Deacidification of Mandarin Orange Juice by Electrodialysis Combined with Ultrafiltration

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Abstract

The effects of electrodialysis (ED) alone or ED plus ultrafiltration (UF) on deacidification of mandarin orange juice were studied by using a commercial ED stack with ion exchange membranes. ED processing, reduced the total acidity of the juices by 30% (0.6 ~ 0.7% as total acidity) after 50 min and by about 60 ~ 70% (0.23 ~ 0.4% as total acidity) after 100 min, as compared to the control juice. However, the acidity reduction after 50 min of ED was determined to be suitable, when considering total acidity (0.6 ~ 0.7%, w/w) and current efficiency. There was no color change in the juices following ED, and the pH and Brix were only slightly decreased. Furthermore, ascorbic acid and citric acid concentrations showed only minor decreases, and amino-N, free sugar, and flavonoid contents remained almost unchanged. Therefore, we concluded that the nutritional integrity of the juice was maintained. ED combined with UF may be effective, not only in preventing membrane fouling, but also in preserving the nutrients, such as ascorbic acid, in citrus juice.

Key words: electrodialysis, ultrafiltration, mandarin juice, deacidification, sugar to acid ratio

INTRODUCTION

As in the rest of the world, citrus in Korea is consumed primarily as the fresh fruit; however, citrus processing has recently become very important in handling the 100 ~ 200 thousand tons of annual overproduction. Mandarin orange (*Citrus unshiu*) is the predominant species of Korean citrus. It is somewhat difficult to produce palatable citrus products from the Korean mandarin orange without acid reduction because it is characteristically too high in acid content and Brix is too low, as compared to other oranges. During the early part of the harvest season, citrus juices have high acidity levels that impart a tart flavor. Therefore, removing the acid from the juice is an important process for improving flavor. The ion-exchange resin filtration process has traditionally been used for acid reduction of citrus juices (1). However, that process has been considered inefficient because it requires regeneration of the resin used and reduces the nutritional quality of the citrus juices due to the removal of all charged ions.

Electrodialysis (ED) is a membrane process wherein ions are transported through ion-exchange membranes from one solution to another under the influence of an electrical potential (2). Under the influence of a direct current, ionic species with their positive and negative charges are driven in opposite directions through selectively permeable ion

exchange membranes. Many ED applications have been developed in various food industries, including: desalination, separation of organic acids, and adjusting the acidity of edible fluids (2,3). The choices and combinations of cation- and anion-exchange membranes mostly reflect material properties and their permeability to cations and anions. Commercial applications have typically utilized a conventional ED mode (with alternating anion- and cation-exchange membranes) for deacidification of fruit juices, although there is an increasing interest in bipolar membranes (2).

The deacidification of citrus juices by ED was reported earlier than by the ion-exchange resin process (4). In that ED process, the citrus juice and a KOH solution flowed through alternate compartments separated by anion exchange membranes. The electric current carried the citrate ions out of the juice into the KOH solution, and OH^- ions moved through the opposite membranes to neutralize the H^+ ions. However, the system (anion exchange membrane alone and strong alkali) was operated only on a pilot scale because of the low efficiency. Acceptable acidity levels have been obtained by deacidifying pineapple juice, orange juice, and grape juice (5) by using a conventional ED mode (with alternating anion- and cation-exchange membranes). ED systems with bipolar membranes have recently been utilized for adjusting the pH of fruit juices (6-9). At present,

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however, the bipolar membrane is costly and susceptible to fouling, limiting its usefulness for commercial applications in the juice industry, as compared to ion exchange membranes (10).

Mandarin orange juice has a high content of large particulate matter, and a high concentration of charged organic molecules. These materials limit ED applications because of concentration polarization and membrane fouling during operation. However, ED combined with ultrafiltration (UF) might prove effective if the offending materials could be temporarily excluded from the juice by UF before ED treatment and then returned to the juice.

The primary objective of this study was, therefore, to obtain basic data for the development of a continuous ED process to de-acidify mandarin orange juices. Changes in physicochemical parameters (conductivity, acidity, pH, and Brix) and major constituent contents in the juice undergoing ED were investigated while operating an ED system with a commercial ion exchange membrane stack alone, and in combination with UF.

