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Comparison of *first principles* model of beer microfiltration to experiments via systematic parameter identification

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ABSTRACT

A first principles microfiltration model based on shear-induced diffusion is compared to experiments performed on the clarification of beer. After performing an identifiability and sensitivity analysis, the model parameters are estimated using global minimization of the sum of least squares. The model is compared to different series of experiments, where either crossflow or permeate flux is varied. This study is concluded with a parameter study on the scaling of the filtration time with various model parameters. We have found that the filtration time primarily depends on two dimensionless numbers, namely the normalized critical distance for cake layer formation, and the dimensionless time required to plug all pores in the selective layer. We have found that there is an optimal setting of these parameters, rendering a maximal amount of filtrated beer in one cycle.

1. Introduction

We are involved in the development of a model on crossflow microfiltration of beer [37,36]. The ultimate purpose of the model is to use it in a model-based control application, which should minimize the consumption of energy and cleaning chemicals of the beer microfiltration process. This research is part of a larger European collaboration program (EU Cafe) on the use of model-based control in food applications [2,3,12]. In this program we develop a conceptual framework for utilizing first principle models in model-based control application. Key elements within this framework are (1) model reduction techniques [24], (2) model parameter identifiability and estimation, (3) optimal experimental design, and (4) optimization methods for control or design.

In our previous paper we have presented our first-principles model [36]. The model incorporates the most recent description of shear-induced diffusion [38,35,39], which is the dominant back-transport mechanism in the boundary layer above the cake layer, deposited on top of the membrane. Shear-induced diffusion is caused by hydrodynamic interactions between micron-sized particles, the suspended yeast cells in the case of beer, which are flowing in the boundary layer above the cake layer. The boundary layer is also known as the concentration polarization layer [9]. Earlier models incorporating shear-induced diffusion in microfiltration have been developed by Romero and Davis [31].

In a parallel research program we have developed the current state-of-the-art *first-principles* model for shear-induced diffusion [33,38,39,35], which is based on the soft matter concept of the *effective temperature* [34,35]. This concept has allowed us to extend the theory to bidisperse suspensions like mixtures of yeast cells and aggregates [39]. In a previous paper we have analyzed the applicability of the bidisperse model for beer microfiltration [36]. There, we have shown that shear-induced diffusion in microfiltrated beer is dominated by the yeast cells, and consequently that the model can be reduced in complexity. The model reduction has been performed via techniques based on scale analysis [14,32,24]. After integration along the radial direction, we have arrived at a one-dimensional model which is quite similar to the earlier models of Romero and Davis, and later models by Bacchin [5]. More details on this approach one finds in our earlier paper [36]. The description on shear-induced diffusion has been complemented with submodels describing (1) the cake layer, and (2) internal fouling of the membrane [36]. These fouling models we have derived via a literature review on models of beer microfiltration [37].

In this paper we will compare the new model with actual experiments. We have performed a large series of experiments, for a variety of different permeate fluxes and crossflow velocities. We note, that the used experiment setup differs in one important aspect compared to the system, we have investigated numerically in our previous paper [36]. Namely, the selective layer of the membrane is now facing the permeate side, similar to the industrial setups for beer microfiltration membranes [27]. Many earlier experimental investigations of beer microfiltration have used the reversed geometry, with the selective layer facing the feed side [10,17,40,37,1].

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In the experiments the permeate flux and crossflow velocity are controlled at constant value, and the transmembrane pressure and pressure drop over the feed channel are recorded in time. We have had no consistent data on the composition of the beer. This makes the number of experimentally measured quantities quite small compared to the number of model parameters, as given in [36]. This situation is comparable to models of biochemistry, which also contain a large number of state variables and model parameters, while the number of experimentally observables is quite limited. Consequently, in systems biology systematic procedures are developed for this challenging problem of parameter identification [15,29,7]. In this paper we will follow a similar systematic approach.

The aim of this paper is to show the application of the systematic approach, as developed within the overall EU Cafe project, to the development of a model of beer microfiltration, which is suited to be implemented in a model-based control application. In this systematic approach we have applied (1) model reduction, (2) sensitivity analysis, (3) model parameter estimation using experimental data, and (4) model parameter study to obtain scaling rules. In the remainder of this paper we subsequently discuss (1) the reduced model is described with special attention to adaptations made, compared to our previous model [36], (2) the setup of experiments used in the parameter estimation, (3) a detailed description of the systematic approach followed, and (4) the results of applied approach, including comparison of the model predictions and the experimental results. The paper is concluded with an overall discussion of our approach and results.

2. Description of beer clarification via crossflow microfiltration

2.1. Fouling during beer microfiltration

The fouling mechanisms occurring during the microfiltration of beer we have depicted schematically in Fig. 1. The hypotheses concerning fouling during beer microfiltration, we have formulated in our review paper [37]. The rough, unfiltrated beer can be viewed to be composed of three classes of particles, each having a distinct size $d_{p,i}$. These classes are (1) yeast cells with size $d_{p,1}$, (2) protein-polyphenol aggregates with size $d_{p,2}$, and (3) macromolecules with size $d_{p,3}$. It follows that $d_{p,1} \gg d_{p,2} \gg d_{p,3}$. Both the yeast particles and aggregates contribute to the turbidity, and in beers like Pilsner they need to be removed during clarification.

