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Original article

Enhancement of oleaster (*Elaeagnus angustifolia L.*) flour properties via instant controlled pressure drop (DIC) technique

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Summary The instant controlled pressure drop (DIC) technology represents a highly efficient and eco-friendly process that enhances the texture of food matrices. In this study, oleaster flour (OF) was subjected to DIC treatment at varying pressures (200 and 500 kPa) and processing times (30, 60, and 120 s) for the purpose of analysing its techno-functional, chemical, concentration of bioactive components, and structural properties. The results demonstrated statistically significant differences between the untreated and DIC-treated OF samples ($P < 0.05$). The application of DIC treatment resulted in a notable enhancement in the water activity, fat, ash, SDF, bound phenolic content, and antioxidant capacity of the OF ($P < 0.05$). The mineral content and structural properties were analysed in two samples of OF (DIC-treated at 200 and 500 kPa for 60 s), which were selected for their superior bioactive components in comparison to the untreated OF. The 200 and 500-kPa 60-s DIC treatment also enhanced the mineral (Cu, Na, Mn, Mg, P, Ca, Fe, and K) and structural properties of OF, thereby rendering it a more functional flour. The utilisation of the DIC method enhances the overall quality of the end product, resulting in a visually acceptable appearance, an extended shelf life and heightened nutritional content, thereby rendering the product more practical and convenient.

Keywords Bioactive component, DIC, functional, nutritious, soluble fibre.

Introduction

The instant controlled pressure drop (DIC) technology is a highly efficient and rapid process, capable of handling high temperatures and pressures. This environmentally friendly approach employs water vapour as a solvent, thereby conserving water and saving energy due to the rapidity of the extraction and heating processes (Chemat *et al*., 2017). This safe and high-quality process results in a reduction in solvent consumption and the promotion of by-product reuse (Louati *et al*., 2019). Moreover, this technology allows for the utilisation of the entire food matrix as a supplement, including phenolic compounds and dietary fibre, without the necessity for any extraction process (Rane *et al*., 2022). It is highly versatile and can be employed for a multitude of applications, including drying, decontaminating microorganisms and extracting essential oils (Mannaï *et al*., 2019).

Martínez-Meza *et al*. (2021) reported that grape stalk powder pre-treated with DIC prior to ethanol-based extraction resulted in an increased concentration of polyphenols, including gallic acid, ellagic acid, quercetin, and resveratrol. Additionally, Louati *et al*. (2019) observed that DIC application notably increased the concentrations of hesperidin from 12.10 to 65.01, rutin from 11.47 to 27, flavone from 0.006 to 0.007, and naringin from 0.0002 to 0.00032 mg/g db. Sánchez-Valdepeñas *et al*. (2015) demonstrated that the concentration of extracted compounds, including gallic acid (up to 198%), quercetin (up to 26%), ellagic acid (up to 103%), and resveratrol (up to 41%), can be significantly affected by the use of DIC, depending on the solvent employed. The findings suggest that DIC pre-treatment has a considerable impact on the properties of the products.

The oleaster is a fruit from the *Elaeagnaceae* family. It offers a wealth of medicinal, dietary, and industrial benefits, boasting a rich profile of essential fatty acids (including palmitic acid, linoleic acid, oleic acid, and stearic acid), protein (globulin and albumin), dietary fibre, vitamins (A, C, and E), and minerals (Ca, Mg, K, Fe, and Zn) (Sahan *et al*., 2015; Şahin, 2023). Its high antioxidant capacity makes it the ideal natural ingredient for enhancing the functional properties of foods (Şahin, 2023). Oleaster flour is a good addition to producing biscuits, yogurt, bread, crackers, and snacks.

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Within the scope of the study, the instant DIC methods were applied to the OF samples. This study aimed to compare the quality of OF samples with the DIC method to enhance their properties, such as the techno-functional, chemical composition, the concentration of bioactive components and microstructure properties, at different pressures (200 and 500 kPa) and processing times (30, 60, and 120 s). The pressure and time parameters for the untreated OF, determined as the control sample, were set to "0" as no treatment was applied. The findings have the potential to facilitate the development of functional food products that are both healthier and more nutritious.

Material and methods

Materials

The *Elaeagnus angustifolia L.*, commonly known as oleaster, was collected from three locations in Karaman (Turkey). It was subsequently dried in an oven at a temperature of 22–24 °C (Nüve, FN055, Ankara). The dried oleaster samples were then separated into three layers using a sieve: the exocarp, the mesocarp (oleaster flour, OF), and the endocarp. The OF, which was <300 μm, was of particular interest. The exocarp and endocarp layers were separated using a sieve.

Experimental design and statistical analysis

The OF is treated with DIC according to 2 (different pressure 200 and 500 kPa) × 3 (different times 30, 60, and 120 s) × 2 factorial design. The pressure and time parameters for the untreated OF, determined as the control sample, were set to "0" as no treatment was applied. A minimum of three measurements were conducted on 13 distinct samples, with the resultant data presented as the mean value. This study employed a randomised experimental design to investigate the impact of DIC treatment on the OF properties. The data were analysed using the statistical software package SPSS (SPSS, Inc., Chicago, IL, USA) for variance analysis (ANOVA), followed by the assessment of group differences using the Duncan multiple comparison test with a 95% confidence interval. Pearson correlation coefficients were determined between time, pressure, colour values, free, bound and total phenolic contents, and EC50 antioxidant capacity.

