

FOULING OF EVAPORATORS IN MAIZE PROCESSING: DEVELOPING A FUNDAMENTAL UNDERSTANDING

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ABSTRACT

Evaporator fouling is a common, chronic problem during maize starch and ethanol production. To compensate for the consequences of fouling, capital costs are increased, operating costs are incurred, productivity is reduced and environmental impact is increased. Despite these issues, fundamental causes of increased fouling in maize processes are not understood. Process streams are biological in origin and have variable compositions. The objective was to develop an improved understanding of components that accelerate fouling in maize processing evaporators.

Two experiments were performed with commercial and model processing streams. In the first experiment, we used model materials (starch and sucrose) to study fouling characteristics of streams having well defined compositions. In a second experiment, commercial thin stillage samples were treated by adding carbohydrate materials (starch and sucrose) and tested in a fouling rig to simulate variation in composition. In the first experiment, sucrose had smaller effects than granular starch on heat transfer fouling. In the second experiment, inclusion of granular starch in thin stillage increased the rates of fouling. These introductory results have broad implications on process strategies.

INTRODUCTION

To remove excess water, evaporation is used in many processes containing biological and agricultural materials. In the USA, fuel ethanol is produced in more than 200 facilities. All use multiple effect evaporation to remove water. About 89% of fuel ethanol is produced using the maize dry grind process, the remainder uses maize wet milling. The dry grind process uses evaporation to concentrate the thin stillage process stream at ~7% total solids (TS) into condensed distillers solubles (35 to 45% TS) prior to mixing with wet grains and drying to produce distillers dried grains with solubles (DDGS). In maize wet milling, liquid material drained from the steeping step, light steepwater, is concentrated

by evaporation to form condensed fermented corn extractives, blended with fiber and dried to form corn gluten feed (CGF). At wet mills that produce ethanol, the material remaining after starch fermentation, distillers solubles, is concentrated in evaporators and typically added to CGF.

The most common multiple effect evaporator configuration used in the maize processing industry is the falling film evaporator. Evaporators used in dry grind and wet milling processes foul and must be cleaned on a regular basis. Information from industry communication is indicative that thin stillage evaporators are cleaned at 7 to 10 day intervals. Due to higher suspended and TS levels, steepwater evaporator cleaning occurs at a maximum of 7 day intervals. Cleaning has multiple negative impacts on the profitability and sustainability of the facility: increased capital investment due to over design of the evaporation process, increased energy use, labor and chemical costs, increased chemical disposal costs and reduced facility capacity since processing must slow during cleaning operations.

There have been relatively few publications on the fouling tendencies of streams from maize processing. For studies involving steepwater and distillers solubles from wet milling and thin stillage from dry grind, the method used to determine fouling tendencies has been an annular fouling probe configuration (Heat Transfer Research Inc., College Station, TX). This configuration has been used in other research to study fouling of heat transfer equipment (Fetissov et al., 1982; Asomaning and Watkinson, 1992; Panchal and Watkinson, 1993; Wilson and Watkinson, 1996).

Singh et al. (1999) measured fouling properties from thin stillage from dry grind and distillers solubles from wet milling. Thin stillage from dry grind had fouling rates that were 3× higher than rates of distillers solubles from wet milling. Distillers solubles from wet milling had lower fat contents; when refined corn oil was added to the distillers solubles, fouling rates increased.

Agbisit et al. (2003) investigated fouling tendencies of steepwater from maize wet milling which had been filtered using a microfiltration membrane (0.1 micron

nominal pore size). Filtered steepwater fouled at a rate 5× lower than unfiltered steepwater even though filtration removed less than 20% of TS. Wilkins et al. (2006) studied the effects of pH (3.5 to 4.5) on fouling characteristics and deposit composition. They found that induction periods (time required for fouling to be initiated) were reduced at pH 3.5. As pH increased, deposit composition was affected; protein concentrations decreased and total ash content increased. Phosphorus was the most abundant mineral in the deposits.

