TANGENTIAL MICROFILTRATION OF ORANGE JUICE IN BENCH PILOT[1]

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SUMMARY

The aim of this study was to introduce the tangential microfiltration (TMF) technique on the production of orange juice (TMFJ), and compare it with pasteurised juice (control) as regards chemical composition and sensorial characteristics. We used a TMF pilot equipped with four monotubular ceramic membranes (0.1, 0.2, 0.8 and 1.4µm) arranged in series with a filtering area of 0.005 m^2 each. Commercial flash-pasteurised orange juice was used as the initial product. Experiments were divided into three parts: a) the characterisation of the TMF pilot; b) optimisation of operational conditions; c) production of the TMFJ. In the second part, membrane with 0.8-µm pores presented best flux followed by those with 1.4-, 0.1-, and 0.2-µm pores. However, to guarantee permeate sterility, we chose the membrane with 0.1-µm pores for TMFJ production. Initially, the orange juice was sieved in order to separate part of the pulp, being subsequently submitted to TMF. A mixture of retentate and pulp was made, and was subsequently pasteurised. We obtained the TMFJ by adding the permeate to the mixture. TMFJ presented soluble solids content (°Brix), pulp, pH, and titrable acidity similar to the initial pasteurised juice (control). Nevertheless, 28% of vitamin C was lost during the TMFJ production. According to the juice taster panel, the control juice presented best sensorial characteristics (greater aroma intensity and fruity flavour) when compared with the TMJF.

Keywords: beverage; fruit; sensorial analysis; membrane.

RESUMO

MICROFILTRAÇÃO TANGENCIAL DE SUCO DE LARANJA EM PILOTO DE BANCADA. O objetivo deste trabalho foi introduzir a técnica de microfiltração tangencial (MFT) na produção de suco de laranja. O suco microfiltrado (SMFT) foi comparado química e sensorialmente com um suco pasteurizado (testemunha). Utilizou-se um piloto de MFT munido de quatro membranas (0,1, 0,2, 0,8 e 1,4µm) cerâmicas monotubulares dispostas em série, cada uma delas com superfície de 0,005m^2. Suco de laranja comercial *flash* pasteurizado foi usado como produto inicial. O trabalho experimental foi dividido em três fases: a) caracterização do piloto de MFT; b) otimização das condições operacionais; c) produção do SMFT. Na fase de otimização, a membrana de 0,8µm apresentou os melhores fluxos de permeado, seguidas pelas de 1,4, 0,1 e 0,2µm. Para garantir a esterilidade do permeado, a membrana de 0,1µm foi escolhida para a terceira fase do trabalho. Na produção do SMFT, o suco de laranja foi peneirado para separar uma parte de sua polpa, sendo em seguida microfiltrado. Depois, a polpa foi misturada ao retentato e a mistura pasteurizada. O SMFT foi obtido adicionando a mistura pasteurizada ao permeado. O SMFT apresentou teor de sólidos solúveis (°Brix), polpa, pH e acidez titulável semelhante ao suco inicial pasteurizado (testemunha); embora, tenha perdido maior quantidade (28%) de vitamina C. De acordo com os provadores do painel, o suco testemunha apresentou melhores características sensoriais em relação ao SMFT, por apresentar maior intensidade de odor e sabor frutoso.

Palavras-chave: bebida; fruta; análise sensorial; membrana.

1 – INTRODUCTION

The 70's and 80's brought much attention to health-oriented foods and natural products, increasing the consumer demand for less-processed products. Concomitantly, producers began the commercialisation of non-concentrated, ready-to-drink orange juice. These products, obtained directly from the fresh orange juice, have presented superior taste and aroma in comparison with those from concentrated juice. Moreover, they possess a 'marketing appeal' since no water is removed or added in its processing, apart from its higher market price. With the increasing popularity of this kind of product, the concept of evaporation or thermal concentration became a synonymous of inferior quality. Thus, consumers continued to demand higher-quality juices with more flavour, aroma, greater natural vitamin, and pulp retention [8, 9].

The application of tangential microfiltration (TMF) with mineral membranes on the process of orange transformation allows the recovery of two new products: a pulpy concentrate and a clarified juice [4]. These two products also possess distinct aroma, where the pulpy concentrate is similar to an orange juice full of pulp. As regards aroma, this product is poor in oxigenated compounds (aldehydes, esters, and alcohols) and rich in hydrocarbons (terpenes), when compared with the juice before microfiltration. The permeate is a clear juice that lacks a turbid appearance. The permeate and the original orange juice have an identical biochemical composition. Furthermore, the permeate is microbiologically stable, and does not present any enzymatic activity when the membrane pore diameter used is equal or inferior to 0.2µm. This clarified juice presents typical aroma of original orange juice, but differs by not presenting body, because of the absence of pulp. The analysis of aroma components reveals a depletion of hydrocarbons and enrichment of oxigenated compounds, in comparison with the original juice.

