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ASSESSING THE CLEANABILITY OF STAINLESS STEEL SURFACES – DEVELOPMENT OF A TESTING METHOD FOR STARCH AND PROTEIN BASED SOILS

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Abstract

To measure the cleanability of stainless steel surfaces under standardized conditions, a test stand was set up. Three stainless steel plates (size 200 x 100 mm) can be fixed to the test stand and sprayed with a water jet from a low-pressure nozzle for a period of several minutes. The plates were previously soiled with a starch or protein film with a pre-defined layer thickness and dried for several hours at 40°C / 50% RH. The progress of the cleaning is determined at set times by removing the plates and taking photographic pictures. When using starch-based soils a staining with a solution of iodine / potassium iodide (Lugol's solution) is necessary. For proteinaceous soils an illumination with UV light is sufficient without staining. With the image processing program ImageJ, the area of the cleaned surface can be identified and quantified.

Preliminary studies show that the method is suited better for starch-based soils than for protein films. A starch film has high adhesion to the stainless steel plate and is rinsed gradually from the surface. Protein films, however, form a strong cohesive soil layer which disintegrates in the region of the spray jet as plaques in an unpredictable way. This results in a high degree of variance of the values that were measured. Our findings indicate that for the test soils used, neither the alloy (AISI 304 L / 316 L) nor the roughness of the surface (0.8 / 0.2 microns) have a significant influence on the cleanability of stainless steel surfaces.

1. Introduction

Manual and automatic cleaning is ubiquitous in food industry. While automatic processes have been designed to clean closed equipment in place (CIP-cleaning), open equipment frequently needs to be cleaned manually. In both cases, the soil that adheres to the surface of the equipment needs to be removed.

Fryer and Asteriadou [1] have classified soils in food industry in a "cleaning map", based on the type of soil and the cleaning mechanism. Three groups of soil are highlighted in this map, as they are frequent cause of problems: high viscous or viscoelastic (water soluble) soils, biofilms, and solid soils (that need dissolving). A further classification is possible, regarding the mechanisms of cohesion and adhesion during the cleaning process [2]. Cohesive forces within a soil layer have to be overcome, as well as adhesive forces between the soil and the surface.

A number of methods have been proposed to measure the cleanability of surfaces. They can be grouped as follows [3]: gravimetric methods, chemical-analytic methods, microbiological methods, and visual inspection methods. Visual methods are fast and do not need special analytical equipment. As digital cameras and image processing software are readily available, it is possible to evaluate pictures taken at various moments of a cleaning process and quantify the progress over time.

To date there has been little agreement on which surfaces are cleaned most easily [4-7]. In general, very smooth surfaces with a roughness of 0.8 µm or less are recommended for food industry. In pharmaceutical industry, electropolished surfaces are preferred to enhance cleaning. Various alloys of stainless steel are used for machinery and equipment, being different in composition, resistance to corrosion, versatility, and price. So far, only few studies have been published on the effect of different surface modifications on cleanability [8, 9].

This paper focuses on the development of a visual method to measure the cleanability of stainless steel surfaces. Soiled stainless steel plates fixed to a test stand are sprayed with a water jet from a low-pressure nozzle for defined time intervals. Subsequent

standardized photography and image analysis allows quantifying the effect of surface roughness and different alloys.

2. Materials and Methods

A special test stand for cleaning experiments was designed by HPM Technologie GmbH (72525 Muensingen, Germany). It consists of a 10 L pressurized container MDJ and three universal spray heads PTR. Three stainless steel plates (size 200 x 100 mm) can be fixed to a rack (Figure 1).

