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REMOVAL FORCE MEASUREMENT AND RHEOLOGICAL CHARACTERIZATION OF SWEETENED CONDENSED MILK DEPOSITS: A PRELIMINARY STUDY

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ABSTRACT

Cleaning of a process plant is costly and time consuming. It is important to understand the removal of fouling deposit. In this study sweetened condensed milk (SCM) is chosen as a commonly encountered proteinaceous deposit in confectionary processing. SCM is an intermediate in the manufacture of chocolate crumb, the precursor to chocolate. During processing, adhesive deposit forms in heat exchanger surfaces which necessitate frequent cleaning. A micromanipulation technique has been used to measure directly the adhesive strength of SCM deposit on stainless steel surface. Result show that adhesion of SCM on stainless steel is higher than cohesion within the soil's bonds. Rheological characterization enables us to understand the nature of the deposit at difference temperature and dilution rates during cleaning and rinsing. SCM was characterized rheologically as Newtonian at ambient conditions. This information is essentially useful in decision making about selection of the best cleaning regime in future cleaning experiments.

INTRODUCTION

Fouling in the food industry is considered to be very severe, especially in the dairy sector where daily cleaning is a routine procedure (Visser and Jeurnik, 1997). Milk fouling is a serious problem that affects dairy processing plants during pasteurisation or ultra heat treatment (sterilisation). Milk fouling deposits consist of a layer of protein aggregate and mineral that can be several millimetres thick. Burton (1968) characterised milk deposits at different temperatures into type A and type B;

> Type A- pasteurisation (70-90 ^{o}C) deposit high in protein content, soft voluminous, spongy, curd-like material, white or cream in colour.
> Type B- ultra higher temperature (UHT) (100-140 ^{o}C) deposit of low protein content, brittle and gritty, grey in colour.

Type A deposit posses a greater problem to dairy processing than type B because of its higher thermal resistance (Burton, 1968). In general the deposit consists of two layers: protein rich outer layer and a layer near the surface of milk processing equipments; rich in calcium and phosphorous (Hege and Kessler, 1986; Britten et al., 1988). On heat exchanger surfaces, when the operation is below 100 ^{o}C, such as during the pasteurisation process, protein is the main constituent of milk fouling deposits as a result of heat-induced protein denaturation and aggregation reaction (Lalande et al. 1985; Grasshoff, 1989). Due to the high heat transfer resistance and the difficulty of removal, proteinaceous fouling is a major concern for cleaning processes.

The formation of milk fouling deposit in heat exchangers, evaporators, and membranes can cause the decline of the efficiency and performance overtime, as well as possible microbial growth problems, preventing a long operation of the equipment. Cleaning such equipment is mandatory, both to maintain high process efficiency and to produce a high quality product consistently. Most food processing equipment needs to be cleaned regularly to maintain thermal efficiency. Historically, cleaning was done manually whereby plant equipment were dismantled and cleaned individually. Today, cleaning is done 'in place', without dismantling or opening of the plant, using sophisticated techniques for controlling and monitoring the whole process (Romney, 1990). The most common approach to a cleaning process in large-scale operation is to employ cleaning-in-place (CIP), a procedure which involves the circulation of cleaning and rinsing solutions without dismantling of the pipe work or any other equipment. The cleaning solutions are commonly used either as single-stage or two-stage processes depending on the processing practices and load of soiling on the process equipment (Timperley and Smeulders, 1987). Due to its high frequency and hygienic requirement, the cost of cleaning is very high, depending on the chemicals, energy, water, product losses, labour and downtime. It was estimated for example that in the dairy industry, the yearly total fouling costs in the Netherlands are approximated to exceed 40 million US dollars (Visser and Jeurnink, 1997) and up to 42 % of available production time may consist of cleaning process (Pritchard et al., 1988).

Cleaning is a multistage process comprising steps that may be controlled by mechanical action, chemical reaction, and mass transfer. Furthermore, the cleaning process is also influenced by other factors such as the nature of the soil and the characteristics of the substrate surface. Bird and Fryer (1991) reported that three layers interact in the removal of proteinaceous deposits:

(i) a solid layer: the surface of the process equipment

(ii) an adherent deposit layer: a network of protein and up to 80% water.

