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A Study of the Effectiveness of Supercritical Fluid Carbon Dioxide in Bitter Almond Oil Extraction

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Abstract

Bitter almond oil, which is rich in aroma and unsaturated fatty acids, has healthful benefits for preventing from cardiovascular diseases. Higher bitter almond oil could be obtained by Soxhlet extraction, but n-hexane remnants are harmful for liver cell. For this purpose, the objective of this research is to investigate how the oil content of bitter almond would be extracted by the supercritical fluid carbon dioxide operated at different temperatures of an indigenous supercritical fluid extractor (ISFE). The operating pressure was 5000 psi, and the ethanol was used as a co-solvent. The results showed that extraction of bitter almond oil was successful using this method in a considerable short period of time. The obtained oil exhibited white color and strong aroma. In addition, the optimal operation conditions of supercritical fluid extraction were obtained through the study. It was found at a temperature of 46℃ and a retention time of 60 min. There was no difference between the oils extracted by ISFE and Soxhlet extraction. Results of this research exhibited that the bitter almond oil could be extracted effectively by supercritical fluid carbon dioxide.
Key words: bitter almond oil, aroma, Soxhlet extraction, supercritical fluid, carbon dioxide

Introduction

Almond oil is a well known example which is used in cosmetics and for medical purposes since many years (Hallabo et al. 1975), but also the fatty acid composition of other Prunus kernels like peach, apricot, plum or cherry has been characterized (Helmy 1990; Femenia et al. 1995; Johansson et al. 1997). The almonds also contain as much as about 50% oil. As one of the most popular vegetable oils, it is rich in mono- and polyunsaturated fatty acids, with oleic and linoleic acids as the major constituents, and contains the naturally occurring Vitamins A, B1, B2, B6 and Vitamin E. This characteristic composition of the almond oil makes it a valuable material for the food industry (Zhang et al. 2009). Depending on variety, they contain approximately 50-150 pMol/g (dry weight basis) of the potentially toxic cyanogenic glycosides amygdalin and prunasin

(Tungel et al., 1990; Brimer et al., 1993). However, recently, interest in amygdalin is gradually increasing due to a derivative of amygdalin, that is, laetrile (vitamin B17), whose use as secondary cancer chemotherapy and antineoplastic agent has been encouraged (Suchard et al., 1998; Tatsuma et al., 2000).

There are many methods of oil extraction with both mechanical and chemical separation processes. Mechanical separation processes lack of efficiency with low yields and chemical processes employ solvents such as hexane, which are harmful environment (Salgın 2008). Steam distillation is commonly used method to extract oil and it’s a simple and easy maintains equipment often use this method in industry. High temperature steam let volatile components come out and cool water can condense volatile components. Finally, the oil and water are separation then we get high concentrations of almond oil. The extraction temperature is too high to affect the oil quality, including oxidative damage, lose their fragrance, which is the largest fatal problem. High-pressure cold pressing method is also one of traditional oil extraction methods; it is the use of physical method. High pressure equipment has simple structure and doesn’t make almond oil heat damage. Although cold press is better than other way but it takes a long time extraction and lack of efficiency. Because it must go through a series of extracts of separation and purification, until almond oil purity. Solvent extraction is the best method of lipid extraction, but it use organic solvents, and produce large amount of solvent wastes. Organic solvent is hazardous and flammable liquid organic solvent; it has potential toxic emissions during extraction, and solvent is costly and high-purity required(Sahena et al. 2009).

Supercritical carbon dioxide extraction is new technology for extraction oil. This method has the advantage of non-burning, non-pollution, non-residue, non-corrosive and there can extract the oil from the natural world, with high purity and low cost. Carbon dioxide (CO_2) is the most commonly used supercritical fluid and it’s non-toxic, non-flammable and is available at low cost with a high degree of purity. Its low critical constants (Tc = 31.2 ℃, Pc = 1070 Psi) allow supercritical operation of thermally labile compounds. After extraction, we reduce pressure at a certain temperature; extract and carbon dioxide will separate. Sometimes we add ethanol to enhance the extraction of polar compound (Salgın 2008; Sanchez *et al*. 2009; Sahena *et al.* 2009; Mezzomo *et al.* 2010; Yilmaz *et al.* 2011; Ozcana *et al.* 2011).