KÖSEOGLU *et al.* [13] worked with ultrafiltration (UF) followed by reverse osmosis (RO) of orange and grapefruit juice, while KÖSEOGLU *et al.* [14] conducted a similar study with celery, carrot, cucumber, and tomato juices. The authors cold-concentrated the UF permeate through the RO process. Afterwards, they sterilised (HTST) the mixture (pulp and UF-concentrated retentate), and

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recombined the permeate with the pulp-enriched retentate, yielding a concentrated juice with sound sensorial qualities. The authors reported no loss of aroma compounds during processing.

HERNANDEZ *et al.* [10] studied the effect of UF with polisulfone membranes on the aroma components and suspended solids contents of orange juices. The authors reported that suspended solids were completely removed by the UF membrane. The great part of pectin and pectinmethylesterase was removed, although they possessed a lower molecular weight than the membrane's cut-off. This fact was explained by the formation of a gel layer over the surface of the membrane, which helped the filtration by reducing the effective pore size of the membrane. The permeate presented lower viscosity in comparison with the concentrated juice mostly due to the removal of the suspended solids. The authors observed that the majority of aroma hydrosoluble components (esters, aldehydes, and alcohols) passed freely through the membrane, whist more apolar compounds (limonene and valencene) could not pass through the membrane. The oxigenated components of the fruity aroma were associated with serum, while the hydrocarbons were associated with the pulp of the orange juice.

JOHNSON *et al.* [12], working with orange juice UF at polysulfone membranes, confirmed the results of BALI, LOZANO [4] and HERNANDEZ *et al.* [10]. Those authors observed that the membrane itself affected the distribution of aroma compounds both in the permeate and retentate. Alcohols were found in the permeates while apolar compounds (terpenes and apolar aldehydes) remained in the retentate. The permeation of intermediate polarity compounds (carbonyls) was between that of alcohols and hydrocarbons. They also mentioned a loss of aroma compounds during processing. On average, 15,6% of the incoming amount of a given alcohol was lost. By contrast, 44,4% of the incoming amount of nonalcohol compounds was lost. Most likely the loss was due adsortion by pulp and/or due to entrapment in the fouling layer.

OLLE *et al.* [15] studied flavour compounds of mango puree submitted to TMF and RO. They conducted a enzymatic treatment (cellulase and pectinase at 50°C/ 2h) and microfiltrated the mango puree in alumina membranes with 0.2-µm pores and subsequently concentrated the permeate by RO. The volumetric reduction ratio (VRR) for TMF and RO was 3.5 and 2, respectively. Gas chromatography revealed that the apolar components (terpenes) present in the original puree did not surpass the TMF membrane and were remained in the retentate. The terpenes, responsible for the flavour and aroma of the fresh mango, represented 98% of the aroma compounds of the mango puree. The authors affirmed that the retention of these hydrocarbons was associated with the insoluble solids in the puree, such as cellular wall remnants. On the other hand, the polar compounds (2%) of the original puree presented variable behaviour under TMF and RO. This compounds were extracted from the start puree, permeate and retentate of TMF and RO. The authors concluded that TMF could be a low-cost option for the concentration of mango puree.

VAILLANT *et al.* [17] studied passion fruit juice TMF. They used ceramic membranes and treated the juice with pectolytic enzymes for the degradation of suspended solids. The permeate flux increased in function of the tangential velocity and of the enzymatic concentration. Although they used enzymes, the authors observed a slight decrease in aroma compounds in the permeate compared with the feeding juice, because few volatil compounds were probably retained by the membrane.

Therefore, we sought to introduce the TMF technique on the production of orange juice using a bench-pilot with four membranes arranged in series, and compare the reconstituted microfiltered orange juice (TMFJ) with the pasteurised juice (referent), by chemical and sensorial analyses.

2 – MATERIAL AND METHODS

2.1 – Material

2.1.1 – Microfiltration pilot

The TMF tests were conducted in a bench pilot with a total volume of 4,4L, and a feeding tank of 3,0 L. The pilot had four tangential filtration modules arranged in series, as depicted in *Figure 1*. The alumina monotubular membranes, were organised in the following sequence: 0.1, 0.2, 0.8, and 1.4µm in relation to the feeding pump. Membranes were 0.2m long, had a diameter of 0.008m, and area of 0.005m^2.

