

## CLEANING MONITORING USING CONTROLLED NANO-VIBRATIONS

A. Pereira<sup>1</sup>, J. Mendes<sup>2</sup>, L.F. Melo<sup>3</sup>

<sup>1</sup> LEPAE, Department of Chemical Engineering, Faculty of Engineering, University of Porto, Rua Dr. Roberto Frias, s/n, 4200-465 Porto, Portugal([aalex@fe.up.pt](mailto:aalex@fe.up.pt))

<sup>2</sup> Department of Mechanical Engineering, Faculty of Engineering, University of Porto, Rua Dr. Roberto Frias, s/n, 4200-465 Porto, Portugal([jgabriel@fe.up.pt](mailto:jgabriel@fe.up.pt))

<sup>3</sup> LEPAE, Department of Chemical Engineering, Faculty of Engineering, University of Porto, Rua Dr. Roberto Frias, s/n, 4200-465 Porto, Portugal([lmelo@fe.up.pt](mailto:lmelo@fe.up.pt))

### ABSTRACT

The monitoring of fouling and cleaning has become very important in order to optimize production cycles, to increase the quality of the final product and to reduce environmental impacts. The aim of the study here presented was to use the MSS – Mechatronic Surface Sensor, to detect the cleaning end-point of hair shampoo removal under different cleaning conditions. It was found that this new monitoring tool was able to clearly identify when the equipment was cleaned, producing different cleaning curves for different cleaning conditions.

### INTRODUCTION

The problems associated with fouling are well known and well documented, and are usually associated with: a decrease of the heat transfer rate, an increase of the pressure drop and can endanger the process sterility. In order to keep the operational efficiency of the process plant, removal of these deposits is required. Both the deposit formation and its removal introduces huge costs (mainly associated with: maintenance, loss of production, production stoppage, water and chemicals consumption and wastewater treatment) to the plant operational cycles.

Different set of approaches are being assessed in order to reduce fouling or to easily remove these unwanted deposits. Such approaches go from 'Computer Fluid Dynamics' (CFD), that can help to optimize the design of the processing equipment and of the production/cleaning cycles (e.g. Asteridou, et al, 2007 and Friis and Jensen, 2002), to the development of new surface materials that show with promising results concerning the reduction of the initial adhesion (Rosmaninho and Melo, 2006).

Monitoring of fouling is required to optimize both the process and the cleaning cycles. Several authors reviewed the most well known fouling monitoring techniques (e.g. Janknecht and Melo, 2003; Hastings, 2002). Hastings (2002) lists temperature and pressure measurements among the most commonly applied industrial monitoring techniques. These techniques can provide valuable indications about the conditions of the deposits on the

surfaces of the processing plant but they have their drawbacks: pressure drop is usually not sensitive enough to detect the first attached layers or accurately assess the cleaning status of the surfaces after CIP, while heat transfer techniques are rather expensive and subject to almost unavoidable errors in wall temperature measurements.

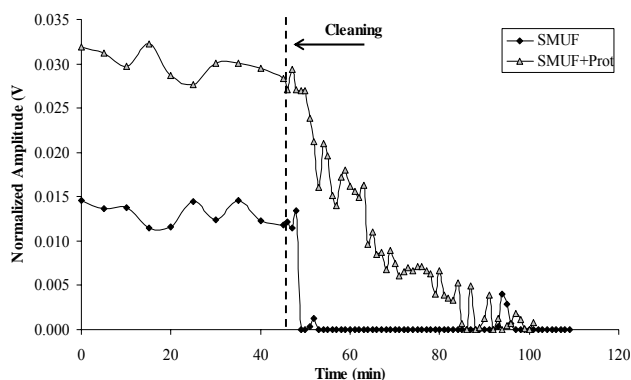
Other methods based on the propagation of mechanical waves have been reported. Hay and Rose (2003) and Lohr and Rose (2003) proposed the use of ultrasounds (such as guided waves, normal beam and pulse-echo) which require more sophisticated and expensive equipments than the MSS (which works with a much lower frequency – around 5 kHz). The work of Merheb et al. (2007) follows the principle previously applied to the MSS by the authors of the present paper (Pereira et al., 2006) although with a different configuration and different measuring parameters.

