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## Use of a Helical Baffle for Red Wine Clarification on a Mineral Membrane

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### ABSTRACT

We present the use of a helical baffle inserted in a mineral membrane (Carbosep) for the clarification of a highly charged red wine. Baffles of different geometries were made of stainless steel by winding a steel wire on a rod. The baffles were centrally placed. The wine was analyzed before and after its clarification for its filtration index, turbidity, color, and microbiological control. Experiments made at different transmembrane pressures and feed flow rates show that the permeate flux increased from  $13 \text{ L/h}\cdot\text{m}^2$  (without baffle) to  $30 \text{ L/h}\cdot\text{m}^2$  (with baffle). Long-term experiments at the same hydraulic dissipated energy gave a mean permeate flux of about  $20 \text{ L/h}\cdot\text{m}^2$  from a baffled membrane compared to  $10 \text{ L/h}\cdot\text{m}^2$  for a membrane without a baffle. The volume of permeate collected during the same time was 145% more for a baffled membrane. It was found that membrane fouling due to polarization concentration was reduced by a factor of 3 with the use of baffles. Analysis of permeate at three optical densities (420, 520, and 620 nm) and turbidity measurements confirm that the quality of the permeate was good. It is concluded that the presence of a baffle in the membrane did not change the characteristics of the filtered wine and that its use is very simple for the enhancement of permeate flux.

### INTRODUCTION

The red wine obtained after the fermentation of grape juice is normally cloudy and contains different types of suspended particles. Young and

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fermented wines contain insoluble pulp particles, insoluble proteins and protein-tannin compounds, active enzymes, live and dead microorganisms, and sometimes salt compounds of low solubility in a crystalline structure as well as other substances of a partly unknown nature in suspended forms (1). In addition, they contain invisible colloids (neutral polysaccharides and acidic polysaccharides) that can aggravate the clarification process (2). Various physicochemical processes and clarification on kieselguhr are used to stabilize these wines before their consumption (3). The content of high molecular compounds and conglomerated substances (colloids) even for optically clear beverages show different filtrabilities ( $V_{max}$ ) (2). Crossflow microfiltration (CFMF) using polymeric and mineral membranes are widely used for the clarification of raw wine (4–6). However, large-scale industrial applications of membranes are still limited because of very low permeate flux and because of the problems which come from frequent membrane cleaning. In general, the permeate flux decreases to a very low value compared to the feed flow in a short filtration time.

In a recent paper (7) we investigated the effect of physical parameters on the microfiltration of red and white wines on a flat polymeric membrane (PVDF). It was found that 0.4  $\mu\text{m}$  pores gave the best compromise between turbidity and flux requirements. With red wine the permeate flux was found to be almost independent of fluid velocity but to increase linearly with transmembrane pressure, reaching up to 50  $\text{L}/\text{h}\cdot\text{m}^2$  at 3 bar. The turbidity was below 0.5 NTU (7). Electrodialysis technique was also used for the stabilization of tartaric acid and deacidification. Different membrane separation techniques (microfiltration, pervaporation, reverse osmosis, and electrodialysis) were compared by Escudier et al. 1988 (8) for their overall quality. A comparative study showed that the clarity and stability of wine with ultrafiltration and traditional filtration is about the same, but the soluble colloids content is strongly reduced by ultrafiltration, which may unfavorably affect taste and tartrate stability of red wine. Some different techniques such as superimposition of pulsating flow on the feed flow and intermittent backwash or backflush have been used to enhance the permeate flux rate and also to control membrane fouling and permeate flux decrease with time (9, 10). The use of turbulence promoters (baffles) or inserts in the tubular membrane was recently reported (11, 12). These promoters can increase both the wall shear rate and mixing of the fluid at the membrane surface.

In this work we present experimental results on the clarification of raw red wine using mineral membranes and helical-shaped inserts. The enhancement in permeate flux by using an insert and by not using an insert at a constant feed flow or at a constant hydraulic dissipated energy has been determined. At a constant feed flow rate the permeate flux increases

for membranes equipped with inserts because of the increase in flow velocity as well as by the turbulence created in the flow field by the presence of the insert, but the hydraulic energy consumption also increases at the same time. In order to justify the use of a turbulence promoter for permeate flux increase, we need to keep the hydraulic dissipated energy constant, i.e., adjusting the feed flow rate and the pressure drop simultaneously. For each experiment the quality of filtrate was analyzed for its turbidity,  $V_{\max}$ , color, and microorganisms.

