Comparative study of the performance of three cross-flow ceramic membranes for water treatment

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Abstract

Several tests using water as effluent are used to analyse the performance of three types of microfiltration cross-flow ceramic membranes. Two of these membranes are commercial (Atech and Membralox/US Filter) and the third one is experimental. The main differences between them lie in their chemical composition (different origin of raw materials) and in their manufacturing process.

The results presented here show the dominant effect of the filtering and the gel layer. Both are formed during operation acting as equalising agent between the three membranes. The membranes tested have similar performances in cross-flow operation, although permeability rates for the membrane Membralox/US Filter were about 15% higher. This increase might be due to the smoother surface formed by a second filtering ultrafiltration layer (0.01 μm) of 10 μm width, which probably contributes to a decrease in the thickness of the gel layer formed during operation.

Using specific raw materials (non-industrial) as well as a second ultrafiltration layer improves the results in operation (performances and cleaning intervals). However, they are uneconomical because of the extra costs involved.

In conclusion, low-cost membranes can achieve similar results to the commercial and more expensive ones opening up their application to new uses and emergent markets.

Keywords: cross-flow, ceramic membrane, filtration, permeability

Introduction

The use of membranes for liquid filtration water treatment processes is very common nowadays. Drinking water supply needs (particularly from recycling) in countries where water is a scarce commodity, and the environmental legal pressure in Europe and USA, have compelled the development of membranes, since increased reduction of turbidity and chemicals for water disinfection is required (EPA, 2001). Generally, conventional processes for splitting liquid streams use chemical treatments (for instance, flocculation or precipitation) or physical treatments (for instance, decantation or centrifugation). From the industrial point of view, membrane filtration is an easy-to-use alternative to these processes.

Membranes have great potential for drinking water production and wastewater treatment, microfiltration (MF) and ultrafiltration (UF). Firstly, they are able to remove particles, micro-organisms (bacteria like *Clostridrium perfringens*, cysts and viruses) and colloidal material, accomplishing the legal requirements of water quality, especially the new EU regulations related to drinking water. Besides, compared with other treatment systems, membranes have the following advantages:

- They do not require the use of any chemicals (such as chlorine)
- They can operate at ambient temperature
- Modular implementation is an option
- Their operation can be automatic.

Commercial MF and UF membranes are, principally, made of either organic polymers or ceramic materials. Nowadays, using polymeric membranes for water purification is more common. However, ceramic membranes have some advantages that could increase their acceptance in water purification matters and also in wastewater treatment in general (urban and industrial wastewaters).

Compared to organic membranes, ceramic membranes have a wide range of advantages such as a better mechanical resistance; they are more resistant to solvents and to micro-organisms. Moreover, they are more durable and they are able to operate under difficult conditions of pH, oxidation, temperature and pressure. Finally, in maintenance cost terms, they are less expensive as they can be cleaned more easily. On the other hand, their manufacturing cost is higher.

The high cost of membranes in general, and the ceramic ones in particular, is doubtless their main drawback as alternative to water treatment. This high cost, in the case of ceramic membranes, is due to the particular manufacturing process. The sintering of materials needs temperatures of as high as 1 600 to 1 800ºC, depending on the grain size (the smaller the grain size, the lower the sintering temperature). Therefore, membrane technologies are basically used in the treatment and recovery of high value-added products like industrial effluents, wine, milk, juices, etc. (Sondhi et al., 2003).

A ceramic membrane usually consists of two elements: Firstly, the filtering layer, a very thin surface layer with fine porosity; and secondly, the membrane support with higher porosity to which the filtering layer is linked. The support itself can consist of up to three layers with varying grain sizes. The relation between them is very important since the physical separation of particles in the fluid takes place in the filtering layer, whereas the main function of the membrane support is to give mechanical support to the filtering layer.

Ceramic membranes are usually of the cross-flow type operation, which implies lower energy needs. The first filtration processes by membranes were 'dead-end', i.e., total filtration,

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Figure 1
Picture of three MF membranes tested. From the left to the right: Membralox/US Filter, Atech and experimental T1-1.

