

Technique to Measure Surface-Fouling Tendencies of Steepwater from Corn Wet Milling

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ABSTRACT

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Surface-fouling tendencies of raw light steepwater (LSW) and membrane-filtered light steepwater (FSW) from corn wet-milling were studied using an annular fouling probe. The probe contained a heated surface to simulate the surface temperature of an evaporator. The heated region caused a fraction of solids in the steepwater to adhere to the surface, thus fouling the probe over time. FSW samples were prepared by filtering LSW using a microfiltration membrane with a nominal pore size of 0.1 μm . Fouling tendencies of both samples were established at an initial probe wall temperature of 99°C. Batches (30 L) were circulated through the fouling probe until the inner surface temperature of the probe reached

200°C. Temperature and power supplied to the probe were measured over time and used to calculate fouling resistance and rate of fouling. Measurement of maximum fouling resistance and fouling rate had a coefficient of variation (COV) of 5.1 and 7.4%, respectively. Maximum fouling resistances attained over a 12-hr period were 0.36 and 0.049 $\text{m}^2 \text{ }^\circ\text{C}/\text{kW}$ for LSW and FSW, respectively. Average rates of fouling were 4.53×10^{-4} and $0.82 \times 10^{-4} \text{ m}^2 \text{ }^\circ\text{C}/\text{kW}/\text{min}$ for LSW and FSW, respectively, showing an 80% decrease in fouling rate using microfiltration to remove 19% of solids.

In the corn wet-milling process, recycled water from milling operations is treated with 0.1–0.2% sulfur dioxide. Steepwater is pumped from tank to tank and finally discharged as light steepwater. Light steepwater (LSW) is concentrated to 45–50% dry solids using multiple effect evaporators (Blanchard 1999). LSW is mixed with fiber from the wet-milling process to form the corn gluten feed coproduct. Steepwater evaporation is one of the most capital and energy-intensive unit operations, consuming $\approx 20\%$ of the total energy for the wet-milling process and typically consisting of $\approx 20\%$ of capital cost for the facility. Components of steepwater (proteins, carbohydrates, and inorganics) bind to the heated surfaces of evaporators, a phenomenon called fouling. Fouling of evaporator surfaces reduces the rate of heat transfer from evaporator surfaces to the material being concentrated, thus increasing energy costs of evaporation. Buildup on evaporator surfaces must be regularly removed for evaporator performance and efficiency, increasing the cost of steepwater concentration through maintenance and operating expenses.

Fouling of evaporator surfaces depends on a number of factors, primarily concentration and composition of the fluid flowing over the surface (Knudsen 1981). At a critical concentration, the rate of diffusion to the heat transfer surface exceeds diffusion away from the surface, which is possible because of the larger magnitude of polymeric units relative to monomeric units in the fluid. It has been theorized that this crowding around the heat-transfer surface causes polymeric concentration to exceed some critical concentration and begin precipitation, leading to fouling (Panchal and Watkinson 1993). According to Lund and Sandu (1981), lipids cause fouling by copolymerizing with proteins and producing dark-colored products that foul heated surfaces. Measurement techniques and causes of fouling have been studied extensively in other processing industries such as the dairy industry (Burton 1967; Knudson 1981; Bouman et al 1992). However, there are no publications that describe the causes of increased evaporator fouling during concentration of LSW that evaluate process alternatives to reduce evaporator fouling in corn processing or that describe quantitative methods to measure fouling of evaporators used to concentrate LSW. The objectives of this study were to 1) observe the reliability of an annular probe used to measure fouling on a heated surface and 2) measure the effect of microfiltration processing on surface fouling tendencies of LSW.

MATERIALS AND METHODS

Experimental Material

LSW results from a complex countercurrent system; characteristics of LSW vary due to system operation and variation in corn quality. Therefore, sufficient material was obtained for testing to provide a uniform supply of LSW for repeatability tests and microfiltration processing. The first sample (200 L) of LSW was obtained for repeatability testing and a second sample (120 L) of LSW was obtained to observe differences in surface fouling tendencies when LSW was filtered using microfiltration membranes. Both samples were obtained from a commercial wet-milling facility. LSW samples were obtained from the process line leading from steep tanks to evaporators. All samples were stored at 2°C before testing. For solids content determination, three 75-mL aliquots were taken from each 500-mL subsample. The subsamples were placed in a 49°C oven for 24 hr, then transferred to a 135°C oven for 2 hr (Approved Methods 44-18 and 33-19, AACC 2000).