(iii) a liquid: the detergent of cleaning solution.

A typical cleaning rate curve for the removal of proteinaceous fouling with alkaline-based solutions includes three stages, namely, a swelling stage, a unifom stage, and a decay stage (Gillham et al., 1997). During swelling stage, there is an induction (delay) time before any deposit is removed after the contacting of the cleaning solution with the deposit. An on-line turbidity measurement of the cleaning kinetics has shown that the initial cleaning rate was zero for milk deposit (Gallot-Lavallee and Lalande, 1985). In the study of removal of whey protein deposits, a delay time was also observed (Bird and Fryer, 1991). The deposit swells after contacting with the alkaline solution to form an open protein matrix with high void fraction. The cleaning rate gradually increases until a relatively constant cleaning rate is reached, where the uniform stage starts. A large portion of the deposit is removed during this uniform stage. The final decay stage occurs when the swollen layer is thin and no longer of a continuous film.

In cleaning process, cohesion forces and adhesion forces are two important and useful properties. Adhesion is the attraction between the surfaces of two different materials. The term is sometimes used to denote the tendency of two adjacent surfaces, with different chemical compositions, to cling to each other. Cohesion refers to the attraction between portions of the same material. The nature of the fouled layer and the surface determine the strength of surface-soil bonds. In the same way, the nature and conditions of the fouled layer material determines the soil-soil cohesion forces. It was proposed that the proteins were responsible for the cohesion of deposits (Tissier and Lalande, 1986).

Corrieu (1981) reported that the adhesive forces of a proteinaceous deposit and stainless steel surface in food processing equipment are considerably larger than the cohesive forces within the deposit, which implies that the breakage of cohesive bond between deposits is more likely than breakage of adhesive bond between deposit and wall. From the thermodynamic point of view, Plett (1985) suggested that during the cleaning procedure, certain chemical and physical bonds have to be overcome. Chemical properties include the pH of the solution, detergent type and concentration, while temperature and flow rate are considered physical properties.

Fouling deposits form as a result of adhesion of species to the surface and cohesion between elements of the materials. The main important factors 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). The force required to remove deposits from heat transfer surfaces is usually measured indirectly as the shear stress inferred from pressure drop data or from empirical correlation (Fryer and Slater, 1987). Some analytical devices, such as the radial flow cell (Klavenes et al., 2002) and atomic force microscopy (AFM) (Parbhu et al., 2002), have been used to investigate the adhesion forces and characterise fouled surfaces.

A micromanipulation equipment has been developed to study adhesion (Chen et al., 1998) as part of other studies on the physical properties of biological systems (Thomas & Zhang, 2000). Liu et al., (2002) further developed this technique to measure directly the force required to disrupt a food deposit (tomato paste) including whey protein (Liu et al., 2006) and egg albumin (Liu et al., 2007) and remove it from a stainless-steel surface. This technique was employed to measure the adhesive strength of cooked SCM onto stainless steel.

Rheology is defined as the science of deformation and flow of matter (Steffe, 1996). The deformation properties relate to solids and flow properties are important to fluids. In food, rheology is important especially in terms of product development. Rheological data are used to gain the information for equipment design, processing methodology, final product quality and product shelf life (Steffe, 1996, Windhab, 1995).

Dairy rheology affects the texture and stability of dairy product (Holcomb, 1991). The rheological properties of dairy product are complex, depending on various factors like temperature, total solids, ingredients and state of dispersed phase (van Vliet and Walstra, 1980). Kristensen et. al. (1997) studied the influence of temperature on the rheological properties of certain dairy products and concluded that the rheology of dairy foods was influenced by temperature to varying degree. Generally, milk and cream exhibit Newtonian behaviour when the product temperatures are above 40^{o}C and the fat content is below 40% (Kristensen et. al., 1997). When any one of these factors change, the product deviates from Newtonian behaviour.

This work aims to get a better understanding of a cleaning process that will be carried out in future work by identifying the adhesive/cohesive force needed to disrupt SCM deposits from stainless steel substrate using micromanipulation. The rheological properties of SCM at different temperature were also studied.

