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# OPTIMIZATION OF NANOFILTRATION PROCESS OF LACTIC ACID SOLUTIONS EMPLOYING STATISTICAL EXPERIMENTAL DESIGN

Ultrafiltration of whey is used to obtain whey protein concentrate, for food and feed formulations. However, wide implementation of this separation method for the recovery of proteins is considerably limited by environmental regulations on whey permeate disposal.

One of the possibilities of utilizing whey permeate is to use it for the production of lactic acid by fermentation. Membrane techniques can be employed successfully for this purpose.

The separation process using nanofiltration dynamically formed zirconium(IV) hydrous oxide-polyacrylate membranes can be used for concentration of dilute lactic acid solutions.

The optimization of membrane processes through conventional investigation of the effects of several parameters at the same time is very inefficient and time consuming. In addition, the combined effects of different variables remain unnoticed. In this work, a $2^4$-factorial experimental design was applied to optimize lactic acid rejection by nanofiltration zirconium(IV) hydrous oxide-polyacrylate membrane (Zr(IV)/PAA). Polynomial models were employed for choosing the optimal levels of operating parameters from the point of view of possible application in concentration of dilute solution of lactic acid using nanofiltration process and dynamically formed Zr(IV)/PAA membrane.

## SYMBOLS

abcd – symbols of independent variable at level +1,
$b_r$ – regression coefficients (equation (4a, b)),
DF – dynamically formed membrane,
$I_j$ – variation interval (table 3),
$k$ – number of independent variables (equation (1)),
MF – microfiltration,
$N$ – number of experiments (equation (1)),

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| | |
|---|---|
| $n$ | – number of levels (equation (1)), |
| NF | – nanofiltration, |
| P | – permeate, |
| PAA | – polyacrylate gel-layer, |
| $p$ | – significance level, |
| $p_{cr}$ | – critical significance level, |
| R | – retentate, |
| $R^2$ | – determination coefficient (table 6), |
| $R_M$ | – resistance of clean membrane to filtration process of deionized water, MPa·s/m, |
| $R_{NaNO_3}$ | – rejection of $NaNO_3$ during formation process of dynamic membranes, |
| UF | – ultrafiltration, |
| $x_j$ | – original value of independent variable (equation (6)), |
| $x_{j0}$ | – value of independent variable at level 0 (equation (6)), |
| $x_{jz}$ | – value of coded independent variable (–1, 0, +1) (equation (6)), |
| $x_1$ $(u)$ | – crossflow velocity, m/s, |
| $x_2$ (pH) | – pH of feed solution, |
| $x_3$ $(\Delta p)$ | – pressure difference across membrane, MPa, |
| $x_4$ $(c_{LA})$ | – lactic acid concentration in the feed solution, mol/dm$^3$, |
| $y_1$ $(r)$ | – rejection of lactic acid, |
| $y_2$ $(J_v)$ | – permeate flux, m$^3$/m$^2$s, |
| Zr(IV)/PAA | – nanofiltration zirconium(IV) hydrous oxide-polyacrylate membrane. |

## 1. INTRODUCTION

Dynamically formed (DF) membranes consist of a porous support and one or more gel layers. In comparison with organic membranes, DF membranes have many advantages: high chemical and thermal stability/strength, possibility of *in situ* membrane replacement, long life time, high permeability, simplicity of construction of membrane modules, and only few disadvantages: relatively high construction costs and considerable weight as well as low rate of compaction of membrane modules. Due to the advantages, application of dynamically formed membranes offers various benefits. Use of such membranes in treatment process of wastewater from different industries is attractive owing to both the environmental protection and recovery of valuable products, which results in lower operating costs.

This paper summarizes the results of tests performed on nanofiltration, dynamically formed membranes, with the use of stainless steel support, in solutions containing a low molecular weight solute (lactic acid). The aim of the research was twofold: 1) obtaining data for analysis of separation characteristics of membranes under investigation, and 2) finding optimum level of operating parameters for nanofiltration (NF) process using nanofiltration dynamically formed zirconium(IV) hydrous oxide-polyacrylate membranes (Zr(IV)/PAA).

