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Process Duration and Water Consumption in a Variable Volume Diafiltration for Partial Demineralization and Concentration of Acid Whey

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Process duration and fresh water consumption were determined (experimentally and statistically) for variable volume diafiltration (VVD) of cottage cheese through a flat sheet membrane (0.046 m²). The VVD process was performed at two volume decreasing ratios ($\alpha=0.75$ and $\alpha=0.5$). The VVD-0.75 process lasts much longer than the VVD-0.5 process if the same concentration degree is required. The VVD-0.5 process lasts longer than the VVD-0.75 process if it is aimed to achieve the same purification degree. At low purification degrees, both processes require similar quantities of fresh water, but better demineralization is possible after higher dilution, which is typical of the VVD-0.75 process. The mathematical model applied in estimating the duration of the process proved very accurate, which cannot be said about the fresh water consumption model.

Keywords nanofiltration; variable volume diafiltration; water consumption; whey

INTRODUCTION

One of the known methods for whey utilization is the separation of valuable components from undesirable ones, especially from the salts having monovalent ions (K^+ and Na^+). The valuable constituents—macrosolutes which prevail are protein (0.6–1.0%) and lactose (4.2–4.5%) accompanied by fat (0.3–0.5%) (1). Inorganic salts containing Ca^{2+} and P ions (0.3–0.5%) are also present. Microsolutes are salts with K^+ and Na^+ , especially $NaCl$, which is added during brining in cheese-making. The results of a great number of investigations have proven that nanofiltration (NF) is a suitable pressure driven process both for concentrating protein and lactose and demineralizing the concentrate (2–12). The basic separation

device is a nanofiltration (NF) membrane, which performs concentration and/or purification of the whey solution containing compounds with molecular weights between 100 and 500 Da. Most of the investigations deal with the optimization of working conditions (4,11,13); some of them consider a selection of adequate membrane (6) while others treat particular phenomena, such as hydrodynamics and mass transfer (6,12). However, all of the investigations mentioned are related to the conventional membrane separation process.

Each conventional separation process consists of several steps. The pre-concentration serves to concentrate the macrosolutes in the retentate according to their rejection coefficients. During this step, the permeate is removed from the system and no additional solvent is added. The diafiltration enables purification of the retentate, i.e., the microsolutes are transferred to the permeate according to their rejection coefficients. During this step, additional solvent is introduced to maintain constant volume. The final concentration of already purified macrosolutes is similar to the pre-concentration. In 1994, Jaffrin and Charrier (14) combined ultrafiltration with diafiltration and applied such an integrated process to albumin production. The process consists of a single diafiltration step but with continuously decreasing volume. Such a process enables simultaneous concentration and purification of solutes. Several authors have investigated possible applications of the VVD process. Various aspects were considered; an estimation of process duration was reported (15–17) and a possibility of decreasing water consumption was considered (16–19). Also, experimental investigation of a semi-batch mode of operation was performed (20). However, solutions of the mathematical models were not compared with the experimental data, which were missing at that time.

The potential of such a combination of NF and diafiltration, for the partial demineralization of whey, is investigated, especially from the point of view of process

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duration and water consumption. The measured values from the two VVD experiments, which differ in volume decreasing ratios, are compared with the values predicted with existing models. The selection of models is based on the references (16,17,20); some other models, based on concentration polarization and/or osmotic pressure (21,22) were not tested.

MATERIALS AND METHODS

Whey

Acid whey (pH = 4.9) was obtained from a local dairy in Szeged. It was a by-product of the production of cottage cheese. Table 1 shows whey composition.

Experimental Set-up

The experiments were performed on laboratory NF equipment at Corvinus University of Budapest, Department of Food Engineering, using a flat sheet membrane (XN45 Polyamide-Urea Nanofiltration Membrane, TriSep Corporation, USA) with an effective area of 0.046 m^2 , sucrose rejection of 96% and nominal molecular weight cut-off of 300 Da. The feed solution was pumped to the membrane by the Hydra – Cell Diaphragm Pump D 10 (Wanner Engineering, Inc. Minneapolis, USA). The transmembrane pressure across the membrane and flow were adjusted using the bypass valve and the main flow valve. The TMP was monitored by manometers (accuracy $\pm 5\%$) while the flow was measured using the rotameter (accuracy $\pm 4\%$, Unirota, Hungary). The temperature was kept constant by using a thermostat and monitored by a digital thermometer (accuracy $\pm 0.5^\circ\text{C}$) on the feed tank.

