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COMPARISON OF WHEY PROTEIN MODEL FOULANTS FOR STUDYING CLEANING OF MILK FOULING DEPOSITS

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The need to generate consistent and reproducible fouling layers for the study of cleaning mechanisms has prompted the use of whey protein solutions as model systems. We have employed both forced flows over heated surfaces—which are thereby fouled—or natural convective flows subjected to thermally induced gelation in sealed vessels. Specifically, deposits were generated from the same whey protein powder (30% WPC) by (1) formation of 'surface deposits', from a solution flowing over a heated surface, (2) gelation, termed 'G80', where the bulk temperature in the sealed vessel remains $<72^{\circ}$C, thus avoiding aggregation in the bulk, and (3) gelation, 'G90', with bulk temperature $>72^{\circ}$C. Evident differences in deposit structure and properties were recorded and related to differences in cleaning behaviour observed using fluid dynamic gauging (FDG) in three modes: (i) quasi-stagnant mode, (ii) quasi-stagnant mode with fluid sampling, and (iii) duct flow with thermal resistance measurements. The surface and G80 deposits showed similarities, while the G90 deposit differed markedly. Larger differences between the surface and G80 deposits were observed under cleaning-in-place conditions.

Keywords: cleaning; cleaning-in-place; fluid dynamic gauging; gelation; whey protein.

INTRODUCTION

Fouling is a serious problem in thermal processing units used in the dairy industry as the deposits reduce operating efficiencies and can compromise the product by contamination and by encouraging unwanted microbiological growth. The mechanisms responsible for deposit generation depend on the operating temperature and are reflected in the composition of the deposit. Burton (1968) labelled the proteinaceous deposit formed at lower temperatures, consisting predominantly of denatured and unfolded aggregates of whey proteins, Type A, while the harder 'milk stone' found at higher temperatures and containing noticeably more mineral salts, especially calcium phosphates, Type B. Detailed accounts can be found in Visser and Jeurnink (1997) and Xin *et al.* (2002). The continued dominance of thermal processing operations for pasteurization and sterilization, where the milk or dairy product is subjected to temperatures higher than those which lead to proteinaceous deposit formation, has resulted in a concommitant need to clean equipment regularly and therefore to understand the mechanisms involved in cleaning (e.g., Changani *et al.*, 1997). One of the challenges involved in this area is the generation of reproducible and representative fouling deposits for subsequent study.

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Using native milk in such studies is problematic, owing to variation in its composition over the lactation cycle and the need to control microbiological activity. Laboratory studies of Type A deposits have lately tended to use foulants generated from whey protein solutions, since these deposits contain mostly water and protein (and predominantly β-lactoglobulin). Many workers have employed solutions based on reconstituted whey protein concentrate (WPC), circulated through a heat transfer system, reflecting the conditions operating in the industrial process (e.g., Bird and Fryer, 1991). The whey protein concentration used is close to that in the native milk or dairy product and the protocols yield reproducible layers for the studying of cleaning. We term these foulant layers 'surface deposits'. Cleaning involves treating the deposit with aqueous NaOH solution, which causes the protein matrix to swell and subsequently dissolve. Cleaning is monitored visually and often by measuring the change in protein concentration in the caustic solution over time. The cleaning rate or flux can then be calculated; for Type A deposits this often exhibits three phases: an initial phase where the rate increases and the deposit swells, a second 'plateau' phase marked by a nearly uniform cleaning rate, followed by a decay phase as the deposit is finally removed.

Recently, Chen and co-workers (Xin *et al.*, 2002, 2004; Mercadé-Prieto and Chen, 2005) have advocated the use of whey protein gels in cleaning studies. These gels are formed by thermal denaturation of higher concentration WPC solutions (typically 25 wt% protein, compared to

3–4 wt% in milk, as Lalande and others (1985) report milk deposit contents of ~25 wt%), in capsules (to study reaction) or inside steel tubes (so that flow effects can also be investigated). The gelation route affords several benefits, including ease of preparation, and facilitation of the study of rheology and kinetics. The reported cleaning rate behaviour reflects that observed for 'surface deposits', formed on heat transfer surfaces from flows of lower concentration WPC solutions, undergoing forced flow.

The aim of the work reported here was to compare the cleaning behaviour of such surface and gel deposits. Formation conditions strongly affect the structure and properties of protein gels (Lefèvre and Subirade, 2000): their impact on subsequent cleaning behaviour has not been reported. Surface deposits are usually formed under conditions of higher shear stress imparted by the flow and can be subject to ageing at the heat transfer surface. We have attempted to reproduce the WPC deposit generation protocols reported by Xin *et al.* (2002) (gel deposits) and Tuladhar *et al.* (2002) (surface deposits). We compare their cleaning behaviour under a set of conditions, namely 0.5 wt% NaOH solution at 20°C, where the rates are relatively slow and differences between the different deposits will be particularly evident.

