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Extraction Kinetics and Composition of Chamomile Flower Extract Obtained by Supercritical CO_2 †

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Abstract

This study aimed to obtain chamomile flower extracts (CFEs) using supercritical CO_2 (200 bar and 40 °C) and analyze their composition by GC-MS. A yield of 2.8 ± 0.3% of CFE was obtained after 122.4 min of extraction. The CFE contained several compounds, the most abundant of which were 4-(4-Hydroxy-2,2,6-trimethyl-7-oxabicyclo [4.1.0]hept-1-yl)butan-2-one (12.9%), (Z)-Tonghaosu (11.8%), 6-hydroxydihydrotheaspirane (11.5%), pentacosane (8.1%), cyclohexanethiol, 2,5-dimethylacetate (5.6%), and tetracontane (5.3%). The SFE process for obtaining CFE compounds is a suitable alternative; however, further studies are needed to evaluate this process and the composition of the extract, especially its most volatile fraction.

Keywords: genus *Matricaria*; volatile compounds; aromatic plant crops; supercritical fluid extraction

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1. Introduction

Chamomile is an aromatic herb that has been studied for its many medicinal properties [1]. The composition profile and biological properties of chamomile may vary depending on its origin of production and variety [2]. Numerous scientific studies have been conducted on chamomile, highlighting its medicinal and therapeutic properties. Some of these properties include its potential to mitigate cancer complications [3], antioxidant activity [4,5], anticancer activity [4,6], anti-inflammatory activity [7], and reduction of dental anxiety in children [8], among other properties [9]. On the other hand, according to the literature review conducted by Ostovar et al. [10], adverse effects are possible due to the use of chamomile. An important finding of this review is that a significant number of studies do not report the characteristics of chamomile products, such as purity. In this

context, we believe that it is necessary to improve the production and characterization of phytochemicals to improve the use of natural products in industry.

The extraction of volatile phytochemicals from chamomile has been performed using conventional technologies, such as hydrodistillation [11]. Kotnik et al. reported other methods, such as steam distillation, maceration, and Soxhlet [12]. Conventional methods have some disadvantages, such as hydrolysis/oxidation of volatile compounds due to operating conditions (extraction time, solvents used, temperatures, etc.) [11]. Various green technologies, such as supercritical fluid extraction, ultrasound-assisted extraction, and microwave-assisted extraction, are available for the recovery of bioactive compounds to improve the yield and preservation of chamomile phytochemicals [13]. Depending on the extraction objective, some emerging techniques may use high temperatures, such as subcritical water extraction for the recovery of phenolic compounds from chamomile, where temperatures ranging from 65 °C to 210 °C were evaluated [14]. Other techniques have been developed to recover temperature-sensitive compounds, such as solvent-free microwave extraction, which has shown better performance and higher essential oil quality compared to the steam distillation method [15]. Supercritical extraction allows extracts with a better composition to be obtained compared with the steam distillation [12] and maceration methods [12]. Supercritical fluid extraction has proven to be an interesting technique for recovering volatile compounds; however, the loss of volatile compounds from the main collection flask was observed during the supercritical extraction process of chamomile flowers using laboratory-scale equipment, probably due to the continuous flow of carbon dioxide [16]. To better understand the losses of volatile compounds from chamomile flowers during supercritical CO_2 extraction, this study aimed to monitor the extraction process and analyze the composition of the chamomile extract.

2. Methods

2.1. Sample Preparation

Chamomile was purchased from a local market in the city of Tarma, Tarma province, Junín region, Peru. The flowers were manually separated and prepared for extraction following the procedure used in our previous work [17].

2.2. Extraction Procedure

A laboratory-scale SCF extraction unit (Singularity Extraction Technologies, Campinas, Brazil) was used for the extraction. The characteristics of the equipment and the CO_2 used in the extraction are described in our previous work [17]. The sample (10.00 ± 0.01 g) was fed into a 100 mL extraction vessel. The extraction was carried out under supercritical conditions of 20 MPa and 40 °C, which allowed for the highest yield in the study by Povh et al. [16]. The solvent flow was set at 5.43 g CO_2/min with a static time of 10 min. During dynamic extraction, the weight of the extract was monitored to construct the overall extraction curve (OEC), up to an extraction time of 122 min and an S (g CO_2)/F (g sample) ratio of 66.

2.3. Composition Analysis

CFE was analyzed by chromatography according to the method developed by Bendif et al. [18]. Details of the laboratory that provided the analysis service, the characteristics of the chromatographic equipment, and some details of the analysis are shown in our previous work [17].