Preparatory experiments (2,23) were performed to find optimal values of the following independent variables: transmembrane pressure (2 MPa), recirculation flow rate (700 L/h), and temperature (40°C). The Permeate was continuously removed from the system while the retentate was recycled to the feed tank. Following the removal of each liter of permeate, fresh water was added to the retentate in the feed tank in alternating amounts of 0.75 L or 0.5 L. This crossflow semi-batch mode of operation, with the partial replacement of the removed

permeate, simulates two alternatives of a VVD process, which will be denoted as VVD-0.75 and VVD-0.5. Other details can be found in the paper by Román et al. (23).

Characterization of Feed, Retentate, and Permeate

Protein, lactose, fat, and total soluble solid content of the samples were analyzed using an Infrared Milk Analyzer Bentley 150 (Bentley Instruments, Inc. Chaska, MN) at the University of Szeged, Faculty of Engineering. Ion composition of the samples was determined with an ICP-MS instrument at the Department of Applied Chemistry, Corvinus University of Budapest.

MATHEMATICAL MODEL OF THE VVD PROCESS

Several mathematical models of the continuous VVD process with an incomplete rejection of the macrosolutes and microsolute have been presented in detail by Tokoš et al. (17). There are two key equations: the continuity equation for the volume changes in time:

$$\frac{dV}{dt} = (\alpha - 1)Q_P \quad (1)$$

and the mass balance equation for macrosolutes:

$$C \frac{dV}{dt} + V \frac{dC}{dt} = -JA(1 - R)C \quad (2)$$

Obviously, $\alpha = Q_D/Q_P$ represents a volume decreasing ratio while R denotes macrosolutes rejection. It is also useful to know that α is related to the concentration degree (C_f/C_o) and purification degree (c_o/c_f) in the following way (17,20):

$$\alpha = \frac{r \ln \frac{C_f}{C_o} + R \ln \frac{c_o}{c_f}}{\ln \frac{C_f}{C_o} + \ln \frac{c_o}{c_f}} \quad (3)$$

After substitution of the volume gradient from Eq. (1) into the mass balance (2) and bearing in mind that: $Q_P = JA$, one obtains:

$$V \frac{dC}{dt} = JAC(R - \alpha) \quad (4)$$

Rearranging Eq. (4), after substitution of volume with the expression:

$$C = C_o \left(\frac{V_o}{V} \right)^R \Rightarrow V = V_o \left(\frac{C_o}{C} \right)^{1/R} \quad (5)$$

results in:

$$t = \frac{V_o C_o^{1/R}}{R - \alpha} \int_{C_o}^{C_f} \frac{dC}{JAC^{(1+R)/R}} \quad (6)$$

TABLE 1
Composition of the crude acid whey

Macro-component	Value (%)	Micro-component	Value ($\text{mg} \cdot \text{dm}^{-3}$)
Total solid content	6.31–6.40	K	1489–1513
Lactose	4.06–4.41	Na	372.1–403.7
Protein	0.57–0.59	Ca	995.7–1004
Total protein	0.83–0.89	Mg	84.71–91.43
Fat	0.27–0.37	P	640.4–721.6