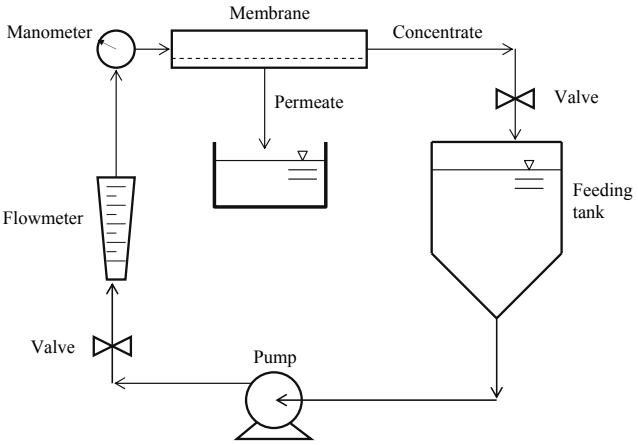

Figure 2
Layout of the experimental laboratory plant

requiring very high pressures (up to 70 bar) to obtain acceptable permeates. Nowadays, almost all filtration processes are cross-flow, with pressures that reach 20 to 30 bar at maximum (in inverse osmosis processes, for instance), but they can operate with pressures as low as 1 to 4 bar (Koros et al., 1996).

We developed a project with the objective to obtain low-cost ceramic membranes to be used as tertiary water treatment (Rodríguez-Grau, 2005). This paper reports the results of a comparative experimental study about the permeability of these experimental membranes compared with two commercial ceramic membranes, namely: Membralox from US Filter, and Atech from Atech Innovations, for water treatment through microfiltration (Fig. 1). In this work the experimental membranes tested have two different compositions: the support of the membrane called T1 is made of 90% tabular alumina T60 Almatis and 10% rutile (TiO_2) R-900 DuPont. On the other hand, the support of the one called T3 is made of 80% corindon CAHPF360 Alcan and 10% rutile R-900 DuPont. Finally, the filtering layer is, in both cases, made of rutile R-900 DuPont with some polyvinyl additives and similar to all membranes tested. Their tubular bodies were obtained by extrusion and then sintered at temperatures of up to 1 300ºC. After that, their filtering layer was applied to the inner part of the sintered tubes by circulating the mixture of rutile and additives with a peristaltic pump and by controlling the application time. Then, the tubes with the filtering layer were sintered again at up to 1 000ºC (Rodríguez-Grau, 2005). Finally, T1 and T3 membranes showed two configurations: monochannel tube and 7-channel tube, which differed only in their surface area available to filtration. Therefore, as far as permeability results are concerned, there are no significant differences between these configurations since the permeability rate is expressed as litres of permeate per hour and per filtration area and per bar of pressure ($\ell \cdot h^{-1} \cdot m^{-2} \cdot bar^{-1}$).

Experimental set-up and procedure

The membrane experiments were carried out in a small laboratory plant (Fig. 2). This plant consisted of a simple hydraulic circuit that operated with just one membrane for each run. According to our methodology on laboratory scale, membranes were up to 360 mm in length and the diameters ranged between 20 and 30 mm. The plant had a centrifugal pump that gave a maximum flow rate of 3.6 $m^3 \cdot h^{-1}$ or a maximum pressure of 4.6 bar. Two manual valves allowed adjustment of the flow rate and pressure of the input flow, with the aid of one flow meter and one manometer. The liquid to be tested was stored in a 120ℓ feed tank where recirculation is possible. Permeate could be either collected in a separate container or recirculated together with the concentrate in the feed tank. The chemical cleaning of the membranes and the entire circuit could be easily achieved. Firstly, it was necessary to replace the feed tank by another tank containing the cleaning product. Secondly, we had to leave it flowing as under normal operating conditions during a given time. Unfortunately, retro-cleaning was not possible with this experimental plant.

The list of the MF membranes tested appears in Table 1. All these membranes were new when the experiments commenced. Before use they were cleaned consecutively with a solution of 2% HCl (30 min), clean water (5 min), solution of 0.5 N NaOH (30 min), and clean water (5 min).

The liquid used for all the tests was drinking water from Terrassa City (located 25 km away from Barcelona, Spain). The heterotrophic plate count (HPC) test (or Standard Plate Count), applied in many variants, is the internationally accepted test to measure the heterotrophic micro-organism population in drinking water, and also other media (Bartram et al., 2003). Heterotrophs are organisms, including bacteria, yeasts and moulds, which require an external source of organic carbon to grow. The HPC test measures only a fraction of the micro-organisms actually present and does not distinguish between pathogens and non-pathogens. In our case, the HPC test carried out at 22 and 37°C on the tap water used in the experiments gave less than 1 $cfu \cdot m\ell^{-1}$ (colonies per mℓ). Therefore, there was no evidence of any living or active bacterial contents. Since permeability is the target function in this study, choosing tap water as fluid minimises the influence of fluid type in the results.