Arora et al. (2010) investigated fouling characteristics of thin stillage (7.0% TS) from a dry grind facility, filtered thin stillage (FTS) and diluted thin stillage (DTS); FTS and DTS each had TS contents of 3.5%. FTS was produced using the same microfiltration membrane used by Agbisit et al. (2003). Filtration reduced solids, protein and fat contents compared to initial thin stillage. FTS had fouling rates 23× and 8× lower than thin stillage and DTS, respectively. After 10 hr testing, FTS had mean heat transfer resistances 10× and 5× lower than thin stillage and DTS streams, respectively. FTS required twice the time to reach the maximum probe temperature of 200°C than did thin stillage. From these data, it was inferred that microfiltration selectively removed thin stillage components that accelerated fouling.

Research on fouling of maize processing streams is relatively young; many questions remain to be answered. Because process operations in either dry grind or wet milling vary daily; effects of process stream variation on evaporator efficiency are not well known. Individual processing methods vary among plants, with unknown effects on evaporator performance. For example, enzyme addition, such as phytase, has become more common to improve starch conversion efficiency; however, the effects of phytase addition on phosphorus and on fouling have not been investigated. Improvements in starch conversion efficiency may be offset by increased evaporator maintenance costs.

Operation of the decanter centrifuge in the dry grind process is considered a balance between sending insoluble solids to the thin stillage evaporator (operated by recovered energy) and to the DDGS dryer (which uses natural gas). Implications of sending additional solids to the thin stillage evaporator are not well understood.

The objectives of this work were to 1) investigate fouling tendencies of individual components of maize processing streams and 2) better understand fundamental causes that accelerate fouling in maize processing streams such as thin stillage.

METHODS

Theory of the annular probe

The annular fouling probe consisted of an annular flow passage with an electrically heated rod in the center. The rod consisted of a stainless steel sheath housing an electrical resistance heater and four thermocouples

located near the interior wall of the rod. The surface temperature on the outside of the rod was calculated as

$$T_s = T_w - \left(\frac{x}{k}\right) \left(\frac{Q}{A}\right) \quad (1)$$

Where T_s was the surface temperature; T_w was the temperature measured at the inner wall of the rod; x/k was the radial distance of the thermocouple from the surface divided by the thermal conductivity of the metal rod, obtained using a procedure given by Fisher et al (1975) and based on a graphical technique by Wilson (1915); Q was the power input (410±10 W) to the electrical heater; A was the area (0.0034 m²) of heated section of the rod based on the outside diameter of the rod.

The ratio x/k was reported as 0.061, 0.091 and 0.10 m²K/kW for each of the three thermocouples (Wilkins et al., 2006) used to measure T_w . The fourth thermocouple was used to control a circuit that would shut off the resistance heater if T_w exceeded 200°C. Individual rod surface temperatures associated with each thermocouple were calculated using T_w and x/k values and averaged for each time interval to calculate a mean T_s .

The overall heat transfer coefficient for the probe (kW/m²K) was determined by

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

Where T_b is the bulk fluid temperature (°C). Fouling resistance at time t is calculated as

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

Where,

R_f was the fouling resistance (at time t), m²K/kW; U_t was the overall heat transfer coefficient at time t , kW/m²K; U_0 was the initial ($t = 0$) overall heat transfer coefficient at the initiation of the fouling test

During fouling, material is deposited on the heated section of the probe. Since deposited material has a relatively low thermal conductivity, the heat transfer coefficient (U_t) is reduced over time, thereby increasing R_f .

Measurement of fouling resistance

Fouling resistance was measured using an annular probe at conditions similar to falling film evaporators. The same probe and associated equipment were used as in previous research (Agbisit et al., 2003; Wilkins et al., 2006; Wilkins et al., 2006; Arora et al., 2010). The annular probe was placed in a test loop consisting of a batch tank, positive displacement pump, flow meter and heat exchanger. Details of the equipment can be found in Arora et al. (2010); dimensions of the annular probe were reported in Agbisit et al. (2003). Thermocouple temperatures (probe and fluid bulk temperatures) and power input to the probe were acquired using a data logger. Prior to data collection, T_b was adjusted to the

desired temperature and the heater activated. When T_s reached 100°C , data collection began.

