Experimental study on lowering oleic acid value and peroxide value of camellia sinensis by scraping film molecular distillation

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Abstract. Camellia oil is a kind of natural product with high added value. In the process of production, processing, and transportation, acidic substances and peroxides that reduce the quality of oil are produced. In this paper, it was refined by scraping membrane molecular distillation device. The factors affecting the molecular distillation results were analyzed and studied through a single factor experiment and orthogonal experiment. On the surface of the experiment, the optimal process parameters were as follows: the temperature was 190℃, the pressure was 1 Pa, the scraping speed was 300r/min, and the feed amount was 900g/h. Under this condition, the acid value in camellia oil decreased from 2.67mgKOH/g to 0.19mgKOH/g, and the free fatty acid removal rate was 92.88%. The peroxide value decreased from 16.78mmol/kg to 1.03mmol/kg, and the peroxide removal rate was 93.86%. The experimental results showed that the surface of the scraping membrane molecular distillation equipment could meet the national standard of acid value and peroxide value under the appropriate technological parameters, and there was no solvent residue, which was a safe physical purification process.

1 Introduction

Scraping film molecular distillation is used to separate materials according to the difference of molecular activity coefficient of different components under the non-equilibrium state of mixture under high vacuum conditions. Under scraper rotor rotation, the material form a thin and uniform on the surface of the evaporation of liquid membrane, and the materials are heated to boiling point. Relying on the theory of molecular thermal motion, the molecular thermal motion of different free paths is intensified under this condition, so that the light molecules escape from the liquid surface of the raw material and condense on the condensation surface, thus completing the separation process of light and light components[1-3]. Scraping membrane molecular distillation technology protects the material activity of original materials on the premise of ensuring polar loss of materials[4-5].

Camellia oil is extracted and pressed from the seeds of the camellia tree, a unique edible oil in south China. As a precious oil with high added value, camellia oil has high medicinal and edible value. It is used in the production of luxury products such as health care products and cosmetics [6-8]. However, the free fatty acids contained in camellia oil can degrade the quality of camellia oil and make it difficult to store. Too long storage time of camellia oil will increase the peroxide content in camellia oil, aggravating the degree of rancidity. To improve the quality of camellia oil, make full use of the resources of camellia oil, based on experiment and purifying of camellia oil, this paper discusses using the blown film type of molecular distillation technology of camellia oil purity to increase at the same time reduce generated during the production of free fatty acid content, with the most gentle way to reduce the damage to the original oil natural ingredients.

2 Experimental and Results

2.1 Materials and instrument

The raw camellia oil was provided by Guangdong Kangdi Green Biotechnology Co Ltd (Guangzhou, China). The acid and peroxide value is 2.67 mgKOH/g and 16.78 mmol/kg, and the boiling point is 230 ℃. Other test reagents are 0.1 mol/L KOH and 0.01 mol/L $Na_2S_2O_3$ standard solution, 1% phenolphthalein indicator, petroleum ether, 95% ethanol, 30ml mixture of chloroform and glacial acetic acid (volume ratio 2:3) and 1ml KI saturated solution were provided by Guangzhou Chemical Regent Co, Ltd., China.

The experiment equipment which is shown in Fig.1 is a VKL70-5FDRR type short-path distillation device of full-heating full-gear pump produced by VTA.

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Fig. 1. The equipment of short-path distillation

2.2 The influence of the scraper speed

When the speed of the film scraper varied from 150 r/min to 400 r/min, the influence of film scraping speed on the acid and peroxide value is shown in Fig.2. When the speed of the scraper decreased, the rate of liquid film renewal decreased, resulting in the decrease of the evaporation rate of light components, which was not conducive to the distillation of free acid. With the increase of rotation speed and turbulent energy, the liquid film became thinner; heat and mass transfer accelerated; the removal of light components increased; the corresponding acid and peroxide values also decreased, so the best speed range was 300 r/min to 325 r/min.