## 2. DYNAMICALLY FORMED MEMBRANES USED IN THE RESEARCH

The nanofiltration (NF) membranes were formed on a permanent titanium dioxide microfiltration (MF) membrane supported by a porous stainless steel tubular module, 16 mm inside diameter, with an area of 0.029 $m^2$, supplied by Du Pont Separation Systems (Seneca, SC, U.S.A.). Characteristics of gel-layers obtained during formation process of final nanofiltration Zr(IV)/PAA membrane are summarized in table 1.

Table 1

The characteristics of dynamically formed membranes used in the research

| Membrane | Support | Gel-layer | Pore radius*/ layer thickness** | $R_{NaNO3}$ | $R_M$, MPa·s/m |
|---|---|---|---|---|---|
| MF, Ti(IV) | Stainless steel tube | $TiO_2$ | 0.03–0.05 μm* | 0.0 | $0.6\times10^4$ |
| UF, Zr(IV) | MF membrane | $ZrO(OH)_2$ | 10 μm** | 0.1–0.3 | $0.9\times10^5$ |
| NF, Zr(IV)/PAA | UF membrane | PAA | 2 μm** | 0.8–0.9 | $0.3\times10^6$ |

MF, Ti(IV) – permanent, titanium dioxide microfiltration membrane; UF, Zr(IV) – dynamically formed hydrous oxide ultrafiltration membrane; NF, Zr(IV)/PAA – dynamically formed hydrous oxide-polyacrylate nanofiltration membrane; $R_M$ – resistance of clean membrane to filtration process of deionized water [MPa·s/m]; $R_{NaNO_3}$ – rejection of $NaNO_3$ during membrane formation processes.

The Zr(IV)/PAA membranes were prepared in a two-step dynamic formation process. In the first step, a metal hydrous oxide layer was formed in a high pressure (5.5 MPa) filtration process, on an MF membrane, from dilute solution of the zirconium nitrate at pH about 4. In the second step, a dilute solution of poly(acrylic acid) was filtered on the hydrous oxide layer beginning with pH 2 and then increasing it gradually to 7.

## 3. INTEGRATION OF MEMBRANE TECHNIQUES FOR IMPROVEMENT OF WASTE LACTOSE FERMENTATION TO LACTIC ACID

Nowadays, the production of new environmentally-friendly products using raw materials based on renewable sources such as plants or waste seems to be very promising. The waste sugars such as lactose from whey can be secondary raw material for manufacture of ethanol (biofuel) or lactic acid (biodegradable polymers) using fermentation. The distinctive feature of bioprocesses is the necessity separating precisely the desired products from multicomponent and usually much diluted systems. Thus, it is separation that is of fundamental importance for such technology and both membrane and adsorption processes can be used for the purposes of upstream and downstream processing as well as bioreactors may be employed. In upstream processing, raw materials are prepared before being placed in a bioreactor. The membranes are used for preconcentration, purification and sterilization. In the bioreactor, membranes are mainly used for immobilization of microorganisms in reaction space, which is important from economical point of view. Another essential role of these membranes is to remove the product which can enhance the reaction kinetics and shift the constant of reaction equilibrium thus leading to a higher conversion of substrate. In downstream processing, the final product is in multicomponent and diluted fermentation broth. The product must be purified and concentrated to the desired level using appropriate techniques.

A general concept of waste lactose conversion to more valuable products such as ethanol or lactic acid is presented in figure 1.

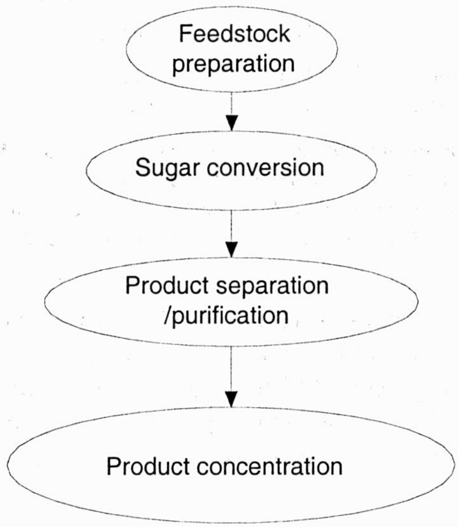


Fig. 1. The main steps in the process of waste lactose conversion to ethanol or lactic acid

There are three main subprocesses in fermentation of sugars: feedstock preparation, sugar conversion, product recovery and concentration which correspond to subsequent steps of the whole hybrid process: upstream processing, bioreactor and downstream processing. The application of environmentally-friendly techniques in each step can lead to a technology of ethanol or lactic acid manufacturing that would be cleaner in terms of environmental impact. A detailed concept of waste lactose conversion to lactic acid is presented in figure 2.

