The Use of Flux Enhancement Methods for High Flux Cross-flow Membrane Microfiltration Systems

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The influence of the two techniques (membrane backflushing and two-phase gas-liquid flow) on permeate flux during the microfiltration of model dispersions on ceramic membrane has been studied.

The experiments were carried out with aqueous titania dispersions filtered through a ceramic tubular membrane. The influence of various transmembrane pressures, feed concentrations, backflushing duration and frequencies, liquid and gas flow velocities, and the influence of periodic gas flow is also presented.

Based on the results of experiments presented in this work, it appears that constant gas-liquid flow has a positive influence on permeate flux. The level of flux enhancement depends both on the gas flow rate and flow pattern. From analysis of experimental results it may be concluded that two-phase flow seems to expand the particle cake as it increases, both, cake porosity and thickness, thus allowing higher fluxes. The enhancement of flux was better observed during the microfiltration of more concentrated dispersions. Periodic gas flow is not so significant for the flux enhancement and strongly depends on the periodic gas flow mode and on the concentration of the dispersion.

This experimental study demonstrated that in cross-flow microfiltration of dispersions, the membrane backflushing could maintain permeate flux at a constant and high level over the duration of an experiment. It was observed that the effect of backflushing on flux enhancement was more pronounced when the backpulse duration was shorter, the transmembrane pressure difference was higher, and the retentate velocity was lower in forward filtration.

Keywords:
Microfiltration; ceramic membrane; backflushing; two-phase gas-liquid flow

Introduction

The membrane filtration processes are currently mostly used in the production of ultrapure water, the processing of food and dairy products, the recovery of electrodeposition paints, the treatment of oil and latex emulsions and in biotechnology oriented applications such as fractionation of fermentation broths and high performance reactors for enzymatic and fermentation processes. However, the present membrane processes for liquid feed streams are complicated by the phenomena of membrane fouling and of concentration polarisation in the liquid boundary layer adjacent to the membrane wall.

Concentration polarisation occurs when a concentration gradient of the retained components is formed on or near the membrane surface. Fouling is the deposition of material on the membrane surface or in its pores, leading to a change in membrane behaviour or even pluggage. These phenomena manifest themselves as such that with time and increased operating pressure, the permeate flux reaches an asymptotic value beyond which further increases in operating time and pressure do not result in increased flux. The severity of the effects of these phenomena varies with the membrane type and the composition of the process stream.

Concentration polarisation is a function of the hydrodynamic conditions in the membrane system. Membrane fouling is usually characterised as irreversible; however, when cross-flow systems are used, the imposed stress of the cross-flow tends to shear the fouling layer. Hence, varying the fluid mechanics of a system is very important in maximising the total capacity of a membrane module. In the past, a number of investigators attempted the manipulation of fluid hydrodynamics or the membrane surface morphology to enhance permeate flux.

There are at least three possible approaches to reduce or control concentration polarisation and fouling:

1) Changes in surface characteristics of the membrane,

2) pre-treatment of the feed and,

3) fluid management methods.

In Ref.[1], a morphological analysis of means of reducing concentration polarisation and fouling is presented. Of the various methods mentioned in Ref.[1], hydrodynamic or fluid management techniques have proved to be quite effective and economical in reducing concentration polarisation and fouling. Recently some studies have pointed out the interest in the area of use of gas-liquid two-phase flow technique in the concentrate stream during ultrafiltration in order to enhance the flux for different applications (biological treatment, drinking water production, macromolecules separation) and different membrane geometries (hollow fibre, flat sheet or tubular).

Application of gas-liquid two-phase flow for microfiltration intensification is based on change of hydrodynamic conditions inside the microfiltration module which positively increase the wall shear stress, preventing the membrane fouling and enhancing the mass transfer of separated compound (solvent, the most frequently water).

Cui and co-workers[2–4] have shown that air sparging can reduce the concentration polarisation in ultrafiltration of macromolecules (dextran, dyed dextran and bovine serum albumin), for flat sheet modules and hollow fibre membranes. The explanation given for the flux enhancement is that air sparging into the liquid stream increases turbulence near the membrane surface, as well as the cross-flow velocity, thus limiting the boundary layer thickness.

