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ABSTRACT

In cleaning, the force required to disrupt the deposit and remove it from the surface tends to be inferred from flow data, rather than being known directly. A novel micromanipulation technique has been developed to measure directly the adhesive strength of food fouling deposits on a stainless steel surface. A T-shaped stainless steel probe pulls fouling deposits away from the surface to which they are attached. The apparent adhesive strength between the fouling deposits and the substrate can be measured as the work required to remove the deposits per unit area from the substrate. Tomato pastes and whey proteins have been used as model deposits. The influences of process variables and different cleaning strategies can be identified, and the differences in cohesive and adhesive behaviour of the materials identified. The results can be compared to larger-scale cleaning processes.

INTRODUCTION

Fouling devices by foods is a severe problem, in which a range of components, both organic and mineral are deposited. Fouling deposits from a range of materials, including tomato juice, grape juice and milk, have been studied; the composition of deposits is different from the fluid (for example, see Robbins et al, 1999). Fouling profiles can be significantly affected by small changes in fluid composition: for example, the addition of calcium phosphate to whey protein concentrate changes the rate, extent and composition of the deposit (Christian et al, 2002). The microscopic appearance of tomato soil was that of clotted tomato with unclotted juice adhering to the denser, sticky soiling film (Cheow and Jackson, 1982). Fouling deposits form as a result of adhesion of species to the surface and cohesion between elements of the material.

The aim of most cleaning research is to devise ways of optimising removal; i.e. to minimise the cleaning time in terms of the effect of flow velocity, cleaning agent chemical concentration and other variables. Cleaning is a critical operation: food processing require protocols that leave surface both microbiologically and physically clean, to eliminate hygiene problems and obstruction of the equipment. Extensive development work has been carried out to produce cleaning-in-place (CIP) equipment and protocols which meets these requirements, but it is not known in practice how optimal these protocols are. One key parameter, being the force required to disrupt the deposit and remove it from the surface, is not known directly. Commonly this is only found indirectly, in terms of surface shear stresses inferred from pressure drop data or from correlation (for example, see Fryer and Slater, 1987). Devices such as the radial flow cell, which provide a range of shear stresses, have been used to study adhesion (Klavenes et al, 2002). On a smaller length scale, atomic force microscopy (AFM) has been used to characterise surface and fouling (such as Prabhu et al, 2002; Weiss et al, 2002).

The forces holding deposits onto surfaces will be both cohesive (between parts of the deposit) and adhesive (between deposit and surface). The principal factors responsible for adhesion between surface and foulant include: (i) van der Waals forces, (ii) electrostatic forces, (iii) and contact area effects; the greater the area the greater the total attractive force (Bott, 1995). Visser (1988) notes that effects of surface hydration and steric hindrance that prevent close approach of particles and surface will act to reduce adhesion. Cohesive forces between deposits may result from covalent bonding such as the disulphide linkage between β-lactoglobulin molecules formed on heating.

Low-adhesion coatings (such as Nejim et al, 1999) have been shown to reduce fouling in some situations such as mineral scales. Zhao et al (2002) demonstrate that biofouling can be reduced by changing surface energy, and link this to adhesive energy between surface and deposits. An understanding of the interaction between deposits and surfaces is clearly critical in cleaning.

This paper describes the development of a micromanipulation technique to measure the adhesive strength between the fouling deposits and the substrate. Micromanipulation equipment has been developed at Birmingham to study adhesion (Chen et al, 1998) as part of other studies on the physical properties of biological systems (Thomas and Zhang, 2000). The technique has been developed to study foods; here we have used both tomato paste and whey protein deposits as model fluids, as both starch and dairy product fouling is widespread in the food industry.

THE MICROMANIPULATION METHOD

Micromanipulation has previously been used to measure the mechanical properties of a biofilm on a glass surface (Chen et al, 1998) and preliminary results of the method have been discussed (Liu et al, 2002). The

technique measures the strength of food fouling deposits using a T-shaped probe made of stainless steel chip, dimension 30 x 6 x 1 mm. The T-shaped probe was connected to the output of a transducer (Model BG-1000, Kulite Semiconductor, Leonia, NJ. USA) which was itself mounted on a three dimensional micromanipulator (Micro Instruments, Oxon, UK). A schematic of the T-shaped probe, fouling sample and stainless steel disc is shown in Figure 1.