Surface-Fouling Tendencies

The annular fouling probe (Heat Transfer Research, Dallas, TX) was the same as used by Singh et al (1999) to study fouling tendencies of thin stillage (Table I). The probe consisted of an annular flow passage with an outer housing that contained an electrically heated concentric rod (Fig. 1). The stainless steel rod contained a resistance heater and four thermocouples located near the heated surface to monitor surface temperature. Surface temperatures were recorded over time with a datalogger (21X(L) Micrologger, Campbell Scientific, Logan, UT) and a computer.

The outer surface temperature of the heated region of the probe was calculated as

$$T_s = T_w - (x/R)Q/A \quad (1)$$

where T_s was the outer surface temperature of the heated rod; T_w was the inner wall temperature measured by the four thermocouples; x/R was a calibration constant unique to each probe and provided by the manufacturer; Q was the power supplied to the heater and measured using a multimeter (Tenma model 72605,

TABLE I
Design Specifications of Annular Fouling Probe

Parameters	Value
Material of construction	SS 316
Diameter of heater rod (A, mm)	10.7
Housing o.d. (B, mm)	25.4
Heated length of rod (C, mm)	102.0
Length from fluid entrance to thermocouple location (mm)	294.0

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Newark Electronics, Springfield, IL) and a clamp-on power meter (model 382060, Extech Instruments, Waltham, MA); A was the surface area of the heated section of the probe. T_w was determined by averaging the four temperatures sensed by the thermocouples inside the heated rod.

Using surface and bulk temperature data, the overall heat transfer coefficient (U) was calculated as:

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

where T_b was the bulk temperature of the test fluid.

Fouling resistance (Knudsen 1982) was calculated as

$$R_f = 1/U_t - 1/U_0 \quad (3)$$

where U_0 was the overall heat transfer coefficient at time $t = 0$ (clean surface) and R_f was the fouling resistance at time t .

The maximum fouling resistance was defined as the measured resistance to heat flow at the time when the inner wall temperature (T_w) reached 200°C. Linear regression analysis was used to determine the fitted least squares lines for estimating average fouling resistance over time for each subsample tested. Fouling rate, defined as the change in fouling resistance over a period of time ($m^2 \text{ } ^\circ\text{C}/\text{kW}/\text{min}$), was derived from the slope of the regression line. A one-way treatment analysis using the general linear model (GLM) was utilized to test for differences in fouling rates among samples.

Repeatability Procedures

To determine repeatability of the fouling technique and to select initial test parameters, one LSW sample was divided into four 30-L batches. This eliminated variation due to LSW characteristics when measuring surface fouling tendencies and evaluating fouling probe operation. Consistency of power supplied to the probe from preliminary work was important in determining the rate of fouling. Hourly adjustments in tests kept power fluctuations at a minimum.

Test parameters used were 30-L batch size, flow rate of 13 ± 0.5 L/min, bulk sample temperature of $40 \pm 1^\circ\text{C}$ and initial probe temperature of $99 \pm 1^\circ\text{C}$. This set of experiments measured reliability of the system using LSW samples.

Each 30-L sample was pumped through the system (Fig. 2) at 13 ± 0.5 L/min. To control bulk fluid temperature, water at 52°C was allowed to flow through the heat exchanger at a flow rate of 30 L/min until bulk fluid temperature reached 40°C . At this point, water through the heat exchanger was adjusted to $43\text{--}44^\circ\text{C}$. Every 20 sec, the datalogger recorded probe temperature at four locations within the probe, and bulk fluid temperature and power consumption. Power input was adjusted so that initial wall temperature of the probe was $99 \pm 1^\circ\text{C}$ and held constant during testing. The system

was monitored for changes in power and bulk temperature for 12 hr or until surface fouling caused the average wall temperature to reach 200°C . Power ($947 \pm 16\text{W}$) and bulk fluid temperature ($40.7 \pm 0.5^\circ\text{C}$) of the steepwater were kept constant throughout the experiment.