Instant controlled pressure drop (DIC) technique

The DIC equipment consists of four key components: a steam generator capable of producing pressurised saturated steam at a pressure of approximately 600 kPa, an 8 mm thick stainless steel processing vessel with a capacity of 10 L, a vacuum chamber with a capacity of approximately 70 L, and a large valve with a diameter of approximately 102 mm that can be opened rapidly (in <1 s). For further details, please refer to the study by Yağcı & Evci (2015), which provides an outline of the schematic diagram and technical details of the equipment. The process commences with the placement of a sample within the processing vessel, followed by the injection of saturated steam until the pressure within the vessel reaches a specified level (either 200 or 500 kPa). This pressure is then maintained for a specified period (indicated by 30, 60, or 120 s). Subsequently, the pressure is rapidly dropped to a vacuum level (maintained at 3 kPa for all treatments) via a pressure drop valve, which causes the water within the product to evaporate. The pressure and time parameters for the untreated OF, which was determined as the control sample, were set to "0" as no treatment was applied. The pressures and times were determined through preliminary experiments, with particular emphasis placed on the values at which differences of the colour in the samples could occur. Approximately 50–60 g of DIC-treated samples were collected and subsequently subjected to drying on a tray dryer maintained at a temperature of 30 °C for a period of 2 h. The samples were then stored at a temperature of −18 °C until such time as further analysis could be conducted.

The physical and functional properties of untreated and DIC-treated OF

The colour of the untreated and DIC-treated OF, as represented by its lightness to darkness (L^*), redness to greenness (a^*), and yellowness to blueness (b^*), was measured using a colorimeter (HunterLab Colour Flex, USA). Total colour difference (ΔE) values were calculated according to the equation of $\Delta E = \sqrt{\left(L_0^* - L^*\right)^2 + \left(a_0^* - a^*\right)^2 + \left(b_0^* - b^*\right)^2}$ (where L_0^*, a_0^*, and b_0^* are the colour of untreated OF, while L^*, b^*, and a^* are the colour of the DIC-treated OF). Additionally, the saturation index (SI) and Hue angle are calculated based on the values of a^* and b^*. The SI is determined by equation of $\mathrm{SI} = \sqrt{\left(a^{*2} + b^{*2}\right)}$ while the Hue angle is calculated using the following formula: if $a^* > 0$ and $b^* > 0$, $\text{Hue angle} = \tan^{-1} b^*/a^*$; if $a^* < 0$ and $b^* > 0$, $\text{Hue angle} = \tan^{-1}(b^*/a^*) + 180^\circ$. Three measurements were conducted, and the results were presented as the mean.

The water activity of the OF was determined utilising the Novasina AW Labmaster device (Aw 1 119 971, Switzerland). The method proposed by Sharma *et al.* (2004) was slightly modified to calculate the water and oil absorption indices (WAI and OAI).

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The 0.5 g of the sample was mixed with 10 mL of water for 10 min and allowed to rest for 10 min at a temperature of 25 °C. Subsequently, the samples were centrifuged at 9000 × ***g*** for 15 min. The WAI was expressed as the quantity of water retained by grams per gram of OF. The 0.5 g sample was mixed with 10 mL of corn oil for 10 min to determine the OAI by OF. The mixture was then allowed to settle for 10 min and subjected to centrifugation at 9000 × ***g*** for 30 min. The weight of the oil retained by the solids was expressed as a ratio of grams per gram of OF.

The chemical composition of untreated and DIC-treated OF

The proximate composition of the untreated and DIC-treated OF was analysed through chemical analysis. In accordance with the standard procedures set forth by the AACC (2002), the moisture, ash, protein, fat, and crude fibre content were determined using the appropriate methods, namely 44-15A, 08-01, 46-12, 30-25, and 32-10, respectively. The total carbohydrate content was calculated on the basis of the mass differences between the proximate compositions. An analysis of the dietary fibre content of the flour was conducted in accordance with the AOAC 960.43 method, utilising the Megazyme Dietary Fibre Determination Kit (K-TDFR-100A, Ireland). The methodology employed for this analysis determined the values of insoluble dietary fibre (IDF) and total dietary fibre (TDF). The value of the soluble dietary fibre (SDF) was calculated by subtracting the IDF from the TDF (AOAC, 2005).

The free and bound (hydrolyzable) phenolics and antioxidants

The bioactive components of the untreated and DIC-treated OF were extracted in both free and bound forms in the samples. The total phenolic and antioxidant content for the untreated and DIC-treated OF were calculated by combining the free and bound phenolic contents and antioxidant capacities. The methodology outlined by Kaya *et al.* (2017) was employed in this investigation. The sample (0.2 g) was prepared by mixing 20 mL acetone and methanol in a 1:1 ratio (v/v), followed by the addition of 13 μL concentrated HCl. Subsequently, the mixture was transferred to a water bath and agitated at 200 rpm for 1 h at 24 ± 1 °C. Subsequently, the supernatant was transferred to a rotary evaporator (Scilogex RE100, USA) in order to evaporate the methanol and acetone. The sediment was dissolved in a solution of ethanol (1 mL), acetone (1 mL) and distilled water (4 mL), and purified through a 0.45 μm membrane filter. The residual substance was subjected to hydrolysis using 10 mL of sodium hydroxide (4 N) in a water bath (200 × ***g***, 3 h) and then extracted with 60 mL of ethyl acetate. The extracts were concentrated, and the sediment was dissolved in a mixture of ethanol (1 mL), acetone (1 mL), and distilled water (1 mL) in order to prepare solutions of a total volume of 10 mL. These solutions were subsequently stored at −18 °C until analysis.

