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Characterization and effect of optimized spray-drying conditions on spray-dried coriander essential oil

Muhammad Abdul Rahim [a,b], Muhammad Imran [a], Faima Atta Khan [a], Fahad Al-Asmari [c], Joe M. Regenstein [d,*], Suliman Yousef Alomar [e], Imtiaz Hussain [f], Elena Bartkiene [g,h], João Miguel Rocha [i,j,k,**]

[a] *Department of Food Science, Faculty of Life Sciences, Government College University, Faisalabad 38000, Pakistan*
[b] *Department of Food Science & Nutrition, Faculty of Medicine and Allied Health Sciences, Times Institute, Multan, 60700, Pakistan*
[c] *Department of Food and Nutrition Sciences, College of Agricultural and Food Sciences, King Faisal University, Al-Ahsa 31982, Saudi Arabia*
[d] *Department of Food Science, Cornell University, Ithaca, NY, USA*
[e] *Zoology Department, College of Science, King Saud University, Riyadh 11451, Saudi Arabia*
[f] *Department of Food Science and Technology, Faculty of Agriculture, University of Poonch, Rawalakot, Azad Kashmir, Pakistan*
[g] *Institute of Animal Rearing Technologies, Lithuanian University of Health Sciences, Tilzes St. 18, LT-47181 Kaunas, Lithuania*
[h] *Department of Food Safety and Quality, Lithuanian University of Health Sciences, Tilzes St. 18, LT-47181 Kaunas, Lithuania*
[i] *Universidade Católica Portuguesa, CBQF—Centro de Biotecnologia e Química Fina—Laboratório Associado, Escola Superior de Biotecnologia, Rua Diogo Botelho 1327, 4169-005 Porto, Portugal*
[j] *Laboratory for Process Engineering, Environment, Biotechnology and Energy (LEPABE), Faculty of Engineering, University of Porto (FEUP), Rua Dr. Roberto Frias, s/n, 4200-465 Porto, Portugal*
[k] *Associate Laboratory in Chemical Engineering (ALiCE), Faculty of Engineering, University of Porto (FEUP), Rua Dr. Roberto Frias, s/n, 4200-465 Porto, Portugal*

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ABSTRACT

Coriander (*Coriandrum sativum* L.) essential oil (CEO) has many beneficial features, including antimicrobial and antifungal properties along with good aroma. It also has an important role in food processing and preservation. However, CEO is highly volatile and sensitive to external factors (heat, light and oxygen), as well as susceptible to lipid oxidation due to environmental and general processing conditions. This limits water solubility, making it difficult to incorporate CEO into aqueous food matrices, which further limits their industrial application. Spray-drying encapsulation may prevent CEO oxidation, increase CEO oxidative stability and improve their physico-chemical properties. In this study, spray-dried CEO (SDCEO) was prepared using a mini laboratory-scale spray-dryer and the processing conditions were optimized. The SDCEO were characterized in respect to free fatty acids (FFA), peroxide values (PV), fatty acid (FA) profiles, Fourier-transform infrared spectroscopy (FTIR) and physical morphology by scanning electron microscopy (SEM). Results indicated that the maximum value of FFA, PV, fatty acid composition (including petroselinic, linoleic and oleic acids) in SDCEO were observed at the following spray-drying conditions: an inlet-air temperature (IAT) of 140 °C, needle speed (NS) of 2 s and the wall-material (WM) at 25%. The minimum values were observed at an IAT of 180 °C, NS of 4 s and WM of 30%. Analysis of variance and the interaction effects of independent factors showed that IAT and WM significantly positively influenced the response for good oxidative stability. Thus, SDCEO is likely to be used as a natural active ingredient in the food processing, cosmetic, nutraceutical and pharmaceutical industries with high stability, and may be stored for a long time without evaporation or oxidation.

Abbreviations: ANOVA, analysis of variance; BBD, Box-Behnken design; C═O, carboxyl groups; COD, chemical oxygen demand; CEO, coriander essential oil; CS, coriander seeds; DF, degree of freedom; EFA, essential fatty acids; EO, essential oil; FA, Fatty acid; FAME, Fatty acid methyl esters; FTIR, Fourier-transform infrared spectroscopy; FFA, free fatty acids; GC-MS, gas-chromatography/mass-spectrometry; IAT, Inlet-air temperature; LA, linoleic acid; MD, Maltodextrin; MS, mean square; MW, molecular weight; MUFA, monounsaturated fatty acids; NS, needle speed; OA, oleic acid; PV, peroxide values; PA, petroselinic acid; PUFA, polyunsaturated fatty acids; SEM, scanning electron microscope/microscopy; SDCEO, spray-dried coriander essential oil; WM, wall-material.

* Corresponding author.
** Corresponding author at: Universidade Católica Portuguesa, CBQF—Centro de Biotecnologia e Química Fina—Laboratório Associado, Escola Superior de Biotecnologia, Rua Diogo Botelho 1327, 4169-005 Porto, Portugal.
E-mail addresses: jmr9@cornell.edu (J.M. Regenstein), jmfrocha@fc.up.pt (J.M. Rocha).

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1. Introduction

Medicinal and aromatic plants have been used as traditional herbs for multiple purposes including the modern food industry because of their health benefits along with their processing benefits such as antioxidant activity. For more than 80% of the world's population, medicinal and aromatic plants are often easy to access, low in cost and easy to prepare (Bajaj, 2012). Coriander (*Coriandrum sativum* L.) seeds (CS) contain beneficial vitamins, proteins, minerals, fats and phytochemicals. Coriander seeds are being used as a supplement in cosmetics and nutraceuticals, as well as for drug development and food processing including dyeing (Ahmad et al., 2021; Imran et al., 2023; Maroufi et al., 2010; Rahim et al., 2023a). Coriander belongs to the apiaceae family, its fruits and fresh leaves are used as a flavoring agent and as a medicinal (Imran et al., 2023; Ramadan, 2023). Coriander seeds are often used as a spice, due to their spicy and citrus flavors, and medicinally for diabetes, stomach pain, immunity disorders, constipation, skin disorders, diarrhea, flatulence, cardiovascular diseases, nausea, acute inflammation and many other infections (Kaefer and Milner, 2008). Coriander seeds contain approximately 0.2 to 1.5% of volatile oil and 13 to 20% of fat oil (Olle et al., 2010; Momin et al., 2012). Coriander seeds contain up to 1% essential oil (EO) (Eikani et al., 2007). Various extraction methods have been used to obtain coriander essential oil (CEO), but the screw-press is generally the most efficient and gives higher yields (Rahim et al., 2022; Rahim et al., 2023b). The fatty acid (FA) of CEO is mainly composed of monounsaturated fatty acids (MUFA) and polyunsaturated fatty acids (PUFA), which together represent near 70 to 75% of the total FA content. MUFA and PUFA potentially have a protective role against many of the disorders listed above and include a decrease of the plasma concentration of glucose and insulin in type-2 diabetes (López-Miranda et al., 2006; Minihane and Lovegrove, 2006; Bhuiyan et al., 2009; Dinu et al., 2018).

