Study on ultrasonic assisted extraction of Shixiang vegetable volatile oil

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Abstract

This test discusses the ultrasonic assisted extraction conditions of Shixiang vegetable essential oil. On the basis of single factor test, orthogonal test was designed to optimize ultrasonic extraction conditions, and the effects of particle size, ultrasonic extraction time and material liquid ratio on the yield of volatile oil from spearmint were investigated. With the yield of spearmint volatile oil as the response value, the results showed that the optimum extraction process condition was 100 mesh, solid-liquid ratio was 1:40 (g:mL), ultrasonic extraction time was 20 min, Under these conditions, the yield of spearmint volatile oil was 8.95%.

Keywords

Ultrasonic assisted method, Shixiang vegetable, Extract; Volatile oil.

1. Introduction

Shixiang, also known as green mint, was a kind of spearmint [1]. It was rich in volatile oil, which can be used in food [2], medicine [3], daily chemical [4], biological control [5] and other aspects. Therefore, the extraction of volatile oil from *Shixiang* had great use value and economic significance.

The common extraction methods of essential oil were steam distillation and organic solvent extraction. Traditional steam distillation and organic solvent extraction have the disadvantages of low yield and high cost [6], Ultrasonic wave was a kind of mechanical wave. Its mechanical effect and cavitation effect caused cell wall to break, which was conducive to the release and dissolution of active components. It was often used in the auxiliary extraction of natural products. This method had the advantages of simple and quick operation, high extraction rate, fast speed, and no damage to product structure [7]. In This experiment, the ultrasonic-assisted organic solvent extraction method was used to extract volatile oil from *Shixiang*, and the effects of various factors on the yield of volatile oil were investigated.

2. Experiment

2.1 Experimental materials, reagents and instruments

Fresh *Shixiang* was picked in the experimental field of zhoukou normal university in mid-april2016. The experimental reagents were all analytical pure. Equipment:kq-500 ultrasonic cleaner (kunshan ultrasonic instrument co., LTD.). AL204 electronic balance (mettler toledo instrument co., LTD.).

2.2 Extraction of essential oil from Shixiang

Fresh *Shixiang* leaves were baked at 40℃ for 24 hours, crushed and screened. Take 1 gram of *Shixiang* powder (m_{SX}) in a bottle(m_1) , add the solvent, the product was extracted after ultrasonic extraction, and then weighed as (m_2) after solvent recovery. The formula for the yield of *Shixiang* volatile oils:

The yield of *Shixiang* volatile oils: = $(m_1-m_2)/m_{SX}\times 100\ \%$

2.3 Single-factor experiment

The effects of three factors, such as particle size (20, 40, 60, 80, 100 mesh), ultrasonic extraction time (10, 15, 20, 25, 30 min), and material liquid ratio [1:10, 1:20, 1:30, 1:40, 1:40, 1:50 (g:mL)], on the extraction rate of essential oil from *Shixiang* were investigated.

2.4 Orthogonal experimental design

On the basis of single factor experiment, the orthogonal experiment of $L_9(3^3)$ was designed to optimize the experimental process.

3. Results and analysis

3.1 Single-factor experiment

3.1.1 Effect of particle size on the yield of volatile oil from Shixiang

Under the condition of ultrasonic extraction time of 20 min, ethanol: ether in solvent of 1:1, material-liquid ratio of 1:20 (g: mL), the influence of raw material particle size on extraction results was shown in Fig. 1. The curve showed an upward trend first and

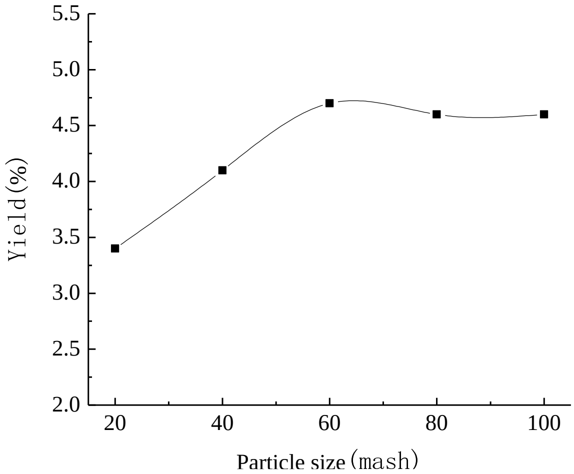

Fig. 1 Effect of particle size on the yield of volatile oil from Shixiang

then a downward trend with the increase of the number of raw materials. When the number of raw materials was about 60 mesh, the yield of volatile oil was the highest, and then decreased slightly with the decrease of the particle size of raw materials. This was due to the fact that the relative surface area of the raw material increases with the decrease of the particle size of the raw material, the raw material and the solvent can be fully contacted, which was conducive to the dissolution of the effective material, and the yield of the target product will be greatly increased. However, when the particle size of raw materials was too small, the accumulation density was large, permeability was poor, easy to sedimentation and consolidation, which was not conducive to the dissolution of the target product, thus reducing the yield of Shixiang essential oil.