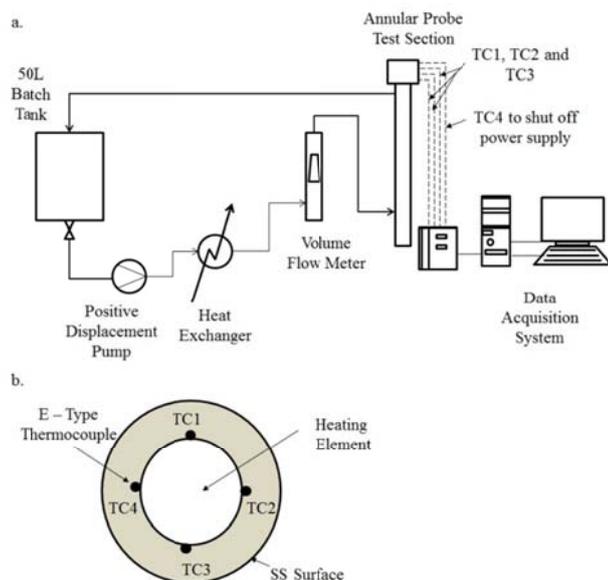


Fig. 1. Schematic of test rig (a) and probe (b).

Experiment A: Model thin stillage using starch and sucrose

Commercial thin stillage can be obtained from dry grind facilities for experimentation. However, composition of thin stillage is complex, varies due to process operations and among facilities, is perishable and changes in fouling characteristics during cold storage. For these reasons, this experiment investigated using model thin stillage which could be formulated in a repeatable manner using commercial corn starch and sucrose purchased at a local grocery supply store.

In this experiment, thin stillage was modeled by creating mixtures of tap water, starch and sucrose in varying ratios of starch (Sta) and sucrose (Suc). During each test (50 L batches), flow rate was 15.1 Lpm and $T_b = 50 \pm 2^\circ\text{C}$. Starch was insoluble in water at the bulk fluid temperature of 50°C and formed a suspension. Sucrose was completely soluble in water at 50°C . Therefore, starch and sucrose simulated soluble and insoluble fractions of thin stillage. There are no published data on the suspended and dissolved solids contents of thin stillage.

For one set of data, the effects of starch were observed by varying TS from 1 to 5% TS ($Re = 500$ to 4500). A second set of data was collected in varying Sta:Suc from 1:9 to 9:1 at 1% TS ($Re = 4500$). A third set varied the Sta:Suc ratio at 10% TS ($Re = 550$ to 1100). Batches (50 L) with varying TS and Sta:Suc were mixed continuously and adjusted to $T_b = 50^\circ\text{C}$. Power (410 ± 10 W) was supplied to the resistance heater of the probe; when T_s reached 100°C , the data collection began. Each test was terminated when T_s reached 170°C or $t = 10$ hr was reached. Treatments were arranged in a

randomized complete block design with at least three replications. At this time, only one replication has been completed.

Experiment B: Commercial thin stillage with added starch or sucrose

In the dry grind process, beer from fermentation is passed through a stripping column to remove ethanol and some water. The remaining material, whole stillage, is sent through a centrifuge to create two streams, wet grains and thin stillage. Processing parameters can affect centrifuge operation: fineness of initial ground corn, liquefaction and fermentation parameters as well as centrifuge operating conditions. Following fermentation, whole stillage, wet grains and thin stillage are routinely tested for total residual sucrose (a measure of all potentially fermentable carbohydrate). Residual sugar levels in DDGS are 6 to 10% db (NRC, 2012). Furthermore, centrifuge operation can change the amount of insoluble materials passing into the thin stillage stream.

