# Modelling the microfiltration of lactic acid fermentation broths and comparison of operating modes

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**Abstract**

This paper deals with the first unit operation of the downstream process for the production of lactic acid: the clarification of fermentation broths by cross-flow microfiltration. Microfiltration experiments conducted under constant transmembrane pressure and under constant permeate fluxes (higher and lower than the critical flux) were represented by the resistance in series model in which the membrane resistance, the adsorption resistance, the bacteria cake resistance and the soluble compounds concentration polarisation resistance were taken into account. The different operating modes were compared in terms of two industrial interest criteria: the productivity and fouling rates. Higher productivities were obtained during constant transmembrane pressure runs whereas the lowest fouling rate was observed during the run conducted with a constant permeate flux lower than the critical flux. However, this fouling was mainly due to adsorption and solute components concentration polarisation.

*Key words:* Microfiltration; Fermentation broths; Productivity; Fouling rate; Constant pressure; Constant flux

## 1. Introduction

Lactic acid and lactates produced by fermentation are increasingly used as natural grade food additives [1] (acidulants, preservatives and flavour enhancers) and as the monomer for biodegradable polymers synthesis [2]. However, the production of lactic acid from fermentation requires the use of an efficient and economic downstream process to recover lactic acid and to isolate it from various impurities present in the fermentation broth [3]. The first step of this separation process is the fermentation broth clarification, it is achieved with filter

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aids in the traditional process. In order to reduce the environmental impact, the fermentation broth clarification by cross-flow micro-filtration was investigated. The aim of this paper is to compare constant transmembrane pressure and constant permeate flow operating modes for batch micro-filtration. The comparison was made in terms of productivity and fouling rates which are criteria of industrial interest. In order to evaluate the contribution of different fouling phenomena, microfiltration operation was modelled by the resistance in series law.

## 2. Material and methods

The lactic acid was produced by fermentation of beet molasses, supplemented with yeast extracts and inoculated with freeze dried *lactobacillus delbrueckii* sp. *lactis*. The pH was regulated at 6.2 by introduction of ammonium hydroxyde. The fermentation broth thus obtained contained 2.6 ± 0.3 g/L of microorganisms; 90 ± 15 g/L of ammonium lactate and 14 ± 9 g/L of sucrose. However, as some broths were reconstituted after clarification by mixing the retentate and the permeate, the initial bacteria concentration could vary. Nevertheless, it has been shown [4] that permeate flux is very slightly dependent on bacteria concentration.

The clarification of the ammonium lactate broth was run on a 0.16 $m^2$ batch filtration rig equipped with a ceramic tubular membrane (B01 BX, Kerasep from Orelis, France). The pilot unit scheme is shown [5]. The nominal pore size was 0.1 μm. Retention of bacteria was complete, while both ammonium lactate and sucrose were fully transmitted to the permeate. Temperature was regulated at 48°C. Cross-flow velocity was 4 m/s. Under these conditions, the critical flux was found to be equal to 50 L $h^{-1}$ $m^{-2}$ [5]. Different runs were carried at constant transmembrane pressure and at constant permeate flowrate with flowrates higher and lower than the critical flux.

## 3. Model development

The standard hydraulic resistance-in-series model has already been applied to represent such processes taking into account the membrane resistance *Rm*, the cake resistance *Rc* and the adsorption resistance *Ra* [6–7]. In the present work, the solute compounds concentration polarisation resistance is included into the model:

$$J = \frac{TMP}{\mu_p \left(Rm + Ra + Rp + Rc\right)} \tag{1}$$

Assuming their additivity, each resistance was independently determined. Membrane resistance was obtained from water flux measurements. The sum of the adsorption and solute compounds concentration polarisation resistances was determined during filtration runs of clarified fermentation broths. It was found to be time dependant. The steady state adsorption resistance $Ra_{ss}$ was measured after immersion of the membrane in the fermented broth:

$$Ra + Rp = \left(Ra_{ss} + Rp_{ss}\right)\left(1 - \exp(-b\,t)\right) \tag{2}$$

The bacteria cake resistance *Rc* was expressed by the relation:

$$Rc = \frac{m}{A}\,\alpha\, TMP^{n} \tag{3}$$

The compressibility index $n$ was measured from frontal filtration experiments. The bacteria mass in the cake per surface unit ($m/A$) was calculated from the mass balance:

