

# Effect of Corn Oil on Thin Stillage Evaporators

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## ABSTRACT

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Removal of the germ at the front end of the dry-grind ethanol process using the Quick Germ process reduces the amount of oil in thin stillage. Thin stillage with 4–6% solids is dewatered to 25–30% solids by evaporation. Thin stillage evaporators in a dry-grind ethanol plant foul and have to be periodically taken down for maintenance and cleaning. Fouling caused by thin stillage containing different amounts of oil was studied using an annular fouling probe. It was determined that the rate of fouling in a dry-grind ethanol plant is three times higher when compared with that in a

wet-milling ethanol plant. The addition of oil to wet-milled thin stillage significantly affected the rate of fouling. Fouling resistance increased with an increase in oil concentration for wet-milled thin stillage up to a concentration of 1.41%. At a concentration of 1.47%, the rate of fouling decreased. As the concentration of oil increased in dry-grind ethanol thin stillage, the rate of fouling decreased. These results suggest that the Quick Germ process will reduce the rate of heat transfer equipment fouling in a dry-grind ethanol plant, which will decrease capital costs and maintenance costs.

Ethanol is produced from corn mainly by two processes: 1) wet-milling and 2) dry-grind (Rendleman and Hohmann 1993). In the dry-grind ethanol process only one coproduct is recovered, which is commonly known as the distillers' dried grains with solubles (DDGS). DDGS is made by combining distillers' wet grains with concentrated thin stillage and drying the mixed product. DDGS is sold as animal feed. Singh and Eckhoff (1996, 1997) combined aspects of the two processes to produce the Quick Germ process where germ is recovered using wet-milling degermination after whole corn is soaked in water for 3–12 hr. The remaining material is ground and processed by normal dry-grind ethanol methods. This change at the front end of the conventional dry-grind ethanol process adds germ (corn oil) as a new coproduct and increases the capacity of the plant. Based on a detailed engineering economic analysis, the Quick Germ process was profitable, with savings in the manufacturing cost of ethanol of 2.69 ¢/L (10.19 ¢/gal or \$0.265/bu) compared with that of the conventional dry-grind ethanol process (Singh and Eckhoff 1997). In an economic analysis, these authors also speculated on some savings associated with reduced fouling of the thin stillage evaporators.

Removal of germ at the front end of a dry-grind ethanol plant reduces the amount of oil in thin stillage. Thin stillage with 4–6% solids is concentrated to 25–30% solids by passing it through evaporators. These evaporators foul and have to be taken down periodically for cleaning and maintenance. Removal of oil from the thin stillage might reduce the fouling of thin stillage evaporators because oil would be present in very small amounts. Lipids are known to cause fouling of heat exchangers. Lipids copolymerize with proteins and produce dark-colored products that foul the heat exchanger surfaces (Lund and Sandu 1981).

Minimizing the fouling would reduce the downtime of heat transfer equipment for maintenance and might increase the plant capacity. Reduced fouling also would result in lower energy costs. However, the extent of reduction in fouling of heat transfer equipment in a dry-grind ethanol plant (due to removal of oil from thin stillage) is not yet known. Significant work on the fouling of heat transfer equipment has been done for the dairy industry (Burton 1968, Lund and Bixby 1975, Sandu and Singh 1991, Georgiadis et al 1998), but there is a lack of any information on the fouling characteristics of the heat transfer equipment in the corn processing or

ethanol industry. The objective of this study was to determine the effect of oil in thin stillage on the fouling of heat transfer equipment.

## MATERIALS AND METHODS

Fouling experiments were performed using an annular fouling probe (Heat Transfer Research Inc., College Station, TX) as shown in Fig. 1. Annular fouling probes have been used by several researchers (Fetisoff et al 1982, Asomaning and Watkinson 1992, Panchal and Watkinson 1993, Wilson and Watkinson 1995) to study the fouling of heat transfer equipment.

The annular fouling probe consists of an annular flow passage with an outer tube containing an electrically heated concentric rod. The design specifications of the fouling probe are given in Table I. The rod consists of a stainless steel sheathed resistance heater that has four thermocouples located close to the heating surface that monitor the surface temperature. The actual surface temperature is then calculated as:

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

where  $T_s$  is the surface temperature;  $T_w$  is the wall temperature;  $x/R$  is the calibration constant, provided by the manufacturer;  $Q$  is the power (watts) and is directly measured from the voltage and resistance measurements;  $A$  is the area of the heated section of the probe.