Lipid oxidation is the most common issue with herbal oil fatty acids, which can cause off-flavors and form toxins during long-term storage. Therefore, the preservation of essential oils against heat, light and moisture is important from both the health and economic perspectives. Various techniques have been used to encapsulate essential oils in foods, alternative medicines, natural therapies and cosmetic applications (Shaaban and Farouk, 2022). Spray-drying has been used for the encapsulation of essential oils due to its simplicity, wide availability, low-price, ease of handling and adjustable microcapsule size. It is a solution for PUFA contain oils as it allows the emulsion to be converted into a powder, while preserving its valuable properties. Due to the large surface area of the droplets, the water evaporates almost immediately and the droplets turn into particles with a satisfactory stability (Sosnik and Seremeta, 2015; Santos et al., 2018; Imran et al., 2023). In this research study, spray-drying conditions were optimized for the encapsulation of CEO. Fourier transform infrared spectroscopy (FTIR) was used to characterize CEO prepared using a cold-pressed oil extraction and spray-dried process (spray-dried coriander essential oil, SDCEO) for further encapsulation and improved bioavailability. Free fatty acids (FFA), peroxide values (PV), FA compositions, FTIR and the physical morphology of SDCEO using scanning electron microscopy (SEM) were measured for the capsules.

2. Materials and methods

Fresh coriander seeds (CS, eagle green) were purchased from the Ayub Agricultural Research Institute (Faisalabad, Punjab, Pakistan). Coriander seeds were cleaned and washed with tap water to remove debris and dust particles, then dried at room temperature (26 ± 1 °C) and stored at room temperature for a maximum of one wk.

2.1. Extraction of coriander essential oil (CEO)

CEO was extracted from coriander seeds using a mini oil press model 6YL-550 (Wuhan Acme Agro-Tech Co., Ltd., Zhengzhou, Henan, China) according to Rahim et al., 2022a,b. Coriander seeds were poured slowly into the feed hopper and a cylindrical barrel continuously pushed seeds forward. The proteins were denatured with the combined impact of the heat and friction generated in the extruder barrel (Ashraf et al., 2020). Oil was collected through the holes (overall dimensions 311 ×179×360 mm) in the collection chamber. The extracted oil was sedimented using centrifugation (Megafuge 8 R Small Benchtop Centrifuge, Thermo Fisher Scientific, Dreieich, Germany) at 30,300g (3000 rpm with a fixed angel rotor) for 30 min at room temperature and a high quality oil was obtained using a series of further processing steps, chiefly degumming, neutralization, bleaching and deodorization at 25–28 °C. Briefly, CEO was heated to 90 °C and thoroughly mixed with 0.20% of phosphoric acid (85% concentration) (Sigma Aldrich, St. Louis, MO, USA) using a homogenizer (FSH-2A, Jiangsu Jinyi Instrument Technology, Changzhou, Jiangsu, China) at 1000 rpm for 5 min. Then, the oil mixture was centrifuged at 30,300 x g for 10 min at room temperature to separate the hydratable phosphatides, specifically phosphatidyl-choline and phosphatidyl-inositol, and other water-soluble impurities. Sodium hydroxide (20%, w/v) was added in the degumming oil to neutralize the FFA and stirred at 180 rpm for 10 min. After stirring, the oil mixture was heated at 60 °C and stirred gently for 10 min. The oil mixture was allowed to stand for approximately 5 – 10 min and the oil was centrifuged at 30,300 x g for 10 min at 25–28 °C to remove the soap. To absorb the residual soap and phospholipids, the neutralized oil was treated with 2% acidic silica (Sigma Aldrich) at room temperature. A predetermined amount of bleaching earth (Sigma Aldrich) was added to the oil mixture and stirred at 250 rpm for 5 min. Then, the acidic silica and bleaching earth were removed from the oil by filtering using Whatman litmus paper (20 to 50 μm) (Cytiva, Marlborough, MA, USA). The final oil was heated at 200 °C for 10 min and allowed to cool at room temperature (Chew and Nyam, 2020). It was then stored in clear glass bottles with a metal cap at room temperature with light access (sunlight and artificial light) for a maximum of two weeks.

2.2. Characterization of coriander essential oil (CEO)

2.2.1. Free fatty acids (FFA)

The FFA were determined according to the AOCS (2017) method number Ca 5a-40 with minor modifications. CEO (10 g) in a 250 mL conical flask was mixed with 50 mL of ethyl alcohol (95%, v/v) at room temperature. Phenolphthalein (1%, 50 μL) indicator in 95% ethanol was added. Furthermore, sodium hydroxide (0.01 N) was added quantitatively with constant stirring until the color changed from colorless to pink. Total FFA was calculated using Eq. (1).

$$\text{FFA} = \frac{\text{Volume of NaOH} \times \text{N} \times 28.2}{\text{Weight of sample}} \quad (1)$$

where N is the normality of NaOH, 28.2 is the molecular weight (MW) of oleic acid, i.e., FFA are measured as oleic acid equivalences.

2.2.2. Peroxide values (PV)

The PV was estimated using the official AOAC method 965.33 (2000) with minor modifications (AOAC, 2000). Briefly, 2 g of CEO was added to a 250 mL conical flask, 30 mL of acetic acid and chloroform solution (1:1, v/v) were mixed for 1 to 2 min. One milliliter of potassium iodide (10%, w/v) was added and mixed for 1 to 2 min. Then, 30 mL of distilled water was added and stirred for 1 min. For the titration, 0.5 mL of starch solution (1%) (high amylose cornstarch, Rafhan Maize Products Co. Ltd., Faisalabad, Punjab, Pakistan) was used as an indicator and mixed until the color changed to a dark blue color. Sodium thiosulfate (0.01 N) was added quantitatively and stirred until the color changed to yellow. PV was estimated using Eq. (2):

$$\text{Peroxide value} = \frac{\text{Volume of NaThiosulfate} \times \text{N} \times 1000}{\text{Weight of sample}} \quad (2)$$

