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Extraction and quality analysis of volatile oils from onions by coupling pilot and laboratory equipment based on multi-rectification

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A B S T R A C T

A technology based on multi-rectification coupling pilot and laboratory equipment was applied to prepare volatile oils from red or pink onions, which were cultivated in Shandong and Gansu province of China. In the present work, golden yellow, clear and sticky onion volatile oil with the yield of 0.033 mL/kg was prepared by rectifying the distillates collected from the pilot device two times in the laboratory device. GC–MS analysis of the onion oils indicated that the content of dipropyl trisulfide was the highest and changed from 10% to 21% with onion types and cultivation areas. Various kinds of sulfides occupied 53–62.1% of the oils. And the content of the sulfide compounds in the dry substances of onions was 0.244–0.306 g/kg. Red skin onion from Shandong province of China and red and pink skin onion from Jiuquan, Gansu province of China have the same components, only a minor discrepancy in relative content.

1. Introduction

Onion (*Allium cepa* L.) is biannual herb and has abundant nutrients. It can improve appetite and activate digestion because of its pungent flavor constituents stimulating the secretion of gastric juice and digestive gland. Volatile sulfides in onion bulbs and leaves possess medical and health care value and have a lot of effects on influenza virus, chill-proof, sterilization, anticancer and hypoglycem [1–7]. In addition, the volatile constituents of onions are widely applied as food additives. However, it is very difficult to obtain onion volatile oils because of its very low content (0.04–0.05% of fresh weight) [8]. Steam distillation [9], solvent extraction, simultaneous distillation extraction (SDE) [10,11] and supercritical carbon dioxide extraction [12,13] were reported to extract volatile oils.

Steam distillation expends huge energy in commercial process. The yield of volatile oils (VO) is inaccurate due to the residual solvents caused by solvent extraction. SDE method may decrease the volume of organic solvent but it needs to be improved so that it can be used in industry. Supercritical fluid carbon dioxide extraction (SFCE) successfully obtained onion volatile oils of rich sulfide flavorings [13]. However the sophisticated equipment, critical operation conditions and expensive operation cost limited the industrial application of SFCE in the production of onion volatile oils.

Our previous research [14,15] showed that rectification is obviously superior to steam distillation in extracting volatile oils from plants with lower VO, such as Rhizoma Chuanxiong (0.5%) and Bupleurum (0.054%). Multi-rectification is wildly used in chemical industry to increase separation efficiency [16]. In the present study, multi-rectification was applied to extract volatile oils from onions of different varieties and growing areas. The products were also analyzed by GC–MS to understand the composition of prepared onion volatile oils.

2. Experiment and principle

2.1. Material, reagent and equipment

Red skin onion cultivated in Shandong province of China, red and pink skin onion, which were cultivated in Jiuquan, Gansu province of China, were collected to prepare onion oils. NaCl, $CHCl_3$ (purchased from Sinopharm Chemical Reagent Co., Ltd. China). CH_3OH (Merk company, Germany). A small scale laboratory device [14] and a pilot equipment (see Fig. 1) based on rectification for preparing VO were designed and made by our research team.

2.2. Principle and equipment of multi-rectification

Rectification is equivalent to multi-distillation, its separation efficiency depends on the height of rectification column, i.e., the number of theoretical plates. The principle of extracting volatile oil and the small scale laboratory extraction device based on recti-

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fication were introduced in our previous work [14,15] for reference (the number of theoretical plates of the laboratory device is 7). Because the content of onion volatile oils is very low and the maximum capacity of the small scale laboratory device is only 300 g, it is not able to obtain onion volatile oils with such small device even by multi-rectification. Limited by the height of our laboratory, the theoretical plate number of the pilot equipment is only 8 and not big enough to extract onion volatile oils by once rectification although it can process 15 kg materials per batch. Since the extraction time and lost VO with the small scale device are less than the pilot equipment, which can process larger amount materials, it is better to prepare VO from onions by combining the pilot and small scale device. Firstly the pilot equipment was applied to extract onion to get initial distillate, then initial distillates were put into the small scale device to enrich onion volatile oils by multi-rectification.