$$\frac{dm}{dt} = \left(JC - \frac{D}{\delta}(C_w - C)\right)A \tag{4}$$

As the bulk concentration $C$ is much lower than the wall concentration $C_w$, it can be neglected. The diffusion term including the diffusion coefficient, the membrane wall concentration and the cake layer thickness ($DC_w/\delta$) was calculated from the steady state permeate flux $J_{ss}$ of constant concentration runs:

$$\frac{D}{\delta}C_w = J_{ss}C \tag{5}$$

Finally, assuming complete retention of cells the variation in retentate bacterial concentration in a batch system was calculated from:

$$C = \frac{C_0 V_0}{V_0 - A \int_0^t J \, dt} \tag{6}$$

The system of Eqs. (1–3) and (5–6) was solved using Matlab software, using an iterative method. The least square method (Leven-Berg-Marquardt algorithm) was used to deter-mine the cake resistance coefficient α. It was determined from constant pressure cross-flow filtration runs by fitting the modelled permeate flux to experimental data as shown in Fig. 1, the mean errors being lower than 10%. The values of each parameter are given [4].

This model was validated with constant permeate flux clarification runs, Fig. 2. Differ-ent transmembrane pressure profiles are observ-ed according to the set value of the permeate flux. For permeate fluxes below the critical flux, transmembrane pressure remained almost constant at a low value during the entire clari-fication run. The value of critical flux was thus valid for bacteria concentrations up to 11 g/L. When permeate fluxes exceeded the critical value, the transmembrane pressure increased continuously during the run. This increase was faster with the higher permeate flux value. The shapes of the transmembrane pressure variation with time were quite accurately predicted in both cases of runs with permeate flux lower and higher than the critical flux with a mean error lower than 32%.

The resistance in-series-model made it possible to calculate the contribution of each phenomena (bacteria deposition, solutes con-centration polarization and adsorption) to mass transfer resistance and for different operating modes, Fig. 3. In all cases, the resistances due to solutes concentration polarization and ad-sorption dominated. Moreover, we can observe that during the constant flux run with flux lower

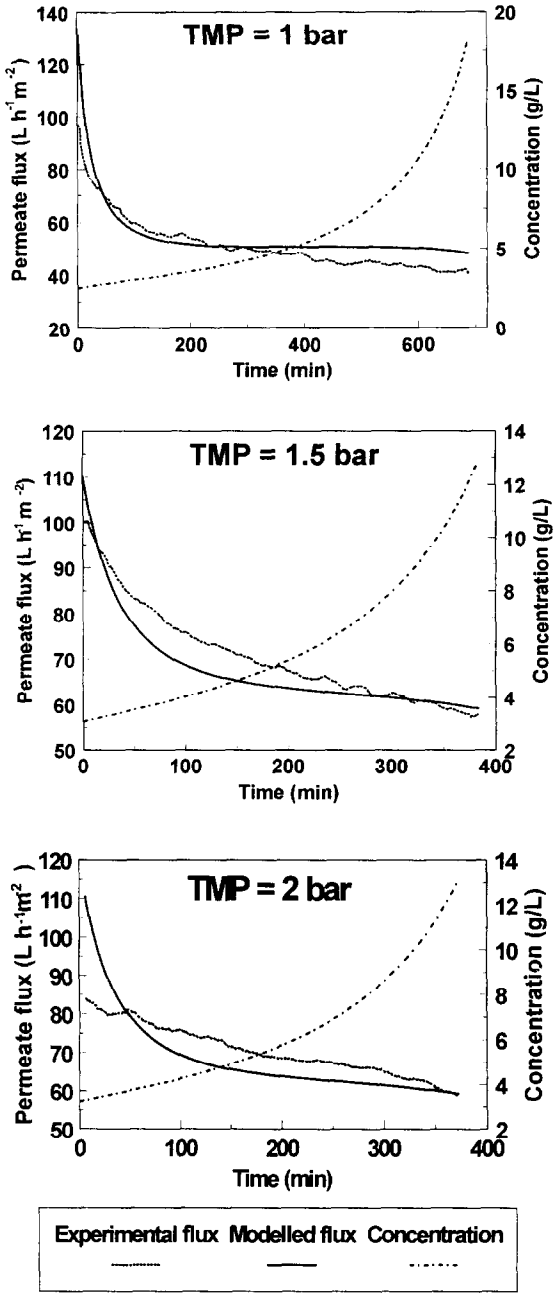


Fig. 1. Constant pressure cross-flow filtration runs. De-termination of parmeter α by fitting modelled permeate flux to experimental permeate flux. TMP = 1 bar, model mean error: 4.9%; TMP = 1.5 bar, model mean error: 9.6%; TMP = 2 bar, model mean error: 8.6%.

