ELSEVIER

Journal of Food Engineering 54 (2002) 95–102

JOURNAL OF FOOD ENGINEERING

www.elsevier.com/locate/jfoodeng

Comparative study of crossflow microfiltration with conventional filtration of sherry wines

V.M. Palacios *, I. Caro, L. Pérez

Department of Chemical Engineering, Food Technology and Environmental Technology, Faculty of Science, University of Cádiz, Campus Río San Pedro, Apdo.40, Puerto Real, 11510 Cádiz, Spain

Received 11 May 2001; accepted 25 September 2001

Abstract

The present work reports, firstly, on the application of the gel-polarisation model to the crossflow microfiltration of sherry wines and brandies, in order to calculate the model parameters. In a preliminary set of experiments, the influence of the transmembrane pressure (TP) on the filtrate flow was studied, at several feed flows. The obtained results show a constant ratio between the optimum TP (TP_{opt}) and the feed flow of 32×10^{-4} l h^{-1} Pa with a TP_{opt} of 11×10^4 Pa, for a feed flow of 360 l h^{-1} for all products. The obtained values of D for the different products can indicate that the average size of the particles in the gel layer is from 10 to 50 nm. Secondly, it has compared crossflow microfiltration process with classic filtration processes, which are used in the standard production system. The results show that the microfiltration has a higher effectiveness than conventional filtration, except in the case of brandy, where conventional filtration is more effective.

Keywords: Crossflow microfiltration; Filter aid filtration; Submerged filtration; Sherry wines; Sherry brandies

1. Introduction

In the last 10 years there has been a general interest in the application of crossflow microfiltration in winemaking (Flores, Heatherbell, & McDaniel, 1990; Mafart & Béliard, 1991; Sánchez-Pineda & Alcain, 1996). Generally, under optimum conditions, crossflow microfiltration can be used satisfactorily in all clarification and stabilisation steps of industrial winemaking, such as clarification of must settlings, clarification of wine lees or partially fermented products lees, microbiological stabilisation of must and physical–chemical stabilisation of wine (Irrmann, 1992).

In membrane filtration, particle retention limits the duration of the filtration cycles by cumulative fouling, consequently the feed fluid should arrive under good filtration conditions; that is to say, not very high in fouling materials. Moreover, if the feed stream contains a high concentration of solids ($> 0.5\%$), the use of microfiltration is recommended in the crossflow configuration (Bertoul & Zanazza, 1986; Eykamp, 1995).

In microfiltration and ultrafiltration, the more common type of membrane is composed of organic polymers (cellulose acetate, polyethylene, polyamides or polysulphone) (Brun & Shaetzel, 1992). However, inorganic membranes of the ceramic type are being applied more today due to their high resistance to chemical degradation (Achotegui, 1993; Larbot, Guizard, Julbe, & Cot, 1992). The main inconvenience of these is their low deformability that makes them mechanically fragile (Renner & Abd-El-Salam, 1991).

Studies in enology recommend the use of microfiltration membranes above those of ultrafiltration, since the latter can reduce some colloidal and phenolic fractions of wine that have a positive influence on the sensorial characteristics of the product, besides giving low filtration yields (Flores, Heatherbell, Henderson, & McDaniel, 1991; Goodwin & Morris, 1991; Peri, Riva, & Decio, 1988). Crossflow microfiltration of wine gives a superior quality of clarification to other filtration techniques (conventional filtration), mainly from the point of view of microorganisms retention. Moreover, microfiltration does not modify in a significant way the sensory qualities of wines in comparison to the common techniques of clarification (Feuillat, Peyron, & Berger, 1987; Serrano, Pontens, & Ribereau-Gayon, 1992).

* Corresponding author. Fax: +34-956-016-411.
E-mail address: victor.palacios@uca.es (V.M. Palacios).

0260-8774/02/$ - see front matter
PII: S0260-8774(01)00189-3

In the microfiltration process, flux decline is caused by concentration polarisation and fouling. Attempts to analyse the fouling phenomenon have shown that the main factors are adsorption of some feed components, clogging of the pores, deposition of solids (Bauser, Chmiel, Stroh, & Walitza, 1989) and chemical interaction between membrane material and colloidal components of the wine (Belleville, Brillouet, Tarodo de la Fuente, Saulinier, & Moutonnet, 1991).

In most of the studies, theoretical calculations in microfiltration have been based on the gel-polarisation model, that uses the relationship among the dimensionless numbers *Sh*, *Re* and *Sc* (Dequian, 1987; Hernández, Tejerina, Arribas, Martínez, & Martínez, 1990; Metref, 1995; Shulz & Ripperger, 1989). The model implies the calculation of the overall mass transfer constant (k), as well as the solute concentration in the gel layer (C_g). This model was demonstrated to be an effective tool in equipment scale-up (Doosnar & Scholz, 1995).