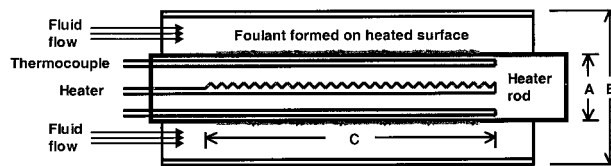


Fig. 1. Annular fouling probe. Dimensions (A, B, C) given in Table I.

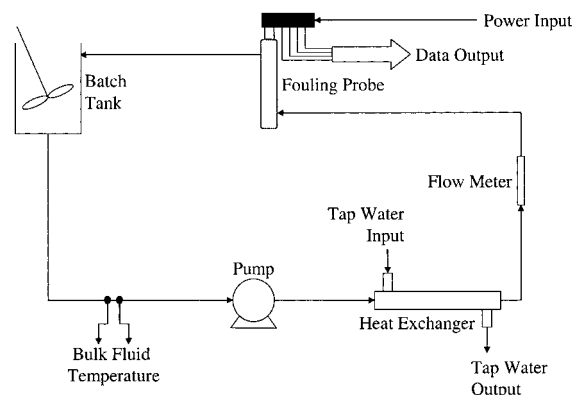


Fig. 2. Apparatus used to measure surface fouling resistance.

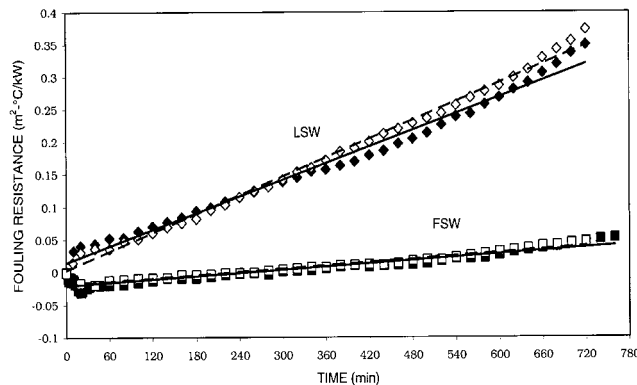


Fig. 3. Fouling resistance of raw light steepwater (LSW) and membrane filtered (FSW) steepwater.

TABLE II
Maximum Fouling Resistance^a and Fouling Rate of Light Steepwater (LSW) Samples

	Sample				Mean	SD	COV (%)
	1	2	3	4			
Max. fouling resistance ($m^2 \text{ } ^\circ\text{C}/\text{kW}$)	0.34	0.38	0.38	0.37	0.368	0.0189	5.1
Fouling rate ($m^2 \text{ } ^\circ\text{C}/\text{kW}/\text{min}$)	5.05×10^{-4}	4.21×10^{-4}	4.60×10^{-4}	4.56×10^{-4}	4.61×10^{-4}	0.34×10^{-4}	7.4
Bulk fluid temperature ($^\circ\text{C}$)	40.3	41.3	40.5	40.6	40.7	0.42	1.0
Power input (W)	1029	908	923	932	948	54.8	5.6

^a Maximum fouling resistance is defined as measured resistance when wall temperature reached 200°C .

TABLE III
Maximum Fouling Resistance and Fouling Rate of Light Steepwater (LSW) and Membrane-Filtered Light Steepwater (FSW) Samples

	Sample 1	Sample 2	Average	SD
LSW samples: maximum fouling resistance ($m^2 \text{ } ^\circ\text{C}/\text{kW}$)	0.35	0.37	0.36	0.0167
Fouling rate ($m^2 \text{ } ^\circ\text{C}/\text{kW}/\text{min}$)	4.24×10^{-4}	4.81×10^{-4}	4.53×10^{-4}	0.4031×10^{-4}
FSW samples: maximum fouling resistance ($m^2 \text{ } ^\circ\text{C}/\text{kW}$)	0.052	0.046	0.049	0.0043
Fouling rate ($m^2 \text{ } ^\circ\text{C}/\text{kW}/\text{min}$)	0.816×10^{-4}	0.823×10^{-4}	0.819×10^{-4}	0.005×10^{-4}

^a Maximum fouling resistance was defined as measured resistance when wall temperature reached 200°C .

