

## Full recovery of value-added compounds from citrus canning processing water

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### ABSTRACT

The citrus canning industry generates large quantities of processing water rich in phytochemical compounds during the sequential acidic and alkaline removal of the citrus segments membrane. However, this processing water was discharged as an effluent causing high chemical oxygen demand (COD). In the present research, a pilot scale process was designed to simultaneously recover pectin and a low molecular weight (Mw) fraction. The pectin was stable in yield and showed a relatively high content of galacturonic acid (~60%) throughout the production season. The low Mw fraction was rich in oligosaccharides (~11 mg/mL) and flavonoids (~3 mg/mL). Oral intake of the low Mw fraction could remarkably inhibit the growing of human non-small cell lung cancer PC-9 cells in nude mice. After treatment, the COD of the water was reduced by more than 95%, allowing more economical disposal of the wastewater. It is estimated that a citrus canning factory could retrieve about 110 tons of pectin in a production season, in addition to large amounts of low Mw fraction. Such an approach to recover valuable compounds from processing water is both beneficial for the environment and economy.

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### 1. Introduction

A growing global population leads to an increasing demand for food production, which consequently leads to the generation of large amounts of food processing waste. However, environmental damage caused by ground water contamination resulting from food processing waste entering landfill can be largely avoided. Unlike the processing waste from other industry, food processing waste usually contains large amounts of edible compounds, which may have health benefits to human. Considering human health and environmental safety, development of novel techniques for the recovery of commercially important biomolecules from food processing waste is a priority. In recent years, technologies for extraction of biologically active molecules from food processing waste have also raised economically interesting prospects.

Citrus fruits are the most important economic crops among the

world. In 2011, the Chinese cultivated area and output of citrus fruits reached 2.29 million hectare and 29.44 million tons, respectively, making it the leading producer in the world (Ministry of Agriculture, 2013). Among the various citrus fruits, sweet orange (*Citrus unshiu*) is one of the most widely consumed. Canning is one of the main ways to preserve citrus products. Peeled segments of the sweet orange (*Citrus unshiu*) are mainly used for canning by the Chinese fruit processing industry. The membrane of the citrus segments are removed by a sequential acidic and alkaline treatment, which yields great amounts of processing water with high chemical oxygen demand (COD) (~10,000 mg/L). In general, the production of 1 ton of peeled segments used for canning will result in about 3.6 tons of high COD processing water as effluent. However, this processing water contains beneficial phytochemical compounds from dissolution of the segments membrane, including high amounts of pectin, oligosaccharides and flavonoids. Thus, improper treatment of the processing water will not only cause severe environmental problems but also incurs a loss of large amounts of valuable food compounds.

Pectin plays an important role in food processing as a safe food

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additive with no limits on acceptable daily intake (Gnanasambandam and Proctor, 1999). Meanwhile, as a dietary fiber, citrus pectin has beneficial effects such as reducing cholesterol (Terpstra et al., 1998), anti-inflammatory (Salman et al., 2008) and playing a role in heavy metal chelation (Eliaz et al., 2007). As one of the most important phytochemicals in food, dietary flavonoids exert a wide range of benefits for human health. Recent research has explored the diverse biological and pharmacological activities of citrus flavonoids, for instance, antioxidant activity (Hertog et al., 1993; Zou et al., 2016), anti-diabetic activity (Shen et al., 2012), anticancer activity, anti-inflammatory activity, cardiovascular protection (Benavente-García and Castillo, 2008; Chanet et al., 2012; Tripoli et al., 2007), and neuroprotective effects (Hwang et al., 2012). Thus, recovery of these compounds from the citrus canning processing water should both benefit human health and generate income.

Currently, the main processing methods used to treat citrus canning processing water do not allow for the retrieval of any beneficial compounds. For example, flocculants such as poly-aluminum chloride has been used to remove pectin (Pavon-Silva et al., 2009), but does not provide an opportunity to recover pectin from the flocculent mixture and produces a secondary pollution stream. Biodegradation methods using microorganism were also examined to degrade the pectin (Tanabe et al., 1986, 1987). However, only a limited reduction in the COD was achieved and no compounds could be recovered. Thus, it is an imperative that the citrus canning industry establishes a system to not only recover the high-value compounds but also realize greener production. The difficult for recovery is a consideration of both technology and economy. Direct recovery of the compounds will cause high expenditure, such as use of ethanol, thus, it is important to mix and concentrate the water. We have successfully established a pilot water reuse system for saving water in the citrus canning processing and achieved a reduction in the amount processing water required (Wu et al., 2016). An attempt to recover pectin from the concentrated water has also been successful in a pilot study. However, after recovery of the pectin, the water still contains large amounts of compounds, including oligosaccharides and flavonoids, which cannot be directly discharged. Furthermore, previous studies, which precipitated pectin from the acidic and alkaline processing water separately, consumed a large amount of alkali and acid for neutralization and were not economic system for pectin recovery.