In several breweries the clarification is performed with membranes using crossflow microfiltration. The membranes used at industrial scale, are cylindrical hollow-fiber membranes with length L. The inner radius of the hollow-fiber is R_0, and its thickness is t. These membranes have two distinct regions: (1) a support layer with large pore of size D_1 and thickness t, and (2) a thin selective layer with small pores of size D_2. In the industrial membranes the support layer is facing the feed side, and is designed to retain the yeast cells, $D_1 < d_{p,1}$. The selective layer has negligible thickness. The pore size of the selective layer is chosen such that it retains the aggregates, but should allow the macromolecules to pass: $d_{p,3} < D_2 < d_{p,2}$.

Due to the retention of the yeast cells, a cake layer will build up. The growth of the cake layer is restrained by the high crossflow, which causes a particle backtransport mechanism [9]. The dimension of the yeast cell is 5 micron, which means that shear-induced diffusion is the dominant backtransport process [9]. A part of the aggregates is assumed to be captured by the cake layer, but the majority of it will pass it – and they will cause the membrane to foul. Due to the reverse geometry of the membrane, most of these aggregates is expected to be captured by the support layer, but some will enter the selective layer and plug the pores. We assume that the aggregates captured in the support layer will form a gel layer, that is still is permeable to water, solutes and macromolecules.

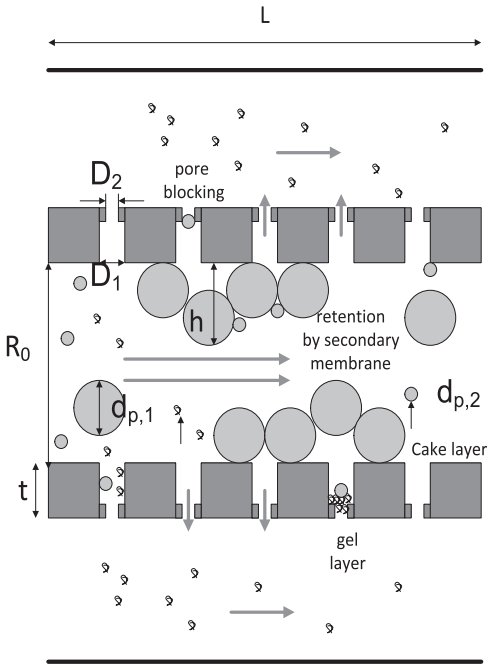

Fig. 1. Fouling modes during microfiltration of fermented beer, containing yeast, polyphenol–protein aggregates, and macromolecules, having sizes $d_{p,1}$ ($d_{p,2}$), and ($d_{p,3}$), respectively. The feed is following laminar through the inside of a hollow fiber membrane. Due to the pressure drop over the membrane the yeast accumulate in the cake layer above the membrane, as their size is $d_{p,1} > D_1$, the pore size of the support layer of the membrane, facing the feed. The smaller aggregates will largely pass the cake layer and the support layer, as $d_{p,2} < D_1 < d_{p,1}$. However, the aggregates cannot pass the selective layer, as $d_{p,2} < D_2$, the pore size of the selective layer. The aggregates will plug the pores of the selective layer. Macromolecules are that small, that most of them will pass through the membrane. A small fraction will absorb to the membrane, and can interact with aggregates – rendering a gel layer.

We note that, contrary to membrane setups with a selective layer facing the feed, we have no back-transport of aggregates or macromolecules. The combined support layer and cake layer has such a large retention capacity, that aggregates will never re-enter the boundary layer above the cake layer. One can say that their "gel polarization" layer are formed in the support layer of the membrane. It is estimated that at the end of filtration, when the selectively layer has become severely fouled, only half of the pore volume in the support layer is filled with aggregates. In industrial practice, the filtration is stopped if the transmembrane pressure exceeds a defined threshold. At this moment membrane cleaning is initiated. In this paper we restrict our analysis to filtration experiments with initially *non-fouled* membranes. The membrane are in the non-fouled state directly after manufacturing or after oxidative, chemical cleaning.

Similar to our previous study with the reversed membrane configuration [36], we expect significant interaction between cake layer build-up and internal membrane fouling, which will induce the downstream movement of the cake layer. Traditionally model studies on crossflow microfiltration consider only one type of particles and constant crossflow and permeate flux. There one has observed that cake layer builds up only when the permeate flux drops down below a critical value [13,30,31]. This critical value is also called the critical flux [18]. The axial position where the cake layer starts is called the critical position x_{cr}. For suspensions with only one type of particles, fouling the membrane via cake layer formation, the critical position is independent of time [31]. However, if multiple fouling modes happen simultaneously, the