2.2.3. FA composition

FA methyl esters (FAME) were estimated using gas-chromatography/mass-spectrometry (GC-MS) (Model 7890-B, Agilent Technologies, Santa Clara, CA, USA) according to Kostik et al. (2013) with minor modifications. Four to five drops of CEO were poured into a chemical oxygen demand (COD) test tube (Thomas Scientific, Swedesboro, NJ, USA). Then, 2 mL of 1 M NaOH in 100% methanol was added. The plastic caps were slightly tightened and the tubes placed in a water bath at 90 °C until they became colorless. Boron trifluoride (3 mL) was added and tubes reheated at 90 °C for 10 to 12 min. After cooling to room temperature, 3 mL of hexane was added. The tubes were vigorously shaken and two phases obtained. The supernatant was put into a clear glass vial for GC. Peak areas were estimated using concentration assuming an equal response for each peak or percent of the total area. The temperature of the injector and flame ionization detector were set at 200 and 250 °C, respectively, using a SP-2560 capillary column (100 m x 0.25 mm id) (Agilent Technologies, model 7890 B), with a flow-rate for He, H_2 and O_2 of 2, 4 and 40 mL, respectively. A FAME 37 (Supelco, St. Louis, MO, USA, a C37 alkane) and SLB-IL111 (Merck, St. Louis, MO, USA) standards were used as internal standards. Fame 37 was used for the fatty acid profile and SLB-IL111was used to distinguish isomers.

2.2.4. Fourier-transform infrared spectroscopy (FTIR)

FTIR was done in the range of 650 – 4000 cm^{-1} using an Agilent Technologies Cary 630. A small drop of sample was dropped onto a flat glass plate. Another plate was placed on top and a quarter turn made to get a good even film. The plate was placed in the sample holder and the spectrum was run at a resolution of 4 cm^{-1} (Berna, 2017).

2.2.5. Spray-drying of coriander essential oil (CEO)

Multivariate optimization (Rahim et al., 2022; Rahim et al., 2023b) was used to evaluate the optimal processing conditions for drying and encapsulation of CEO using a mini spray-drier (Model: TPS-15, TOPTION, Shanghai, China). An emulsion was progressively formed by combining 97 mL of distilled water with 3 mL of toluene. In another beaker, 15 mL of CEO and 85 mL of the above solution were stirred using a magnetic stirrer at 800 rpm for 20 min. Egg lecithin (1%, Sigma Aldrich) and 15 g gum Arabic (Sigma Aldrich) were added and stirred for 10 min. The solution was homogenized using the homogenizer at 1500 rpm for 25 min

2.2.6. Characteristics of spray-dried coriander essential oil (SDCEO)

FFA, PV, FA composition and FTIR analysis were used as previously described. The SEM analysis (Cube 10, Emcraft, Kwangju, Kyonggi-do, South Korea) was used to determine the morphology. For SEM sample preparation a small amount of SDCEO was spread on one side of the conductor using double-sided adhesive tape, such as carbon or copper tape (Agar Scientific, Stansted, Essex, UK). The sample was placed inside the SEM chamber under vacuum and the instrument operated at 15 kV.

2.2.7. Statistical analysis

The response surface method was used to calculate the optimal values of the standard deviation factors. The data were analyzed using Design Expert® (11.1.2.0, Minneapolis, MN, USA) and the level of significance was determined at 5% ($p \leq 0.05$). Analysis of variance (ANOVA) was applied to test the generality of the BBD. Modeling was done with a quadratic model and statistical significance was assessed using the desired F-statistic. The data was analyzed using the sum of squares, degree of freedom and mean square.

A Box-Behnken design (BBD) was used with three independent factors leading to 17 experimental runs, with the experiments randomly organized to minimize the impacts of unexpected variability in the observed dependent variables due to systematic errors. The inlet-air temperature (IAT), wall-material (WM) and needle speed (NS) were selected with high and low coded levels (Table 1). The independent variables and the coded levels were selected based on previous work by Rahim et al. (2022).

3. Results and discussion

3.1. Characterization of coriander essential oil (CEO)

The concentration of FFA in the CEO was 5.3 ± 0.3%, and the PV was 0.92 ± 0.05 meq O_2/kg. The fatty acid composition of CEO showed that it contained 73 ± 2% petroselinic acid (PA), 12.3 ± 0.5% linoleic acid (LA), 5.6 ± 0.2% oleic acid (OA). These results were consistent with Izgi (2020). The latter found 1.03% FFA in CEO and a PA of 72.6%, LA of 14% and OA of 6%. The quality of CEO extracted from coriander fruits using a twin screw extruder was acceptable quality with < 1.5% of FFA and 73% more PA content (Uitterhaegen et al., 2016). Sriti et al. (2012) reported that CEO extracted using a twin-screw extruder was of good quality as compared to CEO extracted using solvent extraction. Ghazanfari et al. (2020) used different methodologies to obtain CEO. Their PV was higher in all samples over 8 days of storage at 60 °C. Agostoni et al. (2013) reported that the amount of PA ranged from 60 to 75% and OA acid from 8 to 15% in CEO.

3.2. Fourier-transform infrared spectroscopy (FTIR) of coriander essential oil (CEO)

The FTIR of the CEO is shown in Fig. 1. CEO contained more than 30 compounds. The FTIR spectrum of native CEO showed a broad peak at 2954 cm^{-1} due to the presence of CH_3, CH_2 and CH bonds, indicating that there were alkanes in CEO, while the peaks at 1748 cm^{-1} showed the presence of carboxyl groups (C═O). The peak shows the occurrence of a dimer carboxylic acid. The peak at 1459 cm^{-1} is the C-C stretch of aromatic compounds. Sulfonyl chloride had a peak value of 1377 cm^{-1}. Furthermore, groups such as C-H, O-H and C═O were observed at 1100–700 cm^{-1} and confirmed the presence of ester groups (Ali et al., 2015). Asif et al. (2013) reported that there was little difference in the chemical composition of CEO obtained from different varieties. The FTIR was consistent with the CEO results of Senrayan and Venkatachalam (2019).

3.3. Characteristics of spray-dried coriander essential oil (SDCEO)

3.3.1. FFA

The effect of the independent variables on the FFA of the SDCEO is shown in Table 2. The IAT, NS and WM values showed that the maximum FFA (7.8 ± 0.4%) were observed at low IAT and WM, while the minimum FFA (4.2 ± 0.2%) were observed at high IAT and NS values. The amount of FFA was significantly reduced with increased IAT and WM. The ANOVA for the optimized model is shown in Table 3. The model was highly significant and the lack of fit was statistically non-significant, suggesting that the model was suitable with the chosen dependent variables. A high coefficient of calculation ($R^2 = 0.99$) and the difference between the adjusted R^2 (0.98) and predicted R^2 (0.93) were small, thus showing the reasonable fit of the model to the response variables. ANOVA indicated that IAT was a highly significant independent variable affecting the dependent variables. This was confirmed by

Table 1
Experimental ranges and their coded levels of parameters chosen for the Box-Behnken design (BBD) factorial design.