MATERIALS AND MATHODS

Juice samples

The primary juice used in this study was a frozen concentrated orange juice (FCOJ) prepared by Jeju Regional Development Company in Korea from mandarin oranges (*Citrus unshiu*) harvested in December 2000. The FCOJ (65 °Brix) was kept frozen during transportation to the laboratory, divided into 1 kg packages, and stored at 25°C in a cold chamber for 2 months (for quality stabilization). The FCOJ (1 kg) was then diluted just before the experiments by adding 5 kg distilled water. The unmodified diluted orange juice was used as the control sample juice (CSJ) in this study. The CSJ had 10.8 °Brix, 1.02% (w/w) total acidity, pH 4.25, and color values of 36.15 (L), 1.49 (a), and 20.82 (b). These values were almost identical to the annual average values obtained from freshly squeezed mandarin orange juices.

Ultrafiltration

To study the combined effects of UF and ED, a UF system (Quixstand Benchtop, A/G Technology Co., Needham, MA, USA) was used. This system has a 500 K Dalton (NMWC) hollow fiber polysulfone membrane (UFP-500-C-4A, A/G Technology Co., Needham, MA, USA) with 650 cm² of effective membrane area. Two liters of the CSJ were applied to the UF system at 7°C. The permeate (1 L) was immediately subjected to ED for deacidification. The retentate (about 1 L) was stored at 0°C and used as a reconstituted and treated juice (RTJ) by combining with equal volumes of deacidified permeate produced by the ED. The amount of juice subjected to dialysis

was, therefore, reduced in half by using only the filtrate for ED and recombining it with undialyzed juice to obtain a juice with an intermediate acid content.

Electrodialysis

The CSJ and UF permeates were deacidified by a laboratory scale electrodialyzer (MicroAcylizer-S3, Asahi Chemical Co., Shizuoka, Japan). The cartridge (AC-230-550, Asahi Chemical Co., Shizuoka, Japan) of the electrodialyzer consisted of 10 pairs of cation- and anion-exchange membranes with a total surface area of 550 cm². The direct current supplied the needed voltage (0-18V) and current (0-3A). The sample, waste, and electrolyte liquids were placed into three 1 L PVC tanks and recirculated through the cartridge stack by pumps. Fig. 1 shows the process flow sheet of the laboratory scale electrodialyzer and membrane stack used in this study.

The sample liquids were circulated through each compartment containing a pair of ion exchange membranes. The anions from the organic acids in the sample are transferred into the waste liquid flow through alternate pair membranes. One liter of CSJ or UF permeate was used for every ED treatment. The electrode liquid and waste liquid were used with 1 L of 3% (w/v) NaNO₃ and pure water, respectively. The ED was operated at room temperature and a constant voltage (9.0 V) for 100 min in an auto-control mode. The pH, current, and conductivity were monitored
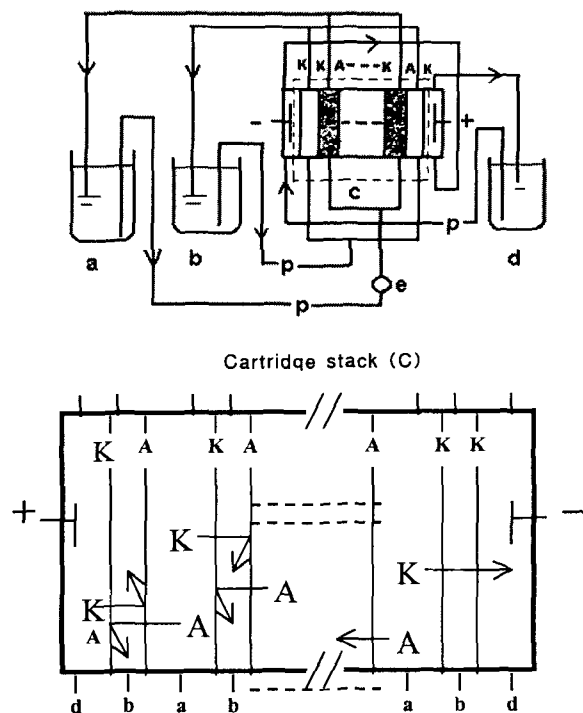

Fig. 1. Process flow sheet and cartridge stack of the electrodialyzer. A: anion and/ exchange membrane. K: cation and/ exchange membrane, a: sample juice, b: waste liquid, c: cartridge stack, d: electrolyte liquid, e: conductivity cell, and p: pump.

t 5 min intervals. The current efficiency of ED was calculated by the equation (11): ς = m F/nlt; where, m is the moles of acid (calculated as citric acid) reduced from the beginning time to time t, F the Faraday constant (96,500 /mol), n the number of cell pairs (10 in this study), and is average current at time t.