The present work treats, in the first place, the application of the gel-polarisation model to the crossflow microfiltration of sherry wines and brandies, in order to calculate the parameters of the model in each case. In second place, it compares the crossflow microfiltration process with classic filtration processes, which are used in the standard production system.

2. Materials and methods

2.1. Crossflow microfiltration system

A pilot crossflow microfiltration system was used for the laboratory experiments and in the industrial plant. The equipment used was a Pellicon Cassette® unit from Millipore (Bedford, USA). This consists of a filtration cell or cassette, a peristaltic pump and two pressure gauges; one located at the feed entrance and the other at the retentate exit.

Twenty microporous membrane films of polyvinylidene difluoride (PVDF) constituted the filtration cassette. The filtration working surface was 0.46 m^2 and the nominal pore size was 0.45 μm. The maximum limit of the transmembrane pressure (TP) that can be reached in the filtration cell is 7 bar and the range of working temperatures is from 4 to 50 °C. The recommended flow across the system was approximately 10^{-4} m s^{-1}.

2.2. Conventional filtration systems

In the industrial experiments using conventional filtration, five different types of filters were used: two sand filters, one plate and frame filter and two cartridge filters. The sand filters utilised a filter aid and were a horizontal leaf filter (HLF) and a tubular candle filter. The cartridge filters utilised submerged filtration and were a coil filter and a tubular filter.

The HLF had a vertical case and a filtration working surface of 2.5 m^2, for an average production of 40 hl h^{-1} of wine (H-1® model, from Echo, Madrid, Spain). This unit incorporates a centrifugal discharge for the removal of the filter cake. Diatomaceous earth of permeability 1.20 Darcy was used as the filter aid at 1 g l^{-1} concentration in the pre-coat formation, and diatomaceous earth of permeability 0.06 Darcy was during filtration at a concentration of 2.5 g l^{-1} in the feed. This sort of filter is commonly used in the filtration of sherry wines of the ''cream'' type and the ''fino'' type.

The tubular candle filter (TCF) used was a Metafilter® unit, from TFB (Madrid, Spain). The pre-coat consisted of 300 g m^{-2} of earth, for an average production of 40 hl h^{-1} of wine. In this case, cellulose fibre and diatomaceous earth with a permeability of less than 1.50 Darcy were used as filter aids in the pre-coating. During filtration, white diatomaceous earth with a permeability of 0.90 Darcy was used in the feed. This type of filter is commonly used in the filtration of sherry brandies.

The plate and frame filter (PFF) used 20 plates of 40×40 cm^2, made from different white cellulose materials (obtained from pine or beech) and purified kieselgur. These plates are specifically dedicated to sterilisation filtration and to the retention of very fine particles. The maximum recommended working flow is 6.5 m^3 h^{-1}.

The submerged coil filter (SCF) was composed of a coiled film, mainly of polypropylene, polyester and thermally purified glass fibre. The equipment used was a Filterite® unit from TFB and the nominal pore size of the film was 3 μm. Each cartridge had a filtration surface of 0.55 m^2 and a flow capacity of 5 hl h^{-1}, so eight cartridges were used in the unit.

The submerged tubular filter (STF) was a tubular cartridge, composed of polypropylene only, with a 1 μm of nominal pore size. The equipment used was a Polygard® unit from Millipore. The filtration surface and the flow capacity were the same as those of the previous unit. Normally, these two industrial filters are used together in series.

2.3. Filtration experiments

For all the crossflow microfiltration and conventional filtration experiments, industrial stocks of sherry wines and sherry brandies were used. The stocks correspond to three tanks of a typical plant of the sherry zone, and consist of three types of products: sherry wine of the ''fino'' type, sherry wine of the ''cream'' type and sherry brandy. These products were chosen due to their different physico-chemical characteristics. The analytical data of these products are shown in Table 1.

Table 1
Characteristics of the three types of liquids filtered in the experiments

	"Fino" wine	"Cream" wine	Brandy
pH	3.31	3.35	4.00
Alcohol grade (v/v)	15.4	17.5	40.3
Sugar content (g l^{-1})	0	150	50
Titratable acidity (gTH_2 l^{-1})[a]	5.34	5.70	0.00
Volatile acidity (gTH_2 l^{-1})[a]	0.27	1.12	0.00
Density (kg m^{-3})[b]	987.5	1032.1	951.2
Dynamic viscosity (Pa s)[b]	1.5×10^{-3}	1.9×10^{-3}	1.8×10^{-3}

[a] TH_2 = tartaric acid.
[b] Measured at 25 °C.