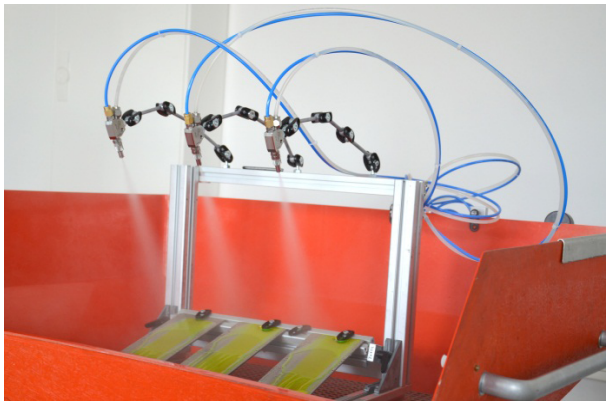

Figure 1. Test stand for cleaning experiments

The spray head nozzles are positioned right above the plates at a distance of 320 mm. Plates were tilted slightly by 20 degrees to allow drainage of cleaning fluid. The angle between the surface and the spray jet was 90 degrees. Partially demineralized water (8 °dH) at room temperature (24 °C) was used for all cleaning experiments. In each nozzle pressurized fluid (1 bar) and process air (1.5 bar) are mixed, resulting in a jet of fine droplets. Before each experiment the flow was controlled (88.6 ± 7.3 mL/min).

Stainless steel plates were provided by Henkel Lohnpoliertechnik GmbH (19306 Neustadt-Glewe, Germany). They were made of different alloys that are frequently used for machinery and equipment in food and pharmaceutical industry (see Table 1). The surface of each plate was either mechanically polished or electropolished. Roughness was measured according to DIN EN ISO 4288. Ra values of mechanically polished plates were 0.8 and 0.2 µm.

Table 1. Stainless steel alloys investigated

AISI 316 L (Mat. No. 1.4404)	AISI 304 L (Mat. No. 1.4307)	Duplex steel (Mat. No. 1.4462)

Each set of experiments consisted of the following steps: pre-cleaning of plates, application of the test soil, drying, cleaning under defined conditions, and, finally, evaluation of the cleaning experiment. For pre-cleaning plates were soaked in alkaline cleaning solution (Grasset by J. Kiehl KG, 85233 Odelzhausen, Germany, dilution 1:100) at 70 °C for 10 min, gently wiped with a cleaning rug and flushed thoroughly with demineralized water. They were dried for 45 min at 60 °C to remove all water.

Starch-based soil was prepared by mixing 5 g of starch (Maize starch K-classic from a local supermarket) with 95 g of water. This mixture was heated to the boiling point and cooked for 30 s. It was directly used after cooling to room temperature. Protein-based soil was prepared by mixing 10 g of milk protein powder (Protein-Concentrate 85, Tartex & Dr. Ritter, 79108 Freiburg, Germany) with 90 g of water. This mixture was kept at room temperature for 24 h prior to use in order to allow for complete solution of protein particles.

Test soil was applied evenly by use of an 8-fold applicator frame (BYK-Gardner GmbH, 82538 Geretsried, Germany) using gap No. 8 (nominal height 203.2 µm). Starch or protein solution was filled in the applicator frame, which was moved slowly over the stainless steel plate (Figure 2).

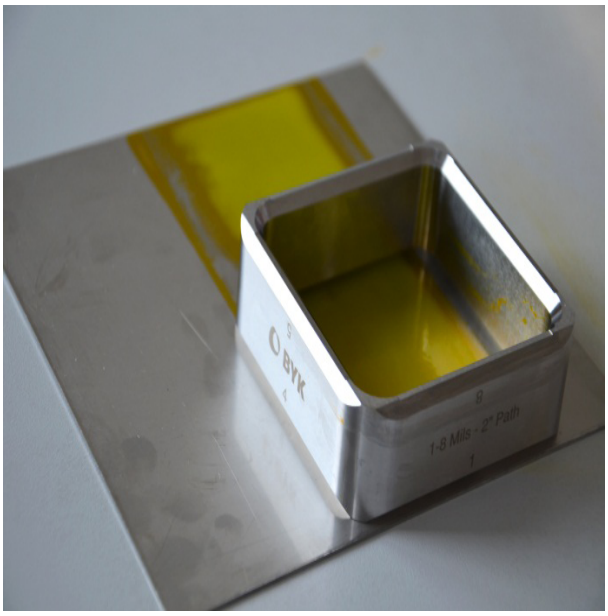

Figure 2. Applicator frame used for soiling the plates

Starch or protein films were dried for 5 h prior to the experiments at 40 °C and 50% relative humidity in a climate chamber. For the cleaning experiment, soiled plates were fixed in the test stand, spray washed as described before, and removed after a given time. After the experiment the plates were gently dried by pressurized air and stained with a solution of iodine / potassium iodide (Lugol's solution) for 5 min. They were rinsed for a few seconds and dried again by pressurized air.

