Crossflow microfiltration of marble processing wastewaters

Maria Diná Afonsoa*, Ana Maria Brites Alvesa, Mousa Mohsenb

aInstituto Superior Técnico, Chemical Engineering Department, 1049-001 Lisbon, Portugal
Tel. +351 (21) 8417595; Fax +351 (21) 8499242; email: Dina.Afonso@ist.utl.pt
bHashemite University, Mechanical Engineering Department, 13115 Zarqa, Jordan

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Abstract

The wastewaters generated during the marble processing (cutting and grinding operations), the average flow rates of which are ca. 15 m3/h for a plant producing 45 m2 of polished marble/h, contain a high load of particulate matter. Furthermore, crushed stone operations sometimes use flotation agents, such as organic amines, fatty acids, and pine oils, in order to remove impurities from the marble. Therefore, the marble processing wastewaters should not be disposed directly into the environment without a suitable treatment in order to prevent negative impacts and comply with the national legislations currently in force. On the other hand, bearing in mind the severe water shortage in Mediterranean countries, which are intensive producers of carbonaceous rock and stone, it would be of utmost importance to recover and reuse the processing wastewaters generated in these plants. In this work, the treatment of marble processing wastewaters by microfiltration (MF) was investigated. Wastewaters from a marble processing plant located in Pero Pinheiro, Portugal, were characterised in terms of pH, conductivity, total solids (TS), and total suspended solids (TSS). A Micro Carbosep 60 module equipped with a mono-tubular mineral MF membrane, Carbosep M45 (nominal pore size = 0.45 µm, ID = 6 mm, L = 0.60 m), was tested at ambient temperature, natural effluent pH, a broad range of transmembrane pressures and crossflow velocities, and up to a 9-fold concentration. MF eliminated the suspended matter from marble wastewaters, allowing the treated water to be recycled into the process, whereas the concentrated stream (rich in particulate matter) can be used elsewhere, e.g. in the ceramic industry. The results from this work clearly show that MF is an efficient and ecologically suited environmental technology for decontamination and recycling of the wastewaters generated by marble processing plants, besides the environmental pollution abatement involved.

Keywords: CaCO3 suspensions; Inorganic membranes; Marble wastewaters; Microfiltration; Water reuse

*Corresponding author.


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1. Introduction

Crossflow microfiltration (MF) is a downstream solid-liquid separation process in which a suspension flows tangentially under moderate pressure through a microporous membrane (nominal pore size = 0.1–10 um). Colloids, microparticles, micro-organisms and macromolecules present in the suspension can be effectively removed by this filtration process. Therefore, crossflow MF has been widely used as a solid-liquid separation technique in the beverage industry (e.g. clarification of beer, wine and juices), food industry, biotechnology (e.g. separation of bacteria from substrate), besides water and wastewater treatment. Purified fluid passes through the membrane as permeate, whereas the retained particles form a polarisation layer and/or a thin cake on the membrane surface. This accumulation of particles close to the membrane surface due to its permselectivity, the so-called concentration polarisation and/or cake build-up, is one of the most serious problems in membrane separation processes. Indeed, the filtration resistance increases and the permeate flux (productivity) decays continuously as the thickness of the polarisation layer or cake increases until a steady-state is reached. However, since the feed flows tangentially to the membrane surface in crossflow MF, the accumulation of particles is limited and the pressure drop is lower in comparison to the conventional filtration process. Even so, the deposition of particles on the membrane surface during crossflow MF of inorganic particles and colloidal matter is the main cause for the permeate flux decline, the steady-state flux being usually 30–50% of the initial flux [1]. A key goal in the design of crossflow microfilters is then to predict the steady-state permeate flux as a function of the operating conditions.