Several crossflow microfiltration experiments were carried out in order to determine the optimum working conditions of the microfiltration system, for each type of product to be filtered. Experiments were performed at different TPs (10×10^{-4}–30×10^{-4} Pa), feed flows (300–500 l h^{-1}), tangential crossflow velocity (3–5 m s^{-1}) and temperature of 25 °C. Considering the technical specifications of the used equipment, it was decided to operate in all the crossflow microfiltration experiments at a feed flow of 360 l h^{-1}, which is the recommended flow in this equipment for crossflow microfiltration.

A complete set of microfiltration experiments was carried out under the optimum operating conditions for each one of the three types of products, to determine the values of the parameters of the gel-polarisation filtration model.

Finally, sets of conventional filtration experiments were run with the three products, in order to compare the effectiveness of the filtration techniques in each case. The conventional filtration used for the "fino" sherry wine was the HLF. For the "cream" sherry wine, the HLF followed by the plate and frame filter was used (HLF + PFF). For the sherry brandy, conventional filtration utilizes the TCF, followed by the SCF, and followed by the STF (TCF + SCF + STF). All these experiments were run at an industrial level at the operating conditions, which have been described before for each system.

Each filtration experiment consisted of a continuous filtration session of 3 h, while samples of the feed stream and filtrate stream were taken periodically (10 min). At the end of each experiment, all the samples were mixed and then submitted to the physico-chemical analysis indicated below.

2.4. Theoretical approach of the gel-polarisation model

Once the working pressure and the feed flow have been fixed, according to the gel-polarisation model, the specific flow of filtrate (J) is a function of the solids concentration in the feed stream (C_i). Both variables are related through the mass transfer constant of the system (k), according to the following equation (Mulder, 1995):

$$J = k \operatorname{Ln}\left(\frac{C_g}{C_i}\right). \tag{1}$$

The specific flow is the volumetric flow divided by the working surface, so the k constant has dimensions of velocity (m s^{-1}). Another constant of the system is the so-called solids concentration in the gel layer (C_g), which depends on the operating conditions. As can be observed in Eq. (1), if the solids concentration in the feed stream is higher than the C_g value, there is a certain filtrate flow, but if solids in the feed streams are more concentrated than this value, there will be no effective filtration.

In accordance with the gel-polarisation model, the following adimensional equation can be used for the determination of D (Dequian, 1987; Hernández et al., 1990):

$$Sh = A\,Re^{\alpha}\,Sc^{\beta}, \tag{2}$$

where Sh is the Sherwood number, Re is the Reynolds number and Sc is the Schmidt number. The letters A, α and β are constants which depend on the geometry and hydrodynamics of the system. As is well known, the definition of these tree numbers is

$$Sh = \frac{kd}{D}, \quad Re = \frac{u\rho d}{\mu}, \quad Sc = \frac{\mu}{\rho D}, \tag{3}$$

where k is the overall mass transfer coefficient of solids from the gel layer to the circulating fluid (m s^{-1}); D is the diffusion coefficient of the solids through the gel layer (m^2 s^{-1}); "d" is the hydraulic diameter of the circulation channel (m); "u" is the linear velocity of the fluid into the channel (m s^{-1}); ρ is the density of the fluid (kg m^{-3}); and μ is the dynamic viscosity of the fluid (Pa s). For rectangular circulation channels, the hydraulic diameter can be calculated as $2ab/(a+b)$, where "a" is the width and "b" is the height of the channel.

Taking into account that the linear velocity of the fluid into the circulation channel is around 4 m s^{-1} and the hydraulic diameter of the channel is around 0.4 mm, for the microfiltration equipment used, the flow conditions of the experiments are clearly laminar flows (with Re from 900 to 1200). As a consequence, under the

experimental conditions, the values of the constants of Eq. (2) given by Grober can be used (Eykamp, 1995) (Mulder, 1995). These values are the following:

$$A = 0.664(d/b)^{1/2}, \quad \alpha = 1/2 \text{ and } \beta = 1/3.$$

Rearranging Eqs. (2) and (3), introducing the Grober constant and detaching D, the following expression can be obtained:

$$D = \left(\frac{k^6 b^3 \mu}{(0.664)^6 u^3 \rho} \right)^{1/4}. \tag{4}$$

The data for D for the different filtered products which are shown in Table 2 have been calculated by means of Eq. (4).

Since the diffusion coefficient is inversely proportional to the molecular diameter of the diffusing compound, it is possible to estimate the average molecular size of solids in the gel layer. According to the well-known Stokes–Einstein equation for spherical particles under diffusion (Mulder, 1995), this relationship is as follows:

$$D = \frac{\kappa T}{3\pi \Phi \mu}, \tag{5}$$

where "κ" is the Boltzmann constant (J K^{-1}); T is the working temperature (K); Φ is the diameter of the particle (m); and μ is the fluid viscosity (kg m^{-1} s^{-1}).