Evaluation of the experiment was done by taking photographic pictures with a digital camera (Canon, PowerShot G11). Special care was taken to align the frame to the orientation of the plates. Pictures of starch films were taken inside a box of translucent white polyethylene on a black cardboard. Thus an even lighting could be provided without direct light that might have caused unwanted reflections. Pictures of protein films were taken in a dark room illuminated by a UV-light (wavelength 312 nm). As proteins naturally show fluorescence, no staining was necessary. All photographic pictures were evaluated using the image processing program ImageJ (source: http://rsbweb.nih.gov/ij).

3. Results and Discussion

The main characteristics of cleaning starch films (left) and protein films (right) on stainless steel surfaces are illustrated in Figure 3.

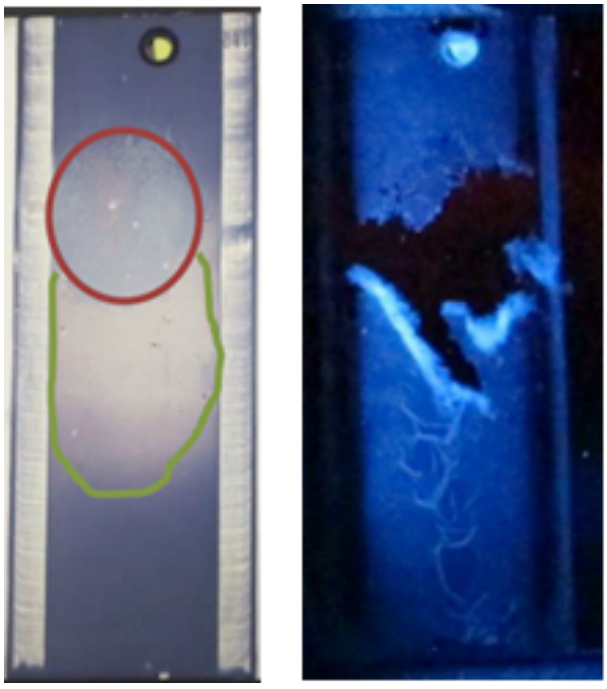

Figure 3. Adhesive layer of starch film (left) and cohesive layer of protein film (right) during cleaning experiments. Red circle: spray area; green line: area cleaned by draining water

A starch film shows high adhesion to the stainless steel plate and is rinsed gradually from the surface. As starch films need to be stained after cleaning, a lighter color indicates a better cleaned surface. The spray area of the jet stream (red circle) can be clearly distinguished from the area cleaned by draining water (green line). Protein films, however, form a strong cohesive soil layer which disintegrates in the region of the spray jet as plaques in an unpredictable way. Therefore the cleaned area has an irregular (jagged) form and does not increase evenly in time. Protein films only need to be illuminated by UV light for detection. Thus, dark areas show the cleaned regions, whereas a lighter color indicates remaining soil.

The steps necessary to evaluate the photographic pictures in a quantitative way are shown in Figure 4 for starch films.