EXPERIMENTAL

Materials

Sample of uncooked sweetened condensed milk (SCM) was obtained from Malbrook Cadbury's factory and stored in a refrigerator for preservation. SCM is an intermediate in the manufacture of chocolate crumb, the precursor to chocolate. During processing, adhesive deposit forms in heat exchanger surfaces which necessitate frequent cleaning. To simulate the processing of the real industrial fouling, one need to prepare a large amount of SCM and such equipment is not available in the lab scale to form the SCM deposit. The nearest is to cook the SCM in stainless steel tube and immersed in water bath to resemb the condition of the pasteurizer. All experiments used the same batch of SCM.

Preparation of SCM fouling deposits

One millilitre uncooked SCM was poured into a stainless steel tube as described in Liu et al., (2007) with 29 mm in diameter, 50 mm length and 1 mm in thickness. The ends of the tube were covered with metal discs and sealed with silicone sealant. Half of the tube was immersed vertically into the water bath at preset temperature of 85 ^{o}C (resembling the pasteurise temperature in plate heat exchanger). Then it was cooked for several sets of time, namely (0 min; control, 120 min, 180 min, 240 min, 360 min, 480 min). After cooling, the tube was opened by removing the top disc. The fouled bottom disc with 26 mm in diameter was carefully removed from the tube prior to the micromanipulation measurement. Discs were weighed before and after the fouling experiment, respectively, to obtain the sample mass before and after cooking.

Micromanipulation measurement

Details of the micromanipulation equipment are given in Liu et al., (2002). In this study, the technique was used to measure the adhesive strength of food fouling using a T-shape probe made of stainless steel chip with dimension of 30x6x1 mm. The T-shape probe was connected to the out put aperture of a transducer (model BG-100, Kulite Semiconductor, Leonia, NJ, USA), which was itself mounted on a three dimensional micromanipulator (Micro Instrument, Oxon UK)

Prior to measurement, the dish containing the fouling 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 100 μm by fine tuning with digital level indicator (model ID-C112mb. Mitutyo, Corp, Japan). The length of the probe was made larger than the diameter of the circular surface of the test disc to minimise edge effects. The probe pulls the deposits horizontally at a constant speed of 1.0 mm/s. The force exerted on the probe was recorded at 100 Hz by a PC 3D data acquisition board (Amplicon Liveline, Brighton, UK) The total work, W (J) done by the applied force, F (t), to remove the deposit may be calculated as the integral of

$$dW = Fdx \tag{1}$$

where, the distance dx is vdt, so that

$$W = \frac{d}{(t_C - t_A)} \int_{t_A}^{t_C} F dt \tag{2}$$

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

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

$$\sigma = \frac{W}{\alpha A} \tag{3}$$

where A (m^2) is the disc surface area, and α is the fraction of that area covered by the sample.

Rheological characterization

Rheological measurements were performed on a controlled stress AR-1000 rheometer (TA Instruments, UK), where the shear stress was set and resultant shear rate was measured. The SCM samples were subjected to different sets of temperatures from 25 ^{o}C to 85 ^{o}C with controlled shear stress of 10.0 Pa. A smooth cone and plate geometry with a cone diameter of 40mm and angle of 4^{o} with gap of 500 μm was employed for all experiments. The samples were covered with moisture trap which contained water to prevent moisture loss. At least two replicates of each measurement were made for test to assess reproducibility.

Experiments were carried out within the steady state shear flow mode and under dynamic oscillatory mode. The flow test was carried out to determine the viscosity of the samples. Within the flow test, assessment under steady state was carried out and the viscosity against shear rate was recorded. The changes in apparent viscosity vs shear rate was also tested for different percentage of SCM at 30 ^{o}C. A series of percentage of SCM composition were prepared by diluting SCM with distilled water. The SCM compositions were 100% (control), 80%, 60%, 50%, 20%, 10% and 5% respectively. For this experiment, a standard-size double concentric cylinder (rotor outer radius 21.96 mm, rotor inner radius 20.38 mm, stator outer radius 20.00 mm, cylinder immersed height 27.50 mm, gap 500 μm) was used.