The experiments consisted of short-duration tests (24 h) and long-duration tests (up to 2 weeks). These periods allowed quality control of the membranes as well as determination of the fluctuation of permeability throughout the operation, taking into account the fouling of membranes.

Operating conditions were kept the same in all the membranes. Trans-membrane pressure was fixed to 1 bar. Cross-flow speed was 3.5 $m \cdot s^{-1}$. The usual range for cross-flow speeds for MF is 2 to 6 $m \cdot s^{-1}$ (Gan, 1999; Bendick et al., 2004). Generally, at high flow rates (close to 6 $m \cdot s^{-1}$) fouling is reduced because the same flow helps to sweep out particles settled on the membrane.

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TABLE 1
Membranes studied

Membrane	Source	Support material	Filtering layer material	Pore size (µm)	Section (outer diameter or side, cm)	Filtration surface (m^2)
Membralox (19 channels)	US Filter	Alumina	Alumina + zirconium oxide	0.1	Hexagonal (1.5)	0.0794
Atech (1 channel)	Atech Innovations	Alumina	Rutile	0.2	Circular (2.5)	0.0163
T1 (1 channel)	own	Tabular alumina	Rutile	0.1 - 0.15	Circular (2.0)	0.0134
T3 (1 channel)	own	Corindon	Rutile	0.1 - 0.15	Circular (2.0)	0.0134
T1 (7 channels)	own	Tabular alumina	Rutile	0.1 - 0.15	Circular (2.5)	0.0346
T3 (7 channels)	own	Corindon	Rutile	0.1 - 0.15	Circular (2.5)	0.0346

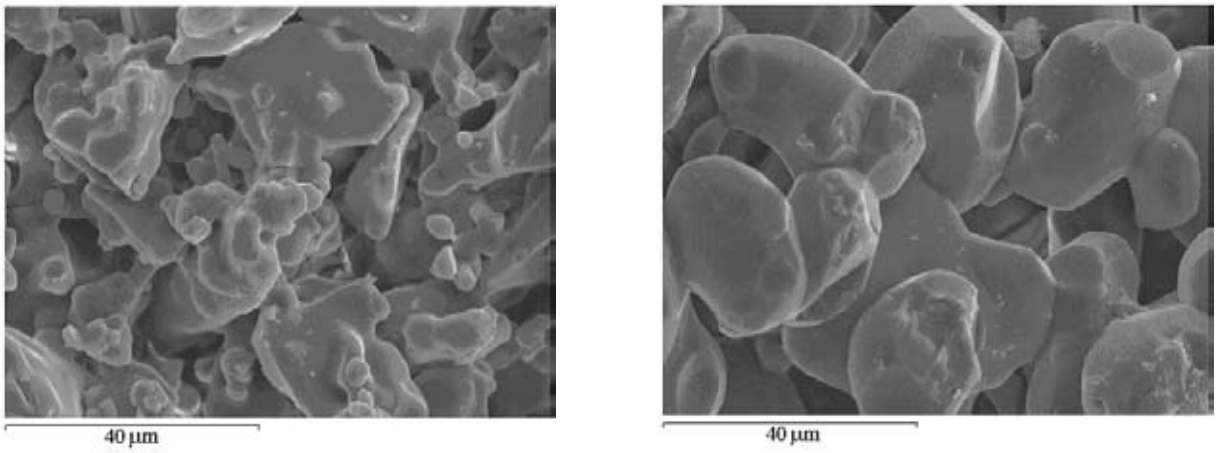

Figure 3
SEM pictures (1400x) of support microstructure of the two commercial membranes tested: a) Atech; b) Membralox/US Filter. Fine round particles can be observed.

In this experiment the highest speed was 3.5 $m \cdot s^{-1}$, due to the available pump power and the cross-section of membranes. As to pressure, 1 bar is a typical value for cross-flow MF (EPA, 2001). In preliminary tests, we observed that the higher the pressure reached, the higher the initial permeability achieved, but then it also decreased more rapidly since fouling was also greater, maybe due to fouling compaction. In most cases, the decrease was dramatic especially when the membrane tested was new.