Because ungelatinized starch is insoluble in water at 50°C , it was hypothesized that starch addition would increase the rates of fouling compared to sucrose addition or untreated thin stillage. Anecdotal information from industry indicates that higher insoluble levels in thin stillage accelerate evaporator fouling. Increased sugar levels were also hypothesized to increase fouling, since industry information has suggested that higher residual sugars in the thin stillage increases fouling.

To observe the effects of changing composition in commercial thin stillage, sucrose or starch was added to commercial thin stillage. Commercial thin stillage was obtained from a dry grind facility located in the Midwest US. All treatments (50 L batches) were adjusted to 7% TS. Thin stillage was diluted to 5% TS, then sucrose or starch was added to result in 7% TS. A control of 7% TS thin stillage was used as a control. During each test, flow rate was 15.1 Lpm, $Q = 410 \pm 10$ W, $T_b = 52 \pm 2^\circ\text{C}$ and $Re = 1600$. The treatments were arranged in a complete block design with four replications per treatment.

RESULTS

Experiment A: Model thin stillage

As percent starch in the model thin stillage increased, the time needed to reach 170°C decreased (Fig. 2). The slurry with 1:0 Sta:Suc (1% TS) required approximately twice the time to reach 170°C as the other treatments at higher TS. Following an initial rapid period of fouling, sloughing of the deposit from probe surface may have resulted in lower fouling resistance at 3% TS starch relative to 1% TS. Lower fluid shear and removal rate higher than the deposition rate may have resulted in lower fouling resistance relative to 1% TS (Fig. 4). At 5% TS starch, initial difference between deposition rate and removal rate was constant but the deposition rate increased after 180 min and R_f was higher than at 1% TS.

Rapid fouling relative to commercial thin stillage may be due to the availability of starch in the simulated mixture to foul the probe while other components in commercial thin stillage interact with the probe surface at a slower rate. Rapid fouling of starch mixtures may indicate that fouling depends strongly on starch content. Additional replication is needed to validate these trends.

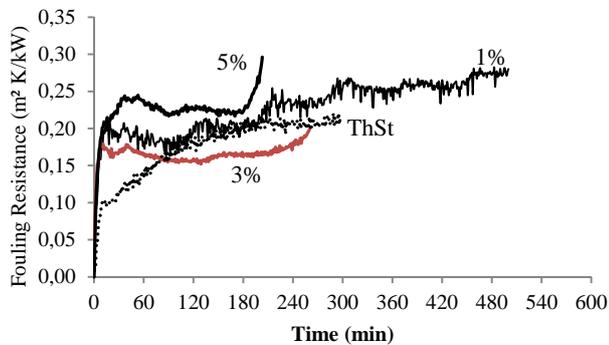


Fig. 2. Effects of starch concentration on fouling resistance. Model thin stillage treatments were composed of starch (1, 3, 5% TS) and tap water. Commercial thin stillage (ThSt) had 7% TS.

For tests conducted at 1% TS (Fig. 3), treatments with mixtures of starch and sucrose had R_f that ranged from 0.25 to 0.30 m^2K/kW at the end of the test period. However, the role of starch content seems less than straightforward as the fouling profiles for low starch (1:9) and high starch (9:1) contents were relatively low and close together (Fig. 3), while intermediate ratios (e.g., 7:3 and 3:7) displayed higher fouling resistances. Initial rapid increases in R_f were observed up to 60 min; thereafter the increases were more gradual and more asymptotic. Rapid increases observed in R_f may be from starch gelatinization on probe surface. If nucleate boiling were present, it is thought that R_f would have been negative at the start ($t < 20$ min) of the experiment, coinciding with rapid growth of vapor bubbles on the probe surface. Negative R_f values have been observed in other work (Singh et al., 1999; Agbisit et al., 2003; Wilkins et al., 2006; Wilkins et al., 2006; Arora et al., 2010) and thought to be caused by particle movement on the probe surface and nucleate boiling dominating heat transfer early in fouling tests (Panchal and Watkinson, 1993; Wilson and Watkinson, 1995).