Prior to measurement, the dish containing the sample was placed on a microscope stage held by a second micromanipulator. The gap between the bottom edge of the T-shaped probe and the surface was adjusted to 10-20 μm by fine tuning with a digital level indicator (Model ID-C112MB, Mitutoyo Corp., Japan). The length of the probe was made larger than the diameter of the test disc to minimise edge effects. The probe pulled the deposits horizontally at a constant speed of 2.6 mm/s. The force exerted on the probe was recorded at 100 Hz by a PC 30-D data acquisition board (Amplicon Liveline, Brighton, UK), and recorded on video (Q600, Leica Cambridge, UK).

FOULING DEPOSITS

Two materials have been used.

Tomato paste

This was purchased from a local supermarket. the composition was (wt%): 4.5 protein, 12.9 carbohydrate, 12.6 sugar, 0.2 fat, 2.8 fibre and 67 water. Experiments were carried out in which paste was spread onto stainless steel discs, heated, and then removal of the baked deposit studied.

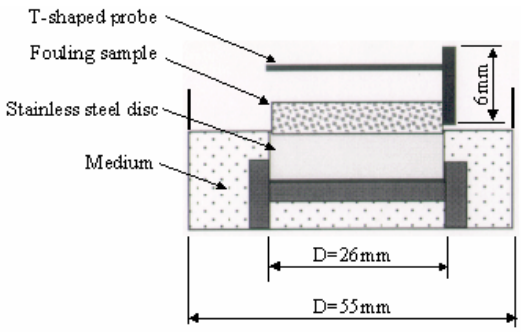


Figure 1: Schematic of the T-shaped probe, fouling sample and stainless steel disc.

Tomato paste (usually 0.8g) was spread evenly over the whole surface of a stainless steel disc (26mm diameter). The roughness of the surface was measured using a Talysurf 120l (Rank Taylor Hobson Ltd, Leicester, UK). R_a, the arithmetic mean of the absolute departure of the roughness profile from the mean line, was found to be 0.2 μm for the surface used, unless otherwise stated below. The discs were then baked in a pre-heated laboratory oven (generally at 100°C for 1 hour). The effect of this is to dry the deposit and bake the paste into a hard form that is difficult to remove. After baking, the discs were allowed to cool to room temperature, and then put into a plastic petri dish. Distilled water was then added to submerge the tomato layer. After a predetermined hydration time (at 20°C unless otherwise stated), the water was removed prior to force measurement.

Milk protein

Deposits were prepared using a plate heat exchanger unit which has previously been described (Christian et al, 2002; Schreier 1995). A sidestream unit was used which consisted of a rectangular flow channel into which six stainless steel disks (20/30 mm diameter) could be inserted. This flow channel was placed at the outlet of the UHT section of the exchanger, where the wall temperature was 90°C and the fluid temperature was ca. 85°C. The discs were fouled with protein [composition: 70 - 80 % protein, 2 % calcium and 1% phosphorus] of amount 0.42 g and about 1.3 mm thick.

TYPICAL EXPERIMENTAL RESULTS

The force required to remove the deposit was measured by drawing the micromanipulation arm across the surface of the deposit, as shown in the video stills of Figure 2(a). A typical curve showing the measured force versus the sampling time is shown in Figure 2(b).

The sample was pulled horizontally from the initial contact point A; points B and C are the centre of the disc and the final edge respectively. As shown in Figure 2(b), during pulling the force measured by the micromanipulator first increased to a maximum (A to B), corresponding to the maximum width of the deposit and then decreased (B to C). The saw-tooth shape of the force curve was attributed to a series of interactions between the deposit and the surface, i.e. to successive waves of sample deformation and detachment during pulling. The pictures show typical deposit removal; the deposit was pulled from the surface in one piece. Once the sample was pulled away from the surface, no significant force was imposed on the probe as shown C to D in Figure 2(b).