Phenolic content analysis

The study used the Folin–Ciocalteu method as explained by Spanos & Wrolstad (1990) to quantify the concentrations of free, bound, and total phenolic contents. To determine the amount of gallic acid equivalents (GAE) per gram of dry weight (DW), a 100 μL sample extract was combined with 900 μL distilled water, 5 mL Folin–Ciocalteu solution (0.2 N), and 4 mL sodium carbonate solution (75 g/L). The mixture was then incubated in a dark room at 24 ± 1 °C for 2 h before measurement using a spectrophotometer (UV-1800; Shimadzu, Kyoto, Japan) at a wavelength of 765 nm.

The EC50 with the DPPH assay analysis

The DPPH assay was performed according to the methodology described by Pellati *et al.* (2004) to analyse free, bound, and total antioxidant activity. To quantify the concentration of an antioxidant, a DPPH radical with a mass of 0.03943 g was dissolved in methanol in a 100 mL flask. Five sample extracts at different concentrations (20, 40, 60, 80, and 100 μL) were then added to a solution of DPPH (600 μL). The mixture was then completed with methanol to a total volume of 6 mL. The solution was then vortexed and incubated in the dark at 24 ± 1 °C for 15 min. The untreated and DIC-treated OF were analysed using a spectrophotometer (UV-1800; Shimadzu) at a wavelength of 517 nm. EC50 values were expressed in mg DW, based on the average of duplicate analyses.

Trolox equivalent antioxidant capacity analysis

The free, bound, and total Trolox equivalent antioxidant capacity of the samples was determined by a modified version of the method developed by Re *et al.* (1999). In summary, 0.0384 g of ABTS was dissolved in distilled water and combined with 2 mL of 12.25 mM potassium persulfate. Subsequently, the solution was completed with 10 mL of distilled water, wrapped in aluminium foil, and stored in the dark at 24 ± 1 °C for 12–16 h. Prior to analysis, the ABTS solution was diluted with phosphate buffered saline (pH 7.4) to an absorbance of 0.700 ± 0.020 at a wavelength of 734 nm. The four distinct concentrations (5,

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10, 15, and 20 μL) of extracts were combined separately with 1 mL of ABTS radical solution. Following a 6-min period of incubation, a measurement was taken at 734 nm. The outcomes were expressed as mM Trolox equivalents per gram of sample, based on the calibration curve of absorbance versus concentration (5–20 μmol) of Trolox.

Mineral content analysis

The DIC-treated samples were selected for mineral analysis and structural properties on the basis of their superior concentration of the bioactive properties, which were found to be comparable to those of the untreated OF.

The mineral content of the two DIC-treated and untreated OF samples was analysed by inductively coupled plasma optical emission spectroscopy (ICP-OES, Vista series). Prior to analysis, the samples were subjected to microwave digestion in a closed vessel microwave digestion furnace (MARS 5, CEM Corporation, USA). The digestion solution consisted of concentrated nitric acid and sulphuric acid. The concentrations of the mineral constituents (Cu, Na, Mn, Mg, P, Ca, Fe, and K) were then determined by ICP-OES (Skujins, 1998).

SEM analysis

The micrographs of the untreated and 200 and 500 kPa 60-s DIC-treated OF were obtained by scanning electron microscopy (SU5000, Hitachi, Japan). Prior to testing, the samples of flour were coated with a 15 nm gold-platinum layer to ensure conductivity. The samples were analysed at an accelerated voltage of 3 kV and a 100 μm working distance.

Results and discussion

Physical and functional properties of DIC-treated oleaster flour

Table 1 illustrates the colour characteristics of both untreated and DIC-treated OF. The pressure and time parameters for the untreated OF, which was selected as the control sample, were set to "0" as no treatment was applied. Two distinct pressures (200 and 500 kPa) and three distinct times (30, 60, and 120 s) were applied to the DIC-treated OF samples. The pressures and times were determined through preliminary experiments, with a particular focus on identifying the values at which differences in the colour of the sample could be observed. Colour measurement of food products is widely used to indirectly assess their quality attributes such as flavour, nutritional content, and pigmentation. This straightforward, speedy, dependable method produces consistent results closely linked to other physicochemical properties.

The relationship between the experimental parameters, namely pressure and time, was determined through the application of the Pearson correlation test. The findings of the study indicated that the colour values exerted an influence on both the pressure and the processing time. The study revealed a negative correlation between pressure and L^* ($R = -0.895$), b^* ($R = -0.852$) and Hue angle ($R = -0.855$) and a positive correlation between pressure and ΔE ($R = 0.897$) at $P < 0.01$. The a^* value demonstrated a positive correlation ($R = 0.593$, $P < 0.05$), while the SI exhibited a moderately negative correlation ($R = -0.679$, $P < 0.01$). The analysis also demonstrated that there was a negative correlation between L^* ($R = -0.635$) and b^* ($R = -0.638$) values and processing time at the

Table 1 Colour and techno-functional properties of untreated and DIC-treated oleaster flour (OF)