## MATERIAL AND METHODS

A small filtration unit was assembled for the clarification of red wine (Fig. 1). The raw wine was pumped by a peristaltic pump at flow rates of less than 1 L/min. The filtrate was not returned to the feed reservoir as it was a small quantity compared to the initial volume of the raw wine used, so the concentration and quality of feed was assumed to be constant. The transmembrane pressure,  $\text{TMP} = (P_1 + P_2)/2 - P_f$ , was regulated by the control valve placed on the retentate side. The temperature of the wine was kept constant at 20°C.

Mineral membranes Carbosep (membrane layer of zirconium oxide on carbon support) of different pore sizes were used. These membranes were 40 cm long and 6 mm in inner diameter. These mineral membranes can easily support chemical cleaning after each experiment and sudden variations in the pressures and temperatures. The raw red wine (Corbière)

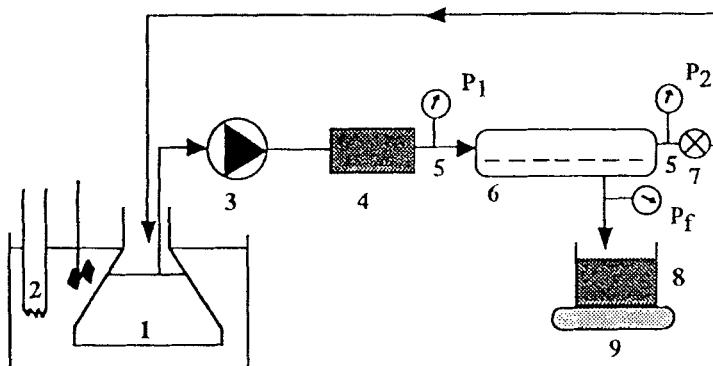


FIG. 1 Laboratory-scale filtration unit. (1) Reservoir, (2) temperature 20°C, (3) feed pump, (4) flowmeter, (5) pressure transducer, (6) filtration unit, (7) control valve, (8) permeate, (9) electronic scale.

was obtained from INRA, France, with initial turbidity > 150 NTU. The different characteristics of this type of wine are given in Jaffrin et al. (7).

Helical shaped baffles (11) made by winding a steel wire of 1 mm diameter on a steel rod of 3 mm diameter and with a different number of turns,  $T$  (2, 4, and 6 turns per 25 mm length of baffle rod) were made and used in the experiments. A rod-shape baffle of 5 mm diameter was also made, which represents an infinite number of turns. These baffles are shown in Figs. 2a and 2b.

The variations with time of filtration of transmembrane pressure (TMP), the head loss ( $P_1 - P_2$ ), and the permeate flux ( $J$ ) were measured through various transducers and were registered on a microcomputer by the help of an interface. The filtrability index ( $V_{max}$ ) was calculated from the equation  $V_{max} = 3/(5/V5 - 2/V2)$ , proposed by Gaillard (13), where  $V5$  represents the filtered volume in 5 minutes and  $V2$  represents the filtered volume after 2 minutes. In these experiments, we used Millipore filters (0.65  $\mu$ m pores) and a pressure of 1 bar. The filtrate was analyzed for its turbidity using a nephelometer (Ratio, Hach) capable of measuring the turbidity between 0.001 and 1999 NTU. It was considered that a filtered wine is not stable if the turbidity value is more than 5 NTU, and is considered

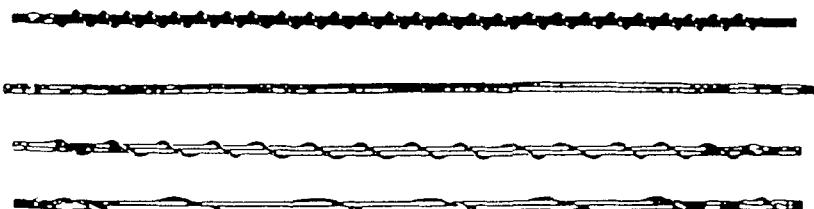


FIG. 2a Helical baffles of different shapes.

