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# Extraction of Coriander Oil Using Twin-Screw Extrusion: Feasibility Study and Potential Press Cake Applications

E. Uitterhaegen<sup>1,2,3</sup> · Q. H. Nguyen<sup>1,2</sup> · K. A. Sampaio<sup>3</sup> · C. V. Stevens<sup>3</sup> · O. Merah<sup>1,2</sup> · T. Talou<sup>1,2</sup> · L. Rigal<sup>1,2</sup> · Ph. Evon<sup>1,2</sup>

**Abstract** This study presents an assessment of the vegetable oil extraction from coriander fruits through mechanical pressing, more specifically twin-screw extrusion. This comprises an evaluation of the oil recovery obtained and its respective quality, as well as the specific mechanical energy, representing an economical point of view. With regard to the extrusion optimization, the screw configuration, the device's filling coefficient and the pressing temperature were varied. The screw configuration was shown to exhibit a key influence on the extraction efficiency and oil recoveries of at least 40 % were reached when the pressing zone was positioned immediately after the filter and consisted of 50 mm long, reverse screws with a -33 mm pitch. Furthermore, with a device's filling coefficient of 39.4 g/h rpm and a pressing temperature of 120 °C, an oil recovery of 47 %, the highest of this study, was reached with concurrent low energy consumption. Next to this, operating parameters of 47.1 g/h rpm and 80 °C resulted in the production of a press cake with the lowest residual oil content (15 %) in this study, although this also involved a significant increase in the filtrate's foot content. All the

produced oils were of acceptable quality (<1.5 % acidity), showed high petroselinic acid content (73 %), and were pleasantly scented.

**Keywords** Twin-screw extruder · Coriander · Vegetable oil extraction · Oil quality · Press cake · Essential oil · Antioxidant capacity

## Introduction

Coriander (*Coriandrum sativum* L.) is an annual herb, native to the eastern Mediterranean and then spread to India, China and the rest of the world. It is commonly used as a condiment or a spice. The fruit has been used as a traditional medicine to treat various medical conditions such as indigestion, worms, rheumatism and joint pain. Indeed, coriander has been shown to exhibit a wide range of biological activities including alterative, antibilious, antispasmodic, aphrodisiac, appetite stimulant, aromatic, carminative, diaphoretic, diuretic, refrigerant, stimulant, stomachic and tonic [1]. The coriander fruits are particularly interesting as they contain both a vegetable oil and an essential oil fraction.

The composition of coriander fruits depends on several factors such as the growing region and maturity stages [2]. Previous research mainly focused on the fruit oil fatty acid composition, triacylglycerols and glycerophospholipids [3, 4], tocopherols and tocotrienols [5], or effects on plasma lipids [6]. The main fatty acid constituent in *Coriandrum sativum* vegetable oil is petroselinic (6Z-octadecenoic) acid, representing between 31 and 75 % of the fatty acid profile. Petroselinic acid is an uncommon isomer of oleic acid and is found in high levels in a restricted range of seed oils, mostly from the Apiaceae family [7]. It presents

✉ Ph. Evon  
Philippe.Evon@ensiacet.fr

<sup>1</sup> Laboratoire de Chimie Agro-industrielle, ENSIACET, Université de Toulouse, INP, 4 Allée Emile Monso, BP 44362, 31030 Toulouse Cedex 4, France

<sup>2</sup> INRA, Laboratoire de Chimie Agro-industrielle, 31030 Toulouse Cedex 4, France

<sup>3</sup> SynBioC, Department of Sustainable Organic Chemistry and Technology, Ghent University, Coupure Links 653, 9000 Ghent, Belgium

a unique oleochemical with high potential for food, cosmetic and pharmaceutical industries and may further allow the synthesis of a large number of interesting platform molecules.

Moreover, vegetable oil from coriander fruits has recently been labeled as a novel food ingredient (NFI) by the European Food Safety Authority [8]. It is now considered as safe to be used as a food supplement for healthy adults, at a maximum level of 600 mg per day (i.e., 8.6 mg/kg bw per day for a 70 kg person). This will lead to significantly higher intakes of coriander fruit oil and petroselinic acid than current background intakes. Therefore, the development of a new process for extracting vegetable oil from coriander fruits is a major challenge for the years to come.

The extraction of vegetable oil on a commercial scale is usually achieved through the application of a two-step process. As a first step, seeds with considerable oil content are pre-pressed by the use of a hydraulic or expeller press, while the second step comprises solvent extraction of the press cake to obtain sufficiently low residual oil contents of typically less than 1 %. However, the continuous extraction of vegetable oil using extrusion technology as a sole method is becoming of increasing importance [9–13]. This commonly involves a single-screw extruder of variable pitch and channel depth, rotating in a cage type barrel [9]. As friction between the material and the screw or barrel surface is a key factor during single-screw extrusion, this process often leads to high energy consumption, overheating and subsequent oil deterioration. Apart from this, complementary special equipment such as breaker bars is often necessary in order to avoid insufficient mixing.

Twin-screw extrusion could provide a solution to these issues, as it exerts a stronger transportation force and enhances mixing and crushing of the seeds during extrusion. In addition, twin-screw extruders have been shown to be significantly more energy efficient [9, 14]. Recently, twin-screw extrusion has been increasingly applied to achieve efficient vegetable oil extraction from various oil seeds [9, 14–23]. Therefore, it was applied for the extraction of vegetable oil from coriander fruits, while aiming to obtain a high level of oil quality, extraction efficiency and feasibility.

Only one previous study has dealt with the use of twin-screw extrusion technology for vegetable oil extraction from coriander fruits by mechanical pressing [24]. The single batch of fruits used in this study was cultivated in the Korba area (North East of Tunisia) and exhibited a relatively low lipid content (only 21.9 % of the dry matter). Both the screw rotation speed and the inlet flow rate of coriander fruits affected the oil extraction. The highest oil recovery was obtained under operating conditions of 50 rpm and 2.3 kg/h, respectively. Nevertheless, it was never more than 45 % and the residual oil content in the

press cakes was at least 16.6 % of the dry matter. At the same time, the filtrate's foot content, i.e., the solid particles forced through the filter, was always high (from 47.5 to 66.0 %). Further, essential oil contents in the press cakes and their composition have not been the subject of any earlier studies although they may present a valuable application of extrusion cakes. The impact of operating conditions on the fatty acid composition of pressed oils was less important. Ten fatty acids were identified, with petroselinic acid accounting for 66–75 %. In conclusion, the use of twin-screw extrusion technology for the mechanical pressing of coriander oil appears promising, even if the process efficiency should be improved.

This study comprises an evaluation of the impact of several operating conditions on the twin-screw extrusion process. These include the screw configuration, the device's filling coefficient, which represents the ratio of the inlet flow rate of coriander fruits to the screw rotation speed, and the pressing temperature. Further, some potential applications for the press cakes obtained are suggested.

## Experimental Procedures

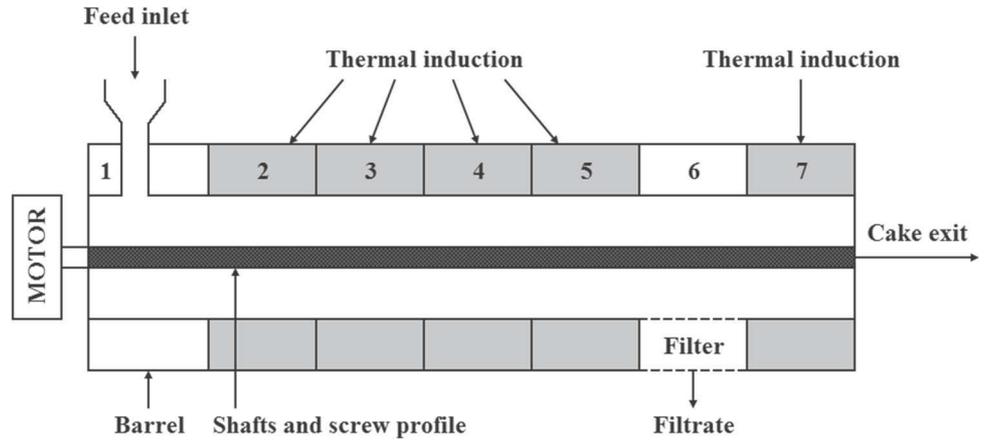
### Materials

All trials were carried out using a single batch (200 kg) of coriander fruits (GSN maintenaire variety), cultivated in the South West part of France and supplied by GSN Semences (Le Houga, France). The moisture content of the coriander fruits was  $9.77 \pm 0.10$  % (French standard NF V 03–903) [25]. All solvents and chemicals were of analytical grade and were obtained from Merck (Germany), Macherey–Nagel (Germany), Sigma-Aldrich (USA) and Prolabo (France).

