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ARTICLE *in* JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY · MAY 1998

Impact Factor: 2.91 · DOI: 10.1021/jf970793h

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Clarification of Pineapple Juice (*Ananas comosus* L. Merryl) by Ultrafiltration and Microfiltration: Physicochemical Evaluation of Clarified Juices, Soft Drink Formulation, and Sensorial Evaluation

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Concentrated pineapple juice reconstituted to 12 °Brix was used to obtain three types of clarified juices by ultrafiltration (UF) and microfiltration (MF) systems with polysulfone and ceramic membranes. They were evaluated by physicochemical analysis, in comparison to the reconstituted juice control, as well as through the sensorial evaluation of the soft drinks obtained by them. The best volume recovery was observed with 50 000 Da polysulfone membranes. Best components recovery was obtained with the 0.22 μm ceramic membrane. The 50 000 Da polysulfone membrane presented better efficiency to remove tannins and pectin. Both membranes of 50,000 D cutoff presented the same performance to decrease turbidity. The 0.22 μm ceramic membrane presented the best overall performance. Clarified juice flow rate was highest in the juice obtained using the 0.22 μm ceramic membrane (52.02 L m^{-2} h^{-1}). The three soft drinks formulated from the clarified pineapple juices presented no differences, at a significance level of 5%.

Keywords: *Pineapple juice; pineapple soft drink; ultrafiltration; microfiltration; physicochemical and sensory evaluation; Ananas comosus*

INTRODUCTION

In 1996, the Brazilian pineapple fruits production was 843 940 fruits in 40 760 ha (yield = 20 705 fruits/ha). The prevision by IBGE (1997) for 1997 is about 939 229 fruits in 42 630 ha of area (yield = 22 032 fruits/ha).

The strength and concentration of pineapple juices are considered byproducts from the canned pineapple industry and are obtained from shells, heart, and central cylinder or from fresh fruits that do not pass quality specification such as overipe (Hulme, 1971; Hodgson and Hodgson, 1993). Thailand continued to be the world's leading producer of pineapple juice concentrated with 87 000 in 1991 according to the USDA (1992). The Brazilian pineapple juice exportations during the first semester of 1992 were about 810 t, considered small as compared to concentrated orange juice that in the same period reached 187 000 t according ASTN (1992). Although the volume of pineapple juice produced and exported from Brazil is inexpressive, the obtainment of clarified pineapple juice for the formulation of soft drinks could be a good alternative. Milnes et al. (1986) reported that the recent tendency of international marketing is toward soft drinks and other beverages that are most welcome when they are without turbidity.

The conventional clarification process involves many steps, such as enzymatic treatment (depectinization), cooling, flocculation (gelatin and bentonite and diatomaceous heart), decantation, and filtration, requiring more costs with personnel, equipment, and physical space (Heartherbell et al., 1977).

The ultrafiltration (UF) and microfiltration(MF) have been applied in vegetal juices and pulps and wines industries, reducing many steps of the conventional clarification (Michaels, 1981). The pectinolitic enzymes can be reduced and sometimes can even be eliminated (Vrignaud, 1983; Breslau, 1984).

According to Barrichello (1977), Short (1983), Cheryan (1986), Rautenbach (1989), and Itoua-Gassaye (1990, 1991a), UF and MF have as a basic principle the separation of the molecules according to size or molecular weight cutoff (MWC) of the membrane, producing a permeate and a retentate.

In the conventional filtration process, the solutions cross perpendicularly through the membrane, while in the UF/MF processes, the feed is pressurized and the solution flows parallel (cross-flow or tangential filtration) at the membrane surface and the permeate passes through it. The solution can sometimes be backflushed in the system, minimizing the fouling or concentration polarization (Milnes et al., 1986; Maldonado, 1991).

Although the UF/MF processes need a high initial investment, we believe that in the special case of fruit juices, they can be used in Brazil by its high performance, since undesirable compounds such as pectins and tannins can be removed by a single pass through the UF/MF system membranes, only depending on the selection of the adequate MWC and structure of membrane, according to the juice to be clarified.

The basic purpose of this research was to evaluate the physicochemical behavior, in terms of constituents loses and recoveries from the reconstituted pineapple juice at 12 °Brix to clarified ones after the different processes in UF/MF systems and different MWC of the membranes available as well as to formulate and evaluate soft drinks obtained from the clarified juices.

