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New electrolyte beverages prepared by the citrus canning processing water through chemical improvement

Sihuan Shen [a], Huan Cheng [a], Ying Liu [c], Yanpei Chen [a], Shiguo Chen [a], Donghong Liu [a], Xingqian Ye [a], Jianle Chen [a,b,*]

[a] College of Biosystems Engineering and Food Science, Ningbo Research Institute, National-Local Joint Engineering Laboratory of Intelligent Food Technology and Equipment, Zhejiang Key Laboratory for Agro-Food Processing, Zhejiang Engineering Laboratory of Food Technology and Equipment, Zhejiang University, Hangzhou 310058, China

[b] NingboTech University, Ningbo 315100, China

[c] Ecology and Health Institute, Hangzhou Vocational & Technical College, Hangzhou 310018, China

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ABSTRACT

In the production of canned citrus, large amounts of processing water were discharged during the segment membrane removal process, causing severe pollution. In order to reduce pollution and recover the bioactive compounds in the processing water, the production of canned satsuma mandarin, sweet orange and grapefruit were studied, and improved acid (0.1% HCl, 0.4% citric acid) and alkali (0.1% KOH, 0.2% NaOH) were used to conduct the new chemical hydrolysis process to remove the segment membrane. The obtained acid and alkali processing water were firstly explored the potential to make novel beverages, which contain electrolytes (Na: 472–945 ppm; K: 208–279 ppm; Cl: 364–411 ppm; citrate: 1105–1653 ppm) and potential prebiotics such as pectin and flavonoids. The improved segment membrane removal process realized the conversion of wastewater into drinkable beverages at low costs. The bioactive compounds were fully recovered without wastewater discharging, which produced great environmental, economic and health value.

Introduction

Citrus is the fruit with the largest cultivation area, highest yield and largest consumption in China. Canned citrus occupies a dominant position in the citrus processing industry. China's export volume of canned citrus is 265,800 tons, accounting for 52.07% of the total export volume of canned fruit (source: USDA). Undoubtedly, the segment membrane removal process is the most important step in the processing of canned citrus. There have been many new methods of removing the segment membrane, the traditional process of chemical hydrolysis with hydrochloric acid and NaOH is the most economical, practical, and suitable for large-scale use, but it consumes lots of water and the acid and alkali canning processing water were discharged (Izumi, et al., 2016).

It is known that the citrus acid and alkali canning processing water are rich in pectin and flavonoids, which result in high chemical oxygen demand (COD) (Chen, et al., 2017). Pectin and flavonoids are now considered as potential prebiotics (Gibson, et al., 2017; Mao, et al., 2019; Ndeh, et al., 2017). Pectin can exert its physiological activities such as immune regulation (Klaenhammer et al., 2012), inhibition of inflammation (Jin, et al., 2021) and anti-cancer (Cui, et al., 2019) through selective fermentation of intestinal microbes. Citrus flavonoids have varieties of biological activities such as antioxidation (Singh et al., 2020), anti-inflammatory (Bell & Wagstaff, 2019; Muhammad, Ikram, Ullah, Rehman, & Kim, 2019), the treatment of metabolic syndrome (Zeng, et al., 2020) and neuroprotection (Hwang et al., 2012).

A recovery line for pectin and flavonoids significantly reduced COD to facilitate the discharge of citrus canning processing water (Yan, et al., 2018). However, it only aimed at the extraction of a single component (pectin or flavonoids), among which the recovered pectin did not meet the requirements of commercial pectin and the bioavailability of dried pectin is poor. Furthermore, the organic reagents used for extraction are highly polluted, and the organic wastewater needs to be further treated. The recovery of pectin and flavonoids from the processing water is high in cost, complicated in process, large in loss and long in treatment time. We try to establish a simpler and faster process to recover bioactive compounds at low costs while achieving zero discharge of the canning processing water.

The citrus acid and alkali canning processing water are theoretically

* Corresponding author at: College of Biosystems Engineering and Food Science, Zhejiang University, Yuhangtang Road 866#, Hangzhou 310058, China.
E-mail address: chenjianle@zju.edu.cn (J. Chen).

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edible after neutralization, but large amounts of NaCl are produced after that, and the taste is too salty to drink. It is hypothesized that if acid such as citric acid and alkali such as KOH can be introduced into the acid and alkali treatments, the segment membrane can be fully removed, the improved acid and alkali processing water can be mixed without salty taste and rich in electrolytes, which has the potential to be prepared into electrolyte beverages. There have been many studies using citric acid and KOH to extract pectin from the skins and pomaces of various fruits (He, Sampers, & Raes, 2021; Mierczyńska, Cybulska, & Zdunek, 2017; Yang, Mu, & Ma, 2018). The principle of segment membrane removal is to break the adhesion of pectin, cellulose and hemicellulose so that the pectin can be dissolved and the structure of the segment membrane is destroyed. This is consistent with the principle of pectin extraction from the cell wall. Therefore, it is technically feasible to remove the segment membrane with citric acid and KOH and to use the acid and alkali processing water as raw materials for electrolyte beverages.

Electrolyte beverages that have significant health benefits are made by dissolving a series of compounds in water. They can not only prevent and relieve cramps (Earp, et al., 2019), but also promote the human body to fully absorb glycogen and improve the activity of human muscles (Rodriguez-Giustiniani et al., 2019). In addition, electrolyte beverages can quickly replenish the body's water consumption, promote metabolism and help eliminate harmful wastes such as alcohol and ammonia to improve body fluid balance (Johannsen, et al., 2014). But the existing electrolyte beverages only contain some electrolytes, sugars and vitamins, usually lack of dietary fiber or functional phytochemicals. If citrus acid and alkali canning processing water are used to make electrolyte beverages, bioactive compounds can be included, which have more comprehensive health benefits to the human body.

In this study, in order to solve the problems of wastewater discharge and the recovery of bioactive compounds, the segment membrane removal process in the processing of canned citrus was improved. While ensuring the success of segment membrane removal, the produced acid and alkaline water were made into electrolyte beverages after seasoning. To reduce the water consumption and enrich the beneficial ingredients such as pectin and flavonoids, the processing water was reused for new batches of segment membrane removal. The reused acid and alkali processing water and electrolyte beverages of three fruits were characterized. This study turns the wastewater into healthy food, which conforms to the concept of green development and produces great economic and healthy value.

Materials and methods

Obtaining citrus segments

Satsuma mandarin (*Citrus unshiu* Marc. Owari satsuma), sweet orange (*Citrus sinensis* (L.) Osbeck) and grapefruit (*Citrus. paradisi* Macf. Changshanhuyou) came from Citrus Research Institute of Zhejiang Province. The three fruits were blanched in the boiling water for 5 s, and then they were peeled and divided while hot. Finally, the tangerine pith was removed to obtain segments for the segment membrane removal process (Fig. 1).