Parameter (Unit)	Symbol	Range and coded level		
		Low (−1)	Medium (0)	High (+1)
Inlet air temperature (°C)	IAT	140	160	180
Needle speed (s)	NS	2	4	6
Wall material (%)	WM	20	25	30

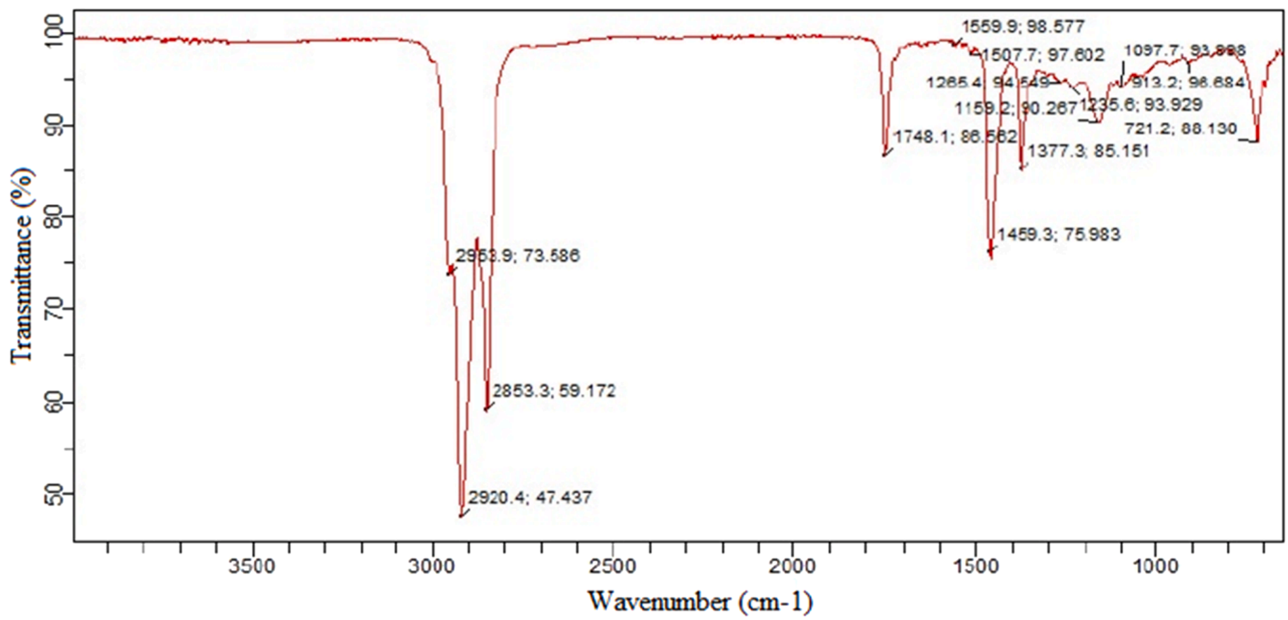

Fig. 1. Fourier-transform infrared spectroscopy (FTIR) of coriander essential oil (CEO).

Table 2
Effect of spray-drying operating conditions on the mean values (mean values ± standard deviation) of free fatty acid (FFA) composition, peroxide values (PV), petroselinic acid (PA), linoleic acid (LA) and oleic acid (OA) of spray-dried coriander essential oil (SDCEO).

Runs	Spray-drying operating factor			Response factor				
	IAT (°C)	NS (s)	WM (%)	FFA (%)	PV (meq O_2/kg)	PA (%)	LA (%)	OA (%)
1	160 (0)	6 (+1)	20 (−1)	6.2 ± 0.3^{g}	1.0 ± 0.1^{m}	63 ± 3^{c}	8.1 ± 0.4^{ef}	4.1 ± 0.2^{k}
2	160 (0)	6 (+1)	30(+1)	5.3 ± 0.3^{h}	0.81 ± 0.06^{mn}	62 ± 3^{d}	7.3 ± 0.3^{fg}	3.8 ± 0.2^{kl}
3	140 (−1)	4 (0)	30 (+1)	6.9 ± 0.3^{fg}	1.1 ± 0.1^{m}	64 ± 3^{bc}	8.5 ± 0.4^{ef}	4.5 ± 0.2^{hk}
4 (C_1)	160 (0)	4 (0)	25 (0)	6.1 ± 0.3^{g}	0.92 ± 0.07^{m}	62 ± 3^{c}	7.8 ± 0.3^{f}	4.6 ± 0.3^{hk}
5 (C_2)	160 (0)	4 (0)	25 (0)	6.2 ± 0.3^{g}	0.91 ± 0.06^{m}	62 ± 3^{c}	7.8 ± 0.4^{f}	4.5 ± 0.3^{hk}
6	160 (0)	2 (−1)	30 (+1)	5.8 ± 0.3^{gh}	0.89 ± 0.06^{mn}	62 ± 3^{cd}	7.7 ± 0.3^{f}	3.9 ± 0.2^{kl}
7	140 (−1)	4 (0)	20 (−1)	7.5 ± 0.4^{fg}	1.3 ± 0.1^{lm}	65 ± 3^{ab}	9.0 ± 0.4^{e}	4.1 ± 0.3^{k}
8 (C_3)	160 (0)	4 (0)	25 (0)	6.1 ± 0.3^{g}	0.92 ± 0.07^{m}	62 ± 3^{c}	7.7 ± 0.3^{f}	4.6 ± 0.3^{hk}
9	180 (+1)	4 (0)	30 (+1)	4.2 ± 0.2^{k}	$0.41 \pm 0.02^{\circ}$	58 ± 3^{e}	5.1 ± 0.2^{h}	3.2 ± 0.1^{l}
10	180 (+1)	2 (−1)	25 (0)	4.9 ± 0.2^{hk}	0.64 ± 0.04^{no}	60 ± 3^{de}	6.6 ± 0.3^{g}	3.5 ± 0.2^{l}
11	140 (−1)	2 (−1)	25 (0)	7.8 ± 0.4^{f}	1.4 ± 0.1^{lm}	66 ± 3^{a}	9.6 ± 0.4^{e}	5.8 ± 0.3^{gh}
12	160 (0)	2 (−1)	20 (−1)	6.7 ± 0.3^{g}	0.85 ± 0.06^{mn}	63 ± 3^{bc}	8.3 ± 0.4^{ef}	4.4 ± 0.2^{k}
13	180 (+1)	6 (+1)	25 (0)	4.5 ± 0.2^{hk}	0.57 ± 0.03^{no}	59 ± 3^{de}	6.3 ± 0.3^{g}	3.3 ± 0.2^{l}
14 (C_4)	160 (0)	4 (0)	25 (0)	6.2 ± 0.3^{g}	0.93 ± 0.07^{m}	62 ± 3^{c}	7.8 ± 0.3^{f}	4.6 ± 0.2^{hk}
15	140 (−1)	6 (+1)	25 (0)	7.3 ± 0.3^{fg}	1.3 ± 0.1^{lm}	65 ± 3^{b}	8.8 ± 0.4^{e}	4.6 ± 0.3^{hk}
16 (C_5)	160 (0)	4 (0)	25 (0)	6.1 ± 0.3^{g}	0.90 ± 0.06^{m}	62 ± 3^{c}	7.8 ± 0.3^{f}	4.6 ± 0.3^{hk}
17	180 (+1)	4 (0)	20 (−1)	5.2 ± 0.2^{h}	0.74 ± 0.05^{n}	60 ± 3^{d}	7.1 ± 0.3^{fg}	3.7 ± 0.2^{kl}