Mercier and co-workers[5,6] obtained significant flux enhancement (200 % of flux increase) by air sparging in ultrafiltration tubular inorganic membranes with two kinds of suspension (bentonite and yeast).

Cabassud and co-workers[7,8] have presented results concerning two-phase gas-liquid flow for particle suspensions (clay suspensions) inside hollow fibres. In that case, flux improvement was linked to hydrodynamic control of the particle deposition on the membrane. Significant increases in permeate flux have been observed, even at a very low air velocity, and for all the concentrations studied. The air injection process led to an increase of flux up to 155 % for specific conditions. However, good results have been obtained for very low air velocities (under 0.2 m s^{-1}).

Lee and co-workers[9] used air slugs entrapped in cross-flow stream to prevent the flux decline during filtration of bacterial cell suspensions. Ultrafiltration and microfiltration flat sheet membranes have been used and the best performances were obtained for the ultrafiltration (maximum enhancement of 200 % is reported).

The membrane backflushing process is carried out by periodic by reversing the direction of permeate flow (in cycles). This is achieved by applying pressure pulses on the permeate side of the membrane, often with the help of an automatic time switch or a microprocessor. Clear liquid is then forced in reverse direction through the membrane, thereby lifting off the boundary layer and washing it out of the membrane surface. Although backflushing gives a loss of permeate to the feed stream, it decreases the effective operating time and it was reported in literature that the average flux per cycle may be much higher than the steady flux at longer time, when the membrane backflushing is not used[10,11,13–15,19].

Initial research showed mainly that backflushing reduces long-term membrane fouling and that the backflush duration of 5 – 20 seconds and pulses of 3 – 10 times per hour were recommended[11]. Further studies showed that frequent transmembrane pressure pulsing could also reduce concentration polarisation resistance, and shorter backflush intervals were suggested where backflush duration was 1–5 s with frequencies of 1–10 times per minute (backpulsing technique)[12–15]. *Wenten*[16] even carried out experiments in which pulse is done every few (1–5) seconds with a backflush time less than 0.1 second (backshock technique).

The objective of this paper is to report the comparison of the gas-liquid two-phase flow and membrane backflushing for flux enhancement during the microfiltration of a model dispersion on tubular ceramic membranes.

Experimental

Membranes

The membranes used in our two-phase gas-liquid flow experiments were asymmetric, multi-layered, ceramic membranes (Terronic a.s., Hradec Králové, Czech Republic). They were configured as single cylindrical tubes 0.1 m long, 6 mm ID and 10 mm OD, consisting of a thin alumina layer deposited on the internal surface of the alumina support. The microfiltration membranes used in our experiments had an average diameter of 91 nm. The pore size distribution of this membrane was determined by the liquid displacement method.[17]

The membranes used for membrane backflushing experiments were obtained from Membralox, SCT Bazet, France. They were configured as single cylindrical tubes 0.25 m long, 7 mm inner diameter, consisting of a thin zirconia layer deposited on the internal surface of the tubular α-alumina support. The average pore diameter of the active layer reported by the membrane producer equaled to 100 nm.

Feeds

The microfiltration experiments were performed with an aqueous dispersion of titanium dioxide (Versanyl B-K7020), obtained from Ostacolor a.s., Pardubice, Czech Republic. The mean diameter of particles was 443 nm, however, the

distribution of particles was very wide (from 210 nm to 850 nm). Concentration of solids in the dispersion was $w = 1$ and 5 %. However, during concentration experiments the concentration of solids in the dispersions varied from $w = 5$ to 20 %.

Equipments

The microfiltration studies were carried out in the membrane filtration units equipped with ceramic membranes. These experimental units were nearly the same for both the flux enhancement methods (see Figs. 1 and 2).

As shown in Fig. 1 the unit can be broken down into two major parts consisting of the circulation loop and the backflushing system.