The improved segment membrane removal process

From the perspective of resource conservation, the ratio of acid/alkaline water to citrus segments was 1:1 (w/w). The acid concentration, alkali concentration and temperature were selected according to the standard requirements of electrolyte beverage and the requirement of pH less than 4.6 after mixing the acid and alkali water to ensure that the beverages will not deteriorate according to GB/T 4789.26–2003.

Acid treatment

In a launder of the citrus canning factory, the segments obtained from *2.1* were immersed in the original acid water (0.4% citric acid, 0.1% HCl) for a certain time (40 min for satsuma mandarin, 50 min for sweet orange and grapefruit) at 30 °C. These conditions were selected based on the results of the previous experiment (Table S1-S7). After that, the acid processing water was collected and used as raw material for beverages. The citrus segments were rinsed thoroughly for the next step of alkali treatment (Fig. 1).

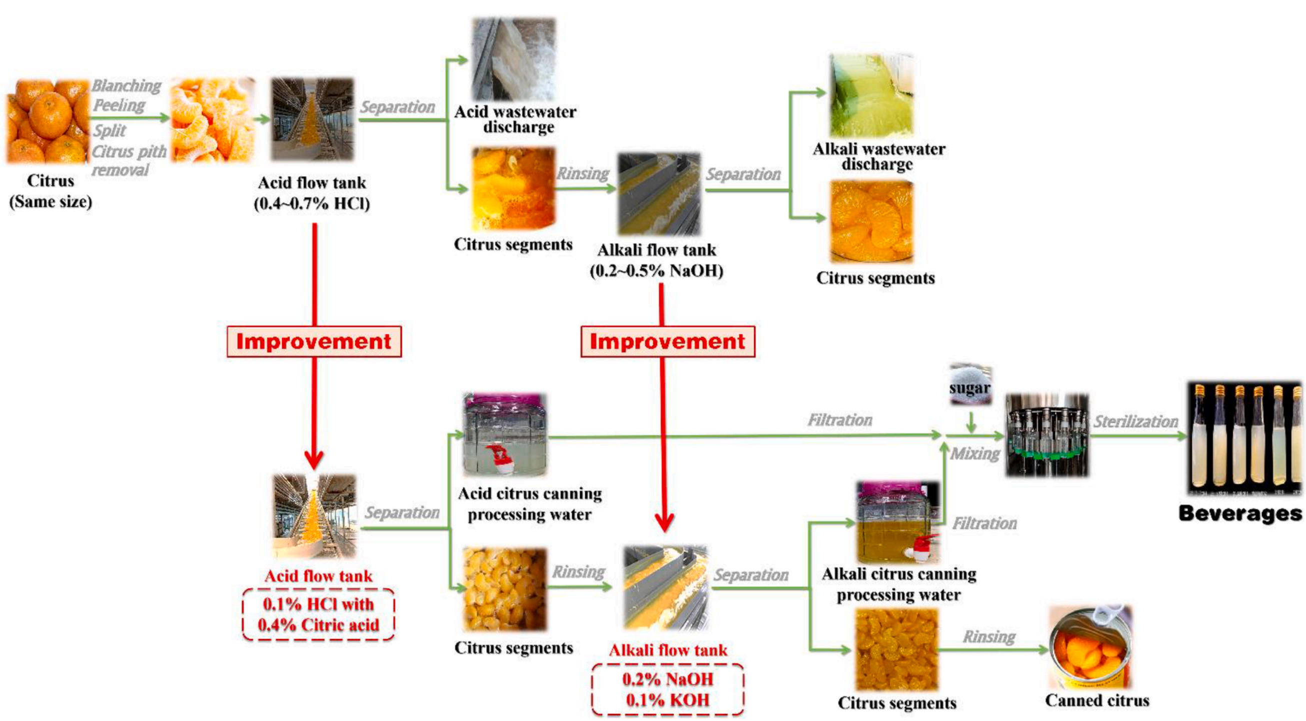

Fig. 1. The process of segment membrane removal and beverage preparation (taking the first batch of segment membrane removal of satsuma mandarin as an example).

Alkali treatment

The segments obtained from *2.2.1* were immersed in the original alkali water (0.2% NaOH, 0.1% KOH) in another launder. All three kinds of fruits were treated for 10 min at 30 °C. The alkali processing water was collected for determination and used as raw material for beverages, too. The citrus segments were rinsed thoroughly again to prepare for canning (Fig. 1).

Recycling of canning processing water collected after acid and alkali treatment

A total of 3 batches of citrus segments were used for the improved segment membrane removal process. In each batch, mixed acid and mixed alkali were added to the collected acid and alkali processing water to make them reach the same titratable acidity and alkalinity as the original acid and alkali water. The replenished acid and alkali processing water were used to remove the segment membrane of a new batch of citrus segments, and they were recycled two times.

Determination of physical and chemical properties of citrus canning processing water

pH

Three batches of acid and alkali processing water were produced by three fruits, a total of 18 samples were uniformly sampled after homogenization. All subsequent assays were sampled as such. The pH of all liquids was measured by a pH meter (METTLER TOLEDO, USA).

Titratable acidity

Refer to AOAC Official Method 942.15, the titratable acidity of all acid processing water was determined and calculated as follows: Titratable acidity=(C × V)/V0 × 100 × 0.07, where C means the concentration of sodium hydroxide standard solution, M; V means the volume of NaOH standard solution consumed during titration, mL; V0 means the volume of samples, mL; 0.07 is the coefficient of conversion to citric acid monohydrate (for citrus fruits).

Titratable alkalinity

All the alkali processing water was titrated with the standard solution of hydrochloric acid (Kirby and Cravotta, 2005). The titratable alkalinity was calculated as follows: Titratable alkalinity=(C × V1)/10 × 43 × 100, where C means the concentration of the hydrochloric acid standard solution, M; V1 means the volume of hydrochloric acid standard solution consumed during titration, mL; 43 means the average molar mass of NaOH and KOH after mixing in 2:1 (w/w).

Pectin content

The content of pectin in the processing water was determined by weighing after precipitation with ethanol (Chen, et al., 2017). The content of pectin was calculated by the formula: Y = W/V, where Y means yield, g/L; W means the weight of dry pectin powder, g; V means the volume of acid or alkaline processing water, L.

Total sugar content

The phenol–sulfuric acid method was used to determine the content of total sugar (DuBois et al., 1956). Under the action of sulfuric acid, the polysaccharide was hydrolyzed into monosaccharides and dehydrated to form aldehyde derivatives, which reacted with phenol to form orange-yellow substances. The absorbance value at 490 nm was determined to calculate the total sugar content of the processing water.