During the ED operation, 5 mL samples were collected very 10 min from the sample tank to measure the soluble olid index (°Brix) and total acidity. The ED-treated UF ermeate was immediately mixed with an equal volume f retentate and stored at 0°C to prepare the reconstituted nd treated juice (RTJ). The CSJ, CSJ treated by ED alone, nd RTJ were frozen and stored at 20°C and small portions vere later used for constituent analysis.

Analysis of the constituents

The color (L, a, b) and soluble solid index (°Brix) were neasured using a Color Difference Meter (TC-1, Tokyo)enshoku Co. Ltd., Tokyo, Japan) and an Abbe Refrecometer (501, Nippon Optical Works Co. Ltd., Tokyo, Ja-an), respectively. After diluting the sample 10 times with listilled water, the total acidity was measured by titrating vith a 0.1 N NaOH solution using 1% phenolphthalein as he indicator. Results were expressed as % citric acid. Citric cid, ascorbic acid, free sugars, and flavonoids of samples vere determined using HPLC (Model 501, Waters Associate Inc., Milford, MA. USA). Samples were filtered hrough 0.45 μm Millipore filter paper and Sep-pak C18 Waters Associate Inc., Milford, MA. USA). Three kinds f columns and mobile phases were used: 1) μ-Bondapak -18 column (3.9×300 mm) with a UV detector (Model 84, Waters Associate Inc., Milford, MA. USA) and 2% KH2PO4 (pH 2.5) for analysis of the citric acid and ascor-ic acid; 2) carbohydrate analysis column (3.9×300 mm) vith an RI detector (Model 410, Waters Associate Inc., Milford, MA., USA) and 80% CH3CN (v/v) for analysis f free sugars; and 3) a Phenomex luna 5 μC-18 column 2×150 mm) with a UV detector and methanol/acetic acid 1000/5, v/v) and H2O/acetic acid (1000/5) by gradient analysis was used for analysis of flavonoids. The above HPLC methods were described in detail elsewhere (12,13).

Each experiment was carried out in duplicate. Statistical analyses (p < 0.05) and graphics were performed using Sigma plot 5.01 (SPSS Inc., Chicago, USA).

RESULTS AND DISCUSSION

Preretreatment of CSJ by UF

CSJ was separated into retentate and permeate (equal volumes) by UF as a pretreatment before ED. The total acidity of both retentate and permeate were decreased to 5~10% of the CSJ, but the ratio of sugar to acid increased by a small amount (Table 1). The difference in measurement values may have been caused by physical changes upon dividing the CSJ into retentate and permeate, and not by chemical changes (14). However, the permeate became clearer (about 3 times) than the original CSJ (L value changed from 36.15 to 11.50). Large organic and colloidal materials in orange juices are troublesome foulants for ED membranes. When feed solutions to an ED stack are clean, the stack can be operated for years with little concern about membrane fouling (2). The fouling of ED membranes is best prevented by a pretreatment such as UF to eliminate foulants. Ideally, the particle size in fluid for ED should be limited to less than 10 μm.

It is, therefore, reasonable to assume that microfiltration to 1~5 μm can be used. However, 500K UF was used in this study because it made only a small difference in filtration efficiency of mandarin orange juice when compared to microfiltration.

ED of UF permeate and CSJ

Flavor enhancement is the primary reason for adjusting the ratio of sugar to acid in mandarin orange juice. Consumer preference surveys reveal that most consumers prefer a Brix (11~12) to total acidity (0.7~0.72%) ratio of around 15, and therefore it is often necessary to increase this ratio in the unprocessed juice (15). Removal of the acid from the juice by deacidification would easily increase this ratio.

