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Research Article

Biochemical Characterization of Coriander Cakes Obtained by Extrusion

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This study was designed to examine the effect of operating conditions such as nozzle diameter on fatty acid, sterol, and tocol composition of coriander cakes. Eight fatty acids were identified, with petroselinic acid accounting for 75–77% of the total fatty acids, followed by linoleic, oleic, and palmitic acids, accounting for 12-13%, 5%, and 3%, respectively, of the total fatty acids. β-Sitosterol was the major sterol in all oils with 33–35% of total sterols. The next major sterols in all oils were stigmasterol (24% of total sterols) and Δ^7-stigmasterol (15% of total sterols). Coriander cake contained higher amounts of total tocotrienol where γ-tocotrienol was the main compound.

1. Introduction

Coriander (*Coriandrum sativum* L.), an annual Apiaceae commonly used as a condiment or a spice in the Mediterranean area, was reported as containing both essential oil (rich in linalool) and vegetal oil (rich in petroselinic acid). All parts of the plant are edible with the fresh leaves and dried seeds most commonly used as culinary ingredients. The fruits, which contain 13–29% vegetable oil [1, 2] and 0.35% essential oil [3], were also used in perfumery, cosmetic, and medicinal applications [4]. A volatile essential oil fraction composed of terpenoid and phenolic phytochemicals that have antioxidant and other medicinal properties were also found in coriander and were transported at least in part into the lipid phase during extraction [2, 4–9]. The main fatty acid constituent in *C. sativum* oil that comprises 31–75% of the fatty acid profile was petroselinic acid, which was uncommon isomer of oleic acid and was found at high levels in a restricted range of seed oils mostly from the Apiaceae family [10].

Numerous researches have been reported on therapeutic properties of coriander [11–15]. Also genetic diversity and chemical variation levels of coriander essential oil have been studied in relation to bioclimatic and geographic location [16–18]. Some other types of research were reported on the case of using different methods for the extraction of coriander components [19, 20].

In general, some work focused on sequenced extractions from coriander fruits respecting the two fractions of interest (vegetal and essential oils), while not penalizing the subsequent valorization of the residual by product. The residue obtained after oil extraction from the seed is called oil cake or oil meal. Recently, oil cakes have been widely used for the production of industrial enzymes, antibiotics, biopesticides, vitamins, and other biochemicals [21]. They have also been commonly used as feed supplement. In the same way, Matthäus and Zubr [22] reported that camelina cake contains remarkable amounts of bioactive substances such as glucosinolates, vitamins, and antioxidants. On the other hand, the value addition of oil cakes, by their utilisation in bioprocesses for the production of industrial bioproducts, was reported [21]. Their application in bioprocesses also offers advantages in bioremediation and biological detoxification of hazardous compounds. Indeed, the effects of extrusion cooking on the polyphenol content and antioxidant activity has been reported in rye bran [23] and in a snack bar composed of chickpea, corn, oat carrot, and hazelnut [24].

Several studies on coriander fruits [2, 3, 25] and leaves, flowers, stems, and roots [26] have been reported recently but

there are no relevant studies on essential oil and antioxidant activity of coriander fruit cake. Therefore, the purpose of the present study was to investigate essential oil of coriander fruit cakes extract fractions and to evaluate their antioxidant activity of methanol cake extracts.

For the first time, the main objectives of this study is set out to evaluate the effects of screw configuration and operating parameters such as nozzle diameter on oil extraction from coriander cakes. Another goal of the work was to determine the effect of nozzle diameter on fatty acid, sterol, and tocol composition from coriander cakes.

2. Materials and Methods

2.1. Plant Material. All trials were carried out using a single batch of coriander fruits obtained from Korba area (North East of Tunisia). In this study, the fruits from coriander were extracted with single screw extruder, the cake samples were immediately collected.

2.2. Extrusion. Extrusion was done by a Single-screw (Model OMEGA 20, France) with a motor (0.75 kW, 230 V of maximal tension, 5.1 A of maximal intensity), a screw length of 18 cm, a pitch screw of 1.8 cm, with an internal diameter of 1.4 cm, a channel depth of 0.5 cm, and a sleeve of 2.5 cm of internal diameter equipped with a filter-pierced outlet for liquid at the end of the screw and at the surface of the nozzles. The filter section was of 2 mm in diameter to separate extracted oil. The feed rate and the screw rotation speed were maintained constant to 15 g/min (0.9 kg/h) and 40 rpm, respectively. Five nozzles of different diameters (5, 6, 7, 8, and 9 mm) were used in the pressing of the coriander seed. The nozzle/screw distance was 3 cm. The screw press was first run for 15 min without seed material but with heating via an electrical resistance-heating ring attached around the press barrel, to raise the screw press barrel temperature to the desired value. Running temperature was adjusted with a thermocouple.

