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ULTRASONIC ADHESION MEASUREMENT OF WHEY PROTEIN FOULING

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ABSTRACT

The knowledge of milk deposit adhesion is a prerequisite for setting innovative operating conditions of processing and strategies of cleaning. Unfortunately, these data are lacking because few methods of characterization of the adhesion quality exist.

In this work, an ultrasonic method toward the quantification of the adhesion of a fouling dairy deposit onto stainless steel surface is proposed. The method is based on the investigation of the shear wave's reflection at the interface between deposit and substrates.

Firstly, the associated theory for quantifying adhesion by a reflection coefficient is introduced and the ultrasonic method proposed is briefly presented.
Then, experiments led with glucose syrups of various viscosities and different substrates (Glass and Stainless steel) for validating the adhesion parameter values are performed and results are compared to classical tests for measuring adhesion strength.

Finally, some preliminary trials for measuring the adhesion of a whey protein fouling onto coated stainless steel were carried out and discussed.

INTRODUCTION

Although cleaning processes are commonplace in the food industry, they are rarely optimised (Fryer et al., 2006). It is for example very difficult to validate the cleaning end-point, in spite of numerous methods of fouling detection level in heat exchangers in the food industry (Wallhausser et al., 2012). There is currently no way to allow the cleaning time of one deposit to be reliability predicted from that of one another. Designing a cleaning protocol for a given situation is still however semi-empirical (Pogiatzis et al., 2012). Cleaning procedures are also difficult to optimise, because of the lack of knowledge for a given soil whether an adhesive or cohesive failure mechanisms are the limiting steps governing the removal of deposit (Liu et al., 2006). Indeed, cleaning happens differently when the critical steps are: the adhesion of species to the surface or cohesion between elements of the material. A systematic quantification of the interaction between deposits and surface and how they are affected by process variables (temperature, composition of the fouling material, interfacial energy and roughness of the surfaces) is highly critical (Rosmaninho et al., 2007; Jimenez et al., 2012). The forces required to disrupt or remove fouling deposits are rarely well understood, both because they are difficult to quantify and also as they are determined by the removal mode, the age and structure of the deposit, and the nature of the deposit-surface interactions (Hooper, 2006).

At present, quantifying the adhesion between a deposit and a solid surface by a non-invasive way is still nowadays a challenge. Few tools are available in this field. A micro-manipulation technique has been developed at Birmingham to quantify the forces involved in deposit removal at the micron/mm scale (Liu et al.,2002, 2006a, 2006b; Akhtar et al., 2010). Data allow deducing the work required to remove deposit per unit area. The same type of measurement can be made using dynamic gauging where deposit is sucked from the surface by the fluid action (Chew et al., 2004; Hooper, 2006; Saikhwan et al., 2007; Gu et al, 2011). Recently, atomic force microscopy was also used to measure deposit-surface interactions (Akhtar et al., 2010). Possibilities to implement these techniques in line are unfortunately limited.

In this paper, a non-invasive Ultrasonic (US) technique to measure the adhesion force between a soil and a solid surface is introduced. Preliminary work on the development of the technique and experimental method are reported here. The ultrasonic test consists in i) propagating ultrasonic shear waves up to the interface when deposit is stick to the substrate ii) measuring the amplitude magnitude of the reflected wave compared to the incident amplitude wave in the absence of deposit. The reflection coefficient (ratio between the amplitude of the reflected and the incident waves) is supposed to be correlated to the adhesion force.

To aid in the development phase and in establishing the existing correlation between the reflection coefficient and the adhesion force, glucose syrups of varying viscosities spread onto two solid surfaces of interest (glass and stainless steel) have been first used as soil. Pulling-force required for removing the aqueous sugar solutions (obtained with a classical traction machine) and corresponding values of reflection coefficient (measured before detachment) are

reported. Then further works discuss adhesion measurements performed with the ultrasonic device when removing whey protein deposits on various coating stainless steel surface.

MATERIALS AND METHODS

Acoustic method principle

The ultrasonic test consists in measuring the amplitude variation of the incident wave (transmitted through the substrate) after its reflection at the interface between the substrate and the fouling deposit. Ultrasonic waves used in this study are shear-waves, because this type of wave is known to be sensitive to interface defects (Ouaftouh et al., 1992). The disturbance induced by such shear waves is an elastic deformation perpendicular to the direction of motion wave (see Fig. 1).