### Twin-Screw Extruder

Twin-screw extrusion was carried out by the use of a Clextral BC 21 (France) co-rotating and co-penetrating twin-screw extruder, comprising two identical, intermeshing screws. The extruder was composed of seven modular barrels, each 100 mm long (Fig. 1), while the screws consisted of several segmental screw elements that were either 25 or 50 mm long. The temperature of modules 2, 3, 4, 5 and 7 was controlled and adjusted through electrical heating and water cooling. Coriander fruits were fed into the extruder inlet port using a volumetric screw feeder (K-Tron Soder KCL-KT20, Switzerland). Through the application of different screw types, different extrusion zones may be created which subject the fruits to a series of operations. In module 1, the forward pitch screws exert a conveying force, while a succession of 10 monolobe and 10 bilobe paddles

**Fig. 1** Schematic modular barrel of the Clextral BC 21 twin-screw extruder used for extraction of vegetable oil from coriander fruits



	1		2		3		4		5		6		7				
Profile 1	T2F 50	T2F 50	C2F 33	C2F 25	DM 10×10 (45°)	C2F 33	C2F 25	BB 10×10 (90°)	C2F 33	C2F 33	C2F 25	C2F 25	C2F 16	C2F 16	CF2C -25	C2F 25	C2F 33
Profile 2	T2F 50	T2F 50	C2F 33	C2F 25	DM 10×10 (45°)	C2F 33	C2F 25	BB 10×10 (90°)	C2F 33	C2F 33	C2F 25	C2F 25	C2F 16	C2F 16	CF2C -25		C2F 33
Profile 3	T2F 50	T2F 50	C2F 33	C2F 25	DM 10×10 (45°)	C2F 33	C2F 25	BB 10×10 (90°)	C2F 33	C2F 33	C2F 25	C2F 25	C2F 16	C2F 16	CF2C -33		C2F 33
Profile 4	T2F 50	T2F 50	C2F 33	C2F 25	DM 10×10 (45°)	C2F 33	C2F 25	BB 10×10 (90°)	C2F 33	C2F 33	C2F 25	C2F 25	C2F 16	C2F 16	CF2C -33	C2F 25	C2F 33

**Fig. 2** Screw configurations for extraction of vegetable oil from coriander fruits. *T2F* trapezoidal double-thread screw; *C2F* conveying double-thread screw; *DM* monolobe paddle-screw; *BB* bilobe paddle-

screw; *CF2C* reverse screw. The numbers following the type of screw indicate the pitch of T2F, C2F, and CF2C screws and the length of the DM and BB screws

ensures thorough trituration in modules 2–4. The fruits are further conveyed in modules 5 and 6, while module 7 represents a pressing zone made up of reverse pitch screws. The filtrate was collected at the filter section in module 6, which is composed of four hemispherical dishes with perforations of 500  $\mu\text{m}$  diameter. A control panel allowed the continuous monitoring of the screw rotation speed ( $S_s$ ), the fruit feed rate ( $Q_s$ ) and the barrel temperature ( $\theta_c$ ).

The screw configurations that were applied during this study (Fig. 2) were based upon those used in Evon *et al.* [22]

for jatropha oil extraction. Variations concerned the pressing zone of the extruder, situated in module 7. The reverse pitch screws used in profiles 1 and 2 were 50 mm long, with a pitch of  $-25$  mm. They were positioned immediately after the filtration module for profile 1, and 25 mm from the end of module 6 for profile 2. The reverse pitch screws used in profiles 3 and 4 had the same length (50 mm), but their pitch was greater ( $-33$  mm instead of  $-25$  mm). They were positioned 25 mm from the end of module 6 for profile 3 and immediately after the filtration module for profile 4.

## Experimental

Ten trials were conducted for the extraction of vegetable oil from coriander fruits in the twin-screw extruder (Table 1). Different operating conditions were tested including the screw profile, the device's filling coefficient and the temperature in the pressing zone. Except for trial 4, the screw rotation speed was 100 rpm for all the experiments, meaning that the device's filling coefficient directly depended on the feed flow of coriander fruits.

In order to ensure stabilization of the extruder and the operating conditions, extrusion was carried out for 30 min prior to sampling. Then, samples consisting of the filtrate and the press cake were collected for a sufficient period of time (30 min) to minimize any variation of the outlet flow rates and to allow a single sampling for each trial. After collection of the filtrate during extrusion, it was subjected to centrifugation (8000  $\times g$ , 15 min, 20 °C) in order to eliminate the solid residue, i.e., the centrifugation foot.

The oil recoveries were calculated from the following formulas:

$$R_L = \frac{Q_F \times T_L}{Q_S \times L_S} \times 100, \quad (1)$$

where  $R_L$  is the oil recovery relative to the total oil content of the fruits (%),  $Q_S$  the inlet flow rate of coriander fruits (kg/h),  $Q_F$  the flow rate of the filtrate (kg/h),  $T_L$  the mass fraction of oil in the filtrate (%), and  $L_S$  the total oil content of the coriander fruits (%).

$$R_C = \frac{(Q_S \times L_S) - (Q_C \times L_C)}{Q_S \times L_S} \times 100, \quad (2)$$

where  $R_C$  is the oil recovery based on the residual oil content of the press cake (%),  $Q_C$  the flow rate of the press cake (kg/h), and  $L_C$  the oil content of the press cake (%).

Although both  $R_L$  and  $R_C$  are expressed in terms of the oil content of the fruit,  $R_C$  is always higher than  $R_L$  because it includes all oil present in the filtrate (pressed oil and oil contained within the foot).

The energy consumed by the motor was determined from the following formulas:

$$P = P_M \times \frac{S_S}{S_{\max}} \times \frac{T}{T_{\max}} \times \cos \varphi, \quad (3)$$

where  $P$  is the electrical power supplied by the motor (W),  $P_M$  the motor's power rating ( $P_M = 8300$  W),  $T$  and  $T_{\max}$  the test torque and maximum torque (100 %) of the extruder motor (%),  $\cos \varphi$  its theoretical yield ( $\cos \varphi = 0.90$ ), and  $S_S$  and  $S_{\max}$  the test speed and maximum speed (682 rpm) of the rotating screws (rpm), respectively.

$$SME = \frac{P}{Q_S}, \quad (4)$$

where SME is the specific mechanical energy consumed by the motor per unit weight of coriander fruits (Wh/kg).

$$SME' = \frac{P}{Q_F \times T_L}, \quad (5)$$

where SME' is the specific mechanical energy consumed by the motor per unit weight of pressed oil (Wh/kg).

## Analytical Methods

Analyses of the fruits or the press cakes obtained were carried out according to the following French standards: moisture contents NF V 03-903 [25]; mineral contents NF V 03-322 [26]; oil contents NF V 03-908 [27]; protein contents NF V 18-100 [28]. Determination of the fruit's oil content and the residual oil content of press cakes was performed by means of a two-step Soxhlet extraction, each of 5 h duration, with *n*-hexane as the extracting solvent. The material was further milled between both steps by the use of a Foss Cyclotec 1093 (Denmark) mill fitted with a 300- $\mu$ m screen. An estimation of the three parietal constituents (cellulose, hemicelluloses, and lignins) of the coriander fruits was made using the ADF-NDF method from Van Soest and Wine [29, 30]. An estimation of the water-soluble components contained in the coriander fruits was made through measurements of the mass reduction of the test sample after 1 h in boiling water. All determinations were carried out in duplicate.

## Oil Quality Analysis

In order to assess the quality of the Soxhlet extracted oil (i.e., the oil extracted from coriander fruits using the Soxhlet extraction apparatus and *n*-hexane as the extracting solvent) and the pressed oils, their free fatty acid content was determined through two parameters: the acid value and the acidity (based on petroselinic acid), expressed in mg of KOH/g of oil and in %, respectively (French standard NF T 60-204) [31]. All determinations were carried out in duplicate.