Thus, the present study describes a pilot scale process to fully recover compounds, including pectin, oligosaccharides and flavonoids, present in processing water resulting from citrus canning. The aim of the study was to fully recover the high-value compounds in the citrus canning processing water and reduce environmental pollution.

## 2. Materials and methods

### 2.1. Analysis of processing water

The processing water including acidic and alkaline water discharged during removal of citrus segments membrane was obtained from a citrus canning factory in Ningbo, China on three dates (26/11/2014, 15/12/2014, 08/01/2015) during the production season (about from November to January of the following year). Water samples were analyzed immediately after collection.

The COD value of the processing water was determined by the COD test tube assay using the Spectroquant<sup>®</sup> NOVA60 (Merck, Germany). Briefly, all the water samples were representatively collected in 1 L volumes and shaken sufficiently before dilution and

removal of 1 mL aliquots into the test tube. The same sampling method was applied in the following tests. The total flavonoid content of the processing water was determined by sodium hydroxide-diethylene glycol colorimetric assay (Davis, 1947). The pectin content of the processing water was determined by ethanol precipitation and then weighed (Yapo et al., 2007). Briefly, a volume of acidic water (or alkaline water) was taken and then filtered through a filtration fabric (400 mesh). The filtered acidic water (or filtered alkaline water) was treated with NaOH solution (or HCl solution) to adjust to a pH of between 4 and 6. Ethanol was added to achieve a final concentration of 50% (v/v) and the solution gently stirred to precipitate pectin. After standing for 30 min, precipitation was complete. The resulting pectin was washed with ethanol, squeezed within the fabric, and dried in an oven at 70–80 °C. The yield of pectin was calculated by the formula:  $Y=W/V$ , where Y represents yield, g/L; W is the weight of dry production powder, g; V is the volume of acidic water (or alkaline water), L. The same recovery method was applied to extract pectin to analyze its quality by the method described in Section 2.4. Other water indices were determined according to standard methods (Chinese Environmental Protection Administration, 2002). All the determinations were conducted in triplicate. The results were all expressed as a range representing the basic characteristics of processing water throughout the production season.

### 2.2. Pilot scale recovery process for high-value compounds

The pilot scale process for recovering the compounds is shown in Fig. 1, and includes two parts:

**Extraction of pectin:** The recovery of the pectin was similar as our previous work (Chen et al., 2017), but some modifications have been made to save acid and alkali use. The acidic and alkaline water were collected in two separate collecting tanks (30 m<sup>3</sup>) and used as the seed material to recover the compounds in the pilot scale process. The water was transferred through the equipment by pump. At the beginning, two filtration steps using brush filters were used to eliminate soluble fibers from the acidic and alkaline water. The filtered acidic water and filtered alkaline water were mixed under stirring to reach a pH of 4–6, which avoided the usage of additional acid or alkali. This mixed water was concentrated at 70–80 °C under vacuum. In brief, mixed water was circulated in pipelines under heating and vacuum circumstance. The mixed water became thicker as it lost water. The concentration process stopped when mixed water flowed with difficulty in the pipelines, which could be judged by observing its flow behavior through pipeline glass window on its circular pathway. The resulting concentrated water was then cooled in a tank. After cooling to room temperature, ethanol precipitation (final ethanol concentration of 50%, v/v) was performed with gentle stirring in a second operation. After standing for 30 min, precipitation was complete. Following filtration and squeezing in a cloth bag, primary pectin and filtered water were obtained. Primary pectin was washed in ethanol for two times and squeezed within the cloth bag again, before vacuum drying at 70–80 °C.

**Production of low molecular weight (Mw) fraction:** The alcohol present in the filtered water was recovered by distillation and the water was then desalted by electrodialysis. For the desalination, in brief, sets of 60 L of sample were used, 10 L as the concentrate (pure water) and 50 L as the diluent solution (pectin-recovered water after eliminating alcohol). Electrolyte solution was 0.05 M NaSO<sub>4</sub>. Flow rate for both solutions was 800 L/h and 400 L/h for the electrolyte. The desalted water was subjected to nanofiltration. In brief, it was performed at a pressure of 0.3–0.5 MPa and flow rate of 450 L/h to retain the low Mw fraction, and the permeate water was discharged.

