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Optimizing refining temperatures to reduce the loss of essential fatty acids and bioactive compounds in tea seed oil

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ABSTRACT

The traditional refining process for tea seed oil (*Camellia oleifera* oil) results in a severe loss of natural bioactive compounds and unsaturated fatty acids owing to the high operating temperatures. This study investigated the relationship between refining temperatures and the loss of various fatty acids and bioactive compounds in tea seed oil. It was found that the optimal refining temperatures should be 35 °C in the degumming stage, 45 °C in the neutralization stage, 85 °C in the bleaching stage, 150 °C at a pressure of 0.3 MPa in the deodorization stage and 7 °C in the dewaxing stage. Results from a factory acceptance test showed that the quality of the oil refined using the optimized temperatures was similar to that of oil refined traditionally. Most unsaturated fatty acids and bioactive compounds were retained in oil refined using the optimized method, but were significantly reduced in oil refined traditionally.

1. Introduction

Over recent years, seed oil from the tea tree (*Camellia oleifera*), mainly grown in subtropical Asia (China, India, Sri Lanka, Indonesia and Japan), has become increasingly favored by East Asian consumers. Approximately 15% of Chinese people use tea seed oil as their primary cooking oil (Ma et al., 2011; AAFC, 2012). Global production of tea seed oil reached almost 200,000 tons in 2011 (Ma et al., 2011). Many studies have shown that tea seed oil has health benefits that prevent many diseases and illnesses in humans (Lee et al., 2007; Tang et al., 2008).

The composition of tea seed oil resembles that of olive oil. It is rich in unsaturated fatty acids, especially oleic (~70%) and linoleic (~10%) acids, as well as vitamins and minerals (Ma et al., 2011). Furthermore, tea seed oil contains sterols, squalene and tocopherols (Ma et al., 2011), which have been characterized as health promoting (Sahari et al., 2004; Lee and Yen, 2006; Zhong et al., 2007). In addition, tea seed oil has been used as an antioxidant to extend the shelf life of other oils (Sahari et al., 2004; Fazel et al., 2008).

Crude tea seed oil is unpalatable owing to its bitter taste and the presence of phospholipids, so it must be refined to produce oil for human consumption. Currently, the traditional refining method for common vegetable oils is widely used for tea seed oil refining. This method comprises several steps: degumming, neutralization, bleaching, deodorization and dewaxing. This method uses high temperatures at every

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step: degumming at 75–80 °C, neutralization at 80–90 °C, bleaching at 110–130 °C under a vacuum of 0.4 kPa, and deodorization at 240–260 °C. The use of high temperature guarantees the removal of impurities from the oil, but unfortunately it also removes most of the bioactive compounds, especially at the deodorization step, which uses a temperature more than 240 °C. Thus, determining the most suitable temperatures would bring significant benefits to tea seed oil refining.

The objective of this study was to find a set of optimized temperatures for tea seed oil refining. Taken together, our results will indicate that the contents of several essential fatty acids and bioactive compounds in the tea seed oil refined through an optimized refining method will be much higher than those in tea seed oil refined traditionally.

2. Materials and methods

2.1. Samples

Tea seeds (*C. oleifera* Abel var. Changeling) were collected from Kaihua Farm in Zhejiang Province, China, then cold-pressed with the oil being filtered through fine cheesecloth by the Health-Coming Camellia Oil Ltd. (Changshan, China). For the laboratory refining experiments, 12 oil samples (1 kg each) were refined consecutively at the processes of degumming, neutralization, bleaching, deodorization and dewaxing. Each refining step was performed at four temperature treatments, with three replicates for each treatment. At each refining step, 100 g of oil was sampled for quality analysis and the remainder subjected to the next step. For the factory refining experiments, two separate samples of 1000 kg of crude oil were refined: the first by the traditional refining method (degumming at 75 °C, neutralization at 85 °C, bleaching at 120 °C, deodorization at 250 °C and dewaxing at 4 °C); the second by the optimized refining method (degumming at 35 °C, neutralization at 45 °C, bleaching at 85 °C, deodorization at 150 °C and dewaxing at 7 °C). Three replicates were made for each treatment.

