



## Exploring the Bioactive Potential of Argan Oil Cake: A Comprehensive Physicochemical Comparison with various Seeds Cakes

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### Abstract

The argan oil industry generates a large quantity of argan oil cake. This residue is traditionally used as livestock feed. In order to explore other potential uses for this by-product, a full physico-chemical characterisation was carried out. The analysis includes mineral profiling, protein content, fiber content, fatty acid composition, sterol content, total polyphenol and flavonoid content, and antioxidant activity, in comparison with other cakes, namely almond, sesame, nigella, sunflower and soybean cakes. The results indicate that potassium (K), phosphorus (P), magnesium (Mg), calcium (Ca) and sodium (Na) are the main mineral elements in the argan oilcake. In terms of protein content, argan oilcake contained the highest content at  $45.90 \pm 0.10$  g/100 g dry weight. The residual oil content was around  $12.61 \pm 0.80$  g/100 g. Oil from argan press cake has high levels of oleic and linoleic acid ( $47.88 \pm 0.20$  and  $32.10 \pm 0.15$  %) respectively. In terms of sterol composition, argan cake is the only one to contain Schottenol, Spenasterol and Stigma-8-22-dien-3b-ol. Argan oil cake showed remarkable values for total phenolic compounds ( $5.11 \pm 0.01$  mg GAE/g) and DPPH antioxidant activity ( $8.06 \pm 0.08$  mg AAE/g). The results suggest that argan cake could be considered a rich source of nutrients and that its extracts have substantial value-added potential, warranting attention in future research and development.



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## Introduction

Food security is a major global challenge, intensified by rapid population growth, particularly in developing countries facing severe food shortages and hunger.<sup>1,2</sup> The most common solution has been to increase food production,<sup>3</sup> but this leads to excessive use of crucial natural resources such as land, water and energy. An increasingly popular alternative approach focuses on social and ecological concerns, including the recovery of by-products, making production more sustainable and profitable.<sup>3</sup> Reducing food waste not only reduces production costs and improves system efficiency, it also enhances the sustainability of the food industry.<sup>4</sup> Food production generates large amounts of waste, often unused, which is traditionally disposed of by methods such as incineration or landfill, contributing to environmental problems and food safety concerns.<sup>4-6</sup> In response, researchers have developed new strategies to reduce food waste, focusing on oilcake, a by-product of oilseed crushing. Oil cake is considered a significant source of proteins, fibers, and minerals, primarily used as animal feed.<sup>7</sup> Numerous studies have encouraged and explored novel applications for by-products from the oil industry, including the formulation of functional drinks,<sup>8</sup> preparation of naturally gluten-free flour,<sup>9</sup> food additives,<sup>10</sup> emulsion stabilizers,<sup>11</sup> sources of essential fatty acids,<sup>12</sup> manufacturing of adsorbents, extraction of fibers like nanocellulose,<sup>13</sup> manufacturing of biocomposite,<sup>14</sup> bioenergy production,<sup>15</sup> protein-based film matrix,<sup>16</sup> and many others use. According to these advances, food waste from the oil industry represents a valuable and profitable reservoir of potentially functional or bioactive compounds. Additionally, these by-products from the food industry exhibit great potential for utilization in both nutraceutical and pharmaceutical applications. This study aligns with the current trend and aims to highlight the opportunities for the valorization of by-products from the argan oil industries. The objective of this work is, therefore, to evaluate the complete physicochemical characterisation, mineral profiling, fatty acid composition, sterol content, total flavonoid content, total polyphenol content, protein and fiber contents, and antioxidant power activity of argan press cake in comparison with almond, sesame, nigella, sunflower and soybean press cakes.

## Materials and Methods

### Plant Material and Processing

This study was conducted on six oilseeds and kernels procured from the local market in Taroudant, Morocco, except argan kernels sourced from the "Taitmatine" women's cooperative in Tiout (Taroudant). The selected six oilseeds and kernels including argan (*Argania spinosa* L. (Skeels)), almond (*Prunus dulcis*), sunflower (*Helianthus annuus* L.), nigella (*Nigella sativa* L.), sesame (*Sesamum indicum*) and soybean (*Glycine max* (L.) Merr). Cold-pressing techniques, employing a screw press, were used to extract oil from the seed, resulting in press cakes. These press cakes were subsequently ground to produce a uniform powder. The obtained powder underwent various analyses, including ash content, moisture content, protein content, mineral profiling, oil yield, fatty acids composition, sterols composition, and antioxidant activities.

### Proximate Composition

#### Residual Oil Content

The residual oil content (OC) was determined using Soxhlet extraction. A total of 20 grams of press cake powder underwent an 8-hours extraction process with hexane. After extraction, the solvent evaporated at 45 °C temperature using a vacuum rotary evaporator (R-200, Buchi, Zurich, Switzerland). The oil content was then determined gravimetrically using the following formula:

$$\text{Oil content (\%)} = \frac{\text{Weight of Oil (g)}}{\text{Sample weight(g)}} \times 100$$

#### Protein Content

The nitrogen content was analysed by combustion using an elemental analyser LECO model (LECO FP628, USA). A conversion factor of 6.25 was applied to derive the protein content, employing the Dumas method as described in the study by Ibourki.<sup>17</sup>

#### Moisture Content, and Ash Content

The moisture content (MC) was determined by drying the sample in an oven at 103 °C until a stable weight was attained. The resulting weight difference was used to calculate MC. Simultaneously, the ash

content (AC) was assessed by incinerating the powder in a muffle furnace at 500 °C for a duration of 4-hours.

#### **Carbohydrate Content and Energy Value**

The carbohydrate content (CC) of each press cake was calculated using the formula derived from the study by Oubannin,<sup>18</sup> subtracting the cumulative content of other compounds such as ash, moisture, oil and protein.

$$CC (\%) = 100\% - (\text{Ash} + \text{Moisture} + \text{Oil} + \text{Proteins})$$

The energy value (EV) for each sample was calculated based on the protein content, oil content, and carbohydrate content, and expressed in kilocalories per 100 g of dried powder. The calculation utilised the following equation from the research by Bouzid.<sup>19</sup>

$$EV (\text{Kcal}/100\text{g}) = (2.62 \times \text{Protein Content}) + (4.2 \times \text{Carbohydrate Content}) + (8.37 \times \text{Oil Content})$$

#### **Minerals Profiling**

Mineral analysis followed the procedure outlined in.<sup>6</sup> One gram of the dried powder was turned into ash at 500 °C using a muffle furnace for 2 hours. Afterward, 4 mL of 65% HNO<sub>3</sub> was added, and the solution was brought to a volume of 25 mL with ultrapure water. The resulting solution was analysed using a Perkin Elmer Optima 8000 DV spectrometry to quantify the concentrations of the following minerals such as zinc (Zn), iron (Fe), boron (B), manganese (Mn), copper (Cu), magnesium (Mg), calcium (Ca), sodium (Na), potassium (K) and phosphorus (P).