Moreover, the average molecular weight of the solid constituents in the gel layer can be estimated if we consider the spherical shape of molecules and use an empirical relationship as the following:

$$Mw = z\Phi^3. \tag{6}$$

Considering an organic nature of solids in the gel layer we can take $z = 6 \times 10^{29}$ g mol^{-1} m^{-3} (Stryer, 1998).

Secondly the value of C_g can be used to estimate the rejection volume of the microfiltration process and so the operating cost of the microfiltration unit applied to each type of product.

Normally, the microfiltration equipment operates on an industrial scale in a semi-continuous mode; that is, the feed stream of the unit goes from a feed tank, which also takes in the rejection stream of the filter. Thus, the concentration of solids in the tank becomes higher as the process progresses. The cycle ends when the concentration of solids in the tank is approximately C_g, because the filtrate flow is then much too low to keep the process running. The rejection (R) is defined as the fraction of the initial volume of liquid that remains in the tank at the end of the cycle. This volume is considered a yield loss, though it is normally recycled to a coarse filtration process.

If we start a cycle with a concentration of solids in the tank C_0 and a volume of liquid to be filtered V_0, and also the final concentration of solids is C_f and the volume is V_f, then the rejection can be calculated based on a solids balance into the tank as follows, assuming that there are no solids in the filtrate stream:

$$C_0 V_0 = C_f V_f, \quad R = \frac{V_f}{V_0} = \frac{C_0}{C_g}. \tag{7}$$

2.5. Analysis of samples

The following physico-chemical analyses were performed on the samples collected:

- Total solids in suspension (TSS), by gravimetric method, using membranes of cellulose acetate with a 0.45 μm of pore size (APHA, AWWA, & WPCF, 1992, Chap. 2).
- Colour index (CI), by determination of absorbance at 470 nm in a spectrophotometer (Terry, 1973).
- Density, using an automatic densimeter model DMA-48 from Anton Paar (Graz, Austria), with a thermostat.
- Dynamic viscosity, by a ball viscosimeter of the Höppler type model B/BH from Haake (Berlin, Germany), fitted with a thermostat.
- Fouling index (FI), according to the method of Descout, Bordier, Laurenty, and Guimberteau, 1976.
- Protein index (PI) by the method of Bayly and Berg, which consists in measuring the sample absorbance at 607 nm, 15 minutes after treating with a phosfomolybdic reagent (García-Barceló, 1990, Chap. 9).
- Particle size distribution (PSD), by automatic counting in a laser counter model HSP8 from Pamas (Boulder, USA), with sensor model HCB-LD-21/23SC, and sensitive in the size range from 0.5 to 100 μm.

Table 2
Microfiltration parameters of the three types of liquids filtered in the experiments

	"Fino" wine	"Cream" wine	Brandy
k (10^{-6} m s^{-1})	30.9	68.0	45.9
C_g (g l^{-1})	46.3	0.4	3.5
D (10^{-12} m^2 s^{-1})	6.3	21.6	11.9
Φ (nm)	46.4	10.3	20.6
Mw (MDa)	60.0	0.7	5.2
Rejection (%)	0.2	24.5	2.8

k: mass transfer constant; C_g: gel layer concentration; D: diffusion coefficient; Φ: particle diameter; Mw: average molecular weight.

- Sugar content, according to the method of Lorenz Miller (1959).
- Alcohol grade and volatile acidity were determined by the OIV standard volumetric method (O.I.V., 1978) and a Dujardin–Salleron type ebulliometer (Amerine & Ough, 1980), respectively.
- Titratable acidity was determined by titration according to the method of the American Society of Enologists (Amerine & Ough, 1980).
- pH was measured with a digital pH meter, equipped with a combined electrode.

3. Results and discussion

3.1. Preliminary experiments in crossflow microfiltration

The filtrate flow rate in a crossflow microfiltration system depends on the TP drop and, at optimum pressure, on the mass transfer coefficient of solids and solutes from the gel layer to the circulating fluid (k), since this is the principal resistance to filtration. This mass transfer depends on certain other factors of the system such as the diffusion coefficient of solids or solutes in the gel layer (D), the viscosity (μ) and density (ρ) of the fluid to be filtered, and the superficial circulation flow of the liquid over the membrane (u) (Eykamp, 1995). To optimise a specific crossflow microfiltration process it is necessary, in the first place, to establish the value of the optimum TP.