Materials and Methods

Materials

Bitter almond used in this study was purchased from Xing Hao Co., Ltd. (Kaohsiung, Taiwan). Ethanol (95%) was purchased from Taiwan Tobacco and Liquor Corporation

(Pingtung, Taiwan). The CO_2 was purchased from Ching Shang Co., Ltd. (Kaohsiung, Taiwan).

Extraction

An indigenous supercritical fluid extractor (ISFE) was used in this study. A schematic diagram of the extractor was shown in Figure 1.

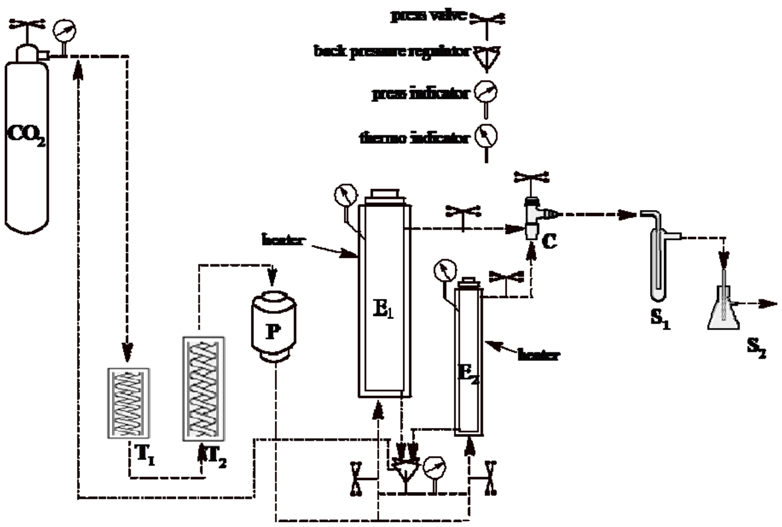

Figure 1 T1:7℃, T2:50℃ temperture control, P: punps, E1: 2L extraction vessel, E2: 0.5L extraction vessel, C: separator control, S1: separator vessel 1, S2: separator vessel 2.

Operation of ISFE

To operate the, semi-batch, indigenous supercritical fluid extractor, first the cooler was set at a temperature of 7 ℃, and a heater was set at a temperature of 50 ℃. The temperature of the cooler was used to control the temperature of the carbon dioxide, so it would be sent out of the gas steel cylinder to the heater for vaporization. The vaporized carbon dioxide would be pressurized and fed into the extraction vessel, where the wet sample volume of 80% had been placed into. Then, the temperature of the extraction vessel was controlled by a cylindrical electrical heater around the vessel. The cylinder contains carbon dioxide was open, and the desired flow rate was adjusted. Then, turn on the high pressure compression motor, and adjust a pressure control to increase the pressure at a slow rate of 2-3 psi/sec.