Parameter	Untreated OF (0 kPa 0 s)	DIC-treated OF					
		200 kPa 30 s	200 kPa 60 s	200 kPa 120 s	500 kPa 30 s	500 kPa 60 s	500 kPa 120 s
L^*	75.14 ± 0.13 a	73.00 ± 0.05 b	68.19 ± 0.03 c	54.17 ± 0.17 d	46.57 ± 0.09 e	34.69 ± 0.12 f	26.50 ± 0.11 g
a^*	2.86 ± 0.03 f	4.81 ± 0.02 e	7.97 ± 0.05 c	13.90 ± 0.06 a	12.44 ± 0.03 b	12.38 ± 0.07 b	7.64 ± 0.09 d
b^*	23.87 ± 0.05 a	22.16 ± 0.05 b	20.85 ± 0.05 c	20.19 ± 0.09 d	17.25 ± 0.17 e	16.28 ± 0.09 f	9.85 ± 0.09 f
ΔE	0.00 ± 0.00 g	3.39 ± 0.09 f	9.14 ± 0.05 e	23.90 ± 0.06 d	30.85 ± 0.06 c	42.21 ± 0.02 b	50.83 ± 0.05 a
SI	24.03 ± 0.03 b	22.66 ± 0.02 c	22.31 ± 0.02 d	24.45 ± 0.09 a	21.21 ± 0.03 e	20.48 ± 0.04 f	12.47 ± 0.01 g
Hue angle	83.16 ± 0.02 a	77.74 ± 0.03 b	69.00 ± 0.08 c	55.44 ± 0.01 d	54.26 ± 0.08 e	52.74 ± 0.01 f	52.07 ± 0.21 g
WAI (g water/g sample)	4.90 ± 0.33 a	3.43 ± 0.11 bc	2.79 ± 0.19 d	3.66 ± 0.05 b	3.36 ± 0.19 bc	4.48 ± 0.84 a	3.65 ± 0.27 b
OAI (g oil/g sample)	2.26 ± 0.07 a	1.60 ± 0.04 bc	1.35 ± 0.14 d	1.57 ± 0.05 c	1.71 ± 0.03 b	1.37 ± 0.06 d	1.13 ± 0.08 e
Water activity	0.31 ± 0.00 ab	0.27 ± 0.01 cd	0.26 ± 0.01 d	0.29 ± 0.01 bc	0.26 ± 0.01 d	0.30 ± 0.03 ab	0.32 ± 0.02 a

The results given represent the mean value of three measurements. Different letters within the same groups indicate significant statistical differences between values, Duncan multiple comparison test, ($P < 0.0.5$).

ΔE, total colour difference; OAI, oil absorption indices; OF, oleaster flour; SI, the saturation index; WAI, water absorption indices.

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$P < 0.05$ level. In contrast, there was a high negative correlation between hue values and processing time ($R = -0.734$, $P < 0.01$), while a^* and ΔE values exhibited a positive correlation ($R = 0.583$, $P < 0.05$ and $R = 0.659$, $P < 0.05$, respectively).

The implementation of the DIC method resulted in notable alterations to the colour characteristics of OF, with both pressure and time variations exerting a discernible influence. However, the findings indicate that pressure exerts a more pronounced impact on colour values than processing time (Table S1 and Figure S1). Li *et al*. (2022) reported results that are very similar to the results of our study. They compared DIC treated apple slices with freeze drying and hot air drying. The DIC apple slices had lower L^* and higher a^* values, leading to significant browning, which was mainly due to melanoidin formation in the Maillard reaction during the higher temperature, pressure drop, and vacuum drying process (Gao *et al*., 2017).

It is paramount to comprehend the water absorption indexes (WAI) and oil absorption indexes (OAI) of a given material to ascertain its capacity to retain water and oil. This is particularly important for powdered food materials, such as flour, which are commonly combined to produce various food products (Mounir *et al*., 2012). The quality and consistency of the final product can be guaranteed by determining the WAI and OAI of the material in question.

The study demonstrates notable alterations in the water and oil absorption characteristics of DIC-treated OF. The water absorption properties were also observed to be lower than those of the control flour, with the exception of the OF treated at 500 kPa for 60 s, which exhibited a water absorption value (4.48 ± 0.84) that was similar to that of the control flour (4.90 ± 0.33). DIC treatment under high pressure and temperature can impact the functional properties through changes in its protein structure. The pressure can cause proteins to unfold, leading to interactions between disulfide (-SS-) and sulfhydryl (-SH) groups. This interaction results in disulfide-bonded aggregates and the exposure of buried hydrophobic groups. These changes in protein conformation can lead to nonpolar interactions with water, causing a decrease in WAC (Schokker *et al*., 2000; Considine *et al*., 2007). According to Wagner *et al*. (2000), protein denaturation increases surface hydrophobicity, promoting water repulsion. This supports the reduction in the WAC of the samples.

Ultimately, the oil absorption results demonstrated a decline with the application of increasing pressure and processing time. The lowest recorded oil absorption value was 1.13 g oil/g sample at 500 kPa for 120 s (Table 1). This reduction may be due to the decrease in the quantity of IDF resulting from the process. Previous studies by Dalgetty & Baik (2003) have reported that the OAC of IDF is higher than that of SDF. Mkaouar *et al*. (2022) also reported comparable outcomes, noting that the untreated and DIC-treated olive leaves exhibited water and oil retention capacities of 2.97 g/g and, 2.10 g/g, and 2.12 g/g and 2.02 g/g, respectively. A reduction of approximately 29% in water retention capacity and 4% in oil retention capacity was observed.

