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Supercritical carbon dioxide extraction of quinoa oil: Study of the influence of process parameters on the extraction yield and oil quality

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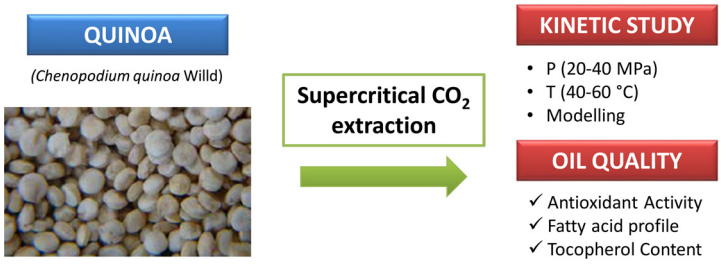
GRAPHICAL ABSTRACT



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ABSTRACT

The supercritical CO_2 extraction of oil from four different quinoa varieties has been studied in this work. For this purpose, the influence of extraction temperature (40–60 °C), pressure (20–40 MPa) and raw material size (250–1000 μm) on the extraction rate has been considered. The extraction rate resulted to be faster the higher the pressure whereas the temperature had less influence on the extraction kinetics. The experimental data were modelled using the Sovova's kinetic model.

The quality of the oil extracted has been evaluated in terms of antioxidant activity (AA), fatty acid profile and tocopherol content. The highest AA was obtained for quinoa oil extracted at 40 MPa and 40 °C; this oil presented high content of polyunsaturated fatty acids (63% of the total) and significant amount of tocopherols (2.5 mg/g oil).

Quinoa oil extracted using CO_2 presented higher antioxidant capacity and tocopherol content than quinoa oil extracted with hexane, regardless the quinoa variety used.

1. Introduction

In recent years there has been a renewed interest in quinoa (*Chenopodium quinoa* Willd) due to its outstanding nutritional properties: it is rich in high quality proteins, vitamins and minerals [1]. Quinoa has been grown in South America for centuries [2], primarily in Bolivia and Peru [3]. This pseudo-cereal has the ability to grow in a wide diversity of environments due to its resistance to weather and soil conditions, unlike other cereals, which reinforces its attractiveness [4].

This renewed interest in quinoa has been fostered by The Food and Agricultural Organization of the United Nations (FAO) after having considered it as *"high nutritive"* due to its high protein content (in the range from 10 to 18%) and high quality oil (in the range from 4.5 to 8.75%) [5]. Moreover, quinoa shows a fatty acid profile rich in unsaturated acids (mainly oleic and linoleic) and a balanced content in aminoacids [6] such as lysine and methionine [1]. The increased

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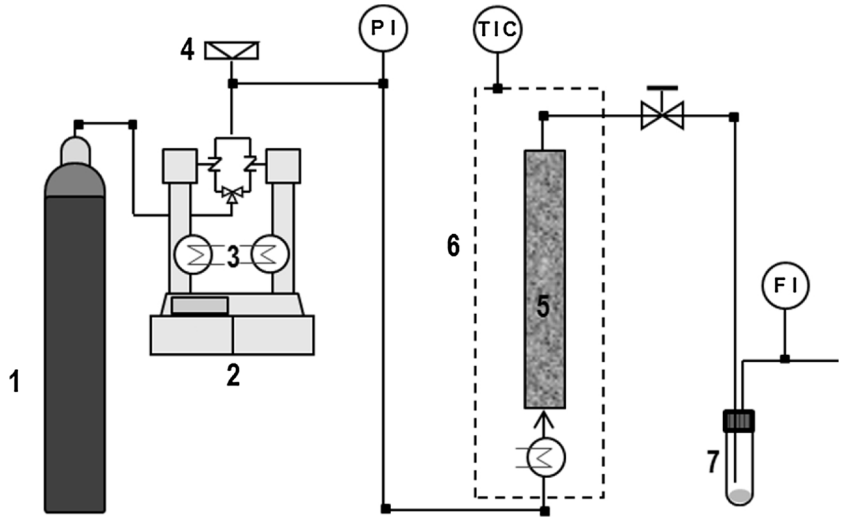

Fig. 1. Supercritical fluid extraction plant. 1: CO_2 reservoir; 2: syringe pump; 3: cryostat; 4: bursting disk; 5: high pressure extractor; 6: oven; 7: separator.

concern that consumers have about food safety and food functionality, willing to try functional and healthier products, is pushing the studying and growing of quinoa in European countries and Japan [7], where there is a great potential for this cultivar. In this sense the presence of essential fatty acids and tocopherols in the oil extracted from quinoa has attracted the attention of the cosmetic, pharmaceutical and food industries [3].