## Glyceride Profile

The glyceride profile of the Soxhlet extracted oil was determined through gas chromatography (GC). Samples of about 0.10–0.15 g were weighed and 0.5 mL of BSTFA (*N,O*-bis(trimethylsilyl)trifluoroacetamide) with a derivatizing agent, i.e., trimethylchlorosilane, was added. Two internal standards were added (0.5 mL). The first one was composed of 8.068 mg/mL betulin in pyridine, while the

**Table 1** Results of the pressing experiments conducted with the Clextral BC 21 twin-screw extruder

Trial number	1	2	3	4	5	6	7	8	9	10
Operating conditions										
Screw profile	3	3	3	3	4	4	4	4	4	4
S <sub>S</sub> (rpm)	100	100	100	133	100	100	100	100	100	100
Q <sub>S</sub> (kg/h)	3.12	4.66	6.19	6.19	3.15	3.94	4.71	4.71	4.71	4.71
C <sub>F</sub> (g/h rpm)	31.2	46.6	61.9	46.5	31.5	39.4	47.1	47.1	47.1	47.1
θ <sub>6</sub> (°C)	83.0 ± 0.0 <sup>e</sup>	79.4 ± 0.8 <sup>f</sup>	77.7 ± 0.6 <sup>g</sup>	77.9 ± 0.6 <sup>g</sup>	95.2 ± 0.8 <sup>h</sup>	90.6 ± 0.6 <sup>b</sup>	89.1 ± 0.3 <sup>c</sup>	84.1 ± 0.9 <sup>d</sup>	82.6 ± 0.8 <sup>e</sup>	76.1 ± 1.1 <sup>b</sup>
θ <sub>7</sub> (°C)	120 (121.0 ± 0.6 <sup>b</sup> )	120 (119.0 ± 0.6 <sup>c,d</sup> )	120 (118.7 ± 0.5 <sup>d</sup> )	120 (118.7 ± 0.5 <sup>d</sup> )	120 (120.4 ± 0.8 <sup>b</sup> )	120 (120.1 ± 0.7 <sup>b</sup> )	120 (118.1 ± 1.1 <sup>e</sup> )	120 (119.5 ± 0.7 <sup>c</sup> )	100 (101.0 ± 1.1 <sup>f</sup> )	65 (79.7 ± 0.9 <sup>g</sup> )
Filtrate										
Q <sub>F</sub> (kg/h)	0.31	0.51	0.49	0.70	0.38	0.52	0.59	0.59	0.59	0.59
T <sub>L</sub> (%)	82.8	87.3	88.3	83.5	83.6	89.3	91.5	91.7	82.1	84.1
T <sub>F</sub> (%)	17.2	12.7	11.7	16.5	16.4	10.7	8.5	8.3	17.9	15.9
H <sub>F</sub> (%)	n.d.	n.d.	n.d.	n.d.	3.32 ± 0.05 <sup>f</sup>	3.60 ± 0.01 <sup>e</sup>	3.74 ± 0.07 <sup>d</sup>	4.59 ± 0.13 <sup>c</sup>	4.99 ± 0.11 <sup>b</sup>	5.78 ± 0.13 <sup>a</sup>
L <sub>F</sub> (% of dry matter)	n.d.	n.d.	n.d.	n.d.	59.33 ± 0.86 <sup>e</sup>	63.34 ± 0.77 <sup>d</sup>	73.48 ± 0.33 <sup>b</sup>	76.21 ± 0.12 <sup>a</sup>	68.74 ± 0.95 <sup>c</sup>	68.90 ± 0.04 <sup>e</sup>
M <sub>F</sub> (% of dry matter)	n.d.	n.d.	n.d.	n.d.	3.80 ± 0.01 <sup>a</sup>	3.35 ± 0.05 <sup>b</sup>	2.71 ± 0.13 <sup>cd</sup>	2.61 ± 0.04 <sup>d</sup>	2.78 ± 0.01 <sup>c</sup>	2.71 ± 0.04 <sup>cd</sup>
Press cake										
Q <sub>C</sub> (kg/h)	2.58	3.84	5.08	5.07	2.61	3.31	3.92	4.00	3.97	4.00
H <sub>C</sub> (%)	2.16 ± 0.02 <sup>g</sup>	3.85 ± 0.07 <sup>f</sup>	4.60 ± 0.04 <sup>d</sup>	4.46 ± 0.06 <sup>d</sup>	2.32 ± 0.14 <sup>g</sup>	3.73 ± 0.08 <sup>f</sup>	4.05 ± 0.06 <sup>e</sup>	4.88 ± 0.22 <sup>c</sup>	8.62 ± 0.27 <sup>b</sup>	8.99 ± 0.19 <sup>a</sup>
L <sub>C</sub> (% of dry matter)	17.63 ± 0.07 <sup>b</sup>	17.06 ± 0.05 <sup>c</sup>	17.62 ± 0.03 <sup>b</sup>	18.10 ± 0.08 <sup>a</sup>	16.48 ± 0.21 <sup>e</sup>	16.82 ± 0.29 <sup>d</sup>	16.06 ± 0.08 <sup>f</sup>	16.46 ± 0.11 <sup>e</sup>	14.99 ± 0.05 <sup>g</sup>	15.23 ± 0.14 <sup>g</sup>
M <sub>C</sub> (% of dry matter)	6.03 ± 0.01 <sup>c</sup>	6.10 ± 0.01 <sup>b</sup>	6.09 ± 0.03 <sup>b</sup>	5.98 ± 0.04 <sup>c</sup>	6.15 ± 0.03 <sup>b</sup>	6.15 ± 0.01 <sup>b</sup>	6.29 ± 0.01 <sup>a</sup>	6.11 ± 0.02 <sup>b</sup>	6.26 ± 0.09 <sup>a</sup>	6.17 ± 0.08 <sup>b</sup>
Oil recoveries (%)										
R <sub>L</sub>	32.5	38.2	28.0	37.8	40.5	46.9	45.8	46.3	41.1	41.8
R <sub>C</sub>	42.9	45.9	44.7	43.3	46.7	45.6	48.7	46.7	53.7	52.8
Energy consumed										
T (%)	16.4 ± 2.2 <sup>i</sup>	42.7 ± 1.6 <sup>e</sup>	57.2 ± 1.6 <sup>a</sup>	32.5 ± 2.1 <sup>g</sup>	25.0 ± 1.7 <sup>h</sup>	37.2 ± 1.4 <sup>f</sup>	45.6 ± 1.5 <sup>d</sup>	47.7 ± 2.0 <sup>c</sup>	54.2 ± 2.2 <sup>b</sup>	54.9 ± 1.3 <sup>b</sup>
P (W)	180.0 ± 23.8 <sup>h</sup>	467.9 ± 17.3 <sup>c</sup>	626.7 ± 17.8 <sup>a</sup>	473.1 ± 30.3 <sup>c</sup>	274.2 ± 18.8 <sup>g</sup>	407.8 ± 15.6 <sup>f</sup>	499.0 ± 16.1 <sup>d</sup>	522.9 ± 21.7 <sup>c</sup>	594.1 ± 23.9 <sup>b</sup>	601.0 ± 14.4 <sup>b</sup>
SME (Wh/kg fruit processed)	57.7 ± 7.6 <sup>g</sup>	100.4 ± 3.7 <sup>d</sup>	101.3 ± 2.9 <sup>d</sup>	76.5 ± 4.9 <sup>f</sup>	86.9 ± 6.0 <sup>e</sup>	103.5 ± 4.0 <sup>cd</sup>	105.9 ± 3.4 <sup>c</sup>	111.0 ± 4.6 <sup>b</sup>	126.1 ± 5.1 <sup>a</sup>	127.6 ± 3.0 <sup>a</sup>
SME' (Wh/kg pressed oil)	710.2 ± 93.8 <sup>h</sup>	1053.8 ± 39.0 <sup>c</sup>	1447.2 ± 41.1 <sup>a</sup>	810.4 ± 51.9 <sup>g</sup>	859.1 ± 58.9 <sup>f</sup>	884.1 ± 33.9 <sup>ef</sup>	925.6 ± 29.9 <sup>de</sup>	960.1 ± 39.9 <sup>d</sup>	1228.9 ± 49.5 <sup>b</sup>	1221.4 ± 29.2 <sup>b</sup>

C<sub>F</sub> is the device's filling coefficient (g/h rpm); it is defined as the ratio of the inlet flow rate of coriander fruits (Q<sub>S</sub>) to the screw rotation speed (S<sub>S</sub>). θ<sub>6</sub> is the barrel temperature measured during sampling at the level of module 6, i.e., the filtration module (°C). θ<sub>7</sub> is the barrel temperature at the level of module 7 (set value first mentioned, plus temperature measured during sampling in parentheses), i.e., the pressing zone (°C). Modules 2-5 were heated to 65 °C for all trials. T<sub>F</sub> is the mass content of the foot in the filtrate (%). H<sub>F</sub> is the moisture content in the foot of the filtrate (%). L<sub>F</sub> is the oil content in the foot of the filtrate (% of the dry matter). M<sub>F</sub> is the mineral content in the foot of the filtrate (% of the dry matter). H<sub>C</sub> is the moisture content in the press cake (%). M<sub>C</sub> is the mineral content in the press cake (% of the dry matter). Means in the same line with the same superscript letter (a-i) are not significantly different at P < 0.05. n.d. non determined

second one was a 8.087 mg/mL tricaprln solution. Next, samples were heated to 80 °C for 30 min.

