Ultrafiltration Performance of Heat-Treated Shamouti Orange [*Citrus sinensis* (L.) Osbeck] Juice

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During ultrafiltration of orange juice with inorganic membranes, heating of the juice prior to the filtration experiment resulted in a significant increase of the fouling indices. The effect of the irreversible fouling (Rif) was always high, whereas the reversible fouling (Rrf) depended on the treatment. It was clearly seen that fouling was reduced after pectin degradation, but the heat treatment applied to the juice before filtration still resulted in reduced fluxes. It is suggested that pectins and proteins that undergo flocculation/coagulation during the heat treatment tend to interact with the membrane-filtering layer and to cause reduction of permeation flux. To clean the membrane to restore its pure water flux, close to the initial one, a proteolitic enzyme detergent wash was needed.

Keywords: *Orange juice; ultrafiltration; proteins; membrane fouling*

INTRODUCTION

Ultrafiltration (UF) for the production of concentrate or clear juice is a technology that is becoming more and more widely used in the juice industry. In general, after passing through an aroma recovery facility at 50 °C, the juice is treated with enzymes to degrade pectin and starch completely, to facilitate economical UF (Stutz, 1993). The potential advantages of UF of orange juice depend on the final product to be produced. These advantages include the following: avoidance of elevated temperatures in the production of clear serum, thus minimizing the likelihood of heat-induced flavor changes (Hernandez et al., 1992); the removal of most of the proteins, thereby rendering unlikely any subsequent enzymatic activity; and, according to recent findings, prevention of any possible interactions of proteins and cloud components that lead to clarification (Shomer et al., 1999a,b).

The major problem associated with UF of most biological fluids is flux reduction because of concentration polarization and membrane fouling. Concentration polarization effects could be reversible as long as changing the operating conditions such as pressure, temperature, and flow velocity can reduce it. Moreover, at low feed concentrations it could be overcome by the self-cleaning action of the cross-flow feed stream. Fouling is also related to flux decline, but it is irreversible. Cross-flow velocity, pressure variations or other operational means cannot remove the gel layer formed on the membrane, comprising mainly proteins, lipids, and other suspended solids. To remove it, the system must undergo physical cleaning (Cheryan, 1986; Dzeizak, 1990; Gésan et al., 1995, 1996). This phenomenon has been thoroughly investigated in the dairy industry (Daufin and Merin, 1995; Marshall and Daufin, 1995; van der Horst, 1995), but researchers in juice filtration have paid little attention to it. Loss of permeation flux has been reported for citrus juice and model solutions (Shomer and Merin, 1984) and was blamed on operating conditions in microfiltration (Todisco et al., 1996). Snir et al. (1995) suggested that pectin methyl esterase (PME) electrostatically binds to soluble pectin and is retained by UF membranes in grapefruit juice.

Orange juice properties, appearance, and consumer acceptability closely involve the subject of cloud stability. Juice filtration is a process that results in two main streams of permeate and retentate. While permeate is a clear aqueous stream, the retentate contains cloud constituents, including several colloidal fractions such as pectin and proteins (Shomer et al., 1985). The major intent in filtering juice is for a subsequent debittering step (Hernandez et al., 1992) and to separate between the clear serum and the cloud matter before heating the latter to achieve stability (Cross, 1989). Cloud instability is a consequence of a series of events such as the action of PME, which causes pectin demethoxylation, that results in the formation of a pectate gel (Baker, 1980; Bruemmer, 1980; Joslyn and Pilnik, 1961; Sinclair, 1984) and to protein interactions, which form complexes and undergo coagulation/flocculation (Shomer et al., 1991; Snir et al., 1995; Thakur et al., 1997). Therefore, it is common to heat treat the juice immediately after its extraction from the fruit to inactivate the enzymes and also to reduce bacterial counts. This process reduces the formation of pectate gels, which lead to clarification. However, in some cases the juice loses its stable opaque appearance despite the above treatment.