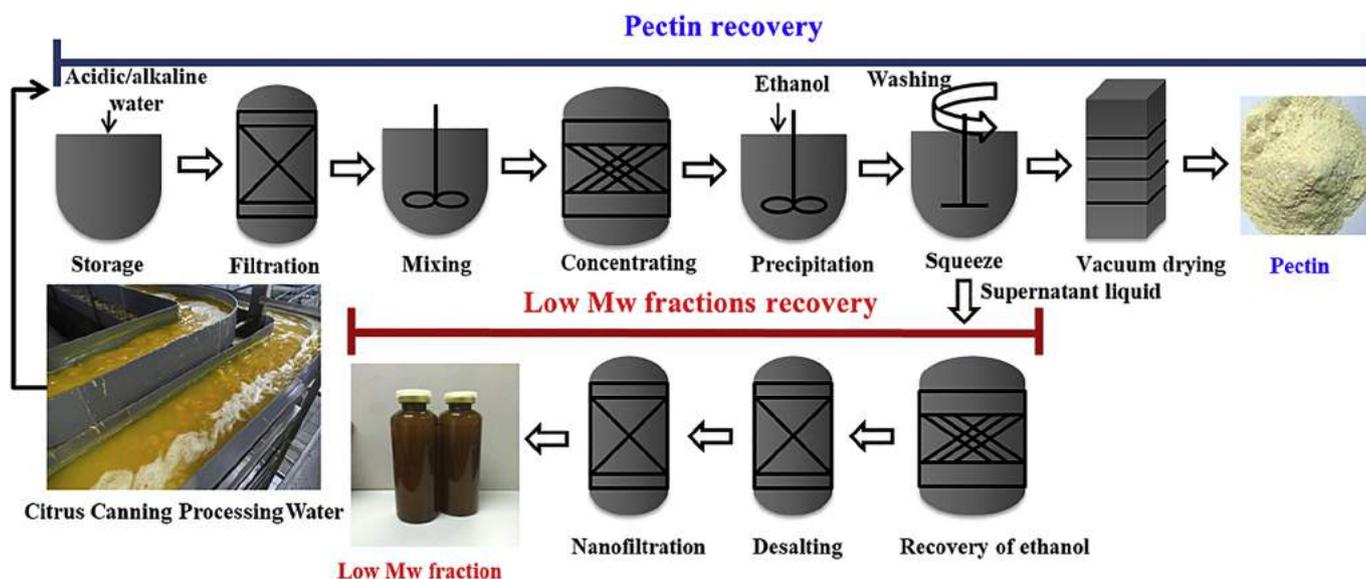


Fig. 1. Process of recovering compounds from the processing water.

### 2.3. Periodical analysis of mixed water and concentrated water in the pilot scale process

Homogeneous samples of the mixed water and concentrated water were taken on seven dates (from 26/11/2014 to 08/01/2015 about every 6 days) in the pilot scale process. Water samples were analyzed immediately after collection.

The total solids and pectin content of the mixed water and concentrated water were analyzed by the method described in Section 2.1. The pectin extracted from the mixed water and concentrated water was analyzed by determining galacturonic acid (GalA) content by the method described in Section 2.4. All the determinations were conducted in triplicate. The results were reported as mean with standard deviation.

### 2.4. Analysis of the recovered pectin

The GalA content and degree of esterification (DE) of the recovered pectin were determined by the acid-base titrimetric method (Guo et al., 2012) with slight modification. Briefly, the pectin sample (5.0 g) was mixed with acidic ethanol (5 mL HCl, 2.7 M and 100 mL ethanol solution (60%, v/v)) and stirred for 10 min. The mixture was filtered through a sand core funnel (G3, 4.9 μm) and washed 6 times with 15 mL acidic ethanol. The HCl was removed from the pectin by washing with ethanol solution (60%, v/v), and subsequently washed with 20 mL anhydrous ethanol before drying at 105 °C for 2.5 h. The dried sample (500 mg) was transferred to a 250 mL flask and was dissolved in 100 mL of carbon dioxide-free water after being moistened with 2 mL of ethanol. After the sample was completely dissolved, five drops of phenolphthalein were added. Then, it was titrated with 0.1 M NaOH solution and the result was recorded as the initial titer ( $V_1$ ). Then, 20 mL of 0.5 M NaOH solution were added, and the sample was shaken vigorously. It was allowed to stand for 15 min before adding 20 mL of 0.5 M HCl solution. The sample was shaken until the pink color disappeared and was titrated with 0.1 M NaOH solution to a faint pink color that persisted after vigorous shaking (end-point). This volume of titration was recorded as the saponification titer (the final titer,  $V_2$ ). The GalA content and DE were calculated from the following formula: GalA content (%) =  $19.41 \cdot (V_1 + V_2) \cdot 1000 / m$ , where  $m$  is the quantity of dried sample, mg; DE (%) =  $V_2 / (V_1 + V_2) \cdot 100\%$ .