The solution of Eq. (6) is an estimate of process duration, which is an important quantity from the point of view of practical process application. Due to solving the integral in the time-equation (6), it is necessary to define how the permeate flux (J) changed depending on the factors which were changed during the VVD process. Some correlations can be found in the literature (14,17). Here, a concentration of the macrosolutes (protein, lactose, and fat) is taken as the control factor while the concentration of the microsolute is not taken into account. Such an assumption seems reasonable in the case of whey concentration and demineralization. To fit the experimental fluxes, the following three-parameter correlation is suggested:

$$(JA)^{-1} = p_1 C^3 C^{1/R} + p_2 C^2 C^{1/R} + p_3 C C^{1/R} \quad (7)$$

It allows for the integral in Eq. (6) to be computed analytically:

$$t = \frac{V_o C_o^{1/R}}{R - \alpha} \int_{C_o}^{C_f} \frac{p_1 C^3 C^{1/R} + p_2 C^2 C^{1/R} + p_3 C C^{1/R}}{C C^{1/R}} dC \quad (8)$$

The final solution of Eq. (8) is as follows:

$$t = \frac{V_o C_o^{1/R}}{R - \alpha} \left[\frac{p_1}{3} (C_f^3 - C_o^3) + \frac{p_2}{2} (C_f^2 - C_o^2) + p_3 (C_f - C_o) \right] \quad (9)$$

The rejection coefficients of the particular macrosolutes (protein, lactose, and fat) were calculated according to Suárez et al. (11) using concentrations in the permeate and the retentate. However, a double-averaging approach was applied:

- (i) to calculate the rejection of mixture of k components, whose mass fractions are known, and
- (ii) to cover the rejection changes during time, divided into n time-increments.

Following equation was applied:

$$R_{i,j} = \left(1 - \frac{C_{i,j,P}}{C_{i,j,R}} \right); \quad R = \frac{\sum_{j=1}^n \sum_{i=1}^k g_{i,j} R_{i,j}}{n} \quad (10)$$

The same equation was used for estimating the rejection of microsolute (salts with K^+ and Na^+) when taking the concentration of their ions into account.

Apart from the process duration, it is important to know how much dialyzation water (V_D) is needed. The equation from the volume balance of the VVD process seems very simple:

$$V_D = \alpha J A t \quad (11)$$

Also, the ratio between the dialyzation water and the initial volume of the suspension is as follows:

$$\beta = \frac{V_D}{V_o} = \frac{\alpha A t}{V_o} \int_{C_o}^{C_f} J dC \quad (12)$$

Before solving the integral within Eq. (12), the relationship between J and C must be substituted. Baring in mind that the three-parameter correlation (7) was suggested, as the appropriate one, this relation might be used for solving Eq. (12). However, its substitution into Eq. (12) leads to a form extremely difficult to integrate.

Therefore, a simplification is suggested, i.e., it is assumed that the permeate flux remains unchanged during the diafiltration. In this case, Krstić et al. (20) derived the equation for β -ratio as follows:

$$\beta = \frac{V_D}{V_o} = \frac{\alpha}{1 - \alpha} \left[1 - \left(\frac{C_f}{C_o} \right)^{\frac{\alpha - 1}{R - \alpha}} \right] \quad (13)$$

Considering that a relation exists between two degrees (purification and concentration):

$$\frac{c_o}{c_f} = \left(\frac{C_f}{C_o} \right)^{\frac{\alpha - 1}{R - \alpha}} \quad (14)$$

the alternative relation between water consumption and the purification degree can be expressed:

$$\beta = \frac{\alpha}{1 - \alpha} \left[1 - \left(\frac{c_o}{c_f} \right)^{\frac{\alpha - 1}{\alpha - r}} \right] \quad (15)$$

RESULTS AND DISCUSSION

Global Data

During the integrated nanofiltration and diafiltration process, the permeate fluxes were measured at two values of volume decreasing ratio ($\alpha = 0.75$ and $\alpha = 0.5$) and two sets of results were obtained. The VVD-0.75 and VVD-0.5 processes resulted in volume concentration ratios of 2 and 2.65, respectively. Chemical compositions of the feed solution, the retentate, and the permeate were determined throughout the experiments. Detailed analysis of flux changes as well as composition changes can be found in a recently published paper (23). Here, factors related to the VVD process itself will be analyzed. Relevant data are shown in Table 2.

