), v is the mean fluid velocity (m/sec), D is the i.d. of annulus housing minus diameter of probe (m) and μ is the absolute viscosity ($\text{kg/m}^2 \cdot \text{sec}$) (Boyce 1997). Density of thin stillage was measured for each batch by weighing a 5-mL aliquot of thin stillage at 50°C (T_b for this experiment). Fluid velocity was determined for each batch by dividing the volumetric

flow rate (measurement as previously described) by the cross-sectional area of the annular region. Viscosity of thin stillage was measured using a viscometer equipped with spindle no. 1 (RVT, Brookfield Engineering, Brookfield, MA) at 50°C .

Three 120-L batches of thin stillage were collected over three months from a DG processing facility. Four subsamples (30 L) were collected from each 120-L replicate batch. For each batch, two subsamples were tested at a flow rate of 11.3 L/min ($Re = 440$, or Re_{440}) and two subsamples were tested at 22.6 L/min ($Re = 880$, or Re_{880}). A flow rate of half the maximum flow rate (11.3 L/min) was chosen for the second treatment level. Flow rates >22.6 L/min could not be achieved with the pump available for this study. The order of analysis of subsamples for each batch was determined randomly. Fouling rate and induction period were determined for each subsample. The T_b was increased from 40°C used in the repeatability measurement to 50°C to better reflect commercial conditions within the limits of the experimental system. Composition of each thin stillage batch and fouling deposits were determined using methods described below.

Composition of Thin Stillage and Fouling Deposits

One aliquot (500 mL) of thin stillage from each batch was analyzed for total nitrogen (TN), ash, and solids concentrations.

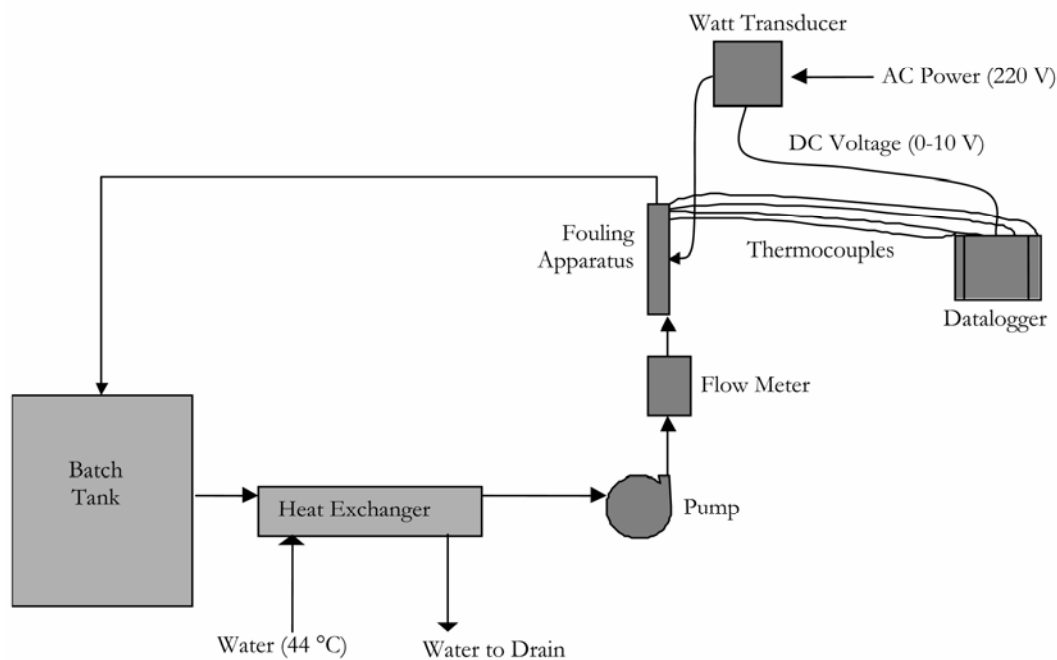


Fig. 2. Schematic of fouling analysis system.