3. Results and Discussion

3.1. Extract Yield and OEC

Figure 1 shows the OEC values of chamomile flowers. The maximum yield achieved during the 2 h of extraction was 2.8 ± 0.3%. In this study, the extract yield was higher than the essential oil yield observed in steam distillation (0.6%) [12] and solvent-free microwave extraction (0.08%) [15]. Kotnik et al. [12] reported a higher yield (3.81%), but with an extraction time more than four times longer than in the present study and a lower CO_2 flow rate (2.5 g/min), which could explain the differences with the results shown in the present study. In another study, a higher yield (4.3%) was obtained when working under the same supercritical conditions as in the present study, but with a CO_2 flow rate of 4.0 g/min and an extraction time five times longer than in the present study [16]. On the other hand, the OEC behavior observed during the first 20 min of extraction (Figure 1) may explain the volatilization of compounds due to solvent flow during continuous extraction. Povh et al. [16] observed the volatilization of chamomile flower compounds during supercritical extraction and adopted a trap to prevent the loss of volatile compounds. In a more recent study, ethanol was used in the collection bottle to prevent the loss of volatile compounds during the supercritical extraction of various plant raw materials [19]. Figure 2A shows a colorless extract during the first few minutes of extraction; part of this extract probably volatilizes as the extraction process progresses. As the extraction process progresses, an amber extract can be observed in the extraction vessel (Figure 2B). The obtained extract has an oily texture and a characteristic chamomile aroma. The results show that the supercritical carbon dioxide extraction process applied to raw materials rich in volatile compounds must be conducted with the volatility of these compounds in mind. The results show an additional challenge to that reported by Dashtian et al. [20] for the commercial production of volatile aromatic compounds using SFE. In another recent study, he successfully evaluated the use of a saline solution as a pretreatment for Hetian rose petals to obtain essential oil with supercritical CO_2 [21]. Finally, the application of supercritical CO_2 for the extraction of volatile aromatic compounds represents a challenge due to the loss of volatile compounds during depressurization in equipment that uses separation vessels and during dynamic extraction in laboratory-scale equipment that only uses an extract collection vessel. The temperature of the separators and the mass flow of the solvent can be studied to determine their effects on the yield and loss of volatile compounds.

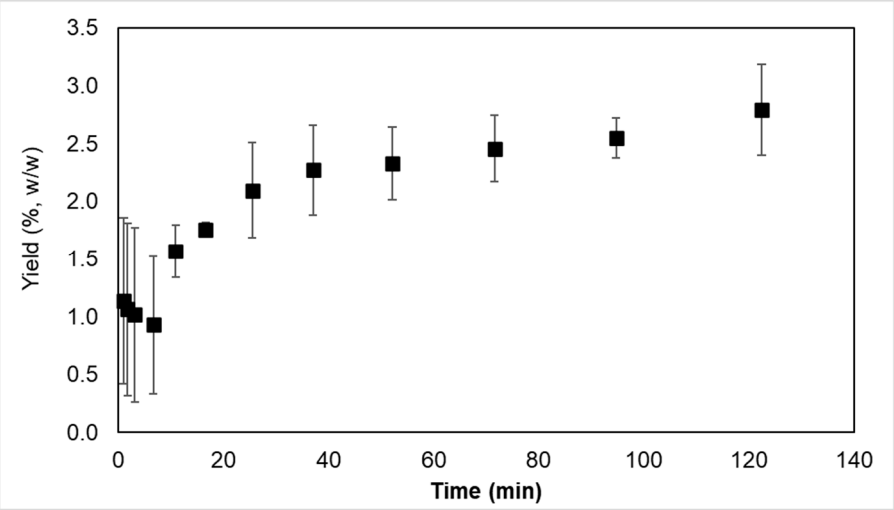

Figure 1. Extraction kinetics at 200 bar, 40 °C, and 65.8 S/F.