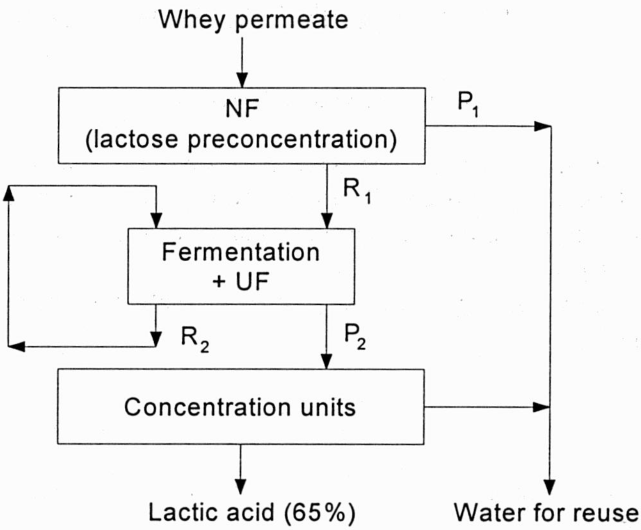


Fig. 2. Scheme of lactic acid production by fermentation of waste lactose (UF – ultrafiltration, NF – nanofiltration, $P_1$ – water for reuse; $R_1$ – preconcentrated lactose; $P_2$ – dilute lactic acid solution; $R_2$ – fermentation broth for reuse)

The possible implementations of selected techniques for improving hybrid processes discussed are presented in table 2.

Table 2

The membrane techniques proposed for improvement of lactic acid manufacturing by fermentation of waste lactose

| General step | Lactic acid manufacturing step | Membrane technique | Implementation goal |
|---|---|---|---|
| Upstream processing | Lactose preconcentration | NF | Increasing lactose concentration with the aim of increasing product concentration in fermentation broth |
| Bioreactor | Bioreactor coupling with membrane module | UF | Continuous removal of product from fermentation broth; recovery of substrate and microorganisms; preliminary purification of product |
| Down-stream processing | Final product concentration | NF | Product concentration at commercial level |

UF – ultrafiltration; NF – nanofiltration.

# 4. EXPERIMENTAL DESIGN

## 4.1. PROBLEM DESCRIPTION

When the aim of the research is the picture of a complete area of investigation, then the factorial design is employed. The main advantages of the factorial design are: great efficiency in estimating the main effects and great scope, since interaction effects may be estimated.

A factorial design involving $k$ factors, each at $n$ levels, will include a total of $N$ treatment combinations:

$$N = n^k , \tag{1}$$

where:

$N$ – number of experiments,

$n$ – number of levels,

$k$ – number of independent variables.

For the membrane system being analyzed we set the number of independent variables $k = 4$ and $n = 2$ levels (+1 and −1), with the resulting number experiments $N = 2^4 = 16$.

The following four independent variables were chosen: cross-flow velocity, $x_1 = u$ [m/s], pH of feed solution, $x_2$ = pH; pressure difference across the membrane, $x_3 = \Delta p$ [MPa] and lactic acid concentration in the feed solution, $x_4 = c_{LA}$ [mol/dm$^3$].

Lactic acid rejection, $r(y_1)$ characterizing membrane selectivity and permeate flux and $J_v(y_2)$ characterizing the efficiency of the process were chosen as the responses.

The effects of independent variables on responses:

$$y_1 = f(x_1, x_2, x_3, x_4), \tag{2}$$

$$y_2 = f(x_1, x_2, x_3, x_4) \tag{3}$$

were determined using the following polynomial models:

$$y_i = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_4x_4 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{14}x_1x_4 + b_{23}x_2x_3 + b_{24}x_2x_4$$
$$+ b_{34}x_3x_4 + b_{123}x_1x_2x_3 + b_{124}x_1x_2x_4 + b_{234}x_2x_3x_4 + b_{1234}x_1x_2x_3x_4 \quad (i = 1, 2). \tag{4a,b}$$

The regression equation coefficients $b_r$ for the functions $y_1$ and $y_2$ were evaluated using equation (5):

$$b_r = \frac{\sum^{N} x_{jz,N}\, y_{i,N}}{N}, \tag{5}$$

where:

$b_r$ – regression coefficient,
$y_{i,N}$ – experimental value of response, $y_1 = r$ and $y_2 = J_v$ (table 5),
$x_{jz,N}$ – coded value of independent variable (equation (6), table 4),
$N$ – number of experiments (equation (1)).

