EXPERIMENTAL INVESTIGATION ABOUT THE MILK PROTEIN BASED DEPOSIT REMOVAL KINETICS

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Abstract: - This paper intends to present a contribution to the experimental investigation about the dairy protein based deposit removal kinetics. The study was based on traditional cleaning technique and on the in-situ electrochemical method. The mathematical model was obtained with the use of a computational program that realize the system identification. The results obtained are adequate to represent the system dynamics.

Key-Words: - Chemical cleaning, system identification, removal kinetics

1 INTRODUCTION

Cleaning-in-Place (CIP) technique is extensively employed in the food industry so that the processing equipments do not need to be dissembled when they are cleaned. A considerable amount of research has been conducted in recent years to seek ways of improving the procedure. Both the chemical reactions and fluid shear play roles in the removal of fouling deposit, but the effect of chemical reaction is one that is of key importance. The dairy industry suffers from specific problems associated with cleaning production machinery, including heat exchangers. Nowadays process equipment involved with the handling of milk and other dairy products must be shut down entirely for an extended period of time during cleaning operations. The cleaning procedure involves flushing the machinery with high levels of caustic cleaning agents through the equipment in order to remove residues build up contamination. Once the cleaning is finished, the machinery must then be rinsed very carefully to remove all traces of these cleaning products (GORMEZANO, 2007).

The current research about CIP systems has taken a different approach, employing electrolysis to improve CIP performance (CHEN at al., 2003). Electrolysis is well known for surface cleaning and polishing (Pie β linger-Schweiger, 2001). The substances are ionized into electrically charged ions, and when an electric current is passed through them by means of conducting electrodes, the ions mode (in the electrolyte solution) towards the oppositely charged electrodes thereby giving up their electric charges to become uncharged atoms or groups, and are either liberated or deposited at the electrode or react chemically therewith.

The purpose of this paper is to present a mathematical model which represents the dairy protein based deposit removal kinetics to be further used as a CIP virtual sensor.

2 PBL – A MODERN APPROACH

The **Problem Based Learning** is probably the best way to teaching at the present, due to its dynamic characteristics. The students are faced with real situations, where they need to present a practical / theoretical point of view to solve the problem presented.

The problem present in this paper is about to obtain a single transfer function for a heat exchanger prototype, using the process response to step changes.

The post-graduate student was the responsible for the experimental methodology adopted in this paper.

A food engineering graduate student was the responsible for the experiments execution and data acquisition.

The experimental results were analyzed for both, post-graduate and graduated students, to get a critical point of view about.

3 PROBLEM FORMULATION

Milk is one of the main raw materials in the food industry. After this natural food product has been processed, the equipment must be cleaned. This is executed by means of a chemical process that removes the residual milk present in the equipment. Thus, residual milk monitoring is needed to assess the CIP cleaning quality in food plants. In a CIP system, an efficient feedback control requires the real-time estimation of the milk concentration. The availability of the measurements is therefore crucial.

XIN, CHENG and ÖZKAN (2003) develop researches about this subject, obtaining a mathematical model for the removal of milk protein deposit to describe the different stages of the cleaning process. Nevertheless, the mathematical model proposed by the authors was very complicated for industrial implementation on commercial control systems as a software sensor.

Meanwhile, although commercial available milk residual concentration sensors are improving, they are still expensive to buy and to maintain. Furthermore, as pointed out by MASSON *et al* (1999), they are not reliable in the long term, causing difficulties in standard feedback control. It is thus essential to estimate the milk residual concentration by other means than the physical sensor, i.e. by a software sensor.

A software sensor computes an estimate of some quantity of interest, based on a mathematical model and other faithful measurements. The computed estimate may be used in place of the measurement when the latter is missing, or as a tool to validate an unreliable physical measurement. In most real world applications, the software sensor estimate will not be as accurate as a carefully tuned physical sensor. If it is designed to replace a physical sensor, the user should be ready to encounter an accuracy loss. But the software sensor has other purposes. It may give predictions of laboratory data, estimates when the measure is missing, and provide a sensor diagnosis when the measure is available.

There are two kinds of software sensors: model-based and data driven. Model-based, or deterministic software sensors can be built when the physical, biological and chemical relations between the measurements are known up to some constants, and that these

constants can be identified. The model is derived from the problem analysis, and the software sensor is build thanks to the estimation of the model parameters. Data-driven, also known as black-box or statistical software sensors are to be used when no accurate model is known. Data-driven methods include kernel and spline smoothers, additive models, projection pursuit and neural networks. These methods estimate the statistical dependence between measurements. For this purpose they require a 'training set' of valid past measures, including the quantity of interest. Hence, the software sensor does not learn the physics of the process, but the behavior of the physical sensor, which had to be installed to provide the training examples.