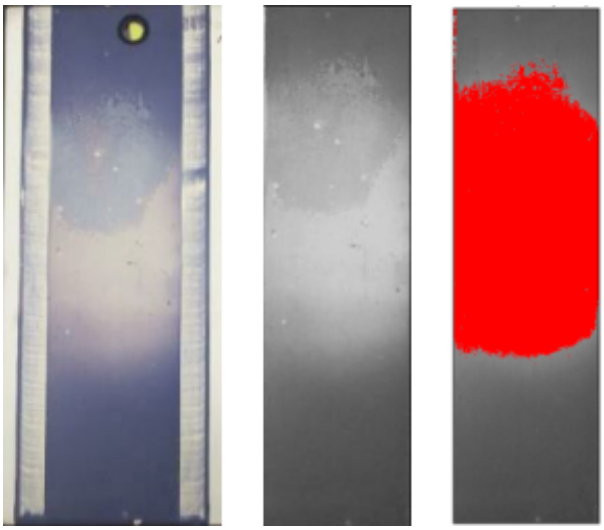

Figure 4. Transformation of original RGB picture of starch soil (left) into 8-bit grey scale (middle). Red area (right) shows the total area cleaned (TAC)

The original RGB picture (left) is first transformed into 8-bit grey scale (middle). In a second step the total area of the cleaned surface (right, red area) is detected by the image processing software ImageJ by using an appropriate threshold level (see below). The same steps are necessary for evaluation of protein films (see Figure 5).

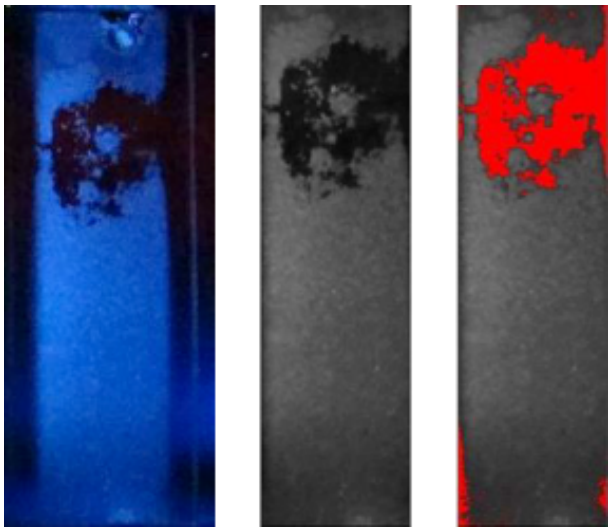

Figure 5. Transformation of original RGB picture of protein soil (left) into 8-bit grey scale (middle). Red area (right) shows the total area cleaned (TAC)

In Figure 6 the determination of the threshold level is explained for starch films.

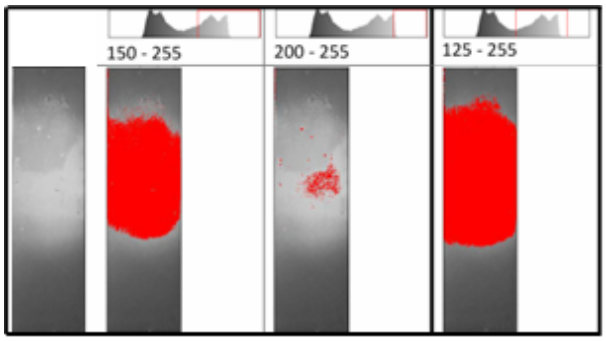

Figure 6. Determination of threshold level for starch films. The lower threshold value was chosen to match the red area best to the original picture (left)

The lower threshold level is varied until a good fit is seen, comparing the grey scale picture to the red area. A threshold level of 125 (of 255) was chosen for all experiments with starch films. The determination of the threshold level for protein films is shown in Figure 7.

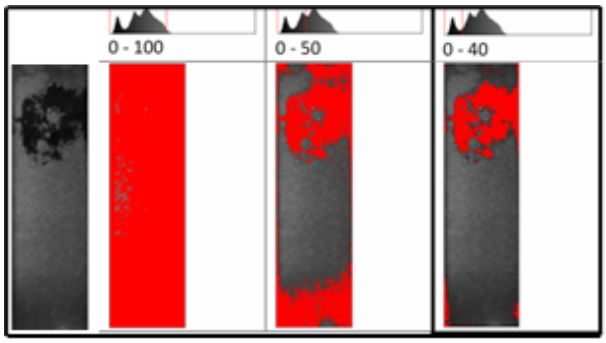

Figure 7. Determination of threshold level for protein films. The upper threshold value was chosen to match the red area best to the original picture (left)

In this case, the upper threshold level is varied for a perfect fit. A threshold level of 40 (of 255) proved best for all experiments with protein films.