During ED of the CSJ and its permeate for 100 min, the deacidification parameters such as total acidity, conductivity, pH, and Brix were measured to evaluate ionic exchanges. As shown in Fig. 2, the total acidity and conductivity were changed in a similar fashion between CSJ and permeate. The total acidity decreased proportionately with time from 1.02% (CSJ) and 0.89% (permeate) at 0 min to 0.39% and 0.24%, respectively, at 100 min. Accordingly, total acidity was reduced by ED by about 32% in the permeate, and 30% in the CSJ after 50 min, and about 73%

Table 1. Changes in physicochemical parameters of CSJ by 500K UF

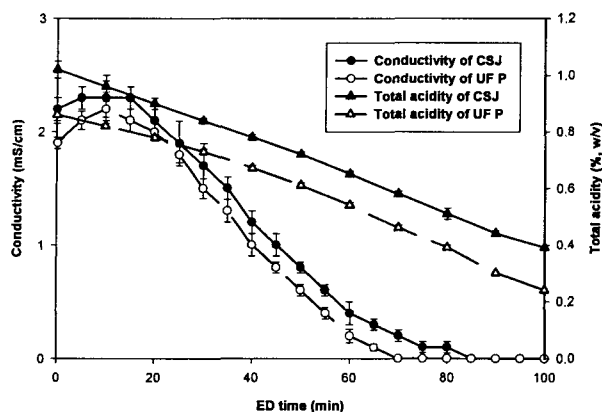| Sample	°Brix (A)	Total acidity, % (B)	(A)/(B)[1]	pH	Color values		
					L	a	b
CSJ	10.8±0.2	1.02±0.05	10.6	4.25±0.05	36.15±1.0	1.49±0.10	20.82±0.50
Retentate	11.0±0.2	0.98±0.03	11.2	4.22±0.06	43.54±1.3	6.75±0.05	26.32±0.20
Permeate	10.0±0.1	0.90±0.02	11.1	4.32±0.05	11.50±0.6	-3.00±0.20	0.43±0.02

[1]Ratio of sugar to acid.

Fig. 2. Changes of conductivities and total acidities of CSJ and UF permeates during ED.

and 62% after 100 min in the permeate and CSJ, respectively; the rate of decrease was almost the same for CSJ and permeate. However, the conductivities dropped sharply from 2.2 (permeate) and 2.3 (CSJ) mS/cm after 5 m to 0 mS/cm after 70 m for the permeate and 85 m for the CSJ. The pH values were also changed during ED, from 4.3 (CSJ) and 3.74 (permeate) at 0 min to 3.80 (CSJ) and 2.86 (permeate) after 100 min (Fig. 3). Brix were also decreased by about 8% (CSJ) and 15% (permeate) after 100 min of ED, respectively, as compared to the control (Fig. 3).

These first results indicated that some parameters needed to be changed to obtain a better balance of brix and acidity. Many of the mineral ions and organic acids in the juice were removed from the juice and transferred to the waste liquid because the membranes used had selectivity for organic acids. The removal of organic acid using anion exchange membranes caused a gradual decrease in the total acidity of the juice.

The total acidity values (0.6~0.7%, w/w) after 50 min of ED are equal to the minimum values to meet the standards for USDA Grade A single strength citrus juice (16). Therefore, 50 min of ED is adequate to deacidify the sam-
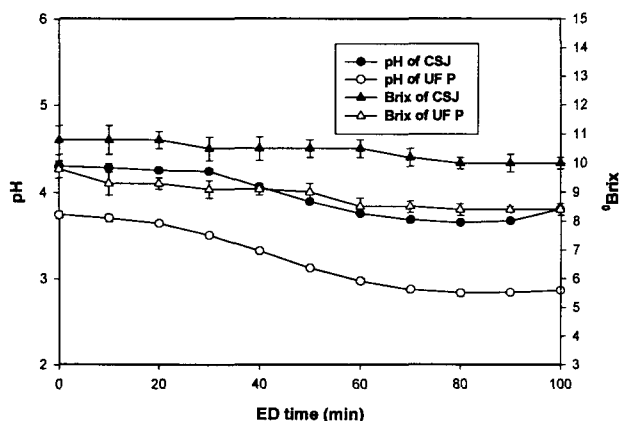
ples under the experiment conditions.