The total work, W (J) done by the applied force, F(t) to remove the deposit may be calculated as the integral:

$$W = \frac{d}{(t_C - t_A)} \int_{t_A}^{t_C} F dt \qquad (1)$$

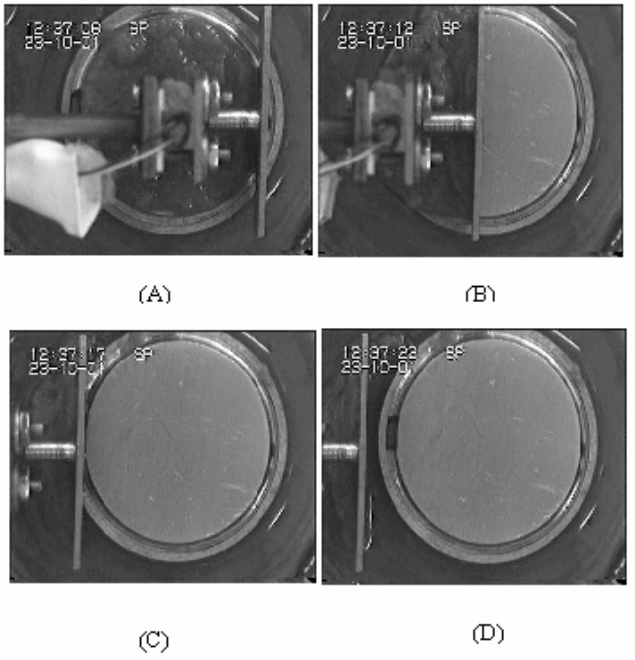
where d is the diameter of the circular disc, and t_A and t_C the first and last times at which the probe touched the fouled surface.

The apparent adhesive strength or 'pulling energy' of a fouling sample, σ (J/m^2), is defined as the work required to remove the sample per unit area from the surface to which it is attached, and is then given by

$$\sigma = \frac{W}{\alpha A} \qquad (2)$$

where A (m^2) is the disc surface area, and α is the fraction of that area covered by the sample measured by image analysis as described above. The relationship between σ and the actual adhesive strength between the surface and the deposit is not clear, as the measured force not only removes the deposit from the surface but also deforms it significantly.

(a)



(b)

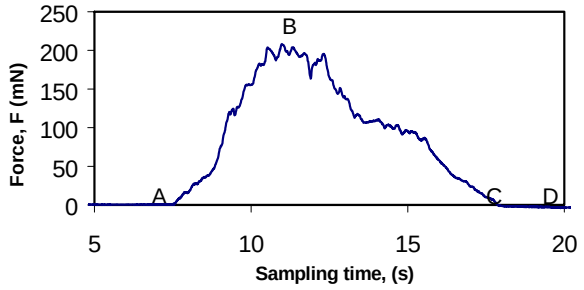


Figure 2 (a) The sequence of fouling sample pulling processes by the T-shaped probe: (A) → (B) → (C) → (D). (b) Typical curve showing force versus sampling time for pulling a fouling sample.

TOMATO STARCHES

Sample baking time

Experiments were carried out in which discs were placed in a pre-heated laboratory oven set at 100°C and baked for times between zero to 240 minutes. The apparent adhesive strength was then measured after the sample was hydrated for 30 minutes. Figure 3 shows the plot of adhesive strength versus baking time. Results are the mean of four samples: the experimental procedure is highly repeatable.

The results indicate that the apparent adhesive strength increases with longer baking time but the change becomes less significant after heating for three hours. Baking makes the sample dry and dark. The longer the baking time, the dryer and darker the sample, however there is little change noticeable in the deposit surface after baking for 200 minutes. On heating, components of the sample undergo chemical reactions (Cheow and Jackson, 1982): caramelisation of sugar, polymerisation of fat and denaturation of protein take place. Longer times generally increase the adhesive strength; the results suggest that after reactions are complete, the strength remains constant.

Hydration time

Sets of experiments were carried out in which 0.8g tomato paste was spread onto the surface and then dried for 1 hour. The dried sample was hydrated for different lengths of time and the resulting apparent adhesive strength then

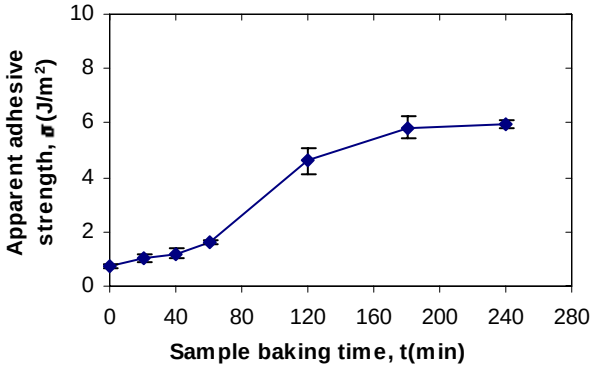


Figure 3 Apparent adhesive strength versus sample baking time. Sample mass = 0.8 g and hydration time = 30 mins. Error bars in all figures represent the standard error of the mean.