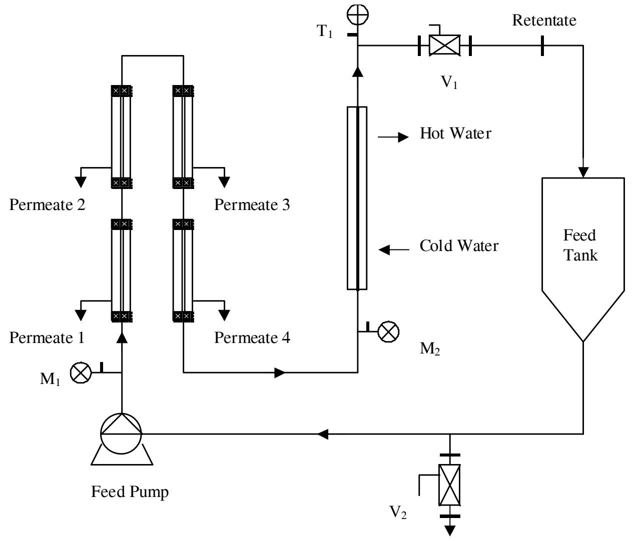

FIGURE 1. Scheme of the TMF pilot.

The simultaneous use of four different membranes in series allows us to increase experimental work productivity, since each one of them could be characterised by their transmembrane pressure (TMP). During the experiments (VRR = 1), we used all of the four membranes assembled in series, except when the VRR > 1, when we operated it with four 0.1-µm membranes.

In the TMF tests, the orange juice that was present in the feeding tank, circulated in the interior of the pilot when the feeding pump was turned on. The pressure of the orange juice at the entrance of the first module and at the end of the last were controlled by the valve V_1, while its temperature was read by the thermometer T_1, and was controlled by the heat changer. The permeates of membranes with pores of 0.1, 0.2, 0.8, and 1.4µm were collected at positions 1, 2, 3, and 4, respectively. In most tests (VRR = 1), the permeate returned to the feeding tank, and the pilot was operated in a closed circuit, in batches. In some tests (VRR > 1), however, the permeate was removed from the pilot at the same time that it was fed an equal volume of orange juice. In this case, the TMF was similar to the continuos process.

Before the TMF experiments, the mean permeability of the membranes with pores of 0.1, 0.2, 0.8, and 1.4µm was 274, 321, 1102, and 1838L/h.m^2.bar, respectively. The average permeability of each membrane was determined in standard conditions of TMP (1.0 bar) and temperature (20°C). The tests were repeated four times. Membranes were considered clean and ready to use when their permeability, measured immediately before the TMF tests, remained within the following interval:

$$\overline{B} - 2 * (\sigma - 1) < B < \overline{B} + 2 * (\sigma - 1)$$

$B \rightarrow$ permeability of the membrane under standard conditions before each orange juice TMF assay, L/h.m^2.bar

$\overline{B} \rightarrow$ average permeability of the membrane, L/h.m^2.bar

$\sigma - 1 \rightarrow$ standard deviation

2.1.2 – Raw material

We acquired a commercial flash-pasteurised orange juice (non-concentrated) at a local market (Montpellier, France). The juice was tetra-packed and fridge-stored. In the laboratory, the juice was stored under -20°C in cold chambers, and defrosted as required.

2.2 – Methods

The orange juice was partially pulped with the aid of a 0.2-mm sieve before being submitted to TMF.

2.2.1 – Repeatability test

Permeate flux values of orange juice TMF were evaluated for repeatability. Four repeats were conducted with fixed conditions: entrance pressure = 1.8 bar, exit pressure = 0 (zero) bar, temperature = 25°C, and VRR = 1. For each test, the measured flux was the average of the values taken at 100, 130, and 160 minutes of filtration. In these time-points, the flux curve presents a slight declination, typical of the orange juice TMF.

2.2.2 – Optimisation of operational conditions

To estimate optimal TMF conditions, two parameters were used: a) permeate flux, and, b) permeate quality. In the first case, maximum flux was expected, whereas guarantee of sterility and the maintenance of original juice chemical characteristics (mainly vitamin C) were expected in the second case. Using these criteria, we chose the adequate membrane pore size, TMP, temperature, and VRR for orange juice MFT.

The following expression defined VRR, which is the relation between the total volume of feed juice and the volume of the retentate at the end of the whole process:

$$VRR = \frac{Vtotal}{Vretentate} = \frac{Vretentate + Vpermeate}{Vretentate} \qquad (1)$$

2.2.3 – Production of reconstituted microfiltered orange juice (TMFJ)

Once the TMF parameters were defined, the TMFJ was produced as shown in the following flux diagram (*Figure 2*).