The velocity and pressure in the retentate loop are varied independently by means of pump controller and an appropriate needle valve. The resulting feed velocities and average transmembrane pressures reach up to 5.8 m s^{-1} and 0.4 MPa, respectively. The circulation loop is constructed of stainless steel and contains a five-liter retentate container, a diaphragm pump, membrane module, and flow control valve at the module outlet. The loop is also equipped with a thermal regulation system, and a pressure and flow monitoring system. The permeate is collected in a reservoir placed on an electronic balance, which is connected to a personal computer.

The backflushing unit uses an air driven piston mechanism mounted on the permeate port of the membrane housing. Furthermore the system contains a timer for setting the frequency and duration of the pulses (computer controlled), and

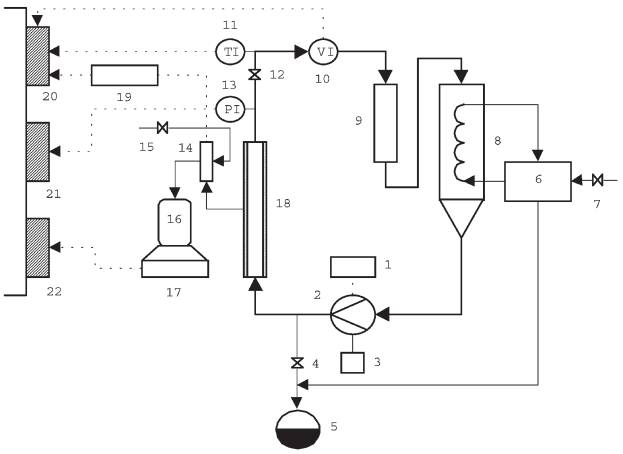

Fig. 1 – *Set up of membrane backflushing experimental apparatus*
1,19 – electrical switches, 2 – pump, 3 – pump speed controller, 4,7,12,15 – valves, 5 – to waste, 6 – temperature regulating system, 8 – retentate container, 9,10 – flowmeter, 11 – temperature indicator, 13 – pressure indicator, 18 – membrane module, 14 – backflushing unit, 16 – permeate reservoir, 17 – electronic balance, 20,21,22 – computer acquisition system

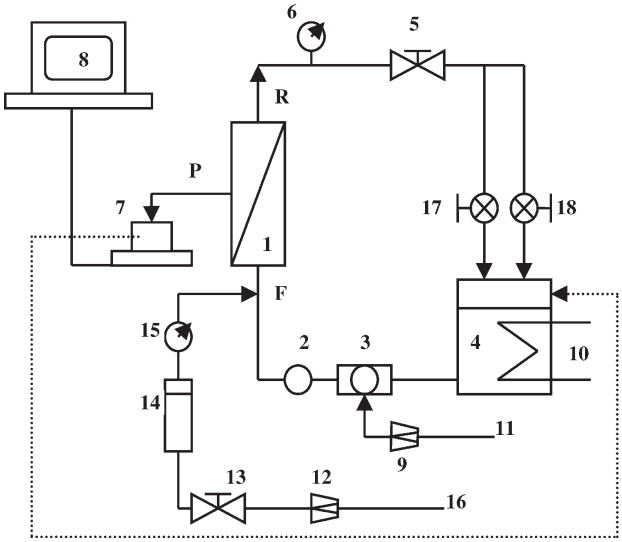

Fig. 2 – *Set-up of two-phase gas-liquid flow experimental apparatus*
1 – membrane module, 2 – pulse dampener, 3 – pump, 4 – storage tank, 5 – regulating valve, 6 – pressure gauge, 7 – electronic balance, 8 – computer, 9 – pump regulating valve, 10 – thermal regulating system, 11 – air inlet, 12 – air regulating valve, 13 – air valve, 14 – flowmeter, 15 – pressure gauge, 16 – air inlet, 17 – by-pass cock, 18 – closing cock, F – feed, P – permeate, R – retentate

pneumatic valves. The set up is capable of handling pulse times of 100 ms and larger. In the beginning of each backflushing cycle the first stage of the piston stroke closes the permeate outlet; than follows the second stage in which piston pushes the permeate back through membrane. The amount of permeate used for backpulsing can be varied in the range from 0 to 5.2×10^{-6} m^3.