Consider three magnitudes of adhesion between a layer of substrate (referenced A and appearing in yellow in Fig. 1) and a layer of fouling deposit (referenced B and appearing in green in Fig. 1). The reflection coefficient at the interface between A and B is described by the relation:

$$r_{A/B} = \frac{Z_B - Z_A}{Z_B + Z_A} \quad (1)$$

with the acoustic impedance:

$$Z_i = \rho_i \cdot c_i^T \quad (2)$$

Z_i, ρ_i and c_i^T are respectively the acoustic impedance, the density and the velocity of shear waves of layer A and layer B (i=A or B).

The impact of the incident shear wave for the three adhesion cases will be the following:

Strong adhesion: At the interface between A and B, particles of layer A are sharply linked to particles of layer B. So stress and strain of both solid (substrate) and fluid (deposit) particles at the interface are the same. The reflection coefficient is $r_{A/B}$.

No adhesion: both layers are not bonded; particles of layer A are not linked to particles of layer B. Fluid particles are not entrained by the movement of the substrate particles. Waves are completely reflected close to the interface. Consequently the reflection coefficient is close to 1.

Intermediate adhesion: the bonding between both layers is low. The fluid particles are partially entrained by the movement of the solid. The reflection coefficient is between $r_{A/B}$ and 1.

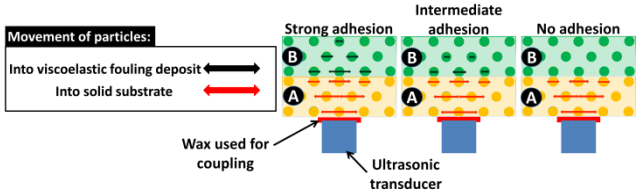

Fig. 1: Sketch describing the amplitudes of the particles movement induced by the propagation of a shear waves for different adhesion cases.

Coupling the ultrasonic transducer with bottom side of the substrate is very critical for the repeatability of the adhesion measurement. Indeed, shear waves propagation are very sensitive to coupling conditions (physical properties of material and thickness). In this study, dental wax (Modelling Wax Cire Pinnacle, DENTSPLY) was used as coupling material between substrate and transducer.

To accurately control the wax thickness, the US transducer was previously fixed to a pneumatic system (AR1000, TA Instrument) allowing the raising or lowering of the transducer to the desired distance from the underside of the substrate (Fig. 2). This automatic adjustment mechanism provides an accuracy of one micrometer.

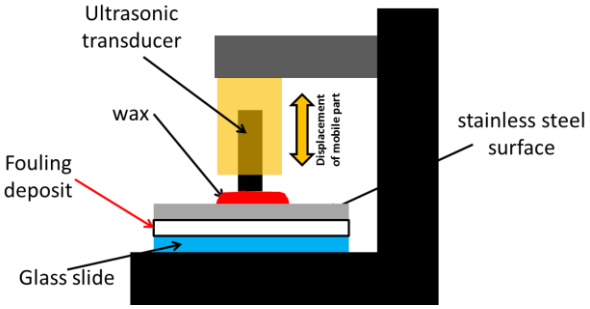

Fig. 2: Coupling device of the ultrasonic transducer with the sample.

The coupling process involves achieving a zero gap between the transducer and the substrate. The substrate on which is placed the adhesive deposit is previously returned and placed onto a glass slide. After zero-gap step is finished, the transducer is lifted and a square wax (with dimension of 1 square centimetre) is deposed onto the solid surface plate. The solid surface plate is heated to 80°C, thanks to a hydraulic heat system, so that the wax melts. The ultrasonic transducer is then brought close to the plate, at 5µm far from the solid surface plate. The solid surface plate is then cooled to 11°C.

This coupling technique requires that the deposit is sandwiched between a stainless steel surface and a glass slide. For coupling the ultrasonic probe with the substrate, liquid wax is deposited on the opposite surface of the coupon with the fouling deposit. The wax could then infiltrate the fouling deposit. To avoid damage of the fouling deposit, the solution was to place the coupon (with the deposit side) on a glass slide with a higher dimension than the coupon. An adhesive tape placed on the edges of the stainless steel coupon protects the fouling deposit against infiltration of wax. The stress applied on the deposit is very low and the area of interest is only the interface between the coupon and the fouling deposit. In addition, the strong heterogeneity of the deposit prevents the propagation of the wave used through it.