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Table 1. Chemical Composition of Pineapple Soft Drinks

ingredients/additives	Brazilian legislation	formulation
soluble solids (°Brix)		10.8
pineapple clarified juices (10 °Brix)	10.0 (min.)	10.0%
sucrose	qsp	qsp
tritable acidity (% w/v) as citric acid	0.07 (min)	0.2%
sodium benzoate	0.05 (max)	0.05 g/100 mL
indigotin blue	0.01 (max)	0.01 g/100 mL
tartrazin	0.01 (max)	0.01 g/100 mL
naturals and identic to natural aroma (g/L)	qsp	0.40
carbon dioxide (vol)	1.0 (min)	2.8
water	qsp	qsp

MATERIALS AND METHODS

Materials. Concentrated pineapple juice (60.5 °Brix) was reconstituted to 12 °Brix and tammied to remove excess suspended pulp.

Equipments. Millipore Ceraflo tubular pilot system composed of one ceramic membrane (α-alumin) MWC 0.22 μm and 50 000 Da (capacity 12 L and 0.14 m^2 filtering area) and a DDS filtration plate and frame pilot system-M20 with 40 units of MWC 50 000 Da polysulfone membranes (capacity 8 L and 0.72 m^2 filtering area).

Operational Parameters. The processes in the different systems and membranes were conducted at room temperature. The operational parameters were setting according to the systems, applying the following inlet, outlet, and transmembrane pressures: for the plate and frame system with 50 000 Da polysulfone membranes 116.0, 29.0, and 72.52 psi, respectively, with 20 min for both processes. For the tubular system, when 50 000 Da ceramic membrane were applied, the inlet, outlet, and transmembrane pressures were 71.0, 44.0, and 56.0, respectively, in 55 min of processes; for 0.22 μm ceramic membrane they were 21.0, 9.0, and 15.0, respectively, in 60 min of process.

Physical and Chemical Analysis. The physical and chemical analysis of the clarified juices were determined in triplicate and compared with the reconstituted pineapple juice control at 12 °Brix according to AOAC techniques for soluble solids, acidity as citric acid, glucose, sucrose, and total solids. The organic acids were determined by HPLC in a UNICAM−Crystal Integrated System with diodine detector. The samples were filtered previously in C18 Sep-Pak cartridges and compared with the organic acid patterns. The absorbance used was 214 nm, and the results were expressed in mg/100 mL. Tannins were determined by a spectrometric AOAC method using the Folin−Denis reagent, and the results were compared with a pattern curve of tannic acid and the absorbance at 760 nm. For pectin, the Bitter and Muir (1962) modified method was used, and the absorbance was at 530 nm. The results of both tannins and pectins were reported in mg/100 mL. The mineral compounds were determined by an atomic absorption Perkin-Elmer spectrometer, and the results were expressed in mg/100 mL. For turbidity measures, the transmittance used was 650 nm (ICUMSA-Copersucar method, 1979). The results of the physicochemical analysis were calculated as mean and standard deviations and submitted to a statistical treatment by ANOVA variance analysis at a significance level of 5%.

Soft Drinks Formulation. Table 1 shows the ingredients and additives used for the three soft drinks formulation prepared according to the Brazilian legislation of the Ministério da Agricultura (1974), with the clarified pineapple juices obtained by the two systems (plate and frame and tubular). The three soft drinks were formulated according to Table 1 and as specified in Materials and Methods. The ingredients were added according to the clarified pineapple juices sugar and citric acid contents.

After formulation, the soft drinks were analyzed considering Brix, pH, tritable acidity, carbon dioxide, and ratio. The results were according to previous theoretical calculations.

Soft Drinks Sensorial Evaluation. Samples of the three soft drinks formulated from the clarified pineapple juices were stored at room temperature for 90 days. After 15 and 90 days of storage, the samples were submitted to a Triangular Difference Test using a pannel of 64 untrained tasters. The results were statistically analyzed using the Roessler Table at a significance level of 5% (Roessler et al., 1978). The Triangle Test presents as an advantage a 1/3 mark of statistical probability for detecting low differences correctly according Garruti (1987). The tasters were previously instructed about the test applied. The tests were carried out in a period of 15 and 90 days after the formulation, and the soft drinks were served cooled at 10 °C.