They were then analyzed through GC using an Agilent Technologies 7890A (USA) gas chromatograph. The different compounds were separated in a J&W DB-5HT GC (Agilent, USA) column (15 m, 0.32 mm i.d., 0.10 µm film thickness) under the following conditions: oven temperature: 50 °C; 50–200 °C (15 °C/min); 200–290 °C (3 °C/min, held 10 min); 290–360 °C (10 °C/min, held for 15 min); flame ionization detector (FID) 380 °C; carrier gas helium (66 kPa).

### Fatty Acid Composition

Determination of the fatty acid composition of the oils obtained was performed by gas chromatography (GC). For this, oil samples were dissolved in *tert*-butyl methyl ether (TBME) to a concentration of 20 mg/mL. Next, 100-µL aliquots of these solutions were converted to methyl esters by the addition of 50 µL of a 0.2 mol/L trimethylsulfonium hydroxide (TMSH) in methanol solution (French standard NF ISO 5508) [32].

The resulting fatty acid methyl esters were subjected to GC analysis by the use of a Varian 3800 (USA) gas chromatograph equipped with a flame ionization detector. Separation of the methyl esters was achieved in a CP Select CB (Varian, USA) fused silica capillary column (50 m, 0.25 mm i.d., 0.25 µm film thickness). The initial oven temperature was held at 185 °C for 40 min, after which it was increased to 250 °C at a rate of 15 °C/min and maintained at 250 °C for 10 min. The temperature of the injector and the detector was kept at 250 °C. Helium was used as the carrier gas with a flow rate of 1.2 mL/min. All determinations were carried out in triplicate.

### Essential Oil Content

Hydrodistillation (or water distillation) was applied to extract the essential oil from the coriander material, i.e., the fruits or the press cakes. 200 g of milled material was mixed with 2 L of water (1:10 ratio) and placed in a distillation flask where it was distilled for 5 h. The installation consisted of a Clevenger-like apparatus. Volatilized compounds and water vapor were condensed through a cooling system, collected, and separated in a separatory tube. All determinations were carried out in triplicate.

### Essential Oil Composition

The composition of essential oils extracted from the coriander fruits and the press cakes was determined by gas chromatography (GC). The essential oils were analyzed using a HP 5890 Series II (USA) gas chromatograph equipped with

a flame ionization detector. The carrier gas was helium with a constant pressure of 15 psi. Compounds of the essential oils were separated in an Agilent VF-5 ms (USA) apolar column (30 m, 0.25 mm i.d., 1 µm film thickness). The injected volume was 0.5 µL. The initial oven temperature was 110 °C and increased at a rate of 7 °C/min to 220 °C. The injector and detector temperatures were 220 °C. Identification of the compounds was based on a comparison of their retention indices relative to a series of *n*-alkanes (C<sub>9</sub>–C<sub>15</sub>) with those of literature (NIST). All determinations were carried out in triplicate.

### Particle Size Distribution of the Press Cakes

The particle size distribution of press cakes was determined by optical microscopy. For this, a Nachet France Z 45 P (France) × 15 binocular magnifier was used and five different photographs were taken of each sample by the use of the Archimed 4.0 (France) software. Next, the particle size distribution was constructed through manual measurement of the diameter of all particles on the five photographs by the use of the ImageJ (USA) software.

### Antioxidant Capacity of the Press Cakes

The antioxidant capacity of the press cakes was measured by means of the DPPH radical scavenging assay, through which the radical scavenging activity of an extract against the stable DPPH (2,2-diphenyl-1-picrylhydrazyl) radical was determined. The applied method was based on the one used by Brand-Williams *et al.* [33] and comprises methanolic extraction and DPPH scavenging assessment through UV spectrophotometry. Methanolic extracts were obtained by methanol Soxhlet extraction for 5 h and subsequent concentration by rotary evaporation. Aqueous extracts were prepared through rotary evaporation of water solutions. Inhibition (INH, %) was calculated from the following formula:

$$INH = \frac{AB - AA}{AB} \times 100 \quad (6)$$

where AB and AA are the absorbances of the DPPH solution and the tested extract solution, respectively.

A 6 × 10<sup>-5</sup> M DPPH in methanol solution was prepared daily, protected from the light and stored at low temperatures (i.e., at about 5 °C). A 10-mg sample of the extract was dissolved in 1 mL of methanol or a 1/1 water/methanol solution for aqueous extracts. The samples were subjected to sonication to ensure complete dissolution. Then, 50 µL of this extract solution was added to 2 mL of DPPH solution, and the samples were put in the dark for 30 min to react. Absorption was measured with UV spectrophotometry at 515 nm. Further, a calibration curve was set up using

methanolic Trolox solutions with known concentrations ranging from 100 to 750  $\mu\text{mol/L}$ . Results were expressed as  $\mu\text{mol}$  of Trolox equivalents (TE) per g of press cake. All determinations were carried out in triplicate.

### Statistical Analyses

All determinations were conducted in duplicate or triplicate, and data are expressed as means  $\pm$  standard deviations. The means were compared by the use of a single-factor analysis of variance (ANOVA) using the GLM procedure of the SAS data analysis software. The comparison between the different individual means was performed using the Duncan's multiple range test at a 5 % probability level.

## Results and Discussion

### Chemical Composition of the Coriander Fruits

The coriander fruits that were utilized during this study were of French origin and were shown to contain

$27.67 \pm 0.57$  % vegetable oil on a dry basis. This value is in accordance with the oil contents of 9.9–28.4 % reported in the literature [3, 4, 24, 34–36]. It was higher than the oil content of the raw material used in the only antecedent study that also dealt with the extraction of coriander oil by twin-screw extrusion, which was 21.9 % of the dry matter for Tunisian coriander [24]. These variations can be attributed not only to the difference in variety but also to some factors such as cultivation conditions, especially the use of fertilizers and irrigation [37]. Mineral and protein contents were  $5.86 \pm 0.11$  % of the dry matter and  $14.07 \pm 0.43$  % of the dry matter, respectively. The three parietal constituents (cellulose, hemicelluloses, and lignins) constituted  $24.38 \pm 0.55$  % of the dry matter,  $17.84 \pm 0.56$  % of the dry matter and  $11.01 \pm 0.25$  % of the dry matter, respectively. Lastly, the water-soluble components represented  $14.32 \pm 0.09$  % of the dry matter in the coriander fruits.

The fatty acid composition of the solvent extracted oil obtained from coriander fruits is shown in Table 2. Petroselinic acid was found to be the major fatty acid, representing 72.6 % of all fatty acids, while significant amounts of linoleic (13.8 %) and oleic acid (6.0 %) were also detected. This is consistent with earlier reports on the fatty

**Table 2** Quality and fatty acid composition of Soxhlet extracted oil, and pressed oils from coriander fruits (trials 2, 4, 7, 8 and 10)