Despite all the excellent properties attributed to quinoa, there is a limited amount of research works about it [2]. The conventional methods to extract oil from seeds are based on the use of organic solvents at relative high temperatures followed by laborious purification procedures. To the best of our knowledge there is a limited number of works dealing with the extraction and characterization of the oil extracted from quinoa [8–10]. Alternatively to the organic solvents, the supercritical fluid extraction is a well-known technology used to extract high added value compounds from many different sources. Supercritical CO_2 (SC-CO_2) is one of the most common supercritical solvents due to its properties: mild critical point (critical temperature 31.7 °C and critical pressure around 7 MPa), non-toxicity, non-explosivity, low price and high selectivity to non-polar molecules such as oils. Its solvent properties can be changed dramatically with small changes in pressure and temperature [11]. SC-CO_2 also separates easily from the extract, once the pressure is released, leaving no traces in the extract. All these properties make SC-CO_2 a potentially successful solvent for the extraction of oil from quinoa [12]. Although it is possible to find in the literature numerous works that successfully deal with the extraction of oil from cereals, such as wheat bran [13], corn [14] or amaranth [15], there is only one work that uses supercritical CO_2 to extract oil from quinoa [3]. Przygoda and Wejnerowska, did not study the extraction of oil from a kinetic point of view: an experimental design was used instead of studying the extraction curves. From that work it is not possible to conclude what mechanism controls the extraction process, whether the internal diffusion, external transport or equilibrium. In that work temperature was varied from 35 to 120 °C, pressure from 8.5 to 28.5 MPa for experiments lasting up to 100 min. The oil yield and the tocopherol concentration were quantified and the effect of the process parameters was studied.

The purpose of this work is to study the influence of several extraction parameters (pressure, temperature and particle size) on the extraction rate of quinoa oil (cv. Titikaka, since it is the most extensively grown in Europe due to its adaptation to the climatic conditions). The Sovovás mathematical model [16] is used to describe the experimental extraction curves. In a second step of the work, the quality and stability of the quinoa oil obtained by SC-CO_2 is analysed and compared to the oil obtained using hexane, in terms of fatty acid profile, tocopherol content and antioxidant activity.

Finally the oil extracted from four quinoa varieties (Pasankalla, Collana and Altiplano and Titikaka) using either hexane or supercritical CO_2 is analysed and compared.

2. Materials and methods

2.1. Raw material

Four different varieties of quinoa (kindly provided by Quinoa Spain Productos Ecológicos S.L. and harvested in Álava (Spain)) were used in this work. The oil content of those varieties, named Titikaka, Altiplano, Collana and Pasankalla, was determined by Soxhlet extraction (Buchi B-8111) using hexane as solvent.

Quinoa seeds were ground in a ball mill (Fritsch) to get different particle sizes in the range from 250 to 1000 μm and study the influence of the particle size on the extractability of the oil by SC-CO_2.

2.2. Supercritical fluid extraction equipment and procedure

The extraction experiments were carried out in a lab-scale plant, whose diagram is shown in Fig. 1.

The maximum specifications of this experimental set-up are 150 °C and 50 MPa. The extractor has a volume of 26.5 mL with ½" internal diameter. In a typical experiment, around 12 g of quinoa were placed in the extractor, which was pressurized with CO_2 (Air Liquide S.A.) up to the extraction pressure. Then, the solvent flowed at the desired pressure and temperature (at a mass rate of 0.14 ± 0.02 kg/h) for the desired time (maximum 3 h). Different combinations of pressure and temperature were tried in order to study their influence on the extraction performance.

2.3. Analytical methods

2.3.1. Determination and quantification of the fatty acids profile

The fatty acids profile was determined by the AOAC official method [17]. According to this method the fatty acid methyl esters were firstly prepared and then analyzed by gas chromatography using a Hewlett Packard (6890N Network GC System) gas chromatograph equipped with an auto-sampler (model 7683B) and a flame ionization detector (FID). Helium was used as a carrier gas at a flow rate equal to 1.8 mL/min. A fused silica capillary column (OmegawaxTM-320, 30 m × 0.32 mm) was used.