Short-duration tests

The main goal of short tests, which were carried out in July 2004, was to ascertain the initial permeability of membranes. The plant operated for 24 h testing each membrane, and re-circulating about 250 ℓ of tap water (Terrassa City). The feed tank was refilled with freshwater before the start of each test.

The membranes tested were the following ones: Atech, Membralox/US Filter, experimental T1-1 and T3-1. For each membrane, at the beginning of each test and every hour during daytime, 1 ℓ of permeate was collected in a gradated tube. The time to fill the tube and temperature of water in the feed tank were also measured. Consequently, permeability could be calculated as $\ell \cdot h^{-1} \cdot m^{-2} \cdot bar^{-1}$. Once measured, the permeate was poured back into the feed tank.

After 24 h , each membrane was cleaned out with chemical products (NaOH 0.5 N and HCl 2%, in this order) replacing the feed tank with the corresponding cleaning tank. The duration of the cleaning process was 30 min with each chemical and 5 min with clean water between each cleaning. Then, the test continued for 4 additional hours in order to understand the ability of each membrane to recover its initial permeability.

Long-duration tests

Since the results obtained at the start of the short-duration tests varied considerably, tests conducted over longer periods were considered appropriate in order to compare the permeability of the membranes over long operating periods. As a result, we can safely say that the permeability of all membranes tested was very similar after a couple of hours operating.

The membranes tested were the experimental T1-7 and T3-7, and the two commercial Atech and Membralox/US Filter used in the short-duration tests.

These tests allowed the determination of the variation of permeability over a duration of not less than 370 h (15 d) for all the membranes, except for the membrane Atech, the test for which was conducted over 140 h (6 d) only.

The tests were carried out between November 2004 and January 2005. During this period, the plant was operating in continuous filtration mode throughout 24 h a day. After the first 24 h, each membrane was cleaned thoroughly as described under short-duration tests. Afterwards, the plant operated for 2 weeks (except for the membrane Atech that was tested for 6 d only).

As in short-duration tests, permeate flow rate was measured and corrected to take into account the effect of temperature.

Microstructure analysis

Some microphotograps by a scanning electron microscope with energy dispersive spectrometry (SEM-EDS) were taken of the

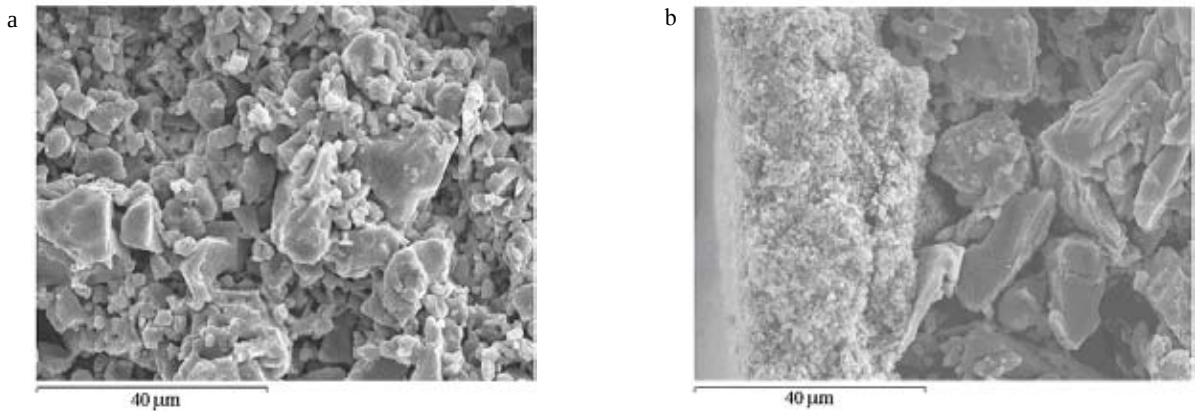

Figure 4
SEM pictures (1400x) of support microstructure of the two own experimental membranes tested: a) T1; b) T3. Industrial matter offers more rugged aspect.