### 2.5. Analysis of the recovered low Mw fraction and permeate water

Two beakers were used to collect about 1 L of homogeneous samples of both the low Mw fraction and permeate water from the pilot scale process on three dates (26/11/2014, 15/12/2014 and 08/01/2015). Water samples were analyzed immediately after collection. The flavonoid analysis of the low Mw fraction was subjected to a HPLC analysis with a reverse phase column (ZORABX SB-C18, 250 × 4.6 mm internal diameter) according to Zhang et al (Zhang et al., 2014), with some modification. Briefly, a two-solvent gradient mobile phase system consisting of water with 0.1% formic acid (A) and methanol (B) was used. The gradient program was performed as follows: 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. The flow rate was 0.7 mL/min, and the column temperature was maintained at 25 °C. The detection wavelengths were set at 283 nm, and 17 kinds of citrus flavonoids were used as standard. Total sugar content of the water samples was determined after dilution by the phenol-sulfuric acid assay (Dubois et al., 1956).

The Mw distribution analysis of the low Mw fraction by HPGPC was performed on a Waters Ultrahydrogel 250 column (3.9 × 300 mm) (Milford, MA, USA) eluted by 0.2 M NaCl aqueous solution at the flow rate 0.5 mL/min monitored with a refractive index detector. Glucan standards are used to determine the Mw of the samples. Other water indices were determined as described in Section 2.1. All the determinations were conducted in triplicate. The results were all expressed as a range reflecting the properties of the water throughout the production season.

### 2.6. Antitumor activity of the low Mw fraction

BALB/c (nu/nu) nude mice, male, SPF, 4–6 weeks old, body weight  $20 \pm 2$  g, were bought from Shanghai Sippr-BK laboratory animal Co., Ltd (Shanghai, China) and were fed for 7 days before experiment. Human non-small cell lung cancer PC-9 cells under logarithmic growth were collected to adjusted the concentration of cell suspension at  $1 \times 10^7$ /mL and then they were took a subcutaneous injection (0.2 mL per mouse) into back of nude mice. After regular feed for 5 days at SPF, they were randomly divided into five groups as tumor model control group, cisplatin (3 mg/kg) control group, high dosage (337.50 mg/kg) of low Mw fraction group,

middle dosage (168.75 mg/kg) of low Mw fraction group and low dosage (84.38 mg/kg) of low Mw fraction group. Three low Mw fraction groups were given the low Mw fraction (15 mg/mL of total soluble solids) and the tumor model group was given saline by oral for five days a week of continuous five weeks. And the cisplatin control group was given cisplatin by intraperitoneal injection for two days a week until the average tumor volume reached over 50 mm<sup>3</sup>. The tumor diameter was the main index and was measured for 3–4 days a time. According to the tumor diameter, the tumor volume (TV) was calculated by the following formula:  $TV = \pi/6 \cdot a \cdot b^2$ , where *a* represents the longest diameter and *b* represents the shortest diameter.

### 2.7. COD analysis of the pilot water samples

Homogeneous samples of the acidic water, alkaline water, filtered acidic water, filtered alkaline water, mixed water, pectin-recovered water (eliminating alcohol), desalted water and permeate water were obtained from the pilot scale process on three dates (26/11/2014, 15/12/2014, 08/01/2015). Their COD values were determined as described in Section 2.1. Water samples were analyzed immediately after collection, and all the determinations were conducted in triplicate. The results were reported as mean with standard deviation.

## 3. Results and discussion

### 3.1. Composition of the processing water

For full recovery of the compounds, the quality of the processing water was first studied (Table 1). The sequential food grade acidic (HCl) and alkaline (NaOH) steeping process dissolved the whole citrus segments membrane, and released insoluble fibers, pectin and flavonoids, resulting in a high COD value in both kinds of processing water. Most of the flavonoids were removed during the acid steep, and less was left in the later alkaline process. However, the alkaline treatment destroyed membrane tissue more easily, generating higher pectin content in alkaline water. Meanwhile, other soluble polysaccharides and unstable methoxyl in the alkaline solution led to a lower GalA content and a lower degree of esterification of the pectin recovered from the alkaline water. In consideration of the similarity of acidic and alkaline water, it is popular to recover compounds together to save additional alkali and acid, and it is important to recover pectin and other compounds to reduce COD. In addition, these segments come from the inner layer of the citrus fruit, which avoids contamination of pesticides and pathogens, and the strong acidity or alkalinity of the processing water inhibits microbial growth, which ensures the safety of the recovery process.

### 3.2. Recovery of pectin

Although the pectin content increased with the water reuse system, it was still not sufficiently economic for the direct recovery

of the pectin by ethanol precipitation, as a large amount of ethanol would be consumed in an industrial-scale recovery process. Thus, an industrial concentrator system was utilized to concentrate the solution. The concentration process was the most important physico-chemical treatment step in the pilot scale process. In previous research, we have succeeded in recovery of pectin from the concentrated acid and alkaline water. However, additional alkali or acid needs to be used for pectin precipitation under appropriate pH. Thus, considering the economic feasibility, the filtered acid and alkaline water were mixed in the present study to achieve requisite pH. Then, before ethanol precipitation, a vacuum thickener was used to concentrate the mixed water in the pilot scale process. Although concentration could save ethanol, the process was limited by the viscosity, which makes circulation of the fluid through the equipment more difficult. Thus, concentration process was judged by observing the flow behavior of concentrating water through pipeline glass window on its pathway.