#### **Fibers Content**

Crude fibers (CF), Neutral Detergent Fibers (NDF), Acid Detergent Fibers (ADF), and Acid Detergent Lignin (ADL) were determined according to the methodology described by Van Soest and Robertson.<sup>20</sup> The analyses were performed using the FIWE 6 fiber extractor (Velp Scientifica, Usmate Velate (MB) – Italy).<sup>20</sup>

#### **Fatty Acids Determination**

The fatty acid composition was determined using gas chromatography. A mixture comprising 1 g of oil, 10 mL of methanol, and 0.4 mL of 2N methanolic KOH underwent reflux for 10 minutes. The resulting mixture was subjected to extraction with 2 mL

of n-hexane followed by washing using distilled water. The methyl esters of fatty acids present in the hexane phase were collected and analysed. Subsequently, fatty acid content was measured using an Agilent Technologies Varian 3800 A gas chromatograph equipped with a flame ionization detector (GC-FID) and the result was expressed as the equivalent methyl ester. The capillary column utilised had dimensions of 30 m length, 0.25 mm internal diameter (ID), and a thickness of 0.25 µm, categorised as the CP-Wax 52CB type. The carrier gas, helium, had a total flow rate of 1 mL/min. A temperature gradient of 4 °C per minute was established, initiating at 170 °C and concluding at 230 °C for the vent. The injector and detector temperatures were set to 220 °C. Employing a fractionation ratio of 1:50 in fractionated mode, the sample injection volume was 2 µL. The outcomes were presented as a percentage of the peak area of each individual fatty acid.<sup>21</sup>

#### **Sterols Composition**

After trimethylsilylation of the crude sterol portion, phytosterol content was determined using a Varian 3800 instrument equipped with a VF-1 ms column (30 m, 0.25 mm internal diameter, 0.25 µm film thickness), helium serving as the carrier gas at a flow rate of 1.6 millimeters per minutes. The injector and detector were maintained at 300 °C, while the column was kept isothermal at 270 °C. Component identification relied on retention time. The results were reported as the relative proportion (g/100 g) of the peak area for each phytosterol peak's.<sup>22</sup>

#### **Bioactive Compounds Extraction**

The extraction of bioactive components followed the procedure described by Ait Bouzid.<sup>23</sup> Each cake powder (1 g) was macerated with 10 mL of 80% aqueous methanol, stirred for 24 hours. After filtering the mixture with Whatman filter paper, the resulting liquid was stored at 4 °C until further use. Phenol, flavonoid content, and antioxidant activity were assessed on the obtained extracts.

#### **Total Phenolic Content Determination**

The determination of total phenolic content (TPC) utilised the Folin-Ciocalteu reagent technique.<sup>24</sup> This involved combining 0.5 mL of each diluted press cake extract with 2.5 milliliter of Folin-Ciocalteu reagent (made in water at 10%) and adding 2 mL of sodium carbonate Na<sub>2</sub>CO<sub>3</sub> (7.5% in water). The mixture

was subjected to incubation in a water bath at 45 °C for 30 minutes. Subsequently, a SCILOGEXSP-UV1100 UV-Vis spectrophotometer (USA, Rocky Hill) was used to measure the intensity of the blue complex that had developed at a wavelength of 765 nm. The calibration curve, established using gallic acid as the standard over a concentration range of 5 to 160 µg/mL, was employed to determine TPC. The regression equation of the gallic acid standard curve was  $y = 0.0071x + 0.0124$  with  $R^2 = 0.9997$ . The TPC was expressed in milligrams (mg GAE/g DM) or as the equivalent of gallic acid per gram matter of dry powder. Each sample underwent three separate analyses.

#### Total Flavonoids Content Determination

Each extract's total flavonoid content (TFC) was determined through a colorimetric assay following the protocol outlined by Bijla.<sup>25</sup> In a 10 mL test flask, 1 mL of the cake extract solution and 0.3 mL of NaNO<sub>2</sub> (0.5 N) were combined, and the mixture was allowed to stand at room temperature for five minutes. Subsequently, the blend was mixed with 0.3 mL of 10% AlCl<sub>3</sub>. After 6-minute incubation, 1 mL of 2N NaOH was added to the blend, and then distilled water was added to reach the gauge line. The absorbance of the samples was measured at 415 nm using a SCILOGEX SP-UV1100 spectrophotometer. The standard curve equation was  $y = 0.0013x + 0.0057$ , where  $R^2 = 0.9994$ . The TFC was expressed in equivalents of quercetin per gram of dry matter (QE/g DM). This procedure was repeated three times for every extract.

#### Antioxydant Activity

##### Ferric Reducing Antioxidant Power (FRAP)

The antioxidant power of the samples, indicative of their reducing capacity, was evaluated following the method outlined by Aryal.<sup>26</sup> In brief, 2.5 mL of 0.2 M sodium phosphate buffer (pH 6.6) and 2.5 mL of 1% (w/v) potassium ferricyanide (K<sub>3</sub>Fe(CN)<sub>6</sub>) solution were mixed with 1 mL of the extract solution. After 20 minutes of incubation at 50 °C, 2.5 mL of 10% (w/v) trichloroacetic acid was added to the mixture. Ferric chloride (FeCl<sub>3</sub>) (0.5 mL at 0.1% w/v) and deionized water (2.5 mL) were combined with the solution's supernatant (2.5 mL). After 30 minutes of incubation, the solution was measured at λ<sub>max</sub> 700 nm against a blank. The FRAP was calculated from the regression equation of the calibration curve ( $y = 0.0108x - 0.0073$ , with  $R^2 = 0.994$ ). The reducing

power of the extract was expressed in milligram equivalents of standard ascorbic acid in grams of dry matter.

#### Free Radical Scavenging Activity DPPH

The antioxidant capacity of the extracts was determined using the 2,2-diphenyl-1-picrylhydrazyl (DPPH) method. This modified method, adopted from,<sup>27</sup> assesses the extracts' ability to neutralize free radicals. A 0.2 mM ethanolic solution of DPPH was prepared and stored in an amber flask away from sunlight. The cake extracts were diluted 1:1000 (v/v) in ethanol. Subsequently, 0.8 mL of DPPH solution were mixed with 4 mL of the diluted samples. Using a SCILOGEX SP-UV1100 spectrophotometer and comparing it to a blank, absorbance was measured at 517 nm. The percentage of free radical inhibition by DPPH was determined using the following formula:

$$\text{Inhibition of DPPH \%} = \frac{Ac - As}{Ac} \times 100$$

Where, "As" represents the sample absorbance, and "Ac" the control absorbance. The results are expressed as milligrams of ascorbic acid equivalent per gram of dry matter. The regression equation of the ascorbic acid standard curve was  $y = 22.718x + 0.6987$  with  $R^2 = 0.9968$ .