Fluid was pumped by a peristaltic pump from a 30-L feed tank through a flow meter to the annular fouling probe (Fig. 1). Another thermocouple in the feed tank measured the bulk fluid temperature. The bulk fluid temperature was maintained by placing the feed tank in a larger tank and circulating cold water in the larger tank. Heating was supplied by an electrical resistance heater so that the device operated under conditions of constant heat flux. The fluid-solid heat transfer coefficient was assumed to remain constant, giving a constant fluid-solid interface temperature. System temperatures and heating load were recorded through a datalogger into a computer and the heat transfer coefficient ( $U$ ) was calculated as:

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

where  $T_s$  is the surface temperature and  $T_b$  is the bulk temperature of the thin stillage.

The fouling resistance (FR) was calculated as:

$$FR = 1/U_t - 1/U_o \quad (3)$$

where  $U_o$  is the heat transfer coefficient at time  $t = 0$ .

Approximately 227 L (60 gal) of thin stillage was obtained from a corn wet-milling plant and a dry-grind ethanol plant. Samples were stored in a cold room at 4°C until tested. Compositional analyses for fat, protein, and crude fiber, and a sugar profile of the

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thin stillage was performed by a commercial analytical laboratory (Silliker Laboratories Group, Cedar Rapids, IA). All samples were tested for fouling within one week of the time received from the plant.

Two different fouling experiments determined the effect of oil in thin stillage on fouling. In the first experiment, four different amounts of refined corn oil (0.0, 0.5, 1.0, and 1.5%) were added to thin stillage from a wet-milling ethanol plant, in addition to the original amount already present in the thin stillage. Fouling data was collected for 12.83 hr. The compositional analysis of the thin stillage from the wet-milling ethanol plant is given in Table II.

In the second experiment, the same amounts of refined corn oil (0, 0.5, 1.0, and 1.5%) were added to thin stillage from a dry-grind ethanol plant. Fouling data was again collected for 12.83 hr. The compositional analysis of the thin stillage samples from the dry-grind ethanol plant is also given in Table II.

No duplicates were made on the first or second set of experiments. To determine the accuracy of the experimental setup, a third experiment determined the reproducibility of the fouling data. Four fouling trials were performed under similar conditions on thin stillage from a corn wet-milling ethanol plant. All of the reproducibility experiments were conducted for 14 hr.

Regression analysis was done using oil as an independent variable to generate a response surface. Analysis of covariance was done with oil as a classification factor to compare the regressions of the response against time for each amount of oil added to thin stillage. Pairwise comparisons of intercepts and slopes tested the homogeneity of the regressions generated. This was done for both the dry-grind and wet-milling experiments separately.

In all of the experiments, the bulk fluid was maintained at  $40 \pm 2.5^\circ\text{C}$ . The flow rate of the feed to the annular fouling probe was maintained at 0.26 m/sec.

## RESULTS AND DISCUSSION

Reproducibility of the fouling data for the same feed (thin stillage from a corn wet-milling ethanol plant) showed the fouling

**TABLE I**  
Design Specifications of an Annular Fouling Probe

Parameters	Value
Material	SS 316
Diameter (mm)	10.7
Annulus o.d. (mm)	25.4
Heated length (mm)	102.0
Heated length to thermo-couple location (mm)	78.0
Length from entrance to thermocouple location (mm)	294.0

apparatus to be very accurate in measuring FR (Fig. 2). A little scatter in the data was due to the characteristics of the peristaltic pump which pumped the feed in a quick pulsating action. However, the increasing trend of FR was the same for all four runs. FR for all four runs was almost negligible for the first 5 hr of testing. After 5 hr, the FR values started to increase, and the final average FR value at the end of 14 hr was  $0.067 \text{ m}^2\text{K/kW}$ .

In the first experiment with thin stillage from a corn wet-milling plant, a significant regression ( $R^2 = 0.813$ ) was obtained for FR:

$$\text{FR} = -0.0186 + 0.0066 \times \text{time} + 0.895 \times \text{oil} + 0.0939 \times \text{time} \times \text{oil} \quad (4)$$

This indicates that there is clearly a significant effect of oil on fouling of thin stillage from a corn wet-milling plant. Negative values of FR during the initial period were due to an increase in the solid-liquid heat transfer coefficient compared with the reference heat transfer coefficient at time  $t = 0$ . This is very common in fouling experiments and is caused by particles disturbing the developing thermal boundary layer (Wilson and Watkinson 1995), deposition that produces roughness (Panchal and Watkinson 1993), or the transients in the power supply.