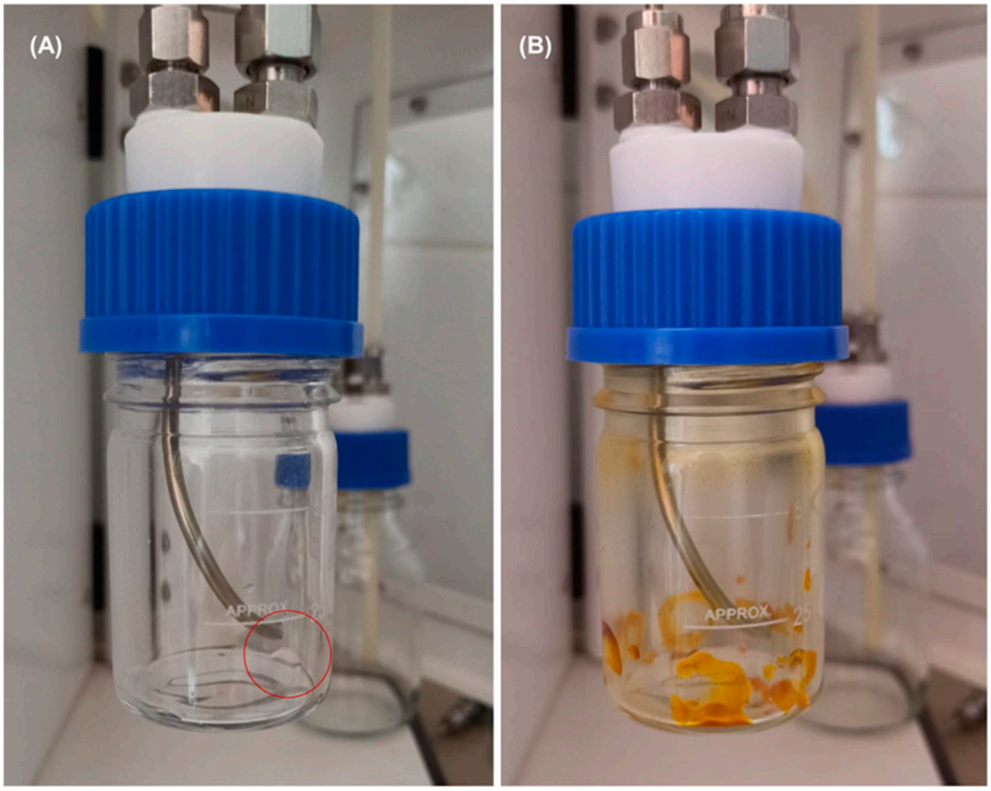

Figure 2. Fraction of colorless (**A**) and orange (**B**) chamomile flower extracts obtained by SFE.

3.2. Composition of the CFE

Figure 3 shows the chromatogram of the identified compounds in the CFE. The CFE contained various chemical components, including 4-(4-Hydroxy-2,2,6-trimethyl-7-oxabicyclo [4.1.0]hept-1-yl)butan-2-one (12.9%), (Z)-Tonghaosu (11.8%), 6-Hydroxydihy drotheaspirane (11.5%), pentacosane (8.1%), cyclohexanethiol, 2,5-dimethyl-, acetate (5.6%), tetracontane (5.3%), 1,6-trimethyl-3-methylene-2-(3,6,9,13-tetramethyl-6-ethenye-10,14-dimethylene-pentadec-4-enyl)cyclohexane (4.9%), hexatriacontane (4.8%), exo-2-Hydroxycineole (3.9%), (E)-beta-Farnesene (3.4%), (E)-Tonghaosu (2.7%), xanthoxylin (2.5%), and other components with an abundance of less than 2%. Among the minor compounds identified were alpha-bisabolol (0.30%), alpha-bisabolol oxide B (0.27%), and 7-methoxycoumarin (0.24%); these compounds were reported in higher concentrations by Rahimi et al. [22]. The composition profile of chamomile flower essential oil reported by Lu et al. [23] shows some similarities with the extract obtained in the present study. In another study, the abundance of (Z)-Tonghaosu was similar to that found in essential oil from shade-dried chamomile flowers and was lower than that found in fresh flowers [24]. The composition of the supercritical extracts reported in this study and the literature consulted can be explained in part by the volatilization of the components of the chamomile flower due to the effect of the CO_2 flow in the collection vessel. The compositions reported by Povh et al. [16], Rahimi et al. [22], and the present study were determined from extracts obtained at mass flows of 6.67×10^{-5} kg/s, 1.16×10^{-4} kg/s, and 9.05×10^{-5} kg/s, respectively. In a study conducted to evaluate the parameters of the SFE process in the yield of carnosic acid and carnosol from sage leaves, the CO_2 flow rate was one of the parameters that significantly influenced the composition of the extracts [25]. In another case, increasing the CO_2 flow rate decreased the extract yield because a higher solvent flow rate results in a shorter residence time of the solvent in the extraction bed [26]. Conversely, in some cases, the solvent flow rate does not significantly affect the yield, as in the case of apple seed oil extraction [27]. The results of this study and previous studies indicate the

need to adequately evaluate supercritical extraction conditions, especially for raw materials rich in volatile compounds.