Several hydrodynamic models have been developed to describe the forces exerted on the particles deposited on the membrane surface by crossflow MF under steady-state and transient conditions. It is well known that after a brief start-up period in which a concentrated layer of particles builds up near the membrane, a steady-state is reached wherein the rate of particles driven toward the membrane surface by the permeate flow must be balanced by some mechanism of particle motion away from the membrane surface. It is the nature of this back transport mechanism that has been the subject of extensive research. Proposed mechanisms leading to steady-state include Brownian diffusion, inertial lift [2], shear-induced diffusion [3,4], axial convection (lateral migration) [5,6], and particles instability [7]. An excellent review of these mechanisms was published by Belfort et al. [2], who concluded that none of the postulated removal mechanisms could be considered dominant for all applications, and that more than one could operate simultaneously depending on the conditions. In general, Brownian diffusion is only important for submicrometer particles and low shear rates, whereas inertial lift is only significant for large particles (>25 µm) and/or high shear rates. For micronmeter particles and shear rates on the order of 10³ s⁻¹, shear-induced diffusion is thought to be dominant [4]. Early attempts to model crossflow MF assumed that the convective flux of particles carried toward the membrane with the permeate is balanced at steady-state by Brownian back-diffusion away from the membrane, but this model underpredicted the measured steady-state permeate fluxes by one or two orders of magnitude. The inertial lift mechanism assumes that under lift and drag forces the particles move from the duct axis and from the porous membrane to develop an annular concentration profile, the ratio of the permeation drag force to the lift force being the criterion for the build-up of a cake on the membrane surface [2]. An alternative model for predicting the permeate flux in crossflow MF controlled by a thin polarisation layer deposited on the membrane surface, yielded better results than the Brownian model by replacing the small Stokes-Einstein diffusivities of the particles with a concentration-dependent shear-induced hydrodynamic diffusivity [3,4]. This model combines the concepts of a concentration polarisation layer controlled by shear-
induced diffusion with a stagnant layer of deposited particles (cake) which exerts a hydraulic resistance on the permeate flux. The model is inappropriate for the crossflow MF of particles that become irreversibly stuck when deposited on the membrane surface due to interparticle adhesive forces. In this case, the filtration may be controlled by local effects at the top surface of the cake and an adsorbing boundary condition should be applied [3]. Romero and Davis [4] further noticed that more highly concentrated suspensions of smaller particles, higher transmembrane pressure drops (or higher permeate fluxes) and lower shear rates led to more rapid flux decline. Vassilieff et al. [5] claimed that the shear-induced diffusion model just supplies an order of magnitude estimate for the steady-state permeate flux, and developed a convective model for crossflow MF, which appears to have solved the so-called “flux paradox for colloidal suspensions”. They assumed the formation of a packed cake on the membrane, which depending upon the shear stress magnitude can be either stagnant or flowing. They neglected diffusion of any kind (Peclet number >> 1), and instead postulated that at steady-state the convection of particles driven toward the membrane is balanced by axial convection along the membrane as a dense flowing cake layer. These authors concluded from crossflow MF experiments conducted with a dilute bentonite suspension in laminar flow regime that the initial transient effects were due to the growth of a flowing cake above the membrane. They further noted that steady-state conditions could be attributed to a tangential outflow from the dense cake with non-Newtonian behaviour. As the previous filtration models did not account for all the significant back-transport forces, Chang et al. [7] developed a more universal model based on particle rolling motion and a momentum balance including lateral lift force, shear-induced force, double-layer repulsion force, van der Waals attraction force, Brownian diffusion, and gravity force, besides the normal and tangential forces acting on the particle. Nevertheless, the theoretical developments of the aforementioned models only hold for laminar flow conditions, thus their practical application is quite limited. Rautenbach and Schock [8] presented a model for crossflow MF of colloidal suspensions (quartz and clay) in turbulent flow conditions ($<u>_t = 2–6$ m/s). They derived the condition for particle motion on the membrane from the equilibrium of the membrane parallel drag forces acting on a particle in the membrane vicinity and a “friction” force resulting from the lateral drag forces. Basically, the drag forces induced by the convective flow toward the membrane force the smallest particles to deposit on the membrane surface and eventually a cake builds up, whereas the drag forces induced by the crossflow tend to move the biggest particles away from the membrane surface. Arnot et al. [9] focused on the mechanisms and modelling of flux decline during cross-flow and dead-end MF of oily-water emulsions in a broad range of operating conditions, covering laminar and turbulent flow regimes ($<u>_t = 0.4–1.0$ m/s). They found out that for a fixed crossflow velocity, the flux decline increased with $\Delta P$ and also that for a given $\Delta P$, it was substantially lower for $<u>_t \geq 0.8$ m/s, thus they set the optimal operating conditions as being 0.6 bar and 0.8 m/s for the module and feed used. In an optimisation study of a back-flush sequence for zeolite microfiltration, the hydrodynamic conditions in the module during forward MF were set as 0.8 bar and 1 m/s [1]. Herath et al. [10] investigated the effect of permeation velocity (suction) on the mass transfer in turbulent flow conditions. The crossflow velocity was varied between 0.9–2.5 m/s in a tubular MF membrane, well within the turbulent flow region (Reynolds numbers between 7,500–19,600). They checked out that the experimental mass transfer coefficient for two viruses’ strains was enhanced by suction, and proposed an equation for the corrected mass transfer coefficient.