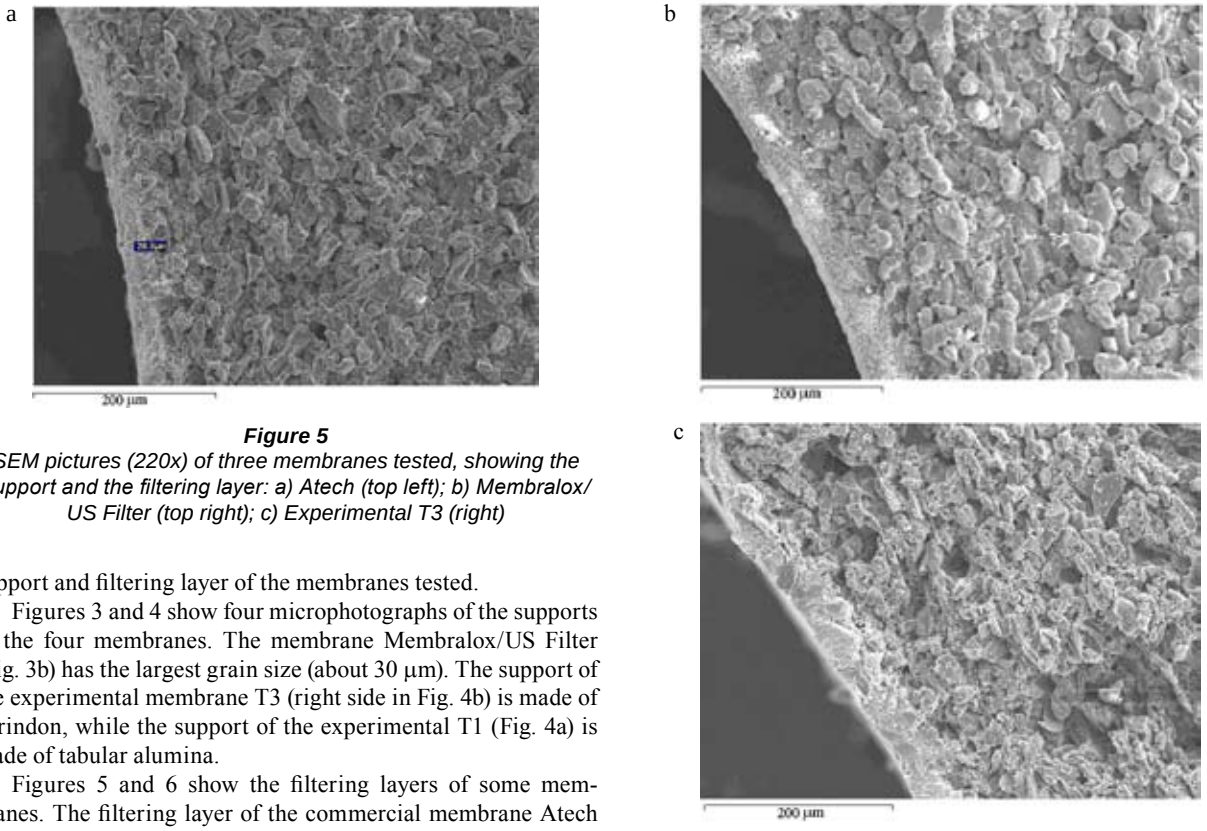

Figure 5
SEM pictures (220x) of three membranes tested, showing the support and the filtering layer: a) Atech (top left); b) Membralox/ US Filter (top right); c) Experimental T3 (right)

support and filtering layer of the membranes tested.

Figures 3 and 4 show four microphotographs of the supports of the four membranes. The membrane Membralox/US Filter (Fig. 3b) has the largest grain size (about 30 µm). The support of the experimental membrane T3 (right side in Fig. 4b) is made of corindon, while the support of the experimental T1 (Fig. 4a) is made of tabular alumina.

Figures 5 and 6 show the filtering layers of some membranes. The filtering layer of the commercial membrane Atech (Fig. 5a) seems to be about 30 µm thick and quite regular. The membrane Membralox/US Filter (Fig. 5b) has two filtering layers: an MF layer with a pore size of about 10 µm and thickness of 35 µm, approximately, and a UF layer with a pore size of 0.1 µm (7 µm thick, approximately). These two layers can be seen in greater detail in Fig. 5b. The thickness of the filtering layer of the experimental T3 (Fig. 5c) is about 45 µm.

Results and discussion

No bacteria, diatoms, etc. were detected in either feed water or permeate. Nevertheless, the mean turbidity of the feed water was 0.6 NTU. Reduction of turbidity was more than 80% in all membranes tested, without significant differences between them (always under 0.1 NTU). Total organic carbon (TOC) was also reduced by about 5% in all cases. There were no problems observed about durability or pathologies in the ceramic membranes.