2.3. Cake Oil Extraction and Fatty Acid Methylation. Cakes from coriander fruit were extracted separately with n-hexane in a soxhlet apparatus. The extraction was protected from light. The extract was then filtered and after evaporation of the solvent under reduced pressure and temperature, the oil content was determined. Then, 20 mg of total extracted oil is rendered soluble in 1 mL ter-butyl-methyl ether (TBME). Before analysis by gas chromatography (GC), fatty acids (FAs) were transformed into their corresponding methyl esters (FAMEs) according to the procedure reported by norm NF ISO 5508 using 50 μL trimethylsulfonium hydroxide (TMSH) in methanol [27]. The sample was analyzed in three replications.

2.4. Unsaponifiable and Sterol Extraction. 5 mg of dihydrocholesterol used as internal standard and dissolved in chloroform were added to 140 mg of oil. Then, 3 mL of KOH 1 M in ethanol were added and mixed to each sample and a heating at 75°C was maintained during 30 min. After cooling at the ambient temperature, 1 mL of distilled water and 6 mL of isohexane were added to the mixtures, followed by a decantation and the recovery of hexane phase was analyzed by GC. All experiments were done in triplicates.

Before GC analysis, samples were silylated by the addition of 1 mL N-methyl-N-trimethylethylsilyl-heptafluorobutyramide (MSHFBA) mixed with 50 μL 1-methyl imidazol and a heating of 5 min at 103°C.

2.5. Gas Chromatography (GC-FID). FAMEs from each sample were analyzed by GC, using a Varian CP-Select CB 3900 flame ionization gas chromatograph, with a fused silica capillary column, CP Select CB (50 m × 0.25 mm, 0.25 μm film thickness). The carrier gas was H_2 with a flow rate of 1.2 mL/min, split ratio was 1 : 100. The initial oven temperature was held at 185°C for 40 min, increased at a rate of 15°C/min to 250°C and then held there for 10 min. The detector and injector temperatures were fixed at 250°C. FAs were identified by comparison of their retention times with those of pure reference standards.

Sterol samples were analysed by GC using a FID-Perkin Elmer chromatograph equipped with a CP-SIL 8CB capillary column (30 m; 0.25 mm; 0.52 μm). The carrier gas was H_2 with a flow rate of 1 mL/min (split-splitless injection was used). Analyses were performed under the following temperature program: isotherm at 160°C during 0.5 min, from 160°C to 260°C at the rate of 20°C/min, 2°C/min up to 300°C, and 45°C/min up to 350°C. Injector and detector temperatures were maintained, respectively, at 340°C and 365°C.

2.6. Tocol Extraction and Analysis. Five grams of cakes of coriander were first ground into a fine powder and combined with 50 mL of hexane. The mixture obtained was centrifuged at 10000 g for 15 min. The organic layer was then recovered and filtered. This procedure was repeated twice. The extract was evaporated first, in a rotary evaporator and then under nitrogen, at room temperature. For determination of tocopherols, a solution of 10 mg oil in 1 mL hexane was directly used for the high-performance liquid chromatography (HPLC) analysis.

Samples were analyzed by high performance liquid chromatography (HPLC) consisting of a pump P680 equipped with a KROMASIL Si-100-S column (Lapeyrouse-Fossat, FR) (5.0 μm, 4.0×250 mm) with fluorometrical detection (Dionex Model RF-2000 Fluorescence Detector, Bretonneux, FR) at 290 and 317 nm of excitation and emission wavelengths, respectively. The mobile phase was isooctane/isopropanol (99.5 : 0.5, v/v) at a flow rate of 1 mL/min. Tocopherols identification was based on the comparison of their retention times with those of standard solutions (Supelco-Sigma) (Figure 1).

2.7. Statistical Analysis. All extractions and determinations were conducted in triplicates and results were expressed on the basis of dry matter weight. Data are expressed as mean ± SD. The means were compared by using the one way and multivariate analysis of variance (ANOVA) followed by Duncan's multiple range tests. The differences between individual means were deemed to be significant at $P \leq 0.05$. All analyses were performed by using the "Statistica v 5.1" software [28].