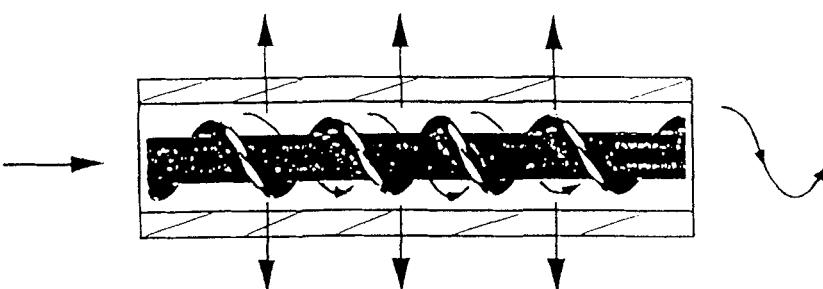


FIG. 2b Flow field in the presence of a helical baffle.

clear or stable if turbidity value is less than 5 NTU; however, it can still contain some colloidal particles. The color intensity was measured by a spectrophotometer at three different wavelengths of 420, 520, and 620 nm. From the spectrophotometer values of filtered wine, we calculated the cloudy nature,  $N = (\text{o.d. at 420})/(\text{o.d. at 520})$ , the color complexion,  $T = (\text{o.d. at 420})/(\text{o.d. at 520})$  and color intensity  $CI = (\text{o.d. at 420} + \text{o.d. at 520})$ , where o.d. represents optical density. A troubled wine is normally considered bad during its taste judgement. It is known that the color intensity of a wine is not the only criteria for its quality, but it may represent the combined effect of the structure, quality, and taste (14).

Microbiological control was made with 100 mL of filtered wine samples collected in a sterilized test tube. This sample was filtered on a Millflex 100 filter from Millipore. This apparatus was equipped with a vacuum pump and a sterile reservoir. On this apparatus and using a membrane of 0.45  $\mu\text{m}$ , we fixed a funnel and filtered the wine sample. The funnel was removed and then fixed on a disk for incubation in a culture medium. The box was kept in the incubator at 26°C for 6 days, and then we counted the differential of the bacteria and the yeast. For each experiment we used a new sample, cultured it in a sterile medium, and counted the number of bacteria.

## RESULTS

### Choice of Membrane Pore Size

Preliminary filtration experiments were made with mineral membranes with pore sizes of 0.14, 0.2, and 0.45  $\mu\text{m}$  at a constant feed flow rate of 1 L/min and at a transmembrane pressure of 1 bar. Figure 3 gives the permeate flux values with filtration time. The comparison of permeate flux after 2 hours of filtration time shows that the flux with 0.45 and 0.2  $\mu\text{m}$  pore sizes was about the same. The flux with 0.14  $\mu\text{m}$  size decreased very fast and became stable at a very small value of about 6  $\text{L}/\text{h}\cdot\text{m}^2$ , whereas for 0.2 and 0.45  $\mu\text{m}$  pore size the stable flux was reached at about 20  $\text{L}/\text{h}\cdot\text{m}^2$ . The filtrate was analyzed for its color, turbidity, and filtration index (Table 1) for 0.2 and 0.45  $\mu\text{m}$  pores. The filtrate quality was about the same for these two sizes; we selected the 0.45  $\mu\text{m}$  membrane for other experiments.