TABLE I
Composition, Fouling Rates, and Induction Periods of Thin Stillage Samples

Batch	Thin Stillage					Fouling Deposits						
	Composition			pH ^b	Power ^{b,c} (kW)	Fouling Rate		Induction Period		Replicate	Composition	
	Dry Matter (% db)	Protein ^a (% db)	Ash (% db)			Mean ^b ($\text{m}^2 \cdot \text{K/kW} \cdot \text{h}$)	CV (%)	Mean ^b (min)	CV (%)		Protein ^a (% db)	Ash (% db)
1	8.11	18.0	9.54	3.99	0.771	0.052	5.33	140	23.1	test 1	19.9	43.6
2	7.78	22.5	9.78	3.96	0.769	0.046	3.11	143	30.0	nd ^d	nd	nd
3	8.90	22.6	9.99	3.76	0.993	0.062	7.03	25.7	88.7	test 2	21.7	39.2
4	7.25	16.8	11.40	3.98	0.985	0.048	5.61	50.0	28.0	test 1	19.6	45.3
										test 2	15.6	40.3
										test 3	18.4	39.6

^a Total nitrogen $\times 6.25$.

^b Means of three tests, except batch 2, which is from two tests.

^c Power was constant ($\pm 15\text{W}$) throughout each test.

^d Not determined.

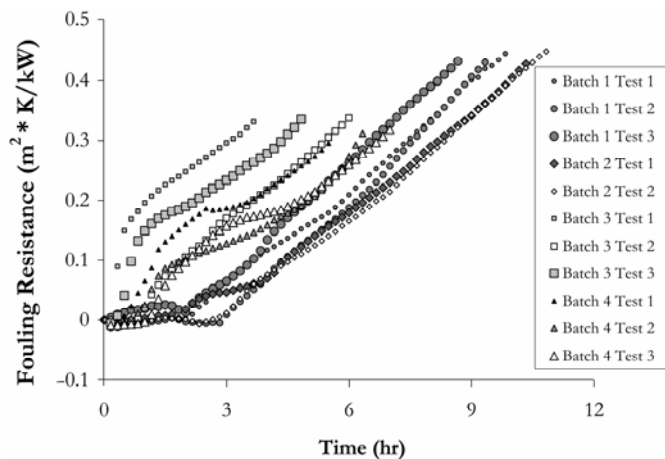


Fig. 3. Fouling resistance curves of thin stillage from replicate batches.

The TN concentration was determined by nitrogen combustion (AACC International 2000); protein was calculated by as TN \times 6.25. Ash concentration was determined by a dry ash method (AACC International 2000). Thin stillage dry solids content was determined by drying a 500-mL sample of thin stillage for 24 hr at 49°C, then drying it further in a lyophilizer until the partial pressure of moisture in the lyophilizer was at a constant minimum. Deposit samples were dried overnight at 49°C. Total nitrogen, protein, and ash were measured on fouling deposits using the same methods as for thin stillage.

Statistical Analysis

Linear regression was performed on R_{ft} versus time data for each test. The slope of each regression line was defined as the fouling rate. Induction period was defined as the time when the 1-min moving average of R_{ft} was $<0.01 \text{ m}^2 \cdot \text{K}/\text{kW}$. Repeatability of the fouling rate and induction period was quantified by the coefficient of variation (CV) for each batch, which is the ratio of the standard deviation to the mean. Fouling rate and induction period were correlated to thin stillage dry solids, protein, and ash concentrations, pH, and power supplied to the probe. Linear regression was performed on these data to assess linear relationships between these factors. In Experiment 2, induction period and composition of selected fouling deposits were determined using a general linear model (release 8.0, SAS Institute, Cary, NC).

RESULTS AND DISCUSSION

Repeatability of Fouling Measurements

Fouling rate CV was from 3.1 to 7.0%, which was similar to the fouling rate CV observed by Wilson and Watkinson (1995) and Agbisit et al (2003) for similar experimental equipment (Table I). Induction period CV was from 23.1 to 88.7%, which was greater than that observed by Wilson and Watkinson (1995). Agbisit et al (2003) did not report values for induction period, but from their figures it is evident that they observed an induction period CV less than what was observed in this study. The reason for the lack of repeatability in induction period measurements is not clear. Power supplied to the probe for each test ($0.06 < \text{CV} < 1.5\%$) was similar among tests from the same batch (data not shown), and pH was constant for each test within the same batch. Linear relationships were not observed between thin stillage composition and induction period, so fluctuation in composition was probably not a major factor in the lack of repeatability in induction period measurement.

