



# Oil recovery in rosehip seeds from food plant waste products using supercritical CO<sub>2</sub> extraction



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## ABSTRACT

Valuable oils in rosehip seeds produced as the solid waste during the process of marmalade production using seed-containing fruits were recovered by supercritical CO<sub>2</sub> extraction method. The influence of the particle size ( $125 \leq D_p < 1000 \mu\text{m}$ ), volumetric flow rate of supercritical solvent (0.75–3.5 mL/min), pressure (20–40 MPa), temperature (40–60 °C) and entrainer concentration (2.5–7.5% vol. ethanol) on the extraction yield in the recovery process was examined. The highest extraction yield determined as 16.5 g oil/100 g dry solid was reached in approximately 150 min by using supercritical CO<sub>2</sub> extraction (30 MPa, 40 °C, 0.75 mL/min,  $355 < D_p < 500 \mu\text{m}$ ) and in the case where 5% vol. ethanol is used as entrainer, it was reached in about 90 min. In similar oil profiles obtained through Soxhlet and supercritical fluid extractions, the ratio of unsaturated fatty acids to saturated fatty acids is about 17. Significant changes were determined in morphological structures of waste seeds compared to unprocessed seeds in marmalade production, by using scanning electron microscope. Apparent solubility values of rosehip oil in supercritical CO<sub>2</sub> did exhibit a significant consistency with Chrastil, del Valle–Aguilera, Adachi–Lu and Sparks solubility models.

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## 1. Introduction

Considering the phytochemical profile and biological potentials, the rosehip fruit has excellent nutrient characteristics. The rosehip fruit, which is one of the richest fruits in nature especially in terms of Vitamin C, contains valuable biological compounds such as tocopherols, phenolics, carotenoids, sugars, organic acids and essential fatty acids [1–3]. Some studies have demonstrated that this fruit exhibits a wide range of bioactivities, such as anti-inflammatory [4–6], antioxidant [7–9], antiproliferative [10,11], anti-obesity and anti-diabetic activity [12]. In this context, the pericarp parts of rosehip fruit are commonly consumed as herbal tea, fruit juice, jelly, jam, marmalade and also used as additive particularly in probiotic beverages, yogurts, soups and baby food today. The rosehip seeds constituting about 30%wt. of the fruit appear as a waste product in the industrial production process of foods mentioned above. The oil content of rosehip seeds varies between 5 and 18%wt. and rosehip seed oil (RHSO) consists of a high amount of unsaturated fatty acids such as linoleic acid (36–55%) in particular and linolenic acid (17–27%) and oleic acid (15–22%) [13,14].

Today, there is a special interest on researches on the recovery of bioactive compounds with high added value from vegetable wastes in food processing processes by using good manufacturing practices (GMP). The fact that some constituents of RHSO exhibit antibacterial, anti-inflammatory, antiseptic and antioxidant activity have a potential importance in the development of versatile products particularly for the cosmetic industry. RHSO is an important natural source involved in the production of skin care and anti-aging products since it contains vitamins (C and E), carotenoids (lycopene and  $\beta$ -carotene) and essential fatty acids (omega 3, 6 and 9) [15–17].

Cold pressing is generally preferred in the production of high-quality RHSO [18]. Oil extraction by cold pressing provides significant advantages in preserving many of the bioactive compounds such as essential fatty acids, phenolics, flavonoids and tocopherol in the oils, but it gives a low oil yield. The oil content of seeds at the end of separation process is between 5 and 15%wt. Cold-pressed oils are considered as healthy oils that are important to human nutrition due to their favorable polyunsaturated fatty acid content, notably  $\alpha$ -linolenic acid and linoleic acid [19]. However, the high amounts of polyunsaturated fatty acids in cold-pressed oils are prone to lipid oxidation [19]. For the solution of these bottlenecks, extraction of the oil in rosehip seeds with supercritical CO<sub>2</sub> (scCO<sub>2</sub>) is an alternative separation process for increasing the yield of the separation and protecting biologi-

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cal potentials of valuable biological molecules in the oil. High-tech separation and purification processes using  $\text{scCO}_2$  in the form of “green solvent” as a separation agent are among alternative processes in the development of industrial processes with GMP used today. In order to increase the yield of oil recovery in the separation process, the physical specifications and transport characteristics of  $\text{scCO}_2$  may be modified as desired by variation of the pressure and temperature conditions of the extraction. And also the polarity can be modified with a polar/non-polar entrainer addition [20–23]. When the pressure condition of stream leaving the extractor is reduced to atmospheric conditions, the products obtained may be separated in a single stage.

The extraction process of oil from seeds isolated from dried rosehip fruit using subcritical and supercritical fluids (such as  $\text{CO}_2$  and propane) have been examined by various researchers [24–31]. Some of these studies were performed to determine the efficiency of oil recovery from the seeds milled in different grinder systems. Furthermore, the effects of operation conditions (such as particle size, pressure, temperature and supercritical solvent flow rate) on the extraction yield were investigated. In addition to the apparent solubility of RHSO in  $\text{scCO}_2$ , the effects of operation conditions on the fatty acid compositions were also examined. However, no detailed study on the recovery of rosehip seed oil from food industry waste by  $\text{scCO}_2$  extraction processes was found in literature.