	Soxhlet extracted oil	Trial number 2	Trial number 4	Trial number 7	Trial number 8	Trial number 10
Oil quality						
Acid value (mg of KOH/g of oil)	$3.58 \pm 0.10^a$	$2.90 \pm 0.08^{b,c}$	$2.92 \pm 0.02^{b,c}$	$2.80 \pm 0.14^c$	$2.90 \pm 0.02^{b,c}$	$3.02 \pm 0.16^b$
Acidity (% FFA as petroselinic acid)	$1.80 \pm 0.05^a$	$1.46 \pm 0.04^{b,c}$	$1.47 \pm 0.01^{b,c}$	$1.41 \pm 0.07^c$	$1.46 \pm 0.01^{b,c}$	$1.52 \pm 0.08^b$
Fatty acid composition (%)						
Caproic acid (C6:0)	$0.1 \pm 0.1$	n.dc.	n.dc.	n.dc.	n.dc.	n.dc.
Myristic acid (C14:0)	n.dc.	$0.2 \pm 0.0^a$	$0.2 \pm 0.0^a$	$0.1 \pm 0.0^a$	$0.2 \pm 0.0^a$	$0.2 \pm 0.0^a$
Palmitic acid (C16:0)	$2.9 \pm 0.1^b$	$3.3 \pm 0.0^a$	$3.3 \pm 0.0^a$	$3.3 \pm 0.0^a$	$3.3 \pm 0.0^a$	$3.3 \pm 0.0^a$
Palmitoleic acid (C16:1)	$0.4 \pm 0.1^a$	$0.2 \pm 0.0^b$	$0.2 \pm 0.0^b$	$0.2 \pm 0.0^b$	$0.2 \pm 0.0^b$	$0.2 \pm 0.0^b$
Margaric acid (C17:0)	<0.1	n.dc.	n.dc.	n.dc.	n.dc.	n.dc.
Stearic acid (C18:0)	<0.1	$0.7 \pm 0.0^a$	$0.7 \pm 0.0^a$	$0.7 \pm 0.0^a$	$0.7 \pm 0.0^a$	$0.7 \pm 0.0^a$
Petroselinic acid (C18:1n-12)	$72.6 \pm 0.1^c$	$72.8 \pm 0.2^c$	$72.9 \pm 0.2^{b,c}$	$73.4 \pm 0.2^a$	$73.3 \pm 0.1^{a,b}$	$73.3 \pm 0.2^{a,b}$
Oleic acid (C18:1n-9)	$6.0 \pm 0.2^a$	$5.5 \pm 0.3^b$	$5.4 \pm 0.1^{b,c}$	$4.9 \pm 0.2^d$	$5.2 \pm 0.1^{b,c,d}$	$5.0 \pm 0.2^{c,d}$
cis-Vaccenic acid (C18:1n-7)	$1.2 \pm 0.1^b$	$1.3 \pm 0.0^a$	$1.3 \pm 0.0^a$	$1.3 \pm 0.0^a$	$1.3 \pm 0.0^a$	$1.3 \pm 0.0^a$
Linoleic acid (C18:2)	$13.8 \pm 0.1^b$	$13.8 \pm 0.1^b$	$13.8 \pm 0.0^b$	$13.8 \pm 0.0^b$	$13.8 \pm 0.0^b$	$13.9 \pm 0.0^a$
Linolenic acid (C18:3)	$0.2 \pm 0.1^a$	$0.1 \pm 0.0^b$	$0.1 \pm 0.0^b$	$0.1 \pm 0.0^b$	$0.1 \pm 0.0^b$	$0.1 \pm 0.0^b$
Arachidic acid (C20:0)	$0.1 \pm 0.0^a$	$0.1 \pm 0.0^a$	$0.1 \pm 0.0^a$	$0.1 \pm 0.0^a$	$0.1 \pm 0.0^a$	$0.1 \pm 0.0^a$
Gadoleic acid (C20:1)	$0.2 \pm 0.1$	n.dc.	n.dc.	n.dc.	n.dc.	n.dc.
SFA	$3.2 \pm 0.0^b$	$4.2 \pm 0.0^a$	$4.2 \pm 0.0^a$	$4.2 \pm 0.0^a$	$4.2 \pm 0.0^a$	$4.2 \pm 0.0^a$
MUFA	$80.4 \pm 0.2^a$	$79.7 \pm 0.2^c$	$79.8 \pm 0.1^c$	$79.9 \pm 0.0^{b,c}$	$80.0 \pm 0.0^b$	$79.9 \pm 0.0^{b,c}$
PUFA	$14.0 \pm 0.1^{ab}$	$14.0 \pm 0.1^{ab}$	$13.9 \pm 0.0^b$	$14.0 \pm 0.0^{ab}$	$13.9 \pm 0.0^{ab}$	$14.0 \pm 0.0^a$
SFA/PUFA	$0.2 \pm 0.0^b$	$0.3 \pm 0.0^a$	$0.3 \pm 0.0^a$	$0.3 \pm 0.0^a$	$0.3 \pm 0.0^a$	$0.3 \pm 0.0^a$
Identified fatty acids	$97.6 \pm 0.1^c$	$97.9 \pm 0.1^b$	$97.9 \pm 0.1^b$	$98.0 \pm 0.0^{ab}$	$98.1 \pm 0.0^a$	$98.1 \pm 0.0^a$

SFA saturated fatty acids, MUFA monounsaturated fatty acids, PUFA polyunsaturated fatty acids. Means in the same line with the same superscript letter (a–d) are not significantly different at  $P < 0.05$ . n.dc. not detected

acid composition of coriander oil [3, 4, 7, 24]. In addition, the oil was shown to exhibit an acceptable free fatty acid (FFA) content of  $1.8 \pm 0.1$  %.

The glyceride profile of Soxhlet extracted oil was determined through GC. The oil contained  $96.25 \pm 1.05$  % of triglycerides (TAG),  $1.03 \pm 0.09$  % of diglycerides (DAG), and  $0.09 \pm 0.02$  % of monoglycerides (MAG). GC analysis also led to a second estimation of the free fatty acid content and the latter was slightly lower ( $1.02 \pm 0.03$  %) than that obtained by titration (French standard NF T 60-204), i.e., 1.8 %. The high amount of TAG and concurrent low FFA content demonstrated that little hydrolysis or enzymatic degradation had occurred in the Soxhlet extracted oil, indicating its good quality.

The coriander fruits displayed an essential oil content of 0.7 % of the dry matter through water distillation (Table 3), and this agrees with the 0.3–1.2 % results reported by Kirilan *et al.* [38]. Further, the essential oil was particularly rich in linalool (71.7 %), a monoterpene alcohol providing the coriander fruits with their characteristic lemony citrus flavor [39]. Such content was comparable to those (41–80 %) mentioned by Asgarpanah and Kazemivash [40] and Sahib *et al.* [41], depending on the variety and origin of coriander. Other important compounds were  $\alpha$ -pinene and  $\gamma$ -terpinene, representing 5.6 % and 5.0 % of the essential oil, respectively.

### Influence of the Operating Conditions on Oil Extraction Efficiency

In order to assess the impact of the extrusion operating conditions on oil extraction, different screw profiles were tested and the device's filling coefficient and pressing temperature were varied. The effect on the extraction efficiency was evaluated through the determination of two oil recoveries ( $R_L$  and  $R_C$ ), the filtrate's foot content and the extruder's energy consumption. Twin-screw extrusion is capable of combining a crushing operation, located at the trituration zone, with a compressing action, performed by the reverse

pitch screws. The former leads to a substantial reduction in the particle size of the material, resulting in a more efficient oil release, while the latter ensures solid/liquid separation through the formation of a counter pressure at the beginning of module 7. The effectiveness of twin-screw extrusion for the extraction of vegetable oil from oil seeds was formerly demonstrated for sunflower oil [16–21], jatropha oil [22] and neem oil [23].

In the first screw profile tested (profile 1), the CF2C reverse pitch screws were 50 mm long, with a pitch of  $-25$  mm, and they were positioned immediately after the filtration module (Fig. 2). With such a screw configuration and a  $120$  °C pressing temperature, a lot of solid particles were rapidly forced through the filter, preventing the oil from draining freely and thus its separation from a press cake. Then, it simply resulted in the clogging of the twin-screw extruder. This phenomenon persisted even when reducing the device's filling coefficient to less than 10 g/h rpm.

A second screw profile (profile 2) was therefore tested consisting of the same CF2C screws with a  $-25$  mm pitch, but positioned 25 mm from the end of module 6 (Fig. 2). However, the same phenomenon, i.e., the clogging of the machine, was observed after only a few tens of seconds. As the CF2C screws with a  $-25$  mm pitch used for screw profiles 1 and 2 were too restrictive, these reverse pitch screws were replaced in screw profiles 3 and 4 by CF2C screws with a higher, i.e., less restrictive, pitch ( $-33$  mm instead of  $-25$  mm).

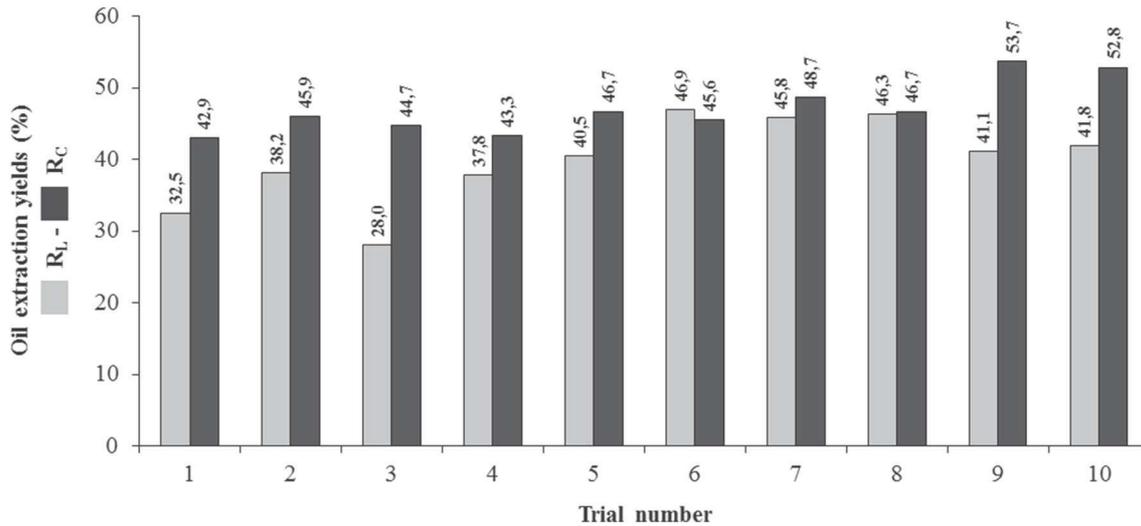
For all experiments conducted with these last two screw profiles, solid/liquid separation at the filter section was effective and the oil content of the press cakes had decreased compared to the coriander fruits. This led to residual oil contents of 18.1–15.0 % of the dry matter and oil recoveries ( $R_C$ ) of 42.9–53.7 %, depending on the operating conditions that were applied during extrusion (Table 1). The mineral content in the press cake was a little higher than in the actual coriander fruits due to the oil pressing, and it varied from 6.0 to 6.3 % of the dry matter