### 3.3. Periodical analysis of mixed water and concentrated water from the pilot scale process

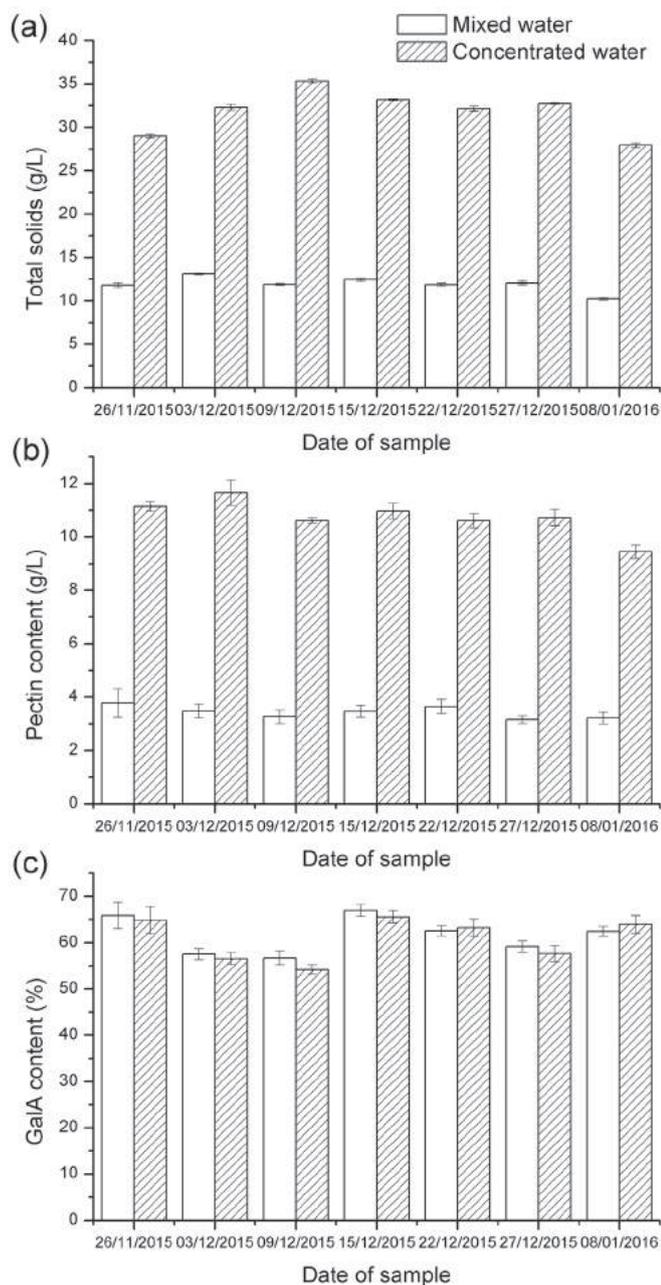
The stability of pectin during the concentration needs to be considered, as these polymers are easily aggregated and loss their ability for gelation. Periodical analysis of mixed water and concentrated water showed the stability of the process throughout the production period. Based on the stable quality of the processing water, mixed water exhibited a stable total solids concentration (~12 g/L, Fig. 2a) and pectin content (~3.5 g/L, Fig. 2b). After concentration, the total solids reached ~33 g/L (Fig. 2a), while the pectin content achieved concentrations of ~11 g/L (Fig. 2b). This indicated that the conversion of mixed water to concentrated water achieved a 3-fold change in total solids, and resulted in no obvious loss of pectin. The GalA content determined the quality of pectin. The GalA content of pectin extracted from concentrated water was close to that of pectin extracted from mixed water (55%–65%, Fig. 2c), which was between the GalA content of pectin extracted from acid and alkaline water. This confirmed that the mixture and concentration process had a limited effect on the quality of pectin. In conclusion, concentration of mixed water was confirmed to be an economical and effective process before pectin precipitation.

### 3.4. Ethanol precipitation and wash

Pectin was usually extracted for use as a food additive by ethanol precipitation (Kalapathy and Proctor, 2001; Wang et al., 2007). The concentrated mixed water was further precipitated by ethanol under an optimized concentration (50%, v/v) to reduce the ethanol consumption according to our previous work (Chen et al., 2017). The primary pectin (initial precipitation) contained large amounts of impurities, mainly flavonoids and salts. Thus, another wash with ethanol improved the quality of pectin and exchanged water within the pectin to avoid caking of the pectin during vacuum drying. The dried pectin was further powdered for better water solubility.