An analysis of global data in Table 2 shows that the VVD-0.75 process starts using 13.8 dm³ of whey solution and ends up at a concentration degree ($C_f/C_o = 2.01$). Such a concentration requires a rather long process duration (18.9 h), especially keeping in mind that, during the process, a significant amount of fresh water is added

TABLE 2
Global data on two VVD processes

Subject	Measured quantity	Two alternatives of VVD process	
		$\alpha = 0.75$	$\alpha = 0.5$
Feed (retentate)	V_o (dm ³)	13.8	9.63
	V_f (dm ³)	6.8	3.63
Dialyzation water total relative	V_D (dm ³)	21	6
	V_D/V_o (l)	1.52	0.62
Conc. ratio	VCR (l)	2	2.65
Rejection (average)	\bar{R} (l)	0.921	0.928
	\bar{r} (l)	0.251	0.272
Microsolute	c_o/c_f (l)	3.06	1.46
Macrosolute	C_o (g · dm ⁻³)	65.7	65.1
	C_f (g · dm ⁻³)	132.4	161
	C_f/C_o (l)	2.01	2.47
Process duration	t (h)	18.9	10

($V_D/V_o = 1.52$). Undoubtedly, this contributes to rather a high purification degree ($c_o/c_f = 3.06$). In contrast, the VVD-0.5 process starts using a smaller feed quantity (9.63 dm³) and ends up at a higher concentration degree ($C_f/C_o = 2.46$). The process duration is 10 h, because of both the small volume at the beginning and a small volume of fresh water, which is added during the process ($V_D/V_o = 0.62$). Quite expectedly, the purification degree is rather low ($c_o/c_f = 1.46$). The mutual dependence of the concentration degree and purification degree is detailed in Fig. 1.

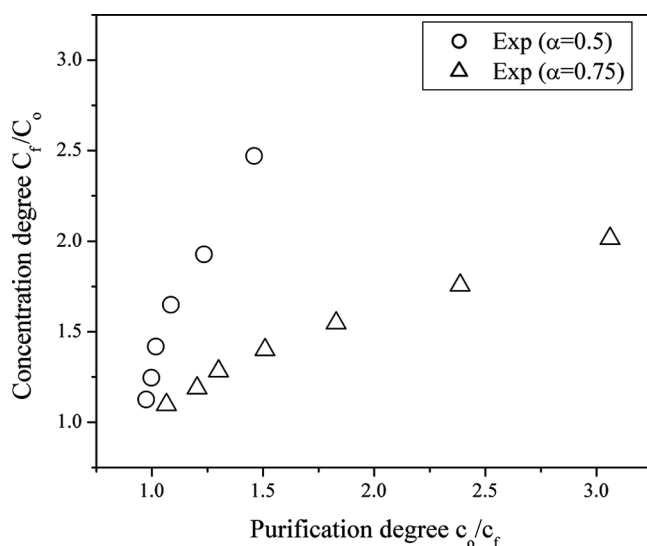


FIG. 1. Concentration degree versus purification degree.

It is evident from the slopes of the two functions in Fig. 1 that the VVD-0.75 is oriented to purification (purification degree changes faster than concentration degree), while VVD-0.5 is better for concentration (concentration degree changes faster than purification degree). For example, at the same concentration degree ($C_f/C_o = 2$), purification is much higher in the VVD-0.75 ($c_o/c_f = 3.06$) than in the VVD-0.5 ($c_o/c_f = 1.25$). On the other hand, at the same purification degree ($c_o/c_f = 1.5$), the VVD-0.5 guarantees a much higher concentration degree ($C_f/C_o = 2.5$) in comparison with the VVD-0.75, which gives almost a two times lower concentration degree ($C_f/C_o = 1.35$).

The average rejections of macrosolutes, given in Table 2, are rather similar, which can be taken as a proof that the rejection depends primarily on the membrane characteristics and only secondarily on working conditions. The same can be concluded regarding the microsolutes rejection. As far as process time is concerned, the VVD-0.75 process, starting with approximately two times larger volume of feed, lasted almost two times longer (18.9 h) than the VVD-0.5 process (10 h). What is more, a lower concentration factor was achieved despite the longer process.