#### Statistical Analysis

The presented results are the averages of analyses conducted in two or three replicates. The values are represented as mean ± standard deviation. The ANOVA was carried out, and mean values were separated at  $p < 0.05$  significance level. Principal component analysis and a correlation matrix were applied to the mean data. For the correlation analyses, the Pearson correlation coefficient method was used to assess the strength and direction of the linear relationships between the variables. All statistical analyses, including the correlation analyses, were performed using Origin Pro "Version 2022" from OriginLab Corporation (Northampton, Massachusetts, USA: OriginLab Corporation).

#### Results

##### Biogenic Elements

Minerals and trace elements play a crucial role as micronutrients essential for the proper functioning of the organism<sup>28,29</sup> and contribute significantly to various physiological functions.<sup>30</sup> However, an

excess of certain trace or certain essential elements can pose serious health risks for humans and animals.<sup>31</sup> Regardless of the source, oilseed cakes serve as valuable suppliers of biogenic elements, offering concentrations up to 9000 ppm, notably in K, Ca, Na, Fe, and Zn.<sup>32</sup> In this study, the analysis focused on ten biogenic elements, including Zn, Fe, B, Mn, Cu, Mg, Ca, Na, K and P, in six different oilseed cakes. The mean values of the results are presented in Table 1. The data obtained reveal that K, P, Mg, Ca, and Na are the predominant elements in the examined oilseed cakes. Potassium (K) emerges as the most abundant element across all samples, with concentration ranging from 11093.97 ± 241.75 mg/kg in Argan Press cake (ARPC) to 20682.98 ± 255.83 mg/kg found in Soybean Press Cake (SBPC). Phosphorus (P) follows as the second most abundant element, with concentration ranging from 5186.33 ± 36.52 (SBPC) to 11787.80 ± 919.43 in Sesame Press Cake (SEPC) mg/kg. Calcium (Ca) takes the third position, varying from 3901.66 ± 316.03 found in Sunflower Press Cake (SFPC) to 7711.12 ± 1.98 mg/kg (SBPC). Magnesium (Mg) ranks fourth, with levels ranging from 2646.93 ± 28.84 (SBPC) to 5534.94 ± 431.71 mg/kg (SEPC). Sodium (Na) occupies the fifth position, ranging from 343.34 ± 1.12 (ARPC) to 80.88 ± 6.31 mg/kg found in Almond Press Cake (ALPC). The average content of the macro elements in all analysed samples is K > P > Ca > Mg > Na. Concerning the microelements (Zn, Mn, B, and Cu), they were detected in low concentrations, except Fe. The highest levels of these mineral elements were obtained as follows: Zn (92.49±7.49, SFPC; 88.42±1.01, ARPC), Mn (61.46±4.79, SEPC), B (36.45±2.84, NIPC; 33.92±0.1, SBPC) and Cu (33.02±2.67, SFPC). Nigella Press Cake (NIPC) exhibited the highest concentration of Fe (659.21±51.42 mg/kg). Notably, the results for sunflower press cake (SFPC) differ from those reported by,<sup>38</sup> particularly for Fe, which is found in small quantities (4.71 ± 2.71 mg/kg) compared to our results (105.35±8.53 mg/kg). Meanwhile, the values of Mn and Zn are higher than our findings. Conversely, the Fe and Zn values in sesame press cake (SEPC) are lower than those obtained by.<sup>39</sup>

### Proximate Composition

#### Ash Content

Ash refers to the inorganic residue left after the combustion or complete acid-facilitated oxidation

of organic compounds in food.<sup>40</sup> The results indicate that SBPC exhibits the highest ash content (5.78 g/100 g), followed by ARPC (5.24 g/100 g). Consequently, these two types of oilcake stand out as the richest in minerals, presenting potential advantages for animal nutrition or soil fertilization. ALPC and SFPC display intermediate ash contents (4.3 and 4.01 g/100 g), while NIPC and SEPC have the lowest ash contents (3.01, 2.7 g/100 g, respectively).

#### Proteins Content

Proteins, essential biological macromolecules found in all living cells, serve as rich sources of numerous essential amino acids and vital macro-nutrients.<sup>42,43</sup> Regardless of their origin, all protein share the same fundamental role within the body.<sup>21</sup> They are recognized as the nutritional elements capable of slowing down or even preventing the loss of muscular strength and mass.<sup>44</sup> Oilcakes with a high protein content offer enhanced nutritional value, making them a substantial protein source for both animals and humans. In the context of the oilcake presented in Table 2, the results reveal that ARPC and SBPC provide the highest protein content (45.90 and 44.85 g/100 g, respectively), imparting significant nutritional value for animal or human consumption. SEPC demonstrates an average protein content (36.43 g/100 g), while ALPC, NIPC, and SFPC exhibit a lower protein content (32.56, 25.62 and 26.43 g/100 g respectively).

#### Residual Oil Content

Vegetable oil stands out as one of the primary energy sources for humans, predominantly used in cooking but also finding application in beauty products, food supplement capsules, and various other uses.<sup>49,50</sup> Residual cake oil represents the amount of oil retained in the cake after extraction from the oilseeds. This quantity is influenced by seed type, extraction method, and the extent of de-oiling. Our research indicates that solvent extraction of oil highlights that ALPC with the highest residual oil content (21.18 ± 0.42 g/100 g), followed by SEPC (18.43 ± 0.25 g/100 g) and NIPC (16.02 ± 0.11 g/100 g), with ARPC trailing (12.61 ± 0.42 g/100 g).