**Table 1**  
The quality of processing water following the segments membrane removal process during citrus canning.

Indices	Visible material	pH	Acidity/Alkalinity (g/L)	Total soluble solids (g/L)	Chemical oxygen demand (mg/L)
Acidic water	Insoluble fibers	0.81–1.00	6.17–7.91	5.56–7.68	10720–13550
Alkaline water	Insoluble fibers	12.65–13.00	3.59–4.71	12.02–16.91	10210–12090
Indices	Total solids (g/L)	Total flavonoids (mg/L)	Pectin (g/L)	Pectin galacturonic acid (%)	Pectin degree of esterification (%)
Acidic water	7.11–9.73	652–839	1.87–3.16	59.14–71.01	17.20–35.21
Alkaline water	15.25–19.16	18–30	3.37–4.58	55.44–63.23	4.32–11.51



**Fig. 2.** Periodical analysis of mixed water and concentrated water from the pilot scale process: (a) total solids (g/L); (b) pectin content (g/L); (c) GalA content (%) of pectin.

### 3.5. Analysis of low Mw fraction and permeate water in the pilot plant

The processing water contained large quantities of flavonoids and oligosaccharides, which could not be recovered by ethanol precipitation. Thus, further steps including recovery of alcohol, desalinization and nanofiltration were employed in the process (Fig. 1).

Nanofiltration was used to concentrate the oligosaccharides and flavonoids while eliminating large volumes of permeate water. The quality of the low Mw fraction and permeate water was determined (Table 2) to evaluate the effect of the process on recovery of the low Mw fraction. Nanofiltration could be difficult when the low Mw fraction was viscous. Thus, a moisture analyzer was used to monitor the total soluble solids of the trapped liquid as an end point of nanofiltration. The low Mw fraction was rich in oligosaccharides

**Table 2**  
The quality of low Mw fraction and permeate water.

Indices	pH	Total soluble solids (g/L)	Total sugar (g/L)	Total flavonoids (mg/L)	Chemical oxygen demand (mg/L)
Low Mw fraction	5.10 –6.30	15.60–18.21	8.68 –12.88	2355–3180	– <sup>a</sup>
Permeate water	6.50 –6.80	<0.2	<0.1	<100	354–422

<sup>a</sup> “–”: not tested.

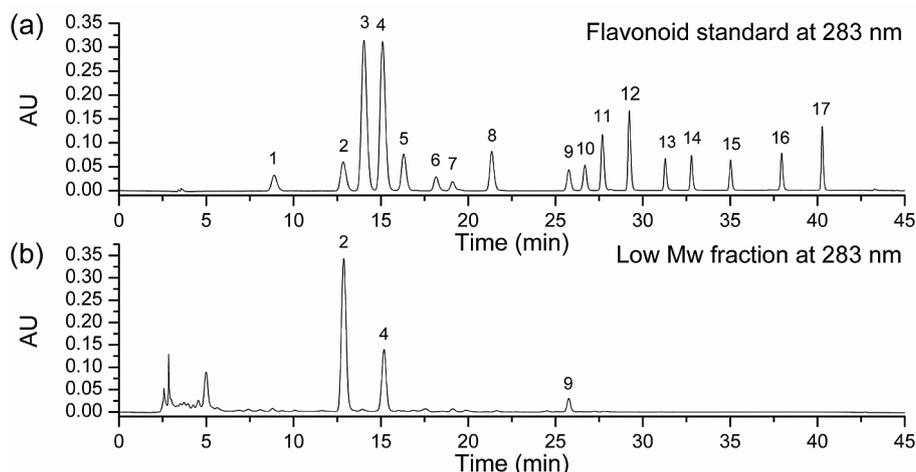
and flavonoids while the permeate water contained little organic matter. The flavonoids were composed of hesperidin, narirutin and small quantities of didymin as examined by HPLC, which mainly came from the acidic water (Fig. 3). This conformed to the distribution of flavonoids in oranges reported by Peterson et al., that hesperidin and narirutin occupied the most of flavonoids in sweet orange (Peterson et al., 2006). The Mw distribution of low Mw fraction was also analyzed by HPGPC (Fig. 4). The oligosaccharides, most of which were under 40 kDa in Mw, were similar to modified pectin. In conclusion, the low Mw fraction could be commercialized as a healthy food. Meanwhile, the permeate water contained almost no organic matter and, consequently, had a low COD value. So, the recovery process for processing water realized the aim of full recovery of compounds and almost no pollution concerns.