The MSS is an on-line fully integrated device based on the effect that the attachment or detachment of deposits has on the (nano) vibrations properties of the monitored surface. It uses a piezo element as an actuator to produce surface vibration. This vibration is then picked up by the sensor (accelerometer) and mathematically processed. The MSS has some intrinsic characteristics that make it an attractive tool to be industrially applied as a fouling and cleaning monitor, namely: a) it is non-intrusive since it can be applied directly on the outer surface of the equipment; b) it can be used with different types of surface materials (e.g. PVC, Stainless Steel, modified surfaces, etc); c) it processes the information collected over a sufficiently large area in order to avoid misinterpretation (misinterpretation is more likely to occur when the monitored area is small); d) it is robust enough to be used in industrial conditions; e) it gives information about the deposit's build-up/removal and about the deposits nature, on-line and in real-time.

### MSS Description

The MSS working principle is based on the analysis of vibration to detect and evaluate the amount and nature of the deposit that is attached to the surface. The adhesion or removal of the deposits affect the amplitude of the output signal, which was found to be inversely proportional to the

amount of deposit attached to the monitored surface. The damping factor is also another wave parameter that, complementarily to the amplitude, reflects the structural nature of the deposit (Pereira *et al.*, 2006). This work also reports that the MSS was found to follow accurately the build-up of a milk mineral solution (Simulated Milk UltraFiltrate- SMUF) and whey protein on a stainless steel surface, through the analysis of the amplitude of the surface wave. As regards the structural characteristics of the deposits, higher damping factors were observed for more elastic deposits (e.g. the whey protein has a more elastic structure than the calcium phosphate, so it presented a higher damping factor). That work (Pereira *et al.*, 2006) also reports the capacity of the MSS to follow different cleaning rates. Figure 1 (taken from Pereira *et al.*, 2006), shows the cleaning rates for a mineral milk deposit (SMUF) and for a mixed deposit of mineral (SMUF) and protein (Whey protein), using 0.5% of diluted nitric acid solution. It shows that the MSS curve obtained for the mixed deposit (based on a mineral-protein layer) has a lower cleaning rate than the mineral deposit, as it could be expected.



**Figure 1:** Cleaning of SMUF and SMUF with whey protein deposition curves assessed by the MSS (from Pereira, et al, 2006)

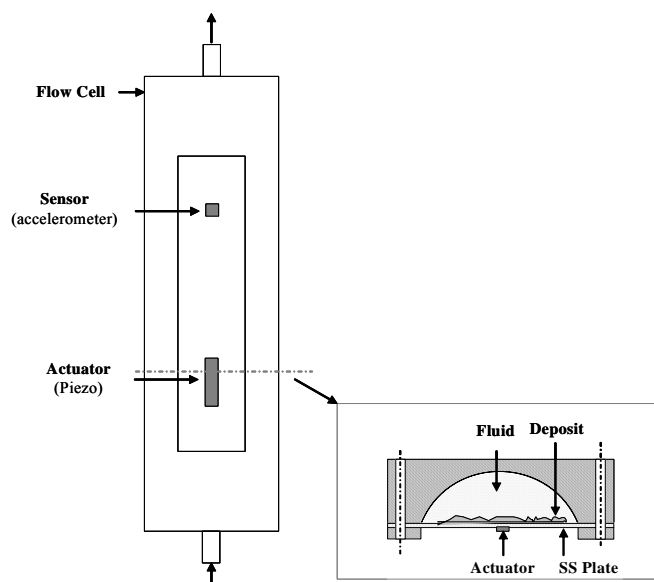
The cleaning end-point is one important issue concerning the optimization and flexibility of manufacturing plants (Wilson, 2005). The question addressed in this paper is to find out if the MSS is able to detect the cleaning end-point of the shampoo films removal on shampoo processing equipment. The problem studied is related with the detection of the removal of the residual shampoo film that stays attached to the equipment walls during cleaning. Since shampoo is quite different (mainly due its viscoelasticity) from water, the vibration that propagates along the tubing is affected by this difference and so different amplitudes are observed for the water and for the shampoo. The absolute

amplitude obtained with shampoo is smaller than the amplitude obtained with water. Based on this fact, the acquired signal was normalized in order to be similar to a typical cleaning curve (decrease with time). This signal varies between 0 and 1, these values corresponding, respectively, to the cleaned stainless steel (SS) surface and to the SS surface filled with shampoo (initial state). The MSS device is presently being patented (Mendes *et al.*, 2005 and Mendes *et al.*, 2006).