### 4.2. PROBLEM SOLUTION

On the basis of preliminary experiments [1]–[3], the factorial space was designed, as presented in table 3.

Table 3

Factor space for $2^4$-factorial experimental design

| Parameter | Level of parameter | | | Variation interval |
|---|---|---|---|---|
| $x_j$ | –1 | 0 | +1 | $I_j$ |
| $x_1$ [m/s] | 1 | 1.8 | 2.6 | 0.8 |
| $x_2$ [–] | 4 | 6 | 8 | 2 |
| $x_3$ [MPA] | 1.4 | 3.45 | 5.5 | 2.05 |
| $x_4$ [mol/dm$^3$] | 0.02 | 0.5 | 1.0 | 0.41 |

The real values of variables were coded using the equation

$$x_{jz} = \frac{x_j - x_{j0}}{I_j}, \tag{6}$$

where:

$x_{jz}$ – coded value of independent variable (–1, 0, +1),
$x_j$ – original value of independent variable ($j = 1, ..., 4$, table 4),
$x_{j0}$ – variable value at level 0,
$I_j$ – variation interval.

The real values of variables for $2^4$-factorial experimental design of nanofiltration process of lactic acid solutions are presented in table 4. The notation abcd means that all parameters are at level +1.

The results of experiments following the statistical experimental design of $2^4$ type are given in table 5 and figures 3–5.

In table 6, equations (4a) and (4b) represent response surfaces for membrane selectivity $y_1$ and membrane efficiency $y_2$, respectively, where $R^2$ is the coefficient of determination. The regression coefficients presented in table 6 were chosen for significance level $p < p_{cr}$, where $p_{cr} = 0.05$. According to the results presented in table 6, the polynomial models obtained for both membrane selectivity and efficiency fit well the experimental data with $R^2$ at the levels of 0.987 and 0.967, respectively. In table 7, the best calculated response of polynomial model for lactic acid rejection (equation (4a)) is presented with the value of $r = 0.85$.

Table 4

Real values of variables for $2^4$-factorial experimental design of nanofiltration process of lactic acid solutions

| Number of measurement $N$ | Symbol of measurement | $x_1$ velocity $u$ [m/s] | $x_2$ pH | $x_3$ pressure $\Delta p$ [MPa] | $x_4$ concentration $c_{LA}$ [mol/dm$^3$] |
|---|---|---|---|---|---|
| 1 | abcd | 2.6 | 8 | 5.5 | 1.0 |
| 2 | abc | 2.6 | 8 | 5.5 | 0.02 |
| 3 | abd | 2.6 | 8 | 1.4 | 1.0 |
| 4 | ab | 2.6 | 8 | 1.4 | 0.02 |
| 5 | acd | 2.6 | 4 | 5.5 | 1.0 |
| 6 | ac | 2.6 | 4 | 5.5 | 0.02 |
| 7 | ad | 2.6 | 4 | 1.4 | 1.0 |
| 8 | a | 2.6 | 4 | 1.4 | 0.02 |
| 9 | bcd | 1.0 | 8 | 5.5 | 1.0 |
| 10 | bc | 1.0 | 8 | 5.5 | 0.02 |
| 11 | bd | 1.0 | 8 | 1.4 | 1.0 |
| 12 | b | 1.0 | 8 | 1.4 | 0.02 |
| 13 | cd | 1.0 | 4 | 5.5 | 1.0 |
| 14 | c | 1.0 | 4 | 5.5 | 0.02 |
| 15 | d | 1.0 | 4 | 1.4 | 1.0 |
| 16 | (16) | 1.0 | 4 | 1.4 | 0.02 |