Ultrasonic device and data acquisition chain.

Shear waves are generated by a transducer electrically linked to the network analyser (Network analyzer 8753E, Hewlett-Packard). The transducer is made in $LiNbO_3$ (Lithium niobate) with a silica delay line, it generates shear

waves with a resonance frequency of 20MHz. Ultrasonic measurements are performed by this last. This measurement uses the S_{11} parameter as function of frequency for a range between 10MHz to 30MHz. The S_{11} scattering parameter is the electric reflection coefficient (ratio between the reflected and the incident electrical wave). The impulse response of the system $S_{11}(t)$ is obtained by computing (Matlab®, Mathworks) the inverse Fourier transform of the $S_{11}(f)$ parameter (Deblock et al., 2005). The measurement chain is shown in Fig. 3.

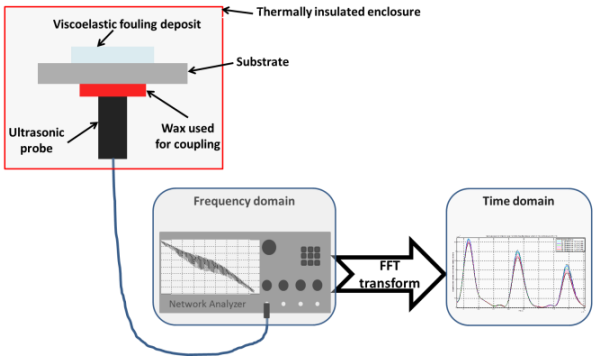

Fig. 3: Sketch of ultrasonic device and data acquisition chain

Substrates and adhesion tests.

Different substrates were used in this work: i) glass slides ii) bare and coated stainless steel surfaces. Glass substrates are slides of 76mm x 26mm with a thickness of 1.1mm. Stainless steel and coated substrates are plates with the following dimensions: 45mm x 15mm with a thickness of 1mm.

Adhesion of glucose syrups. Firstly, glass slides and bare stainless steel substrates were used to measure the reflection coefficient when various glucose syrups were deposed onto these surfaces.

Dilute glucose syrup solutions were obtained by adding deionized water (Millipore, Bedford, MA) to pure glucose syrup. Four solutions of glucose syrups with varying level of viscosity (Fluid 1 to 4) were thus produced.

The viscosity of glucose syrup solutions has been measured by a rheometer (AR2000ex, TA Instrument) at various temperatures. The shear rate range varies from 0.1 to 500s^{-1}. The temperature range investigated in rheometer ranges between 19°C and 22°C. This temperature range corresponds to temperatures encountered during ultrasonic and pulling-force tests. Newtonian behaviors were observed for all glucose solutions.

Viscosity dependence of glucose syrup with temperature measured experimentally is shown in Fig. 4. Power law have been used to take into account this dependence (solid line in Fig. 4):

$$\mu = a\theta^b \qquad (3)$$

Parameters a and b of power law are reported in Table 1.

Table 1 shows also viscosities of the various solutions of glucose syrups at 20.5°C (average temperature at which pulling test were determined). Analysis of Table 1 reveals that: viscosity of fluid 4 is 46 higher than fluid 1, which is 4100 higher than water viscosity.

Pulling tests were also carried out to establish the link between the adhesion indicator delivered by ultrasonic device and the force of detachment measured by conventional fracture test.

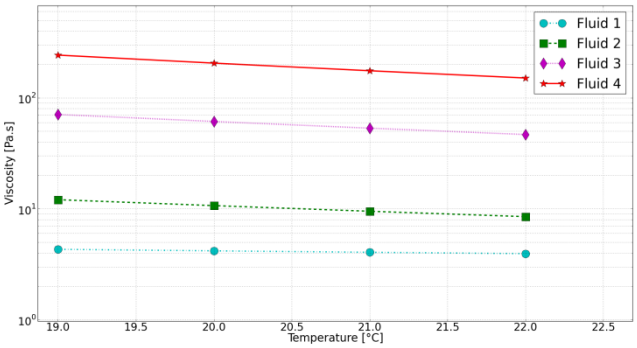

Fig. 4: Viscosities of dilute aqueous solutions of glucose syrup as a function of the temperature.