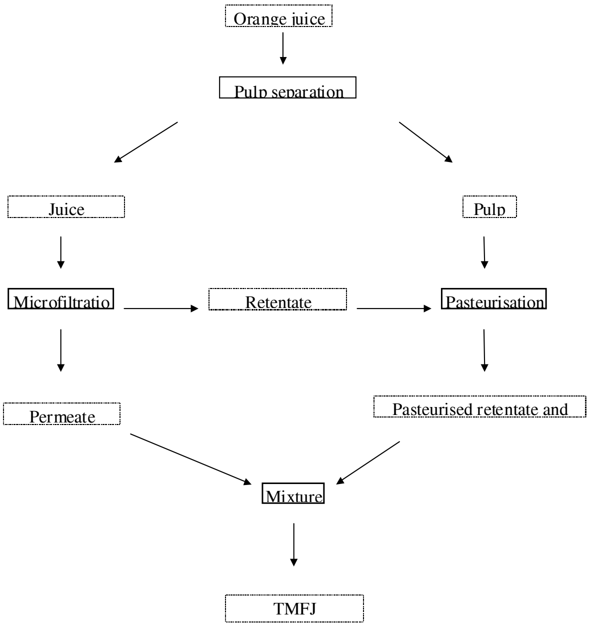

FIGURE 2. Processing of TMFJ.

The pulp separated by the 0.2-mm sieve was mixed with the TMF retentate. Afterwards, this mixture was pasteurised at 83°C/2 min. The TMF permeate was mixed with the pulp-retentate mixture for the production of the TMFJ. This juice was compared by chemical and sensorial analyses with the commercial one (control), which had been submitted to lab pasteurisation (83°C/2min).

2.2.4 – Chemical and sensorial analyses

The original commercial juice, the control juice, the partially pulped feed juice, the permeate, the retentate, and the TMFJ were all submitted to chemical analysis in duplicate. The following analysis were made: pH, titrable acidity (citric acid g/100mL), soluble solids content (°Brix), pulp (insoluble dry residue g/100g), and vitamin C (ppm). Vitamin C was analysed according to the ISIM [11], and the other characteristics according to the AFNOR [2,3].

TMFJ was compared with the control juice for sensorial characteristics using the triangular test and hedonic scale test (structured) in order to evaluate differences between the juices, intensity of sensorial attributes, and preferences of juice tasters for each juice, according to the AFNOR [1]. The juice taster panel was composed of 15 tasters trained in beverage tasting.

3 – RESULTS AND DISCUSSION

3.1 – Repeatability test

Table 1 shows results of the repeatability test for the TMF of orange juice. We observed an inverse relation between the permeate flux and coefficient of variance.

TABLE 1. Variability of permeate flux values in membranes with different pore sizes.

Membrane pore size (μm)	0.1	0.2	0.8	1.4
Mean transmembrane pressure (bar)	1.6	1.2	0.7	0.3
Mean permeate flux (L/h.m^2)	20	5	37	26
Flux standard deviation (L/h.m^2)	3	2	3	6
Flux variation coefficient (%)	15	40	8	23

Temperature = 25°C

The widest dispersion of results was found in the 0.2-μm membrane due to insignificant flux values (5L/h.m^2 mean rate), whereas the lowest variation was observed in the 0.8-μm membrane, which presented the highest flux values (37L/h.m^2 mean rate). As standard deviations for the 0.1- and 0.8-μm membranes were considered acceptable, the subsequent tests were conducted with no repetitions.

Generally, the permeate flux vs. time-curves presented the typical form for the TMF of orange juice. Initially, we observed an accentuated drop followed by a slow attenuation or stabilisation, which continued to the end of the filtration process. This type of curve may be observed in *Figures 4* and *5*, always occurring for the TMF with VRR = 1. As, under these conditions, the permeate returns to the feeding tank, we considered that the retentate presented constant physico-chemical composition, and that the process was conducted in the stationary stage.

The membranes with smaller pore sizes may produce permeate flux values superior to those of bigger pore sizes (*Table 1*). This was verified between the 0.1- and 0.2-μm membranes and between the 0.8- and 1.4-μm membranes for every orange juice test conducted herein. The same characteristic was observed by CHAMCHONG, NOOMHORN [6] who clarified tangerine juice through UF and TMF. These authors noted that the flux through a 0.1-μm membrane (69L/h.m^2) was superior to that of a 0.2-μm membrane (41L/h.m^2). They stated that particles of 0.1 and 0.2μm found in tangerine juice might have caused fouling in the 0.2-μm membrane pores.

3.2 – Membrane pore size and pressure

Four membranes were tested (0.1; 0.2; 0.8; 1.4μm) with six different TMP (2.8; 2.4; 2.0; 1.5; 0.9; 0.3 bar), as showed in *Figure 3*. With the exception of the 0.2-μm membrane, all of the others presented an increasing permeate flux as a function of TMP, up to a limit value. This limit value varied according to the membrane: 1.5, 2.0, and 2.4 bar for the 0.8-, 1.4-, and 0.1-μm, respectively. This observation agrees with those of CHERYAN [7] and TODISCO *et al.* [16] who pointed out that permeate flux increases with TMP. However, after a TMP critical point, flux becomes independent of pressure due to polarisation of macro-molecules and the concentration of suspended material in the membrane surface. TODISCO *et al.* [16] informed that the mechanism of membrane pore blockage is due to the penetration of suspended particles and macromolecules in its interior, and that this is as important as the polarisation layer for the reduction of permeate flux. These mechanisms, responsible for the reduction in flux during TMF, are known as *fouling*. In the same way, CAPANNELLI *et al.* [5] working with orange and lemon juice UF and TMF, pointed out that the permeate flux became independent when TMPs were greater than 2.0 bar.