RESULTS AND DISCUSSION

Flow and Recovery Juice Rates. The comparison of the clarification processes efficiency is shown in Table 2. Operational parameters as well as the flow rate and recovery of clarified juice rate of different membrane system can be observed.

In the two processes with polysulfone membranes, only in the first one was the membrane cleaned before the process. In the first experiment with the 50 000 Da polysulfone membrane, the flow rate increased from 24.58 to 40.83 L m^{-2} h^{-1} between 8 and 10 min of the process. At first, we thought there was possibly a rupture in the membrane pore, but after 2 min the flow was re-established. In the second experiment with the same membranes without previously being cleaned (50 000 Da), the flow increased between 6 and 8 min, and afterwards, there was a oscillation in it that could occur because the polarization concentration of the solutes of the juice was higher in the membrane surface in this second experiment.

In the other two processes using ceramic membranes of 50 000 Da and 0.22 μm after 20 min of operation with 50 000 Da membrane, the flow rate decreased from 51.43 to 49.71 L m^{-2} h^{-1}, and then the juice was recirculated for 5 min into the system to minimize the polarization concentration. The flow rate increased to 104.57 L m^{-2} h^{-1} in 3 min, and after 7 min the flow rate decreased at 47.15 L m^{-2} h^{-1} and remained stable for 55 min. When the 0.22 μm membrane was used, we observed that the flow rate decreased from 63.43 to 50.57 L m^{-2} h^{-1} between 40 and 50 min of operation. The juice was recirculated, and the flow rate was restored until 60 min. Although the time of process was shorter (20 min) in the plate and frame system with polysulfone membranes, we believe the tubular one with the ceramic membrane had best performance presenting higher flow rates than the plate and frame system with an additional possibility of cleaning the equipment without removing membranes for a new process.

The clarified pineapple juices obtained by the two processes using 50 000 Da polysulfone membranes were mixed because there was not enough volume to prepare the beverages (soft drinks) and because they were compatible.

The best juice recovery rate (87.6%) was found in the second process of the 50 000 Da polysulfone membranes, without cleaning the plate and frame system. Thomas et al. (1986) had a juice recovery rate of 85% when applying UF in depectinized apple purée. The clarified juices flow rates were better when using the 50 000 Da and 0.22 μm ceramics membranes (46.8 and 52.0 L m^{-2} h^{-1}) than the 50 000 Da polysulfone ones (25.0 and 22.7 L m^{-2} h^{-1}). Lowest values were reported in depectinized passion fruit juice clarification using polysulfone

Table 2. Operational Parameters, Flow Rates, and Recovery Clarified Juice Rate

membrane/system/MWC	polysulfone plate and frame 50 000 Da (A)	polysulfone plate and frame 50 000 Da without cleaning (B)	ceramic tubular 0.22 μm (C)	ceramic tubular 50 000 Da (D)
inlet pressure (psi)	116.0	116.0	21.0	71.0
outlet pressure (psi)	29.0	29.0	9.0	44.0
transmembrane pressure (psi)	72.5	72.5	15.0	56.0
flow rate (L m^{-2} h^{-1})	25.0	22.7	52.0	46.8
recovery clarified juice rate (%)	77.6	87.6	67.3	69.5

Table 3. Reconstituted Juice and Clarified Juices Composition and Recoveries (Mean and Standard Deviations)

parameters/juices	control juice (A)	polysulfone 50 000 (B)/recovery (%)		ceramic 50 000 (C)/recovery (%)		ceramic 0.22 μm (D)/recovery (%)	
Brix	12.0 ± 0.1	10.3 ± 0.2	85.80	10.3 ± 0.05	85.80	11.3 ± 0.2	94.16
pH	3.73 ± 0.01	3.73 ± 0.0	100	3.73 ± 0.01	100	3.73 ± 0.01	100
acidity (mg/100 mL)	0.48 ± 0.001	0.42 ± 0.001	87.50	0.40 ± 0.005	83.33	0.44 ± 0.002	91.6
glucose (g/100 mL)	3.11 ± 0.002	2.80 ± 0.023	90.03	2.77 ± 0.021	89.06	2.80 ± 0.009	90.03
sucrose (g/100 mL)	7.99 ± 0.003	5.93 ± 0.033	74.21	7.05 ± 0.016	88.23	7.31 ± 0.013	91.48
total sugars (g/100 mL)	10.95 ± 0.012	8.75 ± 0.058	79.90	9.82 ± 0.006	89.68	10.12 ± 0.024	92.42
pectin (mg/100 mL)	0.058 ± 0.008	0.004 ± 0.001	6.89	0.005 ± 0.001	8.62	0.020 ± 0.002	34.48
turbidity (650 nm)	932.5 ± 2.000	70.8 ± 0.702	7.59	73.9 ± 0.251	7.92	133.6 ± 0.351	1.43
tannins (mg/100 mL)	8.60 ± 0.010	2.51 ± 0.007	29.18	5.16 ± 0.009	60.00	6.30 ± 0.012	73.25
ratio (Brix/acidity)	25	24.52		25.75		25.68	