As water re-circulates in the circuit, its temperature increases slightly due to the action of the pump. In MF and UF membranes, the permeate flow rate is inversely proportional to fluid viscosity (Chang and Benjamin, 2003), and it is well known that liquid water viscosity decreases with temperature. Then, the higher the temperature, the higher the permeability. According to literature, this increase can be estimated as 1 to 3.3% per ºC depending on the fluid (Wagner, 2001). Therefore, experimental permeability values should be corrected. Some results published in the literature (for instance, Li, 1972; Marsh and Eriksson, 1988; Pohland, 1988) offer certain expressions to estimate the effect of temperature on flow rate, referred to that for a given temperature (20 or 25ºC). After analysing them, the following expression was considered to be the most appropriate, according to Pohland (1988) and Wagner (2001)'s recommendations:

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TABLE 2
Data and results of the t-Student test for the temperature correction factor k = 0.0202, for a significance level of 0.05

Test no.	Experimental data				ΔT	P_1 / P_2	$(1+k)^{\Delta T}$
	P_1 ($\ell \cdot h^{-1} \cdot m^{-2} \cdot bar^{-1}$)	P_2 ($\ell \cdot h^{-1} \cdot m^{-2} \cdot bar^{-1}$)	T_1 (°C)	T_2 (°C)			
1	285	202	44.0	27.0	17.0	1.408	1.405
2	477	456	17.8	16.4	1.4	1.046	1.028
3	210	152	42.4	26.0	16.4	1.386	1.388
4	126	70	42.0	17.0	25.0	1.808	1.649
5	177	102	43.18	16.5	27.3	1.732	1.726
Average						1.476	1.439
Variance						0.093	0.075
Pearson's coefficient						0.978	
t value						1.194	
P(T ≤ t) one tail						0.149	
Critical value of t (one tail)						2.132	
P(T ≤ t) two tails						0.298	
Critical value of t (two tails)						2.776	

$$P_0 = P\,(1+k)^{(T_0 - T)} \qquad [1]$$

where:

P and P_0 are permeability rates (L h^{-1} m^{-2} bar^{-1}) at temperatures T and T_0, respectively

k is the correction factor

In our case, T_0 = 25 ºC and k = 0.0202.

This k-value was validated by a t-Student test where observed and calculated data obtained with water at different temperatures were compared. This test proved that there were no significant differences between the ratio of two measured permeability rates (for instance, P_1/P_2) and the calculated ratio following Eq. [1], i.e., $(1+k)^{\Delta T}$ (where $\Delta T = P_1 - P_2$). Table 2 shows the results of this statistical test for a significance level of 0.05.

The particle sizes of the support layers of the two commercial membranes (Figs. 3a and 3b) seem to be more regular than those of own membranes (Figs. 4a and 4b). Besides, particles in commercial membranes show rounder edges which may indicate that they have been sintered at high temperatures, modifying the morphology of the grains. Sintering of alumina particles with a diameter of 20 to 30 μm requires temperatures of higher than 1 800ºC (Klein and Hurlbut, 1996).

Although the membranes T1 and T3 were sintered at the same temperature (1 300ºC), the first one shows a more uniform pore distribution with greater pore size. At this temperature, rutile does not change its phase (Lynch et al., 1966; Richerson, 1992) and the porosity measured by weighing the water absorbed in a dried sample was 34% for the support T1 and 41% for T3 (Rodríguez-Grau, 2005). The image analysis of microphotographs confirmed these porosity values.

The filtering layer of the experimental T3 (Fig. 5c), made of rutile sintered at 1 000ºC, seems to adhere well to the support layer although its surface is a bit more irregular than that of the Atech membrane. Nevertheless, size and distribution of pores seem to be similar in both MF membranes (Fig. 6).