The two-phase gas-liquid experimental apparatus used is shown schematically in Fig. 2. The circulating loop was constructed of stainless steel and contained a five-liter retentate container, a diaphragm pump, the membrane module and a flow control valve at the module outlet. The loop was also equipped with a thermal regulating system and pressure, temperature and flow monitoring systems. The velocity and pressure in the retentate loop were varied independently by means of pump controller and an appropriate needle valve. Air was added to the liquid stream at the inlet of the membrane, through a capillary. The airflow rates were controlled using a flowmeter.

Procedure

After the membrane was placed in the membrane module, distilled water was circulated in the test loop at the moderate operating pressure for about 2 hours. During this time a stabilization of the membrane was observed giving relatively stable water permeability. A concentrate of feed substance was then introduced to the unit, preheated to the desired temperature (25 °C), and the

operating pressure as well as retentate velocity were adjusted by the regulation system. The flux through membrane was measured by weighing the permeate and timing the collection period (by use of a balance interfaced with a computer). Both, the retentate and the permeate, were recirculated back into the retentate container. Therefore, the concentration in the recirculation loop remained virtually constant. After each set of experiments the circuit and membrane were rinsed with water and the pure water flux was measured again under the conditions of initial testing until the steady state was obtained. The differences in the steady state pure water flux were taken as a measure of the fouling tendency of the membrane.

Results and discussion

Application of the methods is discussed from the point of view of process efficiency (permeate flux) and the process operating conditions (transmembrane pressure, feed concentration, backflushing duration and frequency, liquid and gas flow velocity, and the influence of periodic gas flow).

Two-phase gas-liquid flow

The direct observations of two-phase gas-liquid flow mode through the transparent tubular pipe (of the same internal diameter as membranes) confirmed published results[5,6,18]. Each flow pattern corresponded to values of the superficial gas velocity, u_G, and the superficial liquid velocity, u, respectively, both of them being calculated as each phase was separately circulating. The main structures, which were observed when the gas velocity was increased for a given liquid velocity, included the bubble flow, slug flow, churn flow and annular flow (Fig. 3).

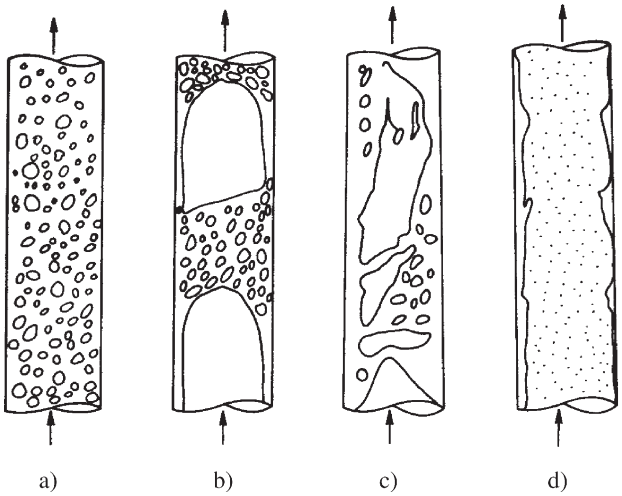

Fig. 3 – *Two-phase flow patterns in vertical tubes a) bubble flow, b) slug flow, c) churn flow, d) annular flow*

For u_G = 0.25–1.25 m s^{-1}, large bubbles were observed with size of the order of the internal diameter of the tube (Taylor bubbles). Due to reduction in the available cross-section for the liquid phase, a thin liquid film always remained over the surface of the membrane and moved in the opposite direction with respect to the main flow. This phenomenon induces a highly variable large shear rate against the pipe wall. It should be noted that for a given liquid flow-rate, the presence of the gas increases the mean longitudinal velocity of the fluid which, in association with the great variations in the wall shear stress and the turbulence existing in the churn flow (u_G = 1.5–2.3 m s^{-1}), can improve the performance. Previous work showed that slug flow is the most efficient regime for significant enhancement of mass transfer[20].