Table 5

Experimental values of responses obtained for NF process of lactic acid solutions following the $2^4$-factorial experimental design

| Number of measurement $N$ | Symbol of measurement | Flux $J_v \times 10^5$ [m$^3$/m$^2$s] $y_2$ | Rejection $r$ [–] $y_1$ |
|---|---|---|---|
| 1 | 2 | 3 | 4 |
| 1 | abcd | 2.10 | 0.58 |
| 2 | abc | 1.20 | 0.41 |
| 3 | abd | 1.82 | 0.30 |
| 4 | ab | 3.45 | 0.32 |
| 5 | acd | 2.50 | 0.35 |
| 6 | ac | 5.20 | 0.25 |
| 7 | ad | 1.25 | 0.18 |
| 8 | a | 0.84 | 0.02 |
| 9 | bcd | 0.81 | 0.28 |
| 10 | bc | 0.87 | 0.82 |

| 1 | 2 | 3 | 4 |
|---|---|---|---|
| 11 | bd | 0.17 | 0.12 |
| 12 | b | 0.39 | 0.79 |
| 13 | cd | 1.20 | 0.19 |
| 14 | c | 3.95 | 0.58 |
| 15 | d | 0.22 | 0.09 |
| 16 | (16) | 0.96 | 0.30 |
| 17 | 0 | 0.90 | 0.51 |
| 18 | 0 | 0.85 | 0.48 |
| 19 | 0 | 0.75 | 0.45 |
| 20 | 0 | 0.70 | 0.49 |

The polynomial model for membrane selectivity is presented graphically in figures 3 and 4 as 3-dimentional graphs, where lactic acid rejection is the function of two parameters with other two variables held at –1, 0 and +1 levels.

The effect of pH and lactic acid concentration ($c_{LA}$, mol/dm$^3$) in the feed solutions on lactic acid rejection is presented in figure 3. The rejection of lactic acid increases with an increase in pH and decreases with an increase of lactic acid concentration in the feed solutions.

Table 6

The evaluated regression coefficients $b_r$ for lactic acid rejection $r$ (equation (4a)) and permeate flux $J_v$ (equation (4b))

| $b_r$ | $b_r(y_1)$ (equation (4a)) | $b_r(y_2)\times10^5$ (equation (4b)) |
|---|---|---|
| 0 | 0.35 | 1.7 |
| 1 | –0.05 | 0.0652 |
| 2 | 0.104 | 0.0652 |
| 3 | 0.08 | 0.0652 |
| 4 | –0.073 | 0.05 |
| 1,2 | 0.11 | – |
| 1,3 | – | – |
| 1,4 | 0.12 | – |
| 2,3 | – | 0.0652 |
| 2,4 | –0.04 | – |
| 3,4 | – | 0.34 |
| 1,2,3 | – | – |
| 1,2,4 | 0.03 | – |
| 2,3,4 | 0.03 | 0.055 |
| 1,2,3,4 | – | – |
| $R^2$ | 0.987 | 0.967 |

The effect of pressure difference across the membrane ($\Delta p$, MPa) and crossflow velocity ($u$, m/s) on lactic acid rejection is given in figure 4. Pressure difference and crossflow velocity have positive and negative effect on the lactic acid rejection, respectively, within the experimental area.

Table 7

The best response of polynomial model for lactic acid rejection, $y_1 = r$ (equation (4a))

| $b_0$ | $b_1x_1$ | $b_2x_2$ | $b_3x_3$ | $b_4x_4$ | $b_{14}x_1x_4$ | $b_{24}x_2x_4$ | $b_{124}x_1x_2x_4$ | $b_{234}x_2x_3x_4$ | $y_1$ |
|---|---|---|---|---|---|---|---|---|---|
| 0.35 | 0.03 | 0.14 | 0.09 | 0.08 | 0.084 | 0.06 | 0.03 | –0.05 | 0.85 |