### Choice of the Helical-Shaped Baffle

We inserted a helical-shaped baffle in the tubular membrane (Fig. 2) and performed long time experiments (3 to 4 hours) with the same type of membrane and wine and at the same feed flow rate and transmembrane

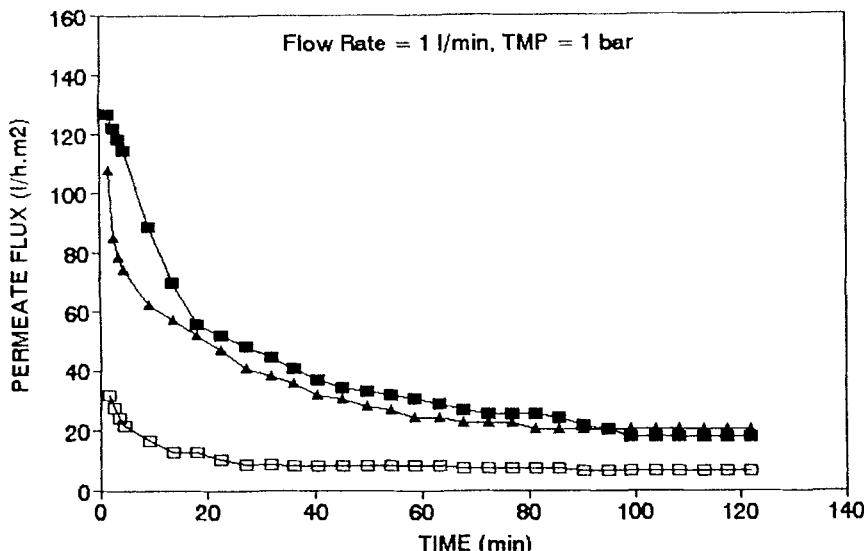


FIG. 3 Variation of permeate flux with time and pore size: (■) 0.2  $\mu\text{m}$ , (▲) 0.45  $\mu\text{m}$ , (□) 0.14  $\mu\text{m}$ .

pressure. The flux increased linearly with the number of helices. The mean filtration values at the end of 3 hours are shown in Fig. 4. The permeate flux increased from about 20  $\text{L}/\text{h}\cdot\text{m}^2$  (without baffle) up to about 50  $\text{L}/\text{h}\cdot\text{m}^2$  with a baffle of 6 turns/25 mm baffle length. It is possible that if we increase number of turns to more than 6 turns/25 mm length, the flux will further increase, but if we go on increasing the number of turns, we will end up with a rod of 5 mm diameter. The flux with such a baffle (rod-type baffle) was found to be lower than that of a 6 turns/25 mm baffle. The detailed explanation of this fact is given in Gupta et al. (11).

TABLE 1  
Quality of Raw and Filtered Wine

Type of wine	o.d. at 520 nm	o.d. at 420 nm	Turbidity (NTU)	$V_{\max}$ (mL)
Raw wine	2.47	2.48	>100	39
Filtered wine:				
0.2 $\mu\text{m}$ pore	2.47	2.48	<1	1500
0.45 $\mu\text{m}$ pore	2.47	2.47	<1	1450

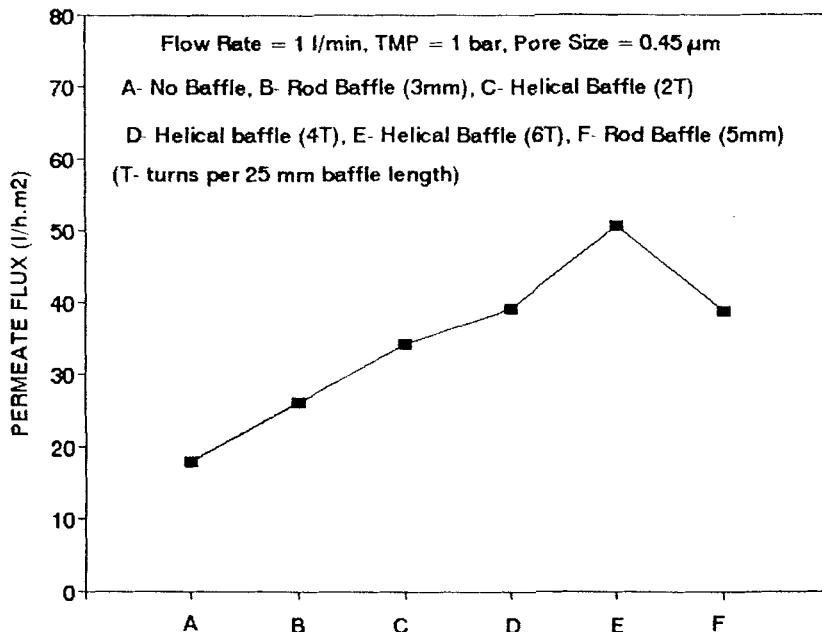


FIG. 4 Mean permeate flux values with membrane configuration.