For the oscillatory test, the samples were first tested over a range of stresses to determine appropriate conditions for non-destructive testing. Stress sweep experiments were performed at oscillatory stress of 0.1 to 100 Pa and a constant frequency of 0.1 Hz to determine the linear viscoelasticity region (LVR). Frequency sweep test carried out at an oscillatory stress of 0.5Pa were performed from 0.1 to 100 Hz. The oscillatory parameters used to compare the elastic properties of SCM; storage modulus (G'), loss modulus (G'') and phase shift angle δ. Dynamic measurements were performed within linear viscoelastic region (LVR).

RESULT AND DISCUSSION

Micromanipulation

Adhesive/cohesive strength

The force required to remove the deposit was measured by drawing the micromanipulation arm across the surface of the deposit (described in detail in Liu et al., 2002). Experiments were carried out to study the effect of cooking time on apparent adhesion strength of SCM deposits. The length of cooking time was chosen to resemble the possibility of SCM deposit embedded on PHE surfaces before its scheduled cleaning. Figure 1 shows the apparent adhesive strength vs cooking time. The results indicate that the apparent adhesive strength increases with longer cooking

time. However change becomes less significant after cooking for 4 hours. It was observed that the colour of the SCM becomes darker (from creamy to brown colour) with longer cooking time. Generally, the apparent adhesive strength increase with longer cooking time. During heating, the food sample undergoes chemical reactions. When reactions are complete, the adhesive strength remains constant (Liu et al., 2002).

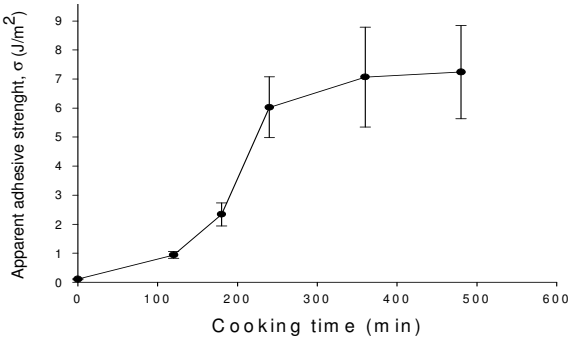

Fig. 1. Adhesion strength vs sample cooking time. Error bars represent the standard error of mean.

In the above experiments (Figure 1) the whole of the deposit was removed from the surface. According to Liu et al., (2002, 2006) it is possible to measure the forces during pulling the T-probe across the deposit by manipulating the gap between probe and substrate. Then it is possible to study the balance between the cohesive and adhesive forces within the deposit. The term 'pulling energy' was used by the authors to differentiate the measured force from that where the deposit is removed. Two types of measurement are possible, namely total removal and partial removal. Total removal measure both adhesive and cohesive forces while partial removal only measure cohesive force. For total removal the gap between the probe and the substrate was set 100 µm and 700 µm for partial removal. The initial thickness of SCM deposit was approximately 1500 µm. Figure 2 compares the results for total removal and partial removal and it was found that the force required to remove the SCM deposit totally from the substrate is more than the cohesive force (partial removal) at different cooking time. The result proposes that for SCM, the adhesive forces between substrate and protein deposit are stronger than forces between elements of the deposit. Figure 3 shows the pulling energy as a function of gap between the probe and surface. Three gap measurements were set at 100, 500, 700 and 1000 µm and the result shows that for SCM deposits the greater the gap between probe and substrate the pulling energy decreases. These observations are in agreement with whey protein deposit but not to the tomato deposit (Liu et al., 2002, 2006). This result indicates that different materials of deposit will behave differently during cleaning processes. This in turn will have an implication on effective cleaning of various deposits.

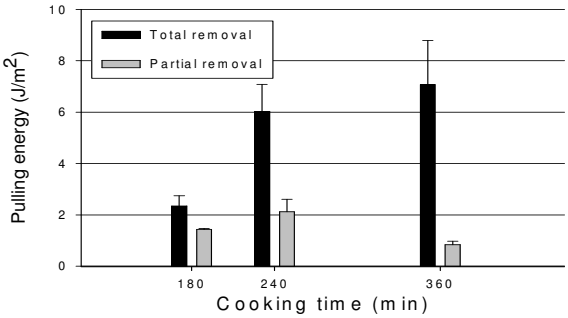

Fig. 2. Pulling energy for partial and total removal of fouling sample versus cooking time