For tests at 10% TS (Fig. 4), as the amount of starch increased from 0:10 to 5:5, fouling rates increased and time needed to reach $170^\circ C$ decreased. At 0:10, sucrose alone did not foul the probe. The treatment using 5:5 ratio rapidly fouled, indicating that starch is a more potent fouling constituent than sucrose in this model system. Tests with 10% TS and a 10:0 Sta:Suc ratio resulted in a viscous material that exceeded equipment limitations and were not conducted.

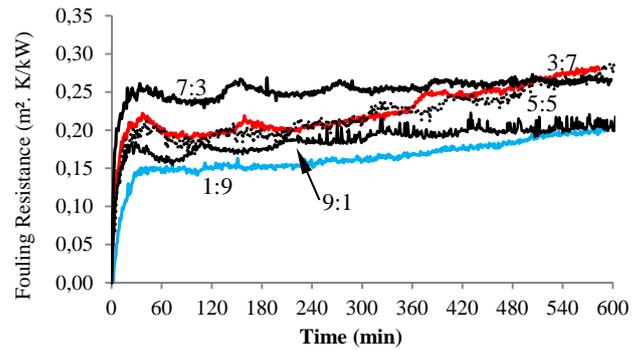


Fig. 3. Effects of starch and sucrose ratio (Sta:Suc) on fouling resistance at 1% TS in model thin stillage streams.

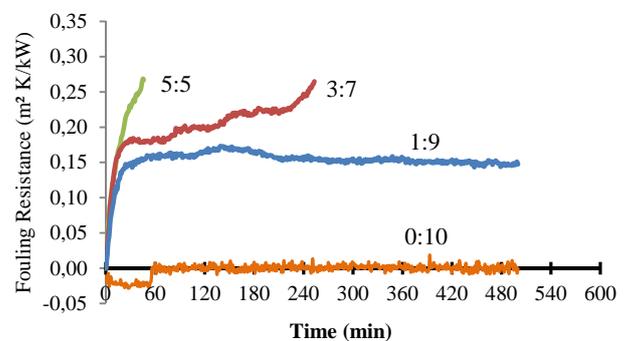


Fig. 4. Effects of starch and sucrose ratio (Sta:Suc) on fouling resistance at 10% TS in model thin stillage streams.

Experiment B: Commercial thin stillage with added starch or sucrose

Added starch in thin stillage increased the rates of fouling relative to thin stillage (Fig. 5). This is supported by increased fouling rates with higher levels of starch reported in Experiment A (eg, Fig. 4). The treatment with additional sucrose had similar or slightly lower fouling resistances. This appears to support observations from Fig. 4 where sucrose alone did not foul the probe under these conditions.

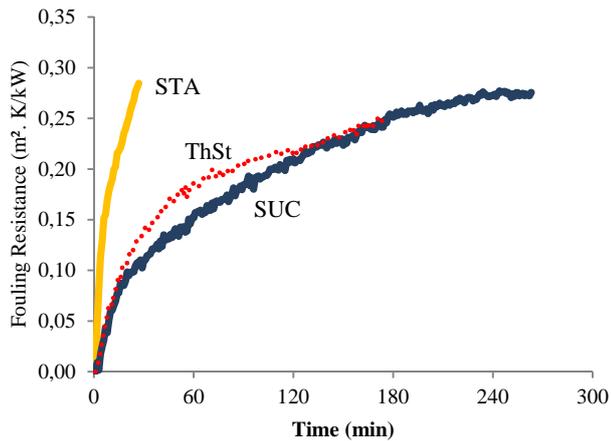


Fig. 5. Mean fouling resistances for commercial thin stillage (ThSt) and for thin stillage with added starch (STA) and added sucrose (SUC) at 7% TS.