**Table 1 : Mean values of mineral elements in the studied press cakes**

Mineral	ARPC	ALPC	NIPC	SEPC	SFPC	SBPC
Zn	88.42±1.01 <sup>a</sup>	75.88±5.88 <sup>a</sup>	71.13±5.54 <sup>ab</sup>	47.77±3.72 <sup>b</sup>	92.49±7.49 <sup>a</sup>	79.19±0.84 <sup>a</sup>
Fe	78.31±0.29 <sup>b</sup>	77.05±6.01 <sup>b</sup>	659.21±51.42 <sup>a</sup>	152.92±11.92 <sup>b</sup>	105.35±8.53 <sup>b</sup>	66.31±0.39 <sup>b</sup>
B	28.23±0.07 <sup>ab</sup>	27.58±2.15 <sup>ab</sup>	36.45±2.84 <sup>a</sup>	19.33±1.51 <sup>b</sup>	32.03±2.59 <sup>a</sup>	33.92±0.15 <sup>a</sup>
Mn	48.04±0.14 <sup>ab</sup>	32.53±2.53 <sup>bc</sup>	32.66±2.54 <sup>bc</sup>	61.46±4.79 <sup>a</sup>	40.92±3.31 <sup>b</sup>	21.15±0.09 <sup>c</sup>
Cu	12.40±0.04 <sup>b</sup>	15.08±1.17 <sup>b</sup>	14.27±1.11 <sup>b</sup>	4.81±0.38 <sup>c</sup>	33.02±2.67 <sup>a</sup>	13.23±0.17 <sup>b</sup>
Mg	4081.39±2.28 <sup>ab</sup>	5103.83±398.08 <sup>a</sup>	3230.88±252.08 <sup>b</sup>	5534.94±431.71 <sup>a</sup>	5218.37±422.68 <sup>a</sup>	2646.93±28.84 <sup>b</sup>
Ca	7576.18±10.94 <sup>a</sup>	4659.77±363.46 <sup>b</sup>	7972.06±621.81 <sup>a</sup>	7855.29±612.72 <sup>a</sup>	3901.66±316.03 <sup>b</sup>	7711.12±1.98 <sup>a</sup>
Na	343.34±1.12 <sup>a</sup>	80.88±6.31 <sup>c</sup>	119.12±9.28 <sup>c</sup>	135.28±10.56 <sup>c</sup>	255.98±20.73 <sup>b</sup>	112.39±3.18 <sup>c</sup>
K	11093.97±241.75 <sup>b</sup>	14137.83±1102.75 <sup>b</sup>	11745.83±916.17 <sup>b</sup>	12495.15±974.62 <sup>b</sup>	15158.56±1227.84 <sup>b</sup>	20682.98±255.83 <sup>a</sup>
P	6022.86±45.02 <sup>c</sup>	9744.01±760.03 <sup>ab</sup>	6807.75±531.03 <sup>bc</sup>	11787.80±919.43 <sup>a</sup>	10511.07±851.39 <sup>a</sup>	5186.33±36.52 <sup>c</sup>

The values presented are the means ± standard deviation of three replicates. Distinct letters within the same column signify statistically significant differences (p < 0.05). ARPC : Argan Press Cake, ALPC : Almond Press Cake, NIPC : Nigella Press Cake, SEPC : Sesame Press Cake, SFPC : Sunflower Press Cake, SBPC : Soybean Press Cake.

**Table 2: Proximate composition (protein content, ash content, residual oil content, total carbohydrate and moisture content), and energy value of the six press cakes studied**

	ARPC	ALPC	NIPC	SEPC	SFPC	SBPC
Protiens	45.90±0.10 <sup>b</sup>	32.56±2.53 <sup>a</sup>	25.62±1.99 <sup>a</sup>	36.43±2.84 <sup>ab</sup>	26.43±2.06 <sup>a</sup>	44.85±1.10 <sup>b</sup>
Ash %	5.18±0.38 <sup>cd</sup>	4.3±0.33 <sup>bc</sup>	3.01±0.23 <sup>ab</sup>	2.7±0.21 <sup>a</sup>	4.01±0.31 <sup>ac</sup>	5.78±0.10 <sup>d</sup>
Residual Oil	12.61±0.8 <sup>b</sup>	21.18±0.42 <sup>e</sup>	16.02±0.11 <sup>c</sup>	18.43±0.25 <sup>d</sup>	1.05±0.55 <sup>a</sup>	1.25±0.35 <sup>a</sup>
Carbohydrates	29.80±0.38 <sup>a</sup>	34.15±0.83 <sup>b</sup>	51.31±0.12 <sup>d</sup>	39.40±0.85 <sup>c</sup>	56.41±0.85 <sup>e</sup>	42.58±0.41 <sup>c</sup>
Moisture	6.46±0.32 <sup>bc</sup>	7.81±0.05 <sup>c</sup>	3.22±0.10 <sup>a</sup>	3.05±0.10 <sup>a</sup>	12.1±0.5 <sup>d</sup>	5.54±0.12 <sup>b</sup>
Energy Value	350.96 ± 8.55 <sup>b</sup>	406.01 ± 13.63 <sup>a</sup>	416.71 ± 6.63 <sup>a</sup>	415.18 ± 13.10 <sup>a</sup>	312.02 ± 0.15 <sup>b</sup>	306.80 ± 1.67 <sup>b</sup>

ARPC : Argan Press Cake, ALPC : Almond Press Cake, NIPC : Nigella Press Cake, SEPC : Sesame Press Cake, SFPC : Sunflower Press Cake, SBPC : Soybean Press Cake.

Table 3 : Average percentages of fatty acids found in press cakes studied

Fatty Acids	ARPC	ALPC	NIPC	SEPC	SFPC	SBPC
Myristic acid (C14: 0)	0.13 ± 0.01 <sup>a</sup>	0.11 ± 0.09 <sup>a</sup>	0.14 ± 0.01 <sup>a</sup>	0.15 ± 0.05 <sup>a</sup>	0.20 ± 0.01 <sup>a</sup>	0.10 ± 0.01 <sup>a</sup>
Palmitic acid (C16: 0)	13.07 ± 0.10 <sup>a</sup>	7.12 ± 0.15 <sup>e</sup>	12.05 ± 0.09 <sup>b</sup>	10.5 ± 0.7 <sup>d</sup>	7.38 ± 0.05 <sup>e</sup>	11.32 ± 0.05 <sup>c</sup>
Palmitoleic acid (C16 :1)	0.10 ± 0.01 <sup>b</sup>	0.62 ± 0.11 <sup>a</sup>	0.21 ± 0.01 <sup>b</sup>	0.25 ± 0.10 <sup>ab</sup>	0.20 ± 0.05 <sup>b</sup>	0.20 ± 0.05 <sup>b</sup>
Stearic acid (C18: 0)	5.78 ± 0.01 <sup>ab</sup>	2.70 ± 0.12 <sup>c</sup>	3.12 ± 0.01 <sup>c</sup>	5.95 ± 0.28 <sup>a</sup>	5.22 ± 0.20 <sup>ab</sup>	4.82 ± 0.22 <sup>b</sup>
Oleic acid (C18: 1)	47.88 ± 0.20 <sup>b</sup>	67.65 ± 0.23 <sup>a</sup>	23.14 ± 0.09 <sup>e</sup>	43.36 ± 0.84 <sup>c</sup>	36.29 ± 0.10 <sup>d</sup>	22.21 ± 0.20 <sup>e</sup>
Linoleic acid (C18: 2)	32.10 ± 0.15 <sup>e</sup>	21.95 ± 0.15 <sup>f</sup>	58.06 ± 0.08 <sup>a</sup>	39.31 ± 0.70 <sup>d</sup>	50.59 ± 0.07 <sup>c</sup>	54.17 ± 0.07 <sup>b</sup>
Linolenic acid (C18: 3)	0.10 ± 0.01 <sup>b</sup>	0.12 ± 0.09 <sup>b</sup>	0.22 ± 0.01 <sup>b</sup>	0.2 ± 0.07 <sup>b</sup>	0.19 ± 0.08 <sup>b</sup>	7.13 ± 0.07 <sup>a</sup>
Arachidic acid (C20: 0)	0.34 ± 0.05 <sup>a</sup>	0.20 ± 0.10 <sup>a</sup>	0.14 ± 0.01 <sup>a</sup>	0.55 ± 0.15 <sup>a</sup>	0.21 ± 0.01 <sup>a</sup>	0.35 ± 0.03 <sup>a</sup>
Galdoleic acid (C20:1)	0.40 ± 0.05 <sup>a</sup>	0.10 ± 0.05 <sup>a</sup>	0.32 ± 0.01 <sup>a</sup>	0.30 ± 0.10 <sup>a</sup>	0.10 ± 0.05 <sup>a</sup>	0.10 ± 0.05 <sup>a</sup>
SFA*	19.32 ± 0.17 <sup>a</sup>	10.13 ± 0.46 <sup>d</sup>	15.45 ± 0.12 <sup>bc</sup>	17.15 ± 1.18 <sup>ab</sup>	13.01 ± 0.27 <sup>cd</sup>	16.59 ± 0.31 <sup>ab</sup>
MUFA**	48.38 ± 0.26 <sup>b</sup>	68.37 ± 0.39 <sup>a</sup>	23.67 ± 0.11 <sup>e</sup>	43.91 ± 1.04 <sup>c</sup>	36.59 ± 0.2 <sup>d</sup>	22.51 ± 0.3 <sup>e</sup>
PUFA***	32.2 ± 0.16 <sup>e</sup>	22.07 ± 0.24 <sup>f</sup>	58.28 ± 0.09 <sup>b</sup>	39.51 ± 0.77 <sup>d</sup>	50.78 ± 0.15 <sup>c</sup>	61.3 ± 0.14 <sup>a</sup>