The water activity (aw) is of great significance as it allows for the quantifying of the quantity of accessible water that can be engaged in biological, chemical, or physical processes within a product. It is of the utmost importance to utilise this metric in order to guarantee enhanced safety, stability, and quality. It offers valuable insights into the intrinsic characteristics of the product, thus facilitating the determination of its suitability for consumption, storage, and processing. Table 1 presents the functional properties of the untreated and DIC-treated OF. In general, the DIC-treated OF exhibited lower water activity values. The DIC-treated OF at 500 kPa for 60 and 120 s exhibited comparable water activity values (0.30 ± 0.03 and 0.32 ± 0.02) in comparison to the untreated OF (0.31 ± 0.00). Although statistically significant ($P < 0.05$), the observed difference was relatively minor. The water binding affinity of materials expanded using the DIC process is greater than that of other materials. This is despite the fact that DIC-expanded materials are more efficient at the drying process. As the specific surface area increases, the polar groups on the external surface also increase, forming stronger bonds with water molecules (Mounir *et al*., 2012). Additionally, the lower water activity is attributed to the amount of water that auto-vaporises. Concurrently, the pressure decreased towards a vacuum, which is higher for the treatment at higher pressure compared to that at lower pressure, resulting in a lower final water content. This reduces water availability for microorganisms and inhibits certain chemical reactions (Arslan & Toğrul, 2005). The findings of our study indicated that DIC-treated OF exhibited a lower water activity, suggesting a more stable flour with an extended shelf life.

Chemical properties of DIC-treated oleaster flour

Table 2 presents a comparison of the chemical properties of untreated and DIC-treated OF. The application of DIC resulted in a notable elevation in the levels of fat and ash present in OF, with a nearly twofold increase observed when the pressure was elevated from 200 to 500 kPa ($P \leq 0.05$). This is attributable to the modified microstructure of the flour, which facilitates the extraction of fat and ash. Furthermore, the SEM analysis corroborates the hypothesis that this method results in the formation of broken-wall cells, thereby

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Table 2 Chemical composition of untreated and DIC-treated oleaster flour (OF)

Parameter	Untreated OF (0 kPa 0 s)	DIC-treated OF					
		200 kPa 30 s	200 kPa 60 s	200 kPa 120 s	500 kPa 30 s	500 kPa 60 s	500 kPa 120 s
Moisture (%)	8.98 ± 0.01 a	7.49 ± 0.19 cd	7.15 ± 0.17 d	7.82 ± 0.07 cd	7.37 ± 0.24 cd	8.65 ± 1.02 ab	8.05 ± 0.49 bc
Fat† (%)	0.43 ± 0.01 c	0.62 ± 0.01 ab	0.53 ± 0.08 bc	0.56 ± 0.07 bc	0.78 ± 0.09 a	0.61 ± 0.12 ab	0.51 ± 0.00 bc
Ash† (%)	1.51 ± 0.03 e	2.41 ± 0.09 bc	2.10 ± 0.05 d	2.28 ± 0.02 c	2.47 ± 00 bc	2.56 ± 0.01 ab	2.75 ± 0.14 a
Protein† (%)	5.67 ± 0.7 a	4.49 ± 0.23 a	4.17 ± 0.25 a	4.49 ± 0.7 a	5.01 ± 0.81 a	5.05 ± 0.37 a	4.70 ± 0.07 a
Total carbohydrate† (%)	83.41 ± 0.74 bc	84.90 ± 0.41 ab	86.00 ± 0.07 a	84.81 ± 0.85 abc	84.23 ± 0.58 bc	83.09 ± 1.18 d	83.76 ± 0.40 bc
SDF† (%)	4.75 ± 0.22 e	4.20 ± 0.42 e	5.16 ± 0.11 de	6.94 ± 0.06 d	10.23 ± 0.16 c	13.02 ± 0.47 b	17.45 ± 0.31 a
IDF† (%)	21.61 ± 0.03 a	22.16 ± 0.42 a	18.37 ± 0.87 b	19.14 ± 0.00 b	16.29 ± 0.01 c	13.23 ± 0.54 d	9.82 ± 0.54 e
TDF† (%)	26.36 ± 0.19 a	26.36 ± 0.84 a	22.91 ± 0.11 b	26.08 ± 0.06 a	26.52 ± 0.17 a	26.25 ± 1.00 a	27.27 ± 0.86 a

The results given represent the mean value of three measurements. Different letters within the same groups indicate significant statistical differences between values. Duncan multiple comparison test, ($P < 0.0.5$).

IDF, insoluble dietary fibre; OF, oleaster flour; SDF, soluble dietary fibre; TDF, total dietary fibre.

†All values are on a dry-matter basis.

rendering the fat and ash more accessible. Mkaouar *et al.* (2022) reported findings that were similar to those presented here and provided detailed information on the chemical composition of untreated olive leaves and DIC-treated olives. The aforementioned research indicates that untreated olive leaves contain 2.7% moisture, 8.62% protein, 3.73% fat, 8.46% ash, 76.49% carbohydrates, and 64.29% fibres per gram of dry matter. In contrast, the DIC-treated olive leaf samples exhibited a moisture content of 5.5%, protein at 6.81%, fat at 8.33%, ash at 10.37%, carbohydrates at 69%, and fibre at 53.57%, all on a dry basis. The protein content of OF exhibited a slight decline following DIC treatment; however, this decrease was not statistically significant ($P > 0.05$). It is possible that the Maillard reaction, which occurred during the treatment, affected certain proteins. A study on cactus pear peel demonstrated that a comparable reduction in protein levels subsequent to DIC treatment could be attributed to a sudden pressure decline, resulting in a decline in overall nitrogen content. This pressure decline may lead to the auto-vaporisation and condensation of volatile compounds, such as inorganic nitrogen (NH4) (Namir *et al.*, 2017). Despite the statistical discrepancy in the total carbohydrate ratio, the values were found to be quite similar.