The changes in conductivity may have been a consequence of the removal of both anions and cations from the samples. However, the rapid decrease in conductivity, combined with the simultaneous slow decrease in acidity, suggests a rapid exclusion of cationic species with strong electro-conductivity, such as Na^+ and K^+ through the cation exchange membranes. In contrast to conductivity, the slow decrease in acidity resulted from a step-by-step exchange of acidic anionic molecules, such as citric acid ions, through anion exchange membranes. This appears to occur because the organic acid anions are slower in migration speed than inorganic cations such as Na^+ and K^+. The more rapid migration of Na^+ and K^+ has been observed by other investigators, who reported that the relative permeation rate of an anion of citric acid was 0.21~0.32 on anion exchange membranes and that of Na^+ was 1 and K^+ was 1.59 on cation exchange membranes (17). The rapid decrease in conductivity during ED will require higher a current density over the limiting current density (LCD), which is defined as the current density at the onset of ion concentration polarization; concentration polarization is caused by low electrolyte concentrations near the membrane surface as opposed to that in the fluid (2). If concentration polarization occurs during ED, conductivity and current efficiency are sharply decreased because of the splitting of water molecules. The splitting of water molecules also result in pH changes of the solution (17). As shown in Fig. 3, ED causes pH to decline after 30 min for CSJ and at 20 min for UF permeate. Therefore, the ED after these times was operated over LCD. In practice, the current efficiencies were calculated to be very low for times longer than 20 min of ED treatment for both samples (Fig. 4). The average current efficiencies at 20 min were 26.23% for CSJ and 28.33% for UF permeate. Therefore, it is necessary for the operating time of ED to be limited to <50 min, not only for maintaining optimal total acidity, but also for maintaining current efficiency under these experimental conditions. Dur-
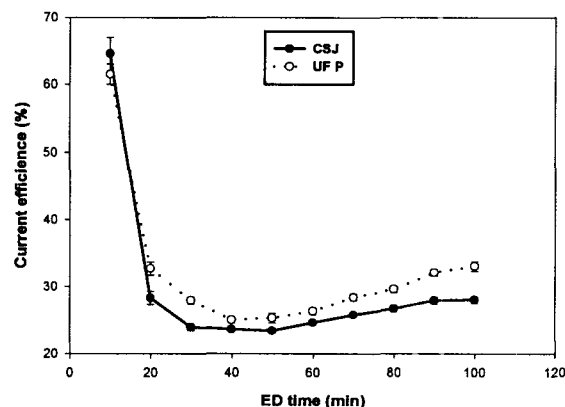

Fig. 3. Changes of pH and °Brix of CSJ and UF permeate during ED.

Fig. 4. Current efficiencies of CSJ and UF permeate during ED.

ng ED of milk samples, the conductivity decreased in a similar manner to the degree of demineralization, and the correlation was significant. As in this study, ED energy efficiency also decreased with demineralization (10). Further work is needed to find ways to increase current efficiency in the ED of citrus juice, since low current efficiency increases energy consumption. The problem might be solved by an exogenous addition of cations (8) or an adjustment of pH. According to one report (18) on the recovery of citric acid from a model solution by ED, the current efficiencies were greatly increased (0.09 ± 0.002 at pH 3, 0.41 ± 0.03 at pH 5, and 0.50 ± 0.01 at pH 7) as operating pH increased.

The results of this study have thus far shown that there is no significant difference between the CSJ and its UF permeate, which was expected. This means that the CSJ sample used in this study did not cause fouling problems for the relatively short operating time (100 min) and low number of batches. However, previous experience has demonstrated that fouling will be a problem for commercial operations. Therefore, this study has demonstrated that UF may reduce fouling problems and extend the life of membranes, and that the UF permeate can be processed more efficiently than CSJ if the membrane was operated continuously. Consequently, under the conditions of this study, both ED alone and the combination of ED and UF effectively reduced the total acidity and increased the ratio of sugar to acid of mandarin orange juice using only ion exchange membranes, without a strong alkali such as KOH or bipolar membranes. The ED operating time of 50 min appears to be suitable for optimizing both total acidity and

current efficiency under the experimental conditions.

Effects on major components of juices

Four juice samples were prepared for quality analysis depending on ED times (0, 50, and 100 min) and whether or not they were reconstituted. The samples included a control sample juice (CSJ) treated by ED alone and a reconstituted juice (RTJ) after treatment with combined UF and ED as shown in Tables 2 and 3. The ratio of sugar to acid increased as much as 2.5 times, from 10.59 to 25.64, depending on the ED time. After 50 min of ED, the ratio increased to as much as 27% (RTJ) ~ 35% (CSJ) of the control juice. The color of the juice was not changed by ED, but pH and Brix were both somewhat lower than those sugar to acid ratio of 16 ~ 17. Therefore, the juice deacidified by ED for 50 min was slightly high in total acidity and had a low Brix to acid ratio as compared to the ideal consumer orange juice. However, the lower quality of this product can be returned to a palatable level by blending with a higher quality juice.