Table 1: Rheological properties of the glucose syrup and parameters of models used to account for their thermal dependence.

Solution of glucose syrup	Parameters of the power law at temperature between 19°C and 22°C		Viscosity at 20.5°C
	a	b	
Fluid 1	26	-0.61	4.1 Pa.s
Fluid 2	14166	-2.4	10.1 Pa.s
Fluid 3	303127	-2.83	58.8 Pa.s
Fluid 4	3371573.8	-3.24	189.6 Pa.s

The equipment used for pulling force measurement is a compression and traction machine (DY 30, Adamel Lhomargy). This apparatus was adapted to allow the gripping of a polydimethylsiloxane (PDMS) cylinder by its mobile part. Substrate is maintained at the machine base by a mechanical system.

The technique of measurement involves two steps. First, a drop of glucose syrup on the substrate (in glass or stainless steel) was deposited and the mobile part is got off until the PDMS cylinder is immersed in the fluid and in contact with the substrate. Second, a constant pulling force is applied to the cylindrical sample until the detachment of cylindrical sample from substrate. The tensile force required to detach the glucose syrup from its substrate was reported in Table 3 (Data Acquisition/switch Unit 34970A, Agilent).

Note that preliminary measurements (not shown here) have been performed to determine the appropriate glucose syrup thickness. It was determined that glucose syrup can be considered as semi-infinite (from an acoustic point of view) for more than 50µm thick. This means that the thickness has no influence on the reflection coefficient. Consequently, we have introduced a drop of glucose syrup on the ultrasonic probe ensuring that the thickness is higher than 50µm.

Adhesion of whey protein fouling. Secondly, bare stainless steel plates were coated to obtain surfaces of various water contact angles. These surfaces were manufactured to observe whether the adhesion of milk deposit measured by US device is sensitive to water contact angle values. To obtain coated surfaces, deposition of precursors was carried out using an atmospheric pressure cold plasma torch (ULS, Acxys technologies). Different precursors (fluorosiloxanes, siloxanes and silanes) and experimental conditions were tested and allowed us to obtain various water contact angles reported in Table 2. More details concerning the antifouling stainless steel fouling surface processes are given elsewhere (Jimenez et al., 2012). A goniometer based on the sessile drop method (DiGidrop Contact Angle Meter, GBX Scientific instruments) was used to measure water contact angles.

Table 2: Stainless steel contact angle and roughness values obtained for different cold plasma coatings.

PECVD treatment	Contact angle (°)	Roughness Ra (nm)
Sample 1	60.7 ±1.23	85 ±28.49
Sample 2	130.0 ±3.38	73 ±13.45
Sample 3	78.7 ±1.61	62 ±7.58
Sample 4	87.9 ±3.14	57 ±30.96
Sample 5	59.5 ±0.69	73 ±6.08
Sample 6	89.4 ±1.97	51 ±8.99
Sample 7	93.0 ±1.28	141 ±119.9
Sample 8	133.4 ±1.52	76 ±7.01

To verify that the surface treatments of stainless steel have not led to excessive changes of surface topography, roughness measurements were performed.

Atomic Force Microscopy (Nanoscope IV, Dimension 3100, Veeco, Tapping mode) was used to analyze the surface morphology and roughness of the prepared coated stainless steel samples. Well known Ra Roughness parameters obtained for the various coated surfaces are reported in Table 2.

A large majority of surfaces have a roughness between 51.3±8.99nm<Ra<88.98±21.86nm. Only the Sample 7 (fluorosiloxane) treatment lead to high and heterogeneous roughness compared to other samples.

Fouling deposit preparation

Fouling deposits were prepared using a pilot-plant installation containing: Pre-heating, heating and holding zones (Fig. 5). A 1% (w/v) of an isolate protein solution (PROMILK 852 FB1 from IDI SAS -62033 Arras -France) was used for this purpose. Final calcium content of the protein solution was adjusted to 96 mg/L. First, the fouling solution was pre-heated from 10 to 60°C, and then heated in the pasteuriser from 60°C to 93°C. Process parameters of heating zone were kept constant for each fouling run (Flow rate of protein solution and hot water, Inlet and Outlet temperatures of protein solution, plate heat exchanger configuration).