Many techniques have been developed recently to reduce membrane fouling, and thus enhance MF performance, e.g. modification in the surface interactions between particles, modification in the membrane hydrophobicity, enhanced
modules design or operation (turbulence promoters, baffles, pulsate flow, intermittent flow, Taylor and Dean instabilities, back-flushing, etc.), and optimisation of the hydrodynamics inside the module to increase the turbulence in the membrane vicinity. In the present work, we were mainly concerned in selecting a combination of favourable operating conditions (sufficiently high velocity and low transmembrane pressure) that would minimise the concentration polarisation and/or the growth of a compact cake on the membrane surface during the concentration of marble wastewaters by crossflow MF, in order to achieve high permeate fluxes. We must emphasise that the comprehensive works of Jaffrin and co-workers [11–15] were extremely helpful in accomplishing our goal as they exclusively focused on surface fouling during crossflow MF of CaCO$_3$ suspensions. They observed that the cake formed by CaCO$_3$ particles is irreversibly compressible (permeate flux hysteresis in cyclic variations of pressure) and that the particles therein stick together in a consolidated state due to interparticle cohesive forces. The build-up of such a cake cannot be reversed, i.e. the particles on the cake top layer cannot be swept away, by lowering the pressure or diluting the suspension (lower concentration), unless a high shear stress (high velocity) exceeding the critical erosion stress (yield stress) is exerted on the cake top layer, a concept similar to the one presented by Vassilieff et al. [5]. As the critical erosion stress is an increasing function of transmembrane pressure, it is actually wise to operate at low pressures and crossflow velocities higher than the critical velocity for cake erosion. It is worth noting that the particular behaviour of a cake formed by CaCO$_3$ particles may not be observed in cakes formed by bacteria or flexible and smooth particles [14]. Another interesting work carried out by Fane and co-workers [6] was the study of the transition from concentration polarisation to cake formation during MF of colloidal silica negatively charged, under controlled flux conditions. They observed that once the critical flux was exceeded, the colloids in the stagnant polarised layer adjacent to the membrane surface formed a structured cake, due to consolidation and aggregation of silica particles, which was resistant to redispersion. They further noted that the depolarisation can be enhanced by increasing pH or crossflow velocity, and highlighted the current interest in controlling hydrodynamics in particular by operating at low to modest pressures in order to minimise fouling and thus maximise the productivity.

2. Experimental

2.1. Experimental set-up

A scheme of the experimental set-up is displayed in Fig. 1. It comprises a standard lab unit equipped with a Micro Carbosep 60 module mounted in the horizontal position such that the gravity effects on the pressure gauge readings are negligible. A mono-tubular mineral microfiltration membrane rated at 0.45 µm pore size, Carbosep M45 (Rhodia-Orelis), was tested in this work. The membrane active layer consists of a thin deposit of ZrO$_2$-TiO$_2$ on a carbon support. The membrane is 6 mm in inside diameter and 0.6 m long (membrane surface area of 0.0113 m$^2$).

The permeate and concentrate flow rates were measured with a stopwatch and a graduated cylinder. The concentrate flow rate and the transmembrane pressure were controlled manually by the by-pass valve and the needle valve at the outlet module.

![Fig. 1. Tubular membrane experimental set-up (V — valves, P — pressure gauges).](image-url)
2.2. Samples collection and characterisation

Three samples of 5 l each were collected from the wastewaters generated in a marble processing industry located in Pero Pinheiro, Portugal. They were taken directly from the recycle loop in between the grinding process and the feeding tanks. A sample was taken from the well water make-up, as well. For comparison purposes, a sample was collected from the decanter recently installed to treat the marble wastewaters and recycle them to the process. None of the referred samples was kept in the refrigerator before their characterisation or MF processing, due to the absence of organic matter.

The collected samples, and the samples from the concentrate and permeate streams of MF experiments were analysed with respect to pH, conductivity, total solids (TS), and total suspended solids (TSS). The density, viscosity and particle size distribution of marble wastewaters were also determined (Table 1).