Process Duration

Beside the process time measurements, the duration of each VVD process was calculated using Eq. (9), after defining the inverse fluxes $(JA)^{-1}$ from Eq. (6). For this purpose, eight representative experimental values for the VVD-0.75 and seven values for the VVD-0.5 were chosen (Figs. 2a and 2b). The parameters (p_1 – p_3) were determined by statistically processing the experimental data (Levenberg-Marquardt method, ORIGIN 6.1). Parameter values are given in Fig. 2 together with their statistical errors generated during data processing. Goodness of fit of the suggested correlation, expressed by the coefficient of determination, is very high ($R^2 = 0.989$ for VVD-0.75 and $R^2 = 0.997$ for VVD-0.5).

Before applying the time calculation procedure, a certain correction of the volume decreasing ratio was made. Namely, for the experiments an approximation of a true VVD process was used because diafiltration water is not added in a continuous manner. The process really performed in the experimental set-up consists of consecutive concentration steps. Each of them starts with a partial replenishing of the outgoing permeate with fresh water. When Eq. (3) was applied to the experimentally determined concentration degree, the purification degree as well as macro solutes and micro solutes rejections (from Table 2), the calculated α -values of true VVD processes were obtained (Table 3). The differences between the experimental and calculated α -values are rather small ($\approx 11\%$ for the VVD-0.75 process and $\approx 6\%$ for the VVD-0.5 process). This shows that the performed experiment plan

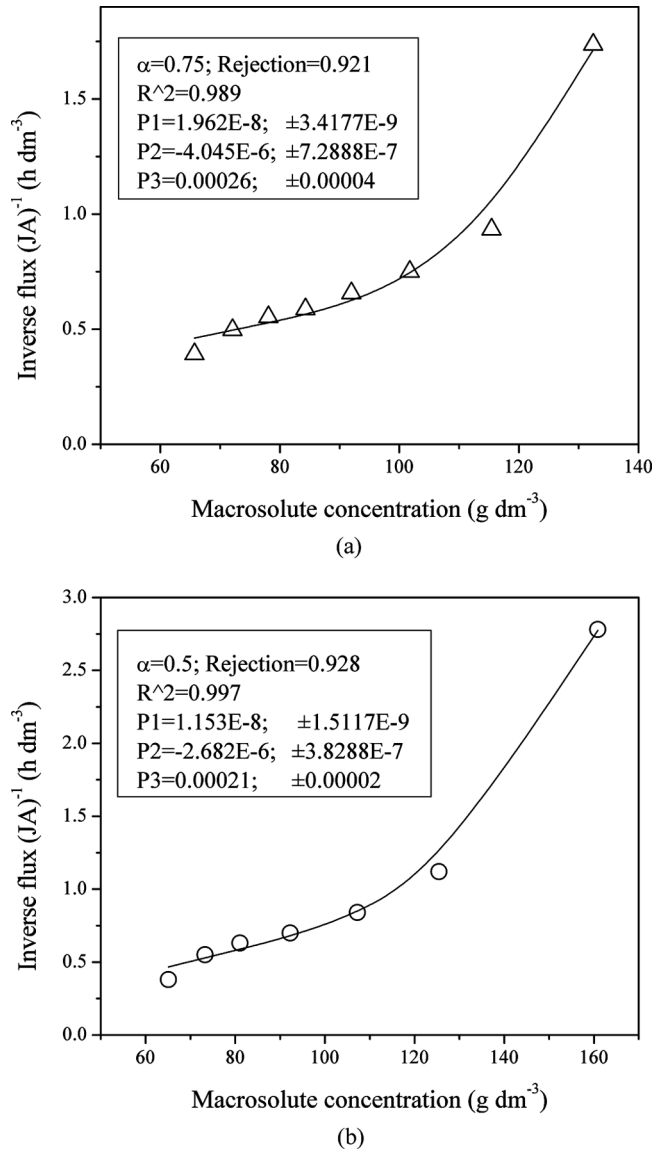


FIG. 2. Experimental and estimated inverse flux values. (a) VVD-0.75 process; (b) VVD-0.5 process.

is a good approximation of a true VVD process. After correcting α -values, the estimates of process time were calculated. Figures 3a and 3b show the comparative values of the calculated and experimentally determined process

durations. It is evident that a very high agreement exists between these sets of data.