Reducing sugar content

The content of reducing sugar was determined by the 3,5-dinitrosalicylic acid (DNS) method (Hu, et al., 2008). DNS reacted with the reducing sugar to produce 3-amino-5-nitrosalicylic acid which turned to brownish-red at 100 °C. The reducing sugar content was calculated by determining the absorbance value at 540 nm.

Total flavonoid content

The modified Davis method was used to determine the content of total flavonoids in satsuma mandarin and sweet orange processing water with hesperidin as the standard and the wavelength at 360 nm. The content of total flavonoids in grapefruit processing water was determined with naringin as the standard and the wavelength at 420 nm (Davis, 1947; Kim, et al., 2016).

Total solid and soluble solid content

Refer to AOAC Official Method 941.08, the drying method was used to determine the total solids content of the processing water. After filtration, the content of total soluble solids (TSS) was measured with a digital refractometer (ATAGO, Japan).

Viscosity

Refer to AOAC Official Method 967.16, the viscosity of the processing water was determined by the Lamb–Lewis capillary viscometer at 24 °C. This viscometer is used as an internal quality standard by the fruit and vegetable juice industry (K. A. Abbas et al., 2010). The time to pass a specified distance in the viscometer was measured. The viscosity constant is 0.1819 mm^2/s^2. The viscosity was calculated as follows: $\eta = c \times t$, where η means viscosity, mm^2/s; c means viscosity constant, mm^2/s^2; t means time, second.

Electrolyte content

After digestion, the assay of Na and K levels was performed by atomic absorption spectrophotometer 240FS AA (Agilent Technologies, USA) (Szymczycha-Madeja, Welna, Jedryczko, & Pohl, 2014; Villagrán, Deetlefs, Pitner, & Hardacre, 2004). After flame atomization, K and Na absorbed the resonance lines of 766 nm and 589 nm respectively.

The level of chloride and citrate was determined by DIONEX AQUION (Thermo Fisher Scientific, America) which is equipped with the AS11-HC guard column (4 × 50 mm, 4 μm) and AS11-HC analytical column (4 × 250 mm, 4 μm). 25 μL sample was injected and the suppression current was 50 mA. 20 mM KOH solution was used as the eluent at a flow rate of 1 mL/minute for 13 min (Penniston et al., 2008).

Preparation of beverages

The acid and alkaline processing water were filtered through the 300-mesh filter cloth to remove impurities and slowly mixed in a certain proportion to ensure that the pH of the mixture was less than 4.6 so that the processing water was maximally utilized and the bacteria were inhibited. After determining the titratable acidity of the mixture, sucrose and citrus essence were added for seasoning. Finally, the beverages were filled and sterilized at 90 °C for 15 min (Fig. 1).

Determination of physical and chemical properties of electrolyte beverages

The determination methods of total solids, soluble solids, pectin, total flavonoids and electrolytes content of the beverages were consistent with those of canning processing water.

Composition and content determination of flavonoids in beverages

The remaining liquid was collected after the pectin was extracted from the processing water. It was concentrated by rotary evaporation at 45 °C and then freeze-dried to obtain crude flavonoids. 12 mL 80% methanol and DMSO (V/V = 1:1) were added to 0.5 g crude flavonoids (Zhang, et al., 2014). After 12 h of mechanical shaking extraction, the extract was centrifuged at 5000 g for 15 min. The residue was extracted with shaking for another 12 h and centrifuged again. Finally, the supernatant was combined and diluted to 25 mL with methanol to determine the composition and content of flavonoids.

21 kinds of standard flavonoids (Flavanones: eriocitrin, narirutin, naringin, hesperidin, neohesperidin, diosmin, eriodictyol, didymin,

poncirin, naringenin, hesperetin; Flavones and polymethoxylated flavones: rhoifolin, luteolin, apigenin, diosmetin, sinensetin, nobiletin, tangeretin; Flavonol: quercitrin, quercetin, kaempferol) were dissolved in 80% methanol and DMSO (V/V = 1:1) and diluted to 0.2, 0.1, 0.05, 0.025, 0.005 and 0.0025 mg/mL.

The Waters e2695 HPLC system (Waters, USA) which is equipped with a 2998 PDA detector (Waters, USA) was used to determine the content of 21 kinds of flavonoids in beverages. The column was ZORABX SB-C18 (4.6 mm × 250 mm, 5 μm, Agilent, USA). The mobile phase was: 0.1% formic acid (A) and methanol (B). Gradient elution was employed at the flow rate of 0.7 mL/minute: 0–20 min, 37%-50% B; 20–35 min, 50%-80% B; 35–40 min, 80%-100% B; 40–50 min, 100% B; 50–60 min, 100–37% B; 60–70 min, 37% B. The column temperature was 25 °C and the injection volume was 10 μL (Zhang, et al., 2014). The detection wavelength was 283 nm for flavanones, 330 nm for flavones and polymethoxy flavones and 367 nm for flavonols.

Descriptive sensory evaluation

In the sensory evaluation laboratory, 10 well-trained panelists aged between 20 and 25 (5 women and 5 men, all of whom have studied food sensory courses and are sensory sensitive) briefly described the beverages and agreed on terms and definitions for their properties. According to the properties of beverages, the standard colorimetric card was used as the reference for the color. Fresh and spoiled citrus juice serves as reference products for appearance and smell. 1–8% sucrose solution, 0.1–0.8% citric acid solution and 0.01–0.08% quinine hydrochloride solution were used as reference products for sweet, sour and bitter taste, respectively. Clear citrus juice and turbid citrus juices with different dilution levels were used as reference products for impurities. Finally, the detailed sensory evaluation intensities (Table S8) were formed, with a total score of 100 points, 20 points for each item, and 4 levels. Panelist proficiency was checked at the end of the training to ensure the consensus, repeatability and discernibility of each panelist (Park et al., 2019).

Within a month, the same 10 panelists evaluated the appearance, color, smell, taste and impurities of these beverages three times. In order to improve credibility, the panelists randomly sampled and evaluated them one by one, and rinsed mouths with water between samples. The final results were obtained by averaging the evaluation results of each judge for each item. The evaluation results of each judge on the appearance, color, smell, taste and impurities of the same beverage were added up and then averaged to obtain the overall descriptive evaluation of each beverage.

Table 1
The improved acid and alkali treatment process of three fruits.