### Effect of Feed Flow Rates

We conducted some parallel experiments at different feed flow rates with a helical baffle (of 6 turns) and without a baffle using a membrane of  $0.45\text{ }\mu\text{m}$  size. Figure 5a presents the results of permeate flux variation at a  $600\text{ mL/min}$  feed flow and a 1 bar transmembrane pressure. The increase in flux was about 250% (without a baffle the stable flux was  $13\text{ L/h}\cdot\text{m}^2$  and with a baffle it was  $30\text{ L/h}\cdot\text{m}^2$ ). The permeate flux values at different flow velocities are shown in Fig. 5b. It was found that the permeate flux increased with an increase of feed velocity. When a baffle was not used, we found that the limiting flux was obtained even at feed velocities  $<1\text{ m/s}$ . However, other authors have reported that the limiting feed velocity can be of the order of 3 to 4 m/s (5). We think that when an insert is used, it is not necessary to use a high feed velocity (high feed flow rates) because the presence of an insert increases the velocity for the same feed flow rate; for example, in this case the feed velocity increased by about 3 times. With the presence of an insert the pressure drop also increases, so it is advisable to check whether this increase in flux is also



related to the corresponding increase in energy consumption. For example, without a baffle and at a flow velocity of 0.35 m/s and a pressure drop of 0.1 bar, the flux was 12 L/h·m<sup>2</sup> and the hydraulic energy consumed was 0.1 W, while with the insert the flux increased to 30 L/h·m<sup>2</sup> but the hydraulic energy consumption increased to 0.35 W, which is relatively more than the increase in flux. This shows that the use of a turbulence promoter is not economical under such experimental conditions. Hence, in order to check its utility, we performed other experiments at constant hydraulic energy consumption.

#### Permeate Flux at the Same Hydraulic Dissipated Energy Consumption

Hydrodynamic methods are normally used to increase the permeate flux, but it should not be done at the cost of extra hydraulic energy. We performed some experiments at the same hydraulic dissipated energy and compared the increase in permeate flux or the increase in the volume of permeate collected during the same time of filtration. Figure 6 shows the results with a helical baffle, a rod-type baffle, and without any baffle. The hydraulic energy was fixed at 0.1 W by adjusting the inlet flow and pressure drop in each case. These experiments show that with a helical baffle

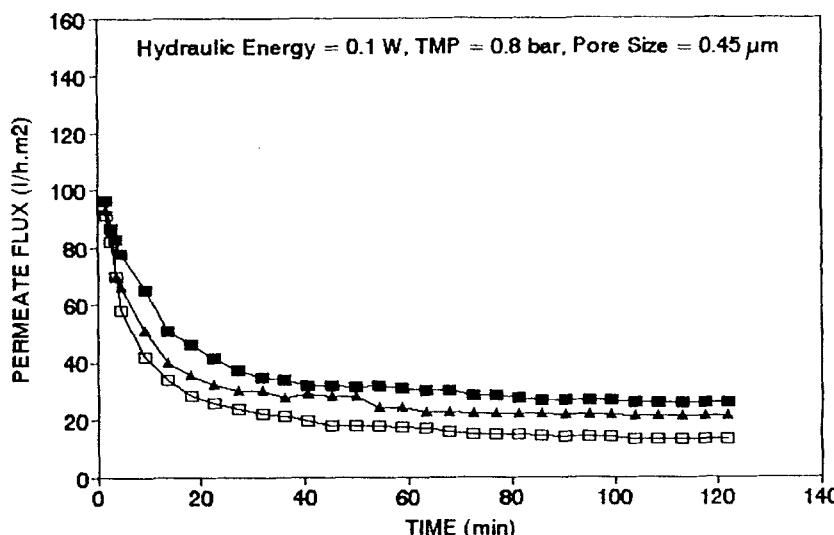


FIG. 6 Comparison of permeate flux variation at same hydraulic energy: (■) helical baffle, (▲) rod baffle, (□) no baffle.