Before industrial processing of orange juice with membranes, the pectin in the juice is degraded by exogenous enzymes to reduce the viscosity of the juice to increase permeation fluxes (Stutz, 1993). Since membrane permeation flux depends on temperature through the temperature dependence of viscosity, it is common in industrial membrane processing to operate at temperatures around 50−55 °C. Since exogenous enzymes are added before the membrane treatment, the residual enzymatic activity must be stopped, which is done by an additional heat treatment. However, heat treatment of natural juice and pectin degradation lead to undesirable processes that result in cloud flocculation

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and clarification (Shomer et al., 1999a,b). Hence, what is of basic importance in relation to filtered juice is to examine the effect of treatments (such as heat and enzymatic pectin degradation) that induce flocculation of the juice components on membrane performance.

In the present study we attempted to explain the effects of heat treatment and enzymatic pectin degradation of orange juice on ultrafiltration performance and on membrane fouling and its removal.

MATERIALS AND METHODS

Shamouti oranges obtained from the orchard of The Volcani Center, Bet Dagan, Israel, were halved and hand-squeezed with an electric squeezer. Immediately after extraction, the juice was screened and kept in an ice bath until the volume collected was heat treated with a plate heat exchanger at 90 °C for 5 min, cooled to 30 °C, and kept until the experimental run. For degradation of the pectin in the juice, enzyme [Ultrazyme-100 G, Novo Ferment, Switzerland (consisting mainly of EC 4.2.2.10)] was added at 0.1% (1000 ppm) level at 30 °C and incubated for various periods of time. Filtration experiments were run with juice after the enzymatic action, with or without additional heat treatment, in which the juice was heated to 90 °C for 5 min using the plate heat exchanger. Permeate and retentate viscosities were measured using a Cannon−Fenske capillary viscometer in a constant-temperature bath.

Filtration Experiments. The filtration rig used was a pilot unit (kindly provided by TechSep, Miribel, France) equipped with a 100−1000 L h^{-1} variable speed positive displacement pump, a flow meter, and three pressure gauges (P_{in}, P_{out}, and $P_{permeate}$). The membrane used was a Carbosep M5 of 10 kDa cutoff, 1.2 m long, 6 mm hydraulic diameter, with a 2.26 × 10^{-2} m^2 ZrO_2 effective filtering layer on a porous carbon support.

Operating conditions were as follows: The rig was operated in a recycle mode with transmembrane pressure (TP) at 0.75 bar, flow velocity (v) at 5.5 m s^{-1}, temperature (T) at 40 °C, and time (t) at 120 min.

From the operating conditions, it was possible to calculate the resistance (R) of the total fouling according to Darcy (Taddéi et al., 1989; Daufin et al., 1991):

$$J = \frac{\mathrm{TP}}{\mu R} \tag{1}$$

where J is the permeation flux (L h^{-1} m^{-2}), TP is the transmembrane pressure (kPa), μ is the viscosity (Pa s), and R is the total resistance (m^{-1}).

According to resistances in series:

$$R = \mathrm{Rm} + \mathrm{Rf} \tag{2}$$

and

$$\mathrm{Rf} = \mathrm{Rif} + \mathrm{Rrf} \tag{3}$$

where Rm is the clean membrane resistance (m^{-1}), Rf is the overall fouling resistance (m^{-1}), Rif is the irreversible fouling resistance (due to intermembrane blocking and adsorption) (m^{-1}), and Rrf is the reversible fouling resistance (due to concentration polarization) (m^{-1}). This enables us to calculate each resistance by itself.

The presentation of the above indices could be done by using the normalized fouling indices, Rrf/Rm and Rif/Rm, which account for changes in Rm that could had influenced the final resistance recorded (Daufin et al., 1993).