Table 1
Measurement devices and analytical methods

<table>
<thead>
<tr>
<th>Measurement devices and analytical methods</th>
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<tbody>
<tr>
<td>Density</td>
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<td>Viscosity</td>
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<tr>
<td>Particle size distribution</td>
</tr>
<tr>
<td>pH</td>
</tr>
<tr>
<td>Conductivity</td>
</tr>
<tr>
<td>TSS</td>
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</tbody>
</table>

2.3. Membrane characterisation

Firstly, the membrane was wetted out by circulating distilled water at 1.5 bar for 2 h, such that the excess of chemicals attached to the membrane surface could be released.

Afterwards, distilled water permeated at pressures up to 1.5 bar, in order to measure the corresponding water permeate fluxes, $J_w$, and assess the membrane hydraulic permeability, $l_p$:

$$J_w = l_p \cdot \Delta P_{oper}$$  \hspace{1cm} (1)

2.4. MF of marble wastewaters

2.4.1. Total recirculation

Two distinct sets of MF runs were performed with marble wastewaters at natural pH, ambient temperature, and total recirculation mode, i.e. both the concentrate and the permeate were recycled back to the feed tank in order to maintain the feed composition nearly constant.

2.4.1.1. Study of the influence of transmembrane pressure and crossflow velocity

In the first set of runs, the operating pressure and the crossflow velocity were varied from 0.25 to 1 bar, and from 0.07 to 1.2 m/s, respectively. At a given set of operating conditions, the permeate flux, $J$, was measured after 15 min of operation, and samples from the feed and permeate streams were collected for analysis, to assess the rejections, $R$, with respect to total solids (TS) and total suspended solids (TSS):

$$R = \left(1 - \frac{C_{permeate}}{C_{feed}}\right) \cdot 100\%$$  \hspace{1cm} (2)

2.4.1.2. Study of the influence of permeation time

In the second set of runs, three permeation experiments were carried out at (1 bar; 0.7 m/s), (0.7 bar; 0.9 m/s), and (0.5 bar; 1.7 m/s), for approximately one hour each, in order to evaluate the fouling tendency of marble wastewaters independent of concentration effect. The permeate flux was measured in intervals of ca. 10 min.

2.4.2. Concentration of marble wastewaters

The concentration of marble wastewaters by MF was conducted at natural pH, ambient temperature, and the optimal operating pressure
and crossflow velocity previously determined, i.e. 0.7 bar, and 0.9 m/s, respectively. During this experiment the permeate was continuously discarded, whilst the concentrate was recycled back to the feed reservoir. The permeate flux was measured and permeate samples were collected for increasing volume reduction factors, VRF, up to 9.

3. Results and discussion

The marble wastewaters consist of suspensions of calcium carbonate (CaCO₃), the average density of which is 1.024 g/l. The rheological behaviour of these suspensions was surprisingly similar to the rheological behaviour of pure water, hence the marble wastewaters may be ranked as a Newtonian fluid, despite its relatively high concentration of solids (25–37 g/l). In fact, Leighton and Acrivos [16] showed that suspensions of rigid spheres exhibit nearly Newtonian behaviour when undergoing simple shear flow, provided that the suspending fluid is Newtonian, and that any colloidal forces are weak. The data of the particle size distribution of marble wastewaters are depicted in Fig. 2. The lognormal size distribution assumed is characterised by an average effective diameter of 2.0 µm and a low polydispersity (0.005), corresponding to a thin half-width of 0.1 µm. Furthermore, there is at least another distribution of macroparticles (big particles and/or particle aggregates), easily observed at naked eye, but impossible to be assessed by the measurement device due to the extreme rapidity of macroparticles to settle down. As soon as the sample is inserted in the analyser, the heavy particles start falling down and deposit on the bottom of the sample cell without being detected by the laser light even for low analysis times (10 s). These macroparticles should result from microparticles agglomeration into larger clusters due to interparticle cohesive forces.