The effects of gas-liquid two-phase flow on permeate flux were measured. In the range of the experimental conditions (liquid flow velocity 1 m s^{-1} and transmembrane pressure difference 100 kPa), the permeate flux obtained with gas flow was always higher than under single liquid conditions, and the enhancement was maximum for a moderate liquid velocity (0.5 – 1.0 m s^{-1}) and a high proportion of injected gas (Fig. 4). It is clear that increasing either liquid velocity or gas velocity will enhance permeate flux. However, the increase of liquid velocity will require more pump power than the increase of gas velocity. Accordingly, the same permeate flux obtained with a higher liquid velocity but without gas slugs can also be achieved with a lower liquid velocity and moderate gas velocity with gas slugs (u_G = 0.8 m s^{-1}), leading to reduced energy consumption.

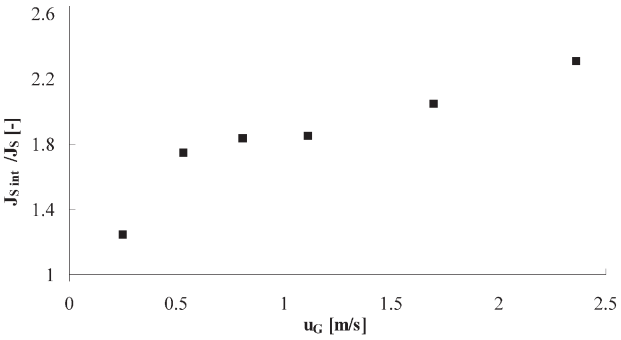

Fig. 4 – *Influence of constant gas flow velocity on normalized steady state permeate flux (u = 1 m s^{-1}, Δp = 100 kPa, x' = 0.01)*

The influence of two-phase flow on cake structure has then been analysed[21]. Figure 5 shows the evolution of cake porosity ε, with gas velocity for a transmembrane pressure difference 100 kPa and liquid velocity 1 m s^{-1}. The cake porosity increases as air is injected, and can reach nearly 0.55. In the presence of air, the cake of particles is very porous. No improvement in porosity is obtained under u_G = 0.5 m s^{-1}.

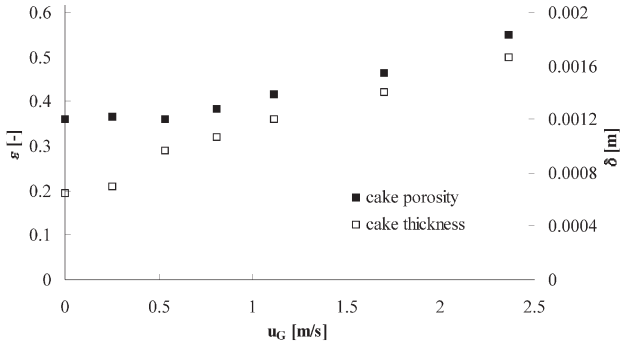

Fig. 5 – *Influence of constant gas flow velocity on cake porosity and cake thickness (u = 1 m s^{-1}, Δp = 100 kPa, x' = 0.01)*

Furthermore, the cake thickness also increases with air injection, as shown in Fig. 5. The cake thickness is nearly 700 μm without air injection. When two-phase flow is used, it can reach nearly 1600 μm, for a gas velocity around 2 m s^{-1}.

These results show that air injection seems to expand the particle cake: the cake obtained is thicker but more porous than without air, and thus allows higher permeation fluxes.

Figure 6 represents the evolution of the permeate flux vs. time for u_G = 0 and for u_G = 0.8 m s^{-1} for a steady and a periodic gas flow mode. The periodic gas flow mode consists in stopping air injection for 10 min every 30 min.