Analysis of covariance also showed that there were significant differences in the slopes of the regression lines of FR versus time for various oil levels:

$$0\% \text{ Oil} \quad \text{FR} = -0.0301 + 0.0068 \times \text{time} \quad (5)$$

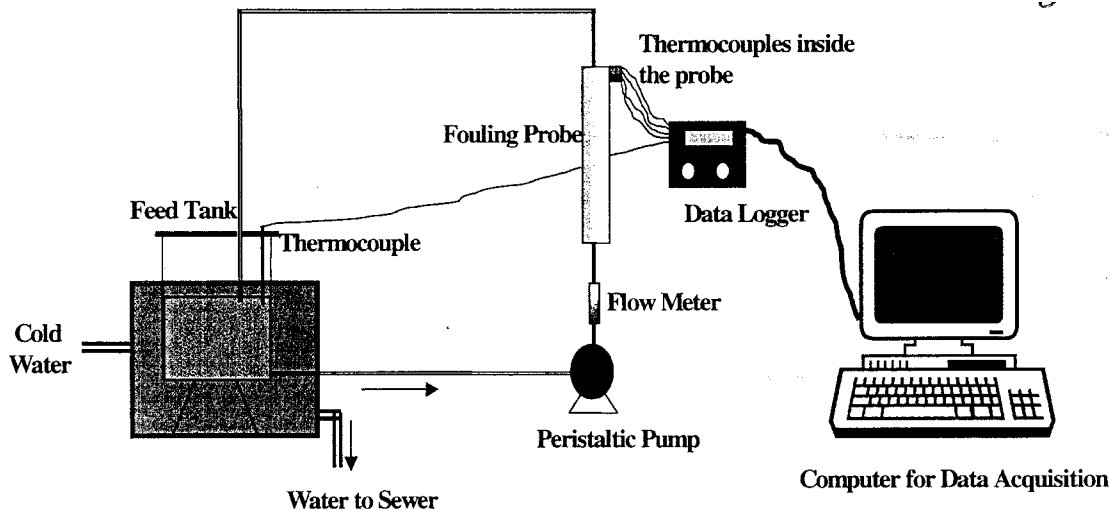
$$0.5\% \text{ Oil} \quad \text{FR} = -0.0334 + 0.0070 \times \text{time} \quad (6)$$

$$1.0\% \text{ Oil} \quad \text{FR} = +0.0060 + 0.0069 \times \text{time} \quad (7)$$

$$1.5\% \text{ Oil} \quad \text{FR} = -0.0192 + 0.0085 \times \text{time} \quad (8)$$

**TABLE II**  
Compositional Analysis of Thin Stillage Samples Obtained from a Corn Wet-Milling and Dry-Grind Ethanol Plant

Composition (% , wb)	Wet-Milling	Dry-Grind
Fat	0.01	1.09
Protein (%)	1.92	1.13
Crude fiber (%)	0.08	0.09
Sugar profile (%)		
DP4+	1.95	1.61
DP3	0.09	0.16
Maltose	0.86	0.32
Lactose	<0.01	<0.01
Sucrose	<0.01	0.02
Dextrose	0.02	0.09
Fructose	0.08	0.06



**Fig. 1.** Schematic diagram of apparatus used to measure fouling resistance of an annular fouling probe.

Intercepts for all of the four regression lines (for different amounts of oil added) were significantly different and the slopes for the first three oil levels were not significantly different (Fig. 3). This shows that the FR increases as the amount of oil increases from 0 to 1.0%. The intercept for the regression line with 1.5% oil was smaller when compared with regression lines for 0.5 and 1.0% oil. However, the slope of the line (with 1.5% oil) was significantly different compared with other regression lines and was steeper (i.e., higher rate of fouling compared with other amounts of oil).

The results from this series of experiments suggest that the fouling characteristics are changed by the presence of oil in wet-milled thin stillage. The FR values increase with the amount of oil present in the thin stillage up to a certain level (1.41% wb with this particular thin stillage). More than 1.41% oil in wet-milled thin stillage will cause the FR values to decrease, but the rate of fouling increases. Why the FR decreases with larger amounts (>1.41% wb) of oil is not known.

Lund and Sandu (1981) suggested that, by themselves, lipids in biological fluids play a minor role in fouling of heat transfer equipment. They react with other components of biological fluids and form compounds that are responsible for the fouling of heat transfer equipment. However, they suggested that large amounts of oil in biological fluids do not exhibit fouling tendencies because lipids get adsorbed onto metal surfaces and alter the character of high-energy surfaces, transforming them into unwettable or even autophobic surfaces. This could be the reason why large amounts of oil in wet-milled thin stillage caused less fouling than did smaller amounts of oil in thin stillage.