While analyzing the duration of the process as a function of purification (Fig. 3a) and the concentration degree (Fig. 3b), one notices that the purification degree of ≈ 1.5 is reached within the same period of time, regardless of which process is used. However, the almost 2.5 times shorter VVD-0.5 process produces a concentration degree of ≈ 2 , compared to the VVD-0.75 process.

Water Consumption

Consumption of fresh water, used during the diafiltration, has a great impact on whether the whole process is

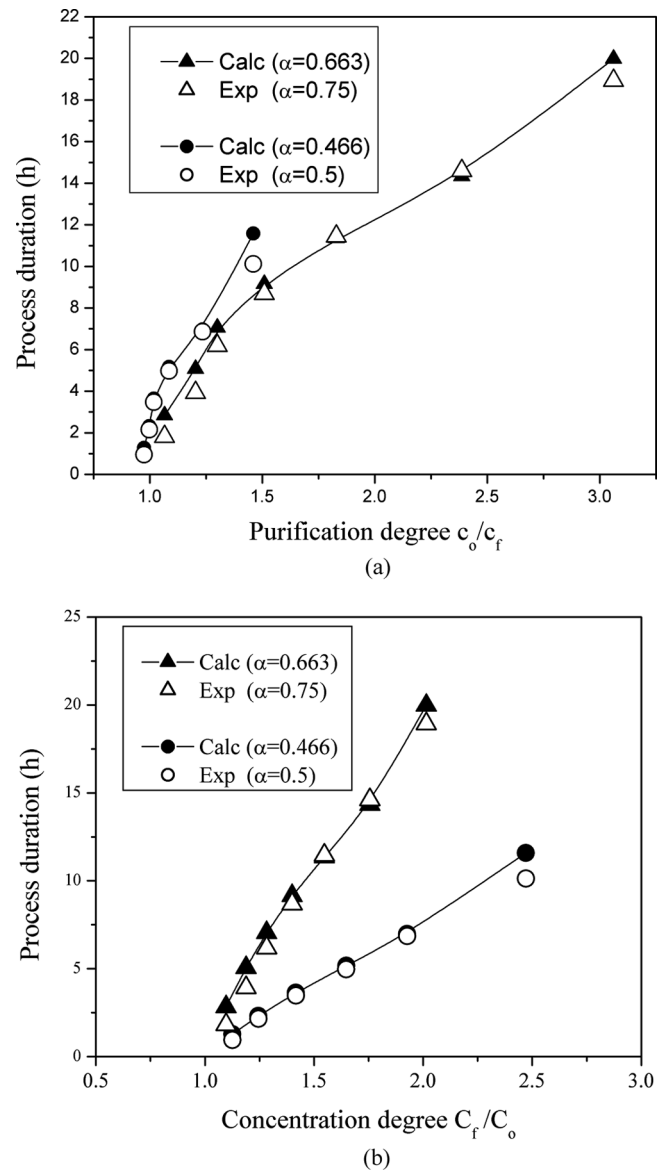


FIG. 3. Process duration as a function of purification degree and concentration degree. (a) VVD-0.75 process; (b) VVD-0.5 process.