The findings of the study demonstrate that the utilisation of DIC resulted in a notable elevation in soluble fibre ($P \leq 0.05$) and a substantial reduction in insoluble fibre ($P \leq 0.05$). Although there was a reduction in total dietary fibre at 200 kPa for 60 s, it remained consistent with the untreated OF for all other pressures and processing times. This may be attributed to the alterations in OF microstructure caused by DIC, which enabled the extraction of soluble fibres by breaking down cell walls that were primarily composed of insoluble fibres (Namir *et al.*, 2017).

Concentration of the bioactive components of the DIC-treated oleaster flour

Table 3 presents the findings of the bioactive components in untreated and DIC-treated OF. The results are classified according to the following categories: free, bound, and total phenolic substances; EC50 antioxidant capacity; and Trolox equivalent antioxidant capacity. The levels of free and total phenolic compounds exhibited a decline during the 200 kPa process, yet demonstrated an increase during the 500 kPa process as compared to the untreated OF. A correlation was observed between the free and total phenolic compound levels and pressure ($R = 0.572$ and $R = 0.620$, $P < 0.05$). These findings are in accordance with those of previous research, as evidenced by Kamal & Allaf (2013), who discovered that elevated pressure levels resulted in a higher total phenolic content (TPC). The lower EC50 value indicates that a reduction in the quantity of antioxidants is necessary to achieve a 50% decrease in DPPH absorbance from the initial level. The application of DIC at 500 kPa for 30 and 60 s resulted in the formation of strong free antiradical properties. The processing conditions of 200 kPa for 60 s and 500 kPa for 30 s demonstrated an increase in the bound and total antioxidant capacity of the flour, which surpassed those of the untreated OF ($P < 0.05$).

Sánchez-Valdepeñas *et al.* (2015) reported that the use of DIC in the treatment of grape stalk powder resulted in an increased presence of bioactive components, including gallic acid, quercetin, ellagic acid, and

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Table 3 Concentration of the bioactive components of untreated and DIC-treated oleaster flour (OF) samples

Parameter	Untreated OF (0 kPa 0 s)	DIC-treated OF					
		200 kPa 30 s	200 kPa 60 s	200 kPa 120 s	500 kPa 30 s	500 kPa 60 s	500 kPa 120 s
Phenolic content mg GAE/g[†]							
Free	28.28 ± 1.89 b	19.71 ± 1.09 d	23.53 ± 0.59 c	18.36 ± 1.05 d	28.72 ± 1.56 b	30.73 ± 1.98 b	34.62 ± 2.07 a
Bound	9.83 ± 0.65 c	6.07 ± 0.34 d	11.59 ± 0.43 b	8.48 ± 0.30 c	12.01 ± 0.65 b	21.33 ± 1.37 a	8.43 ± 0.43 c
Total	38.11 ± 1.24 bc	25.78 ± 0.75 d	35.12 ± 1.02 c	26.83 ± 1.36 d	40.73 ± 2.21 b	52.06 ± 0.61 a	43.04 ± 2.50 b
Antioxidant capacity EC50 mg[†] with DPPH assay							
Free	2.14 ± 0.09 cd	2.53 ± 0.18 bc	2.26 ± 0.16 bc	3.50 ± 0.27 a	1.53 ± 0.06 e	1.78 ± 0.09 de	2.59 ± 0.17 b
Bound	9.28 ± 0.05 b	9.08 ± 0.01 b	7.25 ± 0.52 c	12.57 ± 0.13 a	7.98 ± 0.88 c	9.23 ± 0.19 b	12.99 ± 0.28 a
Total	11.38 ± 0.09 b	11.60 ± 0.18 b	9.51 ± 0.35 c	16.07 ± 0.13 a	9.51 ± 0.81 c	11.01 ± 0.11 b	15.58 ± 0.45 a
Trolox equivalent antioxidant capacity mMTrolox/g[†]							
Free	0.190 ± 0.012 b	0.173 ± 0.009 bc	0.227 ± 0.012 a	0.179 ± 0.009 bc	0.161 ± 0.004 d	0.214 ± 0.002 a	0.233 ± 0.014 a
Bound	0.134 ± 0.009 c	0.132 ± 0.007 c	0.205 ± 0.011 b	0.124 ± 0.007 c	0.216 ± 0.012 b	0.283 ± 0.017 a	0.097 ± 0.006 d
Total	0.325 ± 0.004 d	0.305 ± 0.002 d	0.432 ± 0.001 b	0.298 ± 0.009 d	0.377 ± 0.08 c	0.497 ± 0.016 a	0.329 ± 0.008 cd

The results given represent the mean value of three measurements. Different letters within the same groups indicate significant statistical differences between values Duncan multiple comparison test, ($P < 0.05$).

OF, oleaster flour.

