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The Study on Extraction Technology of Flavonoids from *Tribute Citru* Peel

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Abstract. *Tribute Citru* peel is rich in various natural products. Total flavonoids from *Tribute Citru* peel were extracted by solvent extraction method and the content of total flavonoids was determined by UV spectrophotometer with rutin as standard. The effects of reflux time, solid-liquid ratio, ethanol concentration and temperature on the extraction of flavonoids were investigated. The optimum condition determined by orthogonal test was that *Tribute Citru* peel and 80% ethanol were mixed in the solid-liquid ratio of 1:20, then heated for 4h at 90 °C. Under this condition, the extraction rate yield of flavonoids reached 1.43%.

1. Introduction

Tribute Citru is a natural hybrid of sweet orange and orange, which contains abundant nutrients, such as vitamins, fructose, amino acids [1]. It is popular with people because of cheap price and sweet taste [2]. The famous producing area of *Tribute Citru* is Deqing County, Zhaoqing City, Guangdong Province, China. After eating or processing, a lot of *Tribute Citru* peel residues were produced. It is reported that *Citru* peel is rich in essential oil, flavonoids, pectin, pigment and other natural products, which have high nutritional and medicinal value [3]. In our country, the main methods to deal with orange peel residue is landfill, which will produce a large amount of acidic liquid, pollute the environment and waste resources. Therefore, this research studies the extraction of flavonoids from the *Tribute Citru* peel, to develop new methods and new ideas for the rational development of orange peel residue. It will increase the added value of citrus products and protect the environment.

2. Experimental method

2.1. Material pretreatment

The *Tribute Citru* peels with smooth appearance were selected. After scraping off the white reticulated veins on the inner wall, peels were dried at 50 °C. After grinded and sifted out via 40 mesh, the powder of *Tribute Citru* peels were sealed tightly.



2.2. The standard curve

0.0984g rutin was accurately weighed, and 100 mL 60% ethanol was added to afford 0.984 mg/mL rutin standard solution. 0.00 mL, 2.00 mL, 4.00 mL, 6.00 mL, 8.00 mL and 10.00 mL of rutin standard solution were separately added into 50 mL volumetric flasks. Then 10mL 60% ethanol and 1 mL 5% NaNO_2 solution were added into volumetric flasks, followed shaking for 30s and keeping for 6min. 1 mL 10% $\text{Al}(\text{NO}_3)_3$ solution was transferred to the volumetric flask. After 6 min, 10 mL 5% NaOH solution was added into volumetric flask, which was diluted with 60% ethanol to make 50 mL solution. The solutions were detected via UV spectrophotometry at the wavelength of 500 nm [4]. Standard curve was made according to absorbance and concentration of rutin.

2.3. Extraction of flavonoids

10g of *Tribute Citru* powder and ethanol of different concentrations was added into a round bottom flask. After refluxing for different time, the solution was filtered and concentrated. Then 1.00 mL of concentrated solution was diluted with 60% ethanol in 50 mL volumetric flasks [5]. According to the above preparation method (method 2.2), the absorbance value of total flavonoids of extracting solution was determined.

2.4. Single factor experiment

The effects of reflux time, solid-liquid ratio, ethanol concentration and temperature on the extraction of flavonoids from *Tribute Citru* peel were studied. The reflux time was 1, 2, 3, 4, and 5 h. The solid-liquid ratio was 15:1, 20:1, 25:1, 30:1, and 40:1. The concentration of ethanol was 50%, 60%, 70%, 80%, and 90%. The temperature was set to 75 °C, 80 °C, 85 °C, 90 °C, and 95 °C. According to the above standard curve preparation method (method 2.2), the absorption value of the sample was determined.

2.5. Orthogonal test

According to the results of single factor experiment, the extraction yield of flavonoids in *Tribute Citru* peel was optimized via $L_9(3^4)$ orthogonal experiment, and including four factors of reflux time, solid-liquid ratio, ethanol concentration and temperature.