According to Darcy, Rm and Rif were calculated from the pure water flux of the clean membrane (w) and the membrane after fouling (w′) (Taddéi et al., 1989; Gésan et al., 1995):

$$\mathrm{Rm} = \frac{\mathrm{TP}}{\mu_w J_w} \tag{4}$$

Table 1. Treatments of Various Filtration Experiments and Filtration Indices[a]

exp	enzyme	time	heat inactivation	Rm	Rf	Rrf	Rif
1	fresh juice			34.13	231.08	42.65	188.43
2	+	21 h	−	34.13	55.1	6.22	48.88
3	+	20 h	+	34.13	168.2	10.37	157.83
4	+	2 h	−	36.56	108.83	21.55	87.28
5	+	1 min	+	35.71	487.51	157.59	329.92
6	−	23 h	−	26.03	266.79	118.31	148.48
7	−	22 h	+	27.42	350.63	44.22	306.41
8	+	1 h	+	27.42	350.99	113.65	237.34

[a] Rm, Rf, Rrf, and Rif are × 10^{12} m^{-1}.

and

$$\mathrm{Rif} = \frac{\mathrm{TP}}{\mu_{w'} J_{w'}} - \mathrm{Rm} \tag{5}$$

New membrane conditioning, before an experiment, and membrane cleaning were done according to Daufin et al. (1991). A membrane was considered clean when the cleaned membrane resistance was within 5% of the initial membrane resistance. Water used for membrane conditioning, rinsing, and cleaning was tap water filtered consecutively through 100-μm and 0.2-μm cartridge filters.

Cleaning agents used were NaOH at pH 11 with the addition of 1000 ppm of NaOCl, followed by HNO_3 at pH 1.5. A cleaning cycle was as follows: 5 min, water rinse; 30 min, hydroxide wash; 5 min, water rinse; 15 min, acid wash; 10 min, water rinse.

If, after the final rinse, the water flux did not return to within 5% of the original flux, an enzyme cleaning was performed using Terg-A-Zyme (Alconox Inc.) at a concentration of 1% for 1 h, followed by a short wash cycle (10 min hydroxide, 5 min acid) as above. It should be noted that, after the first experiments, the enzyme cycle was adopted as a routine stage in the cleaning cycle.

Statistical Analysis. The experimental data was analyzed in relation to the main effects of heat and enzyme incubation time as treatments in a random block design with UF runs as experimental units. The calculated total fouling (Rf) after 120 min of filtration was analyzed by ANOVA.

RESULTS AND DISCUSSION

Table 1 summarizes the treatments examined in this study to compare the juice behavior during subsequent UF. The treatment comprised enzymatic treatments of various durations and heat treatments. In Figure 1, the effect of heating on the evolution of the fouling resistance is presented. UF performance with fresh juice was compared with two runs with juice, which had been held at 30 °C for overnight, with and without heating, respectively. Fresh juice and juice after 23-h incubation without added enzyme resulted in a little higher but rather similar fouling evolution, whereas with the 22-h heated juice there was a >30% increase in the fouling resistance. In order, to further assess the effect of heating, samples were prepared with added enzyme, with and without heat inactivation before filtration (Figure 2). After a short (1 and 2 h) or long periods of enzyme action (20 and 21 h), heating resulted in a major increase in fouling resistance. Since filtration efficiency is affected by solution viscosity (as described by Darcy's equation), the juice viscosity was measured and compared between experiments with added enzyme and heat treatment and without either (Figure 3). Enzymatic degradation of pectin resulted in significant change (at 5% level) in viscosity of the juice (Table 2) according to length of enzyme action. Moreover, the