	Processing water	Titratable acid (%)	pH	Acid supplement	pH after supplement	Titratable acid after supplement (%)
Satsumamandarin	Original Acid water	0.58 ± 0.02	1.82 ± 0.14	/	/	/
	MA1	0.56 ± 0.04	1.98 ± 0.27	/	/	/
	MA2	0.54 ± 0.03	2.28 ± 0.28	/	/	/
	MA3	0.43 ± 0.06	2.63 ± 0.49	/	/	/
Sweetorange	Original Acid water	0.58 ± 0.02	1.86 ± 0.13	/	/	/
	OA1	0.53 ± 0.03	1.98 ± 0.25	/	/	/
	OA2	0.50 ± 0.03	2.07 ± 0.26	0.064% Citric acid + 0.016% HCl	1.86 ± 0.07	0.58 ± 0.01
	OA3	0.54 ± 0.02	1.96 ± 0.14	/	/	/
Grapefruit	Original Acid water	0.58 ± 0.02	1.82 ± 0.15	/	/	/
	CA1	0.49 ± 0.02	1.98 ± 0.16	0.072% Citric acid + 0.018% HCl	1.50 ± 0.09	0.64 ± 0.01
	CA2	0.53 ± 0.04	1.89 ± 0.37	/	/	/
	CA3	0.42 ± 0.03	2.19 ± 0.27	/	/	/
	Processing water	Alkalinity(%)	pH	Alkali supplement	pH after supplement	Alkalinity after supplement (%)
Satsuma mandarin	Original basic water	0.29 ± 0.02	13.30 ± 0.11	/	/	
	MB1	0.11 ± 0.02	12.87 ± 0.14	0.063% KOH+0.127% NaOH	12.95 ± 0.12	0.31 ± 0.01
	MB2	0.15 ± 0.03	13.01 ± 0.23	0.05% KOH+0.1% NaOH	12.88 ± 0.15	0.31 ± 0.01
	MB3	0.08 ± 0.01	10.98 ± 0.08	/	/	
Sweetorange	Original basic water	0.26 ± 0.01	13.16 ± 0.08	/	/	
	OB1	0.18 ± 0.02	12.76 ± 0.17	0.027% KOH+0.053%NaOH	13.03 ± 0.16	0.23 ± 0.02
	OB2	0.09 ± 0.01	11.93 ± 0.09	0.056% KOH+0.113%NaOH	13.15 ± 0.15	0.26 ± 0.02
	OB3	0.12 ± 0.03	12.15 ± 0.25			
Grapefruit	Original basic water	0.29 ± 0.01	13.29 ± 0.08	/	/	
	CB1	0.13 ± 0.03	12.67 ± 0.27	0.053% KOH+0.107% NaOH	13.33 ± 0.12	0.31 ± 0.01
	CB2	0.08 ± 0.06	12.32 ± 0.34	0.07% KOH+0.14% NaOH	13.28 ± 0.10	0.31 ± 0.01
	CB3	0.06 ± 0.02	11.89 ± 0.11	/	/	

MA1/MB1: The first batch of satsuma mandarin acid/basic processing water; MA2/MB2: The second batch of satsuma mandarin acid/basic processing water; MA3/MB3: The third batch of satsuma mandarin acid/basic processing water; OA1/OB1: The first batch of sweet orange acid/basic processing water; OA2/OB2: The second batch of sweet orange acid/basic processing water; OA3/OB3: The third batch of sweet orange acid/basic processing water; CA1/CB1: The first batch of grapefruit acid/basic processing water; CA2/CB2: The second batch of grapefruit acid/basic processing water; CA3/CB3: The third batch of grapefruit acid/basic processing water.

Statistical analysis

All experiments were repeated 3 times with 3 parallel samples (except for sensory evaluation), and 9 data were obtained. Sensory evaluation was carried out 3 times, with 10 judges each time, and a total of 30 data were obtained. The data were reported as mean ± standard deviation. One-way ANOVA followed by Duncan's multiple-range test was conducted by SPSS 26.0 to evaluate the significance of differences between the data. *P* less than 0.05 was considered significant.

Results and discussion

Technical feasibility analysis of the improved process for segment membrane removing

It could be seen from Table 1 that the titratable acidity of satsuma mandarin acid processing water did not change much, so no acid was supplemented during the circulation. The titratable acidity of sweet orange and grapefruit acid processing water changed a lot, so additional acid was needed to be added to reach the initial titratable acidity (0.58%) (Table 1). After the improved acid treatment, all three batches of segment membrane were partially dissolved, resulting in white and turbid acid processing water (Fig. 1)

Obviously, the recycling of alkali water led to a significant decrease in titratable alkalinity. Therefore, alkali was supplemented during the recycling to increase the alkalinity to the initial level. After ten minutes of alkali treatment, yellow alkali processing water was produced (Fig. 1). The segment membrane of all three fruits in three batches was completely removed, which showed the feasibility of removing them with low temperature, organic acid and mixed alkali (Fig. 2A).

Analysis of acid and alkali canning processing water produced by three fruits in three cycles

In order to further clarify the new segment membrane removal process and judge whether the acid and alkali processing water can be used to prepare electrolyte beverages, the contents of pectin, total sugar, reducing sugar, total flavonoids, total solids, total soluble solids, Na, K, chloride and citrate and the viscosity of MA1-3, MB1-3, OA1-3, OB1-3, CA1-3 and CB1-3 were determined.

Clarification of the improved segment membrane removal process

Pectin is an important component in the cell wall of the segment membrane. Due to the use of organic acid (pH = 1.82–1.86) and low temperature (30 °C), the damage to the combined and entangled structure of cellulose, hemicellulose and pectin is slight so that little pectin can be released and the content of pectin is lower than that in previous studies when using 0.4% HCl (pH = 1) at 28 °C (Yan, et al., 2018). The content of pectin in acid processing water is much lower than that in alkaline water, suggesting that pectin is mainly dissolved in alkali treatment (Fig. 2B). Because the acidic solution has already processed the cell wall preliminarily, the alkali solution can better extract the remaining polysaccharides from the cell wall of the segment membrane (Zhang, et al., 2018). Among the three fruits, the pectin content in the sweet orange processing water is the highest, and there is little difference between satsuma mandarin and grapefruit processing water, which