3. Result and analysis

3.1. The standard curve

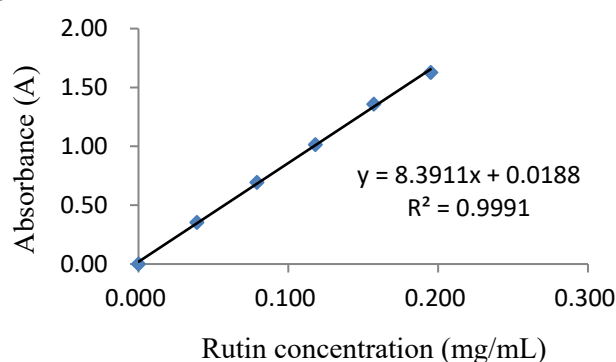


Figure 1. Rutin standard curve

Taking absorbance as ordinate and rutin concentration as abscissa (Figure 1), the standard curve was drawn and the calculation formula was as follow: $y = 8.3911x + 0.0188$ (1).

3.2. The effect of extraction time on the extraction of flavonoids in Tribute Citru peel

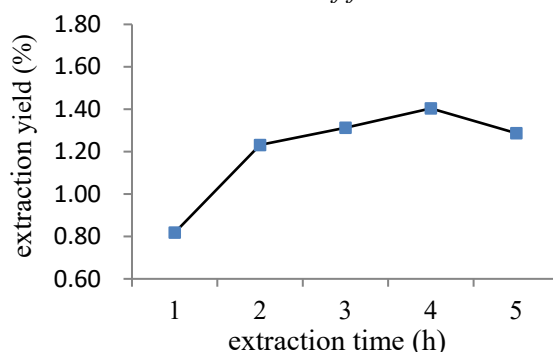


Figure 2. The effect of extraction time on extraction yield of flavonoids

According to Figure 2, the best extraction yield of flavonoids is 1.40% after heating 4 hours. With the increase of extraction time, the extraction yield increased, indicating that the dissolution of flavonoids also increased gradually. After more than 4 hours, the extraction yield was decreased. Therefore, considering the production efficiency and extraction yield, 4 hours was the most appropriate time for extraction.

3.3. The effect of solid-liquid ratio on the extraction of flavonoids in Tribute Citru peel

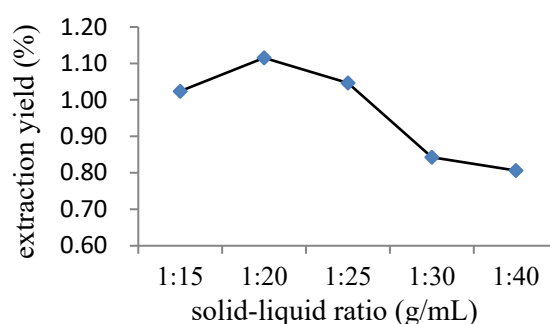


Figure 3. The effect of solid-liquid ratio on extraction yield of flavonoids

According to the experimental data (Figure 3), when the solid-liquid ratio was 1:20, the extraction yield of flavonoids was increased to the maximum. However, when the solid-liquid ratio was continued to increase, the extraction yield of flavonoids decreased slowly.

3.4. The effect of ethanol concentration on the extraction of flavonoids in Tribute Citru peel

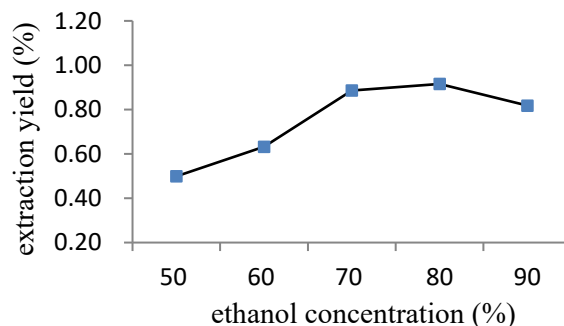


Figure 4. The effect of ethanol concentration on extraction yield of flavonoids

As shown in Figure 4, it was obvious that the extraction yield of flavonoids was increased steadily with the increase of ethanol concentration. When the ethanol concentration was 80%, the extraction results

was best. But the extraction yield of flavonoids was decreases when the ethanol concentration exceeded 80%.