2.2. Oil refining procedures

2.2.1. Degummed oil

Degumming reaction was performed in a degumming vessel (LDZX-1002, LZY Lab Instruments, Shanghai, China). Twelve sets of crude oil samples were degummed at four temperatures: 25, 35, 45 and 75 °C, with three replicates at each temperature. Deionized water heated at 25, 35, 45 and 75 °C (2%, w/w), was added to each sample, then stirred for 15 min to remove phospholipids and held for 3 h before filtration. The degummed ratio (%) was calculated using the formula:

$$\text{Degummed ratio (\%)} = \frac{\text{Phospholipid content of crude oil} - \text{Phospholipid content of degummed oil}}{\text{Phospholipid content of crude oil}} \times 100\%$$

100 mL of 12 samples of the different degumming temperatures (with 3 duplicates) were prepared for chemical analysis. To determine "the best" degumming temperature, the phospholipid content of degummed oil should be lower than that of CNS-TSO, and the contents of the major fatty acids and beneficial bioactive compounds should be not significantly different when compared to the other three treated temperatures.

For preparing the samples at the next refining step, another 14.4 kg of crude oil was degummed at the selected degumming temperature. The degummed oil was separated to 12 sets. Each set of oil was around 1 kg.

2.2.2. Neutralization

Neutralization reaction was performed in a liquid–liquid centrifugal contactor (LDZX-1201, LZY Lab Instruments, Shanghai, China). Twelve sets of oil degummed at 35 °C were obtained using the method described above. Because the theoretically calculated amount of caustic lye was not sufficient for obtaining an optimal result, an excess of the stoichiometric quantity of caustic lye was required. Therefore 3.1 g of 16 Be sodium hydroxide solution (Guide Chemicals, Shanghai, China) with 0.1% (w/w) excess was added to neutralize the free fatty acids. After 30 min of slow stirring, the oil was washed with 15% (w/w) of deionized water heated to four different temperatures of 25, 45, 65 and 85 °C (three replicates for each temperature), and then the resulting soapstock fragments were separated by centrifugation. The mixture was then dried to remove any trace of water from the oil. The neutralization ratio (%) was calculated using the formula:

$$\text{Neutralization ratio (\%)} = \frac{\text{Acid value of crude oil} - \text{Acid value of neutralized oil}}{\text{Acid value of crude oil}} \times 100\%$$

100 mL of 12 samples of the different neutralization temperatures (with 3 duplicates) were prepared for chemical analysis. To determine "the best" neutralization temperature, the phospholipid content of neutralization oil should be lower than that of CNS-TSO, and the contents of the major fatty acids and beneficial bioactive compounds should be not significantly different when compared to the other three neutralization temperatures.

For preparing the samples at the next refining step, another 14.4 kg of crude oil was refined at the selected temperatures of degumming at 35 °C and neutralization at 45 °C. The neutralization oil was separated to 12 sets. Each set of oil was around 1 kg.

2.2.3. Bleached oil

Twelve sets of oil neutralization at 45 °C were prepared using the method described above. Bleaching was performed in a bleacher vessel (conjunction of a vacuum dryer, LDZX-756, LZY Lab Instruments, Shanghai, China). Each sample of neutralization oil was bleached by adding 1.2% (w/w) of acid-activated bentonite (Sigma–Aldrich) at 80 °C with slow stirring. The temperature was then adjusted to 65, 85, 105 and 125 °C and stirred for 60 min to remove chlorophylloid and other pigments. The slurry was filtered after cooling to 60 °C, and then the absorbance of the treated oil sample was measured at 520 nm using a spectrophotometer (721, Shanghai, China). The bentonite acid activation was performed according to the method of Önal and Sarıkaya (2012). The bleached ratio (%) was calculated using the formula:

$$\text{Bleached ratio (\%)} = \frac{\text{Absorbance before decoloration} - \text{Absorbance after decoloration}}{\text{Absorbance before decoloration}} \times 100\%$$