In a preliminary set of experiments, using the microfiltration equipment described above with the "fino" sherry wine, the influence of the TP on the filtrate flow was studied, at several feed flows. Results are shown in Fig. 1. Experimentally, a linear relationship is observed between the filtrate flow and the TP, until a certain pressure value, different for each feed flow. Beyond this point, the filtrate flow becomes constant and independent of pressure, being the maximum possible. This pressure point can be denominated as the optimum TP (TP_{opt}), since this condition offers the highest filtrate flow with the minimum pressure load and sufficient permeate quality (Mulder, 1995).

The obtained results of TP_{opt} for the equipment used at several feed flows show a constant ratio between the optimum TP (Pa) and the feed flow (l h^{-1}). The average value of this ratio is 32×10^{-4}. Thus, this value can be used to well estimate the optimum working pressure in each case, and can be related to the critical value of the ratio permeate flow–wall shear stress (1.0 l h^{-1} m^{-2} Pa^{-1}) found by Le Berre and Daufin (1996) for crossflow microfiltration of skim milk with a ceramic membrane.

For the feed flow of 360 l h^{-1} (recommended in this equipment) corresponds an optimum pressure drop of 11×10^4 Pa. To assure that these are the work conditions for the different types of filter feed liquids, a new set of experiments was carried out with the "cream" sherry wine and with the sherry brandy. The results are shown in Fig. 2. As it can be observed, the value of TP_{opt}

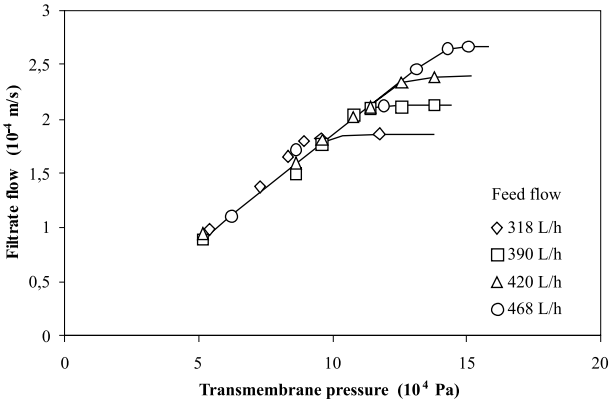


Fig. 1. Filtrate flow in crossflow microfiltration versus TP drop at several feed flows. The filtered product is "fino" sherry wine.

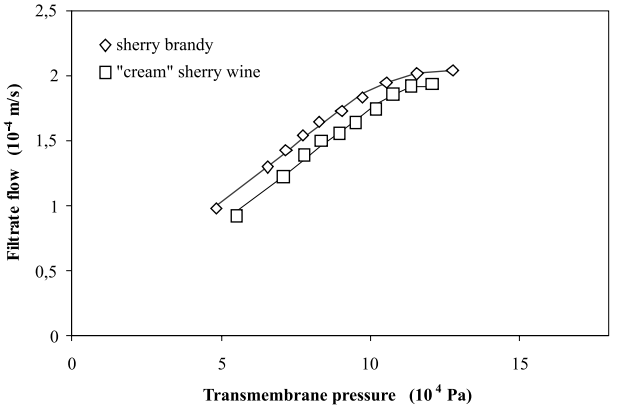
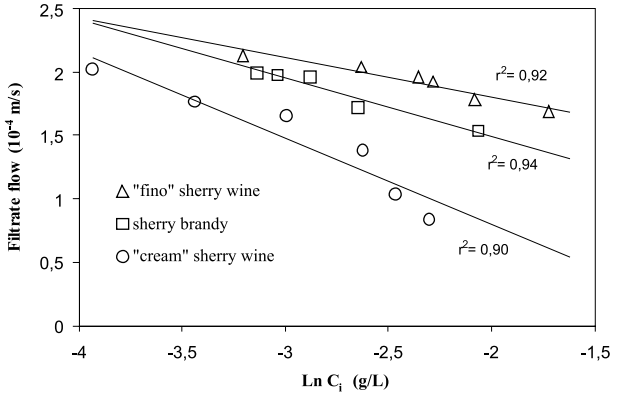


Fig. 2. Filtrate flow versus TP drop for crossflow microfiltration of two different products. The feed flow is 360 l h^{-1}.



C_i : solids concentration in the feed stream

Fig. 3. Specific filtrate flow (J) versus solids concentration in the feed stream (C_i), for crossflow microfiltration of several types of products.