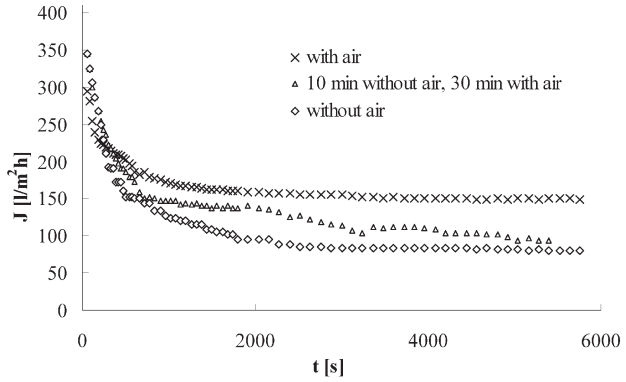

Fig. 6 – *Influence of a periodic gas flow mode on the permeate flux (u = 1 m s^{-1}, u_G = 0.8 m s^{-1}, Δp = 100 kPa, x' = 0.01)*

The first thing is that even for the periodic gas flow mode, the permeate flux after 1.5 h is increased in comparison with the flux without air. But after each interruption, the permeate flux decreases sharply. Within the first minutes of airflow interruption, on the membrane surface, a particle deposit is created which is difficult to remove when the air injection is restored. Then after filtering for 1.5 h the permeate flux reaches 170 l m^{-2} h^{-1} with a steady gas flow, whereas with the periodical gas flow mode it barely amount to 90 l m^{-2} h^{-1}. The aim of each process is different: the steady process permits to prevent a cake deposit, whereas in the periodical gas flow mode air has to eliminate the deposit built up during the air flow interruption. In similar experimental operating conditions a steady injecting process is more efficient than a periodic one, for which higher air velocities may be necessary to sweep the deposit.

Backflushing

To facilitate comparison of experimental data, the measured average permeate flux $J_{S\ int}$ was firstly normalized with respect to the steady state flux J_S without backflushing at the corresponding operating conditions (transmembrane pressure difference and retentate velocity). Calculated values of normalized flux were then plotted as a function of the duration of forward filtration t_F, duration of backpulses t_P, forward filtration transmembrane pressure difference Δp_F, reverse flow transmembrane pressure difference Δp_R, and feed flow velocity u, respectively. Figure 7 shows, for example, the normalized flux vs. duration of forward filtration t_F for w = 5%. Titania dispersion operated at duration of backpulses t_P = 0.2 s at different retentate velocities. The magnitude of the reverse transmembrane pressure difference had a relatively small effect and all presented results are given for Δp_R = 550 kPa.

The membrane backflushing results demonstrated that a 1.5-fold increase in the permeation flux could be maintained (operating at low tangential velocity) over the long-term flux in the absence of membrane backflushing.

It can be observed in Fig. 7, that with retentate velocity increasing the normalized permeate flux increased, however, values were strongly velocity dependent. At lower retentate velocities (0.5 – 1 m s^{-1}) the resistance due to boundary layer and/or particle cake layer was high and could easily be removed during reverse flow period. Thus effectiveness of membrane backflushing was high under these conditions. Of

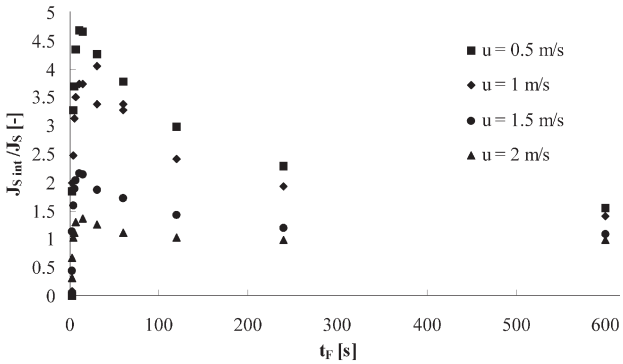

Fig. 7 – *Normalized permeate flux J_{Sint}/J_S as a function of forward filtration time t_F for various feed flow velocities u (Δp = 100 kPa, t_P = 0.2 s, x' = 0.05)*

course, if the retentate velocities were too low, the removed particles would not be swept out of the retentate channel by axial flow and the permeate flux would fall down progressively. On the other hand, the higher the retentate velocity (1.5 – 2 m s^{-1}) the thinner the temporary boundary layers on the membrane surface, and the permeate flow would be membrane controlled; also, the loss of permeate into the retentate stream would become significant, and consequently the influence of bacflushing decreased.