TABLE 3

Comparison between experimental and calculated α - values

	Volume decreasing ratio (α)	
Experimental	0.75	0.5
Calculated from Eq. (3)	0.663	0.466

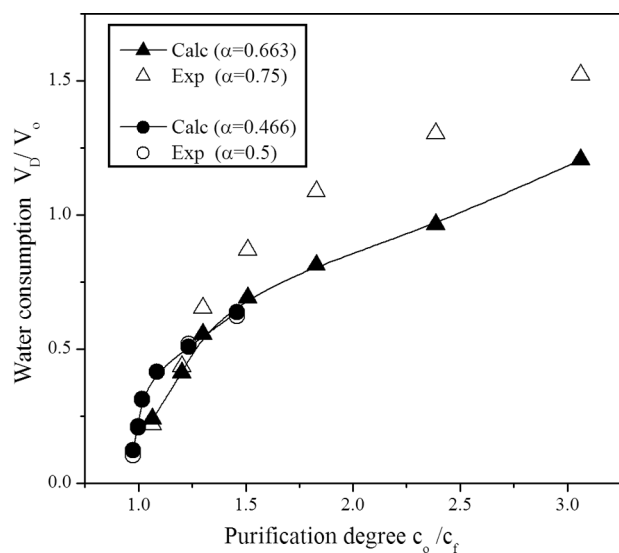


FIG. 4. Consumption of fresh water.

economical. It is a factor that has to be taken into account when considering the application of any separation process. As follows from the experimental data presented in Fig. 4, rather similar water consumption can be observed in the case of both VVD processes at purification degrees not higher than 1.5. However, in the case of the VVD-0.75 process, further improvements of the retentate purity require higher quantities of fresh water.

Apart from the experimentally determined water amounts, the dialyzation water quantity was estimated using the equation (15). The estimates, presented in Fig. 4, are close to the experimental values for both VVD processes as long as one assumes that the permeate flux is constant. However, the differences between the calculated and measured water requirements become greater when such an assumption does not correspond to the real conditions, which is typical of the VVD-0.75 process.

A comparison of two processes of integrated concentration and diafiltration shows that high dilution from the beginning to the end significantly increases water consumption. This conclusion, confirmed by the presented experiments, is in agreement with the suggestion of Jaffrin and Charrier, (14) who have found that the optimal process may consist of a pre-concentration phase where a certain degree of macrosolute concentration is reached, followed by diafiltration with continuously decreasing volume. A similar conclusion was offered by Foley (19), who stated that a significant reduction in water usage can be achieved “without incurring a large time penalty” by performing VVD after an initial concentration step (UF/VVD).

CONCLUSION

Two important and possibly critical factors (process duration and consumption of dialyzation water) were

considered in the case of variable volume diafiltration for partial demineralization and concentration of acid whey. Two processes of simultaneous concentration and diafiltration, one with lower volume decreasing ratio ($\alpha = 0.75$) and one with higher ($\alpha = 0.5$), are compared.

The comparison shows that high dilution from the beginning to the end contributes to higher purification but significantly increases both the process time and the water consumption.

Further investigations are suggested in which the NF concentration could be combined with the VVD process, as a possible optimization of the process as a whole.

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NOMENCLATURE

A	surface of the membrane used (m^2)
c	microsolute ions concentration ($\text{g} \cdot \text{dm}^{-3}$)
C	macrosolutes concentration ($\text{g} \cdot \text{dm}^{-3}$)
g	mass fraction (—)
J	permeate flux ($\text{dm}^3 \cdot \text{m}^{-2} \cdot \text{h}$)
k	number of components
n	number of time-increments
p	parameter in the correlation (6)
Q	flow rate ($\text{dm}^3 \cdot \text{h}$)
r	microsolutes rejection (—)
R	macrosolutes rejection (—)
R^2	coefficient of determination (—)
t	process duration (h)
V	volume (dm^3)

Greek Letters

α	volume decreasing ratio (—)
β	ratio between dialyzation water and initial volume (—)

Subscripts

$1-3$	index of parameters in the correlation (6)
$calc$	calculated
D	dialyzation
exp	experimental
i	i -th component
j	j -th time increment
f	final
o	initial
P	permeate
R	retentate

ABBREVIATIONS

NF	nanofiltration
VVD	variable volume diafiltration
VVD-0.75	variable volume diafiltration with decreasing volume $\alpha = 0.75$
VVD-0.5	variable volume diafiltration with decreasing volume $\alpha = 0.5$
UF/VVD	ultrafiltration followed by the variable volume diafiltration

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