## MATERIALS AND METHODS

### MSS Measuring System

In the present configuration, the MSS is attached to the outer surface of a stainless steel (SS) plate that replaces the flat base of a semi-circular flow cell made of Perspex (internal diameter=30mm, equivalent diameter=18.3 mm; h= 350 mm) – Figure 2.



**Figure 2:** Schematic representation of the MSS inserted into the flow cell

The actuation (piezo) and the sensing (accelerometer) elements are glued to the outer surface of the SS plate at a distance of 9 cm, allowing a monitoring area of 18 cm<sup>2</sup>. One of the most important characteristics of the MSS is the fact that it takes advantage of the resonance frequency of the system, guarantying that the maximum output signal is obtained. The MSS measures changes in the vibration characteristics of the surface due to fouling formation/removal (or in the present case due to the shampoo cleaning). Both the actuation and the sensing processes are automatically carried out via a data acquisition board

(National Instruments, NIDAQ-Card PCI-6221) inserted into the computer. The SS plate is actuated by a piezo element (BM 70/25/200 M, Piezomechanik) with a sinusoidal wave with frequency = 5.1 kHz (which matches the resonant frequency of the system) and the vibration response is then captured by an accelerometer (ADXL-103, Analog Devices) and mathematically processed (to determine the amplitude and the damping factor).

### Experimental cleaning conditions

The work reported in this paper shows the cleaning of commercial shampoo with water at different velocities and temperatures – Table 1. The main goal of these experiments was to determine if the MSS could be used as a monitoring device to provide information about the cleaning end-point of shampoo.

**Table 1:** Experimental conditions used to clean the shampoo

Temperature (°C)	Velocity (m/s)	Reynolds number
31	0.142	2915
31	0.212	4373
51	0.142	4220
51	0.472	14066

The experiments have been performed with commercial hair shampoo with the trade name SUNSILK® (color radiant).

### Cleaning procedure

All the experiments followed the same cleaning procedure:

1) The water baseline was determined by recirculating the water at the defined temperature and velocity:  $A_{inv} \approx 0$  (amplitude correspondent to the clean flow cell);

2) The flow cell was filled with shampoo. The amplitude was determined for the shampoo at room temperature and with no recirculation –  $A_{inv} \approx 1$ .

3) The shampoo film was removed (with water under the conditions stated in point 1) and the amplitude of the stabilization zone was determined. When the MSS signal reaches the zero value ( $A_{inv} \approx 0$ ), it means that the flow cell is cleaned. On the other hand if an output signal between  $0 < A_{inv} < 1$  is obtained, it means that there is still a shampoo film attached to the sensor surface. It is important to note that the cleaning experiments were carried out without recirculating the water.

### Cleanliness confirming methods

In some of the experiments the cleanliness of the flow cell was confirmed by: visual inspection, absorbance and contact angle measurements.

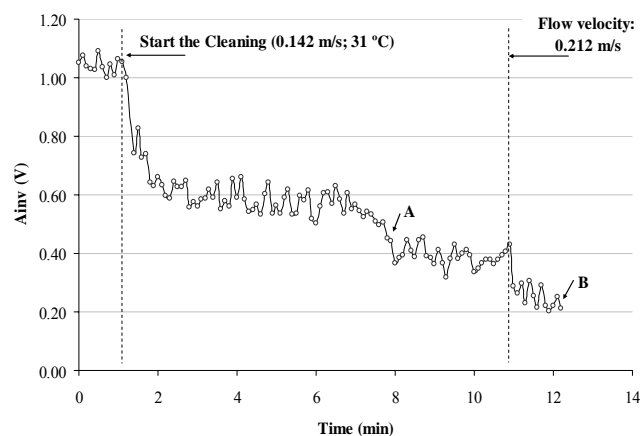
- **Visual inspection:** a few photos and movies were taken during the cleaning process. The movies allow checking what happened in terms of the shampoo removal at a given cleaning time for different removal conditions.

- **Spectrophotometry:** at the end of the experiment, the water was drained out of the flow cell. The absorbance of this bulk water was determined using a spectrophotometer (T80 UV/VIS Spectrometer, PG instruments Ltd) at  $\lambda = 225$  nm. The blank (control) sample was tap water (the tap water used to clean the flow cell).

- **Contact angle (CA) measurements:** the basic procedure used to determine this parameter was similar to the one used to evaluate the absorbance of the bulk fluid. After draining the water out of the flow cell, the SS plate was unscrewed and the CA was determined on the MSS surface (fluid side). The CA values were determined by the sessile drop method in a contact angle equipment (DataPhysics OCA15 Plus) with water as reference liquid. The evaluations of the contact angle values have been done using an image analyzing system. The comparison between the CA measurements for the initially clean SS plate and after running the removal experiments are a measure of the cleanliness of the surface.