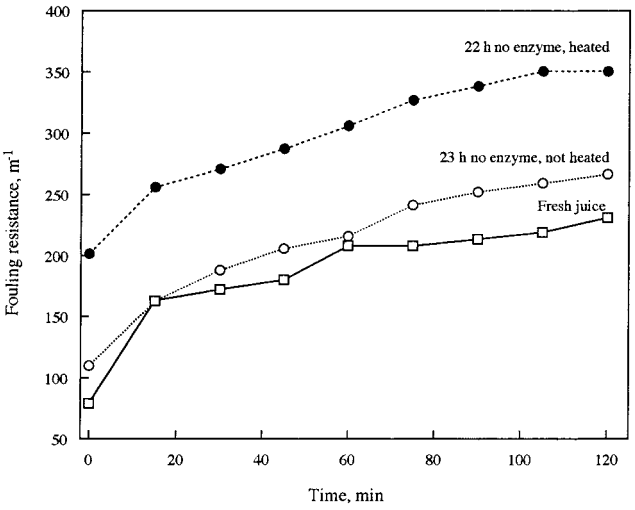

Figure 1. Fouling evolution with time as affected by heat treatment of the juice. □, freshly squeezed juice (control); ○, 23-h incubation without added enzyme, without heat treatment; ●, 22-h incubation without added enzyme, with heat treatment.

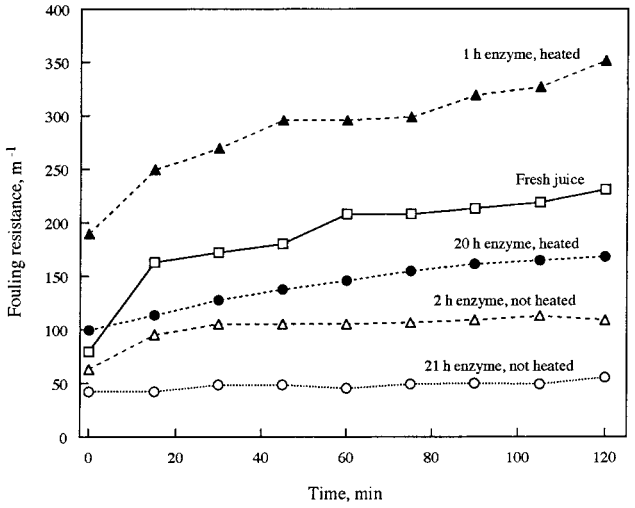

Figure 2. Fouling evolution with time as affected by enzyme addition, with and without heat treatment of the juice. □, freshly squeezed juice (control); ○, 21-h incubation with added enzyme, without heat treatment; ●, 20-h incubation with added enzyme, with heat treatment; Δ, 2-h incubation with added enzyme, without heat treatment; ▲, 1-h incubation with added enzyme, with heat treatment.

additional heat inactivation did not change the viscosity but always resulted in increased fouling resistance (Figure 1). The duration of the enzyme action (2 vs 21 h) reduced fouling by 50% (Figure 4) in what could be attributed to the reduction in viscosity caused by the enzyme action (Figure 3). Nevertheless, the major effect was due to the heat treatment applied to each sample, a phenomenon that can be clearly seen by comparing the fouling resistance when the juice was heat-treated immediately after the enzyme addition, and filtered and when the fresh juice was filtered without treatment (Figure 4). It is clearly seen that heating by itself resulted in tripled fouling. Comparison between the viscosities measured in the two experiments underlines the effect of heating, since the viscosity of the heat-treated juice was lowered after a short time, but the juice caused much greater fouling (Figure 5).

Membrane fouling can be considered as a series of resistances, as explained in Materials and Methods.

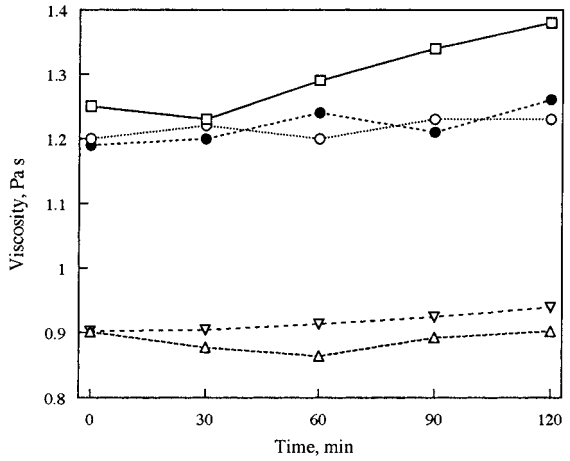

Figure 3. Retentate viscosity during filtration. □, freshly squeezed juice (control); ○, 23-h incubation without added enzyme, without heat treatment; ●, 22-h incubation with added enzyme, with heat treatment; ▽, 21-h incubation with added enzyme, without heat treatment; △, 2-h incubation with added enzyme, without heat treatment.