**Table 3** Essential oil content in coriander fruits and in press cakes from trials 2, 4, 7, 8 and 10, and antioxidant capacity of press cakes 2, 4, 8 and 10 (before and after hydrodistillation)

	Coriander fruits	Press cake number 2	Press cake number 4	Press cake number 7	Press cake number 8	Press cake number 10
Essential oil content (% of the dry matter)	$0.67 \pm 0.10^a$	$0.14 \pm 0.00^c$	$0.14 \pm 0.01^c$	$0.14 \pm 0.00^c$	$0.18 \pm 0.01^c$	$0.31 \pm 0.02^b$
Antioxidant capacity ( $\mu\text{mol}$ of TE per g of press cake)						
Before hydrodistillation	n.d.	$3.7 \pm 0.2^c$	$4.2 \pm 0.0^b$	n.d.	$5.1 \pm 0.1^a$	$5.0 \pm 0.2^a$
After hydrodistillation	n.d.	$2.4 \pm 0.2^a$	$2.8 \pm 0.3^a$	n.d.	n.d.	n.d.

Means in the same line with the same superscript letter (a–c) are not significantly different at  $P < 0.05$

n.d. Not determined



**Fig. 3** Variation in oil recoveries for trials 1–10

(Table 1). In addition, for the two screw profiles tested successfully, the lower the oil content in the press cake, the higher its mineral content. The oil recovery ( $R_L$ ), defined as the ratio of the pressed oil to the total oil contained within the fruit, varied from 28.0 to 46.9 % (Table 1). It was lower than the oil recovery ( $R_C$ ) due to the oil retained in the filtrate foot (Fig. 3) and the difference between these two oil recoveries was minimal with the lowest filtrate foot contents (trials 6–8).

Moreover, the mass content of the foot in the filtrate was never more than 18 %, instead of 47–66 % for oil pressing from coriander fruits of Tunisian variety [24], meaning that the screw configurations used in this study were much more suited. Further reduction of the filtrate foot content could be accomplished through the application of a filter section with smaller perforations, but this was not possible during the course of this study. Low foot contents are important for the stability and the capacity of the extrusion process, as the centrifugation foot is commonly reintroduced at the inlet of the extruder, thus forming an additional feed flow.

For all screw profiles tested, the trituration zone was composed of a succession of 10 monolobe paddles and 10 bilobe paddles. It was the same as the one in the optimized screw profile used for the extraction of oil from jatropha seeds by mechanical pressing [22]. These elements ensure a profound reduction in the particle size of the material, leading to the rupture of the cell structures. In the third screw profile tested (profile 3), CF2C screws with a –33 mm pitch were still positioned 25 mm from the end of module 6 (Fig. 2). Four experiments were conducted with this profile (trials 1–4). However, when using screw profile 3, there was an accumulation of oil between the end of the filter section and the beginning of the pressing zone which

could not drain freely. This presumably led to a decrease in the filtrate’s flow rate as well as in the oil recovery ( $R_L$ ). A fourth screw profile was therefore tested (profile 4), with the CF2C screws with a –33 mm pitch being positioned immediately after the filtration module (Fig. 2). This profile was applied for trials 5–10. Comparing trials 1 and 5, conducted with screw profiles 3 and 4, respectively, while maintaining the same operating conditions, screw profile 4 was found to be significantly more effective in oil extraction. It further resulted in a significantly lower residual oil content in the press cake and a slight reduction in the filtrate’s foot content, leading to an increase in both oil recoveries (Table 1). However, the increase in the motor’s torque resulted in a modest augmentation of the specific mechanical energy per unit weight of pressed oil (+21 %), which was shown to be significant through statistical analysis. In conclusion, screw profile 4 was considered as the optimized screw configuration for this study.

Next, the influence of the device’s filling coefficient on oil extraction efficiency was assessed for screw profiles 3 and 4. When comparing trials 1–3, using the third screw profile, the increase in the inlet flow rate of coriander fruits from 3.12 kg/h (trial 1) to 6.19 kg/h (trial 3) directly affected the device’s filling coefficient, increasing it from 31 to 62 g/h rpm (Table 1). Because it was more filled, the crushing ability of the trituration zone certainly diminished and the size reduction of solid particles became less significant, thus resulting in a decrease in the filtrate’s foot content. Furthermore, the degree of filling of the CF2C screws increased as the device’s filling coefficient increased, resulting in an enhanced pressure buildup inside the extruder and thus to an improved solid/liquid separation. As a result of this, the motor’s torque significantly increased, resulting

in a higher specific mechanical energy per unit weight of pressed oil and a lower residual oil content of the obtained press cake, the latter being significantly different from the residual oil content of press cake from trial 1 according to the statistical analysis. Consequently, the oil recovery ( $R_C$ ) increased with an increase in the device's filling coefficient. In addition, the oil recovery ( $R_L$ ) showed an even more distinct increase due to the concurrent decrease of the filtrate's foot content. Similar findings were reported for the mechanical pressing of sunflower and jatropha oil [17, 18, 22].

For trial 3, the device's filling coefficient reached 62 g/h rpm. However, even though the motor's torque still increased, this did not result in a decrease in the press cake's residual oil content, the latter exhibiting a slight increase. With such a device's filling coefficient, the solid particles accumulated more upstream from the pressing zone, obstructing part of the filtering screens and thus reducing the filtration surface. This resulted in a decrease of the filtrate's flow rate, causing a significant reduction in the oil recovery ( $R_L$ ) and an important increase in the specific mechanical energy. With screw profile 3, the machine became, in fact, too highly filled with a 62 g/h rpm device's filling coefficient, and higher filling coefficients were not tested to avoid clogging of the twin-screw extruder.

Comparing trials 2 and 4, the device's filling coefficient was the same (47 g/h rpm), but the screw rotation speed and the inlet flow rate of coriander fruits increased in the same proportions for trial 4: from 100 to 133 rpm and from 4.66 to 6.19 kg/h, respectively (Table 1). The increase in the screw rotation speed led to a more effective size reduction of the solid particles near the trituration zone, resulting in an increase in the foot content in the filtrate. Although the press cake's residual oil content was significantly higher from a statistical point of view, which is due to a less efficient compression of the mixture in the reverse screw elements, there was only a marginal reduction effect on both oil recoveries ( $R_L$  and  $R_C$ ). Furthermore, the increase in the screw rotation speed significantly reduced the motor's torque, further reducing the specific mechanical energy per unit weight of pressed oil. In conclusion, a device's filling coefficient close to 50 g/h rpm was better suited for such a screw configuration, and higher productivity of the extruder resulted in the production of similar oil yields at a lower cost.

The effect of the device's filling coefficient on the oil extraction efficiency was further evaluated for the fourth screw profile through trials 5–7. As previously observed with screw profile 3 (trials 1–3), a decrease in the filtrate's foot content was observed with the increase of the device's filling coefficient, which is due to a lowering of the crushing ability of the trituration zone, thus leading to larger solid particles at the outlet of this zone. A significant

increase in the motor's torque was observed at the same time due to the increase in the degree of filling of the CF2C screws and thus resulting in a better pressing action on the matter in this location. Comparing trials 5 and 6, this further led to an improvement in the liquid/solid separation, as illustrated by the relative increase in pressed oil quantity, resulting in an increased oil recovery ( $R_L$ ) without any significant effect on specific mechanical energy per unit weight of pressed oil, as shown by the statistical analysis. Trial 6 was also the most efficient experiment of the entire study for oil extraction, exhibiting an oil recovery ( $R_L$ ) of 46.9 % and producing a press cake with good quality (residual oil content of 16.8 % of the dry matter).