The aim of this study is to recover the oil in the rosehip seeds emerging as a waste product in an organic marmalade production process by using a process with GMP. RHSO in waste seeds were separated by  $\text{scCO}_2$  extraction process. The effects of main process parameters on the extraction yield were investigated. The effects of operation conditions on the initial extraction rate and the apparent solubility of the oil in the solvent under supercritical conditions were also investigated. Furthermore, the compatibility of some semi-empirical solubility models based on pressure and temperature with experimental data were tested. The changes in morphological structures of seeds before and after the separation process were examined using a scanning electron microscope at the microscopic level. Fatty acid profiles of the extracted oil were analyzed by gas chromatography.

## 2. Material and methods

### 2.1. Materials

As the plant material, seeds of *Rosa canina* L. fruits included in the subspecies of Rosaceae family were used. The seeds were supplied from İlay-Öztemiz Tarım Orman Gıda Ltd. Şti. (Şarkışla, Turkey), which is a company producing organic rosehip marmalade. Fruits containing the seeds were harvested in Şarkışla region in October 2014. Rosehip fruits are used as a whole together with its seeds during the process of marmalade production. In this process, the seeds used together with the fruit are subjected to a heating process under a temperature of 72 °C and a vacuum pressure of 0.05 MPa, and appear as a solid waste product. The *n*-hexane (<0.004%  $\text{H}_2\text{O}$ ) and ethanol (<0.01%  $\text{H}_2\text{O}$ ) used as the organic solvent in the extraction process was purchased from Merck KGaA (Darmstadt, Germany) and the liquid  $\text{CO}_2$  was purchased from HABAŞ Company (İzmir, Turkey).

### 2.2. Seed pretreatments

After foreign bodies other than the seeds were removed, the seeds were washed with distilled water, and then dried to a constant weight under atmospheric conditions. Dried seeds were ground in a laboratory scale mill (Polymix PX-MFC 90D Model, Kinematica AG, Switzerland) which was equipped with

a hammer-grinding attachment and a 1.52 mm sieve. Ground seeds were divided into four fractions with particles sizes of  $125 < D_p < 355 \mu\text{m}$ ,  $355 < D_p < 500 \mu\text{m}$ ,  $500 < D_p < 1000 \mu\text{m}$  and  $D_p > 1000 \mu\text{m}$  by using a sieve shaker (Vibratory Sieve Shaker AS 200 control, Retsch, Germany). Milling operation and sieve analysis were made in a cold room at 4 °C. The fractionated samples were stored in airtight amber glass bottles at 4 °C in a fridge up to the time of extraction experiments.

### 2.3. Determination of moisture and volatile component quantity

Moisture and volatile component contents of rosehip seeds were determined according to ISO-659 standard [32] in order to define the extraction yield on dry solid base. Experiments were repeated three times.

### 2.4. Determination of oil content

Oil content in rosehip seeds was determined according to ISO 659 standard [33]. In the experiments conducted with Soxhlet extraction apparatus, about 10 g of triturated rosehip seed was used together with 250 mL *n*-hexane as the organic solvent in an extraction thimble made of cellulose. The organic solvent in the oil solution obtained as a result of the extraction process was removed in a rotary evaporator (Rotavapor R-114 Model, Büchi Labortechnik AG, Switzerland) working at a temperature of 40 °C *in vacuo*. The mass of the oil obtained at the end of this process was determined gravimetrically. Experiments were repeated three times.

### 2.5. Supercritical fluid extraction

Extraction of RHSO with  $\text{scCO}_2$  was performed in an ISCO SFX 220 Model (ISCO Company, USA) supercritical fluid extraction system operated continuously for fluid phase but batch for solid phase (Fig. 1). An extraction column (316–stainless steel) with a volume of 10 mL was loaded with about 3.25 g rosehip seed. There are frits with a pore size of 2  $\mu\text{m}$  at the end parts of the extractor column. Liquid  $\text{CO}_2$  was taken from the  $\text{CO}_2$  tank was loaded to the extraction unit with a high pressure syringe pump (ISCO 100 DX Model, USA). During the process of pressurization, the cooling fluid at 4 °C was continuously circulated through heat exchangers surrounding the piston heads of syringe pumps by means of a cooled circulator (Haake C25P Phoenix II, Thermo Electron Co., USA). Before being fed to the extractor, the pressurized  $\text{CO}_2$  was brought to the desired operating temperature by means of electrical heat exchangers on pipe lines. The extract was brought to atmospheric pressure by passing the flow of extract leaving the extractor through a temperature and flow rate controlled micro valve (restrictor). Thereby, the RHSO isolated from its solvent was accumulated on glass wool inside the product collection unit. The temperature of restrictor was kept around 70 °C depending on the flow rate. In order to determine the change in the amount of extracted oil with the extraction time, the product collection unit was changed at certain operating times (in particular, once in every 5 min during the first 30 min and once in every 10 min during the second 30 min). Extracted oil samples were dried under a nitrogen atmosphere at 40 °C for about 12 h and then, the mass of extracted oil was determined gravimetrically. The second high pressure syringe pump in the system was used to investigate the effect of entrainer concentration. The control unit is able to feed ethanol/ $\text{CO}_2$  at a desired volumetric ratio under a constant pressure of fluids in pumps.  $\text{scCO}_2$  and ethanol at the pump outlet were fed to a micro mixing unit and mixed together therein and the mixture was fed to the extractor. Experiments were repeated 3 times for each condition. The values in figures are arithmetic mean of findings in experiments repeated for each condition.

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