3.5. The effect of temperature on the extraction of flavonoids in Tribute Citru peel

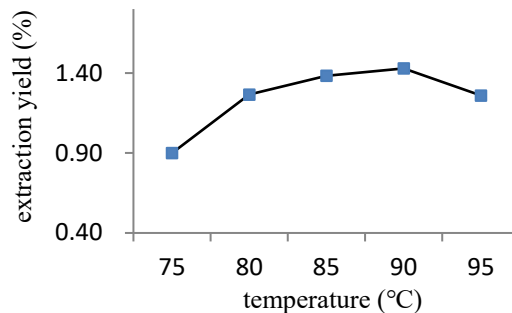


Figure 5. The effect of temperature on extraction yield of flavonoids

According to the experimental data, it indicated that extraction yield of flavonoids increased along with the temperature was raised. When the temperature was 90 °C, the extraction yield was reached the highest (1.43%). However, the extraction yield gradually declined once the temperature exceeded 90 °C. It was explained by the dissolved flavonoids might decompose at excessive temperatures.

3.6. The result of orthogonal test

According to the data of single factor experiment, the factor levels were shown in Table 1, and the results of orthogonal experiment were shown in Table 2. From the range R, it could be concluded that the degree of four factors affecting the extraction yield of flavonoids was: C > B > A > D. That is to say, factor C (extraction time) had a maximum impact on extraction yield, followed by factor B (solid-liquid ratio), factor A (ethanol concentration), and factor D (temperature). According to K value, the optimal extraction condition was A₂B₂C₃D₃. The optimum conditions were *Tribute Citru* peel and 80% ethanol solution were mixed at the solid-liquid ratio of 1:20, which was heated for 4h at 90 °C. Three validation experiments were carried out under optimal extraction conditions. And the average value was 1.43%, indicating the process was feasible.

Table 1. Factors and levels of orthogonal experiments design

	A	B	C	D
	ethanol concentration (%)	solid-liquid ratio (g/ml)	extraction time (h)	temperature (°C)
1	70	1:15	2	80
2	80	1:20	3	85
3	90	1:25	4	90

Table 2. Results and analysis of orthogonal experiments

entry	ethanol concentration (%)	solid-liquid ratio (g/ml)	extraction time (h)	temperature (°C)	extraction yield (%)
1	70	1:15	2	80	0.62
2	70	1:20	3	85	1.32
3	70	1:25	4	90	1.41
4	80	1:15	3	90	1.08
5	80	1:20	4	80	1.32
6	80	1:25	2	85	0.99
7	90	1:15	4	85	0.80
8	90	1:20	2	90	0.79
9	90	1:25	3	80	0.89
K ₁	3.34	2.49	2.40	2.83	
K ₂	3.39	3.43	3.28	3.11	

K ₃	2.47	3.29	3.53	3.27
k ₁	1.12	0.83	0.80	0.94
k ₂	1.13	1.14	1.09	1.04
k ₃	0.82	1.10	1.18	1.09
Range	0.31	0.31	0.38	0.15
order	C>B>A>D			
optimization levels	A ₂	B ₂	C ₃	D ₃
optimization condition	A ₂ B ₂ C ₃ D ₃			

4. Conclusion

In this experiment, the extraction of flavonoids from *Tribute Citru* peel was carried out by solvent extraction method. The optimal extraction condition was determined by orthogonal test. It was the solution of *Tribute Citru* peel and 80% ethanol at a solid-liquid ratio 1:20 was heated at 90 °C for 4 h, under which the extraction yield was 1.43%. The experimental condition was mild, the equipment was simple. This experiment could provide experimental support for extracting of flavonoids from *Tribute Citru* peel, which would further investigate the physiological activities of flavonoids. It would contribute to promote the industrialization development of citrus products and make rational use of resources.

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