For a higher device's filling coefficient (47 g/h rpm, trial 7), the relative quantity of pressed oil slightly decreased compared to trial 6, illustrating the same phenomenon as that observed more significantly for trial 3 with the third screw profile, i.e., the obstruction of part of the filtering screens by an accumulation of solid particles. Despite a slightly excessive filling of the machine, the effect on the oil recovery ( $R_L$ ) was quite limited and the corresponding specific mechanical energy only increased by 5 % compared to trial 6. At the same time, the press cake was of better quality with a significantly reduced residual oil content, resulting in an increase in the oil recovery ( $R_C$ ). Therefore, analogous to what was found for screw profile 3, the 47 g/h rpm device's filling coefficient was considered to be a good compromise for screw profile 4 between oil extraction efficiency and the press cake's reduction in lipids.

As a second important operating parameter for twin-screw extrusion, the effect of the pressing temperature on the oil extraction efficiency was assessed for screw profile 4 using the optimized device's filling coefficient through trials 7–10. The applied pressing temperatures were 120, 100, 80 and 65 °C, respectively (Table 1). Contrary to what has been reported for sunflower and jatropha seeds [17, 18, 22], decreasing the pressing temperature from 120 to 65 °C did not substantially improve the oil extraction efficiency. A decrease in the pressing temperature of the extruder leads to a significant increase in the material viscosity, resulting in a profound impact on the residence time and energy consumption [42]. As the increased viscosity of the material flow causes a higher degree of filling of the extruder, the residence time and the energy input increased with decreasing pressing temperature. This led to an increase in both the motor's torque and the specific mechanical energy per unit weight of fruit processed, rendering the pressed oil slightly more expensive to produce. However, the oil extraction efficiency and filtrate's foot content at 100 °C (trial 8) were quite similar to that obtained at 120 °C (trial 7). At the same time, the press cake's oil contents were nearly comparable between trials 7 and 8, further resulting in similar oil recoveries ( $R_C$ ).

Conversely, when the pressing temperature was only 80 °C (trial 9) or 65 °C (trial 10), the flow of material across the CF2C screws became much more viscous, as illustrated by the high values for the motor's torque (Table 1). This led to a better compression of the matter near the reverse pitch screws, and thus to a better reduction of the lipids in the press cakes. Moreover, press cakes from trials 9 and 10 revealed the two lowest residual oil contents of the entire study: 15.0 and 15.2 % of the dry matter, respectively, resulting in the two highest values for the oil recovery ( $R_C$ ): 53.7 and 52.8 %, respectively. However, because solid particles accumulated more upstream from the pressing zone for these two lowest pressing temperatures, a significant increase in the foot content in the filtrate was also observed. Therefore, oil recoveries ( $R_L$ ) slightly decreased compared to trials 7 and 8, further contributing to a significant increase (+30 % on average) in the specific mechanical energy.

In conclusion, the highest oil recovery ( $R_L$ ) for this study was 46.9 %, obtained under the following operating conditions (trial 6): profile 4 screw configuration, 39.4 g/h rpm device's filling coefficient and 120 °C pressing temperature (Table 1). In addition, the residual oil content in the press cake was 16.8 % of the dry matter. The specific mechanical energy was 103 Wh/kg fruit processed or 884 Wh/kg pressed oil, which is quite low compared to other samplings in this study. It should however be noted that these values of energy consumption need further confirmation when twin-screw extrusion of coriander fruits is executed on a larger scale. The amount of foot in the filtrate was only 10.7 % under these optimal conditions.

The lowest press cake's residual oil content (15.0 % of the dry matter) was obtained under the following operating conditions (trial 9): profile 4 screw configuration, 47.1 g/h rpm device's filling coefficient and 80 °C pressing temperature (Table 1). The oil recovery ( $R_C$ ) was then maximal (53.7 %). However, the amount of foot in the filtrate was much more important for these conditions (17.9 %). The oil recovery ( $R_L$ ) was slightly lower than for the optimal conditions (41.1 % instead of 46.9 %). Moreover, the corresponding pressed oil was more expensive to produce (126 Wh/kg fruit processed or 1229 Wh/kg pressed oil). Reducing the diameter of perforations in the filter section should diminish the filtrate's foot content.

The abrupt shutdown of the extruder when all experiments were finished allowed the observation of material along the screw profile. From this, it could be seen that near modules 1–5, the trituration zone was significantly more filled than the conveying zones. Furthermore, the particle size of the material was found to be reduced substantially after passing through the monolobe and bilobe paddles, even if no accurate measurement of the particle size was carried out. Additionally, the pressing zone containing the

reverse screw elements showed the highest filling due to its inherent pressing action on the material. And, as it was previously observed in the case of jatropha oil pressing [22], this led to an additional size reduction of the material due to the strong shearing force imposed by the reverse screws.

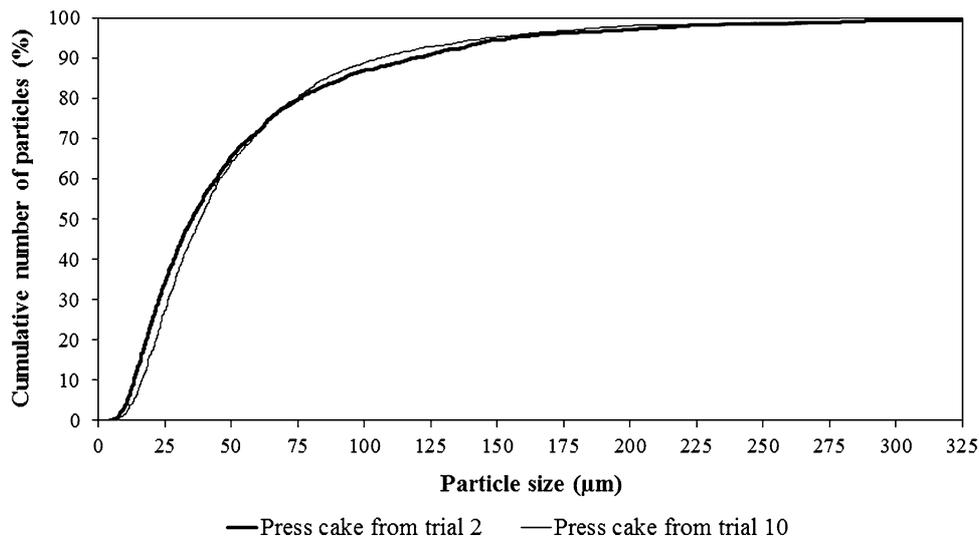
In summary, the extraction of vegetable oil from coriander fruits originating from France by mechanical pressing in a twin-screw extruder was improved compared to results obtained in the previous study, i.e., with fruits of Tunisian origin [24]. Indeed, even if the French variety was richer in vegetable oil than the Tunisian one (27.7 % of the dry matter instead of 21.9 %), the lowest residual oil content in the press cake (15.0 % of the dry matter) was lower than for the Tunisian variety (16.6 %). This means that the reduction in lipids was much more effective, leading to a better oil recovery ( $R_C$ ): 54 % instead of only 45 % for the Tunisian variety. Furthermore, the filtrate's foot content was never more than 17.9 % due to the use of an optimized screw configuration, while it was at least 47.5 % in the previous study [24]. This considerably facilitates the pressed oil isolation, i.e., elimination of the foot from the filtrate by centrifugation.

### Influence of the Operating Conditions on Oil Quality

The quality of pressed oils was examined through two parameters: the acidity and the petroselinic acid content (Table 2). For screw profile 3, the two pressed oils that were analyzed were those from the two most effective trials in terms of oil recovery ( $R_L$ ), i.e., trial 2 and trial 4. For screw profile 4, this included the three pressed oils (trials 7, 8 and 10) that were produced with varying pressing temperatures: 120, 100 and 65 °C, respectively.

The oil acidity varied between 1.41 and 1.52 % for all pressed oils that were analyzed, corresponding to acid values between 2.80 and 3.02 mg of KOH/g of oil (Table 2). Thus, these virgin-type vegetable oils exhibit a higher quality than the Soxhlet extracted one (1.80 % acidity). Their acidity was slightly higher than that measured for pressed oils from the Tunisian variety of coriander fruits, which was situated between 1.54 and 2.21 mg of KOH/g of oil [24]. However, they were of excellent quality. Next to this, as revealed by the statistical analysis, it appeared that screw configuration had no significant effect on oil quality. Moreover, a decrease in the pressing temperature in the twin-screw extruder did not induce an oil quality improvement. This could be due to the fact that pressed oils did not properly come into contact with the pressing temperature, but rather with the temperature at the filter section (i.e., module 6). The latter did not exhibit high temperature variation between different trials, fluctuating between 76 and 89 °C for the pressed oils analyzed (Table 1).