The schematic diagrams of the small scale device please see the references [14,15]. The profile map of the pilot equipment for preparing VO was illustrated in Fig. 1. It was mainly made of stainless steel 316L, the principal parts were connected with flanges and spacers etc. Electric steam generator was used to heat the equipment with the power of 3000 W and provide 0.2–0.5 MPa of steam. The generated steam comes into the interlayer between the bottom and the sidewall of the extracting tank, which had independent switches, respectively. The temperature and pressure of the extracting tank were monitored by temperature controller and pressure controller. The inner diameter, height, volume and handling capacity of the extracting tank were 300 mm, 700 mm, 40 L and 1–15 kg, respectively. Rectification column was 1000 mm tall, 80 mm diameter and filled with θ ring (φ = 2.5 mm) fillers coated with asbestos outsides. The height and diameter of the cylindrical condenser were 450 mm and 20 mm, respectively. Pipe bundle condenser was installed inside the cylindrical condenser in order to increase condensation area. The number of theoretical plates of the pilot device was calculated to be 8 with graphic method and Fenske method [17].

2.3. GC–MS conditions

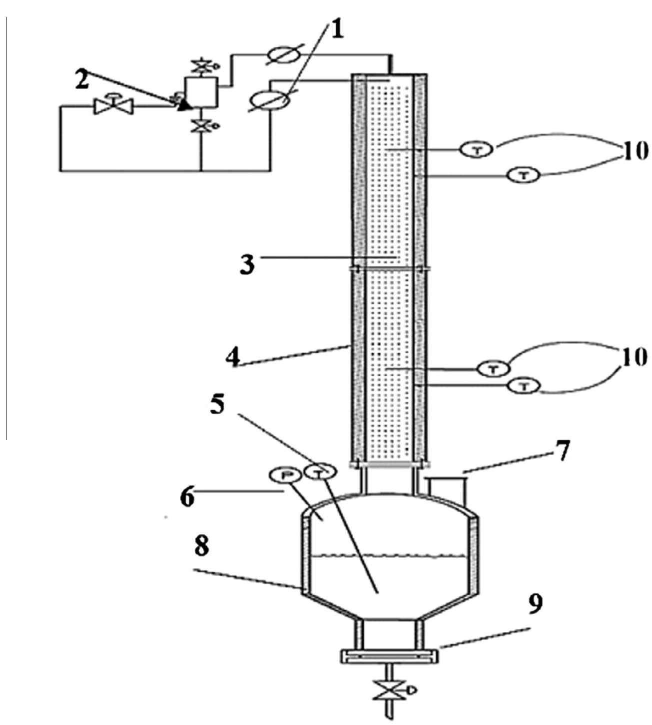
GC–MS instrument: Clarus 500 (Perkin Elmer, USA). EI ion source with the temperature of 250 °C and electron energy of 70 eV. Helium was used as carrier gas at a constant flow of 1 ml/min. a HP-5 MS capillary column (30 m × 0.25 mm i.d.) coated with 0.25 μm film 5% phenyl methyl siloxane was used for separation, and MS search V.2.0 (2) software was used to analyze volatile oil components.

The column temperature was maintained at 60 °C for 3 min then programmed at 16 °C/min to 300 °C, kept at 300 °C for 5 min. injection volume: 0.2 μL, splitting ratio:50:1, input temperature: 250 °C; Mass scan range: 33–600 amu

2.4. Preparation of onion volatile oils by coupling the pilot and small scale device of multi-rectification

The procedure for preparing onion volatile oils by coupling the pilot and small scale devices based on multi-rectification was shown in Fig. 2.

Because there is more than 90% water in fresh onions [18], it is not necessary to add water into the extraction tank of the pilot equipment during the extraction process. Crash 15 kg of onions until they become homogenate prior to putting them into the extraction tank. This process took 2.5 h. Then rectify the onion homogenate and collect distillates from the pilot device one after another. As the small scale device accommodates 1500 ml distillate at most, gather the distillate of 1500 ml per batch, which was called as the first distillate and recorded as RL1, until the ultraviolet



1 mechanical reflux pump; 2 oil-water separator; 3 fillers; 4 distillation towers; 5 thermometer of extracting tank; 6 pressure gauge of extracting tank; 7 feed part of extracting tank; 8 extracting tank; 9 discharge gate of extracting tank; 10 temperature control module

Fig. 1. Cutaway chart of the pilot equipment for preparing onion volatile oils.