membranes with 100 000 MWC (Chiang and Yu 1987; Sheu et al., 1987; Chao et al., 1992). The membrane structure and the configuration of the two systems (Plate and frame and tubular) could be a limiting factor since polysulfone membrane structure is less resistant. This may cause a rupture in the membranes pores causing an enlargement around them and, consequently, increasing the flow rate but without the necessary efficiency. Sometimes fouling may still occur and/or concentration polarization may decrease the flow rates. Rautenbach et al. (1989), Merlo et al. (1993), and Fane (1994) reported that the concentration polarization and fouling can be minimized through the pressure decreasing, turbulence increasing, or still using some operations such as backwashing, pulsed feed flow, or intermittent on−off.

With regarding to the recovery rate of clarified juices, the lowest values were in the ceramic membranes because the juice that remained (retentate) in the system was about 2 L instead of the 0.74 L retained in the 50 000 Da polysulfone membranes ones.

The mixture of clarified pineapple juices obtained by the two processes using 50 000 MWC polysulfone membranes (10.3 °Brix) were evaluated by physicochemical composition as well as the clarified pineapple juices obtained from 0.22 μm (11.3 °Brix) and 50 000 Da (10.3 °Brix) ceramic membranes separately.

Table 3 shows the physicochemical composition results of the three final clarified (ultrafiltered) pineapple juices, done in triplicate, as well as the standard deviations.

According to ANOVA statistical analysis, there were significance differences at a level of 5% among the clarified and the control pineapple juices (reconstituted to 12 °Brix) for all the parameters analyzed except for pH.

pH. There was no significance differences at a level of 5% for the pH values in the three juices according to Milnes (1986).

Soluble Solids. The soluble solids values decreased to a maximum value of 1.7 °Brix in clarified juices obtained by ceramic and polysulfone membranes systems. When 50 000 Da membranes (polysulfone and ceramic) were used, this values decreased from 12 to 10.3 °Brix. Studies involving physicochemical composition after MF and UF in depectinized pineapple juice reported by Itoua-Gassaye et al. (1991b) showed that there were no changes in Brix values. Wu et al. (1990) reported the highest differences for total solids in the polysulfone membranes systems, during the clarification of depectinized apple juices by UF/MF, using 0.1 μm ceramic membrane and 5000 and 50 000 Da polysulfone membranes. This result showed that a concentration polarization and fouling in the membrane surface during the processes may occur, causing loses in the process efficiency according to the membrane pore size (MWC). To minimize this effect the backflushing can be done during both processes with the tubular ceramic membranes system and in the polysulfone membranes systems according Doko et al. (1992).

Tritable Acidity. The highest tritable acidity as citric acid reduction (0.48 mg/100 mL to 0.40 mg/100 mL) was observed in the clarified juice ultrafiltered with 50 000 Da ceramic membranes (83.33% of recovery) showing that the citric acid content reduces as the MWC of the membranes decreases as well as (Gherardi et al., 1978; Krueger, 1992). It could also be observed with the glucose, sucrose, and total solids contents in the clarified juices by UF/MF.

Reducing and Nonreducing Sugars. The best recovery rates for reducing sugars (as glucose) of 90.03% and 91.49% for nonreducing sugars (as sucrose) were found in the ultrafiltered pineapple juices obtained from the 0.22 μm ceramic membrane system. Itoua-Gassaye et al. (1991a,b) reported similar values in pineapple juice clarification by UF.