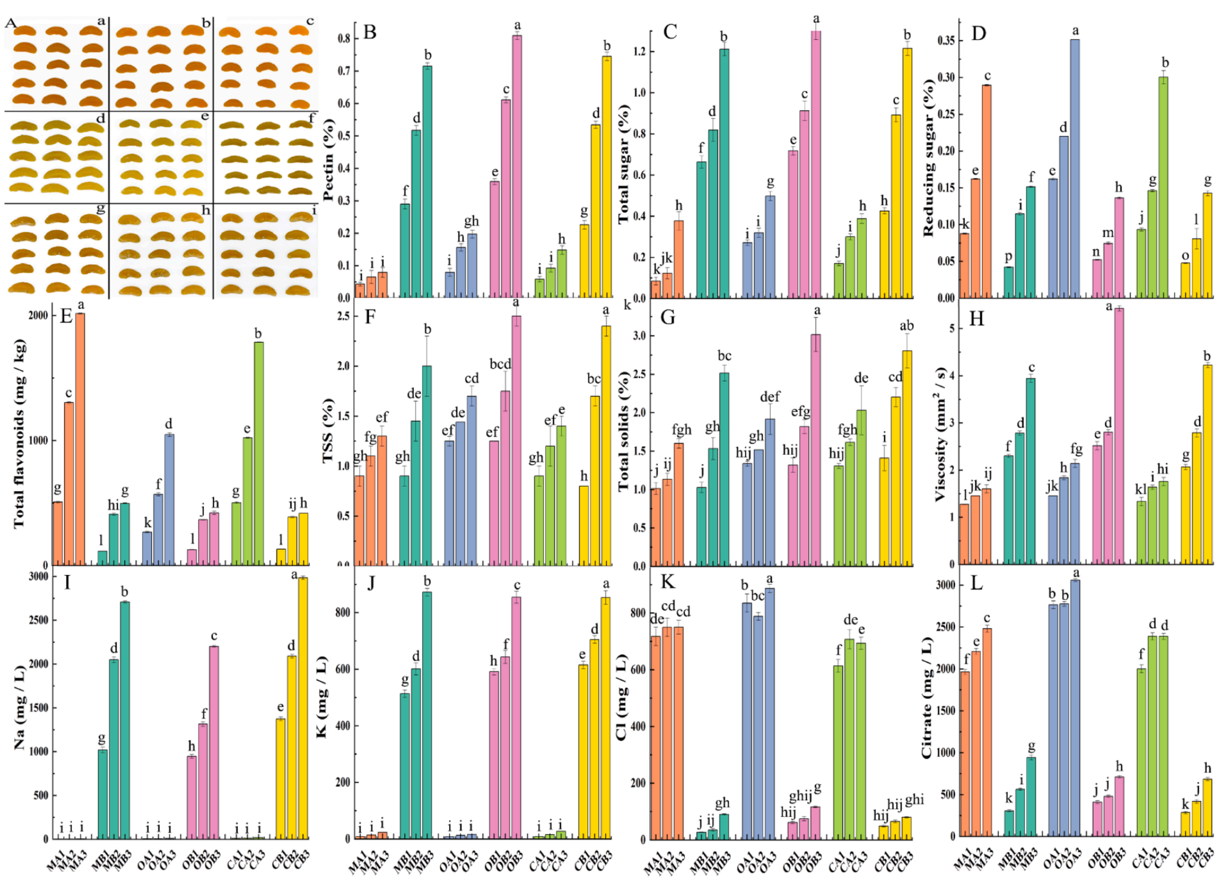

Fig. 2. (A) The citrus segments after the improved segment membrane removal process: (a-*b*-c) The first, second and third batches of mandarin segments; (d-e-f) The first, second and third batches of orange segments; (g-h-i) The first, second and third batches of grapefruit segments. Comprehensive properties of three batches of acid and alkali processing water produced by three fruits are exhibited including the levels of pectin (B), total sugar (C), reducing sugar (D), total flavonoids (E), total soluble solids (F), total solids (G), viscosity (H), Na (I), K (J), Cl (K) and citrate (L). Different lowercase letters indicate significant differences ($P < 0.05$) between different processing water.

might be caused by the difference in fruit varieties (Liu, et al., 2001).

The content of total sugar in alkaline water is significantly higher than that in acid water (Fig. 2C), because the segment membrane is mainly removed during the alkali treatment and the content of pectin is higher. However, the content of reducing sugar in acid water is significantly higher than that in alkali water (Fig. 2D). Therefore, it can be inferred that in the first step of acid treatment, due to mild conditions, the segment membrane can only be partially softened and dissolved, and most of the small molecules of free sugars are dissolved. After that, the alkali treatment dissolved most of the large molecules of polysaccharides, such as pectin.

As another important bioactive compound in citrus segment membrane, the content of flavonoids in acid water is much higher than that in alkaline water (Fig. 2E). This is an interesting phenomenon because flavonoids are generally soluble in alkali water but insoluble in acid water. It is speculated that after the first step of acid treatment, the cell wall is destroyed and most of the flavonoids enter the acid water (in an insoluble state). Then, the remaining flavonoids are dissolved in the second step of alkali treatment. Among the acid processing water of three fruits, the content of flavonoids in the satsuma mandarin acid processing water is the highest, and that of sweet orange acid processing water is the lowest; among the alkali processing water, the content of flavonoids in satsuma mandarin alkali processing water is slightly higher, sweet orange and grapefruit had no significant difference (Fig. 2E).

Therefore, the acid treatment process initially destroys the structure of the segment membrane, resulting in the release of large amounts of free sugars and flavonoids (in an insoluble state), but little of the pectin can be dissolved due to the mild conditions. Because of the first step of acid treatment, most of the pectin in the segment membrane can be better dissolved and the remaining small part of flavonoids are dissolved during the subsequent alkali treatment.

Evaluation on the feasibility of acid and alkali processing water to electrolyte beverages

The total solids and soluble solids contents in the acid and alkali processing water are higher than the added acid (0.1% HCl, 0.4% citric acid) and alkali (0.2% NaOH, 0.1% KOH) solids contents (Fig. 2F, 2G), indicating that the insoluble fiber and functional components in the segment membrane are released during the sequential acidic and alkaline steeping process. In addition, the solids in MA1, MA2, MB1, MB2, OA1, OA2, OB1 and OB2 are basically soluble solids (Fig. 2F, 2G), which proves that the segment membrane has been fully dissolved and there is little residue in the processing water. The insoluble solids content in MA3, MB3, CA3 and CB3 increases, which proves that although the third batch of segment membrane removal can remove the segment membrane from the surface of the segments, the membrane cannot be completely dissolved in the processing water. In the grapefruit acid and alkali processing water, the total solids content is always higher than the soluble solids content. The segment membrane of grapefruit cannot be fully hydrolyzed in the processing water, resulting in the insufficient release of beneficial ingredients.

The viscosity of alkali processing water is significantly higher than that of acid processing water, the viscosity of sweet orange processing water is the highest, and there is no significant difference between the viscosity of satsuma mandarin and grapefruit processing water (Fig. 2H). This corresponds to the pectin content in the processing water (Fig. 2B), the higher the content of pectin, the greater viscosity of the processing water. Meanwhile, the viscosity of the processing water increases with the number of cycles (Fig. 2H). The third batch of processing water of the three fruits was too viscous to flow and formed gel after mixing, it was not suitable to be directly used as materials for preparing beverages.