### 3.6. Antitumor activity of low Mw fraction

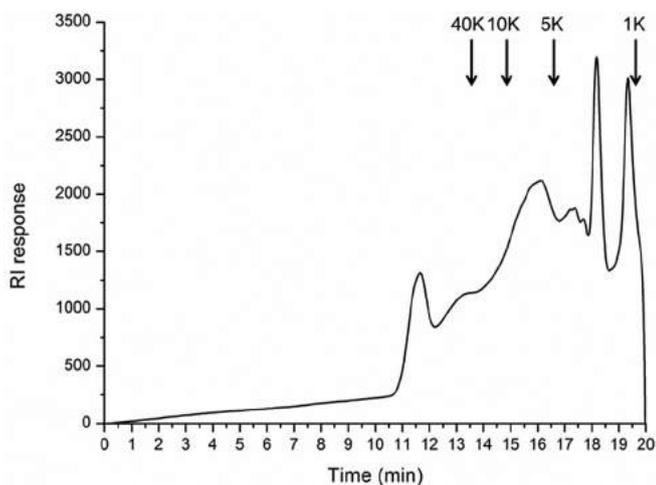
Citrus flavonoids have been reported diverse biological and pharmacological activities including anticancer activity (Benavente-Garcia and Castillo, 2008). And pectic oligosaccharides also show anticancer activity (Olano-Martin et al., 2003; Takei et al., 2010). Thus, in order to make sure the new recovery method is worthy of developing isolated compounds as an anticancer functional food, a preliminary animal experiment was undertaken to confirm its positive effects. The antitumor activity of recovered low Mw fraction compared with cisplatin was showed in Fig. 5. The antitumor activity of low Mw fraction was obviously dosage-dependent. After continuous 35 days of oral intake of low Mw fraction at high dosage (337.50 mg/kg), the growth of tumor from subcutaneous injection of human non-small cell lung cancer PC-9 cells into back of nude mice was obviously restrained at 53.9%. The low Mw fraction at middle dosage (168.75 mg/kg) had certain antitumor activity of inhibition ratio at 23.3%. However, the low Mw fraction at low dosage (84.38 mg/kg) had no certain antitumor activity of indistinctive inhibition ratio at 5.0%. The pectic oligosaccharides and flavonoids contributed to the antitumor activity of low Mw fraction.

### 3.7. The COD of different water samples in the recovery process

The COD of typical water samples during the process were shown in Fig. 6. After filtration, the COD of both acidic water and alkaline water were reduced by 30.0% and 16.3%, respectively. For acidic water, which contained more insoluble fibers than alkaline water, the COD value was further reduced following filtration. The COD of mixed water was between that of filtered acidic water and filtered alkaline water, since the COD was directly related to the content of organic matter. After extracting pectin, the COD value was reduced by 63.9%, since large amounts of organic matter was removed. Desalinization had no impact on the COD. Finally, through nanofiltration, almost all of the oligosaccharides and flavonoids were trapped and concentrated while the COD of permeate water was reduced to under 500 mg/L.



**Fig. 3.** The flavonoids analysis by HPLC of low Mw fraction: (a) flavonoids standard; (b) low Mw fraction. Peaks label: 1 Eriocitrin, 2 Narirutin, 3 Naringin, 4 Hesperidin, 5 Neohesperidin, 6 Rhoifolin, 7 Quercitrin, 8 Eridictyol, 9 Didymin, 10 Poncirin, 11 Naringenin, 12 Hesperetin, 13 Kaempferol, 14 Diosmetin, 15 Sinensetin, 16 Nobiletin, 17 Tangeretin.



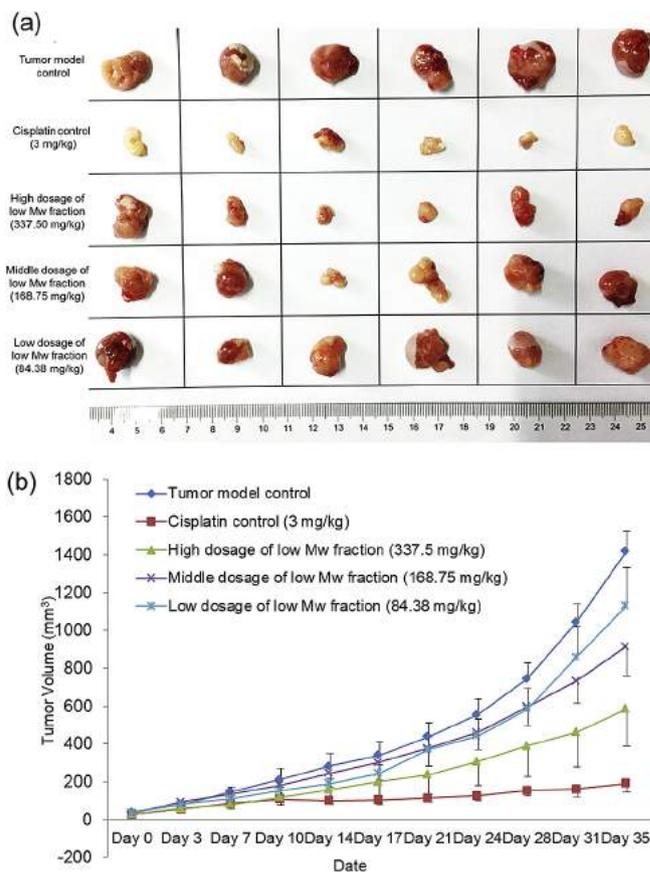
**Fig. 4.** The Mw distribution of low Mw fraction.