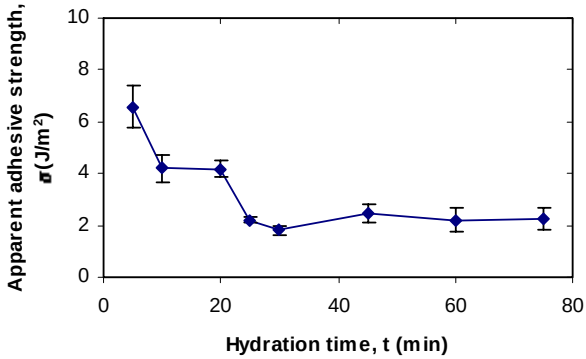


Figure 4: Apparent adhesive strength versus hydration time. Sample mass = 0.8 g and baking time = 1hr.

measured. Figure 4 plots adhesive strength versus hydration time. The error bars in all figures represent the

standard error of the mean. The apparent adhesive strength decreases with hydration time by a factor of about three, from values in the region of 7 J/m^2 down to a value of 2 J/m^2 after 30 minutes of hydration time: it then remains essentially constant.

The nature of the chemistry which takes place on hydration of tomato pastes is unclear. Hydration of starch results in swelling and disruption of granules. In addition, in the interaction between the particles and the surface, the total surface free energy will change due to the wetting of the metal surface. After hydration starts, the sample appears swollen, as a result of the starch swelling. The longer the hydration time the more water will diffuse into the solid. The attractive force between material and the surface will decrease as the effective thickness of the liquid bridge increases. After the surface is saturated, no further change can be seen, after about 30 minutes. The results suggest that removal of deposit becomes easier the longer that the samples are left to soak in water; this is a common observation domestically.

Effect of hydration temperature

A set of samples baked for an hour were immersed in a water bath attached to a temperature controlled system, and the adhesive strength after 10 minutes hydration time measured for different hydration temperatures. This time was chosen as the shortest that produced significant hydration, as shown in Figure 4. Figure 5 shows that the apparent adhesive strength decreases as the hydration temperature increases: the line through the points is a guide only, but shows that it is possible to fit an exponential decay curve to the data.

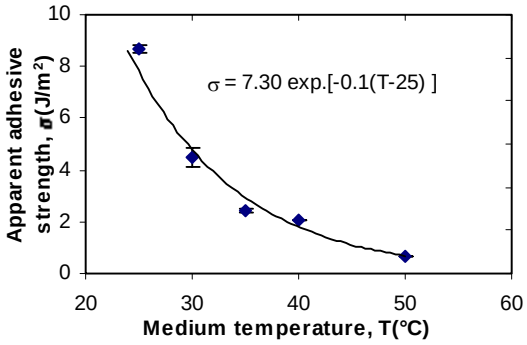


Figure 5: Apparent adhesive strength versus medium temperature. Sample mass = 0.8 g and hydration time = 10 mins. Line shows an exponential fit to the data.

Cohesive and adhesive strength of fouling deposits

The above demonstrates that the forces measured by the micromanipulation technique vary with process variables. A fully hydrated deposit has an apparent adhesive strength on the order of 2 J/m^2. As noted, however, the measured force will result both from the force required to remove the deposit from the surface, and that required to deform the deposit. It is not clear to what extent cohesive and adhesive properties control removal in this case: sets of experiments were carried out to clarify this.

Two stainless steel surfaces were made by polishing and roughening the surfaces respectively, and the roughness of each surface measured with the Talysurf 120l. R_a, the arithmetic mean of the absolute departure of the roughness profile from the mean line, was found to be

- 0.1 μm for the fine surface
- 32.5 μm for the rough surface

which compares to an R_a of 0.2 μm for the normal surface a set of experiments were then conducted in which the apparent adhesive strength was measured for removal of 0.8g of deposit, baked for one hour, as a function of hydration time. Results are shown in figure 6. it is clear that the roughness affects the adhesive strength, in that the rough surface has an apparent adhesive strength nearly twice that of the smooth surface after 10 minutes of hydration (14 as opposed to 7 J/m^2). The polished surface gives adhesion strengths very close to that of the 'normal' unpolished steel. as the hydration time increases, the magnitude of both adhesive strengths decreases, although the ratio between the two stays reasonably constant. at the highest hydration time, the values are very close to each other, at about 2 J/m^2, which appears to be the fully saturated value of the adhesive strength. Once the surface is fully hydrated, the surface does not seem to have a strong effect on adhesion. These results show that adhesion to the surface is significant; if there was no effect it would mean that cohesive forces between sections of the deposit were controlling removal.

