The Study on Extraction Technology of Essential Oil from *Tribute Citru*

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Abstract. The essential oil from *Tribute Citru* peel was extracted by steam distillation. The extraction method was studied by single factor test and orthogonal test. According to the experiment results, the order of influence was: distillation time, sodium chloride solution concentration, soaking time, material-liquid ratio. The optimum condition determined by orthogonal test was 3.0% sodium chloride solution in the solid-liquid ratio of 1:16 and distillation time was 1.5h without soaking. Under this condition, the extraction yield of essential oil from *Tribute Citru* peel reached 9.29%.

1. Introduction

*Citrus reticulata* Banco, a citrus subfamily of Rutaceae Botany, is one of the most important agricultural products because of its rich species, wide planting area and high yield [1]. As the world's largest fruit production, a large amount of peel residue will be produced after eating or production and processing, which is approximately 30%~50% of the total weight of citrus. If pericarp is treated as waste, it is not only a great waste of resources, but also causes environmental pollution. Therefore, the comprehensive utilization of citrus peel residue and improving the added value of *Citrus* has become crucial in processing industry. Taking advantage of natural product from citrus peel sufficiently, such as essential oils, natural carotenoids, pectin, flavone and edible cellulose [2], which can be developed as food additives, chemical materials. The citrus essential oil is one of the most widely used natural flavor and fragrances [3]. The research showed that citrus peel essential oil has the functions of antibacterial[4], anti-inflammatory[5], anti-oxidation[6], insecticidal[7], promoting gastrointestinal peristalsis and secretion of digestive fluid[8]. The main extraction methods of citrus peel essential oil are steam distillation, expelling, solvent extraction, microwave radiation and supercritical extraction. Among them, steam distillation can be widely used in industrial production because of its simple equipment, low cost and high output.

*Tribute Citru* is a natural hybrid of sweet orange and orange. It has abundant nutrition and a unique taste, which is a popular fruit in citrus. Zhaoqing City is the main production area of *Tribute Citru*, and
the supply can’t satisfy the market demand. After eating or production and processing, *Tribute Citrus* peel is also valuable and rich in natural products. At present, the application of natural products in *Tribute Citrus* is still in primary stage. Flavonoids from *Tribute Citrus* were reported by Deng hongmei[9]. The extraction of essential oil from Tribute Citrus has almost not been reported yet. Therefore, the extraction of essential oil is imperative which has enormous potential in food and pharmaceutical industry.

2. Experimental method

2.1. Material pretreatment
The *Tribute Citrus* peel was selected with smooth appearance, which has no insect eyes, rust spots. The branches and the pulp were removed and the white inner pulp was scraped off. After completely dried at 50 °C, the *Tribute Citrus peel* was put into medicine grinder to crush. Then powder was passed 40 mesh screen and sealed storage.

2.2. The extraction processing
Steam distillation[10] was chosen as extraction method. 15g of *Tribute Citrus peel* powder was weighed accurately and added into a 250 mL round bottom flask which has zeolite. Then a certain volume of different concentrations sodium chloride solution was added according to the solid-liquid ratio, and round bottom flask was connected the volatile oil extractor and condenser tube. After soaking for different gradient time, the solution was refluxed for different gradient time. The volume of essential oil was readed after the essential oil and water are completely layered in the volatile oil extractor and the upper essential oil was collected.

2.3. Single factor experiment
The effects of soaking time, solid-liquid ratio, distillation time and NaCl concentration on the extraction of essential oil from *Tribute Citrus* peel were investigated. The soaking time was 0 h, 1.0 h, 2.0 h, 3.0 h and 4.0 h. The solid-liquid ratio of 1:12, 1:14, 1:16, 1:18 and 1:20 were studied respectively. The distillation time was set to 0.5 h, 1.0 h, 1.5 h, 2.0 h, 2.5 h and 3.0 h. And the NaCl concentration was 0%, 1.5%, 3.0%, 4.5% and 6.0%. The method (2.2) was adopted to extract the essential oil of *Tribute Citrus* peel, and each experiment was repeated 3 times.

2.4. Orthogonal test
The factors of extracting essential oil from Tribute Citrus peel were determined by single factor experiment. In order to further optimize the extraction conditions, the orthogonal experiment of L9 (3^4) was designed considering the factors of soaking time, solid-liquid ratio, distillation time, NaCl concentration.

3. Result and analysis

3.1. The effect of different soaking time on the extraction of essential oil

![Figure 1. The effect of soaking time on essential oil yield](image-url)
Under the condition of 3.0% sodium chloride solution in the solid-liquid ratio of 1:16 that distilled for 1.5 h, the single factor test of soaking time was taken to investigate the effect on the extraction yield of essential oil (Figure 1). The results showed that the longer the time of soaking time, the lower extraction yield was obtained. Therefore, the desirable result could be obtained without soaking.

3.2. The effect of solid-liquid ratio on the extraction of essential oil

Under the conditions of 3.0% sodium chloride solution in the solid-liquid ratio of 1:16 that distilled for 1.5 h without soaking, the single factor test of solid-liquid ratio was taken to investigate the effect on the extraction yield of essential oil (Figure 2). From the data, it showed that the influence of solid-liquid ratio on the extraction yield increased first and then decreased. When the solid-liquid ratio is less than 1:16, the extraction yield of essential oil increased as the solid-liquid ratio increased. When the solid-liquid ratio is 1:16, the extraction rate of essential oil reached the highest 9.06%. However, when the solid-liquid ratio is higher than 1:16, the extraction results decreased sharply with the increase of solid-liquid ratio. According to the result, the optimum solid-liquid ratio is 1:16.