The area of the clean surface was quantified by the software ImageJ and expressed as fraction of the area cleaned (AC) using formula 1:

$$AC = \frac{TAC}{RA} \cdot 100\% \quad (1)$$

with TAC: Total area cleaned (mm^2) as detected by ImageJ

and RA: Rectangular area between set bars (mm^2)

It is evident that the value of AC depends on the position of the bars (yellow lines) in Figure 8.

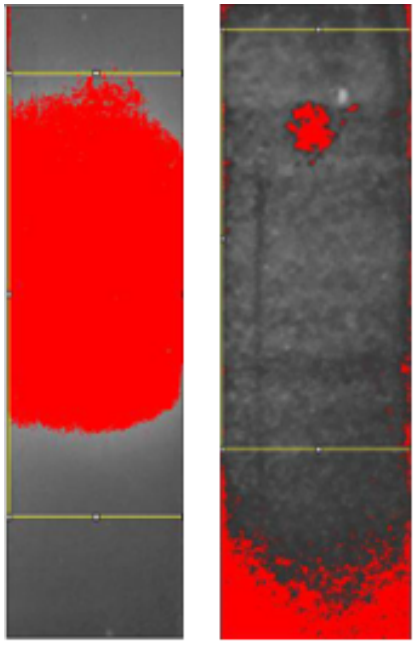

Figure 8. Positions of the bars (yellow lines) for the rectangular area (RA) for starch films (left) and protein films (right)

Therefore the bar position was set in the same way for all experiments with starch films (left) and protein films (right).

Typical results of cleaning experiments with starch-based soils are shown in Figure 9.

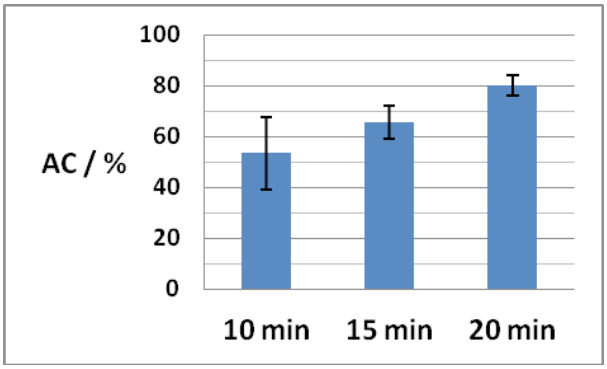

Figure 9. Area cleaned (AC) of starch films for various cleaning times. Stainless steel AISI 304 L (Mat. No. 1.4307), electropolished surface

Stainless steel used was AISI 304 L, the surface was electropolished. As can be seen the fraction of the area cleaned (AC) rises with an increase of cleaning time from 10 to 20 minutes: 10 minutes, 53.4 ± 14.3 % (n = 3); 15 minutes, 65.7 ± 6.5 % (n = 3); 20 minutes, 80.0 ± 4.2 % (n = 3). A similar increase can be seen in Figure 10 with protein films.

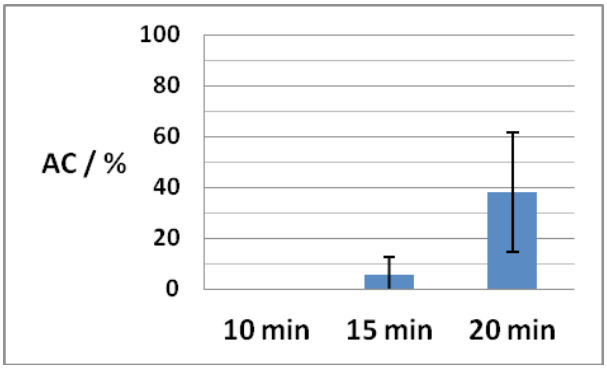

Figure 10. Area cleaned (AC) of protein films for various cleaning times. Stainless steel AISI 304 L (Mat. No. 1.4307), electropolished surface