3.1.2 Effect of ultrasonic extraction time on the yield of volatile oil from Shixiang

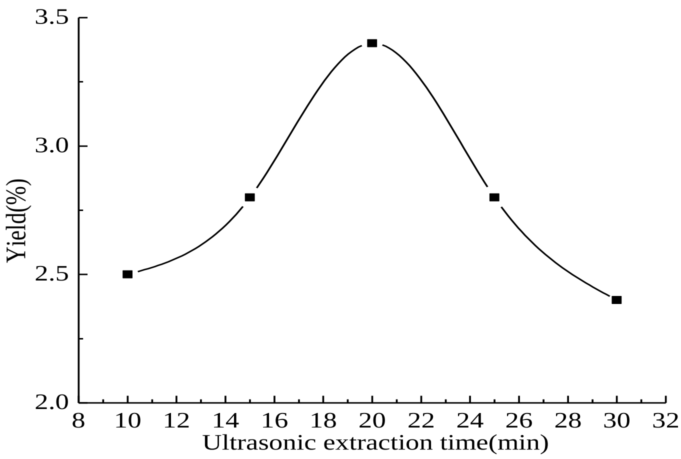

Fig. 2 Effect of ultrasonic extraction time on the yield of volatile oil from Shixiang

The effect of ultrasonic extraction time on the extraction results was shown in Fig. 2. From Fig. 2, it can be seen that the yield of volatile oil increased with the increase of ultrasonic extraction time at first, and reached the highest at about 20 minutes, then decreased with the increase of ultrasonic extraction time. This was because with the increase of the extraction time of ultrasound, the cells were fully ruptured and effective substances were dissolved more frequently. However, the continued extension of ultrasound time, the increase of impurities in the solution, the increase of solution viscosity and the increase of mass transfer resistance were not conducive to the dissolution of effective components, On the other hand, with the increase of ultrasound time, the temperature of solution increased and some volatile oil lost. Therefore, under certain conditions, the highest yield of essential oil could be obtained by ultrasonic time of 20 min.

3.1.3 Effect of material liquid ratio on the yield of volatile oil from Shixiang

The effect of different material liquid ratio on the yield of volatile oil was shown in Fig. 3. From the fig. 3, It can be seen that the yield of volatile oil increases with the increase of the material liquid ratio. When the ratio of material to liquid reached 1:40(g/mL), the yield of volatile oil did not increase obviously. From the perspective of cost saving, the proportion of 1:40(g/mL) was selected for subsequent experiments.

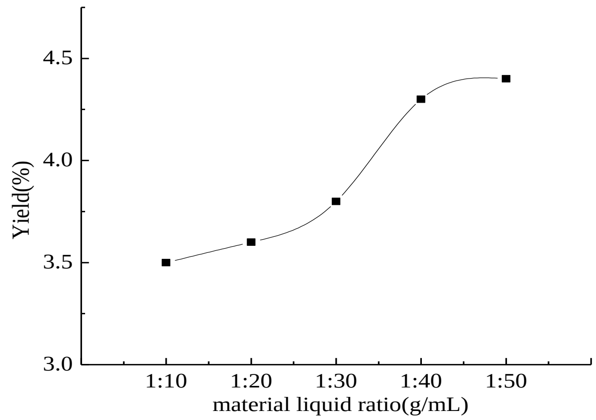

Fig. 3 Effect of material liquid ratio on the yield of volatile oil from Shixiang

3.2 Orthogonal experimental

The results of orthogonal test and range analysis were shown in Table 1.

From table 1, it can be seen that the order of the influence of the three factors on the yield was A> B > C, that was, the particle size of the raw material had the largest

Table 1 $L_9(3^3)$ Orthogonal experimental results and range analysis

NO.	A Particle size (mash)	B Ultrasonic extraction time (min)	C Material liquid ratio (g:mL)	Average yield (%)
1	1（60）	1（15）	1（1:30）	5.6
2	1（60）	2（20）	2（1:40）	5.7
3	1（60）	3（25）	3（1:50）	6.9
4	2（80）	1（15）	1（1:30）	6.4
5	2（80）	2（20）	2（1:40）	6.9
6	2（80）	3（25）	3（1:50）	7.9
7	3（100）	1（15）	1（1:30）	8.2
8	3（100）	2（20）	2（1:40）	8.9
9	3（100）	3（25）	3（1:50）	8.6
K1	6.067	6.733	7.467	
K2	7.067	7.167	6.900	
K3	8.567	7.800	7.333	

Range	2.500	1.067	0.567	
Factor	A>B>C			
The optimal scheme	A_3B_3 C_1			

influence on the yield, followed by the ultrasonic extraction time, and the material liquid ratio had the least influence. The optimal extraction process was composed of $A_3B_3C_1$, with particle size of 100 meshes, extraction time of 25 min and material-liquid ratio of 1:30 (g:mL). The experiment was repeated for 3 times under this condition, and the average yield was 8.95%.

4. Conclusion

In this study, the ultrasonic-assisted extraction process of volatile oil from *Shixiang* was investigated. The results showed that the optimum extraction process was obtained when the particle size of *Shixiang* powder was 100 mesh, the ultrasonic time was 25 minutes, and the ratio of *Shixiang* to ethanol solvent was 1:30 (g:mL). The yield of volatile oil from *Shixiang* was about 8.95%, this result was higher than other test combination. To sum up, the ultrasonic-assisted extraction process of volatile oil from *Shixiang* was rapid, efficient, simple, easy to operate, solvent-saving, economical and feasible.

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