Table 3
Total solids and fouling index (FI) in samples of sherry wine and sherry brandy, before filtration and after filtration

	Total solids				Fouling index					
	"Cream" wine		Brandy		"Fino" wine		"Cream" wine		Brandy	
	mg l^{-1}	%	mg l^{-1}	%	FI	%	FI	%	FI	%
FS	126		30		140		400		26	
CMF	20	15.9	14	46.7	1	0.7	1	0.2	2	7.7
CF[a]	35	27.8	10	33.3	40	28.6	27	6.8	4	15.4

FS: feed stream; CMF: crossflow microfiltration; CF: conventionally filtered; % in relation to the value of the feed stream.
[a] HLF + PFF for "cream" wine, TCF + SCF + STF for brandy and HLF for "fino" wine; HLF: horizontal leaf filter; PFF: plate and frame filter; TCF: tubular candle filter; SCF: submerged coil filter; STF: submerged tubular filter.

for a feed flow of 360 l h^{-1} is also around 11×10^4 Pa in both cases.

3.2. Theoretical calculations in crossflow microfiltration

The dependence of the specific filtrate flow (J) on the solids concentration differs for the three different types of products (Fig. 3, Table 2). Thus, it can be observed that the wine of the "fino" type is more readily filtered than the "cream" type and than the brandy.

The obtained k data can be used to estimate the values of the diffusion coefficient of solids through the gel layer (D). Moreover, the obtained values of D for the different products can indicate the nature of the filtered solids in each case. The data of Φ obtained by Eq. (5) are shown in Table 2. As it can be observed the average size of the particles in the layer is from 10 to 50 nm.

The molecular weights shown in Table 2 have been estimated using Eq. (6). As it can be observed, the solids involved in the formation of the gel layer consisted of molecules of small proteins (700,000 g mol^{-1}) to nucleic acids, large globules of polymers or cell components (60 MDa).

A set of microfiltration experiments in semi-continuous cycles was carried out in order to determine the rejection volume for each product under study. Several cycles were tried in each case, starting with a solids concentration of 0.1 g l^{-1} and ending when the filtrate flow was negligible. The average rejections obtained are shown in Table 2. As it can be observed, the experimental rejections agree well with those expected from Eq. (7). The "cream" sherry wine has a higher solids concentration in the gel layer than that of sherry brandy and so than that of "fino" sherry wine. Thus, the rejection volume is also higher in the case of "cream" wine and the industrial operation gives higher yield losses.

In conclusion, the behaviour of the microfiltration equipment at pilot or industrial scale for sherry wines can be well predicted by applying the gel-polarisation model and the proper design equations to the data of the operating conditions and using the calculated values of the mass transfer coefficient (k) and the solids concentration in the gel layer (C_g).

3.3. Comparison of crossflow microfiltration and conventional filtration

The comparative results obtained for the different filtration techniques in relation with TSS are shown in Table 3. As it can be observed, in the case of "cream" sherry wine, microfiltration has a higher effectiveness than conventional filtration but in the case of brandy, conventional filtration is more effective. These results are due to the three consecutive filtration steps applied conventionally in brandy and the lower solids concentration that usually exists in the feed stream of this product.

Furthermore, the effect of the particle size on the effectiveness of solids retention has been studied. The mass retention for each particle size in conventional filtration and crossflow microfiltration is plotted in Fig. 4. As is shown, a lower mass retention was found at smaller particle sizes. This is logically due to the selective effect of the nominal pore size of the membranes. However, the observed overall retention is different in each case. As was said previously, microfiltration of "cream" wine shows higher effectiveness, but conventional filtration of brandy is more effective.

The fouling index of a suspension is indicative of the quantity and nature of the particles present in the

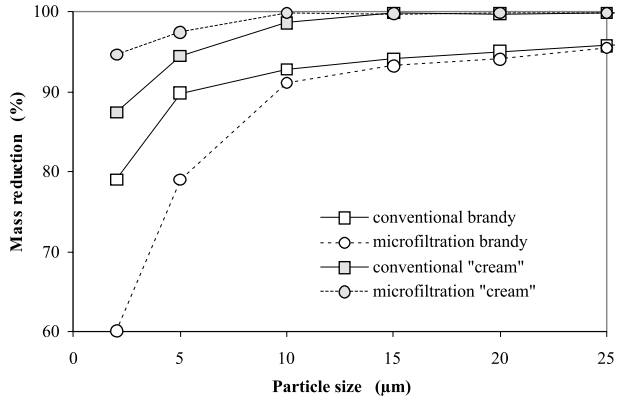


Fig. 4. Mass reduction at different particle sizes for crossflow microfiltration and conventional filtration of two different products.