**Fig. 4** Cumulative number of particles as a function of particle size for the press cakes from trials 2 and 10



The fatty acid composition of pressed oils was also shown to be nearly independent of the operating conditions used for twin-screw extrusion, i.e., screw configuration and pressing temperature (Table 2). Similar to the oil obtained through solvent extraction, all analyzed pressed oils were very rich in petroselinic acid, exhibiting contents between 72.8 and 73.4 %, while also similar contents of linoleic and oleic fatty acids were found. The minor differences between samples constituted a small variation in the petroselinic/oleic acid ratio. This possibly resulted from a complex integration of GC results, as retention times of both petroselinic and oleic acid were very similar. In conclusion, on the basis of the two quality criteria examined, it was clear that all analyzed pressed oils were high-quality vegetable oils.

### Potential Press Cake Applications

In order to establish an economically favorable extrusion process, it is of key importance that extraction by-products such as the press cakes find some industrial applications. The ten press cakes were fine powders composed of almost spherical particles and their particle size distribution was estimated for press cakes from trials 2 and 10, each corresponding to the highest oil recovery ( $R_C$ ) obtained for the two screw profiles used successfully (i.e., profiles 3 and 4, respectively). It appeared that the particle size distribution of press cakes was independent on the screw profile that was applied (Fig. 4). Indeed, the mean diameter of particles in press cakes from trials 2 and 10 was 35  $\mu\text{m}$  and 38  $\mu\text{m}$ , respectively.

Press cakes still contained part of the essential oil from coriander fruits. However, its content was very low for all operating conditions tested, varying from 0.14 to 0.31 % of the dry matter (Table 3). Further, the residual essential oil

content in the press cake mainly depended on the pressing temperature applied in the twin-screw extruder, increasing with its decrease. A maximal essential oil content of 0.31 % was found inside the press cake from trial 10, corresponding to the lowest pressing temperature tested (i.e., 65 °C). Such temperature did not allow strong evaporation of the essential oil contained within the press cake. The residual essential oil content amounted in that case to 40 % of the essential oil in the starting material, leading to the conclusion that the remaining 60 % was co-extracted with the vegetable oil, rendering it pleasantly scented. The simultaneous extraction of both vegetable oil and essential oil through mechanical pressing is an interesting novelty of this research work. The residual essential oil in the press cakes could be extracted by means of hydrodistillation. Its composition was rather similar to that of the essential oil extracted from the fruits (Table 4), linalool being the main component. Linalool is one of the most commonly applied fragrance ingredients in cosmetics, perfumes, shampoos, soaps and even household detergents due to its fresh and flowery scent and it displays a worldwide use of over 1000 tons per year [43]. Further applications include its use as a repellent against mosquitoes [44]. However, because of the low quantities of volatile oil that could be extracted using hydrodistillation and the lack of a strong market for this essential oil, using the press cakes as raw materials to extract the remaining essential oil may seem too ambitious. Its application on an industrial scale would currently not be economical. It might therefore be more interesting to leave the volatile oil within other end products such as agromaterials, providing them with an added value.

Another possible use of the press cakes could be the isolation of some natural antioxidants through methanolic extraction, these being potentially interesting due to their beneficial impact on health. The antioxidant capacity of the

**Table 4** Essential oil composition in coriander fruits and in press cakes from trials 2 and 4

	Coriander fruits	Press cake number 2	Press cake number 4
Essential oil composition (%)			
$\alpha$ -Pinene	5.6 $\pm$ 0.1 <sup>a</sup>	1.2 $\pm$ 0.2 <sup>b</sup>	1.2 $\pm$ 0.2 <sup>b</sup>
Camphene	0.8 $\pm$ 0.0 <sup>a</sup>	0.2 $\pm$ 0.0 <sup>b</sup>	0.2 $\pm$ 0.0 <sup>b</sup>
$\beta$ -Pinene	1.1 $\pm$ 0.2 <sup>a</sup>	0.4 $\pm$ 0.0 <sup>b</sup>	0.4 $\pm$ 0.0 <sup>b</sup>
Myrcene	0.6 $\pm$ 0.0 <sup>a</sup>	0.2 $\pm$ 0.0 <sup>b</sup>	0.2 $\pm$ 0.0 <sup>b</sup>
<i>p</i> -Cymene	1.6 $\pm$ 0.2 <sup>a</sup>	0.8 $\pm$ 0.0 <sup>b</sup>	0.7 $\pm$ 0.1 <sup>b</sup>
Limonene	1.9 $\pm$ 0.2 <sup>a</sup>	0.9 $\pm$ 0.1 <sup>b</sup>	0.8 $\pm$ 0.1 <sup>b</sup>
$\gamma$ -Terpinene	5.0 $\pm$ 0.5 <sup>a</sup>	3.3 $\pm$ 0.2 <sup>b</sup>	2.8 $\pm$ 0.3 <sup>b</sup>
Linalool	71.7 $\pm$ 1.4 <sup>b</sup>	77.0 $\pm$ 1.0 <sup>a</sup>	77.4 $\pm$ 1.1 <sup>a</sup>
Camphor	4.2 $\pm$ 0.1 <sup>b</sup>	4.8 $\pm$ 0.0 <sup>a</sup>	4.8 $\pm$ 0.1 <sup>a</sup>
Borneol	1.2 $\pm$ 0.0 <sup>b</sup>	1.6 $\pm$ 0.0 <sup>a</sup>	1.5 $\pm$ 0.1 <sup>a</sup>
$\alpha$ -Terpineol	0.4 $\pm$ 0.0 <sup>b</sup>	0.5 $\pm$ 0.0 <sup>a</sup>	0.5 $\pm$ 0.0 <sup>a</sup>
Carvone	n.dc.	0.2 $\pm$ 0.0 <sup>a</sup>	0.2 $\pm$ 0.0 <sup>a</sup>
Linalyl acetate	2.9 $\pm$ 0.2 <sup>b</sup>	4.1 $\pm$ 0.2 <sup>a</sup>	4.2 $\pm$ 0.3 <sup>a</sup>
2-(E)-decenal	n.dc.	n.dc.	0.1 $\pm$ 0.0
Geranyl acetate	3.0 $\pm$ 0.1 <sup>b</sup>	4.8 $\pm$ 0.3 <sup>a</sup>	4.9 $\pm$ 0.3 <sup>a</sup>

Means in the same line with the same superscript letter (a–b) are not significantly different at  $P < 0.05$

n.d. Not detected

press cakes was determined through DPPH analysis and results varied from 3.7 to 5.1  $\mu\text{mol}$  of TE per g of press cake (Table 3), though still representing rather low antioxidative activity. Press cakes from trials 8 and 10 revealed a better antioxidant capacity due to the decrease in pressing temperature, while for these two press cakes, the antioxidant capacities were shown not to be significantly different. The antioxidative activity of press cakes from trials 2 and 4 was also determined after they had further been subjected to hydrodistillation. This consistently led to a reduction in the antioxidant capacity (Table 3), possibly caused by the high temperatures applied through hydrodistillation for 5 h. However, this may also be a consequence of the migration of some water-soluble antioxidants such as anthocyanins to the hydrodistillation water phase.

The coriander press cakes obtained displayed significant residual oil contents of 15–18 % (Table 1), which may be unfavorable for some applications. However, this does not impose any disadvantages when the cakes are converted into usable energy through combustion, gasification or pyrolysis, possibly rendering the extrusion process self-supporting [45, 46]. In addition, the press cakes could be used as a renewable resource for the production of value-added agromaterials through thermo-pressing [21, 47–51]. Finally, they may be interesting for the biocomposite industry as they could be incorporated into

biodegradable polymers such as polycaprolactone and polylactic acid where they could act as reinforcing fillers [52, 53].

## Conclusion

The application of twin-screw extrusion for the extraction of vegetable oil from coriander fruits was shown to be effective and resulted in considerable oil recoveries while maintaining low foot contents. The extrusion operating conditions were found to have a significant impact on the oil recovery and energy consumption of the extruder. The highest extraction efficiencies were achieved with a pressing zone situated immediately after the filter and containing -33 mm reverse pitch screws that are 50 mm in length. Further, with a device's filling coefficient of 39.4 g/h rpm and a pressing temperature of 120 °C, an oil recovery of 47 % and a low foot content of 11 % were obtained. The press cake resulting from this trial displayed a residual oil content of 17 %, while a residual oil content of 15 % was reached with an increased device's filling coefficient (47.1 g/h rpm) and a decreased pressing temperature (80 °C). Here, however, the filtrate's foot content was considerable at 18 %, leading to a decrease in the oil recovery based on the filtrate. Because part of the essential oil is co-extracted by mechanical pressing with the vegetable oil, twin-screw extrusion of coriander fruits consistently produced an agreeably scented vegetable oil of good quality with less than 1.5 % free fatty acids and a high petroselinic acid content of 73 %. Potential applications of the press cakes obtained concern the essential oil content, which is rich in linalool, its antioxidant activity and the possible use as a biocomposite.

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