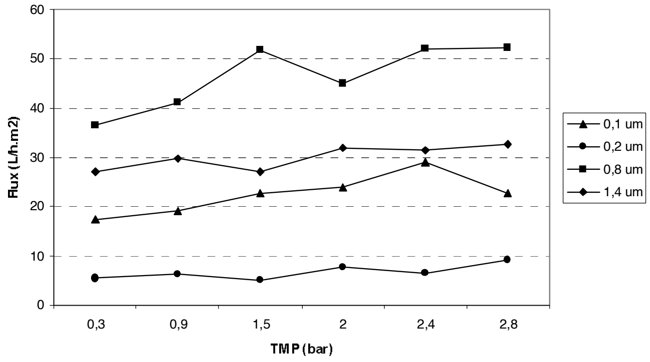

FIGURE 3. Permeate flux of partially pulped 0.2-mm sieved orange juice, filtered through four different membranes at different TMP, 25°C and 6.7m/s.

Figure 4 shows the variation in flux for the four membranes at 2.4 bar. Flux decreased abruptly for every membrane in the first 40 minutes, after what flux remained relatively stable. This is a typical model of membrane fouling.

The 0.8-μm membrane presented the highest flux under all tested pressures, while the opposite was found for the 0.2-μm membrane, and the 1.4- and 0.1-μm membranes presented intermediate values. Only the

0.8-µm membrane reached a flux > 50L/h.m^2 – the minimum reference value for commercial TMF membranes.

Nevertheless, because of the purpose of this study, we chose the 0.1-µm membrane to guarantee permeate sterility together with the 2.4 bar pressure where it yielded the best microfiltration flux.

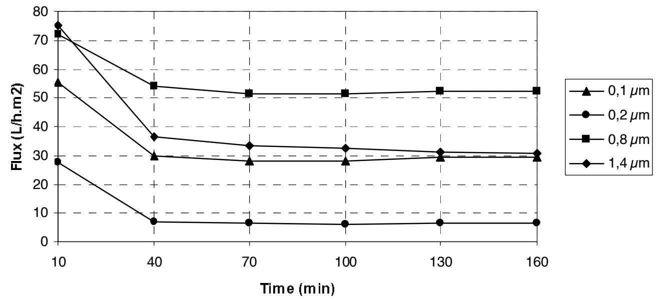

FIGURE 4. Permeate flux of partially pulped 0.2-mm sieved orange juice, filtered through four different membranes at 2.4 bar, 25°C, and 6.7m/s.

3.3 – Working temperature

Only the 0.1-µm membrane was used for testing the best working temperatures (20, 25, 30, and 35°C). The permeate flux increased from 25 to 40L/h.m^2 when the temperature was raised from 20 to 35°C (*Figure 5*). This behaviour was expected once the viscosity of the feed juice, the permeate, and the retentate was inversely related to temperature, resulting in higher flux values, as anticipated by Darcy's law [6].

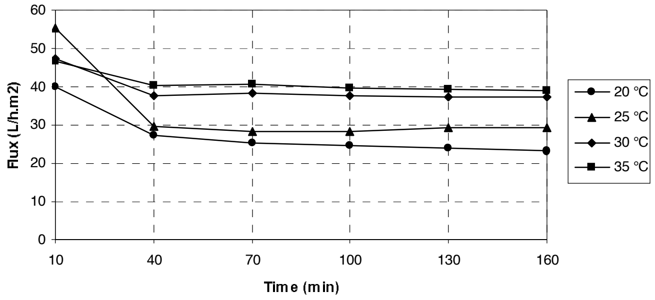

FIGURE 5. Permeate flux of partially pulped 0.2-mm sieved orange juice, microfiltered with a 0.1-µm membrane, at four different temperatures, 2.5 bar, and 6.7m/s.

Feed juice, permeate, and retentate were chemically analysed (*Table 2*). Except for vitamin C, we may affirm that temperature did not change chemical characteristics such as pH, titrable acidity, °Brix, or pulp content in the retentate or the permeate. Vitamin C content dropped due to an increase in TMF temperature and the permeate showed the lowest content. In the retentate, vitamin C loss ranged from 6 to 18% when temperature changed from 20 to 35°C, respectively, whereas in permeate this loss was between 10 and 18.5%. We adopted 30°C to continue the process, as higher temperature lead to oxidation of ascorbic acid during orange juice TMF. The 14% vitamin C loss in the retentate was considered acceptable. At 30°C, the 0.1-µm membrane permeate flux was 37.5L/h.m^2, practically the same as 39.3L/h.m^2 at 35°C.