The flow rate of whey protein solution was 350L/h. The ratio of flow rates of hot water and whey protein solution was also kept constant and equal to 2. The pasteuriser (holding zone) consists in five passes of one channel for the two sides used in a counter-current configuration (Model V7 of Alfa-Laval Vicarb, France).

The length between the two frames was fixed at 47mm which means that the equivalent space between two plates was equal to 3.93mm. During fouling experiments, the inlet hot water temperature was adjusted to ensure a constant outlet product temperature close to 92°C. The holding zone is a square pipe in which stainless steel plates are inserted.

After a fouling run of two hours, plates were carefully remove from the channel and quickly placed in Petri dishes at room temperature before performing the adhesion tests (Fig. 6). The thickness of fouling deposits was evaluated at 150µm by profilometer (MarSurf XR 20, Mahr).

Fouling deposit samples are then coupled to the ultrasonic transducer as described in the "Acoustic method principle" part. Ultrasonic measurements are performed in a room with controlled temperature.

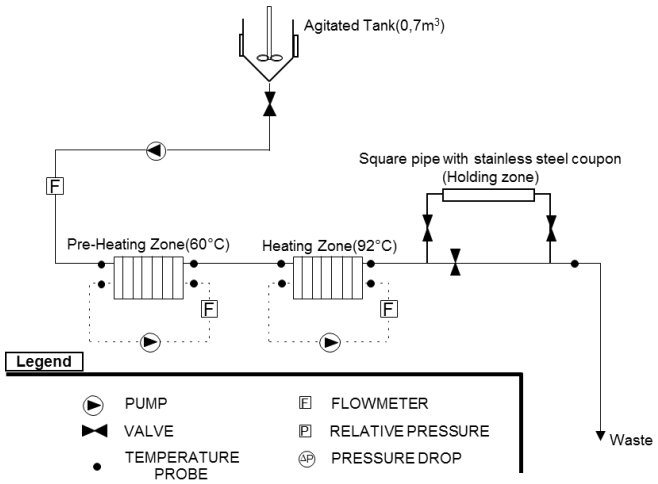

Fig. 5: Sketch of pilot-plant installation used to obtain whey protein fouling deposits.

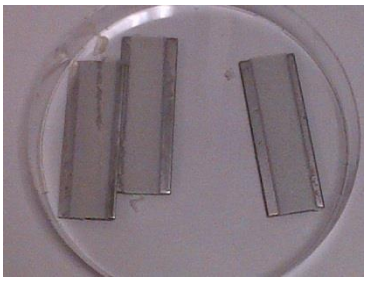

Fig. 6: Picture of fouling deposits onto coated stainless steel surfaces after fouling runs.

RESULTS

The adhesion data collected are presented in two sections.

Firstly, some results illustrating the ability of the ultrasonic test to evaluate adhesion values of viscoelastic fluid/substrate are shown. Secondly, the ultrasonic test was

used for determining milk fouling deposit adhesion onto coated stainless steel plates with various surfaces properties.

Adhesion of glucose syrup: reliability of ultrasonic test for measuring adhesion strength.

Fig. 7 is an example of four envelops of the impulse response at the interface glass/glucose when ultrasonic tests are performed. Each dashed line stands for a different level of dilution for the glucose syrup solution. The black solid line corresponds to the reference signal (i.e. the reflection signal at the interface glass/air when glass surface is not covered by a glucose syrup solution).

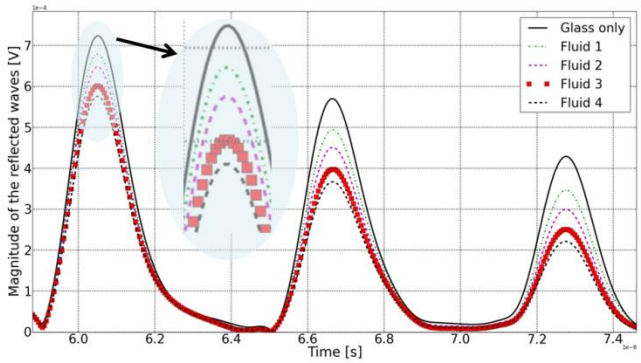

Fig. 7: Shape of the reflected waves at the interface between the glass slide and fluids as function of time.