the significant linear and quadratic term of the model.

The mutual interactions of IAT, NS and WM are shown in Table 3. Koç et al. (2015) similarly showed that the increasing spray-dried IAT (110 to 118 °C) had a variable effect, leading to increased FFA of spray-dried olive oil microcapsules. Similarly, Rahim et al. (2022) showed that the concentration of FFA in spray-dried microcapsules of chia and fish oil blends were significantly increased with decreasing IAT (185 to 125 °C) of spray drying. Laohasongkram et al. (2011) also reported that the increase in the air drying temperature lead to increased FFA. Özyurt et al. (2022) reported that IAT was not significant for FFA for spray-dried microencapsulated fish oils with maltodextrin (MD) and sodium caseinate.

SDCEO = Spray-dried coriander essential oil; IAT = Inlet-air temperature; NS = Needle speed; WM= Wall-material; FFA= Free fatty acids; PV= Peroxide value; PA= Petroselinic acid; LA= Linoleic acid; OA= Oleic acid; C1–C5 represent the central points of the BBD analysis; a–o = different superscripts in a column differ significantly ($p \leq 0.05$).

3.3.2. *Peroxide values (PV)*

Peroxide values is a widely used quantitative assay for estimating the primary oxidation of lipids to yield lipid peroxidation products after processing. Lipid peroxidation reduces the bioavailability of essential fatty acids (EFA) (Wang et al., 2023). The oxidative stability of CEO was improved using spray-drying. Results indicated that the lowest PV of SDCEO was (0.41 ± 0.02 meq O_2/kg) at 140 °C and the highest (1.4 ± 0.1 meq O_2/kg) at 180 °C (Table 2). The oxidative stability of SDCEO was significantly improved compared to CEO. PV increased with decreasing IAT and NS. The ANOVA results are shown in Table 3. The R^2 and adjusted R^2 were both higher than 0.9, and the difference between predicted R^2 and adjusted R^2 suggested that the interaction effects of IAT and WM were statistically significant for PV (Fig. 2). This was consistent with the higher PV which occurred due to the additional energy provided at higher IAT (180 °C) of spray drying (Aghbashlo et al., 2013). Tonon et al. (2011) found that an increasing drying temperature of a spray dryer resulted in higher lipid oxidation. According to Ferreira et al. (2016), the PV of palm oil encapsulated in gum Arabic and whey protein concentrate increased significantly with increasing spray-drying

Table 3
Analysis of variance (ANOVA) of spray-drier operating conditions effect on dependent factors of spray-dried coriander essential oil (SDCEO).

Source of variation		DF	FFA (%)		PV (meq O_2/kg)		PA (%)		LA (%)		OA (%)	
			MS	p-value	MS	p-value	MS	p-value	MS	p-value	MS	p-value
Model		9	1.82	< 0.01	0.11	0.01	8.21	< 0.01	1.58	< 0.01	0.71	0.04
Linear	A-IAT	1	14.3	< 0.01	0.95	< 0.01	63.8	< 0.01	12.5	< 0.01	4.68	< 0.01
Effects	B-NS	1	0.45	0.04	0.01	0.06	1.53	0.01	0.34	0.04	0.34	0.01
	C-WM	1	1.45	< 0.01	0.06	0.07	7.41	< 0.01	1.07	0.02	0.38	0.07
Interaction	AB	1	0.02	0.65	0.04	0.77	0.12	0.20	0.06	0.01	0.21	0.04
Effects	AC	1	0.04	0.01	0.04	0.09	0.30	0.06	0.07	0.09	0.02	0.94
	BC	1	0.01	1.00	0.01	0.49	0.04	0.44	0.02	0.73	0.03	0.76
Quadratic	A^2	1	0.04	0.06	0.04	0.03	0.10	0.02	0.03	0.02	0.05	0.27
Effects	B^2	1	0.01	0.04	0.03	0.42	0.13	0.01	0.07	0.09	0.10	0.14
	C^2	1	0.10	0.01	0.01	0.15	0.43	0.03	0.02	0.30	0.59	0.05
Residual		7	0.01	—	0.04	—	0.06	—	0.02	—	0.03	—
Lack of Fit		3	0.02	0.40	0.09	0.45	0.12	0.25	0.04	0.59	0.08	0.67
Pure Error		4	0.03	—	0.01	—	0.01	—	0.01	—	0.01	—
Cor. Total		16	—	—	—	—	—	—	—	—	—	—

ANOVA = Analysis of variance; SDCEO = Spray-dried coriander essential oil; FFA= Free fatty acids; PV = Peroxide value; PA = Petroselinic acid; LA = Linoleic acid; OA = Oleic acid; DF = Degree of freedom; MS = Mean square; A = Inlet-air temperature; B = Needle speed; C=Wall-material.