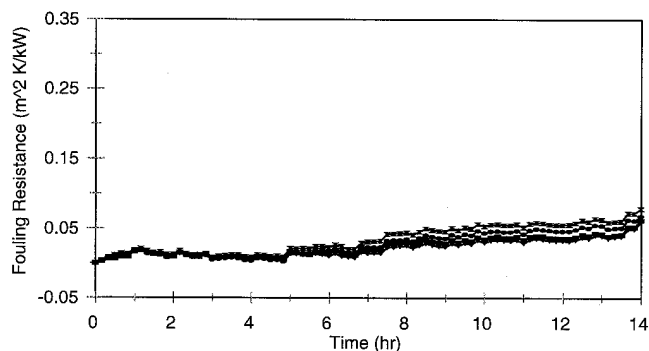


Fig. 2. Reproducibility testing of fouling apparatus in measuring fouling resistance of thin stillage obtained from a corn wet-milling plant.

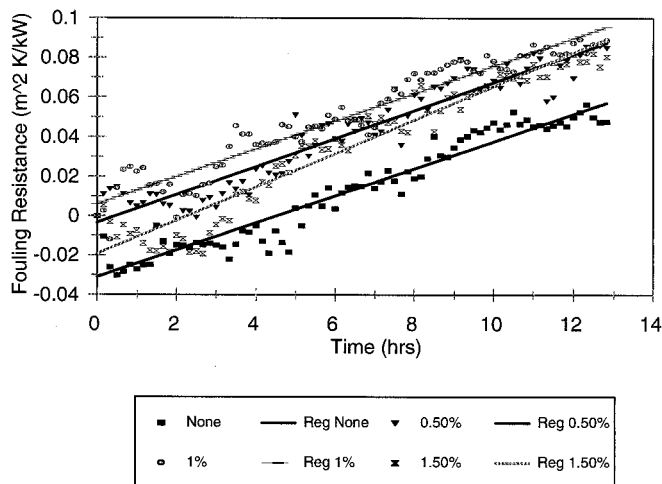


Fig. 3. Fouling resistance of wet-milled corn thin stillage with different (0, 0.5, 1.0, and 1.5%) amounts of oil added.

The effect of oil in thin stillage from a dry-grind ethanol plant was also significant ( $R^2 = 0.946$ ) on the fouling of heat transfer equipment (Fig. 4) and the response surface obtained was:

$$FR = 0.0425 + 0.0217 \times \text{time} - 3.708 \times \text{oil} - 0.429 \times \text{time} \times \text{oil} \quad (9)$$

Analysis of covariance showed that the intercepts for the regression line with 0% oil was significantly higher than for regression lines with other amounts of oil. Intercepts for regression lines with 0.5, 1.0, and 1.5% were not significantly different from each other.

$$0\% \text{ Oil} \quad FR = +0.0621 + 0.0208 \times \text{time} \quad (10)$$

$$0.5\% \text{ Oil} \quad FR = -0.0043 + 0.0201 \times \text{time} \quad (11)$$

$$1.0\% \text{ Oil} \quad FR = +0.0029 + 0.0188 \times \text{time} \quad (12)$$

$$1.5\% \text{ Oil} \quad FR = -0.0020 + 0.0141 \times \text{time} \quad (13)$$

Slopes of these regression lines were also significantly different from each other except for the regression lines with 0 and 0.5% oil. The slopes of regression lines with 1.0 and 1.5% oil were lower when compared with the regression line with 0% oil. This experiment suggests that an increase in the oil content of thin stillage from a dry-grind ethanol plant significantly reduces the fouling of the heat transfer equipment. Since the thin stillage obtained from a dry-grind ethanol plant already has a large amount of oil (1.08% wb more compared with that in wet-milled thin stillage), any additional amount of refined oil will reduce the fouling of the heat transfer equipment.

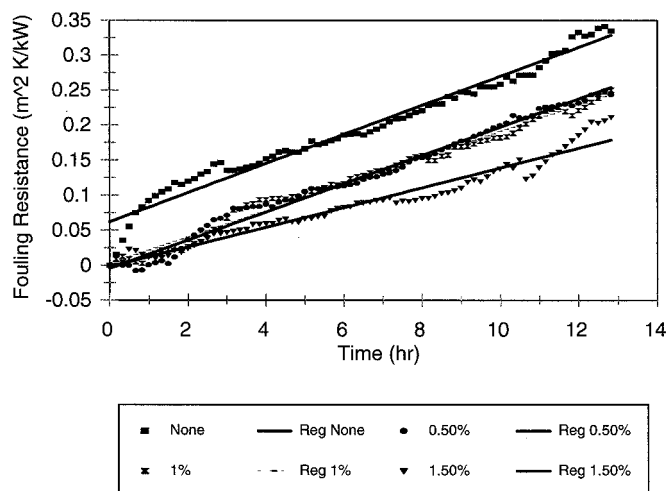


Fig. 4. Fouling resistance of dry-grind corn thin stillage with different (0, 0.5, 1.0, and 1.5%) amounts of oil added.