Total Sugars. The total sugars best recovery (92.4%) was observed when the 0.22 μm ceramic membrane system was used.

Pectin. Pectin content reduced after UF/MF in both systems and in accordance with the decrease of the membranes MWC. When the 0.22 μm ceramic membrane system was used, 65.5% of the pectin was removed from the pineapple juice while 93.1% was removed when the 50 000 Da polysulfone membrane system was used. In previous studies about apple juice clarification, the enzymatic treatment step before UF/MF were suppressed, similar results were reported by Vrignaud (1983). Milnes et al. (1986) using a UF system for the clarification of depectinized apple juices obtained clear juices without turbidity.

Tannins. The tannins content was reduced in 70.82% when the 50 000 Da polysulfone membrane system was

Table 4. Organic Acids and Minerals Compounds

parameters	reconstituted juice (A)/recovery (%)	polysulfone 50 000 Da (B)/recovery (%)		ceramic 50 000 Da (C)/recovery (%)		ceramic 0.22 μm (D)/recovery (%)	
		Organic Acids (mg/100 mL)					
malic	247.81 ± 0.03	227.43 ± 0.03	91.77	219.84 ± 0.04	88.71	233.53 ± 0.03	94.23
citric	443.57 ± 0.04	423.87 ± 0.03	95.55	428.61 ± 0.10	96.62	437.98 ± 0.10	98.73
ascorbic	4.27 ± 0.13	0.81 ± 0.04	18.96	0.69 ± 0.04	16.16	0.81 ± 0.02	18.96
		Minerals Compounds (mg/100 mL)					
phosphorus	7.40 ± 0.05	5.88 ± 0.09	70.45	5.55 ± 0.08	75.0	6.13 ± 0.1	82.8
calcium	10.65 ± 0.04	10.20 ± 0.06	95.77	9.37 ± 0.06	87.98	10.66 ± 0.02	100
iron	3.00 ± 0.10	3.00 ± 0.15	100	1.00 ± 0.10	33.33	1.00 ± 0.10	100
magnesium	12.29 ± 0.10	10.61 ± 0.14	86.33	10.55 ± 0.08	85.84	9.81 ± 0.09	79.82

used. Contradicting, these data differ from the results reported by Pepper (1987) when, clarifying apple juice using UF 25 000 polysulfone membranes, the tannins content were not reduced. The tannins are responsible for the juice browning and astringency and must be removed.

Turbidity. Both processes using the two membrane systems reduced the turbidity from the reconstituted juices. The best result (92.41%) was found when the 50 000 Da polysulfone membrane systems were used. Previously, work had shown that turbidity decreasing was observed in ultrafiltered apple juices at the same wavelength (650 nm), but the MWC, membrane structure and systems used were not mentioned (Milnes et al., 1986; Itoua-Gassaye et al., 1991b).

Organic Acids. Table 4 shows the contents of organic acids and mineral compounds. The best results in recovery were found in the malic (94.24%) and citric (98.74%) acid contents (233.53 and 437.98 mg/100 mL) when the 0.22 μm ceramic membrane was used. Recent determinations done by Low et al. (1994) in 19 samples of commercial pineapple juices concentrated and reconstituted to 12.8 °Brix showed that the citric acid content was from 532 to 788 mg/100 mL and from 156 to 242 mg/100 mL for malic acid. The lowest recovery values were found in both 50 000 Da membrane systems as follows: 423.87 mg/100 mL of citric acid obtained by 50 000 Da polysulfone membrane clarification system and 219.84 mg/100 mL of malic acid by ceramic membrane system. Gherardi et al. (1978) and Krueger et al. (1992) reported similar results of citric acid content, 0.46−1.21 and 0.39 mg/100 mL, respectively. Substantial loses of ascorbic acid could be observed after the UF/MF with 50 000 Da ceramic membranes (83.8%), from 4.27 (reconstituted juice control) to 0.69 mg/100 mL (ultrafiltered juice). Although the temperatures applied during the processes were below 40 °C, Vrignaud et al. (1983) reported that the ascorbic acid content in the juice could be oxidized by air incorporation during the process and/or by the polifenoloxidase natural presence that can still rapidly oxidize ascorbic acid.

Minerals. The mineral compound recoveries can be observed in Table 4. The iron contents were reduced (66.67%) from 3.00 mg/100 mL in reconstituted and 50 000 Da polysulfone membrane clarified juices to 1.00 mg/100 mL in 50 000 Da and 0.22 μm ceramic ones.