Negative R_{ft} values were observed during several tests (Figs. 3 and 4). Negative R_{ft} values have been reported by others (Wilson and Watkinson 1996; Singh et al 1999; Agbisit et al 2003) and are thought to be caused by particles disrupting the thermal boundary

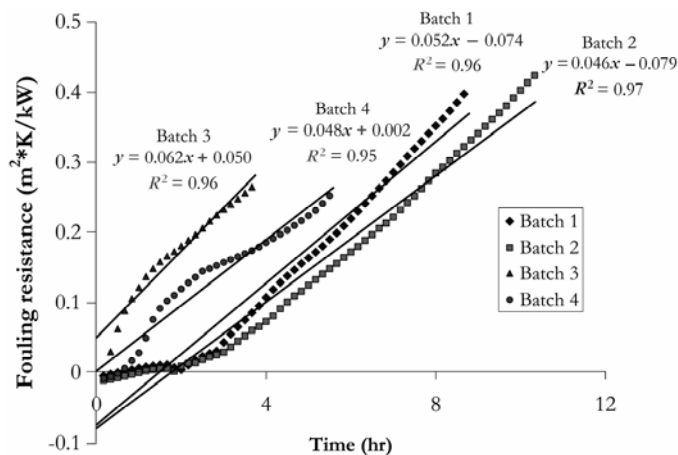


Fig. 4. Mean fouling resistance curves for thin stillage batches.

layer (Crittenden and Alderman 1988; Wilson and Watkinson 1996), power fluctuations, or depositions that produce roughness (Panchal and Watkinson 1993).

Increased power supplied to the probe led to decreased induction periods (Table I). A linear correlation between induction period and fouling probe power was observed ($R^2 = 0.98$). In dairy fluid processing, increased power leads to more denaturation and aggregation reactions among proteins. Aggregates then adsorb onto the probe surface (Georgiadis and Macchietto 2000). Increased heat transfer to the bulk fluid increases production rate of aggregates that can adsorb to the surface and initiate fouling, which could decrease the induction period. Tests of samples from batches 1 and 2 required less power to achieve the initial T_{ic} of 100°C than did tests of samples from batches 3 and 4 for reasons that are not clear.

Effect of Variation of Thin Stillage Composition and pH

Fouling rate increased with increasing thin stillage dry solids composition (% w/w) ($R^2 = 0.83$). Linear relationships were not observed between thin stillage protein (% dry solids) and ash (% dry solids) compositions and fouling rate ($R^2 < 0.14$) (data not shown). Linear relationships were not observed between thin stillage dry solids, protein and ash compositions, and induction period ($R^2 < 0.36$) (data not shown). Fouling rate decreased with increasing pH ($R^2 = 0.76$). There was not a strong linear relationship between pH and induction period ($R^2 = 0.46$). Because batches had similar pH values, except for one batch, relationships between pH and the fouling behavior parameters of fouling rate and induction period were difficult to determine.

Thin stillage compositions and pH were not controlled during the study. Variations in composition and pH were caused by variations in the operation of the DG facility from which the thin stillage was obtained. This variation can be caused by changes in composition of maize entering the facility and in processing parameters such as acid addition rates, enzyme loadings, temperatures, and fermentation conditions. Variability in composition of maize processing streams has been observed in other work (Belyea et al 2004; Rausch et al 2005). Observing fouling rates with varying dry solids concentration and pH indicate that tighter control of these factors may better control thin stillage fouling behavior.

Effect of Re

For thin stillage at 50°C, the mean absolute viscosity for all batches was measured as 0.0145 kg/m²·sec and the mean density was 0.96 kg/m³. The viscosity and density measurements were the same for each batch. Dry matter contents of thin stillage batches were from 8.4 to 8.7%, protein contents of thin stillage batches were from 18.1 to 22.1% db, and ash contents of thin stillage

TABLE II
Dry Matter, Protein, and Ash Concentrations
of Three Thin Stillage Batches Used to Study the Effect
of Reynolds Number on Fouling Properties

Batch	Dry Matter (% wb)	Protein (% db)	Ash (% db)
1	8.72	22.1	10.6
2	8.50	21.8	9.6
3	8.36	18.1	21.4

TABLE III
Induction Periods and Compositions of Fouling Deposits
for Tests at Re_{440} and Re_{880} ^a

Re	Batch	Fouling Rate ($m^2 \cdot kW/K \cdot hr$)	Induction Period (min)	Protein Concentration ^b (% db)	Ash Concentration (% db)
440	1	0.115	41.9	18.5	36.0
	2	0.073	2.50	22.4	25.7
	3	0.067	5.00	19.8	30.5
880	1	0.059	78.6	20.0	43.7
	2	0.050	111.8	18.6	38.2
	3 ^c	0.051	136.5	18.0	42.2

^a Means of two analyses.

^b Total nitrogen $\times 6.25$.

