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Short communication: Evaluation of a sol-gel–based stainless steel surface modification to reduce fouling and biofilm formation during pasteurization of milk

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ABSTRACT

Milk fouling and biofilms are common problems in the dairy industry across many types of processing equipment. One way to reduce milk fouling and biofilms is to modify the characteristics of milk contact surfaces. This study examines the viability of using Thermolon (Porcelain Industries Inc., Dickson, TN), a sol-gel-based surface modification of stainless steel, during thermal processing of milk. We used stainless steel 316L (control) and sol-gel-modified coupons in this study to evaluate fouling behavior and bacterial adhesion. The surface roughness as measured by an optical profiler indicated that the control coupons had a slightly smoother finish. Contact angle measurements showed that the modified surface led to a higher water contact angle, suggesting a more hydrophobic surface. The modified surface also had a lower surface energy (32.4 ± 1.4 mN/m) than the control surface (41.36 ± 2.7 mN/m). We evaluated the susceptibility of control and modified stainless steel coupons to fouling in a benchtop plate heat exchanger. We observed a significant reduction in the amount of fouled layer on modified surfaces. We found an average fouling weight of 19.21 mg/cm^2 and 0.37 mg/cm^2 on the control and modified stainless steel coupons, respectively. We also examined the adhesion of *Bacillus* and biofilm formation, and observed that the modified stainless steel surface offered greater resistance to biofilm formation. Overall, the Thermolon-modified surface showed potential in the thermal processing of milk, offering significantly lower fouling and bacterial attachment than the control surface.
Key words: milk deposit, bacterial attachment, thermal processing, surface treatment

Short Communication

Milk fouling and biofilms are expensive and persistent problems for the dairy industry (Baier, 2006; Mérian and Goddard, 2012). Fouling of milk components is a common occurrence across many types of dairy processing equipment (e.g., plates, pipes, and flow channels; Sadeghinezhad et al., 2015). Previous studies have stated that the effect of fouling on the dairy industry accounts for up to 80% of total operating costs (Bansal and Chen, 2006). Fouling during pasteurization of milk on the stainless steel surfaces of plate heat exchangers can be classified as type A fouling, consisting of 50 to 60% proteins and 30 to 35% minerals. Fouling necessitates frequent clean-in-place, leading to increased down time and reduced production (de Jong, 1997).

The fouling layer on stainless steel surfaces accelerates the adhesion of bacteria and encourages the development of biofilms (Simões et al., 2010). The formation of biofilms during milk processing can lead to food spoilage and economic losses (Bremer et al., 2006). The major components of biofilms include bacteria and extracellular polymeric substances produced by bacteria (Flint et al., 1997; Mittelman, 1998). The fouled layer and extracellular polymeric substances guard the microorganisms and help them survive most of the cleaning protocols used in the dairy industry. Biofilms formed by *Bacillus* spp. are resistant to high stress, are very hydrophobic, and can easily attach to processing equipment (Faille et al., 2002; Simões et al., 2010).

One way to control undesirable fouling and biofilms is to modify the surface properties of the processing equipment. Sol-gel surface modification converts inorganic liquid substances into a gel that can be applied on metal surfaces to improve the surface properties. Thermolon is a sol-gel-based surface modification developed from an inorganic ceramic polymer. According to its manufacturer (Porcelain Industries Inc., Dickson, TN), "It is environmentally friendly, durable, and most importantly, it has been approved by the FDA as a food contact surface (FDA 21CFR 175.300)." The objective

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of the present study was to evaluate the effectiveness of Thermolon surface modification on mitigating fouling and adhesion of microorganisms during the thermal processing of milk.

Stainless steel 316L coupons (25.4mm × 25.4mm × 0.5 mm) with a 2B finished surface were provided by Stainless Supply (Monroe, NC) and used to mimic the surface of a typical plate heat exchanger. Thermolon surface modification was done by Porcelain Industries (Dickson, TN).

We measured the contact angles of 3 liquids with known surface tension (Costanzo et al., 1990): water (72.8 mN/m), 1-bromonaphthalene (44.4 mN/m), and ethylene glycol (48 mN/m) on the stainless steel control and Thermolon-modified surfaces using a static method with a FTA 1000 B Drop Shape instrument (Portsmouth, VA) at room temperature.