The values are reported as mean ± standard deviation based on three replicates. SFA refers to saturated fatty acids, MUFA stands for mono-unsaturated fatty acids, and PUFA denotes poly-unsaturated fatty acid. ARPC : Argan Press Cake, ALPC : Almond Press Cake, NIPC : Nigella Press Cake, SEPC : Sesame Press Cake, SFPC : Sunflower Press Cake, SBPC : Soybean Press Cake.

**Table 4: Mean values of individual sterols (%) as well as total sterols found in the studied press cakes. The values are expressed as mean ± standard deviation based on three replicates**

Phetosterols	ARPC	ALPC	NIPC	SEPC	SFPC	SBPC
Cholesterol	0.51 ± 0.05 <sup>b</sup>	0.51 ± 0.10 <sup>b</sup>	1.26 ± 0.05 <sup>a</sup>	0.22 ± 0.01 <sup>b</sup>	0.10 ± 0.10 <sup>b</sup>	0.50 ± 0.10 <sup>b</sup>
Campesterol	0.20 ± 0.05 <sup>d</sup>	3.50 ± 1.50 <sup>cd</sup>	11.61 ± 0.03 <sup>ab</sup>	17.83 ± 0.15 <sup>a</sup>	9.5 ± 1.05 <sup>bc</sup>	16.5 ± 2.50 <sup>ab</sup>
Stigmasterol	-----	3.12 ± 0.5 <sup>b</sup>	ND	6.45 ± 0.10 <sup>b</sup>	8.5 ± 1.50 <sup>b</sup>	15.5 ± 1.50 <sup>a</sup>
β-Sitosterol	-----	73.5 ± 3.50 <sup>a</sup>	49.24 ± 0.05 <sup>b</sup>	61.5 ± 2.25 <sup>ab</sup>	60.5 ± 2.25 <sup>ab</sup>	54.5 ± 3.50 <sup>b</sup>
Δ-5-Avenasterol	-----	11.5 ± 2.50 <sup>a</sup>	11.10 ± 0.04 <sup>b</sup>	7.55 ± 1.10 <sup>c</sup>	3.70 ± 1.51 <sup>d</sup>	2.01 ± 0.51 <sup>e</sup>
Schotenol	46.02 ± 0.17 <sup>a</sup>	-----	-----	-----	-----	-----
Spenasterol	38.19 ± 0.09 <sup>a</sup>	-----	-----	-----	-----	-----
Stigma-8-22-dien-3b-ol	4.12 ± 0.5 <sup>a</sup>	-----	-----	-----	-----	-----
Δ-7-Stigmasterol	-----	2.5 ± 0.50 <sup>bc</sup>	0.78 ± 0.08 <sup>c</sup>	0.35 ± 0.12 <sup>c</sup>	7.51 ± 0.50 <sup>a</sup>	3.50 ± 0.50 <sup>b</sup>
Δ-7-Avenasterol	5.24 ± 0.09 <sup>a</sup>	1.5 ± 0.51 <sup>b</sup>	2.47 ± 0.07 <sup>ab</sup>	0.15 ± 0.01 <sup>b</sup>	4.57 ± 1.21 <sup>ab</sup>	3.07 ± 1.50 <sup>ab</sup>
Others	5.72 ± 0.05 <sup>a</sup>	3.7 ± 1.51 <sup>a</sup>	8.04 ± 0.05 <sup>a</sup>	5.65 ± 2.50 <sup>a</sup>	5.62 ± 1.50 <sup>a</sup>	4.42 ± 1.50 <sup>a</sup>
Total sterol mg/100g	185.25 ± 15.50 <sup>c</sup>	350 ± 20.50 <sup>b</sup>	156.9 ± 5.5 <sup>c</sup>	565 ± 10.55 <sup>a</sup>	365 ± 15.50 <sup>b</sup>	285 ± 25.50 <sup>b</sup>

values are expressed as mean ± standard deviation based on three replicates.

ARPC : Argan Press Cake, ALPC : Almond Press Cake, NIPC : Nigella Press Cake, SEPC : Sesame Press Cake, SFPC : Sunflower Press Cake, SBPC : Soybean Press Cake.