The best value of lactic acid rejection at a level of 0.85 was obtained for the following independent variables:

$$x_1(u) = 1.3 \text{ [m/s]},$$

$$x_2(\text{pH}) = 8.6 \text{ [–]},$$

$$x_3(\Delta p) = 5.65 \text{ [MPa]},$$

$$x_4(c_{\text{LA}}) = 0.04 \text{ [mol/dm}^3\text{]}.$$

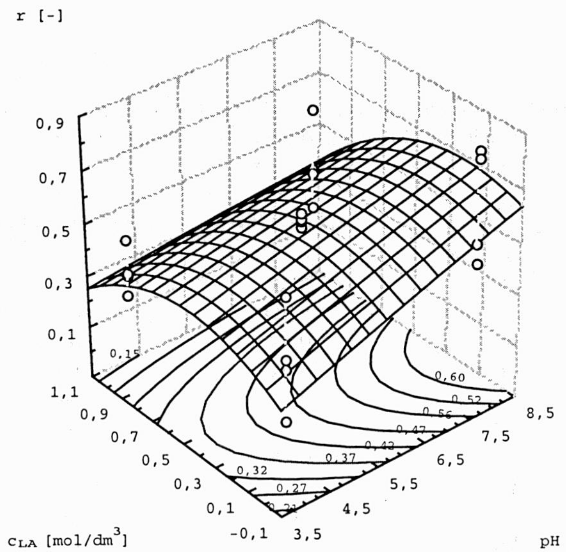


Fig. 3. Lactic acid rejection (o – experimental results from table 5) as a function of pH and lactic acid concentration $c_{\text{LA}}$ [mol/dm$^3$] for $u = 1$ m/s and $\Delta p = 5.5$ MPa

The strong positive effect of pH on lactic acid rejection was observed, with regression coefficient $b_2 = 0.104$ (table 6). Positive but less substantial effect of pressure difference across the membrane on membrane selectivity was also observed, with

$b_3 = 0.08$. The next two variables, crossflow velocity and lactic acid concentration in the feed solutions, have negative effect on lactic acid rejection, with regression coefficients $b_1 = -0.05$ and $b_4 = -0.073$.

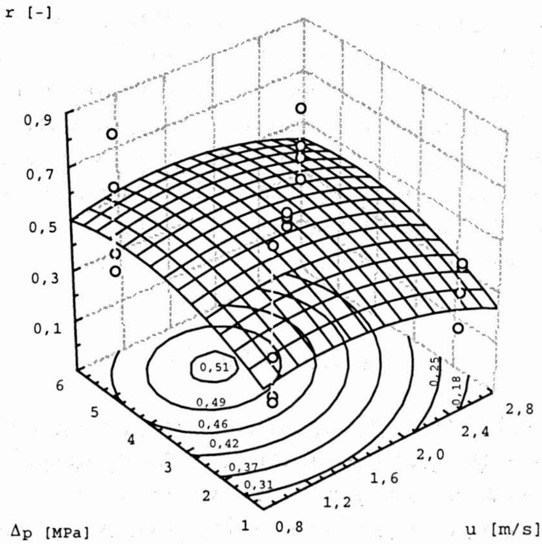


Fig. 4. Lactic acid rejection (o – experimental results from table 5) as the function of pressure difference across the membrane $\Delta p$ [MPa] and crossflow velocity $u$ [m/s] for $c_{LA} = 0.02$ mol/dm$^3$ and pH = 8.0

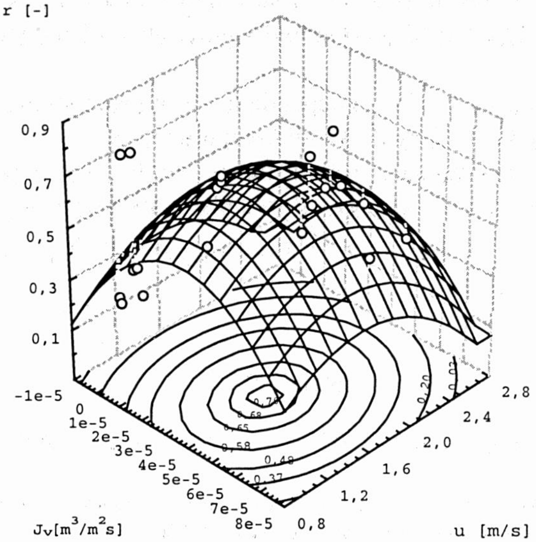


Fig. 5. Interdependence of lactic acid rejection $r$ and permeate flux $J_v$ [m$^3$/m$^2$s] as a function of crossflow velocity $u$ [m/s] for pH = 8, $\Delta p$ =1.4 MPa and $c_{LA} = 0.02$ mol/dm$^3$ (o – experimental data)

The visualization of interdependence of lactic acid rejection and permeate flux as a function of crossflow velocity is shown in figure 5. The crossflow velocity has opposite effect on permeate flux ($b_2$ = 0.065, table 6) and lactic acid rejection ($b_2$ = –0.05, table 6). The optimization consists in finding the highest level of both dependent variables. In such a case, lactic acid rejection will be lower than 0.85. From practical point of view it is better to choose the highest level of lactic acid rejection. The essential level of flux can be easily reached by increasing the membrane area.