Three reflected echoes are shown on Fig. 7. As expected, the amplitude of the first echo received is higher than the following.
For the first three echoes, it can be seen that:

- The amplitudes of reflected wave in the absence of deposit (i.e. at the interface glass/air) are the highest.

-The amplitudes of the reflected wave measured are correlated with the level of dilution of glucose syrups. Indeed, for the four glucose syrup solutions tested, the amplitude of the reflected wave increases with the rate of dilution of glucose solution.

Fig. 8 is an example of the amplitude evolution of the reflected wave measured at the interface glucose syrup/glass at different temperatures.

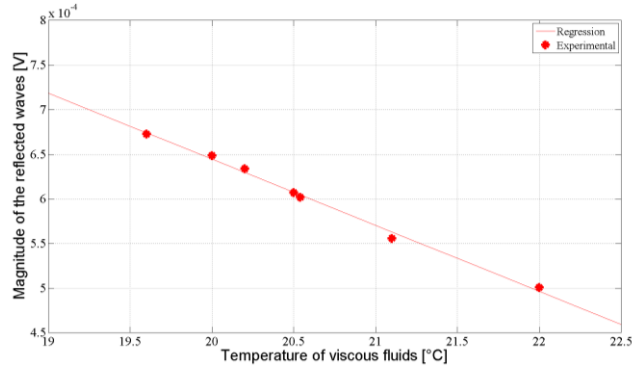

Fig. 8: Amplitude of reflected waves (1st echo) at the interface glass/fluid 3 as function of temperature.

Analysis of Fig. 8 shows that amplitude of reflected wave is highly dependent of fluid temperature. This trend was expected since viscosities of glucose syrup solutions (and consequently adhesion strength) are temperature-dependent.

The same temperature dependency is observed when wave reflections are measured at the interface bare stainless steel/glucose solution and for the other glucose solutions studied (data not shown here).

All these results support the hypothesis that it is possible to discriminate adhesion cases by measurement of the magnitude of the ultrasonic reflected wave at the substrate/fluid interface.

Fig. 9 reports for the two substrates investigated the evolution of reflection coefficient measured by US test as a function of the glucose syrup viscosity.

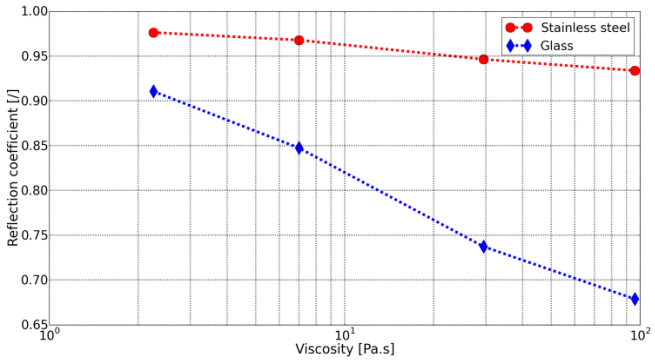

Fig. 9: Evolution of reflection coefficient at the interface substrate/glucose syrup versus viscosity.

For a given substrate, the reflection coefficient depends on mechanic properties of the material. For each substrate the reflection coefficient increases with the viscosity value.

It can also be noted that the reflection coefficient doesn't achieve a unique value for a given fluid viscosity. Indeed, the substrates being different, it is normal to have different reflection coefficients. In addition, the adhesion strength is not only dependent on the viscous properties of the deposit but also on surface substrate properties. Slip effects for the two substrates are quite different and may induce different adhesion mechanisms.

Table 3 presents pulling-force results obtained when glucose syrup solutions were removed from substrates. Classification of adhesion based on the reflection coefficient is consistent with pulling test results.

Indeed, when the pulling strength increases, the reflection coefficient decreases.

Reflection coefficient seems to be a good parameter for characterising the adhesion between a solid substrate and a fluid. Indeed the adhesion classification delivered by ultrasonic device is in agreement with both the force of detachment measured by conventional fracture test and Newtonian viscosity.

Adhesion characterisation of whey protein fouling.

Adhesion of the whey protein deposits formed on various coated surfaces has been measured using ultrasonic technique. Experimental results are plotted in Fig. 10.