**Table 5: Fibers content (%) of the analyzed samples**

	ARPC	ALPC	NIPC	SEPC	SFPC	SBPC
Crude Fiber	9.76 ± 2.93	41.14 ± 12.34	12.91 ± 3.87	13.50 ± 4.05	12.50 ± 3.75	10.58 ± 3.17
NDF	38.00 ± 11.40	16.40 ± 4.92	34.60 ± 10.38	19.20 ± 5.76	54.40 ± 16.32	17.20 ± 5.16
ADF	29.20 ± 8.76	18.30 ± 5.49	33.80 ± 10.14	20.30 ± 6.09	47.50 ± 14.25	16.60 ± 4.98
ADL	18.80 ± 5.64	9.10 ± 2.73	9.40 ± 2.82	13.00 ± 3.90	19.00 ± 5.70	2.20 ± 0.66

ARPC: Argan Press Cake, ALPC: Almond Press Cake, NIPC: Nigella Press Cake, SEPC: Sesame Press Cake, SFPC: Sunflower Press Cake, SBPC: Soybean Press Cake

**Table 6 : Total phenolic and flavonoids content as well as the antioxidant activities of the examined press cakes**

Press cakes	ARPC	ALPC	NIPC	SEPC	SFPC	SBPC
TPC (mg GAE /g DM)	5.11 ± 0.01 <sup>b</sup>	0.46 ± 0.01 <sup>f</sup>	3.53 ± 0.06 <sup>c</sup>	0.76 ± 0.02 <sup>e</sup>	9.82 ± 0.02 <sup>a</sup>	2.51 ± 0.04 <sup>d</sup>
TFC (mg QE /g DM)	0.63 ± 0.07 <sup>e</sup>	2.21 ± 0.19 <sup>d</sup>	9.13 ± 0.11 <sup>b</sup>	3.94 ± 0.08 <sup>c</sup>	15.44 ± 0.04 <sup>a</sup>	3.36 ± 0.11 <sup>c</sup>
FRAP (mg AAE /g DM)	1.17 ± 0.01 <sup>b</sup>	0.56 ± 0.03 <sup>b</sup>	2.68 ± 0.12 <sup>b</sup>	1.6 ± 0.2 <sup>b</sup>	8.90 ± 1.2 <sup>a</sup>	1.57 ± 0.06 <sup>b</sup>
DPPH (mg AAE /g DM)	8.06 ± 0.08 <sup>b</sup>	7.98 ± 0.17 <sup>b</sup>	1.37 ± 0.01 <sup>c</sup>	6.63 ± 0.17 <sup>b</sup>	34.05 ± 0.8 <sup>a</sup>	8.49 ± 0.34 <sup>b</sup>
DPPH %	19.03 ± 0.19 <sup>b</sup>	18.84 ± 0.38 <sup>b</sup>	3.81 ± 0.02 <sup>c</sup>	15.76 ± 0.3 <sup>b</sup>	78.07 ± 1.92 <sup>a</sup>	19.99 ± 0.76 <sup>b</sup>

ARPC: Argan Press Cake, ALPC: Almond Press Cake, NIPC: Nigella Press Cake, SEPC: Sesame Press Cake, SFPC: Sunflower Press Cake, SBPC: Soybean Press Cake.

### Carbohydrates

Carbohydrates are compounds primarily composed of carbon, hydrogen, and oxygen molecules, although they can also contain nitrogen and phosphorus.<sup>56</sup> Constituting over 50% of daily energy needs,<sup>57</sup> carbohydrates, along with fats and proteins, play a crucial role in providing the energy essential for human sustenance, forming an integral part of a balanced diet.<sup>19</sup> The results of our analysis show that the carbohydrate content varies significantly between the six cakes studied. SFPC has the highest carbohydrate content (56.41 ± 0.85 g/100 g), followed by NIPC (51.31 ± 0.12 g/100 g) and SBPC (42.58 ± 0.41 g/100 g).

### Energy Value

The energy value (EV) was also determined in the course of this study. Our results show that NIPC has the highest energy value (416.71 ± 6.63 kcal/100g), followed by SEPC (415.18 ± 13.10 kcal/100g) and

ALPC (406.01 ± 13.63 kcal/100g). These three types of oilcakes have a high residual lipid content, which gives them a high energy value. ARPC and SFPC respectively recorded average values (350.96 ± 8.55; 312.02 ± 0.15 kcal/100g), while SBPC showed the lowest value of all the oilcakes studied (306.80 ± 1.67 kcal/100g).

### Moisture

Moisture content is an essential factor in maintaining the stability of an oilcake over long periods of time.<sup>59</sup> A moisture level under 12% is deemed safe for storage as it inhibits the rapid proliferation of mold.<sup>38</sup> NIPC and SEPC stand out with the lowest moisture levels (3.22 ± 0.10 g/100 g and 3.05 ± 0.10 g/100 g, respectively), indicating excellent resistance to mould growth and high storage stability. On the other hand, SFPC has the highest moisture content (12.1 ± 0.5 g/100 g), requiring extra vigilance during storage due to the increased risk of mould growth. ARPC

has the lowest moisture content after Nigella and Sesame cakes ( $6.46 \pm 0.32$  g/100 g), while ALPC shows respectable stability with a moisture content of  $7.81 \pm 0.05$  g/100 g. SBPC, with a moisture content of  $5.54 \pm 0.12$  g/100 g, is in the intermediate range. The results found for SEPC are close to those obtained by.<sup>60</sup>

### Fatty Acids Composition

In our study, analysis of the oilcake identified nine different fatty acids representing more than 99.00 g/100 g of the total oil. A significant difference was observed between the various oilcakes studied (Table 3). In general, the oils under examination exhibit a notable presence of unsaturated fatty acids (oleic acid and linoleic acid), ranging from 80.58 g/100 g in ARPC to 90.44 g/100 g in ALPC. Simultaneously, they demonstrate a lower concentration of saturated fatty acids, varying from 10.13 g/100 g to 19.32 g/100 g found in ALPC and ARPC, respectively. Among saturated fatty acids found in the results of this study, the most abundant were Palmitic acid (C16:0) which varies considerably, with high concentrations in ARPC oil ( $13.07 \pm 0.10$  g/100 g) and NIPC oil ( $12.05 \pm 0.09$  g/100 g), while ALPC oil has the lowest content ( $7.12 \pm 0.15$  g/100 g). This variation can influence the physical and chemical properties of oilcakes, thus influencing their use in industrial applications. However, these findings are in harmony with the results declared by<sup>15,68</sup> in ARPC and ALPC, respectively. The second highest concentration of saturated fatty acid is stearic acid (C18:0), which is present in the samples analysed in varying proportions, when SEPC, ARPC and SFPC oils stand out here with a significant content ( $5.95 \pm 0.28$ ;  $5.78 \pm 0.01$ ;  $5.22 \pm 0.20$  g/100 g) respectively. Regarding myristic acid (C14:0), its levels are relatively similar between the different oil cakes, with values ranging from 0.10 to 0.20 g/100 g. Nevertheless, the present study revealed that the myristic acid content in SEPC oil is slightly higher than those reported by.<sup>60</sup>

In terms of unsaturated fatty acids, our findings reveal a high predominance of oleic and linoleic acids. These two fatty acids emerged as the main constituents, present in significant quantities compared to the other unsaturated fatty acids examined. In fact the highest content of oleic acid (C18:1) was identified in ALPC ( $67.65 \pm 0.23$  g/100 g), ARPC ( $47.88 \pm 0.20$  g/100 g), SEPC ( $43.36 \pm$

$0.84$  g/100 g) and these findings align with the results reported in earlier studies,<sup>68,15,60,68</sup> which underlines their particular nutritional potential. Nevertheless, oleic acid (C18:1) is considered to be the most common MUFA in the human diet.<sup>69</sup> Linoleic acid (C18:2), which is one of the polyunsaturated fatty acids in the omega-6 family and is an essential component of various food sources, such as vegetable oils, nuts and seeds. Notably, this fatty acid cannot be synthesised by the human body,<sup>69</sup> underlining its essential status and the need for its intake through the diet. The highest concentration of this fatty acid was found in NIPC oil ( $58.06 \pm 0.08$  g/100 g); this was closely followed by SBPC oil and SFPC oil, which also showed high levels of this fatty acid ( $54.17 \pm 0.07$ ;  $50.59 \pm 0.07$  g/100 g) respectively. ALPC oil, on the other hand, stands out for its relatively low linoleic acid content ( $21.95 \pm 0.15$  g/100 g).