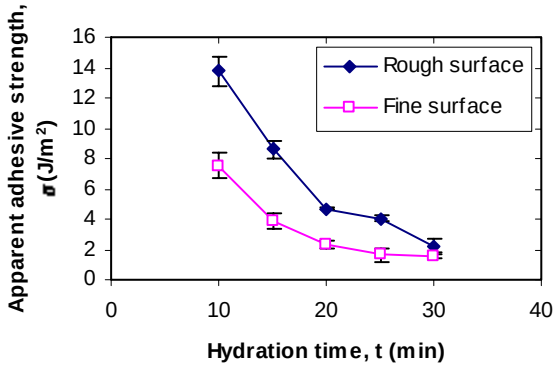


Figure 6: Apparent adhesive strength versus hydration time for two surfaces with different roughness.

Partial removal of deposit

In all of the previous experiments the whole of the deposit was removed. It is possible, by setting the micromanipulation probe to pass 50 μm above the disc, to leave deposit on the surface. In this case the force measured by the probe will corresponds to that required to break the cohesive forces between parts of the deposit. The thickness of the layer on the surface in the

experiment was 900 μm (M = 0.8g). Figure 7 compares the results for full and partial removal. In all cases, the force required for partial removal of the deposit *exceeds* that for the total removal, showing

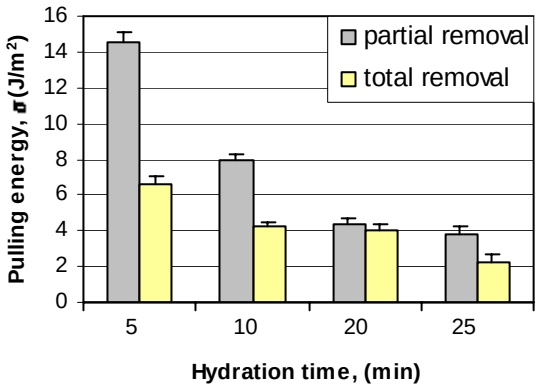


Figure 7 Pulling energy per unit area for partial removal and total removal of baked tomato paste samples at different hydration time. Sample mass = 0.8 g.

clearly that the cohesive forces between the deposit exceed those of adhesion between surface and deposit. Again, as hydration time increases the forces decrease; that for partial removal decreases more rapidly than that for total removal.

MILK PROTEIN DEPOSITS.

The force required to disrupt and remove the milk protein deposits was measured under two conditions: without cleaning chemical being used and having cleaning chemical added. A solution of a single stage cleaner (LQ32, Diversey-Lever, UK) containing 0.5 wt% NaOH was used. The effect of the cleaning chemical was observed by submerging the deposit into the 0.5 wt% NaOH solution for different lengths of time and at different temperatures before the force measurement was carried out.

Effect of cleaning chemical

The force required to remove the milk protein deposit was measured using the same procedure as described for the tomato pastes. Two typical curves showing the measured force versus the sampling time for water alone and with added cleaning chemicals are shown in Figure 8. For the measurement of the effect of chemical, the deposit was submerged into chemical solution for 5 minutes and then the removal of deposit was studied. A, B, C and D in figure 8 denote the same positions as those shown in Figure 2 (a) .

The force profile for the condition where no chemicals are added shows a shape with a number of peaks, which is significantly different from that seen with chemicals added, where one maximum force value was found corresponding to the time which the probe had reached the middle position of the circular disc. It was also found difficult to remove all the deposits from the substrate when no chemicals were used, however the whole deposit could be removed from the surface after the deposit was submerged in 0.5 wt% NaOH solution. The irregular force profile when no chemicals were used was thought to be due to irregular interactions (both cohesive and adhesive) in the protein deposit system. The addition of chemicals clearly modifies the interfacial properties between the deposit layer and the substrate