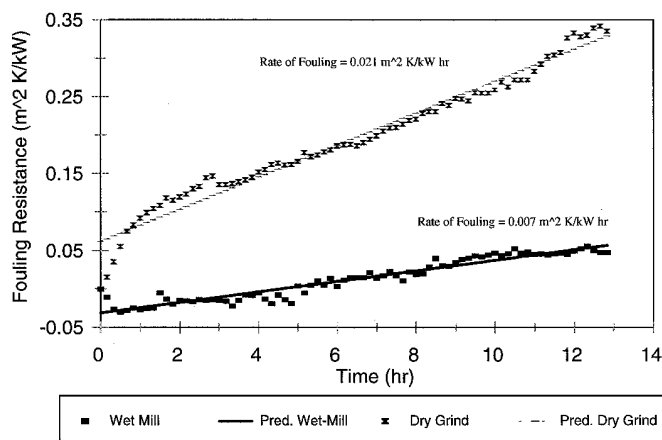


Fig. 5. Fouling resistance of thin stillage from a corn wet-milling and a dry-grind ethanol plant.

Comparison between refined corn oil and crude corn oil shows that there are some waxes and trace amounts of other compounds in crude corn oil that precipitate when heated (CRA 1996). Refined corn oil is, therefore, thermally stable compared with crude corn oil. Any excess amount of refined corn oil would, therefore, decrease the fouling of thin stillage, as suggested by Lund and Sandu (1981).

When comparing the FR values of thin stillage from a wet-milling and a dry-grind ethanol plant (with no additional amount of oil added to the thin stillage), the FR in the dry-grind ethanol plant was an order of magnitude higher as compared with that in the wet-milling plant (Fig. 5). The rate of fouling (change in the FR per unit of time, or the slope of the regression line with 0% oil addition) of thin stillage from the dry-grind ethanol plant was three times higher when compared with that from thin stillage from the corn wet-milling plant.

The biggest difference in the compositional analysis (Table II) of the thin stillage from both plants (dry-grind and wet-milling) was in the oil content. The thin stillage from the dry-grind ethanol plant had an oil content of  $\approx 1.09\%$  wb (16.5% db), while the thin stillage from the wet-milling ethanol plant had an oil content of 0.01% wb (0.14% db). Compositional analyses other than protein content were quite similar for both thin stillage fractions. The protein content in the dry-grind thin stillage was  $\approx 0.8\%$  wb lower when compared with that in the wet-milled thin stillage. Unconverted starch, which is also a known contributor to fouling of heat transfer equipment (Madson and Monceaux 1995), was also 0.34% lower in the dry-grind ethanol thin stillage.

Although the thin stillage from the Quick Germ process would be different from wet-milled thin stillage, Singh and Eckhoff (1996) found that germ yields with the Quick Germ process were comparable to those of the conventional wet-milling process. This suggests that the thin stillage from the Quick Germ process will have an oil content comparable to or slightly higher than that observed in corn wet-milled thin stillage, but will have considerably lower oil content when compared with conventional dry-grind thin stillage. Based on the comparison of the fouling data between wet-milling and dry-grind thin stillage, we believe that there would be significant reduction in fouling with thin stillage produced from the Quick Germ process.

## CONCLUSIONS

Fouling resistance increases with an increase in the amount of oil in wet-milled thin stillage up to a certain level, beyond which it starts to decrease. As the amount of oil increases in thin stillage

from a dry-grind ethanol plant, the rate of fouling decreases. The rate of fouling in a dry-grind ethanol plant is three times higher when compared with that in a wet-milling ethanol plant. Based on these results and the previous work done on comparison of the germ yields for the Quick Germ and conventional wet-milling processes, we believe that the thin stillage from the Quick Germ process would contain a smaller amount of crude oil and, therefore, would result in reduced fouling of evaporators. The exact amount of reduction in fouling with the Quick Germ thin stillage is not known. More research with actual Quick Germ thin stillage samples is required to determine the exact reduction in fouling and the savings associated in maintenance and energy costs.

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