Table 2. ANOVA Table of Viscosity of Juice of Various Treatments[a]

exp	viscosity (Pa s)[b] ($\times 10^{-4}$)	variance ($\times 10^{-10}$)
1	13.000[a]	0.387
2	8.872[b]	2.687
3	8.842[b]	4.937
4	9.164[c]	2.353
5	9.836[d]	2.843
6	12.200[e]	2.300
7	12.200[e]	8.500
8	9.680[d]	56.500

[a] For details on experiments and viscosity, see Table 1 and Figure 3. [b] Means (of five measurements along the filtration experiment) marked by different letters are significantly different ($p < 0.05$).

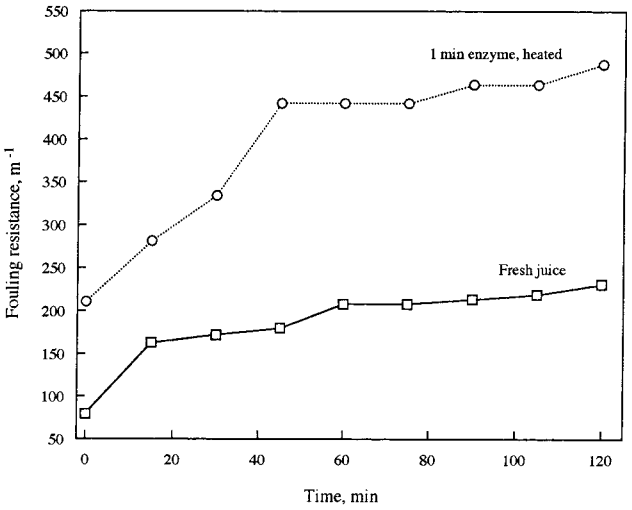

Figure 4. Immediate effect of heat treatment after enzyme incubation. □, freshly squeezed juice (control); ○, 1-min incubation with added enzyme, with heat treatment.

Calculation of the reversible (Rrf) and the irreversible (Rif) fouling resistances also points to the influence of the heat treatment. This is in addition to the presence of a substantial irreversible fouling layer in most of the treatments, which is indicative of surface interaction between fouling material from the juice and the zyrconia filtering layer of the membrane. Membrane fouling during juice filtration has been considered to be mainly

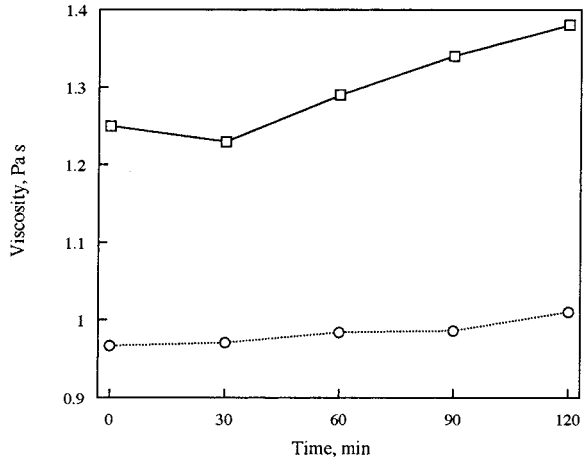

Figure 5. Retentate viscosity after immediate heat treatment and enzyme incubation. □, freshly squeezed juice (control); ○, 1-min incubation with added enzyme and with heat treatment.