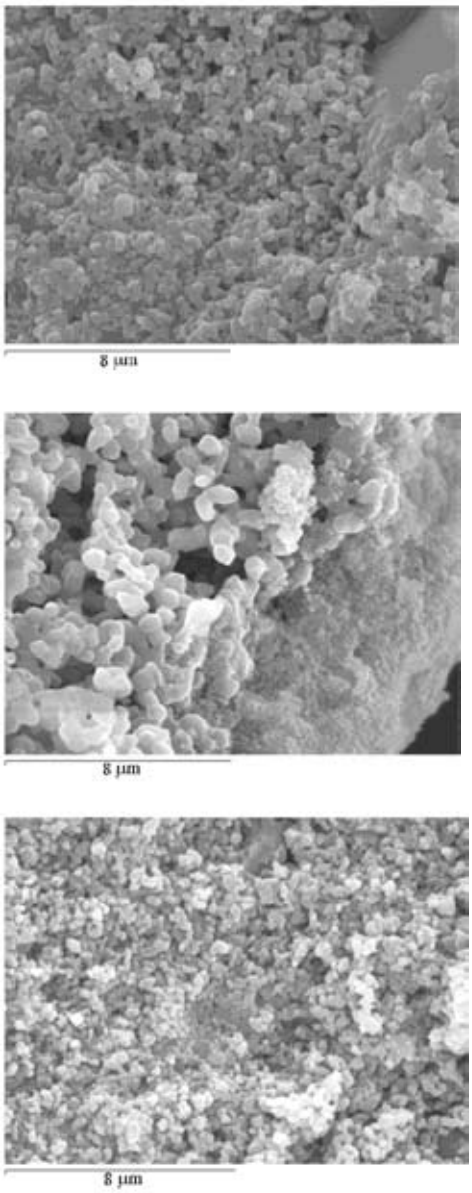

Figure 6 (right)
SEM pictures (7500x) of the filtering layer of three membranes tested: a) Atech; b) Membralox/US Filter; c) Experimental T3

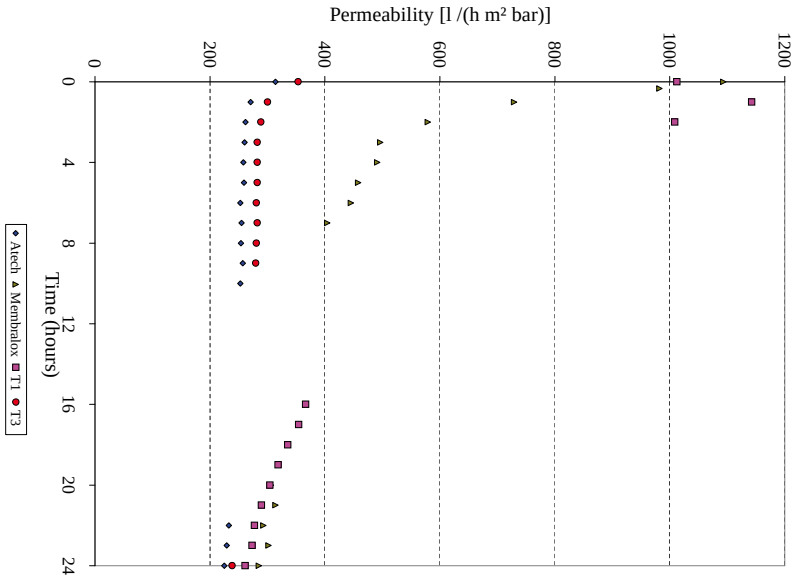

Figure 7
Comparative results of permeability rates with tap water for the 24 h test. Values have temperature correction.

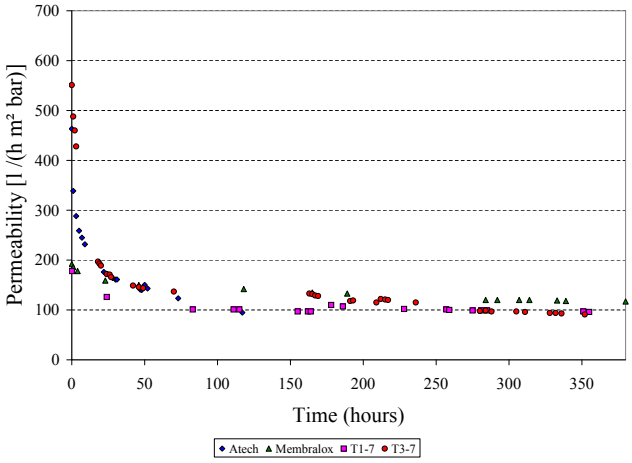

Figure 8
Permeability rates for the long tests, after filtration during 24 h and subsequent chemical cleaning. Values have temperature correction.