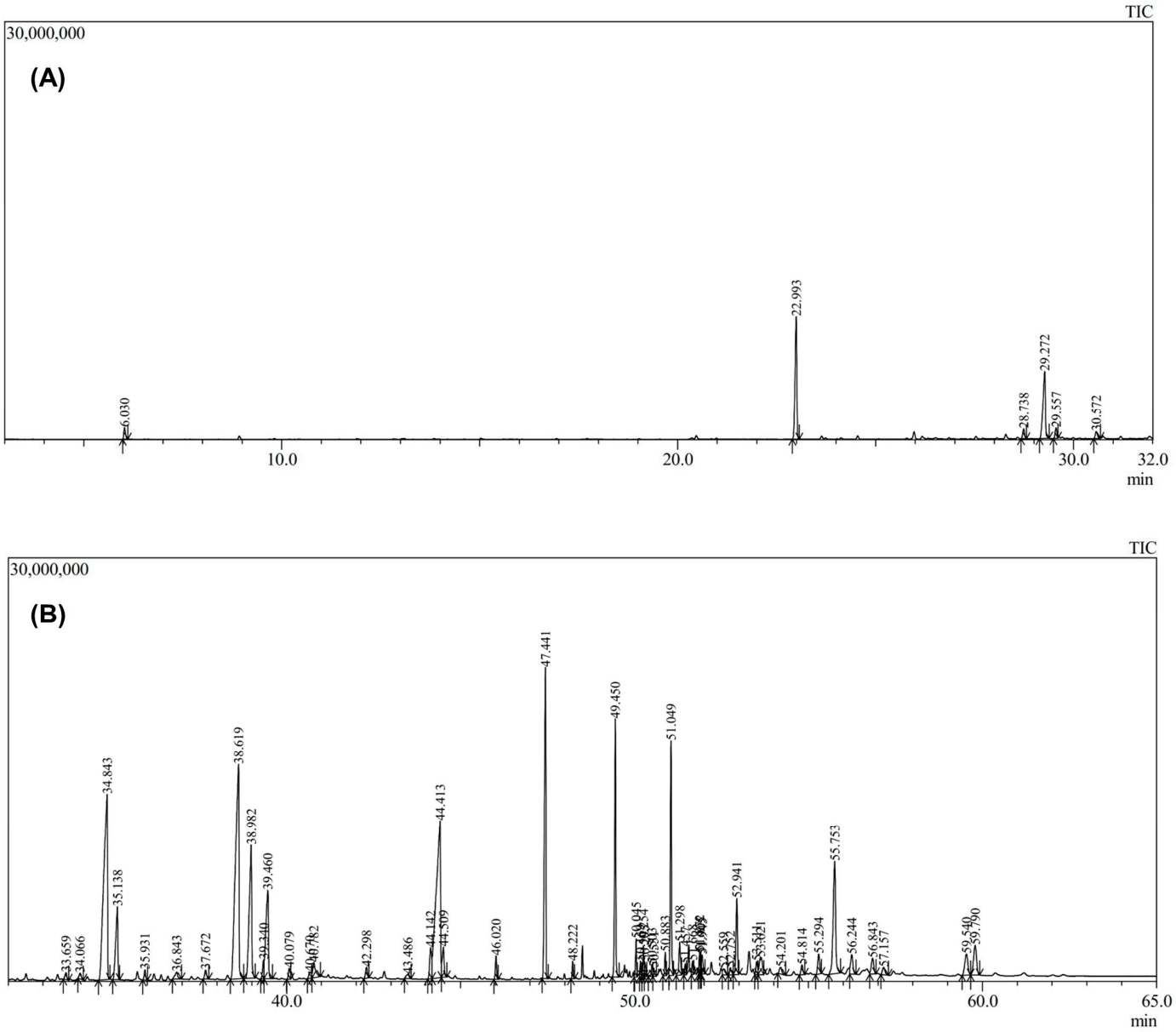

Figure 3. GC-MS chromatograms up to a retention time of 32 min (**A**) and 65 min (**B**) of the SFE extract of chamomile flower.

4. Conclusions

Supercritical CO_2 extraction enables the isolation of chamomile flower extracts rich in chemical compounds. During the extraction process, extract losses were recorded, probably from the most volatile fractions. The results of this study indicate the need for further research into supercritical fluid extraction to improve the recovery of volatile compounds from chamomile and other volatile compound-rich raw materials.

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M.A.A.M. and L.O.C.-P.; Project administration, G.W.Q.S., W.J.C.C. and L.O.C.-P.; Funding acquisition, L.O.C.-P. All authors have read and agreed to the published version of the manuscript.

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