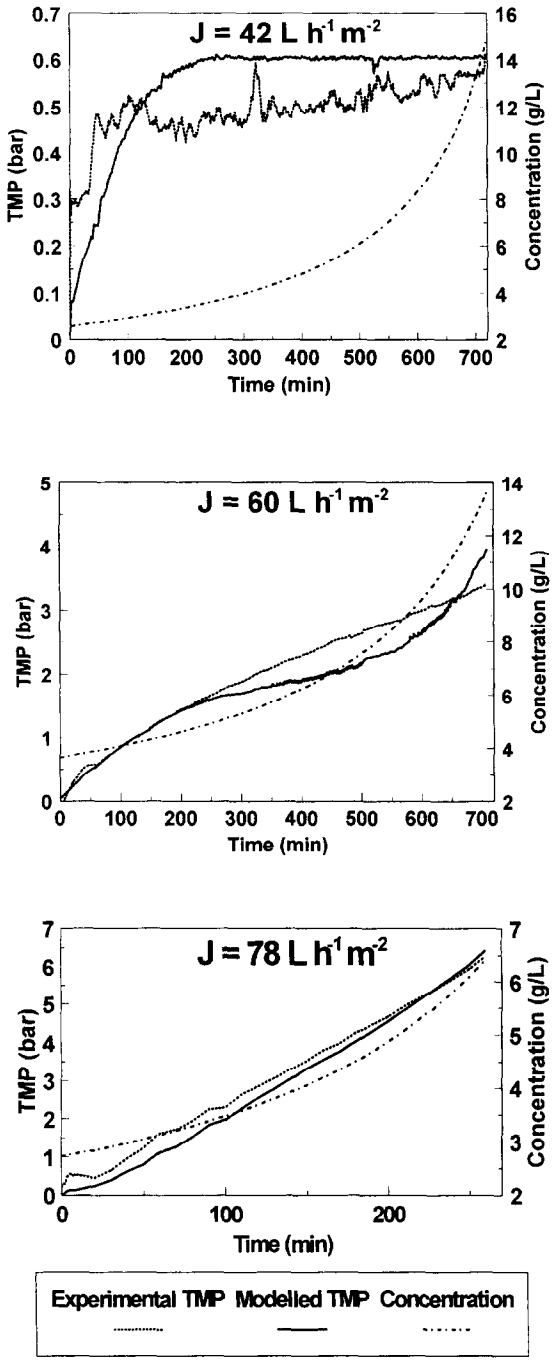


Fig. 2. Constant flux cross-flow filtration runs. Model validation. J = 42 L $h^{-1}$ $m^{-2}$, model mean error: 20%; J = 60 L $h^{-1}$ $m^{-2}$, model mean error: 14%; J = 78 L $h^{-1}$ $m^{-2}$, model mean error: 32%.

than the critical value, the cake resistance was negligible: there was no deposition of bacterial cells at the membrane wall [8].

## 4. Comparison of operating modes

The comparison of different operating modes was made on the base of filtration runs which the objective was the production of 70 L of permeate and the initial volume of the fermentation broth was about 100 L. The produced permeate volumes vs. time are shown in Fig. 4. The expected 70 L production could not be reached during the run with a permeate flux very higher (78 L $h^{-1}m^{-2}$) than the critical flux because the fouling rate, and consequently the transmembrane pressure, increased very quickly, limiting the filtration time.

Obviously, the relationship between permeate volume and time was linear for the constant permeate flux runs. Higher production or productivities (permeate volume divided by time, Table 1) were obtained during the constant transmembrane pressure runs. The lowest productivity was observed during the run conducted with a constant permeate flux lower than the critical flux. This productivity value was about twice as low as those obtained during the constant transmembrane pressure runs. Nevertheless, it is worth noting that these productivity values depend on the starting procedure of the unit.