Cleaning was monitored by the fluid dynamic gauging (FDG) technique developed by Tuladhar *et al.* (2000, 2002). Figure 1 illustrates the principle of the technique. A detailed description is given in the aforementioned papers. The layer to be studied is located on a rigid, impermeable surface and is immersed in a Newtonian liquid: the gauging nozzle of throat diameter d_t is moved close to the layer surface and suction applied so that liquid is continuously withdrawn through the nozzle and its flow rate measured. This flow rate is very sensitive to the clearance, h, between the nozzle and the layer surface when the nozzle is close to the surface ($h/d_t < 0.25$). The location of the gauge relative to the impermeable surface, h_o, is measured accurately, separately, so that the thickness of the layer, δ, can be calculated from $\delta = h_o - h$. The accuracy of δ measurements in this work was $\pm$ 20 μm.

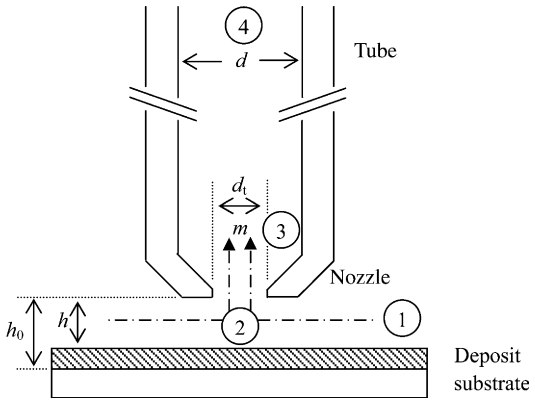

Figure 1. Principle of fluid dynamic gauging. Imposing a siphon effect at (4) results in the process liquid being sucked from the surrounding reservoir (1) into the nozzle of the gauge (2); the fluid travels through the gauge (3) and is discharged at position (4). For a defined set of conditions a calibration curve of nozzle-film surface distance, h, versus the discharge flow rate, m, can be obtained experimentally.

Deposits were formed on stainless steel plates and studied using FDG in a quasi-stagnant environment, where the only flow present was that due to the gauge, and in duct flow, replicating cleaning-in-place (CIP), as reported by Tuladhar *et al.* (2002). A new mode is reported here where the gauge is also used as a sampling system: the liquid withdrawn through the gauge is taken from the 'boundary layer' of fluid above the deposit layer. The gauge is operated at almost constant flow rate and so can be used to track the evolution of solubilised protein over time (in effect, a local version of a cleaning rate assay). The Xin *et al.* protocols could not be reproduced exactly because the FDG technique currently requires the gel to be located on a flat plate, whereas they coated the inside of a tube by partially filling and rotating it during the gelation period. FDG allows monitoring of the thickness of the fouling layer in real time and tracking of the onset of deformation, i.e., disruption of the layer by the stresses imposed by the flow.

METHODS AND MATERIALS

Deposit Formation

Surface deposits

These were generated using a protocol and apparatus similar to that reported by Tuladhar *et al.* (2002). Whey protein solutions with a protein concentration of 3.5 wt% were produced in 15 L batches by dissolving WPC30 powder (30 wt% protein, Volac International Ltd, UK) in reverse osmosis (RO) water at room temperature. The pH was adjusted to 6.0 using 0.1 M HCl. The solution was recirculated through the deposition cell shown in Figure 2(a). This laboratory co-current heat exchanger consists of two Perspex blocks each containing a duct of 15 mm square cross-section, separated by a gasketed 1 mm thick stainless steel plate. The blocks are clamped together to seal the system and the flows are vertical to inhibit air bubbles from attaching to the plate. An insulated brass entry duct, of length 450 mm, ensures that the process fluid flow is fully developed.

The WPC solution is pumped from a holding tank through a coiled copper heating tube (13 mm i.d. ×12 m) held in a water bath before entering the deposition cell. The bath temperature of 85°C ensures a WPC inlet temperature of 74°C and thus the occurrence of protein denaturation (Belmar-Beiny *et al.*, 1993), except during the initial 30 min period as the system warms up. The WPC mean velocity was maintained at 0.23 m s^{-1} (inlet Reynolds number ~9000). Hot water is recirculated at 90°C and 0.19 m s^{-1}on the utility side, giving rise to an initial plate temperature of 82°C, which can increase to 86°C as the fouling layer develops (Tuladhar, 2001).

The test plates used were made from 1 mm thick 316 stainless steel of exposed dimensions 150 mm long × 15 mm wide. Plates were cleaned by immersion in 0.5 wt% NaOH at 40°C for 30 min, followed by immersion in room temperature 0.1 M HCl for 30 min, rinsing after each immersion and storing in RO water until use. After a fouling run, the plate was rinsed in RO water to remove excess solution, drained and weighed, rinsed and then stored in a humid environment at 4°C for up to 2