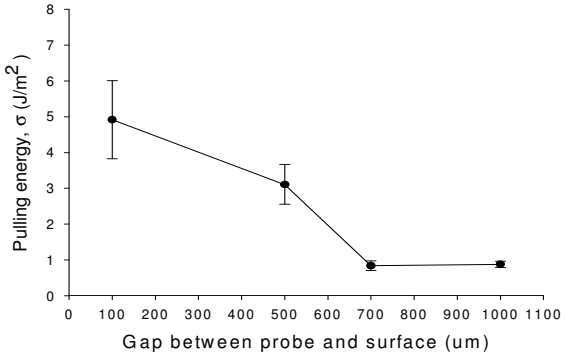

Fig. 3. Pulling energy as a function of gap between probe and surface.

Rheology of SCM

Flow Characteristics

Figure 4 shows the flow curves of SCM with varying temperature from 25 to 85 °C and the curves of shear rate vs shear stress is shown. All the samples exhibited Newtonian behaviour. Figure 5 shows changes in viscosity as a function of shear stress at 30 °C. Viscosity reduces with decreasing of SCM percentage. Viscosity data at 5 and 10% SCM gives unusual results and this is may be due to the sensitivity of the rheometer as the viscosity of the solution is very thin, resembling water, so it can be concluded that no time dependency is being observed.

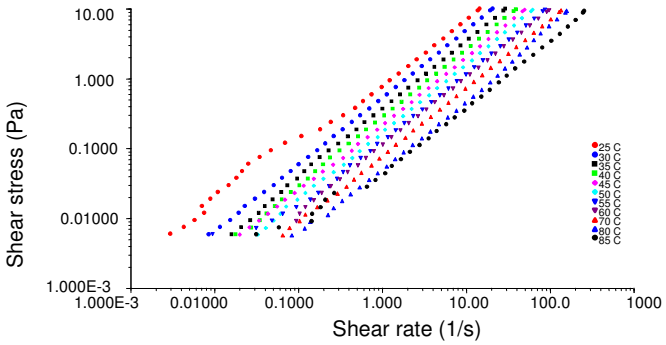

Fig. 4. Changes in apparent viscosity as a function of shear stress for SCM at different temperatures from 25 °C to 85 °C

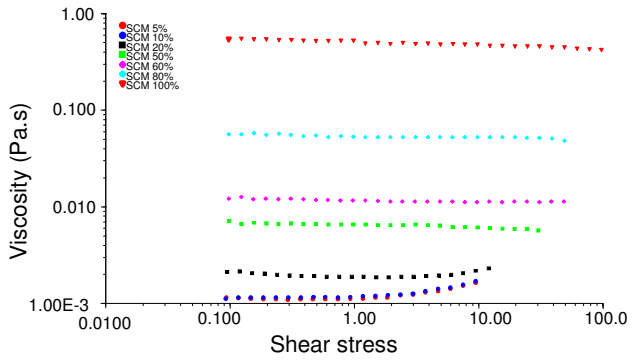

Fig. 5. Changes in apparent viscosity as a function of shear stress at different percentage of SCM at 30 °C

Dynamic measurements

Dynamic measurement was conducted to obtain a better description of rheological properties of SCM under studied. To ensure storage modulus (G') and loss modulus (G") are reliable and accurate a stress sweep test was first conducted. Stress sweep test were performed at the 0.1 Hz at an oscillatory stress of 0.01 to 100 Pa. The linear region, where the dynamic parameters (G', G'' and phase shift angle δ) are independent of the magnitude of applied stress, was investigated and appropriate measuring parameters was selected. The use of phase shift angle or delta degree (δ) in viscoelastic system is based on the measurements of G' and G" modulus' Thus in purely viscous system (i.e.water) δ is 90° , and subsequently G'= 0 and G" = G*, where G* is complex modulus. Consequently, if the system is purely elastic δ is 0° , and subsequently G'= G* and G" = 0. Figure 6, 7 and 8 shows the frequency dependence of G'and G'' modulus and phase angle (δ) for SCM at 20 °C and 30 °C respectively. Both temperatures studied exhibited a fluid-like behaviour at low frequencies where loss modulus (G'') was higher than storage modulus (G') and the delta degree (δ) increased, suggesting that fluid-like viscous behaviour dominates solid like elastis behaviour. According to Rao (1999), at low frequencies, when G'' is much higher than G', the energy used to deform the material is dissipated viscously and the material's behaviour is liquid-like.