[†]All values are on a dry-weight basis.

resveratrol. The amounts of these components present varied according to the solvent used. The ABTS method represents an effective means of measuring the Trolox equivalent antioxidant capacity. The DIC-treated OF exhibited a greater Trolox equivalent antioxidant capacity than the untreated OF under processing conditions of 200 and 500 kPa for 60 s ($P < 0.05$). Mounir *et al.* (2011) observed that the quantity of quercetin present in apples treated with DIC was increased by up to 700% in comparison to untreated fresh samples. The treatment of orange by-products using DIC (drying-induced changes), which involves partial drying and texturing, has resulted in a notable increase in the concentration of active molecules. As an example, the levels of hesperidin increased from 12.10 to 65.01 mg/g db, while those of rutin rose from 11.47 to 27.10 mg/g db. Similarly, the levels of flavone increased from 0.006 to 0.007 mg/g db, and those of naringin increased from 0.0002 to 0.00032 mg/g db. It can be reasonably inferred that the aforementioned active molecules are correlated with the presence of broken-wall cells, as well as free and total phenolic content. This hypothesis has been corroborated by SEM analysis (Louati *et al.*, 2019).

The results of this study demonstrated a notable increase in the quantity of bound phenolic substances and antioxidant capacity when the DIC method was utilised, particularly under 200 and 500 kPa for 60 s ($P < 0.05$). The non-extractable phenolic contents associated with dietary fibres can still display various antioxidant capacities, which are collectively referred to as 'antioxidant dietary fibre' (Saura-Calixto, 1998). Oleaster is a functional food product with a high dietary fibre and phenolic content, in which catechin and its derivatives are the most abundant (Karkar & Şahin, 2022). Phenolic components interact with other food components in a reversible or irreversible manner (Rocchetti *et al.*, 2022). The degree of hydroxylation, methylation, methoxylation, esterification, glycosylation, hydrogenation, molecular weight, and fibre types all exert a significant influence on the binding affinity of phenolic components to dietary fibres (Rocchetti *et al.*, 2022). In a study by Liu *et al.* (2019), it was found that pectin, a soluble fibre, has a greater capacity for adsorbing catechin in comparison to cellulose. This is due to the extended molecular chain structure of pectin, which provides a greater number of favourable binding sites for catechin. However, food processing can have a beneficial or detrimental impact on the bioaccessibility and bioavailability of phenolic components, which in turn affects their beneficial properties for human health (Cilla *et al.*, 2018). The effect of processing and fermentation on fibre-associated phenolic components after in vitro gastrointestinal digestion remains limited. Further studies are needed to investigate the interactions between dietary fibre and phenolic compounds, specifically during processing.

Mineral content

The DIC-treated OFs, which were treated at 200 and 500 kPa for 60 s, were selected for analysis of their mineral and structural properties due to their superior bioactive properties, which were comparable to those of the untreated OF. Despite the existence of significant differences in terms of techno-functional and

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chemical attributes between the control sample and the treated OFs, the most noteworthy distinction was observed in the concentration of the bioactive components.

The mineral content of the untreated OF was determined to be 304.2 ppm Cu, 330.97 ppm Na, 384.1 ppm Mn, 50.88 ppm Mg, 73.28 ppm P, 141.07 ppm Ca, 1620.5 ppm Fe, and 566.21 ppm K. The application of DIC treatment resulted in a notable enhancement in the mineral content of the OF ($P < 0.05$, as illustrated in Fig. 1). A recent study by Mkaouar *et al.* (2022) demonstrated that the application of DIC treatment to olive leaves (*Olea europaea L.*) led to a significant increase in the levels of Ca, K, Mg, and Na in the final product. However, a notable decline was observed in the iron content. The levels of Cu, Zn, and Mn were found to be almost identical in the processed samples.

Rousseau *et al.* (2020) reported that the processing techniques employed may either enhance or reduce the mineral content. The observed enhancement may be attributed to the removal of antinutrients, leaching into processing liquids, or the activation of enzymes. Conversely, the reduction may be attributed to mineral leaching, loss of cell wall integrity, or phytase inactivation during thermal processing. Additionally, the microstructure of the flour underwent modification, potentially enhancing its ability to extract mineral content.

SEM analyses

In particular, samples subjected to 200 and 500 kPa of pressure for 60 s were compared to the control sample, demonstrating notable differences in the resulting outcomes (Fig. 2). Upon examination of the SEM micrographs, it becomes evident that there is a considerable difference in the structure of DIC-treated and untreated OF. The structure of the untreated OF appears more holistic, whereas that of the DIC-treated OF is fragmented. In particular, the DIC-treated OF at 500 kPa and 60 s exhibited severe disintegration.