However, mean values of the area cleaned (AC) are much lower, while standard deviation was considerably greater. The fraction of the area cleaned (AC) rises from 5.8 ± 6.9 % (n = 3) to 38.3 ± 23.4 % (n = 3) with an increase in cleaning time from 15 to 20 minutes. Due to a technical problem, values of 10 minutes could not be obtained.

To assess the effect of different alloys the cleanability of stainless steel AIS 316 L, AISI 304 L, and Duplex steel was compared. Results obtained from experiments with starch films are shown in Figure 11. As there was no significant effect of surface roughness (see below), data of all experiments with Ra = 0.8 µm, Ra = 0.2 µm, and electropolished surface could be combined to increase statistical power.

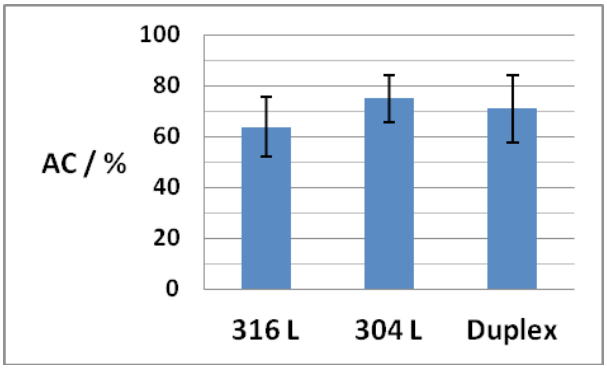

Figure 11. Area cleaned (AC) of starch films for various alloys of stainless steel. AISI 316 L (Mat. No. 1.4404), AISI 304 L (Mat. No. 1.4307), Duplex steel (Mat. No. 1.4462)

It is apparent from this figure, that there is little difference between mean values of the area cleaned (AC). Mean value of AISI 304 L was slightly higher (75.0 ± 9.3 %) than of AISI 316 L or Duplex steel (63.7 ± 11.7 % and 71.0 ± 13.3 %, resp.). The one-way ANOVA (n = 9) did not show any significant differences ($p \leq 0.05$) between different alloys.

Figure 12 presents similar experimental data with protein films.

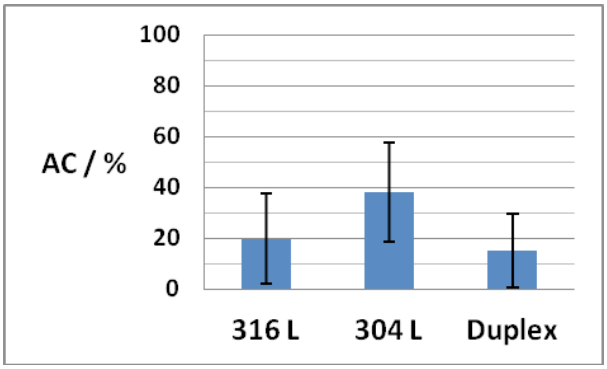

Figure 12. Area cleaned (AC) of protein films for various alloys of stainless steel. AISI 316 L (Mat. No. 1.4404), AISI 304 L (Mat. No. 1.4307), Duplex steel (Mat. No. 1.4462)

As can be seen, mean values of AC are much lower, again, with a larger variation of mean values compared with starch films. As described before, the standard deviation for each set of experiments is much larger. It seems obvious, that the mean value of AC is higher for AISI 304 L than for AISI 316 L or Duplex steel. However, due to the large values of standard deviation, the one-way ANOVA (n = 9) did not show any significant differences ($p \leq 0.05$) again.

To test the influence of surface roughness the same data was now grouped by this parameter, again. Figure 13 compares the experimental state for starch films.