Microfiltration Processing

Testing was conducted using the above parameters to measure surface fouling tendencies of LSW and FSW. Two tests were done for each treatment. Material for comparing LSW and FSW fouling rates was obtained from a 120-L sample taken from a commercial facility. Two FSW samples were prepared by filtering a 60-L batch of LSW using a stainless steel tubular microfiltration membrane having a nominal pore size of 0.1 μm (Graver Separations, Glasgow, DE), using 140 kPa transmembrane pressure and 3.5 m/sec crossflow velocity as recommended by the manufacturer.

RESULTS AND DISCUSSION

Repeatability

LSW samples showed small differences in mean wall temperature profile for the first 5 hr of testing, with growing differences in the later stages of testing. Power input and bulk temperatures were relatively constant for all tests (Table II). Fouling resistance profiles exhibited linear behavior with time (R^2 0.94–0.98, data not shown). Sample 1 exhibited a slightly higher fouling rate, primarily because average power input was slightly higher than for other samples. However, statistical analysis determined average fouling rates were not different from each other ($P = 0.46$). Maximum fouling resistance and fouling rate were 0.368 $\text{m}^2 \text{ }^\circ\text{C}/\text{kW}$ and 4.61 $\times 10^{-4} \text{ m}^2 \text{ }^\circ\text{C}/\text{kW}/\text{min}$, respectively. Maximum fouling resistance was defined as the resistance when mean inner wall temperature (T_w) reached 200 $^\circ\text{C}$.

Microfiltration Processing

Based on computed fouling resistances (R_f), maximum fouling resistances were 0.36 and 0.049 $\text{m}^2 \text{ }^\circ\text{C}/\text{kW}$ for LSW and FSW, respectively (Table III). Average fouling rates were 4.53 $\times 10^{-4}$ and 0.82 $\times 10^{-4} \text{ m}^2 \text{ }^\circ\text{C}/\text{kW}/\text{min}$ for LSW and FSW, respectively. The fouling rates for LSW and FSW samples were significantly different ($P = 0.0056$). Values from linear regression analysis were $R^2 = 0.90$ – 0.99 for the different samples. At a test interval of 600 min, resistance to heat transfer caused by surface fouling was ≈ 10 times greater for LSW than for FSW (Fig. 3). Average rate of fouling determined by linear regression was 5.3 times higher for LSW than for FSW.

Decreases in wall temperatures reflected a decrease in fouling rate of FSW in the early stages of the tests. As a result of decreases in temperature, negative values of fouling resistance for FSW were observed during the initial period of testing (Fig. 3). During this period, the solid to liquid heat transfer coefficient was relatively higher compared to the reference heat transfer at $t = 0$, leading to negative values of fouling resistance. This condition has been reported by others (Wilson and Watkinson 1995; Singh et al 1999) and has been theorized as a phenomena due to particles disturbing the developing thermal boundary layer, to deposition that produces roughness (Panchal and Watkinson 1993), or fluctuations in power input to the probe. However, negative fouling resistances appear to be unique to FSW for this study.

Mean solids content of samples were 15.05 and 12.89% dry solids by weight for LSW and FSW, respectively. In samples subjected to microfiltration, fouling rate decreased 80% while solids content decreased only 19%. The level of solids in the material being tested for fouling tendencies appears to have a direct influence on fouling rate but requires additional work to determine which components were responsible for fouling.

CONCLUSIONS

Measurement of maximum fouling resistance and fouling rate had a coefficient of variation (COV) of 5.1 and 7.4%, respectively, indicating the technique has stable operation. Average rate of fouling for LSW was 5.3 times larger than for FSW (4.53×10^{-4} and $0.819 \times 10^{-4} \text{ m}^2 \text{ }^\circ\text{C}/\text{kW}/\text{min}$, respectively). Maximum fouling resistance of LSW was 10 times larger than FSW. Fouling rate decreased 80% while solids content decreased only 19% in samples subjected to microfiltration. This indicates that microfiltration removed steepwater constituents responsible for fouling and a more rapid fouling rate. Further analysis should be done to determine materials in steepwater responsible for fouling of heated surfaces, composition of material removed during microfiltration, and effect of solids content on fouling tendencies. Membrane filtration of steepwater before evaporation should have a positive impact on economical operation of evaporators used to concentrate steepwater.

This study indicates the potential usefulness of this technique to quantify effects of process changes on surface fouling and heat transfer efficiency. Additional work is needed to establish how the technique can be used to simulate actual fouling rates of commercial evaporator.

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