3.3. The effect of distillation time on the extraction effect

Under the conditions of 3.0% sodium chloride solution in the solid-liquid ratio of 1:16 without soaking, the single factor test of distillation time was carried out to investigate the effect on the extraction yield of essential oil (Figure 3). The results showed that with the extension of distillation time, extraction yield increased or remained unchanged, which is maintaining at 8.55%. Unfortunately, when the distillation time was more than 2.5h, the extraction yield of essential oil was decreased slightly. A plausible explanation is that the damage degree of the cell wall was increased by heating with longer distillation time, leading to significant increase of extraction yield. When the distillation time is 1.5 h, maximum damage of the cell wall was reached, and the extraction yield remained unchanged despite distilled for the longer time. However, the extraction yield of essential oil decreased slightly when the heating time was longer than 2.5h. The longer time may cause heat-sensitive component of essential oil decomposed, resulting in lower extraction yield. Considering energy saving and extraction yield, the optimal distillation time is 1.5h.
3.4. The effect of NaCl concentration on the extraction effect

The osmotic pressure of orange peel cells would be changed by adding sodium chloride solution, which affecting the extraction yield of essential oil (Figure 4). Under the standard condition, NaCl concentration of 0%, 1.5%, 3.0%, 4.5%, 6.0% were investigated. The results showed that with the increase of NaCl concentration, the extraction yield of the essential oil increased. Because the osmotic pressure of the orange peel cells increased correspondingly, it accelerated the cracking and breaking of the cell wall to give a higher yield. The highest extraction rate of essential oil is 8.55% under the condition of 3.0% NaCl concentration. However, with the further increase of the NaCl concentration, the extraction yield of essential oil shows a downward trend. Therefore, the optimal concentration of sodium chloride solution is 3.0%.

3.5. The result of orthogonal test

According to result of the single factor experiment, L9 (3^4) orthogonal experiment was adopted to optimize the extraction process conditions. The factor levels are shown in table 1 and the results are shown in table 2. The degree of influence of each factor on the extraction of essential oil was as follows: distillation time > NaCl concentration > soaking time > solid-liquid ratio. So the best extraction conditions was A_1B_3C_1D_2, that is, the optimum conditions were the 3.0% sodium chloride solution in the solid-liquid ratio of 1:16 without soaking, then the solution was distilled for 1.5 h. Under this condition, the extraction yield of essential oil in Tribute Citrus peel reached 9.29%.

Table 1. Factors and levels of orthogonal experiments design

|   | A soaking time (h) | B solid-liquid ratio (g/ml) | C distillation time (h) | D NaCl concentration (%) |
|---|-----------------|------------------|-----------------|-----------------|
| 1 | 0               | 1:12             | 1.5             | 1.5             |
| 2 | 1.0             | 1:14             | 2.0             | 3.0             |
| 3 | 2.0             | 1:16             | 2.5             | 4.0             |

Table 2. Results and analysis of orthogonal experiments

| entry | soaking time (h) | solid-liquid ratio (g/ml) | distillation time (h) | NaCl concentration (%) | extraction yield (%) |
|-------|-----------------|------------------|-----------------|-----------------|-------------------|
| 1     | 0               | 1:12             | 1.5             | 1.5             | 8.27              |
| 2     | 0               | 1:14             | 2.0             | 3.0             | 8.10              |
| 3     | 0               | 1:16             | 2.5             | 4.5             | 8.61              |
| 4     | 1               | 1:16             | 1.5             | 3.0             | 8.67              |
| 5     | 1               | 1:12             | 2.0             | 4.5             | 7.65              |
| 6     | 1               | 1:14             | 2.5             | 1.5             | 7.70              |
| 7     | 2               | 1:14             | 1.5             | 4.5             | 8.44              |
| 8     | 2               | 1:16             | 2.0             | 1.5             | 7.76              |
| 9     | 2               | 1:12             | 2.5             | 3.0             | 8.78              |
| K_1   | 24.98           | 24.70            | 25.38           | 23.73           |                   |
| K_2   | 24.02           | 24.24            | 23.51           | 25.55           |                   |
| K_3   | 24.98           | 25.04            | 25.09           | 24.70           |                   |
4. Conclusion

In conclusion, we have studied the extraction of essential oil from *Tribute Citrus* peel by steam distillation. The optimum extraction conditions were the 3.0% sodium chloride solution in the solid-liquid ratio of 1:16 without soaking, then distilled for 1.5 h. Under this condition, the extraction yield of essential oil in *Tribute Citrus* peel reaches 9.29%. The materials and equipment involved in this test are cheap and easily available. Meanwhile, the reaction conditions are mild, environment friendly in the test. The citrus essential oil is widely used and has great potential in the field of medicine and food, which is worth further exploring. At the same time, making full use of the orange peel can improve the added value of *Tribute Citrus* and reduce the environmental pollution.

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