There were no significant changes in the concentrations of amino-N and free sugars of the juice because the uncharged amino acids and sugars do not migrate during ED (Table 3). Only a major osmotic pressure gradient between two compartments (a pair of membranes) could force them to migrate through membranes (9). Of the three primary flavonoids in orange juice (representing >90% of the flavonoid content of mandarin orange juice) (13), naringin, and hesperidin were assayed and neohesperidin was not. The results show that ED has almost no affect on the flavonoid compounds, although there was a slight, but not sig-

Table 2. Changes in the physical parameters of CSJ and RTJ by ED alone or by UF and ED combination treatments

Sample	°Brix (A)	Total acidity (%) (B)	A/B[1]	pH	Color Values		
					L	a	b
CSJ	10.8 ± 0.2	1.02 ± 0.05	10.59	4.25 ± 0.05	36.2 ± 1.0	1.49 ± 0.10	20.8 ± 0.5
CSJ, ED50 only	10.5 ± 0.1	0.73 ± 0.03	14.38	3.88 ± 0.02	36.1 ± 0.5	1.06 ± 0.10	20.8 ± 0.7
CSJ, ED100 only	10.0 ± 0.1	0.39 ± 0.02	25.64	3.80 ± 0.03	35.4 ± 0.4	1.02 ± 0.15	20.2 ± 0.6
RTJ, after UF + ED50	10.5 ± 0.1	0.78 ± 0.01	13.46	4.04 ± 0.04	36.3 ± 0.8	1.29 ± 0.08	20.7 ± 0.4
RTJ, after UF + ED100	9.5 ± 0.1	0.57 ± 0.02	16.66	3.9 ± 0.02	36.0 ± 0.8	1.28 ± 0.08	20.6 ± 0.5

[1]Ratio of sugar to acid. ED50 and ED100 mean for 50 and 100 min of ED treatment.

Table 3. Changes in major components in CSJ and RTJ by ED alone or UF and ED combined treatment[1]

Samples	Amino-N (mg%)	Free sugar (%)			Organic acid		Flavonoid (mg%)	
		Fructose	Glucose	Sucrose	Ascorbic acid (mg%)	Citric acid (%)	Naringin	Hesperidin
CSJ	31.0 ± 1.4	1.79 ± 0.00	1.42 ± 0.01	5.59 ± 0.05	30.6 ± 1.6[a2]	1.41 ± 0.04[a]	2.01 ± 0.23	18.82 ± 0.33
CSJ, ED50 only	29.0 ± 1.4	1.65 ± 0.05	1.35 ± 0.02	5.14 ± 0.15	30.0 ± 0.5[a]	1.09 ± 0.01[b]	1.71 ± 0.06	16.78 ± 0.16
CSJ, ED100 only	31.0 ± 0.0	1.66 ± 0.01	1.32 ± 0.01	5.36 ± 0.06	27.2 ± 0.5[b]	0.92 ± 0.14[b]	1.59 ± 0.13	16.73 ± 0.16
RTJ, after UF + ED50	30.0 ± 1.4	1.69 ± 0.03	1.41 ± 0.05	5.36 ± 0.15	30.1 ± 4.1[a]	1.20 ± 0.01[c]	2.10 ± 0.40	18.66 ± 0.34
RTJ, after UF + ED100	29.0 ± 1.4	1.64 ± 0.12	1.33 ± 0.13	5.16 ± 0.28	30.9 ± 0.7[a]	0.90 ± 0.04[b]	1.62 ± 0.41	18.39 ± 0.40

[1]Means ± standard deviations.
[2]Any two means in the same column followed by the same letter are not significantly (p > 0.05) different by Student's t-test.

nificant, decrease in the naringin content ($p > 0.05$). The phenolic compounds in apple juice have also been shown to be preserved in their original state during ED, because they carry little or no charge, which makes migration during ED difficult (9). The only major changes, with significant differences ($p > 0.05$) occurred with ascorbic acid content after 100 min of ED and for citric acid level in all samples (Table 3). The decrease in the total acidity of juices after ED appears to be due to a reduction in citric acid content greater than 50%. The results of the ascorbic acid data suggests that shorter ED times are preferable for preserving nutritional quality, particularly when the ED process is used alone. The mineral content (such as K and Na) may have changed significantly, but they were not measured in this study.

In conclusion, ED combined UF may be effective, not only for preventing membrane fouling, but also for preserving ascorbic acid and mineral content, which are important nutrients in fruit juices.

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