the permeate flux increased by about 2 times compared to those with no baffle. In order to check if this increase was consistent for long durations, we did experiments for 8 hours. The results in Fig. 7 show that even in the presence of an obstacle, particle deposition on the membrane (on the surface or in a pore) is not avoided completely but was less compared to the no-baffle case. After 8 hours of experiment the increase in permeate flux was about 250%. The total permeate volume obtained was about 1.5 L with a baffle compared to 0.6 L without a baffle. The experiments made at other transmembrane pressures also confirmed this increase in flux (Table 2). All these results confirm that at different transmembrane pressures the presence of an obstacle can partially control membrane fouling and that it is possible to increase the permeate flux by a minimum of 150%.

### Calculations of Different Membrane Resistances

The membrane resistance can be calculated by the equation of Darcy (15),  $J = \text{TMP}/\mu R$ , where  $R$  represents the total resistance ( $R = R_m + R_i + R_e$ ),  $R_m$  is the clean membrane resistance,  $R_i$  is the resistance from pore plugging, and  $R_e$  is the resistance on the membrane surface (mainly from concentration polarization).  $R_m$  was initially obtained from experi-

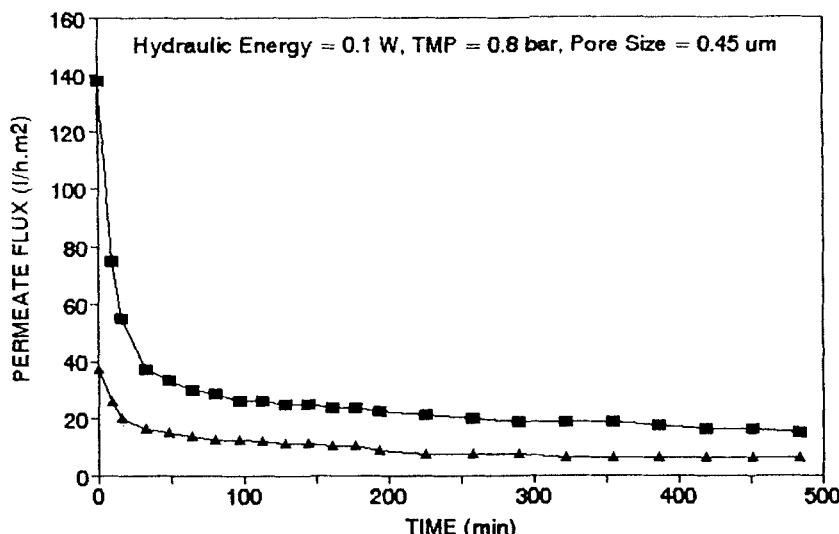


FIG. 7 Comparison of permeate flux variation with helical baffle (■) and without baffle (▲).

TABLE 2  
Total Permeate Volume Collected at Different Transmembrane Pressures

	TMP (bar)			
	0.17	0.5	0.8	1.0
Permeate volume (mL):				
Without baffle	—	350	375	350
With baffle (6 turns/25 mm)	245	445	446	540

ments with distilled water.  $R$  was calculated from Darcy's law by using the stable permeate flux, the transmembrane pressure and the viscosity values ( $\mu = 1.6$  cP). After the experiment the membrane was rinsed with demineralized water. Filtration experiments with demineralized water gave the value of  $R_m + R_i$ , which can be used to calculate the resistance of the deposition of particles on the membrane surface and in the pores. The polarized layer, which is reversible, is supposed to be washed away by rinsing. Finally, from the values of these resistances, it was possible to calculate  $R_e$ . We used the same fluid velocity while rinsing the membrane as was used in the filtration experiment so that the membrane was not overcleaned by rinsing. Table 3 gives the values of different resistances. The calculation of error from different experiments show that the total resistance values can vary up to 15% and the permeate flux values up to 3.5%. We have plotted these resistances for different configurations in Fig. 8. This figure shows that the presence of a baffle decreases both  $R_e$  and  $R_i$ , with the effect more pronounced for  $R_e$ . This rapid decrease in  $R_e$  can be explained by the type of flow field (helicoidal) generated inside the membrane from the presence of a helical baffle against simply tangential flow without a baffle. We also observed that the increase in flow velocity did not improve the decrease in membrane fouling. In order