## RESULTS AND DISCUSSION

**Figure 3** presents the removal curve obtained with the MSS for the cleaning of shampoo with water at 31 °C and 0.142 m/s.



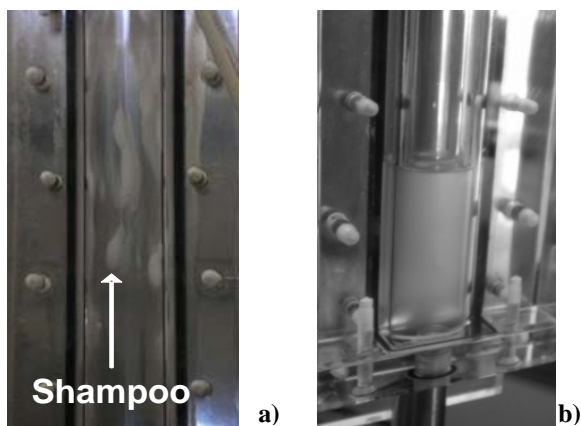
**Figure 3:** Cleaning of attached shampoo film with water at 0.142 m/s and 31 °C. At 11 min the velocity was increased to 0.212 m/s.

As soon as the water started to circulate through the flow cell, the MSS output signal ( $A_{inv}$ ) started to decrease. This is according to what was expected since, as mentioned in the ‘Material and Methods’ section,  $A_{inv} = 0$  corresponds to the

cleaned state of the SS plate. It can also be seen, that the first stabilization zone (between 8 and 11 min) is around 0.40 V, indicating that the plate is not completely cleaned. A picture taken at point A (Figure 4 a), confirms the existence of attached shampoo film to the upper walls of the flow cell.

Around 11 min, an increase on the water flow velocity from 0.142 m/s to 0.212 m/s was imposed. This perturbation resulted in a decrease of  $A_{inv}$ , since an additional layer of the attached shampoo has been removed (visually confirmed). Two independent methods, spectrophotometry and contact angle measurement, have been used to confirm the cleaning state of the flow cell at point B (end of this experiment). At point B (Figure 3) the water solution was drained out of the flow cell – Figure 4b, and its absorbance was determined – Table 2. Figure 4b shows that the drained water was very turbid, this fact confirmed in Table 2 by the high absorbance value (0.253), which indicates that the bulk water still contained shampoo. Additionally, contact angle measurements have been determined on the fluid side of the MSS surface (after being dried). The CA values obtained at point B and for the clean SS are shown in Table 2 indicating the existence of a remaining film on the MSS surface since the CA value at the end of the experiment (point B) is smaller than the one obtained for the initially clean surface.

The absorbance and the contact angle measurements confirm the existence of shampoo on the flow cell and, furthermore, that some of this shampoo attached to the SS plate (which is what mostly affect the MSS measurement). This conclusion confirms the MSS information at point B,  $A_{inv}=0.22$  V.

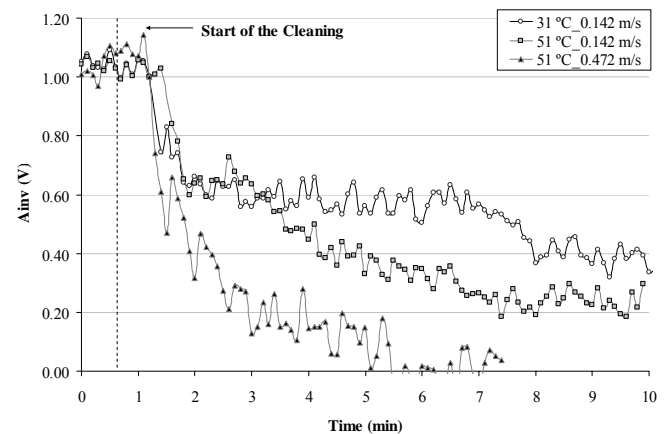


**Figure 4:** Images of the flow cell: a) during the shampoo cleaning process- point A in Figure 3; b) during the draining of the water out of the flow cell – point B in Figure 3.