As essential ingredients in electrolyte beverages, the contents of Na and K in alkaline processing water are much higher than that in acid water (Fig. 2I, 2 J), because Na and K mainly come from the 0.2% NaOH and 0.1% KOH added in the alkali treatment process. The Na in acid water mainly comes from fruits themselves, which contain small amounts of Na (Szymczycha-Madeja & Welna, 2013). However, the content of K in the acid water is not as low as the content of Na (Fig. 2J), which should be due to the higher content of K in the three fruits themselves (Ani and Abel, 2018). Because in the alkali treatment of the three fruits, alkali was replenished in every batch (Table 1), so the contents of Na and K in the alkali processing water increase with the increase of the number of cycles.

The chloride and citrate in processing water are other essential ingredients of electrolyte beverages, basically come from the hydrochloric acid and citric acid which are added in the acid treatment, and the contents of chloride and citrate in acid processing water are much higher than that in alkaline processing water (Fig. 2K, 2L), which come from the fruits themselves and the residual chloride and citric acid after acid treatment. And the replenishments of acid between OA2-OA3 and CA1-CA2 (Table 1) show a significant increase in chloride and citrate content (Fig. 2K, 2L).

In conclusion, the electrolytes and bioactive compounds are abundant in the acid and alkali processing water of three fruits, but the third batch of processing water cannot be made into beverages directly because of its high viscosity. Compared with the first and second batches of grapefruit processing water, the first and second batches of acid and alkali processing water of satsuma mandarin and sweet orange are more suitable as raw materials for electrolyte beverage preparation because the ingredients of the segment membrane are fully released in the processing water, and the flavonoids content in satsuma mandarin processing water is the highest, the pectin content in sweet orange processing water is the highest.

The recipes of new electrolyte beverages

Only the first and second batches of acid and alkali canning processing water were used for beverage preparation. It was found that the pH value was less than 4.6 when the acid and alkali water were mixed at the ratio of 1:1 (w/w), the beverages were not easy to deteriorate and the full utilization of acid and alkaline water was realized. According to the sugar: acid ratio of 40, sucrose was added for better taste and calories. And 0.02% essence was added for aroma giving (Table S9).

Analysis of the physical and chemical properties of beverages

The contents of Na, K, chloride and citrate in beverages are basically equal to the sum of half of the contents in the corresponding acid and alkali processing water (Fig. 3A, 3B, Fig. 2I-2L). The contents of Na and K in beverages made from the second batch of processing water are significantly higher than those made from the first batch of processing water. The contents of Na in BM2 and BC2 are significantly higher than in other beverages, and the content of Na in BO1 is the lowest (Fig. 3A). All the beverages (Na: 471.83–945.07 ppm) meet the requirement for Na content (50–1200 ppm) in the electrolyte beverage standard. The content of K in BC2 (278.95 ppm) is significantly higher than that of other beverages, which does not meet the requirement for K content (50–250 ppm) in the electrolyte beverage standard.

There is no significant difference in the chloride content of all beverages (Fig. 3B). The citrate contents in BO1 and BO2 are higher than others (Fig. 3B), because the citrate content in sweet orange processing water is higher (Fig. 2L). It also can be seen from Fig. 3B that the citrate content in BM1 and BM2 has no significant difference, and the citrate content of BC2 is significantly higher than that of BC1 because there is no acid replenishment between MA1 and MA2, but the acid replenishment was carried out between CA1 and CA2 (Table 1). Because of their high electrolyte contents and reasonable proportions, the beverages can maintain the body's electrolyte balance after oral administration, quickly relieve fatigue (von Duvillard et al., 2004), help the body fully absorb the sucrose in beverages and improve the mobility of muscles (Chryssanthopoulos, et al., 2002). After absorbing the sucrose and

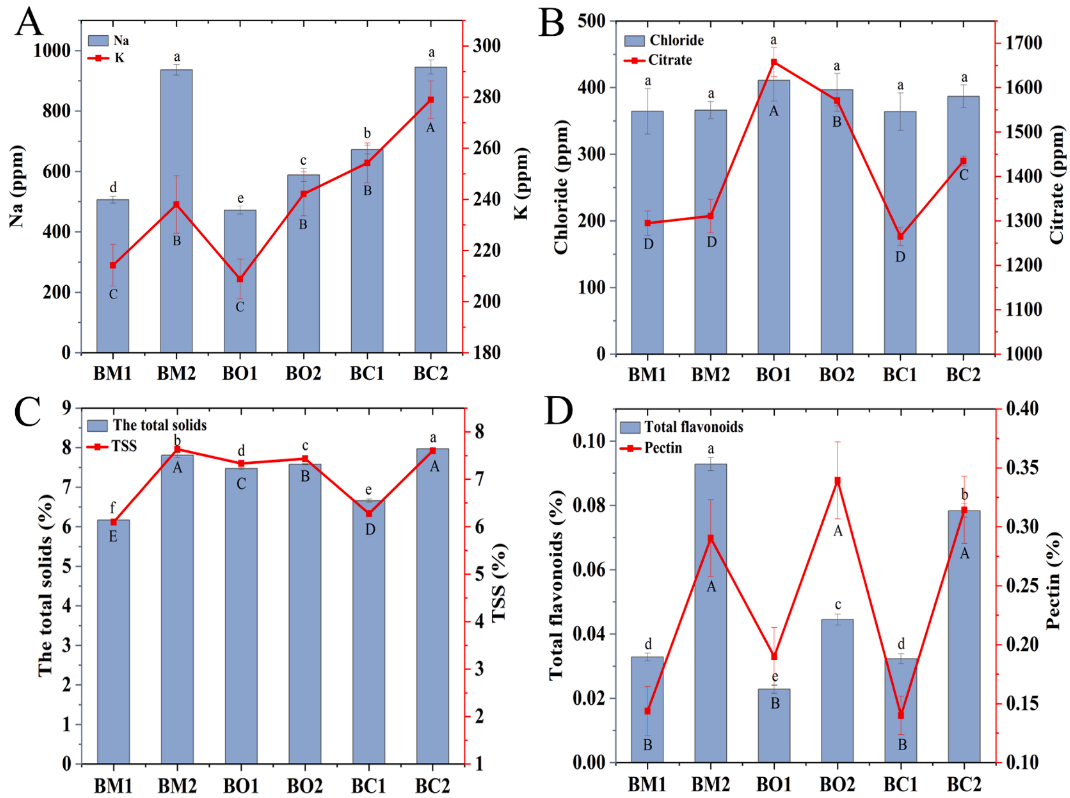

Fig. 3. The contents of important bioactive compounds and electrolytes in beverages. (A) The contents of Na and K. (B) The contents of chloride and citrate. (C) The contents of total solids and soluble solids. (D) The contents of total flavonoids and pectin. BM1/2: beverages made from the first/second batch of satsuma mandarin acid and alkali processing water; BO1/2: beverages made from the first/second batch of sweet orange acid and alkali processing water; BC1/2: beverages made from the first/second batch of grapefruit acid and alkali processing water. Different lowercase letters indicate significant differences ($P < 0.05$) between the bar groups; Different capital letters indicate significant differences ($P < 0.05$) between the line groups.

citrate in beverages, they can be quickly converted into glycogen and stored in the liver or muscle to reduce the formation of cholesterol (Williams & Rollo, 2015).