The experiments reported in Fig. 7 were carried out at a fixed transmembrane pressure difference of 100 kPa during the forward filtration period. This was a practically convenient value of driving force in order to perform experiments in the region of boundary layer control regime. It could be seen in the literature[19] that the effect of backpulsing was much more pronounced when the transmembrane pressure in forward filtration was higher enough to obtain a polarized layer of solids that control mass transfer phenomena. This was not surprising considering, that in the initial period of forward filtration (flow through clean membrane), the permeate flux was increasing the function of the transmembrane pressure difference (hydrodynamic controlled region). If transmembrane pressure difference was increased over a critical value, it could result in the irreversible membrane fouling (e.g. internal pore blocking) with the reverse flow unable to restore the original flux[19].

Comparison of the methods

The gas-liquid two-phase flow and membrane backflushing were effective for cross-flow microfiltration intensification and for each method optimal experimental conditions were found (transmembrane pressure difference Δp = 100 kPa and feed flow velocity u = 1 m s^{-1}) for the comparison. Flux enhancement distinctness has mainly depended on feed concentration for other process parameters, constant and optimal.

Figures 8 and 9 illustrate the advantage of constant gas-liquid two-phase flow and backflushing methods for aqueous titania dispersions (mass fraction 1 and 5 %) respectively. As in convectional single-phase cross-flow microfiltration the initial flux decline could still be observed. The presence of gas flow and/or backflushing does not modify the general behaviour of the flux variation with time. The steady state flux was generally increased and the flux decrease was slower.

There is a difference in the steady state flux between the two intensification methods. In the case of the feed mass fraction 1 %, the flux enhancement of both intensification methods has been nearly twice higher. The hydraulic resistance, which is added to the membrane resistance, could significantly be decreased by both flux enhancement methods. However the effect was more dramatic for gas-liquid two-phase flow than for

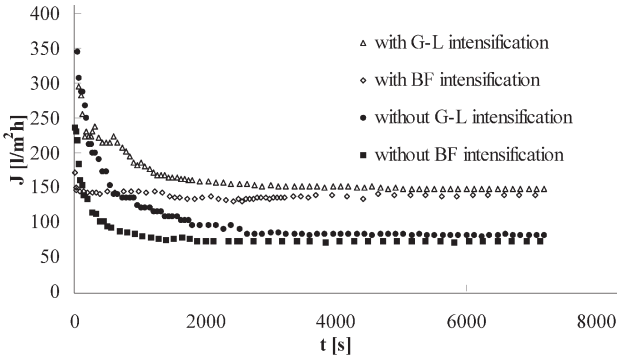

Fig. 8 – *Influence of gas-liquid flow and membrane backflushing on permeate flux in microfiltration of dispersion (u = 1 m s^{-1}, Δp = 100 kPa, x' = 0.01)*

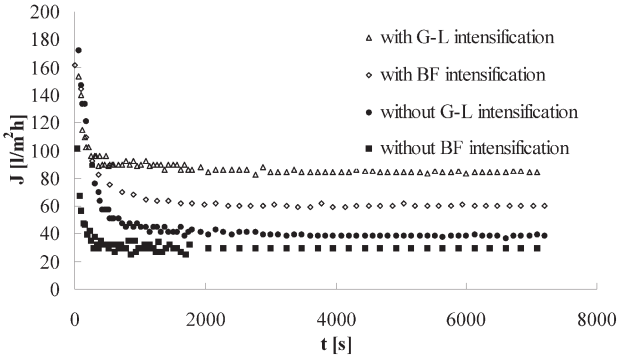

Fig. 9 – *Influence of gas-liquid flow and membrane backflushing on permeate flux in microfiltration of dispersion (u = 1 m s^{-1}, Δp = 100 kPa, x' = 0.05)*

membrane backflushing. Under similar experimental conditions gas-liquid flow in the vicinity of 0.8 m s^{-1} reduced the resistance by a factor of 2 for mass fraction of dispersion 1 % and by factor of 3 for mass fraction of dispersion 5 %.