The chemical characterisation of marble wastewaters, marble decanted wastewaters and well water is presented in Table 2. As shown in this table, the pH of marble wastewaters is much higher than that of well water, whilst the conductivity is much lower. The aeration of marble processing wastewaters by flowing in open pipes at high velocities towards the feed tank promotes the release of carbon dioxide, and since this is an acidic gas (in equilibrium with carbonic acid, H₂CO₃), its removal tends to decrease [H⁺] and thus raise the pH of marble wastewaters to between 8 and 10, in accordance with Eq. (3) [17]:

\[
\text{CO}_2 + \text{H}_2\text{O} \leftrightarrow \text{H}_2\text{CO}_3 \leftrightarrow \text{H}^+ + \text{HCO}_3^- \quad (3)
\]

On the other hand, the behaviour of weak acid-base systems is also intimately involved in the precipitation phenomena. In natural waters containing appreciable amounts of Ca²⁺, calcium carbonate precipitates when the carbonate ion concentration, according to Eq. (4):

\[
2 \text{HCO}_3^- \leftrightarrow \text{CO}_3^{2-} + \text{H}_2\text{O} + \text{CO}_2 \quad (4)
\]

becomes great enough so that the CaCO₃ solubility product is exceeded:

<table>
<thead>
<tr>
<th>Table 2</th>
<th>Marble wastewaters</th>
<th>Marble decanted wastewaters</th>
<th>Well water</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td>8.4–9.5</td>
<td>10.6</td>
<td>7.6</td>
</tr>
<tr>
<td>Conductivity, µS/cm</td>
<td>427–500</td>
<td>498</td>
<td>931</td>
</tr>
<tr>
<td>TS, g/l</td>
<td>25–37</td>
<td>1.9</td>
<td>—</td>
</tr>
<tr>
<td>TSS, g/l</td>
<td>25–37</td>
<td>1.6</td>
<td>—</td>
</tr>
</tbody>
</table>

Fig. 2. Microparticle size distribution of marble wastewaters.
Ca^{2+} + CO_3^{2–} ↔ CaCO_3  \quad (5)

This precipitation usually occurs before pH levels have exceeded 10, which limits the pH values obtainable [17–20]. Therefore, the high pH of marble wastewaters favour the retention of suspended CaCO_3 by MF, since such wastewaters are oversaturated and thus tend to precipitate CaCO_3, as outlined by Kedem, Gilron and co-workers [21–24]. These authors have developed a new softening method, the so-called “compact accelerated precipitation softening” (CAPS), which consists basically of raising the pH to 8–10.5 to reduce calcium and carbonate alkalinity by accelerated precipitation of CaCO_3 from the supersaturated basic water, followed by filtration or MF.

Moreover, the removal of CO_2 by aeration leads to the reduction of [HCO_3] and [CO_3^{2–}], resulting in a lower conductivity of marble wastewaters in comparison to well water. As shown in Table 2, decantation removes the majority of the suspended matter present in marble wastewaters, but the separation is not complete and a little amount of solids still remain in the treated waters.

The hydraulic permeability of the membrane Carbosep M45 is 177 l/(m^2·h·bar). The results of MF experiments relative to the effect of crossflow velocity and transmembrane pressure upon the permeate flux are plotted in Fig. 3. The conclusions to be drawn are the following:

- For high velocities in the range of 0.8–1.2 m/s (turbulent regime), the permeate flux, \( J \), increases linearly with pressure revealing the total absence of concentration polarisation or cake build-up. Amazingly, at high pressures, it is clear that \( J \) is higher than \( J_w \), most likely due to operation at a slightly higher transmembrane pressure in comparison to that for pure water permeation, as a result of the lower pressure drop presented by marble wastewaters (higher density) and higher stability of the pressure gauge readings.

- For low velocities in the range of 0.07–0.14 m/s (laminar regime), the permeate flux, \( J \), increases linearly with pressure up to 0.4 bar. At higher pressures, \( J \) is no longer linear, revealing a strong effect of concentration polarisation and/or cake build-up. Although the deviation between \( J \) and \( J_w \) gets wider as the pressure increases, \( J \) does not show a trend to reach a threshold.

- The TS and TSS rejections, 98.6 and 99.9% respectively, are extremely high and constant irrespective to the operating conditions. The pH and conductivity of the feed and permeate samples are quite similar.