When it comes to palmitoleic acid (C16:1), which is one of the principal MUFAs in the omega-7 ( $\omega$ -7) family,<sup>69</sup> ALPC has a significantly higher concentration ( $0.62 \pm 0.11$  g/100 g), among the oil cakes studied, followed by SEPC oil ( $0.25 \pm 0.10$  g/100 g). Whereas the last value of this fatty acid was registered in ARPC oil ( $0.10 \pm 0.01$  g/100 g). The obtained results in ARPC, SEPC and SFPC are higher than those reported in other studies conducted by<sup>15,60,73</sup> respectively.

### Phytosterols Composition

Phytosterols, also known as sterols, represent the second class of lipids studied. They are widely distributed in plants and are similar to cholesterol in their chemical structure and physiological properties.<sup>62,74</sup> Phytosterols have considerable nutritional and health value, as they simultaneously reduce blood cholesterol levels and the risk of cardiovascular disease.<sup>19,25</sup> In actual fact, over 100 different phytosterols have been identified in vegetable oils, of which  $\beta$ -sitosterol, campesterol, stigmasterol, brassicasterol and  $\Delta$ 5-avenasterol were the main types.<sup>62</sup> The phytosterol composition of the different press cake oils studied is shown in Table 4. Among the oils studied, SEPC oil had the highest concentration of phytosterols, with a quantity of  $565 \pm 10.55$  mg/100 g, followed by SFPC, ALPC and SBPC oils, which had concentrations of  $365 \pm 15.50$  mg/100 g,  $350 \pm 20.50$  mg/100 g and  $285 \pm 25.50$  mg/100 g respectively, whereas NIPC oil has

the lowest value ( $156.9 \pm 5.5$  mg/100 g). Ten sterols were detected, including  $\beta$ -sitosterol, stigmasterol, campesterol,  $\Delta$ 5-avenasterol,  $\Delta$ -7-stigmastenol and  $\Delta$ -7-avenasterol.  $\beta$ -Sitosterol was the main phytosterol in press cakes oils studied, and the highest amount of  $\beta$ -sitosterol was presented in ALPC oil ( $73.5 \pm 3.50$  mg/kg), followed by SEPC oil ( $61.5 \pm 2.25$  mg/kg), SFPC oil ( $60.5 \pm 2.25$  mg/kg), then SBPC oil ( $54.5 \pm 3.50$  mg/kg) and finally in NIPC oil ( $49.24 \pm 0.05$  mg/kg). Other predominant sterols included  $\Delta$ -5-avenasterol in ALPC and NIPC oils and campesterol in other press cakes oils were identified. SEPC and SBPC oils stood out as the richest sources of campesterol (17.83 and 16.5 mg/kg respectively). However, high levels of  $\Delta$ -5-avenasterol were recorded in ALPC and NIPC oils ( $11.5 \pm 2.50$  and  $11.10 \pm 0.04$  mg/kg respectively). On the other hand, a low cholesterol content, less than 1% by weight of sterols, was found in all press cake oils, with the exception of NIPC oil, which contained  $1.26 \pm 0.05$  mg/kg. The  $\Delta$ 7-sterols ( $\Delta$ 7-avenasterol and  $\Delta$ 7- stigmasterol) constituted an important group of phytosterols. With regard to  $\Delta$ 7-avenasterol, high values were observed in ARPC oil ( $5.24 \pm 0.09$  mg/kg), while low values were recorded in SEPC oil ( $0.15 \pm 0.01$  mg/kg). As for the  $\Delta$ 7-stigmastenol content, it varied from 0.35 mg/kg in ALPC to 7.51 mg/kg in SFPC. ARPC stood out as the only one to contain schotenol (46.02 mg/kg), spenasterol (38.19 mg/kg) and stigma-8-22-diene-3b-ol, (4.12 mg/kg).

All sterol composition values for ARPC oil are within the standard range for extra virgin argan oil, except for cholesterol with a slight increase of 0.11.<sup>75</sup> Several studies confirm the predominant sterol compound in argan oil was spinasterol (47.35 %),<sup>76</sup> (45.5%–46.5%)<sup>77</sup> and ( $44.52 \pm 0.07$  % -  $46.22 \pm 0.07$  %).<sup>18</sup> In terms of abundant sterol ( $\beta$ -sitosterol), ALPC oil is similar to *Ziziphus lotus* almonds ( $71.99 \pm 0.82$  %) <sup>19</sup> and to almond germplasm (72.4 – 73.9 %).<sup>78</sup>

### Fibers Content

Crude fiber is the residue obtained by subjecting the feed to a dual hydrolysis process involving both acid (with  $H_2SO_4$ ) and alkaline (with KOH) treatments.<sup>83</sup> The results (Table 5) show that ARPC has the lowest fiber content, with an average of 9.76