**Table 2:** Values obtained with the MSS and with the two independent methods (determined at the beginning- Clean SS plate- and at the end of the experiment - Point B)

Measured Parameter	At point B	Clean SS plate
Absorbance	0.253	Blank Sample
Contact angle (°)	33.3 ± 4.6	46.6 ± 2.0
MSS Value – $A_{inv}$ (V)	0.22	0.00

Two of the most often studied parameters for the improvement of cleaning strategies are the temperature and the flow velocity. With the purpose of evaluating the MSS response to the removal of the shampoo under different cleaning conditions, the water temperature and flow velocity have been changed. Figure 5 compares the cleaning curves assessed with the MSS, for water recirculating at the: 31 °C and 0.142 m/s (curve already present in Figure 1); 51 °C and 0.142 m/s and 51 °C and 0.472 m/s.



**Figure 5:** Shampoo cleaning curves assessed with the MSS. Cleaning conditions: 31 °C and 0.142 m/s, 51 °C and 0.142 m/s, 51 °C and 0.472 m/s

#### Different temperatures with the same flow velocities

Figure 5 shows that according to the cleaning conditions (different temperatures and flow velocities), different shampoo cleaning curves were obtained. The comparison between the two curves with the same velocity (0.142 m/s) and different cleaning temperatures (31 and 51 °C) indicates that the initial removal phase (between 0.5 and 1.3 min) is rather similar. This initial phase corresponds to the removal of the shampoo in the bulk, which is the first to be flushed out of the flow cell. It was visually observed that the removal of the shampoo from the flow cell walls, at these conditions, starts from the bottom to the top of the flow cell. The period between 1.3 and 2.8 min, which has a  $A_{inv}$  constant value in Figure 5, corresponds to the removal of

the shampoo in the zone between the flow cell entrance and the begin of the actuator (piezo element). This result is in accordance to what was expected, since the measuring area (as already mentioned in the introduction) is just between the actuator and the sensor. After 2.5 min, it can be seen that the removal curves are substantially different. In fact, after the same period of time (10 min),  $A_{inv}$  is respectively around 0.20 V and 0.40V for the removal at 51 °C and 31 °C. This result indicates that at higher temperature the final removal was more efficient than at 31 °C, which can be explained by the fact that higher temperatures are better to dissolve shampoo components.

#### Same temperature with different flow velocities

When the cleaning was performed at the same temperature (51 °C) but with different flow velocities (0.142 m/s and 0.472 m/s, respectively, laminar and turbulent flow), different shampoo removal curves were obtained. Similarly to what happened on the case previously described (different temperatures with the same flow velocity),  $A_{inv}$  only starts to decrease after removing the shampoo from the bulk. As expected, the cleaning of the shampoo at 51 °C and 0.472 m/s is much more efficient than at lower flow velocity. According to the cleaning curves presented in Figure 5, the flow cell after 7 min was completely cleaned (at 0.472 m/s). This fact was confirmed by determining the absorbance of the drained water and the contact angles of the dried MSS (fluid side) surface. The results are presented in Table 3, and show that the values at the end-point are very similar to the ones determined for the corresponding cleaned state.

**Table 3:** Values obtained with the MSS and with the two independent methods (determined at the beginning- Clean SS plate - and at the end of the experiment - Point C)

Measured Parameter	End-point	Clean SS plate
Absorbance	0.006	Blank Sample
Contact angle (°)	41.6 ± 3.8	46.6 ± 2.0
MSS Value – $A_{inv}$ (V)	0.00	0.00

#### CONCLUSIONS

The MSS is a fully integrated monitoring device that provides information about the amount of build-up/removal and nature of deposit. This paper shows that the MSS can also be used to detect cleaning end-points on shampoo cleaning processes. It was also demonstrated that different cleaning conditions lead to different curves, which are accurately monitored by the MSS. The end-points determined by the MSS have been positively confirmed by spectrophotometry and contact angle measurements.

The characteristics of the MSS respond to three important questions regarding the optimization of the production and cleaning procedures: a) When should the plant be cleaned; b) How should the plant be cleaned; c) When is the cleaning end-point achieved. Answering these questions contributes to optimize the cleaning procedures, improving the whole operational cycles of the plant, increasing the quality of the final product and saving in processing costs (mainly associated with water, chemicals, energy and wastewater treatments).

#### NOMENCLATURE

$A_{inv}$  – Inverse Amplitude (V)

CA – Contact Angle

MSS – Mechatronic Surface Sensor

SMUF – Simulated Milk Ultrafiltrate

SS – Stainless Steel

$\lambda$  – Wavelength (nm)

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