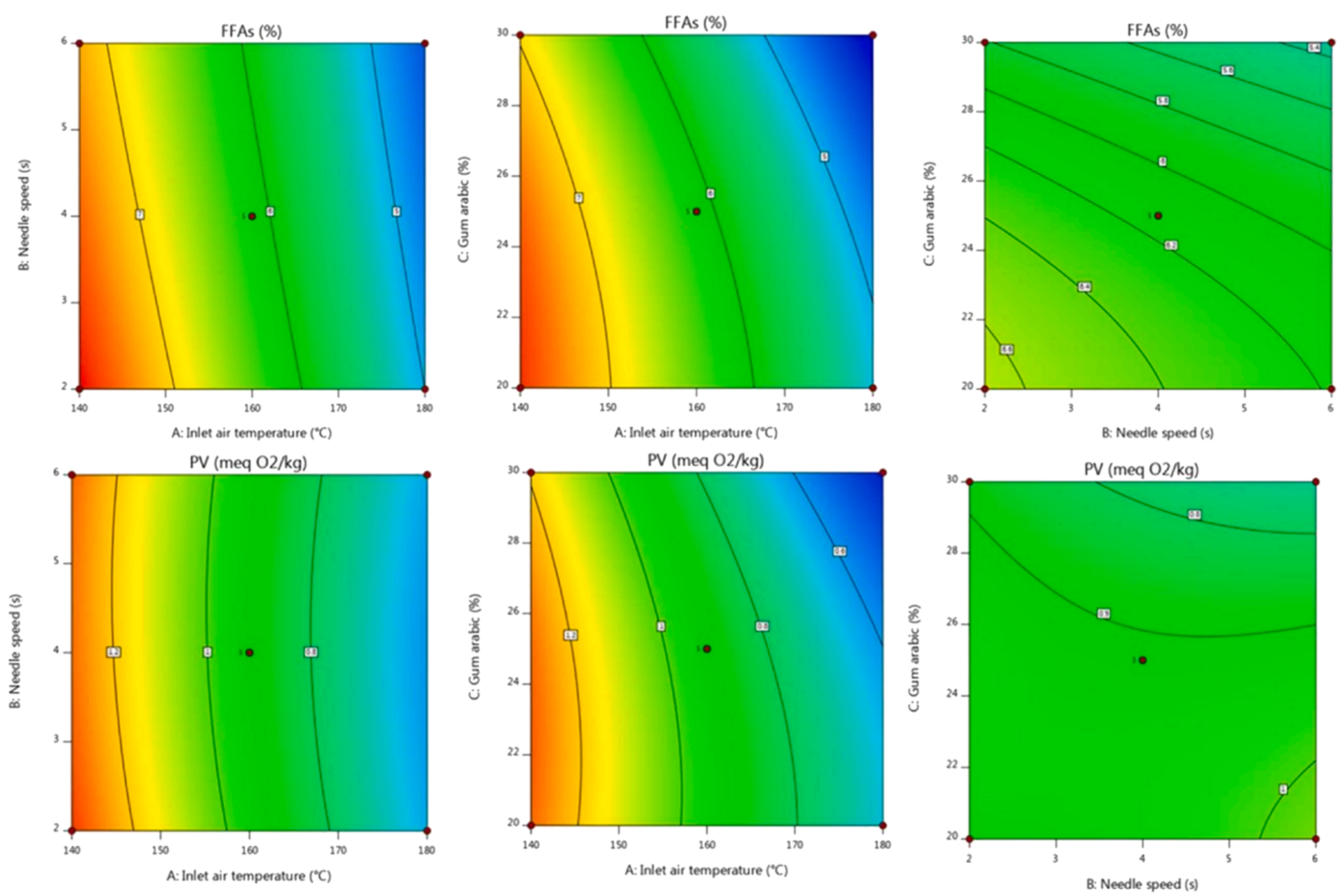

Fig. 2. Contour plots for free fatty acids (FFA) and peroxide values (PV) of spray-dried coriander essential oil (SDCEO).

temperature. Rahim et al. (2022) also showed that the use of a higher IAT increased the PV. Caihong et al. (2023) also found that the PV decreased when the spray-drying temperature was lowered during encapsulation.

3.3.3. Fatty acid (FA) composition

The FA composition of SDCEO was estimated using GC-MS. The PA decreased with increasing temperature. Reduction in the concentration of FA profile may be due to oxidation during the spray drying process at higher temperatures. As seen in Table 3, the p-value was lower than 0.001, which confirms the significance of the model and difference between the predicted and adjusted R^2 was lower than 2. The p-values of IAT, NS and WM were lower than 0.05, which shows their statistically significant linear effects. The p-value of the lack of fit was statistically non-significant, thus indicating that the model had a good fit with the response variables. Interactions between two independent variables indicated that the IAT and WM significantly impacted the response variable, while interaction effect between IAT and NS, and NS and WM were not statistically significant for PA (Fig. 3). The concentration of LA decreased with increased IAT and reduced NS. The p-value of the model indicated that the model was highly significant and that IAT, NS and WM were significant terms (Table 3). Similarly, the lack of fit p-value indicated a good model. The adjusted R^2 and R^2 were close to 1 because the independent variables were statistically significant along with the predicted model. IAT and NS, and IAT and WM significantly interacted. The WM and NS interaction was not significant (Fig. 3). The OA increased significantly with increasing IAT. The highest amount of OA was observed at IAT of 140 °C, NS of 4 s and WM at 30%, while the lowest amount was observed at IAT of 180 °C, NS of 2 s and WM at 25%. Table 3 indicates its coefficient of determination R^2 and adjusted R^2 for the quadratic terms were also statistically significant. The RSM contour plots are shown in Fig. 3. Mutual interactions indicated that the IAT had the most significant effect on LA. With increasing IAT and decreasing NS, the OA increased. The IAT × NS significantly influenced OA, while IAT × WM and NS × WM were not statistically significant.

Similarly, Rahim et al. (2022) showed that spray drying conditions did not significantly impact the FA composition of the spray dried microcapsules of fish and chia oil. The authors found a significant loss of EFA but the total amount of PUFA did not change significantly. This trend in FA composition was also noted in an experiment by Ogrodowska et al. (2017) who encapsulated pumpkin seed oil using spray drying. However, the observed changes were not statistically significant for PUFA losses. Lavanya et al. (2019) studied the microencapsulation of chia and fish oil by spray-drying using whey protein as a WM and showed that the FA were reduced at higher temperatures due to oxidation. Overall, spray-drying helped with the retention of the FA because higher IAT increase the crust formation rate around the oil droplets, allowing the diffusion of water. Temperature and physical