The membrane hydraulic permeability measured immediately after this set of experiments was 179 l/(m^2·h·bar), thus there was no need for prolonged washing. This fact shows that no irreversible fouling of the membrane (adsorption, pore plugging or whatsoever) occurred at the operating conditions employed. This conclusion is reinforced by the study of the permeation time effect upon the permeate flux displayed in Fig. 4. As shown in this figure, the permeate flux at three different pressures and high velocities, was similar to the pure water flux under equivalent pressures, and remained nearly constant over time, except at 1 bar for which \( J \) is clearly higher than \( J_w \) and slightly decayed with time. This flux decline was the main reason for selecting a lower pressure, namely 0.7 bar, to conduct the concentration experiment.
The results of marble wastewaters concentration by MF are plotted as a function of the volume reduction factor, $VRF$, in Fig. 5. The following conclusions can be drawn:

- The MF membrane tested efficiently concentrates the particulate matter since the TSS rejections are 100% throughout the experiment, meaning that no particles pass through the membrane (complete separation). The pH and conductivity of the permeate samples remained constant throughout the experiment, whereas the TS rejections slightly increased from 99.4% to 99.8%.
- For total recirculation ($VRF = 1$), the permeate flux is nearly equal to the pure water permeate flux, and it remained constant throughout the experiment up to a 9-fold concentration (ca. 1.5 h later on), which is the most striking result from this work.

The membrane hydraulic permeability measured after the concentration experiment was 169 l/(m²·h·bar), thus there was no need for extensive washing to restore its initial value.

Some experimental data presented in [13, 14, 25] are similar to our own results. Ould-Dris et al. [14] investigated the stability of cakes formed by calcium carbonate particles on MF organic membranes by imposing cyclic variations of transmembrane pressure, crossflow velocity and particles concentration. They reported the absence of fouling during MF of CaCO$_3$ suspensions until the transmembrane pressure reached 0.9 bar, at a crossflow velocity of 0.8 m/s and a concentration of 100 g/l. Moreover, they noted that at a pressure of 1 bar and the same concentration, the critical velocity at which the cake starts getting eroded (1.1 m/s) is higher than the velocity at which the cake starts to form (0.83 m/s). Furthermore, they observed that the maximum permeate flux attained at 1 bar for each concentration, in the range of 60–100 g/l, was close to the pure water flux. In a complementary study, Ould-Dris et al. [13] pointed out that at 1 bar the permeate flux of a CaCO$_3$ suspension 100 g/l decayed rapidly with time at a velocity of 0.1 m/s, whereas it remained nearly constant and equal to the pure water permeate flux for crossflow velocities higher than 0.8 m/s. Bouzerar et al. [25] found out that the permeate flux of a suspension of ferric hydroxide 9 g/l through a flat MF zirconium oxide membrane mounted in a dynamic crossflow device (rotating disk) raised linearly with pressure with a slope equal to the membrane hydraulic permeability,
confirming the absence of a deposited layer on the membrane, although the system operated in the laminar boundary layer regime. Besides, the permeate flux did not decay with time nor presented hysteresis in cyclic variations of rotation speed. They further observed that the permeate flux rose up to 50% with the concentration increase from 9 to 39 g/l, presumably due to the transmembrane pressure increase caused by the increase of the suspension density. The permeate flux decreased thereafter, as the pressure rise effect was surpassed by fouling caused by the concentration increase up to 130 g/l. The authors highlighted that operating at low transmembrane pressure is essential when the cake is compressible, as the critical erosion shear stress for cake removal increases with transmembrane pressure.

4. Conclusions

The microfiltration of wastewaters from a marble processing industry was investigated. A tubular mineral membrane 0.45 μm nominal pore size, Carbosep M45, was tested in this work. For moderate transmembrane pressures and high velocities, the MF permeate flux of marble wastewaters does not decay with respect to pure water permeate flux, remaining constant either with time and a 9-fold concentration rise, revealing the total absence of concentration polarisation and/or cake build-up. This is a remarkable finding, meaning that there is no need for membrane washing during crossflow MF of marble wastewaters, at least up to a 9-fold concentration. The high permeate fluxes and particles separation achieved, especially throughout the concentration run, clearly points out that MF is technically suited to accomplish the aimed goals, i.e. environmental pollution abatement from the marble wastewaters, recycle of water into the process and potential reuse of the concentrate stream elsewhere, e.g. in the ceramic industry.

Continuous experiments should be carried out at higher concentration factors and longer operation periods, in order to get better insight of the concentration effect upon the concentration polarisation phenomena and/or cake build-up and thereby optimise the water recovery rate during microfiltration of marble wastewaters.

References