In the present work, the lactic acid rejection varied from 0.02 (table 5, measurement 8) to 0.82 (table 5, measurement 10) depending on the operating parameters. The highest membrane selectivity expressed by lactic acid rejection was computed with the use of polynomial model at a level of 0.85. The measurement 10 (table 5) is experimental confirmation of this result.

## 5. CONCLUSIONS

Statistical orthogonal $2^4$-factorial experimental design with four replications of the independent variables at the center point was used for optimization of nanofiltration process of lactic acid solutions using Zr(IV)/PAA membranes. This led to a total of 20 experiments, 16 experiments for two levels of independent variables (–1 and +1) and 4 experiments for level 0. Polynomial models of the form of $y_i = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_4x_4 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{14}x_1x_4 + b_{23}x_2x_3 + b_{24}x_2x_4 + b_{34}x_3x_4 + b_{123}x_1x_2x_3 + b_{124}x_1x_2x_4 + b_{234}x_2x_3x_4 + b_{1234}x_1x_2x_3x_4$ were obtained. The regression equations correlating rejection and permeate flux with four independent variables provide:

– analysis of the effects of direction and significance of independent variables on responses,

– evaluation of optimal level of independent variables enabling achievement of the highest rejection of lactic acid by Zr(IV)/PAA membrane.

The data obtained are the basis for further research using experimental design of higher order with response of polynomial models of the form of $y_i = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_4x_4 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{14}x_1x_4 + b_{23}x_2x_3 + b_{24}x_2x_4 + b_{34}x_3x_4 + b_{11}x_1^2 + b_{22}x_2^2 + b_{33}x_3^2 + b_{44}x_4^2$.

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## OPTYMALIZACJA PROCESU NANOFILTRACJI ROZTWORÓW KWASU MLEKOWEGO Z ZASTOSOWANIEM METODY PLANOWANEGO EKSPERYMENTU

Obecnie systematycznie zaostrza się te kryteria ochrony środowiska, które mają uniemożliwić odprowadzanie serwatki, odpadu z przemysłu mleczarskiego, bezpośrednio do środowiska. Serwatka jako ściek jest bardzo uciążliwa; z drugiej jednak strony może być traktowana jako źródło wielu cennych składników, m.in. laktozy, z której można w wyniku fermentacji otrzymać kwas mlekowy.

Istnieją dwie główne metody biochemicznej produkcji kwasu mlekowego: fermentacja cukru w warunkach albo ustalonego, albo zmiennego pH. W warunkach zmiennego pH kwas mlekowy jest selektywnie wydzielany z roztworu fermentacyjnego dzięki zastosowaniu odpowiedniego procesu membranowego. Otrzymuje się wtedy rozcieńczone roztwory kwasu mlekowego (1–2%). Nanofiltracja przez membrany cyrkonowo-poliakrylowe formowane dynamicznie może być stosowana zarówno do wydzielania rozcieńczonego kwasu mlekowego w procesie fermentacji, jak i do jego koncentracji.

Optymalizacja procesów membranowych dzięki użyciu tradycyjnych badań wpływu kilku parametrów po kolei jest bardzo czasochłonna. Ponadto łączny wpływ różnych zmiennych pozostaje niezauważony. W tej pracy do optymalizacji procesu nanofiltracji roztworów kwasu mlekowego został zastosowany czynnikowy eksperyment planowany typu $2^4$. Otrzymane modele wielomianowe wykorzystano do wybrania optymalnych poziomów parametrów, biorąc pod uwagę ich przydatność do zagęszczania rozcieńczonych roztworów kwasu mlekowego z użyciem procesu nanofiltracji i dynamicznie uformowanej membrany Zr(IV)/PAA.