Table 4
Protein index (PI) and colour index (CI) in samples of "cream" and "fino" sherry wines, before filtration and after filtration

	Protein index				Colour index					
	"Cream" wine		"Fino" wine		"Fino" wine		"Cream" wine		Brandy	
	PI	%	PI	%	CI	%	CI	%	CI	%
FS	2.3		1.6		0.058		0.101		0.063	
CMF	0.0	0.0	0.0	0.0	0.054	93.1	0.074	73.3	0.049	77.8
CF[a]	0.7	30.4	0.7	43.8	0.056	96.6	0.089	88.1	0.061	96.8

FS: feed stream; CMF: crossflow microfiltration; CF: conventionally filtered; % in relation to the value of the feed stream.
[a] HLF + PFF for "cream" wine, TCF + SCF + STF for brandy and HLF for "fino" wine; HLF: horizontal leaf filter; PFF: plate and frame filter; TCF: tubular candle filter; SCF: submerged coil filter; STF: submerged tubular filter.

sample. This value shows the volume of suspension that can be easily filtered. The results are shown in Table 3.

As is shown, after filtration, the fouling index descends rapidly all the cases and microfiltration reduces the index to a lower level than conventional filtration. This point is clear in the case of the "fino" wine and even more in the case of the "cream" wine, but it is not so clear for brandy. However, it must again be taken into account that the feed stream of brandy has even less fouling index than the other filtrates.

Another interesting filtration parameter in this type of product is the protein index, which should be the lowest possible to avoid later decay of the medium. The comparative results are shown in Table 4. As it can be observed, continuous microfiltration reduces the index, while conventional filtration reduces the index by a lesser amount.

The parameter colour index is also very important in relation with the sensorial properties of the products. The results are listed in Table 4. As is shown, crossflow microfiltration reduces the colour of the "cream" sherry wine and the sherry brandy by approximately 25%, while conventional filtration only reduces it by 12% and 5%, respectively. In the case of the "fino" wine, which has the lowest colour intensity, the effect is less marked. These data indicates that crossflow microfiltration produces an important retention of the colloidal compounds responsible for the colour of wines, higher than conventional techniques in all the cases.

Finally, the application of microfiltration techniques, instead of conventional filtration, can lead to a higher rationalisation of the solid wastes management of the filtration stage, due to the elimination of solid filtration residues such as diatomaceous earths, filtration plates, etc.

4. Conclusions

First of all, the calculated values of the crossflow microfiltration parameters for sherry wines and brandy lead to an average size of the particles in the gel layer from 10 to 50 nm. As a consequence, the molecular weight of the compounds involved in its formation can correspond to molecules from small proteins to nucleic acids, large globules of polymers or cell components.

Moreover, it has been shown that "cream" sherry wine has a higher solids concentration in the gel layer than sherry brandy and so than "fino" sherry wine. Thus, the rejection volume is also higher in the case of "cream" wine and the industrial operation gives higher yield losses.

Secondly, the microfiltration has a higher effectiveness than conventional filtration, except in the case of brandy, where conventional filtration is more effective. It has been shown that crossflow microfiltration confers higher physico-chemical stability than conventional filtration on sherry wines. In particular, the "fino" sherry wine shows better analytical parameters in the filtrates from crossflow microfiltration than those from conventional filtration. However, the "cream" sherry wine suffers an important colour reduction when it is microfiltered.

Finally, crossflow microfiltration has been shown to be an appropriate technique to substitute for conventional filtration, having many advantages.

References

APHA, AWWA & WPCF (1992). *Métodos normalizados para el análisis de aguas potables y residuales* (pp. 83–85). Madrid: Díaz de Santos.

Achotegui, A. (1993). Microfiltración con membranas cerámicas. *Ingeniería Química, 6*, 169–172.

Amerine, M. A., & Ough, C. S. (1980). *Methods for analysis of must and wines* (pp. 45–48). New York: Wiley.

Bauser, H., Chmiel, H., Stroh, N., & Walitza, E. (1989). Interfacial effects with microfiltration membrane. *Journal of Membrane Science, 11*, 321–326.

Belleville, M. P., Brillouet, J. M., Tarodo de la Fuente, B., Saulinier, L., & Moutonnet, M. (1991). Differential roles of wine colloids in the fouling of a cross flow microfiltration alumina membrane. *Kdie Wein-Wissenschaft, 46*, 100–107.

Bertoul, E. C., & Zanazza, P. (1986). La filtrazione in flusso tangenzialle. *Enotecnico, 2*, 199–204.

Brun, J. P., & Shaetzel, P. (1992). Membranes organiques. *Revue Française d'Oenologie, 137*, 77–91.

Dequian, R. (1987). Cleaning and regeneration of membranes. *Desalination, 62*, 363–366.

Descout, J. J., Bordier, J. L., Laurenty, J., & Guimberteau, G. (1976). Contribution à l'étude des phénomènes de colmatage lors de la filtration des vinssur filtre écran. *Connaissance Vigne Vin, 10*(1), 93–123.