The experimental method is the use of carbon dioxide and supercritical fluid extraction of ethanol for the work, first of all the pressure slowly compressed pressure 5500 psi, 5500 psi is unknown at this time the best standards set pressure, the pressure from this set go for the best extraction temperature. Because the temperature supercritical conditions of ethanol in 42 ℃, then skip the carbon dioxide, 31.8 ℃, because the effect of ethanol co-solvent effect, 42 ℃ below the temperature will not have to test. Therefore, the temperature experiments started directly from more than 42 ℃, almond oil productions by the experimental conditions, the temperature started increasing from 43 ℃, 47 ℃ in temperature almond oil production began to decrease. Also adjust the pressure to find the maximum extraction of the best barrel production, in which case the fluid within the overall adhesive force and activity to achieve a certain balance. That above the optimal range of temperature and pressure but reduce the total output, because the whole almond oil molecules too large to be separated from carbon dioxide and ethanol co-solvent into a single molecule, single molecule fatty acids have been hundreds of molecules of carbon dioxide and ethanol Surrounded. The original carbon dioxide and ethanol molecules Brown campaign, by the strong collision force becomes floating state. Carbon dioxide and ethanol molecules adsorbed on the fatty acids, it will not necessarily impact the loss of the original direction, must be floating the kinetic energy of the whole molecule. Temperature control in the extraction process is to maintain the power of floating, the temperature is not affected by the extraction of reducing the effect of pressures in the extraction process will be gradually reduced, which is semi-batch supercritical fluid extraction machine features. Less than optimal output conditions, fatty acids with smaller molecular weight of more output products, when compared with the best conditions, this time at the same time extracting the amount of output would be less. Another output is greater than the best conditions, the fatty acids with larger molecular weight of more output products, when compared with the best conditions, when total extraction yield at the same time will be less. The adjustment from 3500 to 5500 psi pressure conditions in the search, the experiment is to test each additional 100 psi as a unit, and eventually found 4500 psi is the largest almond oil output. At this time a total of carbon dioxide and ethanol soluble extract maximum effect, the resulting ethanol extract is white with a very fine aroma of suspended particles occurs, the net for 24 hours after the fine will be white gelatinous precipitate, ethanol extract at this time Contains little aroma. Will be recorded in the literature amygdalin dissolved in ethanol (Lapis Lazuli Light 2011; Tunpl *et al.* 1995), amygdalin extracted by supercritical ethanol, the ethanol concentration in the amygdalin is saturated. Wet in the process of raw materials level is 1/4 to 1/3 ranges, so the more humid the amount of ethanol is more the amount of amygdalin was extracted, residual amount of amygdalin in the almond in the less.

Results and Discussion

Preliminary tests of operation of an indigenous supercritical fluid extractor for bitter almond oil were completed in this study. By subjective judgment on oil obtained, the operating pressure of

ISFE was 5000 psi, when the ethanol was used as a co-solvent. The obtained oil exhibited white color and strong aroma, and shown in Figure 2.

Figure 2 Appearance of extracts of bitter almonds by ISFE

The optimal operation conditions of the indigenous supercritical fluid extractor, a temperature of 46℃ and a retention time of 60 min, were obtained through different tests. There was no difference between the oils extracted by ISFE and Soxhlet extraction. It was found that the bitter almond oil could be extracted effectively by supercritical fluid carbon dioxide.

Absorption ethanol extraction would slowly move the oil of the bitter almonds inside extraction vessel. There was a phenomenon of oil moving gradient shown in Figure 3. If the extraction time and the number of extraction were increased, the color of bitter almonds became a pure white, and the left oil content was very low. For some reason, the defatted bitter almonds were crushed into powder passing through an ASTM #80 sieve. Crude almond powder and then by the second crushing process is shown in Figure 4.

Figure 3 Appearance of bitter almonds taken out of the extraction vessel with a moving oil gradient.

Figure 4 Appearance of the powder crushed with defatted bitter almonds.

The upper pale yellow liquid shown in Figure 5 containing alcohol. The middle layer showed white color with gelatinous texture. It might be alcohol-soluble proteins, although the protein is only soluble in alcohol 50 in 75% ethanol. The white gelatinous extract will be further examined in the future.

Figure 4 Another picture showing different layers of extracts of bitter almonds using ISFE.

Conclusions

Using ethanol as a co-solvent, the extraction of bitter almonds oil by an ISFE was fount successful. The extracts obtained need more experiments to verify its composition. Powder obtained by crushing the defatted bitter almonds was found with fine texture, and would be suitable for commercial usage.

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