Fig. 5 makes it possible to compare the total resistances to mass transfer as a function of the produced permeate volume. Different behaviors can be observed. For constant transmembrane pressure runs, total hydraulic resistance was high from the start of experiment and increased very moderately. For the constant flux runs with fluxes higher than the critical value, it increased progressively during the runs, becoming higher than the constant transmembrane pressure resistances. For the constant flux run with flux lower than the critical value, fouling rate was very low and almost constant. However, the resistance in series model showed that

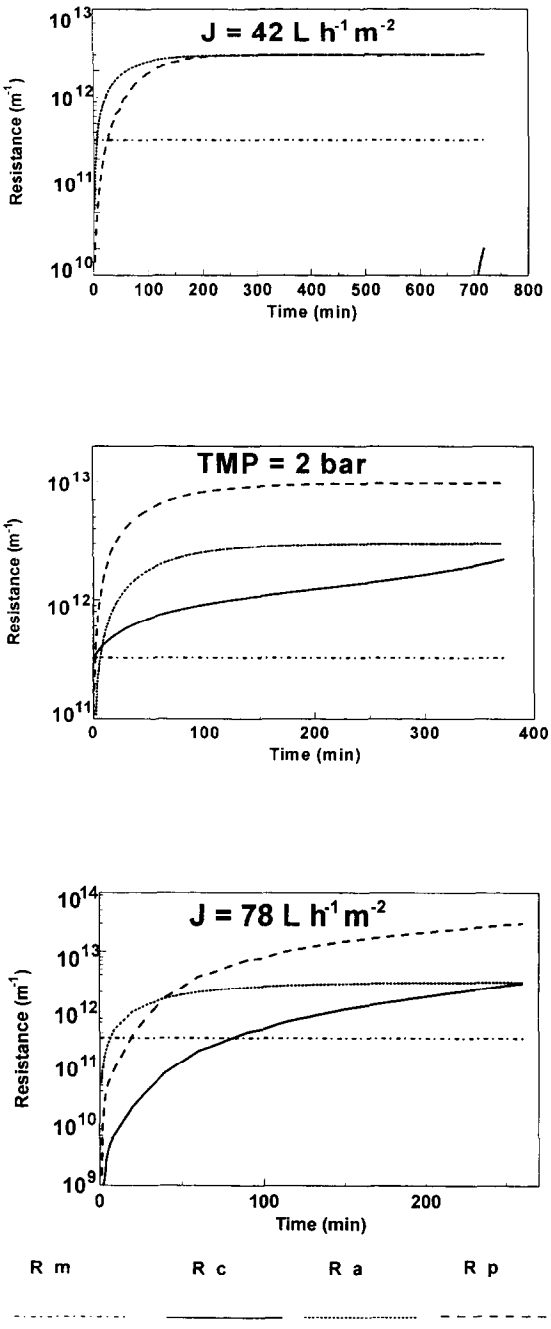


Fig. 3. Modelled hydraulic resistances for different operating modes.

Table 1
Productivity values of constant transmembrane pressure and constant permeate flux

| Run | Time required to obtain 70 L of permeate, min | Mean productivity, L/h |
|---|---|---|
| TMP=1.5 bar | 366 | 11.5 |
| TMP =2 bar | 359 | 11.7 |
| J=42 L $h^{-1}$ $m^{-2}$ | 670 | 6.3 |
| J=60 L $h^{-1}$ $m^{-2}$ | 440 | 9.5 |
| J=78 L $h^{-1}$ $m^{-2}$ | — | 11.2* |

* calculated for 49 L permeate vol. instead of 70 L for other values

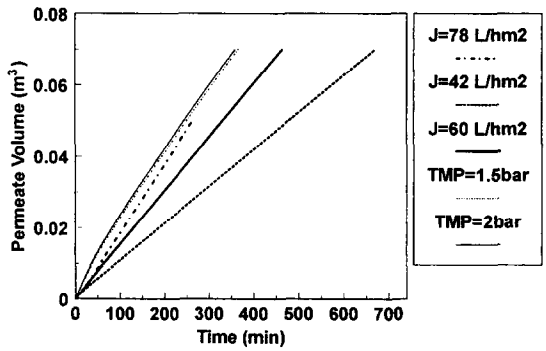


Fig. 4. Production obtained using different modes of cross-flow filtration of lactic acid fermentation broths.