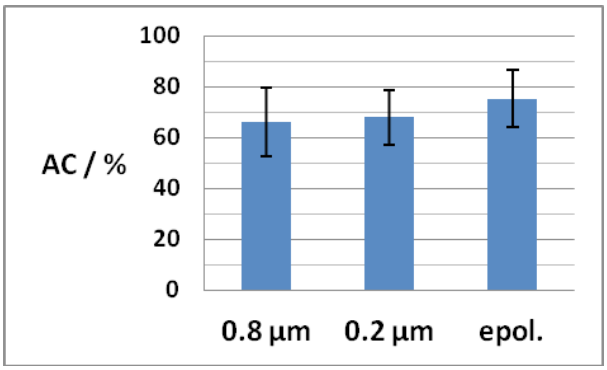

Figure 13. Area cleaned (AC) of starch films for various surface roughness. Electropolished surface (epol.); mechanically polished with Ra = 0.2 and 0.8 µm

It is quite evident, that there is little variation of the mean values of the area cleaned (AC). AC ranges from 66.3 ± 13.5 % (Ra = 0.8 µm) to 68.0 ± 10.7 % (Ra = 0.2 µm) for mechanically polished surfaces, while AC rises to 75.2 ± 11.2 % for electropolished surfaces. However, the one-way ANOVA (n = 9) did not show any significant differences ($p \leq 0.05$).

Figure 14 shows the data for protein films.

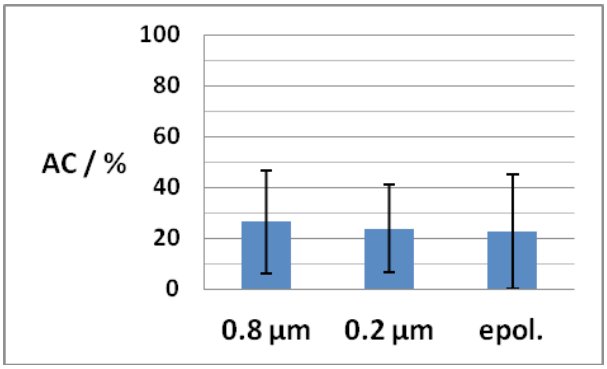

Figure 14. Area cleaned (AC) of protein films for various surface roughness. Electropolished surface (epol.); mechanically polished with Ra = 0.2 and 0.8 µm

As in the case before, there is almost no variation of the mean value of area cleaned (AC). Highest value was 26.4 ± 20.4 % for Ra = 0.8 µm, while AC was 23.8 ± 17.2 % for Ra = 0.2 µm and 22.7 ± 22.5 % for electropolished surfaces. As before, standard deviation of protein films was almost doubled compared to starch films. Again, the one-way ANOVA (n = 9) did not show any significant differences ($p \leq 0.05$).

4. Conclusions

The purpose of the current study was to develop a testing method for assessing the cleanability of stainless steel surfaces used in food industry. The method uses photographic pictures and the image processing program ImageJ to identify and quantify the area of the cleaned surface in experiments under standardized conditions. This study has shown that it is possible to measure the effect of cleaning time for starch-base soils and protein films on stainless steel surfaces. This will enhance our understanding of cleaning processes and allows the quantification of kinetics needed for mathematical modeling.

However, it must be noted that this method is suited better for starch-based soils than for protein films. A starch film has high adhesion to the stainless steel plate and is rinsed gradually from the surface. Protein films, however, form a strong cohesive soil layer which disintegrates in the region of the spray jet as plaques in an unpredictable way. This results in a high degree of variance of the values that were measured. Nevertheless, our findings indicate that for the two test soils used, neither the alloy (AISI 304 L / 316 L) nor the roughness of the surface (0.8 / 0.2 microns) have a significant influence on the cleanability of stainless steel surfaces. The development of the standardized testing method, however, will facilitate the expansion to other soils, cleaning agents, physical conditions and surface characteristics, ultimately improving our understanding of cleanability.

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