3.4 – Determination of the volumetric reduction ratio

For the determination of VRR, the 0.2-, 0.8-, and 1.4-µm membranes were substituted by three 0.1-µm membranes. Due to the superior permeability of these new membranes, a higher flux was observed in the beginning of VRR testing (above 50L/h.m^2) compared with the 37.5L/h.m^2 flux with the original membrane setting (*Figure 5*).

TABLE 2. Temperature effect on the chemical characteristics of permeate and retentate partially pulped 0.2-mm sieved orange juice, microfiltered in 0.1µm membrane, at 2.5 bar, and 6.7m/s.

	Temperature = 20°C		
Analysis	Feed Juice	Retentate	Permeate
Vitamin C (ppm)	358	336	324
Soluble solids (°Brix)	10.6	11.9	11.4
Pulp (%m/m)	0.25	0.27	0
pH	3.61	3.61	3.60
Titrable Acidity (% m/v)	0.70	0.79	0.77
	Temperature = 25°C		
Analysis	Feed Juice	Retentate	Permeate
Vitamin C (ppm)	435	396	366
Soluble solids (°Brix)	11.5	11.5	11.2
Pulp (%m/m)	0.28	0.27	0
pH	3.52	3.51	3.49
Titrable Acidity (% m/v)	0.83	0.83	0.80
	Temperature = 30°C		
Analysis	Feed Juice	Retentate	Permeate
Vitamin C (ppm)	403	347	337
Soluble solids (°Brix)	11.6	11.8	11.5
Pulp (%m/m)	0.27	0.22	0
pH	3.58	3.57	3.56
Titrable Acidity (% m/v)	0.78	0.79	0.78
	Temperature = 35°C		
Analysis	Feed Juice	Retentate	Permeate
Vitamin C (ppm)	395	322	322
Soluble solids (°Brix)	11.6	11.4	11.8
Pulp (%m/m)	0.27	0.23	0
pH	3.58	3.59	3.59
Titrable Acidity (% m/v)	0.79	0.79	0.79

When VRR reached 5, the permeate flux was reduced from 50 to 20L/h.m^2. This drop in flux was already expected because of membrane fouling, which increases resistance to TMF and viscosity in retentate once suspended particles that cause viscosity do not pass through membrane. According to Darcy's law, permeate flux is inversely proportional to membrane resistance and viscosity of the product to be filtered [6].

The experimental results showed that permeate flux is related to VRR by a second-degree polynomial equation, as shown in *Figure 6*. A greater dispersion of the results was observed during the first two hours of testing (up to VRR $\leq$ 2).

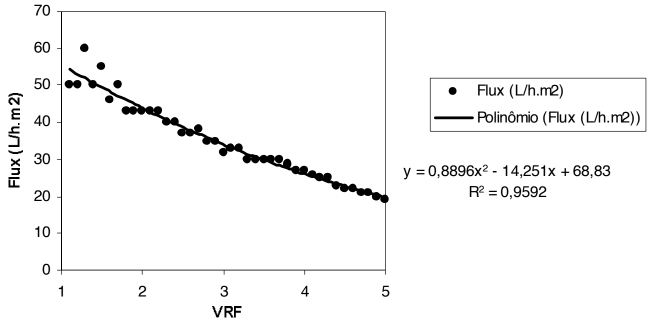

FIGURE 6. Permeate flux of partially pulped orange juice as function of VRR, in 0.1-µm membrane, at 2.4 bar, 6.7m/s, and 30°C.

Figure 7 shows the evolution of VRR and permeate flux values as functions of time. Note that, as in the previous case (*Figure 6*), permeate flux declined as VRR increased. Permeate flux remained practically constant at 30L/h.m^2 by fixing VRR at 3.5 through the recycling of filtrate.

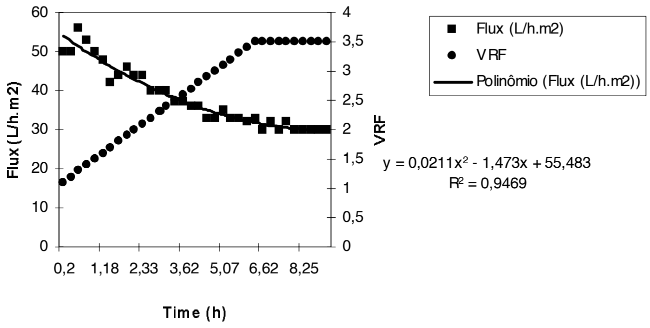

FIGURE 7. Relation between VRR and permeate flux of partially pulped orange juice, microfiltered in a 0.1-µm membrane, at 2.4 bar, 6.7 m/s, and 30°C.