Because $\gamma_s^{TOT} = \gamma_s^d + \gamma_s^p$ (where γ_s^d and γ_s^p are the dispersive and polar components of the solid surface energy, respectively), solid surface energy can be determined by combining Young's equation (Equation [1]) and the Owens-Wendt approach (Equation [2]; Santos et al., 2004):

$$\gamma_{sl}^{TOT} = \gamma_s^{TOT} - \gamma_l^{TOT}\cos\theta, \quad [1]$$

where γ_{sl}^{TOT} is the total interfacial surface tension between solid and liquid, γ_s^{TOT} and γ_l^{TOT} are the surface tension of the solid and the liquid, respectively, and the contact angle θ; and

$$\gamma_{sl}^{TOT} = \gamma_s^{TOT} + \gamma_l^{TOT} - 2\left(\gamma_l^d\gamma_s^d\right)^{\frac{1}{2}} - 2\left(\gamma_l^p\gamma_s^p\right)^{\frac{1}{2}}, \quad [2]$$

where γ_l^d and γ_l^p are the dispersive and polar contributions of the liquid (Table 1).

We determined the surface roughness of different substrates using a Wyko NT1100 Optical Profiler (VEECO, Tucson, AZ). The field of view was 450 × 592 μm, and the results were reported as an average of duplicate samples, with 5 scans of each sample.

We conducted fouling experiments in a laboratory-designed benchtop plate heat exchanger as shown in Figure 1, fitted with control and modified coupons, respectively. Different batches of raw milk were collected from the dairy plant at Kansas State University and kept at 4°C before use. Each batch of milk was divided into 2 for tests using control and Thermolon-modified stainless steel coupons. The milk inlet temperature was set at 40°C, and the hot water temperature in the second water bath was maintained at 88–90°C to maintain the milk outlet temperature at ~85°C. Raw milk was pumped through the benchtop plate heat exchanger for 7.5 h, with a flow rate of 22 mL/min. After each test, the plate heat exchanger was dissembled, and the weight of the milk deposit on the coupons was measured after air drying for 15 min by recording the difference in weight of the clean plates versus the air-dried fouled substrates.

We carried out scanning electron microscope analysis using the Hitachi S-3500N (Tokyo, Japan). Clean stainless steel coupons (control and modified) were analyzed directly at a 20-kV accelerating voltage. Milk fouling on the coupons was air-dried at room temperature and then coated with a 10-nm layer of 99% gold. The fouling layer was then observed at a 10-kV accelerating voltage.

We used aerobic spore-forming *Bacillus licheniformis* (ATCC 6643) to develop biofilms on control and modified stainless steel coupons at 50°C using the method described in our previous study (Jindal et al., 2016). The biofilm embedded cells in 72 h, and matured biofilms formed on control and modified stainless steel coupons were enumerated by swabbing an area of 6.45 cm^2 and plating on brain heart infusion (BHI) agar plates (Jindal et al., 2016).

Milk fouling and biofilm formation tests were conducted in triplicate. We calculated milk deposit weight and bacterial counts as mean values and standard deviations. We compared fouling, bacteria attachment, and surface property results using SAS software (version 9.4; SAS Institute Inc., Cary, NC) and set the least significance difference at $P < 0.05$.

Table 1. Surface tension values (mN/m; Costanzo et al., 1990; Santos et al., 2004) and contact angle for the liquids used on control and modified coupons[1]

Liquid	γ_l^{TOT}	γ_l^d	γ_l^p	Contact angle (°)	
				Stainless steel	Thermolon
Water	72.8	21.8	51	82.9 ± 1.2	105.5 ± 0.9
1-Bromonaphthalene	44.4	44.4	0	21.7 ± 1.0	44.9 ± 1.5
Ethylene glycol	48	29	19	65.9 ± 1.4	72.4 ± 0.8

[1]Values are an average of 9 measurements (3 distinct regions on 3 independent samples) ± SE. γ_l^{TOT} is the surface tension of the liquid; γ_l^d and γ_l^p are the dispersive and polar contributions of the liquid.