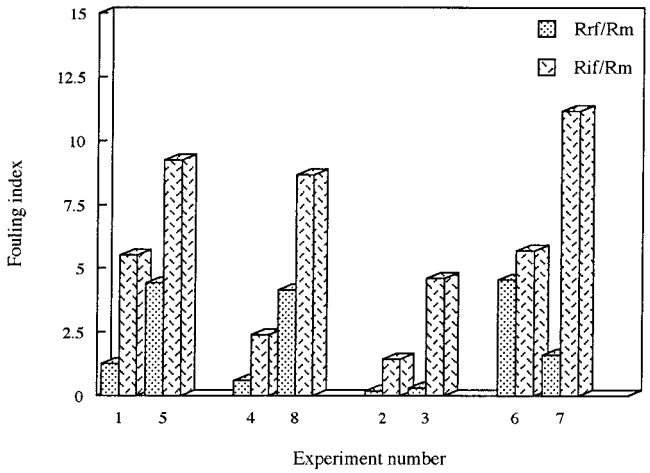

Figure 6. Fouling indices for the various experiments after after the end of filtration.

exp	enzyme	time	heat inactivation
1	fresh juice		
5	+	1 min	+
4	+	2 h	−
8	+	1 h	+
2	+	21 h	−
3	+	20 h	+
6	−	23 h	−
7	−	22 h	+

Rif, irreversible fouling ($\times 10^{12}$ m^{-1}); Rrf, reversible fouling ($\times 10^{12}$ m^{-1}).

Table 3. ANOVA Table for Final Rf Values of Filtration Experiments with and without Heating for Different Incubation Periods

not heated			heated		
exp	incubation time (h)	Rf ($\times 10^{12}$ m^{-1})	exp	incubation time (h)	Rf ($\times 10^{12}$ m^{-1})
1	0.017[a]	231.08	5	0.017	487.51
4	2	108.83	8	1	350.99
2	21	55.10	3	20	168.20
6	23	266.79	7	22	350.63

source of variation	*df*	sum of squares	*F* ratio	prob > *F*
heat	1	60378.125	15.4060	0.0294
incubation time	3	69944.375	5.9490	0.0886
error	3			

[a] Fresh juice was considered equal to 1 min holding before filtration.

due to pectins (Shomer and Merin, 1984; Stutz, 1993; Thomas and Barefoot, 1989) and pectin−pulp deposits (Capannelli et al., 1994). The finding of the present study and those reported by Shomer et al. (1999a,b) indicate a possible involvement of proteins in the fouling process of orange juice, which could be related to the intensified and accelerated flocculation due to heating (Shomer et al., 1999a,b). This might be the outcome of some protein denaturation by heating that results in differently structured flocculated proteins and its association with pectin, as has been observed in peel extract (Shomer, 1988; Shomer et al., 1991). Such a change in the cloud structure as exhibited by flocculation due to possible protein−pectin association (Shomer et al., 1999a,b) might also be responsible for the increased fouling that was observed whenever the juice was heat-treated. The involvement of proteins in the fouling process is supported by the need to use proteases in the cleaning cycle for full water flux restoration after UF runs.

Examination of the normalized fouling indices, Rrf/Rm and Rif/Rm (Daufin et al., 1993), at the end of the experiment reveals the impact of the heat treatment, which almost doubled Rif/Rm except for exp 6 (Figure 6).

The statistical analysis given in Table 3 reveals a significant effect of the heat treatment at the 5% level ($F = \sim 3\%$), while the incubation time was not significant. It should be noted that in exp 1 the fresh juice was compared to exp 5 of 1 min enzyme action since enzyme addition always contributed to fouling. The comparison performed for the other pairs, despite the longer time of enzyme action, is justified by the consistent contribution of time to lowering fouling (Table 1).