Soon after fixing the VRR at 3.5, the layer of particles, which had deposited on the membrane surface, remained in dynamic equilibrium, and thereafter the pores had no additional obstruction. However, the quantification technique used here in did not allow analysis of flux changes on filtration. We cannot predict for how long permeate flux will remain oscillating around 30L/h.m^2. We know however that flux should decline slowly over filtration time. Probably, the concept of steady state is only valid when time intervals are short.

Also, we noted an elevated dispersion of the measurements in the first two hours of TMF (up to VRR=2), and the best-fitted curve was a second-degree polynomial equation.

3.5 – Production of reconstituted microfiltered orange juice (TMFJ)

The TMFJ was obtained from the 3.5 VRR test (*Figure 7*) according to manufacturing protocols shown in *Figure 2*, whereas the control juice was obtained by the pasteurisation of the original juice. *Table 3* shows that the chemical composition of both the control and MFTJ were not altered by the treatment. Differences in the vitamin C content of MFTJ and control juice were readily apparent in this study. It is probable that the aeration observed in the feed tank, because of the return of retentate, is responsible for most of vitamin C loss during the TMF of orange juice. Recycling for more than 9 hours in the TMF pilot caused the orange juice to lose 28% vitamin C, whereas only 5% were lost from the control juice in comparison with the original juice.

TABLE 3. Chemical analyses of original, control, and TMFJ.

	Original juice	Control juice	MFTJ
Vitamin C (ppm)	398	380	286
Extract (°Brix)	11.6	12.5	12.0
Pulp (% m/m)	0.54	0.54	0.47
Acidity (% m/v)	0.88	0.88	0.83
pH	3.68	3.50	3.49

3.5.1 – Sensorial analysis

In the triangular test, every taster was successful in differentiating the samples, as the TMFJ held a greater quantity of thick pulp on its surface. Despite the apparently richer pulp, the TMFJ had notwithstanding less pulp than the control juice, as revealed by chemical analysis (*Table 3*). The tasters also indicated that the control juice contained greater odour intensity and fruity taste than the TMFJ (*Table 4*). Maybe this was the reason the majority opted for the control juice.

TABLE 4. Sensorial comparison between pasteurised and reconstituted microfiltered juices, through structured scale test for attributed intensity and preference for the tested products.

	Pasteurised juice		Microfiltered juice		Statistical results	
	Average	Deviation	Average	Deviation	T Student	Est. sig.
1. Odour	3.42	0.79	2.33	1.07	2.81	P < 0.01
2. Colour	3.33	0.78	3.00	0.60	1.17	ns[1]
3. Sweetness	2.66	0.89	2.50	1.00	0.43	ns
4. Acidity	2.83	1.11	2.00	1.54	1.52	ns
5. Bitterness	1.33	0.65	1.67	0.98	0.98	ns
6. Astringent C	1.25	0.75	1.50	1.17	0.62	ns
7. Sourness	1.08	1.31	1.42	1.38	0.61	ns
8. Spiciness	0.83	0.94	0.92	0.90	0.22	ns
9. Fruitiness	3.08	1.08	2.00	1.13	2.40	P < 0.05
10. Preference	7.58	1.88	4.75	1.14	4.46	P < 0.001

[1] ns: no statistic significance.

It is likely that part of the aroma compounds were lost during the TMF of orange juice because of aeration inside the feeding tank, in a similar fashion to what happened with vitamin C. Likewise, JOHNSON *et al.* [12] and VAILLANT *et al.* [17], observed aroma losses during tangential filtration processes, for orange and passion fruit juice, respectively. Alternatively, one may avoid juice aeration by changing the pilot design. Namely, we propose that the retentate should not reach the feed tank by its superior part, but should return to the process immediately before the feed pump (*Figure 1*).

On the other hand, several authors do not mention the problem of losing aroma compounds [4, 10, 13, 14, 15]. Different processing (pilot, membrane, raw material, and experimental conditions) could account for the discrepancies observed between our results and those of others.