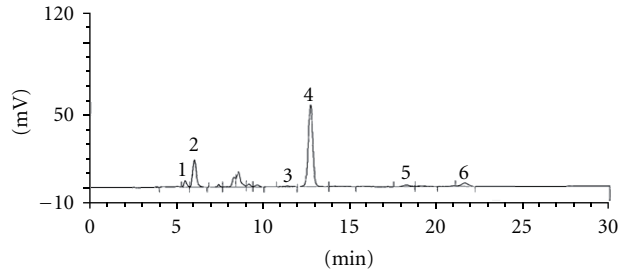

Figure 1: RP-HPLC chromatogram type of *Coriandrum sativum* cake. Signal was collected at 290 nm and 317 nm. Peaks numbers corresponding to: 1: α-tocopherol, 2: α-tocotrienol, 3: γ-tocopherol, 4: γ-tocotrienol, 5: δ-tocopherol, 6: δ-tocotrienol.

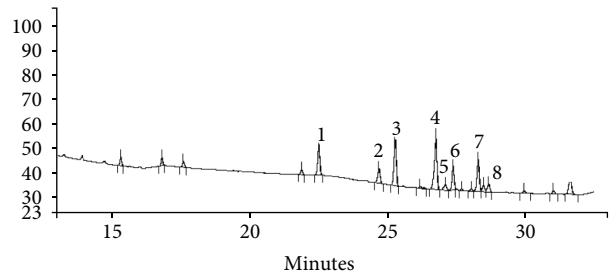

Figure 3: Chromatographic profiles of coriander cake of sterols. Peaks numbers corresponding to: 1: cholestanol, 2: campesterol, 3: stigmasterol, 4: β-sitosterol, 5: Δ^5-avenasterol, 6: Δ^5,24-stigmastadienol, 7: Δ^7-stigmasterol, 8: Δ^7-avenasterol.

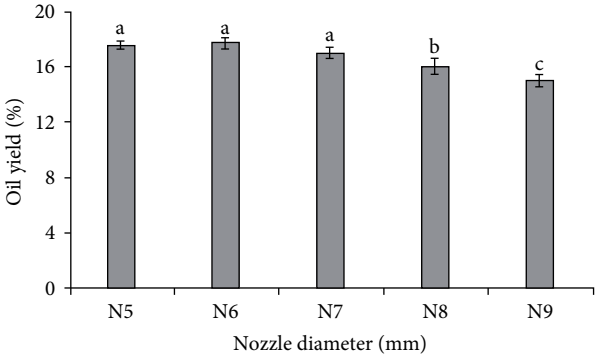

Figure 2: Effects of nozzle diameter on the oil yield (%) of *Coriandrum sativum* cake. Oil yield values with different subscript (a–c) were significantly different at $P < 0.05$ (Duncan test). N: nozzle.

3. Result and Discussion

3.1. Oil Yield. Figure 2 presents the effects of nozzle diameter on the oil yield of coriander cake oil as expressed on the basis of dry weight. Results obtained showed that the yields of the oils were significantly ($P < 0.05$) affected by the diameter of nozzle. The highest oil yield (17%) was obtained by the diameter of the nozzle was 5 mm, 6 mm, and 7 mm. The lowest yield (15.01%) was obtained in the nozzle diameter at 9 mm.

On the other hand, our results showed that oil yields of 5 mm and 6 mm diameter of nozzle were not significantly different ($P < 0.05$). The increase of the empty diameter inside the extruder caused a greater oil loss increase, probably as a consequence of an increase in the operating pressure. Besides, it is worth to highlight the relatively opposite evolution in the oil yield extracted directly of coriander fruit and those extracted of coriander cake [29].

These authors showed that the maximum oil yield was obtained for nozzle diameter 9 mm and reached 15.73%. The reason for extracting oil from the cake was for the efficiency of pressing and achieves the optimum conditions to have the total exhaustion of the fruit.

3.2. Fatty Acid Composition. As can be seen in Table 1, fatty acid composition pointed out the abundance of four fatty acids (petroselinic C18:1, linoleic C18:2, oleic C18:1, and palmitic C16:0) which accounted 98%. The unsaturated fatty acids (C18:1 and C18:2) were more abundant than the saturated ones with the highest rate of the petroselinic varying from 77% to 75% of the total fatty acids (TFA), followed by the linoleic one ranging from 12.47% to 13.61% of TFA. The oleic and palmitic acids were present at a percentage from 5% and 3% of TFA, respectively. These results showed that there were not significant differences among different samples, permitting to deduce that nozzle diameter did not affect fatty acid composition but affected essentially minor compounds of oil such as essential oil (Table 1).