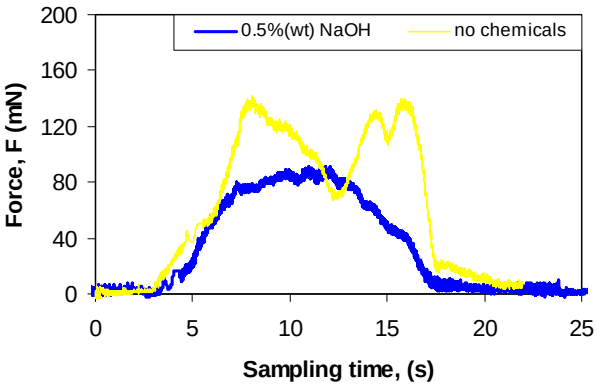


Figure 8 Force versus sampling time for pulling milk protein deposits both with and without chemicals. The deposit was submerged into 0.5% NaOH solution for 5 minutes.

Effect of the time and temperature

Figure 9 shows the evolution of the apparent adhesive force for a range of times after the sample was submerged into the cleaning solution at 20°C (293K) and 70°C (343K) respectively. In both cases, results indicate that the force was reduced after the sample was submerged in chemical solution. The force was eventually reduced to a minimum value and kept unchanged after submerging 50 minutes at 20°C and 5 minutes at 70°C. However, it can be seen that both the minimum force value and the rate at which the force reduces is different for the two different solution temperatures. At the high temperature of 70°C, the force reduces at ca. 1.2 J/m^2 min over a period of about five minutes to a minimum force of around 0.8 J/m^2. However, at the lower temperature of 20°C, the rate of reduction of the adhesive force is about 0.1 J/m^{2}min, over about 50 mins, to a higher final value of about 3 J/m^2. The diffusion of chemical agent into the deposit will be a function of temperature as the diffusion coefficient is thermally activated; any chemical reaction which reduces the deposit strength will also be thermally activated.

The result suggests that during the diffusion and swelling phase, wherein the cleaning agent has to be transported through the deposit and molecular bonds have to be broken, chemical and thermal process are dominating.

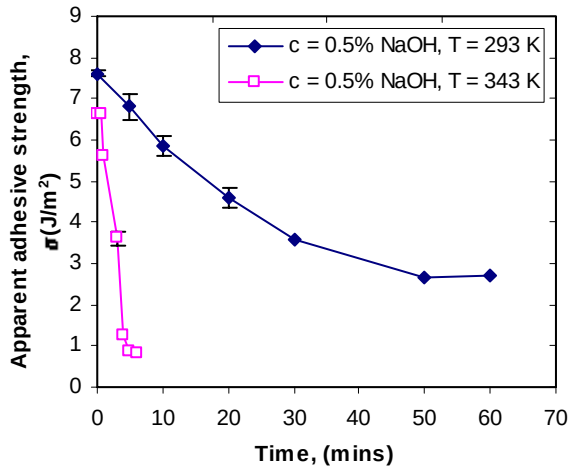


Figure 9: Apparent adhesive force as a function of the time the deposit is submerged in a 0.5% wt NaOH solution prior to measurement. Two solution temperatures, of 293K and 342K have been tested.

Effect of cohesion/adhesion

By setting the micromanipulation probe to pass over the disc and leave a complete layer of protein deposit on the surface. it is possible to study the balance between the cohesive and adhesive forces within the deposit. In this case the force measured by the probe will correspond to that required to deform the deposit above the probe, and to break the cohesive bonds between the protein deposit removed and the layer left on the surface.

The initial thickness of the deposit layer was around 1300 µm. Four force measurements were taken after leaving the gap between the probe and substrate to 900, 600, 100 and 20 µm respectively. A complete layer was left during the first two measurements which kept the gap at 900 µm and 600 µm respectively. A partial layer was left when the gap was set at 100 µm; the deposit deforms under and around the probe, making the final layer of deposit rather rough; this again is different to the removal of the tomato paste, and is perhaps connected to the nonuniformity in the deposit shown in the removal curve without cleaning chemical. The deposit layer was completely removed after the gap was set at 20 um.

The measured pulling energy as a function of gap was shown in Figure 10. The result indicates that the pulling energy measured by the probe increases with decreasing gap distance. The pulling energy obtained at the gap of 900 and 600um correspond to the apparent cohesive strength values since only deposit-deposit bonds were involved in the pulling process. The pulling energy obtained at the gap of 100 um was the joined effort of both cohesive and adhesive energy since both the deposit species and substrate were involved in the pulling process. The pulling energy obtained at the gap of 20 um corresponded only to the apparent adhesive energy since the deposits were completely removed from the substrate.