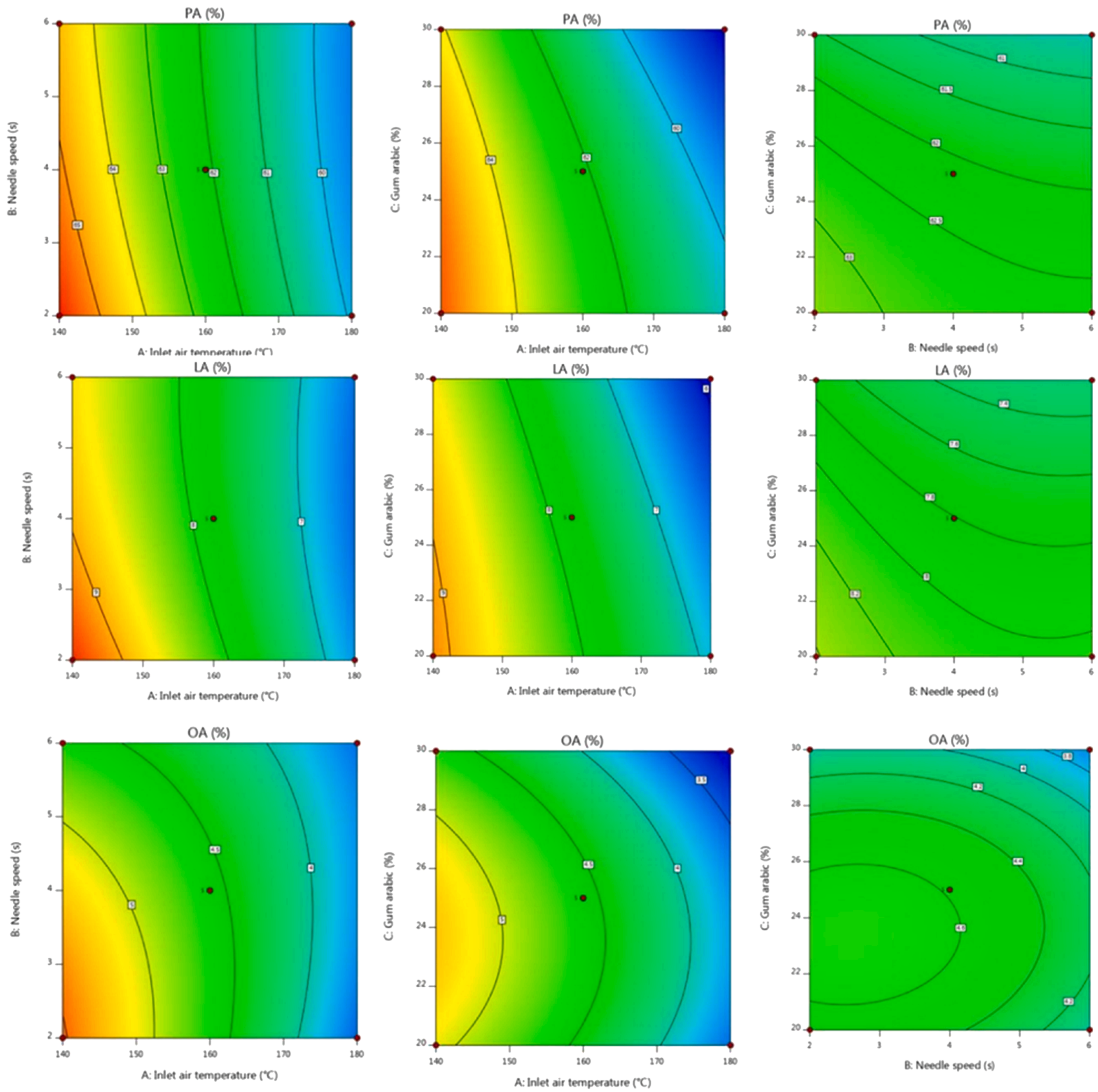

Fig. 3. Contour plots for the fatty acids profiles of spray-dried coriander essential oil (SDCEO) with the three independent spray-drying factors.

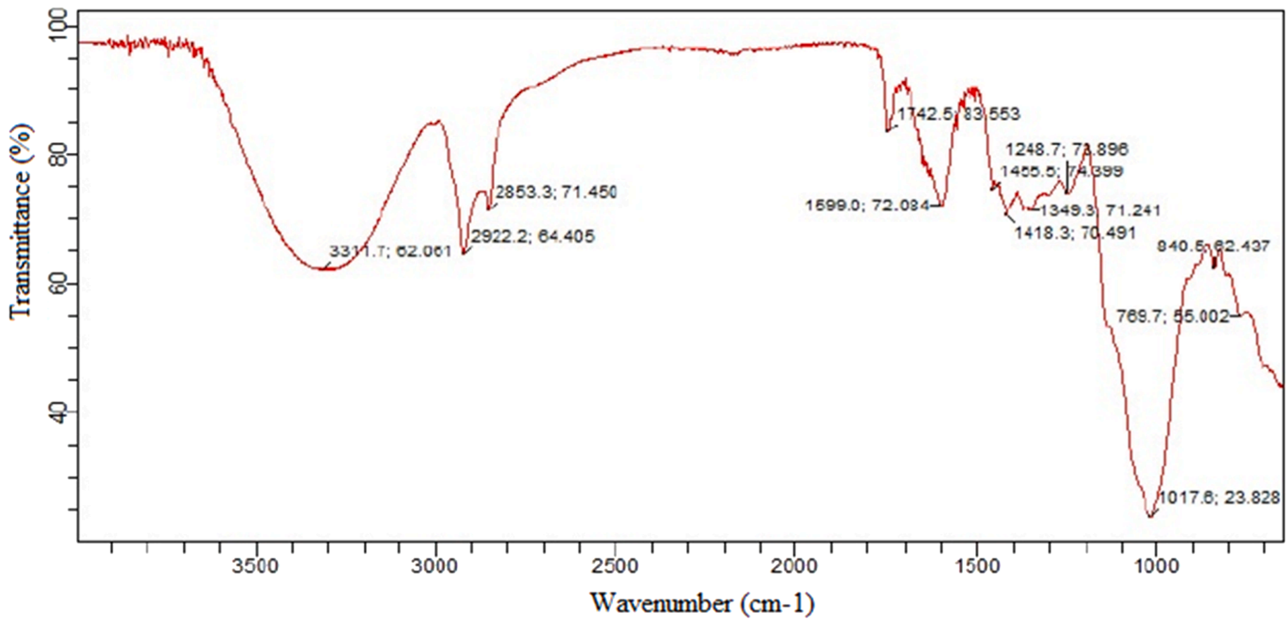

Fig. 4. Fourier-transform infrared spectroscopy (FTIR) of spray-dried coriander essential oil (SDCEO).

stress during emulsion preparation and spray-drying can affect FA composition (Gharsallaoui et al., 2007; Karthik and Anandharamakrishnan, 2016).