Doosnar, M., & Scholz, T. (1995). Scaling up crossflow filtration processes for biotech manufacturing. *Genetic Engineering News, 9*, 20.

Eykamp, W. (1995). Microfiltration and ultrafiltration. In R. D. Noble, & S. A. Stern (Eds.), *Membrane Separation Technology. Principles and Applications* (pp. 1–40). London: Elsevier.

Feuillat, M., Peyron, D., & Berger, J. L. (1987). Influence de la microfiltration tangentielle des vins sur leur composition physicochimique et leurs caractères sensoriels. *Bulletin de l'OIV, 673–674*, 227–244.

Flores, J. H., Heatherbell, D. A., & McDaniel, M. R. (1990). Ultrafiltration of wine: effect of ultrafiltration on white riesling and gewürztraminer wine composition and stability. *American Journal of Enology and Viticulture, 41*(3), 207–214.

Flores, J. H., Heatherbell, D. A., Henderson, L. A., & McDaniel, M. R. (1991). Ultrafiltration of wine: effect of ultrafiltration on the aroma and flavor characteristics of white riesling and gewürztraminer wines. *American Journal of Enology and Viticulture, 42*(2), 91–96.

García-Barceló, J. (1990). *Técnicas analíticas para vinos* (pp. 19–22). Barcelona, Spain: GAB.

Goodwin, C. O., & Morris, J. R. (1991). Effect of ultrafiltration on wine quality and browning. *American Journal of Enology and Viticulture, 42*(4), 347–353.

Hernández, A., Tejerina, F., Arribas, J. Y., Martínez, L., & Martínez, F. (1990). *Microfiltración, ultrafiltración y ósmosis inversa* (pp. 43–78). Murcia, Spain: Universidad de Murcia.

Irrmann, R. (1992). Aplications en œnologie. *Revue Française d'Oenologie, 137*, 17–19.

Larbot, A., Guizard, C., Julbe, A., & Cot, L. (1992). Matériaux et membranes 1: les membranes minérales. *Revue Française d'Oenologie, 137*, 65–75.

Le Berre, O., & Daufin, G. (1996). Skim milk crossflow microfiltration performance versus permeation flux to wall shear stress ratio. *Journal of Membrane Science, 117*(1–2), 261–270.

Lorenz Miller, G. (1959). Use of Dinitrosalicylic acid reagent for determination of reducing sugars. *Annalitical Chemistry, 31*(3), 426–428.

Mafart, P., & Béliard, E. (1991). *Génie industriel alimentaire* (Vol. II) (pp. 165–172). Paris: Technique et Documentation Lavoisier.

Metref, S. (1995). *Contribution à l'étude expérimentale, à la modélisation et á la simulation du couplage fermentationlmicrofiltration tangentielle* (pp. 145–156). Thesis, École Centrale du Paris, Paris.

Mulder, M. H. V. (1995). Polarization phenomena and membrane fouling. In R. D. Noble, & S. A. Stern (Eds.), *Membrane separation technology. Principles and applications* (pp. 45–82). London: Elsevier.

Office International de la Vigne et du Vin (O.I.V.) (1978). *Recueil des methodes internationales d'Analysis des vins* (4th ed., pp. 75–79). Paris: OIV.

Peri, C., Riva, M., & Decio, P. (1988). Crossflow membrane filtration of wines: comparison of performance of ultrafiltration, microfiltration, and intermediate cut-off membranes. *American Journal of Enology and Viticulture, 39*(2), 162–168.

Renner, E., & Abd-El-Salam, M. H. (1991). *Ultrafiltration membranes in application of ultrafiltration in the dairy industry* (pp. 30–46). London: Elsevier.

Sánchez-Pineda, M. T., & Alcain, F. J. (1996). Técnica de filtración tangencial y su aplicación en la ingeniería enológica. *Alimentación Equipos y Tecnología, 12*, 105–109.

Serrano, M., Pontens, B., & Ribereau-Gayon, P. (1992). Étude de différentes membranes de microfiltration tangentielle. Comparaison avec la filtration sur précouche de diatomées. *Journal International des Sciences de la Vigne et du Vin, 26*(2), 135–141.

Shulz, G., & Ripperger, S. (1989). Concentration polarization in crossflow microfiltration. *Journal of Membrane Science, 40*, 173–187.

Stryer, L. (1998). *Biochemistry* (Vol. I) (3rd ed., pp. 71–90). New York: Freeman.

Terry, J.C. (1973). *Contribución al estudio de los compuestos polifenólicos en vinos de la Denominación de Origen Jerez-Xérèz-Sherry* (pp. 123–134). Thesis, Universidad de Sevilla, Sevilla, Spain.