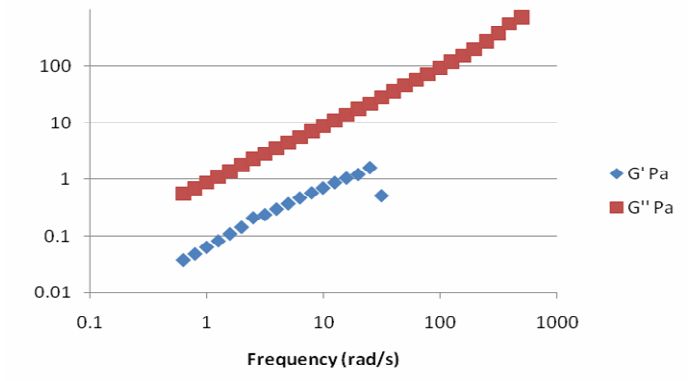

Fig. 6. Frequency dependence of G' and G'' modulus for SCM at 20°C

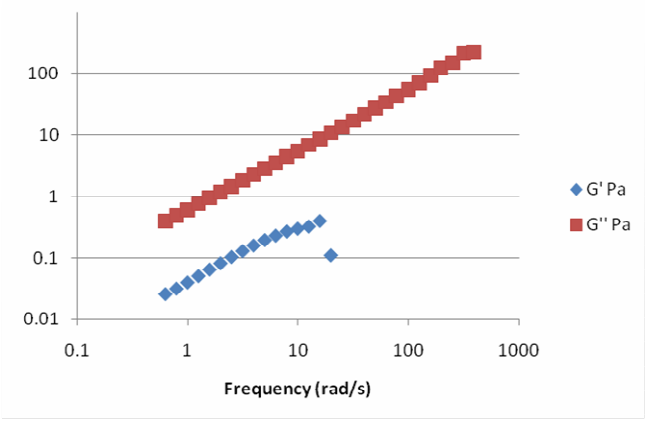

Fig. 7. Frequency dependence of G' and G'' modulus for SCM at 30°C

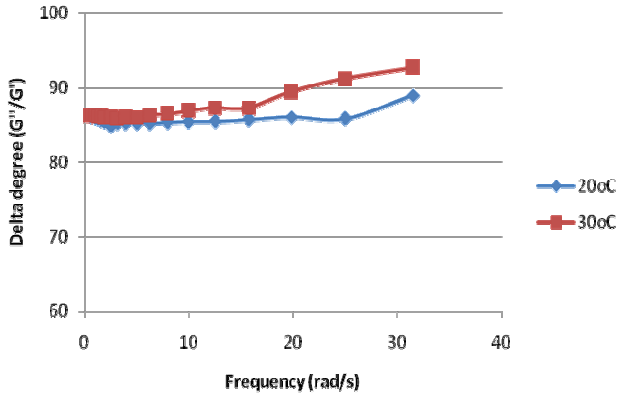

Fig. 8. Frequency dependence of the delta degree δ of SCM at 20°C and 30°C.

CONCLUSION

Micromanipulation equipment makes it possible to study the forces required to separate adhesive and cohesive effect in the cleaning of protein fouling particularly sweetened condensed milk deposit. It was found that the SCM deposit is adhesive in nature and strongly adheres to the surface of stainless steel disc. SCM behaviour is more liquid-like and viscous. Rheology characterization enables us to understand the nature of the deposit at different temperature and dilution rate during cleaning and rinsing. With adhesion and dilution properties, this suggests that the deposits will be removed from top layer down with fluid flow during cleaning. Future studies include an investigation of the cleaning behaviour using a bench type cleaning rig where variables such as flow rate, temperature and chemical concentration are study in obtaining cleaning optimisation.

NOMENCLATURE

A disc surface area (m^2)
d diameter or the circular disc (m)
$F(t)$ force measured by the manipulation probe (N)
t_A, t_C first and the last time at which the probe touches the fouled surface(s)

W work done in deposit removal (J)
G' storage modulus
G'' loss modulus

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

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