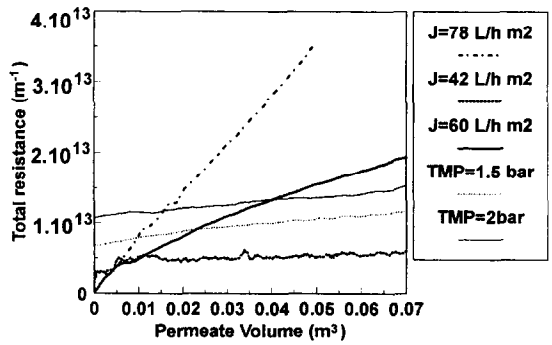


Fig. 5. Total resistance to mass transfer observed using different modes of cross-flow filtration of lactic acid fermentation broths.

this fouling was mainly due to solute components concentration polarisation and adsorption phenomena, see Fig. 3. Fouling by adsorption is the most difficult to remove and cleaning procedures are the same whatever the operating mode.

Considering the productivities and the fouling rates, it is thus preferable to operate the batch microfiltration of the lactic acid fermentation broth under constant transmembrane pressure. However, the transmembrane pressure should be higher than 0.5 bar in order to be under limiting flux conditions [5].

## 5. Conclusion

The clarification of the lactic acid fermentation broth was modelled by the resistance in series law in which the membrane resistance, the adsorption resistance, the bacteria cake resistance and the soluble compounds concentration polarisation resistance were taken into account. In all cases, the resistances due to adsorption and solutes concentration polarization dominated. We found that constant transmembrane pressure mode was preferable for the batch microfiltration, unless the clarification is coupled to another unit operation which requires a constant feed flow rate.

## Symbols

$A$ — Membrane area, $m^2$
$b$ — Constant in equation 2, $s^{-1}$
$C$ — Bacteria concentration in the retentate, $kg.m^{-3}$
$Cw$ — Bacteria concentration on the membrane wall, $kg.m^{-3}$
$D$ — Shear induced diffusion coefficient, $m^2/s$
$J$ — Permeate flux, $m\ s^{-1}$ or $l\ h^{-1}\ m^{-2}$
$m$ — Weight of bacteria in the cake, kg
$Ra$ — Adsorption resistance, $m^{-1}$
$Rc$ — Cake resistance, $m^{-1}$
$Rm$ — Membrane resistance, $m^{-1}$
$Rp$ — Solute concentration polarisation resistance, $m^{-1}$
$t$ — Time, s
$TMP$ — Transmembrane pressure, Pa
$V$ — Retentate volume, $m^3$

*Greek*

$\alpha$ — Cake resistance coefficient, $m\ kg^{-1}\ Pa^{-0.63}$
$\delta$ — Cake thickness, m
$\mu_p$ — Permeate dynamic viscosity, Pa s

*Subscripts*

$ss$ — Steady state
0 — Initial


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## References


[1] T.B. Vick Roy, Lactic acid, in: M. Moo-Young (Ed.), Comprehensive Biotechnology, Vol. 3, Pergamon Press, Oxford, 1985.
[2] R.L. Evangelista and Z.L. Nikolov, Appl. Biochem. Biotechnol., 57/58 (1996) 471–480.
[3] A.S. Kertes and C.J. King, Biotech. Bioeng., 28 (1986) 269–282.
[4] H. Carrère, F. Blaszkow and H. Roux de Balmann, J. Membr. Sci., 186 (2001) 219–230.
[5] S. Milcent and H. Carrère, Separation and Purification Technology, 22/23 (2001) 393–401.
[6] Y. Shimizu, K.I. Shimodera and A. Watanabe, J. Fermentation Bioeng., 76 (1993) 493–500.
[7] T. Tanaka, K. Usui, K. Kouda, K. Nakanishi, J. Chem. Eng. Japan, 29 (1996) 973–981.
[8] R.W. Field, D. Wu, J.A. Howell, B.B. Gupta, J. Membr. Sci., 100 (1995) 259–272.