TABLE 3  
Different Membrane Resistances<sup>a</sup>

	Membrane resistance ( $m^{-1}$ )		
	$R \times 10^{12}$	$R_i \times 10^{12}$	$R_e \times 10^{12}$
Prefiltered wine (WB)	11.0	3.0	7.0
Raw wine (WB)	16.0	3.8	11.0
Raw wine (B)	8.6	4.7	3.0

<sup>a</sup> WB = without baffle; B = baffled.

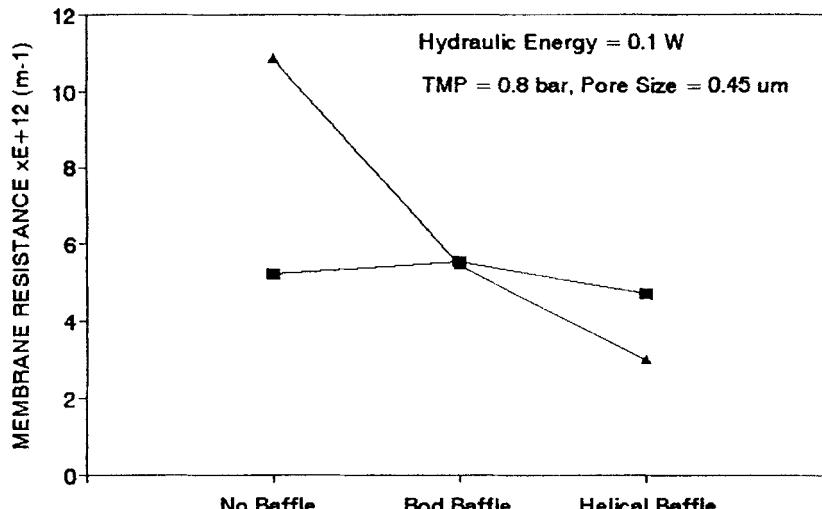


FIG. 8 Variation of membrane resistance with membrane configuration: (■) pore plugging, (▲) membrane surface.

to better understand this, we used a pre-filtered wine sample for filtration and a membrane without a baffle. The permeate flux improved very slightly (from 10 to 15  $\text{L}/\text{h} \cdot \text{m}^2$ ) and the external resistance (due to concentration polarization) was decreased by 50%, whereas there was no effect on the internal resistance. It can be concluded that membrane fouling mainly depends on the quality of wine and its interaction with the membrane (physicochemical interaction), whereas the external fouling can be controlled by the presence of an insert. The helical-shaped baffle controlled fouling on the membrane surface, and so the concentration polarization layer build up was reduced.

### Analysis of the Quality of the Permeate

During all experiments we analyzed the quality of the permeate by measuring the optical densities at 420, 520, and 620 nm with a spectrophotometer. We also controlled the quality by measuring the turbidity values and filtration index of raw and filtered wine. The turbidity of the filtered wine decreased very rapidly in less than 10 minutes to <1 NTU and then it remained constant with filtration time. The turbidity of raw wine was of the order of 100 NTU. The filtration index was found to be more than 1000 mL for the filtered wine against 15 mL for the raw wine. The control

TABLE 4  
Analysis of Raw and Filtered Wine<sup>a</sup>

Results of analysis	RW 1	Conf 1	RW 2	Conf 2	RW 3	Conf 3
Turbidity (NTU)	30	<1	94	<1	97	<1
o.d. at 620 nm	0.97	0.53	1.54	0.88	1.33	0.86
o.d. at 520 nm	2.47	2.47	2.48	2.48	2.49	2.49
o.d. at 420 nm	2.47	1.77	2.48	2.48	2.5	2.5
Color intensity	4.94	4.24	4.96	4.96	4.99	4.99
$V_{max}$	58	2970	58	2900	58	2900
Opacity	0	0.7	0	0	0	0

<sup>a</sup> RW = raw wine, Conf = membrane configuration. 1:  $Q = 600$  mL/min, no baffle. 2:  $Q = 400$  mL/min, rod-type baffle. 3:  $Q = 320$  mL/min, helical baffle of 6 turns/25 mm.

of yeast and number of bacteria show that the filtrate has no yeast after 20 minutes of filtration and the number of bacteria was reduced to about 0 in the filtrate. The value of yeast (cells) in raw wine was about  $10^8$ /mL, and the bacteria count was about  $3 \times 10^8$ /mL.