It can be seen from Fig. 3C that the total solids and soluble solids contents of the six beverages are close, and there are few insoluble solids in beverages, among which the insoluble solids content in BC1 and BC2 are relatively higher. The contents of TSS in all beverages (6.10–7.63%) meet the requirement for TSS content in the electrolyte beverage standard (3–8%).

As ingredients that are not present in ordinary electrolyte beverages, there is no significant difference in pectin content among BM1, BO1, BC1, and among BM2, BO2, BC2. The pectin contents in beverages made from the second batch of processing water are significantly higher than that made from the first batch of processing water (Fig. 3D). Beverages made from the second batch of processing water had significantly higher levels of flavonoids than beverages made from the first batch of processing water. Among the beverages made from the first batch of processing water, the total flavonoids content of BO1 is the lowest, and there is no significant difference between BM1 and BC1; among the beverages made from the second batch of processing water, the total flavonoids content: BM2 > BC2 > BO2. Therefore, the content of total flavonoids is significantly highest in beverages made from satsuma mandarin acid and alkaline processing water, but lowest in beverages made from sweet orange processing water (Fig. 3D). The flavonoids in beverages mainly come from the segment membrane, and the content of flavonoids in the satsuma mandarin segment membrane is indeed higher than that in the orange segment membrane (Abeysinghe et al., 2007).

Therefore, satsuma mandarin processing water is most suitable for making electrolyte beverages. The beverages made from it meet the standard requirements for electrolyte beverage, with high and balanced electrolyte contents; the pectin content is not significantly different from the beverages made from the other two kinds of fruit processing water, and all are high; the total flavonoid content is the highest. The grapefruit acid and alkali processing water may not be suitable for making electrolyte beverages. And, due to its higher contents of electrolytes and bioactive compounds, the second batch of processing water is more suitable for beverage production than the first batch of processing water. The new electrolyte beverages not only have the main components of ordinary electrolyte beverages but also increase the bioactive compounds (pectin and flavonoids) contents in beverages at a low cost, providing more comprehensive protection for the human body.

Composition and content analysis of flavonoids in beverages

The HPLC chromatograms of 6 beverages (Figure. S1-S3) were compared with those of 21 standard flavonoids (Figure. S4), and the contents of various flavonoids in the beverage were calculated according to the corresponding standard curve equations (Table S10).

Obviously, the total flavonoids content in beverages made from the second batch of processing water is higher than that made from the first batch. The beverage with the highest total flavonoids content is BM2, while the lowest is BO1 (Fig. 4A), which is consistent with the total flavonoids content determined by the Davis method (Fig. 3D). The high content of flavonoids in BM1 and BM2 are hesperidin, narirutin and didymin, while BO1 and BO2 are the same. The contents of naringin, neohesperidin and narirutin are high in BC1 and BC2 (Fig. 4A). The composition of flavonoids in the six beverages is consistent with the known components of flavonoids in the peel and pulp of the three fruits, but at higher levels, because of the higher content of flavonoids in the segment membrane. This may be because the ancestor of satsuma mandarin and sweet orange is *Citrus reticulata*, while the ancestor of grapefruit is *Citrus maxima (Burm) Merr*, which lead to the great