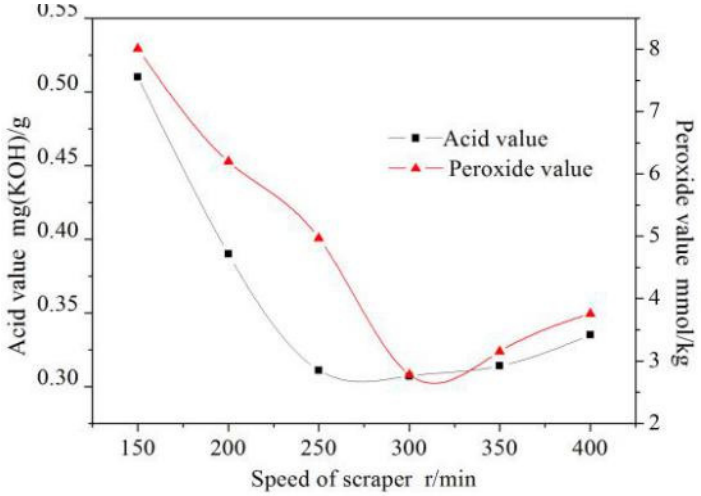

Fig. 2. Variation curves of acid and peroxide value with the scraper speed

2.3 The effect of the feed speed

As shown in Fig.3, when the feed speed increased from 500 g/h to 1000 g/h, both of the acid value and peroxide value were on the rise. With the increase of feed speed, the acid value and peroxide value were increased. The more light components did not reach the separation condition, the thicker and shorter the liquid film and residence time became. The rate of feed was supposed to 900 g/h by taking the factors into account, such as light component yield, quality, and treatment volume.

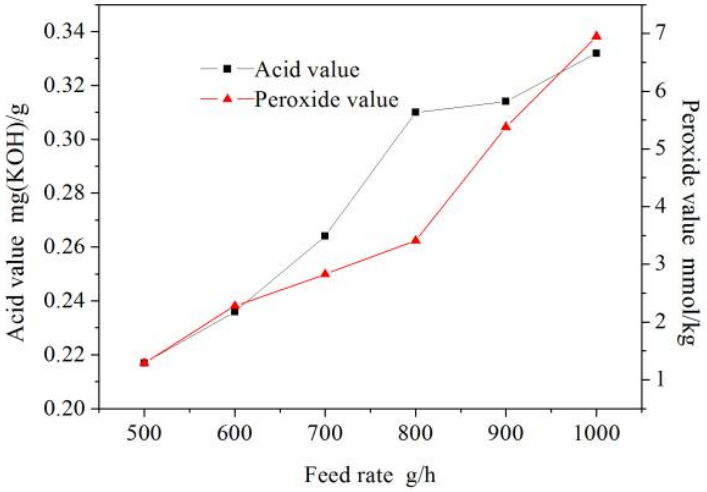

Fig.3. Variation curves of acid and peroxide value with the feed speed

2.4 The influence of temperature

The boiling point of camellia oil is 230℃. The higher temperature can not only destroy the effective components of camellia oil [9-10], but also cause the evaporation of the heavy components. In a certain range, the higher the evaporation surface temperature is, the higher the distillation efficiency is.

