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# Application of nanofiltration models for the prediction of lactose retention using three modes of operation

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#### ABSTRACT

The desal 5 DL membrane (Dow Chemical, USA) was used to recover lactose from whey ultrafiltration permeate. Lactose and ions rejection and permeate flux were measured using ultrafiltered sweet whey as feed solution. Three different operation modes (total recycle mode, concentration mode and continuous diafiltration mode) were considered. Lactose retention was predicted by means of the Donnan Steric Partioning model (DSPM) and the Kedem–Spiegler model (KSM). The best fit for the total recycle and concentration modes was obtained with the KSM. Moreover, the KSM predictions were worse for the CDM. These models contribute to a better understanding of the transport mechanisms involved in nanofiltration processes and to optimize the process at industrial scale. The two models can be considered as a useful tool to improve the recovery, demineralization and concentration of lactose.

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#### 1. Introduction

Bioactive substances present in whey such as lactose and lactose derivatives have many applications in both the pharmaceutical and food processing industries (cheeses, drinks, soups, diet food and infant formulations). Whey is a wastewater that shows a very high chemical oxygen demand (COD) value (between 40,000 and 60,000 mg  $\rm O_2/L$ ), due to the presence of compounds such as lactose, proteins, vitamins, fat and mineral salts among others. Thus it cannot be drained without a treatment. Therefore, there is a wide interest in obtaining the most economically viable processing conditions for whey reuse purposes. Lactose from whey is a good candidate for both recovery and valorization purposes decreasing the environmental problems related to this wastewater. On the other hand, membrane technologies are separation methods used in the food processing industry to concentrate and/or purify different streams.

Several models have been proposed to predict ion rejection in nanofiltration. Most of them are based on the extended Nernst-Planck (ENP) equation. One of those models is the Donnan Steric Partioning pore model (DSPM) Labbez et al. (2003) and Van der Bruggen and Vandecasteele (2002). Another model that has also been used is the Kedem-Spiegler model (KSM) (Yaroshchuk et al., 2000; Koyuncu and Topacik, 2002). These models try to explain the separation mechanisms of the nanofiltration process (Cavaco Morão et al., 2008). It has also been demonstrated that the proper-

ties of the feed solutions and the characteristics of the membrane play an important role on the process (Mohammad and Takriff, 2003).

Although there are many studies on modelling salt transport in NF membranes (Wang et al., 1995; Xu and Spencer, 1997; Schaep et al., 2001; Koyuncu and Topacik, 2002; Tay et al., 2002; Szymczyk and Fievet, 2005; Otero et al., 2006; Santos et al., 2006), not many of them have been performed with real feed solutions (Cavaco Morão et al., 2008; Cuartas-Uribe et al., 2009; Oatley et al., 2005). In addition, it is possible to optimize lactose recovery and salt removal using transport models.

Several authors worked in the utilization of nanofiltration to perform whey concentration and demineralization with different operation modes. Räsänen et al. (2002) and Suárez et al. (2006) worked in the concentration mode (CM) whereas Minhalma et al. (2005) worked in the total recycle mode (TRM) and CM. However, none of these authors compare the three modes of operation at the same time. No details about the effect of the membrane intrinsic properties on the degree of demineralization were reported in those works and the KSM or the DSPM were not used to predict or explain the experimental results obtained.

In this work the membrane technique of nanofiltration (NF) has been used to remove ions (especially polyvalent ions) and lactose from whey ultrafiltration permeate as reported by Cuartas-Uribe et al. (2009). Three different NF operating modes (total recycle, concentration and continuous diafiltration) were compared from the point of view of lactose retention and permeate fluxes. The concentration and diafiltration processes were able to concentrate lactose and remove monovalent ions from sweet whey wastewaters.

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#### Nomenclature

CM concentration mode Ps solute permeability (m/s) COD chemical oxygen demand real solute retention  $R_{\rm real}$ CDM continuous diafiltration mode **TMP** transmembrane pressure DSPM Donnan Steric Partioning mode TRM total recycle mode ENP

extende Nerst-Planck

hindrance factor for convection  $K_{i,c}$  $K_{i.d}$ hindrance factor for diffusion **KSM** Kedem-Spiegler mode nanofiltration membrane

Greek letters

reflection coefficient pore radius (m)

 $\Delta x/A_k$ thickness-porosity radius (m)

However, it should be noted that the retention of polyvalent ions is much higher. Therefore, to achieve a high whey demineralization degree, the combination of CM and CDM is necessary. It is worth to mention that previous works in the literature used whey as a feed whereas in this paper the whey was previously ultrafiltered. i.e., the feed stream used in this work has been the permeate obtained after the ultrafiltration of whey. The use of ultrafiltration prior to nanofiltration is expected to reduce fouling of nanofiltration membranes and therefore achieve a better performance of these membranes in terms of permeate flux. The aim of this work was to predict lactose retention by means of mathematical modelling.