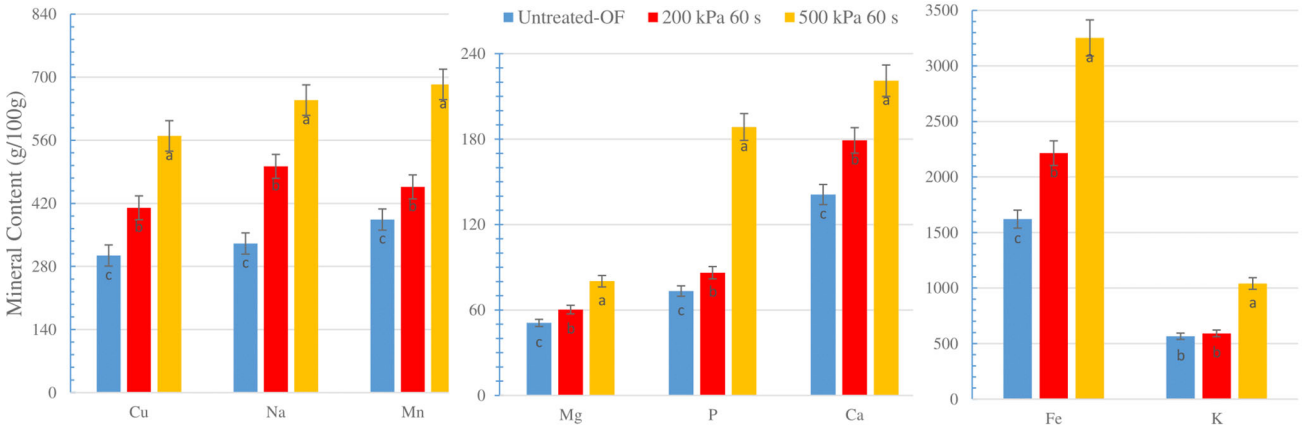


Figure 1 Mineral contents of untreated and DIC-treated oleaster flour (OF). Different letters indicate significant statistical differences between values ($P < 0.05$).

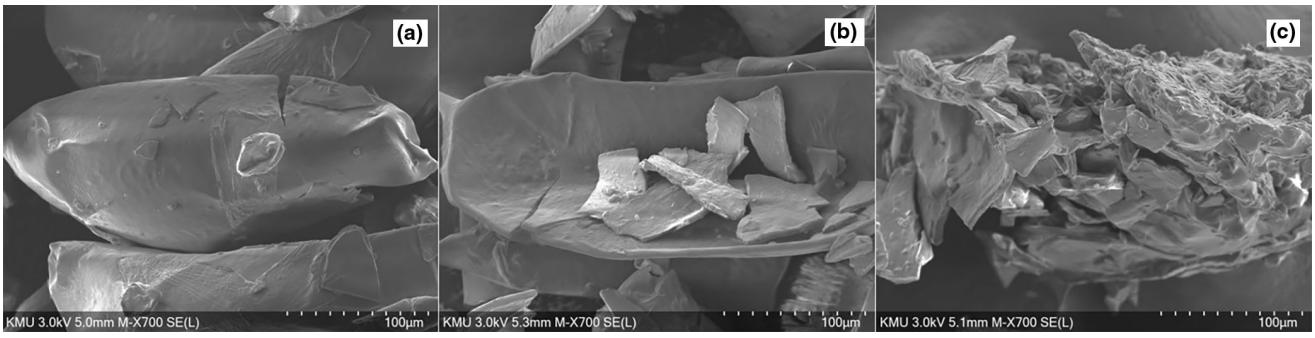


Figure 2 SEM micrographs of untreated OF (a) 200 kPa 60 s DIC-treated OF (b) and 500 kPa 60 s DIC-treated OF (c).

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As reported by Louati *et al.* (2019), the DIC treatment of orange by-products has been found to significantly increase the concentration of active molecules. It can be reasonably inferred that the active molecules are correlated with the presence of broken-wall cells, as evidenced by SEM. Romero-Cano *et al.* (2016) observed that the application of DIC to orange peels resulted in the collapse of cell walls, thereby creating additional openings. The duration of the treatment is directly correlated with the number of openings that are created. Furthermore, this transformation results in an increase in the quantity of R-OH and R-COOH residues, thereby creating additional openings. The findings demonstrate that alterations in the product structure, including fragmentation, breakage, and gap formation, have a considerable impact on the characteristics of the product. Consequently, such changes can result in enhanced extractability, reduced functional properties, and elevated bioactive components (Mounir *et al.*, 2011; Sánchez-Valdepeñas *et al.*, 2015; Romero-Cano *et al.*, 2016; Louati *et al.*, 2019).

Conclusion

Controlled pressure drop treatment is environmentally friendly and has been applied to modify the texture of food matrices of fruits, vegetables, and by-products. In this study, OF was subjected to DIC treatment at different pressures (200 and 500 kPa) and processing times (30, 60, and 120 s). The results were compared with untreated OF. The DIC treatment led to considerable alterations in colour attributes and techno-functional properties compared to the control. It enhanced the content of fat, ash, soluble fibre, and various minerals (Cu, Na, Mn, Mg, P, Ca, Fe, and K). The concentration of bioactive compounds in free, bound, and total forms varied based on process variables. Substantial differences in structure were also noted. Upon thoroughly evaluating the results, the DIC method improved the quality of the final product, resulting in a visually appealing product with a long shelf-life, high nutritional value, and more functional and convenient products.

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Author contributions

Nazlı Şahin: Conceptualization; investigation; methodology; validation; writing – original draft; writing – review and editing; visualization; formal analysis; data curation.

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Conflict of interest

The authors declare that they have no conflicts of interest.

Ethical statement

Ethics approval was not required for this study.

Peer review

The peer review history for this article is available at https://www.webofscience.com/api/gateway/wos/peer-review/10.1111/ijfs.17552.

Data availability statement

All data generated or analysed during this study are included in this published article.

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Our study emphasised the effect of the DIC method on the phenolic content. Karkar and Şahin (2022) characterised the phenolic compound profile of oleaster samples and supported our findings.

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The study established the potential for producing chickpea and wheat snacks using various DIC-processing conditions, including processing pressure, time, and material moisture content. Their specific impact on selected physicochemical and nutritional parameters of the final product was determined. This study outlines the schematic diagram and technical details of the equipment and DIC-treatment method.

Supporting Information

Additional Supporting Information may be found in the online version of this article:

Table S1. Correlation table between the pressure, the processing time, color properties, phenolic content and EC50 antioxidant capacity of OF.

Figure S1. The images of untreated and DIC-treated oleaster flour (OF).

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