### 3.8. Estimation of the economic value for full recovery

For a factory producing 10,000 tons of canned citrus during a production season, the discharge of acidic and alkaline water used for removal of the segments membrane could be estimated to be 250 ton/day (20,000 ton/production season) and 200 ton/day (16,000 ton/production season), respectively. Thus, estimation for the amount of pectin, which could be recovered during a production season, is shown in Table 3. The pilot scale process developed in this study indicated that implementation of the recovery process to processing water could lead to the production of large amounts of pectin with great economic value.

Apart from the pectin, the production amount of the low Mw fraction was similar to that of pectin in quantity, which was about 1 ton/day, with about 15 mg/mL total solids content. It has shown remarkable effect on the antitumor actives and could be further develop as antitumor functional foods, which would have potential large economic value.

Lastly, the processing water after full recovery of the compounds could be reused, which occupied more than two-thirds of the initial quantity. This not only caused less pollution to the environment, but also reduced the use of water, which benefited both of the environment and economic.



**Fig. 5.** The antitumor activity of low Mw fraction.

## 4. Conclusions

The demand for environmental protection and efficient utilization of resources has continued to rise and enterprises are encouraged continually to adopt new technologies to reduce pollutants. The citrus canning industry has become one of the main effluent water discharge industries. After a systematic analysis of the quality, the processing water produced during removing segments membrane has showed great potential for recovery of high value compounds.

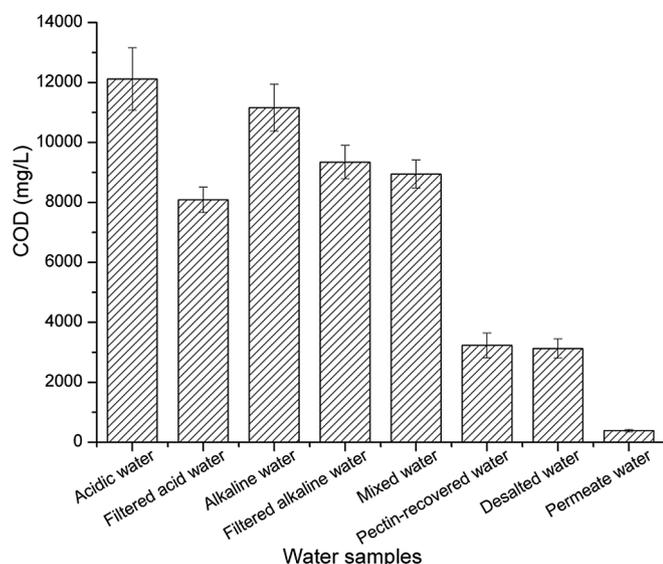


Fig. 6. The COD of different water samples on recovery process.

Table 3

The estimation of recovered pectin during a production season.

Indices	The discharge of processing water	Recovered pectin
Acidic water	250 ton/day	610 kg/day <sup>a</sup>
Alkaline water	200 ton/day	774 kg/day <sup>b</sup>
Total	450 ton/day	1384 kg/day
	36,000 ton/production season	110.72 ton/production season

<sup>a</sup> The acidic water pectin content was estimated at 0.224% (w/w).

<sup>b</sup> The alkaline water pectin content was estimated at 0.387% (w/w).

A pilot scale process including filtration, mixture, concentration, extracting pectin, recycling alcohol, desalinization and nano-filtration was designed, to recover pectin and low Mw fraction in the processing water, while reducing its COD from over 10,000 mg/L to lower than 500 mg/L. More than two-thirds of the initial quantity of the processing water could be reused. For a factory canning 10,000 tons of citrus in a production season, implementation of the process would allow recovery of about 110 tons of pectin, which has significant economic value. A similar amount of low Mw fraction was recovered, which showed an inhibition of human non-small cell lung cancer PC-9 cells growing in nude mice when administered orally, and might be further developed as an anticancer functional food for human health. Besides, compared with the traditional method of pectin extraction, our method avoids the use of hot acid or alkali reducing the chemical byproducts of pectin and flavonoids. Thus, we consider these compounds are safer and of higher quality than those produced by using traditional methods. This study identified a safe and effective way of recovering edible compounds from the processing water produced during citrus canning processing, which provides a case study for the food industry. The study is beneficial to develop a coordinated and sustainable society.

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