The three operating modes tested are discussed in order to show that the DSPM and KSM are a useful tool not only in the prediction of the process performance, but also in the optimization of the process conditions (TMP, operating mode). These conditions can be selected to maximize lactose retention. However, it is worth to mention that the background of these models is an idealistic system that does not exist and therefore they do not have a physical meaning in terms of fitting parameters. In order to investigate the fitting of the model to the experimental data, permeate flux and lactose retention were determined at different ultrafiltered sweet whey (UFSW) concentrations. Therefore, the present study was divided into two parts: (1) estimation of model parameters and (2) comparison between the experimental data on lactose retention and model predictions for the different operation modes.

#### 2. Materials and methods

#### 2.1. Whey composition

Ultrafiltered sweet whey from a dairy industry was used as feed in the nanofiltration experiments. The ultrafiltration of sweet whey was performed as described in Cuartas-Uribe et al. (2007). Feed and permeate were analyzed for lactose, ions, fat and protein content. The analytical methods used to quantify fat, protein, lactose and ions content were those described in Cuartas-Uribe et al. (2009).

#### 2.2. Nanofiltration experiments

The membrane used in the experiments was a spiral wound membrane (desal 5 DL) supplied by Dow Chemical (USA). Membrane characteristics and a detailed description of the NF pilot plant were previously reported elsewhere (Cuartas-Uribe et al.,

Three modes of operation were tested: TRM (0.5-2.5 MPa); CM (1 and 2 MPa) and CDM (1 and 2 MPa). These experiments were carried out as described in Cuartas-Uribe et al. (2009). It must be pointed out that the feed that was remaining in the feed tank after the experiments in the CM was later used as feed for the CDM tests. In the CDM water addition was carried out continuously in order to maintain the feed volume constant. The volume dilution factor was studied in the range of 0-8 until a feed conductivity of 1 mS/cm was achieved. Experimental values on permeate flux decline and salt removal versus the concentration factor were previously reported by Cuartas-Uribe et al. (2009). The real solute retention  $(R_{\text{real}})$  and permeate flux  $(J_p)$  were determined in all the runs according to the equations that appear in Cuartas-Uribe et al. (2007).

#### 2.3. Application of the models

The models were verified following the procedure described in Cuartas-Uribe et al. (2007). However, different modelling techniques were used. In the DSPM model the dependence of the thickness-porosity ratio  $(\Delta x/A_k)$  with the viscosity of the feed was also considered. The solute permeability  $(P_s)$  and the reflection coefficient  $(\sigma)$  in the KSM were obtained by the regression analysis of real rejection and flux data. For that purpose the procedure proposed by Koyuncu and Topacik, (2002) was considered. Moreover. a comparison between model predictions for the three modes of operation is carried out.

#### 3. Results and discussion

#### 3.1. Model parameters obtained for the KSM and the DSPM

The membrane parameters ( $P_s$ ,  $\sigma$ ,  $r_p$ ,  $\Delta x/A_k$ ,  $K_{i,c}$  and  $K_{i,d}$ ) for the two models used (the KSM and the DSPM) and the three operation modes were estimated by means of the procedure described in Cuartas-Uribe et al. (2007). The values of  $r_p$ ,  $\Delta x/A_k$ ,  $K_{i,c}$  and  $K_{i,d}$ parameters are shown in Table 1. These values were very similar for each operation mode tested.  $K_{i,c}$ ,  $K_{i,d}$  and  $r_p$  can be estimated from the  $\boldsymbol{\sigma}$  values by means of an iterative method. The parameter  $\Delta x/A_k$  was calculated with the Hagen-Poiseuille equation. A detailed description of the procedure is found in Cuartas-Uribe et al. (2007). Therefore, the similarity of the values of the parameters can be attributed to a slight variation of membrane characteristics with the operating modes.

Table 2 shows that the parameters  $P_s$  and  $\sigma$  changed for each mode of operation tested. Both parameters depend on concentration (Bason et al., 2009; Cuartas-Uribe et al., 2007). The highest val-

Calculated parameters for the DSPM for the three modes of operation.

Parameters	Operation mode		
	TRM	CM	CDM
$r_{\rm p} \times 10^{-10}  ({\rm m})$ $\Delta x/A_k \times 10^{-6}  ({\rm m})$	5.025	5.050	5.005
$\Delta x/A_k \times 10^{-6} \text{ (m)}$	3.427	3.561	3.450
$K_{i,c}$	1.980	1.985	1.979
$K_{i,c}$ $K_{i,d}$	0.018	0.017	0.018

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