The optical density values for the different raw and filtered wines, for the different configurations used are given in Table 4. It was not possible

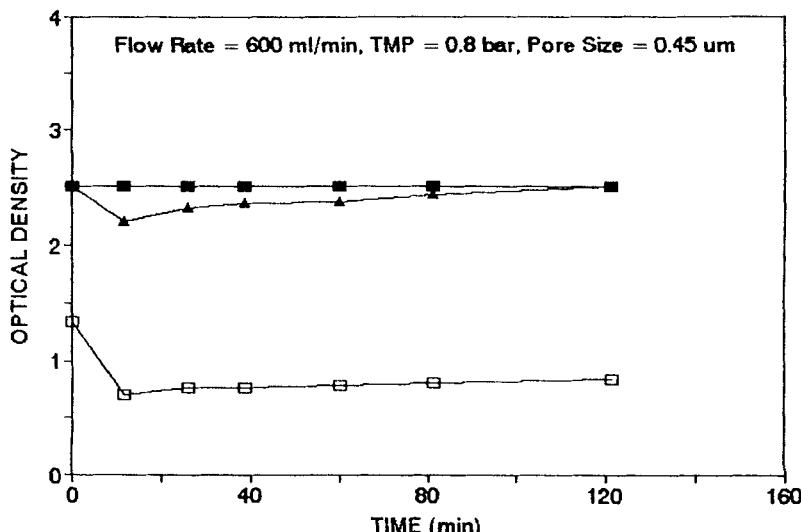


FIG. 9 Comparison of filtered wine at different optical densities: (■) 420 nm, (▲) 520 nm, (□) 620 nm.

to analyze the quality of permeate at each wavelength and for each type of molecule. The uncolored polyphenols were analyzed at 420 nm and the anthrocyanates at 520 and 620 nm. It was noticed that the optical density of the filtrate at 420 nm decreased during the first 20 minutes of filtration and then increased but was inferior to the initial value. This decrease in optical density during the first 20 minutes may be because of the dilution of filtrate with water trapped in the membrane pores while cleaning the membrane, but this decrease is important in showing that the membrane has stopped some types of molecules and then released them, or that certain types of polyphenols changed their physicochemical characteristic because of the higher wall stresses imposed on the membrane surface.

The optical density at 620 nm also decreased for about 20 minutes and then increased to a constant value (Fig. 9). This shows that certain types of molecules are retained by the membrane at the beginning of filtration. The majority of the particles analyzed at 620 nm are those deposited at the membrane surface due to adsorption. In general, we found that the quality of the filtrate was not modified by the presence of the obstacle.

## CONCLUSIONS

It has been found that a mineral membrane of 0.45  $\mu\text{m}$  pore size is suitable for the microfiltration of raw red wine without any pretreatment. A permeate with a very small adsorption of certain molecules was found to be of good quality, and so its treatment with a mineral membrane is acceptable. The filtration experiment performed at a low transmembrane pressure ( $10^5$  Pa) and at a flow velocity of less than 1 m/s gave a low flux of about  $10 \text{ L/h}\cdot\text{m}^2$ , whereas the use of a helical-shaped baffle under the same hydrodynamic conditions increased the permeate flux rate up to  $25 \text{ L/h}\cdot\text{m}^2$ , and even at the same dissipated hydraulic energy an increase of about 200% was possible. It was also found that the presence of an obstacle in the membrane reduced the membrane resistance due to concentration polarization, and has no bad effect on the quality of filtered wine compared to the filtrate obtained without an obstacle.

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