Table 3: Pulling force results for removing the glucose syrup from glass and bared stainless steel.

Solution of glucose syrup	Pulling-force between glass slide/fluid at 20.5°C	Pulling-force between stainless steel/fluid at 20.5°C
fluid 1	5.88 N	7.02 N
fluid 2	7.29 N	8.17 N
fluid 3	7.68 N	8.99 N
fluid 4	8.05 N	9.94 N

An increase of the reflection coefficient with the contact angle value of the coated surface is observed. These results are consistent with the literature. Indeed, different adhesion forces of the fouling deposit are expected since one approach to reduce fouling and to increase the removal of formed deposits is the defined modification of the energetic and topographic surface properties (Boxler et al.). Moreover, It is widely admitted in literature that, the surface adhesion decreases when the contact angle is elevated (Jimenez et al.).

At this stage, it is difficult to conclude concerning the scatter of the data. Indeed, firstly fouling deposit samples obtained are heterogeneous and have a high porosity that may induce dispersion in the reflection coefficient determination. Another hypothesis is that the roughness of coated surfaces is not equivalent (Table 2). These roughness range differences affect significantly the relationship between adhesion and water contact angles.

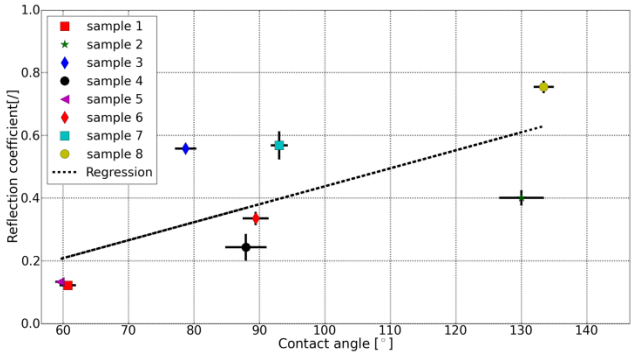

Fig. 10: Correlation between reflection coefficient and contact angle measurements for different cold plasma coatings.

CONCLUSIONS

In this study, an ultrasonic technique has been proposed to characterise the adhesion of fouling deposits. The ultrasound test consists in monitoring the reflected wave at the deposit/substrate interface after shear waves had been propagated through the substrate containing the deposit. The feasibility of the technique could be established.

It was shown that the determination of the ultrasound reflection coefficient at the interface viscoelastic material/ substrate is an effective parameter for tracking adhesive interactions, making it possible to distinguish deposit adhesion forces.

Up to now, the ultrasonic technique has been used as a laboratory analytical method for classifying adhesion of well-known viscoelastic materials and starts to be employed to study adhesion of whey protein deposit on various coated stainless steel.

Even if the device required further validation, the first experimental results presented here are promising since the method go towards non-invasive and in-line characterization of fouling deposit adhesion.

This ultrasonic diagnostic could be very efficient for industrial application to induce better cleaning and dairy processing strategies in order to reduce fouling.

However further validations are required. To obtain different qualities of fouling deposit adhesion onto stainless steel, modulating the amounts of calcium during manufacturing processes samples will be considered in the future. Indeed, some authors have shown that a small change in the calcium concentration has an important impact upon the fouling behaviour (Guérin et al., 2007). The structure and appearance of the fouling deposit is highly dependent on calcium concentration since this mineral salt content has been shown to affect strongly the denaturation kinetics of beta-lactoglobulin (Petit et al, 2012).

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SUBSCRIPT

$S_{11}(t)$	Scattering parameters in the time domain of the input port [V].
$S_{11}(f)$	Scattering parameters in the frequency domain of the input port [V].
$r_{A/B}$	Reflection coefficient at the interface between the layers A and B [/].
Z_A	Acoustic impedance of the layer A [MRay].
Z_B	Acoustic impedance of the layer B [MRay].
Z_i	Acoustic impedance of the layer *i* [MRay].
ρ_i	Density of the layer *i* [Kg.m^{-3}].
c_i^T	Velocity of shear waves into a layer *i* [m.s^{-1}].
μ	Fluid viscosity [Pa.s].
a	Constant parameter used in equation 3.
b	Constant parameter used in equation 3.
θ	Fluid temperature [°C].
Ra	Arithmetic average deviation of surface profile [nm].

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