Gas-liquid two phase flow and membrane backflushing were able to eliminate part of fouling and partly reduce the overall resistance at a lower retentate velocities (0.5 – 1 m s^{-1}), which was not possible to reduce by increasing liquid flow velocity or transmembrane pressure difference during the microfiltration of dispersions.

A more extensive study of the effect of the concentration of the dispersion on permeate flux was made in studies comparing the permeate flux of two-phase flow and membrane backflushing systems (Fig. 10). The results show that an operational run time produces a reasonable flux where it may be assumed that the concentration corresponding to the steady state flux value is reached. To facilitate comparison between the two different systems considered the normalized permeate flux was plotted as a function of the concentration of the dispersions (see Fig. 11). The intensification effects of both methods were nearly the same. For feed mass fraction 10 % the permeate flux was three times higher then flux without intensification.

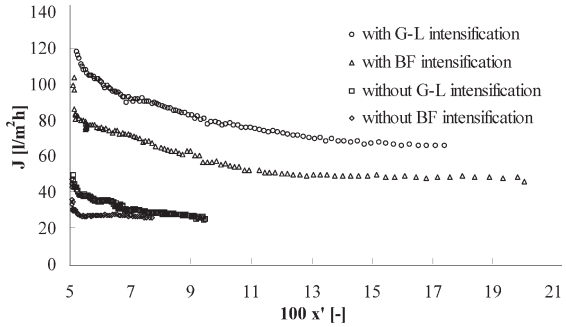

Fig. 10 – *Permeate flux vs. feed concentration during microfiltration of dispersion (u = 1 m s^{-1}, Δp = 100 kPa)*

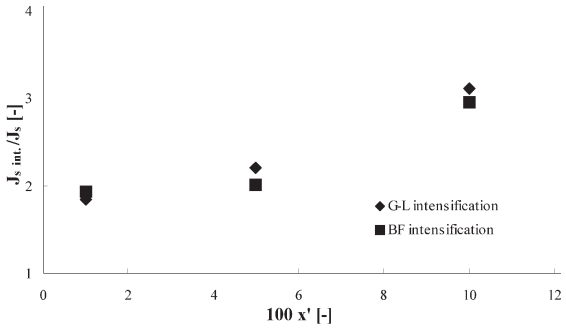

Fig. 11 – *Normalized permeate flux J_{Sint}/J_S as function of the feed concentration (u = 1 m s^{-1}, Δp = 100 kPa)*

Conclusions

The results of this experimental study demonstrated that in cross-flow microfiltration of dispersions, the gas-liquid two-phase flow and membrane backflushing could maintain the permeate flux at a constant and high level over the duration of an experiment.

The experiments under a large range of flow conditions showed that the constant gas-liquid two-phase flow enhances microfiltration flux better than periodic gas flow. However, this phenomenon depends on the periodic gas flow mode and on dispersion concentration. This effect is probably due to the high and transient wall shear stress induced by the gas flow. The hydrodynamic regime inducing the largest enhancement in flux, is a slug flow in the case of aqueous titania dispersion, where a permeate flux plateau is reached at the beginning of the slug flow regime.

It was observed that the effect of backflushing was much more pronounced when the backpulse duration was shorter, the transmembrane pressure difference was higher, and the retentate velocity in forward filtration was lower.

ACKNOWLEDGEMENTS

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List of symbols

J – permeate flux, lm^{-2} h^{-1}
J_S – steady state permeate flux without intensification, lm^{-2} h^{-1}
$J_{S\,int}$ – steady state permeate flux with intensification, lm^{-2} h^{-1}
Δp – transmembrane pressure difference, kPa
Δp_R – reverse transmembrane pressure difference, kPa
t – time, s
t_P – duration of backpulses, s
t_F – duration of forward filtration, s
u – superficial liquid velocity, m s^{-1}
u_G – superficial gas velocity, m s^{-1}
x' – mass fraction of dispersion
δ – cake thickness, m
ε – cake porosity
w – percentage by weight

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