3.3.4. *Fourier-transform infrared spectroscopy (FTIR)*

SDCEO obtained at a low temperature (140 °C) was used for FTIR analysis due to minimum FA losses and was more stable as compared to CEO. The results of FTIR analysis is shown in Fig. 4. SDCEO showed peaks at 3311, 2922, 2853, 1742, 1599, 1455, 1418, 1349, 1248, 1017, 840 and 769 cm^{-1}. The peak with a high intensity at 3311 cm^{-1} was characteristic of carboxylic groups (CH−function on a C−C-triple bond and O−H peaks). The spectrum showed strong characteristic bands of lipids from 3000 − 2800 cm^{-1}, including peaks at 2922 and 2853 cm^{-1}, which are attributable to C–H stretching modes of the methyl ($-CH_3$) and methylene ($-CH_2$) backbones of lipids (Shi et al., 2017; Mukhametov et al., 2023). The peaks at 1742, 1599, 1455, 1418, 1349, 1248

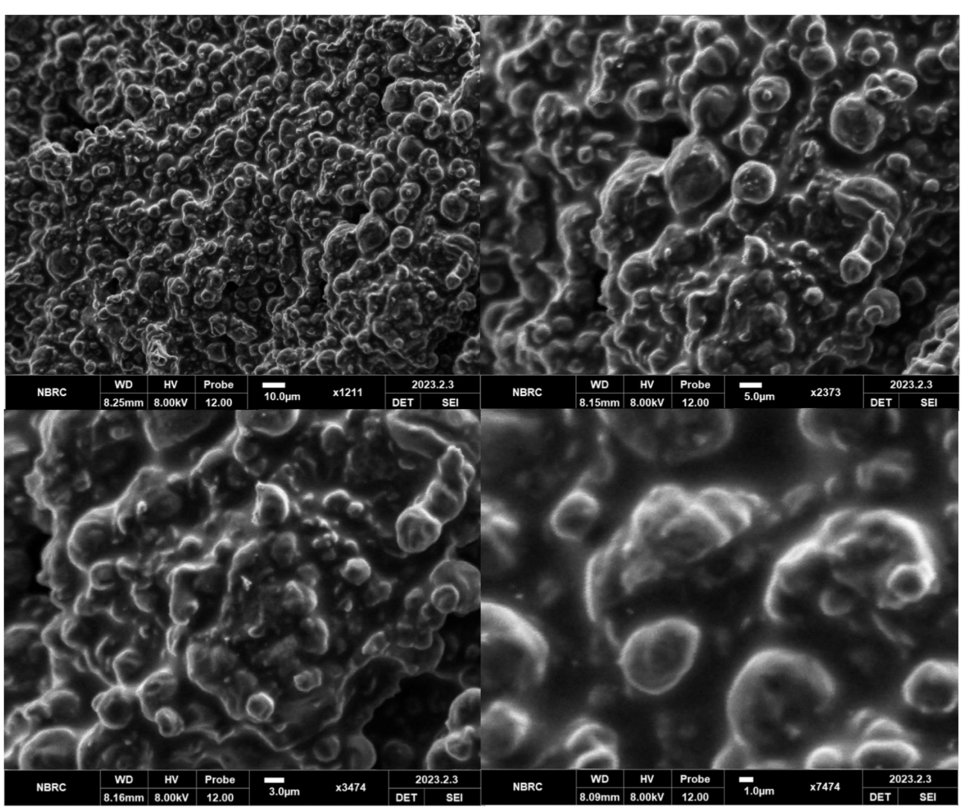

Fig. 5. Scanning electron microscope/microscopy (SEM) images of spray-dried coriander essential oil (SDCEO) at different width of 1, 3, 5 and 10 µm to determine the particle-size distribution.

and 1017 cm^{-1} characterize carbonyls. The sharp peak at 1742 cm^{-1} was assignable to C−O stretches of ester functional groups from lipids and FA. The strong single peak of the aromatic ring C−C skeleton vibration was observed at 1599 cm^{-1}. The peaks at 1455 and 1418 cm^{-1} were assigned to the methylene groups ($-CH_2$), while a strong peak at 1349 cm^{-1} was assigned to methyl groups ($-CH_3$). The peaks between 1000 and 1300 cm^{-1} were attributed to the stretching vibrations of the C−O−C of ethers and esters. The peaks below 1000 cm^{-1} were characteristic for alkenes (hydrocarbons) and arenes (aromatic hydrocarbons) groups. Duman and Kaya (2016) did FTIR of encapsulated CEO, and found peaks at 2921, 1378, 1259, 1062, and 891 cm^{-1}. The highest peak was observed at 3356 cm^{-1} with chitosan used as a WM. Bai et al. (2019) found similar peaks with spray-dried microencapsulated soybean oil powder.

3.3.5. SDCEO morphology

The image size of the SDCEO particles were all in the range of 1–10 μm (Fig. 5). The particles were semi-spherical. Similar morphology was reported by Bae and Lee (2008) for avocado oil encapsulated using spray-drying in whey protein and maltodextrin. The spray-dried powders were highly agglomerated small particles rather than discrete particles. Jiménez-Martín et al. (2015) obtained microencapsulated fish oil powders in chitosan and maltodextrin. They showed the powder had a spherical shape. Yingngam et al. (2019) reported that microcapsules, both blanks and with citronella oil, had uniform morphology and spherical particles. Bellache et al. (2023) used SEM and indicated that prickly pear seed oil loaded with microcapsules of polyhydroxy butyrate-co-valerate gave spherical shaped particles.

4. Conclusions

Characterization of CEO indicated that it contained higher amounts of MUFA and PUFA. FTIR indicated that many different compounds were present. Optimization of spray-dried coriander essential oil (SDCEO) showed that maximum FA were obtained at 140 °C, while the minimum was observed at the highest temperature of 180 °C. A similar trend was observed for the peroxide values. It was concluded that IAT was a major independent factor that significantly influenced the dependence on this response factors compared to other independent factors. SEM morphology showed the semi-spherical shape of SDCEO. Thus, SDCEO can be used as an active ingredient in foods, cosmetics, drugs, nutraceuticals and the medical industries with good oxidative stability. Future studies should focus on analyzing the in vitro and in vivo of SDCEO for applications in food models.

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CRediT authorship contribution statement

Muhammad Abdul Rahim: Software, Investigation. **Muhammad Imran:** Software, Investigation. **Faima Atta Khan:** Writing – original draft, Funding acquisition, Conceptualization. **Fahad Al-Asmari:** Writing – original draft, Funding acquisition, Conceptualization. **Joe M. Regenstein:** Writing – original draft, Supervision, Formal analysis. **Suliman Yousef Alomar:** Software. **Imtiaz Hussain:** Supervision, Formal analysis. **Elena Bartkiene:** Writing – review & editing, Validation. **João Miguel Rocha:** Writing – review & editing, Validation.

Declaration of Competing Interest

none.

Data availability

Data will be made available on request.

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Data Avail ability Statement

Data is contained within the article.

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