^c Data from one observation.

batches were from 9.6 to 21.4% db (Table II). Ash content of batch 3 (21.4%) was more than twice the ash contents of batches 1 and 2. The Re was calculated to be 440 at a flow rate of 11.3 L/min (Re_{440}) and 880 at a flow rate of 22.6 L/min (Re_{880}). Both Re values were in the laminar flow region ($Re < 2,100$), which were achievable and practical in a laboratory setting but are lower than Re found in commercial processes.

Induction periods were longer for Re_{880} tests than for Re_{440} tests. Rapid fouling within the first 30 min was observed for some tests at Re_{440} . Fouling rates were greater for Re_{440} tests than for Re_{880} tests. Induction periods >1 hr followed by an increase in fouling were observed for Re_{880} tests (Fig. 5, Table III). After the induction period, fluctuations in fouling resistance were observed at both Re values for some of the batches. There were no linear correlations between thin stillage dry solids, protein and ash concentration, and fouling rate or induction period (data not shown).

At higher flow velocity and Re, shear forces increase and the thermal boundary layer thickness along a heated probe surface decreases (Belmar-Beiny et al 1993). Increased shear force disrupts the adsorption of fouling deposits onto the heated rod surface. Decreased boundary layer thickness reduces the volume of fluid at temperatures suitable for reactions associated with fouling to occur.

Fouling deposit protein concentrations were from 18.5 to 22.4% db for tests at Re_{440} and 18.0 to 20.0% db at Re_{880} (Table III). Fouling deposit ash concentrations were 25.7 to 36.0% db for tests at Re_{440} and 38.2 to 43.7% db for tests at Re_{880} . Protein and ash concentrations of fouling deposits from different batches were not different, despite the fact that thin stillage from batch 3 had twice the ash content of batches 1 and 2.

Re observed in the current study indicate that Re should be controlled during measurement of heat transfer fouling. Though Re observed in this study were not in the turbulent region due to limitations in equipment, the results indicate reductions of fouling rate and increases of induction period for DG thin stillage can be achieved by increasing Re in the laminar region. In the range of Re observed for this study, it was also observed that fouling deposit protein concentrations did not change as Re increased, but fouling deposit ash concentrations increased when the Re was increased. Within these test parameters, protein adsorption to the

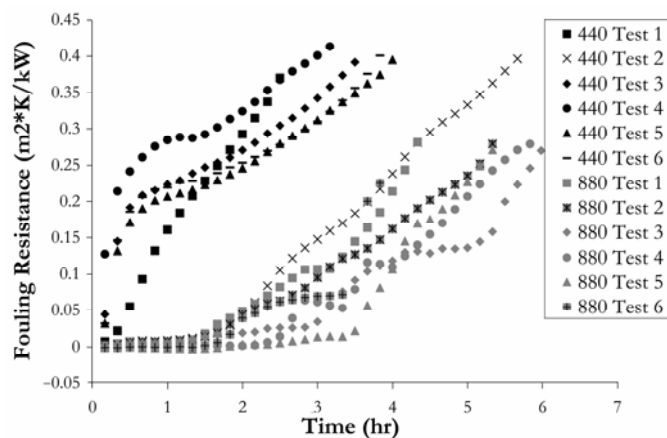


Fig. 5. Fouling resistance curves for thin stillage at two Reynolds numbers in the laminar region.

heated surface appears to be more affected by increasing shear at higher Re than is ash adsorption. Belmar-Beiny et al (1993) observed reductions in fouling from whey solutions as Re increased from 1,800 to 7,200 but they did not measure the ash concentration of the deposits from different Re.

CONCLUSIONS

Repeatability of fouling rate measurement within batches of thin stillage with an annular fouling probe as measured by CV was similar to other studies ($CV < 7.0\%$), while repeatability of induction period measurement was larger ($23.1 < CV < 88.7\%$) than that reported in other work. Increasing power to the probe decreased induction periods but did not affect fouling rate. Reasons for increased power requirements in later tests to achieve the same initial temperatures as earlier tests are unclear. Fouling rate increased as dry solids concentration of thin stillage increased. Increasing Re from 440 to 880 reduced fouling rate, increased induction period, and increased fouling deposit ash concentrations, while fouling deposit protein concentrations were unchanged. Effect of Re should be considered an important parameter when measuring fouling characteristics. Ash concentrations were greater than protein concentration in fouling deposits from all tests, indicating mineral deposition plays an important role in thin stillage fouling.

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