The cakes obtained after extrusion of coriander fruit were characterized by the presence of a high proportion of monounsaturated fatty acids (MUFA) (82% of TFA). Polyunsaturated (PUFA) and saturated fatty acids (SFA) represented 13 and 4% of TFA, respectively. To the best of our knowledge, the effect of nozzle diameter on fatty acid composition is investigated for the first time in coriander oil cake. The ratio of saturated fatty acids to polyunsaturated fatty acids (SFA/PUFA) not varied with different nozzle diameter. However, this ratio was 0.33 (Table 2). These result showed that the cake maintained the same fatty acid composition of coriander fruit [25] and the vegetal oil extracted by single screw extruder [29].

3.3. Sterol Composition. The analysis of the sterols provides rich information about the quality and the identity of the oil investigated and for the detection of oil and mixtures not recognized by their fatty acids profile [30] (Figure 3). This fraction has been considered as the major unsaponifiable fraction in many oils [31]. No significant differences were observed between the total unsaponifiables content of the oil extracted of the cakes (Table 2).

β-Sitosterol (33–35% of total sterol (TS)) represents the main component followed by stigmasterol (24% of TS). The next major components were Δ^7-stigmasterol (15% of TS) and Δ^5,24-stigmastadienol (10% of TS). Campesterol was present at a level of 8% of TS, whereas Δ^7-avenasterol and Δ^5-avenasterol were detected in lower amounts with approximately 4 and 2% of TS, respectively. The composition of sterol content in the oil cake was also compared with oil extraction

Table 1: Effect of nozzle diameter on fatty acid composition (%) of coriander cake.

Fatty acid	Nozzle diameter (mm)				
	5	6	7	8	9
C16:0	3.68 ± 0.01[a]	3.81 ± 0.02[a]	3.66 ± 0.03[a]	3.68 ± 0.00[a]	3.78 ± 0.02[a]
C16:1(n-7)	0.23 ± 0.00[b]	0.24 ± 0.00[a]	0.24 ± 0.01[a]	0.24 ± 0.00[a]	0.25 ± 0.00[a]
C18:0	0.89 ± 0.02[a]	0.82 ± 0.04[a]	0.73 ± 0.00[b]	0.72 ± 0.01[b]	0.73 ± 0.01[b]
C18:1(n-12)	77.28 ± 0.36[a]	76.57 ± 0.59[a]	76.84 ± 0.09[a]	76.80 ± 0.14[a]	75.54 ± 0.02[b]
C18:1(n-9)	5.25 ± 0.24[a]	5.22 ± 0.56[a]	5.16 ± 0.05[a]	5.15 ± 0.13[a]	5.86 ± 0.01[a]
C18:2(n-6)	12.47 ± 0.11[b]	13.12 ± 0.01[a]	13.17 ± 0.01[a]	13.19 ± 0.01[a]	13.61 ± 0.00[a]
C20:0	0.09 ± 0.00[a]	0.09 ± 0.00[a]	0.08 ± 0.00[a]	0.09 ± 0.00[a]	0.09 ± 0.00[a]
C18:3(n-3)	0.12 ± 0.00[b]	0.12 ± 0.01[b]	0.12 ± 0.01[b]	0.13 ± 0.00[ab]	0.14 ± 0.00[a]
SFA	4.66 ± 0.02[a]	4.72 ± 0.05[a]	4.47 ± 0.03[a]	4.49 ± 0.01[a]	4.60 ± 0.03[a]
MUFA	82.75 ± 0.13[a]	82.04 ± 0.04[a]	82.24 ± 0.03[a]	82.19 ± 0.01[a]	81.65 ± 0.02[b]
PUFA	12.59 ± 0.10[a]	13.24 ± 0.01[a]	13.29 ± 0.00[a]	13.32 ± 0.00[a]	13.75 ± 0.00[a]
SFA/PUFA	0.37	0.35	0.33	0.33	0.33

C14:0: myristic acid; C16:0: palmitic acid; C16:1(n-7): palmitoleic acid; C18:0: stearic acid; C18:1(n-12): petroselinic acid; C18:1(n-9): oleic acid; C18:2(n-6): linoleic acid; C20:0: arachidic acid; C18:3(n-3): α-linolenic acid. SFA: saturated fatty acids; MUFA: monounsaturated fatty acids; PUFA: polyunsaturated fatty acid. Data are expressed as mean ± SD of three replicates. Values with different superscripts (a,b) are significantly different at $P < 0.05$.