In all the runs, the irreversible phenomenon was always greater than the reversible, which indicates the formation of fouling substances that could not be removed solely by the shear force of the water rinse. It should be noted that each pair of treatments (except for exps 1 and 5) comprised a control and a heat treatment. This points to the possible occurrence of some interactions between the filtered juice and the membrane-filtering layer after heating. The reversible fouling was very low after a long period of enzyme action (Figure 6, exps 2 and 3), and it may be that some of the pectin also participates in the reversible fouling and that, after the degradation of the pectin by the enzyme, its contribution was substantially reduced. Of major interest is the influence of heat treatment after 1 min of enzyme action (Figure 6, exp 5). It is compared to the fresh juice experiment (Figure 6, exp 1), which was not heat treated. It is assumed that the difference in fouling between the experiments is not the contribution of the added enzyme, since in comparison to exp 4 (Figure 6) we can discard the added enzyme as the major contributor to Rif or Rrf. Thus, heating by itself, even after such a short period of time, points to a possible involvement of the temperature in the formed juice−membrane interactions (Figure 6). Furthermore, when juice is set at 30 °C for overnight without enzyme (Figure 6, exp 6) there was greater fouling than with EPD-treated juice (Figure 6, exp 4). It is assumed that endogenous PE is active at 30 °C for such a period of time and exposes charges on the pectic polymer that might interact with the cloud proteins, thus leading to increased fouling (Imeson et al., 1977; Thakur et al., 1997).

Table 4. Pure Water Flux and Recovery of Membrane Cleanliness after Two Consecutive Cleaning Cycles (exp 5)

wash cycle	pure water flux (L h^{-1} m^{-2})	Rm ($\times 10^{12}$ m^{-1})	% of previous Rm
1st cycle			
after enzyme	42.48	95.98	38
after acid	61.06	66.77	55
after base	92.92	43.88	83
2nd cycle			
after enzyme	84.96	47.99	76
after acid	92.92	43.88	83
after base	108.85	37.41	98

In fouling by whey, it is usually considered that Rif is caused mainly by proteins, while Rrf is due to minerals (Taddéi et al., 1989; Daufin et al., 1992a,b; Daufin and Merin, 1995). Such a contribution of proteins to the fouling during orange juice filtration might be possible since heating in the presence of high molecular weight pectin and soluble proteins, caused coagulated proteins to appear as a deposit on the pectic polymer. This phenomenon has been observed both in model solutions (Imeson et al., 1977; Shomer et al., 1982) and in tissue extracts (Shomer, 1988).

Membrane Cleaning. Cleaning of the membrane was assessed by comparing its initial Rm with its Rm after cleaning (Daufin et al., 1991). In a preliminary experiment, it was noted that membrane cleaning by a standard cleaning procedure was insufficient, and a second wash cycle was required to achieve an acceptable Rm. In certain cases, even a second cleaning was not enough, so the membrane was left soaking in the enzyme solution for 1 h before an acceptable Rm value was obtained. It was concluded that, for efficient cleaning of zirconia membranes after juice filtration, an enzyme cleaning cycle is essential and must be performed routinely. As presented in Table 4, two consecutive cleaning cycles including enzyme were necessary to obtain an Rm value within 5% of the previous one. The use of proteases in the enzyme detergent for the removal of the fouling layer and the restoration of flux increases the likelihood that proteinacous material formed part of the fouling layer, since only after enzymatic action was full membrane performance restored.

CONCLUSIONS

During ultrafiltration of orange juice with inorganic membranes, heating of the juice prior to the filtration experiment resulted in an increase of the fouling indices. Irreversible fouling was always doubled while reversible fouling depended on the treatment. It is clearly seen that heating after pectin degradation resulted in lowered fouling, but nevertheless, any heat treatment given to the juice before filtration resulted in lower fluxes. The fouling is suggested to be the outcome of cloud particles that interact with the membrane-filtering layer.

It seems that UF of orange juice is restricted by membrane fouling as is true for most other food liquids. When better performance of the filtration is desired, industrial processes use enzymes to reduce juice viscosity, thus enhancing permeation flux. However, when enzyme addition is accompanied by a complimentary heat treatment to stop the enzyme action, an increase of fouling could be expected as was found in the present study. For efficient membrane cleaning and flux restoration, a proteolytic enzyme detergent wash cycle is needed.

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