4 – CONCLUSIONS

Within this study's scope and for the conditions tested herein, we may conclude the following:

- During the TMF of orange juice, the 0.1- and 0.8-µm membranes present greater permeate fluxes in comparison with the 0.2- and 1.4-µm membranes, respectively, in all of the tested TMPs;
- The permeate flux is proportional to TMP for the 0.1-, 0.8-, and 1.4-µm membranes, but only up to a critical value (2.4, 1.5, and 2.0 bar, respectively), after what the flux becomes independent of the pressure;
- As temperature rises (from 20 to 35°C) during the TMF of orange juice, vitamin C content decreases proportionally both in the permeate and retentate;
- The TMF of orange juice does not alter the chemical composition of the permeate and retentate, with the exception of the insoluble compound content, which reached the null in the permeate, due to the retention of those particles in the membrane;
- The 0.1-µm membrane was chosen for TMFJ production because it guaranteed permeate sterility and presented an acceptable flux (37,5L/h.m^2);
- Permeate flux falls in function of increases in VRR, albeit when the latter is fixed at 3.5, the permeate flux practically stabilises at 30L/h.m^2;
- The TMFJ presents a lower concentration of vitamin C and aroma compounds, when compared with the control juice, probably because of the aeration observed in the feed tank during TMF of orange juice;
- The control orange juice is preferable to the TMFJ because of its greater odour intensity and fruity flavour.

5 – REFERENCES

[1] AFNOR – Association Française de Normalisation. Contrôle de la Qualité des Produits Alimentaires: Analyse Sensorielle. 5 ed. Paris: AFNOR, 400p.,1995.

[2] AFNOR – Association Française de Normalisation. Produits derives des fruits et legumes – Determination de l'acidité titrable. Paris: AFNOR, NF V 05-101, p. 1-4, 1974.

[3] AFNOR – Association Française de Normalisation. Produits derives des fruits et legumes – Détermination du résidu sec insoluble dans l'eau. Paris: AFNOR, NF V 05-102, p. 1-3, 1974.

[4] BALI, R.; LOZANO, Y.F. Comparaison de la composition aromatique des produits nouveaux issus du jus d'orange traité par microfiltration en flux tangentiel sur des membranes minérales de developpement récent. **Fruits** v. 47, p. 273-274, 1992.

[5] CAPANNELLI, G.; BOTTINO, A.; MUNARI, S.; LISTER, D.G.; MASCHIO, G.; BECCHI, I. The use of membrane processes in the clarification of orange and lemon juices. **Journal of Food Engineering** v. 21, p. 473-483, 1994.

[6] CHAMCHONG, M.; NOOMHORM, A. Effect of pH and enzymatic treatment on microfiltration and ultrafiltration of tangerine juice. **Journal of Food Process and Engineering** v. 14, p. 21-34, 1991.

[7] CHERYAN, M. Ultrafiltration Handbook. Lancaster: Technomic Publishing Co., 375p., 1986.

[8] DECIO, P.; GHERARDI, S. Freshly Squeezed Orange Juice. **Confructa – Studien**: v. 36, n. 5/6, p. 162-167, 1992.

[9] FOX, K. New technology in citrus processing. **Confructa Studien** v. 35, n. 5, p. 124-135, 1991.

[10] HERNANDEZ, E.; CHEN, C.S.; SHAW, P.E.; CARTER, R.D.; BARROS, S. Ultrafiltration of orange juice: effect on soluble solids, suspended solids, and aroma. **J. Agric. Food Chem.** v. 40, p. 986-988, 1992.

[11] ISIM – Institut des Sciences de l'Ingenieur de Montpellier. Analyse Alimentaire: travaux pratiques. Montpellier: ISIM, p. 9-10. (Practical class brochure), 1998.

[12] JOHNSON, J.R.; BRADDOCK, R.J.; CHEN, C.S. Flavor losses in orange juice during ultrafiltration and subsequent evaporation. **Journal of Food Science** v. 61, n. 3, p. 540-543, 1996.

[13] KÖSEOGLU, S.S.; LAWHON, J.T.; LUSAS, E.W. Use of membranes in citrus juice processing. **Food Technology** v. 44, n. 12, p. 90-97, 1990.

[14] KÖSEOGLU, S.S.; LAWHON, J.T.; LUSAS, E.W. Vegetable juices produced with membrane technology. **Food Technology** v. 45, n. 1, p. 125-130, 1991.

[15] OLLE, D.; BARON, A.; LOZANO, Y.F.; SZNAPER, C.; BAUMES, R.; BAYONOVE, C.; BRILLOUET, J.M. Microfiltration and reverse osmosis affect recovery of mango puree flavor compounds. **Journal of Food Science** v. 62, n. 6, p. 1116-1119, 1997.

[16] TODISCO, S.; PENA, L.; DRIOLI, E.; TALLARICO, P. Analysis of the fouling mechanism in microfiltration of orange juice. **Journal of Food Processing and Preservation** v. 20, n. 6, p. 453-466, 1996.

[17] VAILLANT, F.; MILLAN, P.; O'BRIEN, G.; DORNIER, M.; DECLOUX, M., REYNES, M. Crossflow microfiltration of passion fruit after partial enzymatic liquefation. **Journal of Food Engineering** v. 42, p. 215-224, 1999.

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