The permeability rates obtained from the 24 h test are shown in Fig. 7 for the four membranes. Initial values are high, but permeability rates decrease due to fouling throughout the test. This reduction is drastic during the first hours and much slower at the end of the test, by following a decreasing exponential curve. It can be also observed that initial values are quite different depending on the membrane (from 314 to 1 093 $\ell \cdot h^{-1} \cdot m^{-2} \cdot bar^{-1}$), but final values are very similar (225 to 285 $\ell \cdot h^{-1} \cdot m^{-2} \cdot bar^{-1}$). Final permeability rates for the mono-channel experimental membranes T1 and T3 range between those for the two commercial ones. After the last chemical cleaning, the four membranes recovered approximately similar rates (319 to 463 $\ell \cdot h^{-1} \cdot m^{-2} \cdot bar^{-1}$), although they were much lower than those at the start of the test, when the membranes were new and clean. All the values decay in a similar way tending to reach equilibrium after time.

In the case of long tests (Fig. 8), time = 0 corresponds to the beginning of filtration after the chemical cleaning. Initial permeability values are more similar between them, although in general they are lower (190 to 550 $\ell \cdot h^{-1} \cdot m^{-2} \cdot bar^{-1}$) than those obtained in the short-duration tests. The low value obtained for the membrane Membralox/US Filter is noticeable. In our opinion, this may be due to an incomplete cleaning cycle before the start of the test. This membrane has the largest surface area per unit length (see Table 1), so the chemical cleaning procedure adopted here would not be sufficient compared to the other membranes.

All the membranes show a similar behaviour as regards permeability rates, which can be fitted reasonably well to a decreasing exponential curve. Equations of the corresponding fitted curves are:

Membralox/US Filter:	$P = 171.7\ t^{-0.0553}$	($R^2 = 0.83$)
Atech:	$P = 287.2\ t^{-0.1686}$	($R^2 = 0.86$)
Experimental T1-7:	$P = 137.4\ t^{-0.0587}$	($R^2 = 0.91$)
Experimental T3-7:	$P = 358.8\ t^{-0.2028}$	($R^2 = 0.83$)

where:

P is permeability rate ($\ell \cdot h^{-1} \cdot m^{-2} \cdot bar^{-1}$)

t is time (hours)

By extrapolating these curves, it can be observed that membranes Atech, T1-7 and T3-7 tend to a permeability rate of 100 $\ell \cdot h^{-1} \cdot m^{-2} \cdot bar^{-1}$, approximately, whereas the membrane Membralox/US Filter tends to a value of about 120 $\ell \cdot h^{-1} \cdot m^{-2} \cdot bar^{-1}$ (after 400 h of filtration).

Higher performance of the membrane Membralox/US Filter can be explained by its multilayer filter (see Fig. 5b), which on the one hand improves the surface finish (decreasing the possibility of fouling), but on the other hand increases the manufacturing cost.

Conclusions

The main goal of this study has been to compare the performance during cross-flow operation of three types of microfiltration ceramic membranes (Atech, Membralox/US Filter and experimental). In contrast to commercial membranes, the experimental ones were produced from standard industrial raw materials and with low-cost sintering processes (1 300°C). Differences between the three types of membranes in permeability values, fouling rates and durability are the determining factors in the assessment of a possible industrial process in order to obtain low-cost ceramic membranes. It can be concluded that:

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- The membranes tested have similar performances during cross-flow operation, although permeability rates for the membrane Membralox/US Filter were about 15% higher
- The filtering layer seems to play a dominant role in permeability depending on the mineralogical and morphological features of the support layer
- The superior performance of the membrane Membralox/US Filter may be due to the presence of a second filtering layer of ultrafiltration (0.01 μm) of 10 μm width, which would contribute to a decrease in the thickness of the gel layer formed in operation.

The co-adjuvant effect of rutile combined with alumina and/or corindon, allows operation at relatively low temperatures because the experimental membranes are synthesised (for both support and filtering layer). Therefore the cost-savings are double: the investment cost is lower for the furnace and also the energy costs are lower. We estimate that the energy savings are about 30%. Furthermore, the ceramic mixtures used allow for extrusion at low pressure using standard equipment. In short, the experimental membranes can be manufactured by using the current equipment of a conventional ceramic factory.

Therefore, it would be possible to obtain low-cost ceramic membranes from industrial raw materials through low-cost industrial processes. Filtration plants that would operate with these membranes could have comparable performance and durability to the ones with commercial membranes. In addition, the use of ceramic membranes could be extended to applications which are presently not feasible, mainly because of their high costs (residual water, skimmed milk, etc.).

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