4 MATERIALS AND METHODS

The preliminary experiments were conducted using a system shown in Figure 1. The system studied consists of: (*i*)- 20 L capacity reservoir containing a 0.5 % wt solution of sodium hydroxide; (*ii*)- centrifugal pump; (*iii*)- rotameter (0 - 5 L/min); (*iv*)- $\frac{1}{4}$ " 316 stainless steel tubing; (*v*)- adjustable output DC power supply and the (*vi*)- 316 stainless steel test section.

First of all, the powdered milk solution was prepared dissolving two spoons of NINHO[®] powdered milk with 200 mL of distillated water. This milk solution and the test section were transferred to a pan of adequate dimensions and then stove heated for 30 minutes after the boiling point. The pan was constantly agitated during the heating step. The coated tube was then cooled from outside using running tap water and inspected in order to ensure that it had a continuous uniform film before being put in to the cleaning loop. Further, the milk embedded test section was replaced in to the system presented at Figure 2.

The 316 stainless steel test section used with the system studied has 15 mm internal diameter and 150 mm length. It was pre-coated with milk protein film as described above. A NaOH reservoir maintained at ambient temperature was used to storage the cleaning solution prepared. The flow velocity of the cleaning solution is controlled by adjusting the electrical current furnished to the DC drive pump motor by means of a DC power supply. The flow was monitored by a float type flow meter. In the once-through cleaning system, the cleaning solution containing the removed deposits was continuously discarded and an effluent sample was automatically collected at 1 s intervals to monitor the whole cleaning process.



Figure 1 – System studied for CIP purposes

The electrolysis device was implemented into the 316 stainless steel test section as shown at Figure 2. The stainless steel test section provides an electrochemical apparatus comprising

an electrolyte to be applied to in-situ metallic components and a voltage control means for applying a voltage to an electrolyte (i.e. the chemical cleaning solution) with the metallic components acting as either anode or cathode. When an electric field is applied to a fouled metal surface, it can remove the fouling. Gas bubbles are formed as a result of electrolyzing of the solution on the surface of the electrodes. The electrochemical reactions also provide the formation of protons (H^+) and the formation of hydroxyls (OH) depending on anode or cathode sides. This causes swelling of protein deposits making it easier to remove them. The gas bubbles formed provide an additional physical force to dislodge the attached foulant, whether mineral or protein. This last action might be the primary mover of the fouling deposit.



Figure 2 – The test section studied for CIP purposes

In a further step, rinse water was used after the detergent. In this case, after the use of NaOH solution, rinse water was used in the same flowrates as detergent solution. A laboratory pH meter (MICRONAL, model B474) was employed to measure the the pH profile. This pH meter was adapted to work as a continuous analyzer. The sample rate used was approximately equal to 1 second, with Hyperterminal® of WINDOWS®. Figure 3 presents the pH meter used in this paper and Figure 4 shows its adaptation to work on-line.



Figure 3 - pH meter used in the assays



Figure 4 – pH meter adaptation

All of the tests were executed by a student of the Food Engineering graduation course, that is the responsible for get the experimental data to be used in this paper, according to the professor orientations. This student receives all the technical orientation for the experiments development. All the experiment preparation was also executed by the graduation student.

5 RESULTS AND DISCUSSION

The assays were executed using the following flow rates: $2,3 \ L/min$, $2,5 \ L/min$ and $3,0 \ L/min$. The cleaning operations were executed during 5 minutes for each flow rate considered in this paper. The assays were realized tree times each one. Figure 5 shows the results obtained for the cleaning with conventional technique (without the applying electrochemical influence).



Figure 5– Cleaning profiles (ambient temperature) for milk protein in rinse water

The system identification was executed with the use of a CONNOISSEUR[®] software (SIMSCI, 2005) which adopted that the assays were executed using the following flow rates: 2,3 *L/min*, 2,5 *L/min* and 3,0 *L/min*. The cleaning operations were executed during 5 minutes for each flow rate considered in this paper. The assays were realized tree times each one. Figure 6 shows the results obtained for the system identification of the cleaning procedure with conventional technique (without the applying electro-chemical influence).



Figure 6 – Cleaning profiles (ambient temperature) for milk protein in cleaning solution without using eletrolysis technique (modeled and real curves)

The equations that represent the mathematical model of milk protein based deposit removal kinetics were obtained with the use of CONNOISSEUR[®] software resources (SIMSCI, 2005). Equation (2) represents the kinetics of milk protein removal for the conventional procedure to 2,5 L/min and equation (3) for 3,0 L/min flow rates.

The system model using the identification procedure employed in this paper, is presented in Figure 7 (CONNOISSEUR, 2005).



Figure 7 – Mathematical model of the cleaning process studied

6 CONCLUSIONS

The mathematical models presented above suggest a first order dynamic behavior for the process studied. Although the mathematical expressions show a second exponential term, its influence in the final result is negligible. The experimental data obtained by CHEN et al. (2003) also suggest this kind of dynamical behavior. This approximation is frequently observed for most of the control systems of industrial processes, assuring adequate performance for the process control. So, the mathematical model could be replaced by a simple transfer function that considers this approximation and make easy its implementation on commercial control system equipment.

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