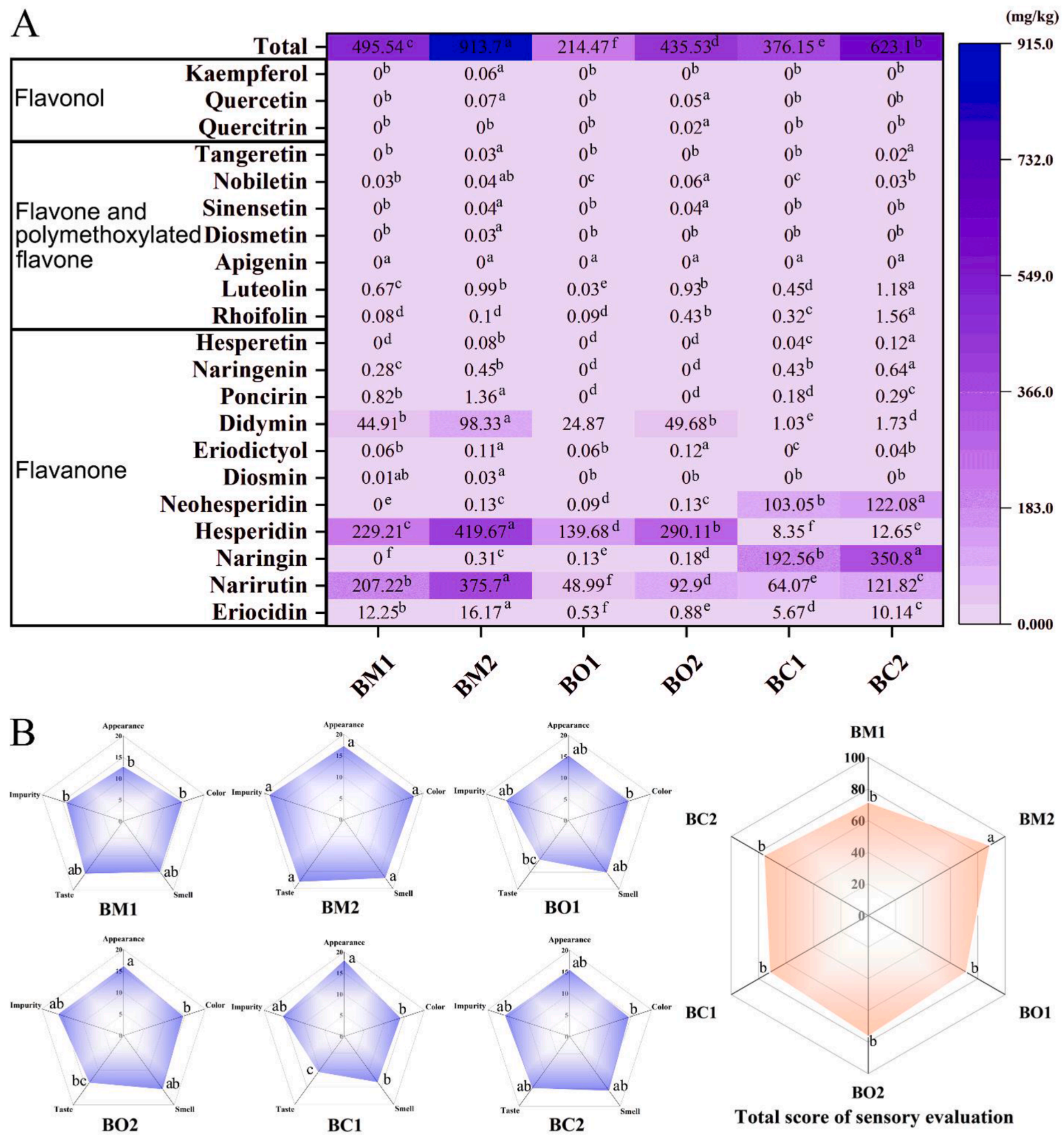

Fig. 4. (A) Heat map of the composition and content of flavonoids in beverages. (B) Sensory evaluation on the appearance, color, smell, taste and impurity of 6 beverages and an overall sensory evaluation. Different lowercase letters indicate significant differences ($P < 0.05$) between different beverages.

difference in the composition of flavonoids (Wang, et al., 2016). Flavonoids in beverages are mainly composed of flavanones. Flavones, polymethoxyflavones and flavonols are low in content and almost absent in BO1 and BC1(Fig. 4A).

Sensory characteristics of beverages

Sensory evaluation of the six beverages was carried out in five aspects. The total score of sensory evaluation of BM2 is significantly higher than that of other beverages (Fig. 4B). And the evaluation score of each descriptive item is also slightly higher, especially the taste. BM2 has suitable turbidity, uniform texture, and no precipitation; the color is like fresh citrus, bright and consistent; there is a clear citrus scent, no peculiar smell; the taste is in harmony with sweet and sour, with an obvious citrus beverage taste and there are no obvious impurities.

Therefore, BM2 meets the standard requirements of electrolyte beverages, has high contents of bioactive compounds, and is the most recognized in descriptive sensory characteristics, the industrialization of BM2 can be considered.

Economic estimates

0.4% citric acid, 0.1% HCl, 0.2% NaOH and 0.1% KOH are required for the optimized segment membrane removal process, while 0.6% HCl and 0.3% NaOH are needed in the traditional process. The cost is calculated based on processing 1 ton of citrus segments. It can be seen from Table 2 that the new process costs $1.74/ton of citrus segments more than the traditional process.

BM2 is selected as the most suitable beverage for industrial production according to the results above. Taking its production process as an example and calculating beverage production costs based on processing one ton of segments. Nearly two tons of beverages were produced by mixing acid and alkali water at a ratio of 1:1 (w/w). The added sugar and essence are calculated at 6.21% and 0.02%, respectively (Appendix Table 1). It can be seen from Table 2 that the beverage costs mainly consist of bottles, sugar, heating steam, etc. (where bottles are the main costs). If each bottle of beverage is priced at $0.08, the cost of each bottle of beverage is about $0.066, then the profit is about $0.014/bottle. Two tons of beverages (40,000 bottles) generate a profit of $560.

Through costs accounting, the costs of the new segment membrane removal process are only $1.74/ton of citrus segments higher than the

Table 2
Cost comparison of the new and traditional segment membrane removal process and beverage production ($/ton citrus segments).

Items	Cost comparison of segment membrane removal process Traditional	New
Citric acid [a]	0	2.82
30% hydrochloric acid [b]	2.20	0.36
30% NaOH [c]	1.88	1.25
KOH [d]	0	1.38
Heating steam	the same	the same
Water [e]	1.26 + rinse water (the same)	1.27 + rinse water (the same)
Costs of labor	the same	the same
Total	5.34 + rinse water + costs of labor	7.08 + rinse water + costs of labor
	Cost comparison of beverage production	
Items	Traditional	New
Heating steam costs	/	93.90
Sucrose [f]	/	106.81
Essence [g]	/	4.80
Bottle (50 mL) [h]	/	2400
Labor [i]	/	31.25
Total	/	2636.76

[a] The price of citric acid (food grade) is $704/ton.
[b] The price of 30% hydrochloric acid (food grade) is $110/ton, which is used to prepare acid water.
[c] The price of 30% NaOH (food grade) is $188/ton, which is used to prepare alkali water.
[d] The price of KOH (food grade) is $1377/ton.
[e] The price of water is $0.64/ton.
[f] The price of sucrose is $860/ton.
[g] The price of essence is $12/kg.
[h] It takes 40,000 bottles to fill 2 tons of beverages and the price of each bottle is $0.06.
[i] 5 workers are required at a maximum, and each worker is paid $50 for working 8 h a day in China. The beverage production process takes 1 h.

traditional one. However, the acid and alkali processing water produced by the traditional process are discharged, the processing water produced by the new process can be directly mixed to prepare electrolyte beverages and obtain a profit of $560/ton of citrus segments. Therefore, it is of great economic value to adopt the improved segment membrane removal process to prepare canned citrus and use the canning processing water for beverage production.

Conclusion

This study optimized the segment membrane removal process of citrus on the premise of ensuring that the segment membrane could be completely removed. After changing the chemical composition of acid and alkali processing water, the costs of the process did not increase, the mixture of acid and alkali water had no salty taste and was directly made into products rich in electrolytes and bioactive compounds (pectin and flavonoids) which had great health benefits and market profits. At the same time, three kinds of canned fruits were also produced. Among the three fruits, satsuma mandarin is the most suitable one for the new segment membrane removal process to prepare canned citrus and electrolyte beverages, while grapefruit is relatively unsuitable. This study has achieved the goal of zero discharge of citrus canning processing water while recovering bioactive compounds at low cost and made into beverages, which has great environmental and economic benefits.

CRediT authorship contribution statement

Sihuan Shen: Investigation, Writing – original draft, Formal analysis, Visualization. **Huan Cheng:** Investigation, Formal analysis. **Ying Liu:** Investigation. **Yanpei Chen:** Visualization. **Shiguo Chen:** Conceptualization, Supervision. **Donghong Liu:** Data curation. **Xingqian Ye:** Conceptualization, Supervision, Funding acquisition, Project administration. **Jianle Chen:** Conceptualization, Supervision, Writing – review & editing, Investigation, Methodology.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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