Table 2: Variation of sterol composition (mg/100 g oil) in different screw configurations.

	Nozzle diameter (mm)				
	5	6	7	8	9
Campesterol	8.76 ± 0.21[a]	8.14 ± 0.11[a]	8.74 ± 0.81[a]	8.58 ± 0.32[a]	8.36 ± 0.04[a]
Stigmasterol	24.12 ± 0.52[a]	24.34 ± 0.73[a]	24.70 ± 0.27[a]	25.18 ± 0.56[a]	24.38 ± 0.09[a]
β-Sitosterol	33.71 ± 0.60[b]	34.61 ± 0.09[a]	35.22 ± 0.78[a]	33.29 ± 0.85[b]	35.78 ± 0.22[a]
Δ^5-Avenasterol	2.79 ± 0.39[a]	2.32 ± 0.09[a]	2.62 ± 0.05[a]	2.56 ± 0.12[a]	2.62 ± 0.04[a]
Δ^5,24-Stigmastadienol	12.23 ± 0.11[a]	10.52 ± 0.76[b]	9.67 ± 0.23[c]	10.37 ± 0.30[b]	9.48 ± 0.49[c]
Δ^7-Stigmasterol	14.42 ± 0.09[a]	15.31 ± 0.08[a]	14.72 ± 0.09[a]	15.52 ± 0.32[a]	15.42 ± 0.45[a]
Δ^7-Avenasterol	3.97 ± 0.02[b]	4.75 ± 0.01[a]	4.34 ± 0.02[a]	4.50 ± 0.06[a]	3.95 ± 0.05[b]

Data were means ± SD of three replicates. Values with different superscripts (a–c) are significantly different at $P < 0.05$.

Table 3: Tocopherol and tocotrienol content (mg/100 g oil) of coriander cake.

	Nozzle diameter (mm)		
	7	8	9
α-Tocopherol	3.44 ± 0.10[a]	2.45 ± 0.01[b]	3.72 ± 0.21[a]
α-Tocotrienol	19.77 ± 1.31[a]	12.29 ± 1.60[b]	19.61 ± 0.65[a]
γ-Tocopherol	1.37 ± 0.06[b]	2.72 ± 0.07[a]	1.29 ± 0.10[b]
γ-Tocotrienol	71.51 ± 2.05[b]	76.42 ± 1.09[a]	71.11 ± 1.55[b]
δ-Tocopherol	0.92 ± 0.02[c]	1.54 ± 0.05[a]	1.10 ± 0.03[b]
δ-Tocotrienol	2.99 ± 0.11[c]	4.58 ± 0.22[a]	3.16 ± 0.11[b]

Data are expressed as mean ± SD of three replicates. Values with different superscripts (a–c) are significantly different at $P < 0.05$.

using single screw extruder [29]. The comparison was carried out to the most influential mechanic press and to check the quality of the processed oil product. The result showed that the coriander oil cake maintained similar composition of the vegetal oil extracted by single screw extruder which β-sitosterol was the major compounds [29].

3.4. Tocol Composition. The compositions of tocols in coriander cake analyzed using HPLC are summarized in Table 3. Coriander cake contained higher amounts of total tocotrienol where γ-tocotrienol was the main compound. The percentage of this main compound increased when nozzle diameter at 8 mm (76.42%), whereas it decreased significantly when nozzle diameter with 7 mm and 9 mm (71%). The second main compound, α-tocotrienol, decreased under the increased the nozzle diameter with 7 mm and 9 mm; its lowest percentage is detected in the trial of 8 mm of nozzle diameter (12.29%), and the highest percentage is unregistered at 7 mm with 19.77%. As for δ-tocotrienol, its highest percentage was obtained in the trial of nozzle diameter 8 mm and reached 4.58%. The major tocopherol was α-tocopherol, the maximum percentage detected under nozzle diameter 9 mm. The percentage of γ-tocopherol increased significantly whenever the nozzle diameter was 8 mm and reached 2.72%. The rates of δ-tocopherol when increased the nozzle diameter increased 7 mm to 9 mm and reached 1.10%.

4. Conclusion

This study revealed coriander cake as a source of natural bioactive compounds which could be attractive to the food or pharmaceutical industry. Indeed, use of oil cakes offers good alternative to traditional applications by their exploitation

in the production of environmentally friendly green biofuel. Another key point to be noted is that the bioprocess utilizing oil cakes is attractive due to relatively cheaper availability of the oil cakes throughout the year, making it even more favourable when economics is considered.

Conflict of Interests

The authors declared no conflict of interests.

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