The lowest value for recovery of phosphorus content (5.55 mg/100 mL) was found in the clarified juice obtained by the process with 50 000 Da ceramic membrane system when compared with reconstituted pineapple juice control (7.40 mg/100 mL). The lowest calcium and magnesium contents (from 10.65 to 9.37 mg/mL) was obtained in the 50 000 Da ceramic membrane system and (from 12.29 to 9.81 mg/100 mL) in the clarified juice obtained from the 0.22 μm ceramic membrane system, respectively. These results are compatible with those reported by Low et al. (1994). They observed variations in the calcium content from 8.7 to 22.7 mg/100 mL and from 10.3 to 20.1 mg/100 mL for magnesium content in commercial concentrated and reconstituted to 12.8 °Brix pineapple juices, respectively.

Table 5. Soft Drinks Sensorial Evaluation[a]

	storage at room temperature (day)					
	15			90		
test	no. of P	no. of M	results	no. of P	no. of M	results
soft drink 1 × 2	36	11	ns	36	11	ns
soft drink 1 × 3	36	17	ns	36	13	ns
soft drink 2 × 3	36	14	ns	36	11	ns

[a] no of P, panelists number; no. of M, panelists marks; ns, no significance at 5%. 1 × 2, soft drink from ultrafiltered juice of the 50 000 Da polysulfone membranes against 0.22 μm ceramic membrane. 1 × 3, soft drink from ultrafiltered juice of the 50 000 Da polysulfone membrane against 50 000 Da ceramic membrane. 2 × 3, soft drink from ultrafiltered juice from the 0.22 μm ceramic membrane against the 50 000 Da ceramic membrane.

Soft Drinks Sensorial Evaluation. The three soft drinks formulated from the clarified juices were tested in their stability, after storage for 15 and 90 days at room temperature, where the ingredients interaction occurs and can be observed with the top and bottom clear in this product. Turbidity or sedimentation were not observed. After this period, the sensorial tests were applied.

The Triangle Test consisted of presenting once the three soft drink samples to the 64 tasters on the 15th and 90th day after the formulation (128 repetitions).Two samples were identical, and one was different; the different one had to be identified. After the tasters had identified the different sample (soft drink), they had to answer some questions as to verify the difference in the grade of the taste: none (0); very little, but perceptible (1); very little, but defined (2); moderate (3); high (4); and extremely high (5). When the grade of difference was equal to or higher than 1, the taster had to describe the difference in a best way. No differences were detected by the tasters.

Table 5 shows that for the results found according to Statistical Roessler Tables (1978) there were no significance differences among the soft drinks at a 5% level (Meilgaard et al., 1987).

CONCLUSIONS

The different systems applied for the pineapple juice clarification were efficient since the obtained clarified juices presented the desirable limpidity when the MF/UF processes were used.

The clarified juices physicochemical parameters were reduced according to the MWC of membranes decrease.

The 0.22 μm ceramic membrane clarified juice presented the best recoveries in soluble solids, sugars, and acidity; its color and limpidity were similar to the 50 000 Da (ceramic and polysulfone membranes) clarified ones. According to the analysis of variance (ANOVA), there were significance differences at a 5% level among all the membrane processes.

The clarification process where the composition was near the pineapple reconstituted juice control at 12 °Brix was obtained by the 0.22 μm ceramic membrane UF system.

The sensorial evaluation by the Triangle Difference Test, applied in the three soft drinks formulated from the clarified pineapple juices through different systems and membranes, showed that there were no significance difference among them at a level of 5% of significance in storage periods of 15 and 90 days.

We believe that a previous juice depectinization before UF/MF processes could give the highest flow rates, minimizing concentration polarization and fouling in the membranes surface during the processes, as well as could remove the pectin more easily.

Studies about economic availability about these kinds of UF/MF systems seem to be necessary as well as the acceptance tests and soft drink desirable characteristics in terms of future consumers market.

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Received for review September 15, 1997. Revised manuscript received January 20, 1998. Accepted March 10, 1998. This work was supported by grants from Conselho Nacional de Desenvolvimento Científico (CNPq/UFRRJ) and Coca-Cola Indústrias Ltda.

JF970793H