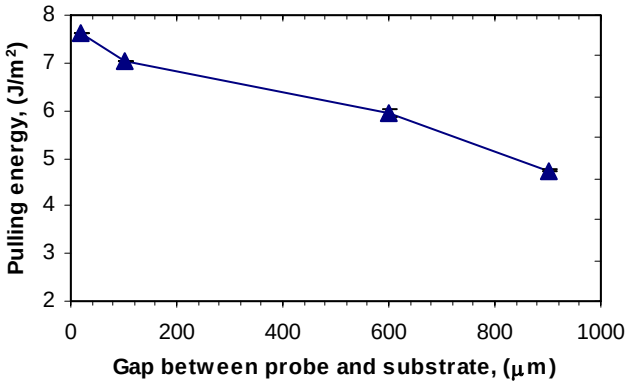


Figure 10. Pulling energy for partial removal and total removal of protein deposits. The gap between probe and substrate was kept at 900, 600, 100 and 20 µm respectively.

The results suggest that in this case the cohesive forces between elements of the deposit are weaker than those of adhesion between surface and protein deposits. This is opposite to the behavior of tomato starch shown above, in Figure 7, which shows that for tomato pastes that it is easier to remove the whole of the deposit than it is to remove a surface layer. This indicates that the micromanipulation technique can identify that different fouling species show different mechanical properties.

This type of result has strong implications for cleaning. In tomato pastes, removal of the whole deposit is possible once the low forces binding it to the surface have been broken, whilst for the milk deposit the final layer is the most adhesive. This is reflected in cleaning (Christian, 2003): on occasion, the whole of a tomato deposit is removed in one go, whilst this is less common in milk fouling.

It may be that the cohesive and adhesive forces result from the way in which the deposit has been created; for milk, the temperature gradient during deposit formation is through the metal disc, whilst for the tomato paste the heat was applied in an oven. Alternatively the different forces may reflect the chemistry of the two systems. Further work is underway to identify whether the chemistry or the deposit preparation route is the critical factor. This type of result will be invaluable, for example if novel coatings are to be used successfully.

In addition, many of the cleaning problems of the food industry are not associated with the type of flow problem which is familiar to chemical engineers. Deposition in dry areas, such as deposits of starch in bakeries, are significant.; this can pose a problem in terms of cross-contamination of products with allergens, for example. The type of measurements made here would be able to study both this type of deposit (which is normally removed by wiping and brushing) as well as flow-removed deposits. A range of modified surfaces have been proposed to prevent adhesion and/or enhance

removal; preliminary results are given here to show how micromanipulation can quantify the effects of the surface.

CONCLUSIONS

A micromanipulation technique, using a specially designed stainless steel T-shaped probe, has been developed to directly measure the apparent cohesive and adhesive strength of food fouling deposits. The method has been tested using baked samples of tomato paste and milk protein deposits. Variation in the adhesive strength as a function of processing conditions can be measured. For tomato paste, the effects of sample heating time, sample hydration time, surface roughness, and medium temperature can be identified. At room temperature, the adhesive strength of tomato paste is smaller than the cohesive strength. For milk protein deposits, the effects of cleaning chemicals, deposit submerging time and solution temperature have been determined. The adhesive strength of milk protein deposits is greater than the cohesive strength at room temperature.

The results show that the technique can be used to study a variety of food deposits from different food processing conditions. The method can help in optimizing removal of food deposits in term of food cleaning protocols. The results also suggest that the technique will be valuable in measuring surface properties, and in relating cleaning behaviour to surfaces. Work is currently underway to exploit the method. Modelling work will also be needed to relate the micromanipulation measurements to the rheological properties (including adhesive and cohesive strength) of fouling deposits, and to predict the shear stresses (pressure drops) required to remove them in fluid flows.

Further work is also required to identify the relationship between the force measured by the probe and the adhesive force between deposit and the surface. The extent of mechanical properties of the fouling materials contributed to the deformation prior to detachment of deposit layer needs to be identified.

NOMENCLATURE

A disc surface area (m^2)
d diameter of the circular disc (m)
$F(t)$ force measured by the manipulation probe (N)
t_A, t_C first and last times at which the probe touches the fouled surface (s)
W work done in deposit removal (J)

Greek symbols
α fraction of disc covered
σ apparent adhesive strength (J/m^2)

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