DISCUSSION

We conducted tests involving starch and sucrose to evaluate the roles of these two components in fouling heated surfaces. In commercial thin stillage, concentrations of intact granular starch, starch polymers, dextrans, maltodextrins and simple sugars are tested and quantified as a single parameter, total residual sugar. During ethanol production, processing steps attempt to convert granular starch to sugar, but a percentage of the starch is not converted, and thus is not fermented into ethanol. This affects DDGS composition and nutritional characteristics (NRC, 2012), but also evaporator performance. Quantifying degree of polymerization is time consuming and costly and therefore not done on a routine basis. Therefore, our understanding of polymers on thin stillage fouling is limited. From anecdotal observations and personal communication with industry, it has been observed during commercial operations that higher total sugars in the stillage result in accelerated fouling. Our data are indicative that sugar alone would not cause rapid fouling (Fig. 4). Data obtained from our experiments suggest that starch had a higher impact on fouling rates when compared to sugar. Higher levels of starch in thin stillage increased fouling relative to thin stillage and sucrose added thin stillage. Experimental results suggest that compounds such as starch accelerate fouling and cause relatively more fouling when compared to simple sugars; more research is needed to validate this point. Processors should make certain that unconverted starch should not pass downstream following fermentation, not only to maximize biofuel yields, but also to increase processing efficiency through reduced fouling.

These experiments provide an initial understanding of how starch based compounds affect fouling. But these compounds are only a part of a complex mixture that comprises the thin stillage, steepwater and distillers solubles streams. Previous work (Rausch et al., 2005;

Belyea et al., 2006; Rausch et al., 2007) has shown that these streams in particular have relatively high concentrations of inorganic material (phosphorus, sulfur) that may interact with the organic components during evaporation and cause fouling. Other carbohydrates (e.g., fiber), amino acids and proteins (corn and yeast based) and inorganic components also may contribute to fouling characteristics, either individually or by interacting with one another. Wilkins et al (2006) found fouling deposits were up to 6× higher in ash content than thin stillage and that protein and ash contents of the deposits were affected by pH of the thin stillage. In the food processing and dairy industries, stream composition was reported to be a major factor influencing fouling. Components in the process stream interact with each other as well as at the heat transfer surface (Bansal and Chen, 2006).

In previous work, the annular probe has been used exclusively to simulate fouling of maize processing streams and has been found to be a useful tool (Singh et al., 1999; Agbisit et al., 2003; Wilkins et al., 2006; Wilkins et al., 2006; Arora et al., 2010). Additional work is needed to understand how the data from fouling probe experiments could be transferred accurately to fouling film evaporator operations.

CONCLUSIONS

1. In model thin stillage, the fouling resistance increased with increased starch concentration.
2. Higher levels of starch in model thin stillage shortened time needed to reach 170°C.
3. Model thin stillage containing only sucrose did not foul during 10 hr of testing.
4. In model thin stillage, there was a larger effect on fouling tendencies from starch content than sucrose content.
5. With increased starch levels in thin stillage, the rates of fouling were increased.

NOMENCLATURE

A	area of rod heated section, m ²
CGF	corn gluten feed
DDGS	distillers dried grains with solubles
DTS	diluted thin stillage
FTS	filtered thin stillage
Q	power input to the electrical heater, W
Re	Reynolds number, dimensionless
R _f	fouling resistance, m ² K/kW
STA	starch
SUC	sucrose
T _b	bulk fluid temperature, °C
T _s	surface temperature of the rod, °C
T _w	temperature measured at the inner wall of the rod, °C
ThSt	thin stillage
TS	total solids
U	overall heat transfer coefficient for the probe, kW/m ² K

U_0	initial ($t = 0$) overall heat transfer coefficient, kW/m ² K
U_t	overall heat transfer coefficient at time t , kW/m ² K
W	watts
x/k	calibration constant for the probe, m ² K/kW

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