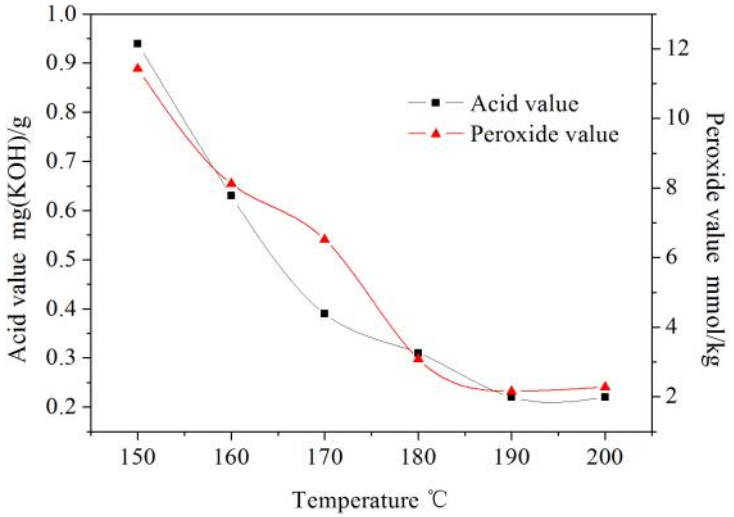

Fig.4. Change of acid valence and peroxide value with temperature

The change of the acid value and peroxide value with the temperature of evaporation is shown in Fig.4. As the temperature rose from 150℃ to 200℃, the acid and peroxide value dropped, and they fell most rapidly at the range of 150℃ to 180℃. When the temperature arrived at 190℃, the change of acid and peroxide value were not pronounced and trended to be stable, And the active ingredients in camellia oil can be protected from being destroyed.

2.5 The effect of pressure

The lower the system vacuum is, the higher separation efficiency will be obtained. But the lower the vacuum degree is, the more energy will be consumed. At low pressure, light molecules did less work overcoming molecular forces in the liquid membrane, so the possibility of escape of light components was greatly increased. Besides, from the perspective of molecular dynamics, the mean free path of molecules increases as the pressure decreases. The initial kinetic energy was large, so the average free path of movement increased, which make it quickly reach the condensation surface [11].

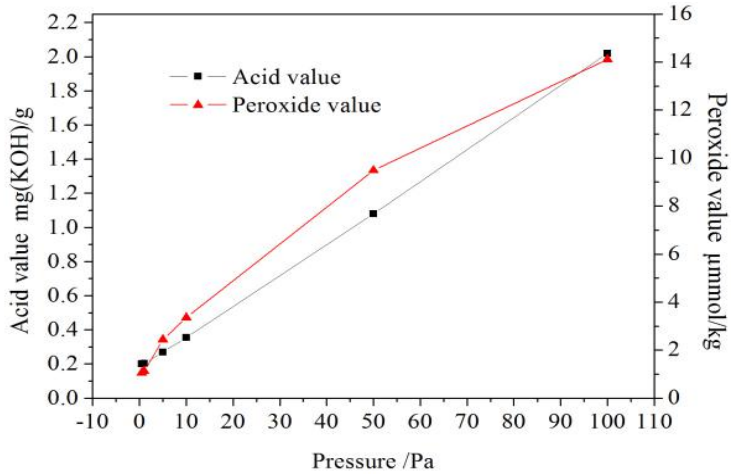

Fig.5. Curves of acid valence and peroxide value with pressure

The condition of pressure was changed, and the other conditions remained the same. It is shown in Fig.5 that the acid and peroxide values dropped rapidly with the pressure declined. When the pressure was 100 Pa, the acid value was 2.02 mgKOH/g, and the peroxide value was 14.11 mmol/kg, which had a not obvious downward trend compared with the raw oil. When the system pressure dropped to 1.0 Pa, the acid value was 0.203 mgKOH/g, and the peroxide value was 1.13 mmol/kg, which could reach the requirements.

3 Conclusion

The experimental data and the quality requirements of the product are analyzed, and the factors of energy consumption are considered. When the process parameters are 190 ℃, 1.0 Pa, 300 r/min, and 900 g/h, the acid value will decrease from 2.67 to 0.19 mg KOH/g, which reduces 92.88%, and the peroxide value decreases from 16.78 mmol/kg to 1.03 mmol/kg, which reduces 93.86%. Compared with the traditional camellia oil refining process (pressing method and leaching method), short-path distillation has greatly improved the oil yield of tea seeds without going through the steps of

degumming, deacidification, decolorization, fractionation, and deodorization. There are no organic solvents and other chemical residues in the distillate, which protects the ingredients of camellia oil to the greatest extent and the quality of camellia oil can be effectively improved.

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