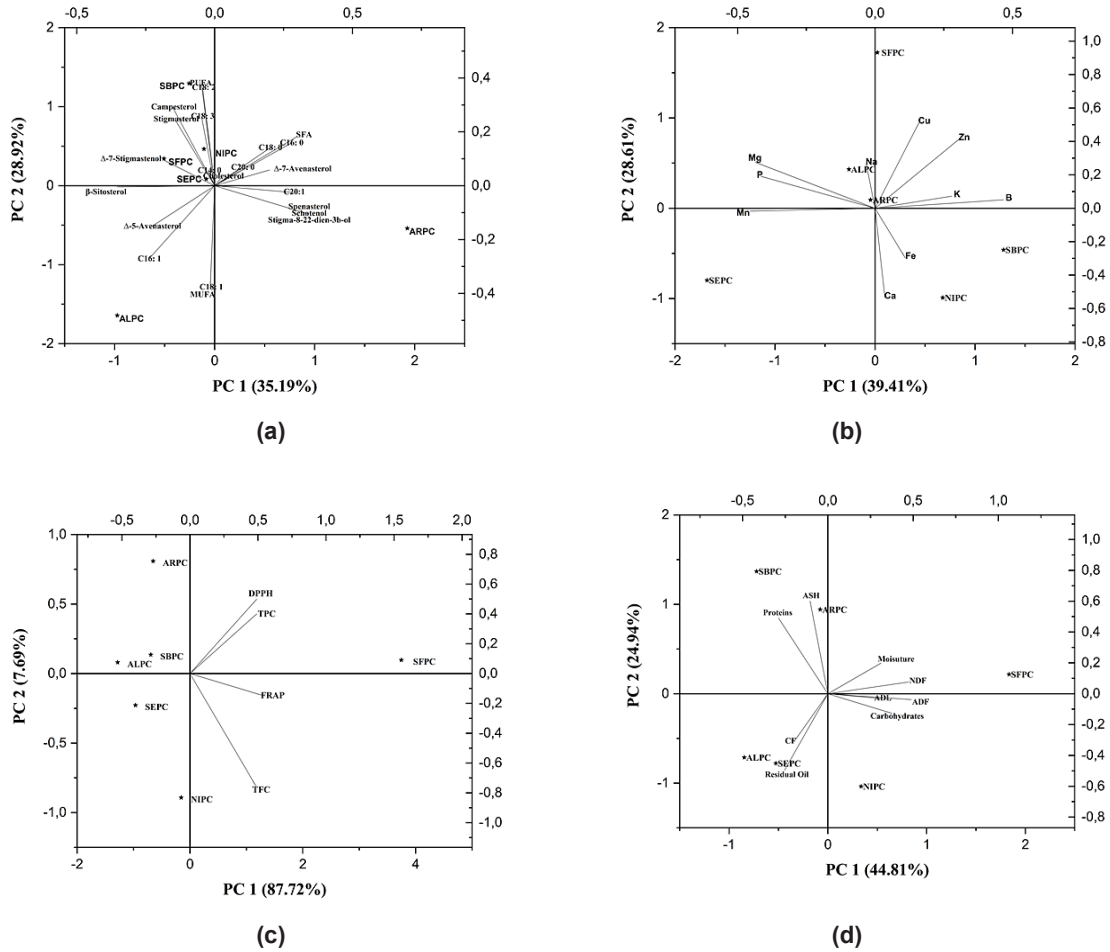
%. Conversely, ALPC had the highest fiber content, with an average of 41.14%. NIPC, SEPC and SFPC have intermediate fiber contents, with averages of 12.91, 13.50 and 12.50 % respectively. SBPC has the second lowest fiber content, with an average of 10.58%.

### TPC, TFC and Antioxidant Activities of Press Cakes Extract's

Flavonoids and other phenolic compounds are frequently recognized as secondary metabolites in plants, characterised by an aromatic ring containing at least one hydroxyl group.<sup>90</sup> These plant-derived compounds have been attributed with various properties, including antioxidant, anticancer, antibacterial, cardioprotective, and anti-inflammatory effects.<sup>19,91</sup> The total phenols content (TPC) and total favonoids content (TFC) quantified spectrophotometrically in different oilseed cakes are presented in Table 6. As can be seen in Table 6, TPC values in the studied press cakes were in the range of  $0.46 \pm 0.01$  mg GAE/g DM (ALPC) to  $9.82 \pm 0.02$  mg GAE/g DM (SFPC). However, the ranges of contents of TFC were from  $0.63 \pm 0.07$  mg QE/g DM (ARPC) to  $15.44 \pm 0.04$  mg QE/g DM (SFPC). SFPC demonstrated exceptionally high levels of TPC and TFC, as shown in Table 6. This finding contrasts with results obtained in other studies, including that conducted by.<sup>92</sup> However, it is important to note that, despite these high levels, our results for TPC are lower than those reported by,<sup>93</sup> who documented a value of  $17.21 \pm 0.89$  mg GAE/g DM. For SEPC, TPC was  $0.76 \pm 0.02$  mg GAE/g DM. It is lower than values obtained by,<sup>60</sup> but it is smaller than the finding of.<sup>92</sup> In literature, <sup>94,95</sup> reported a value of TPC of  $7.9 \pm 0.6$  and  $7.9 \pm 0.7$  mg GAE/g, respectively in ARPC, which are slightly higher than our finding ( $5.11 \pm 0.01$  mg GAE/g DM). However, our results found in NIPC are in line with those reported by Kadam.<sup>96</sup>

### Principal Component Analysis

Principal Component Analysis (PCA) is widely utilised in various fields, encompassing pomological investigations, chemometrics, and food science. In this study, PCA was employed to complement the results derived from the Analysis of Variance (ANOVA).



**Fig.1: Graphical representation of the projections of the two main components with the highest influence on the press cakes studied. The black segments symbolise the dependent variables, while the plotted points represent the mean values. TPC: total phenolic content, TFC: total flavonoid content, ADF: acid detergent fiber, NDF: neutral detergent fiber, ADL: acid detergent lignin, CF: crude fiber, DPPH: 2,2-diphenyl-1-picrylhydrazyl, and FRAP: ferric ion reducing antioxidant power**

The graphical representation in Figure 1-a illustrates the distribution of press cake oils with respect to their fatty acids and sterol profiling. Figure 1-a visually emphasizes the clustering of samples along PC1 (35.19%) and PC2 (28.92%), collectively explaining 64.11% of the variance. PC1 effectively segregates samples based on the content of analysed sterols. Notably, samples positioned to the left of the plot exhibit higher concentrations of the targeted compounds compared to those located on the right. Conversely, PC2 appears to differentiate samples based on their distribution of fatty acids, with samples at the top of the graph being richer

in targeted compounds than those at the bottom. Further examination reveals specific associations between samples and their respective compositions. For instance, the sample labeled 'ARPC' is linked to elevated levels of schoterol, spenasterol, and stigma-8-22-dien-3b-ol. In contrast, 'SBPC' is characterized by higher levels of polyunsaturated fatty acids (PUFA), notably (C18:2), along with increased concentrations of campesterol and stigmasterol. 'NIPC' is associated with a notable abundance of saturated fatty acids (SFA), particularly (C16:0), (C18:0), and (C20:0), while 'ALPC' is distinguished by higher amounts of monounsaturated fatty

acids (MUFA), primarily (C18:1). This observed data distribution aligns perfectly with previously discussed findings concerning the fatty acid and sterol composition of the press cake oils. Figure 1-b illustrates the distribution of press cakes based on their mineral profiling, thus providing the basis for construction of a data matrix suitable for carrying out a principal component analysis (PCA) representing the first two principal components (PCs) of the samples. The resulting plot, as depicted in Figure 1-b, reveals a distinct separation of press cakes along PC1 (39.41%) and PC2 (28.61%). Notably, samples from 'ARPC' and 'SBPC' exhibit clustering toward positive values of PC1, indicating higher concentrations of Fe, Ca, and Mg. Similarly, 'ARPC,' 'SEPC,' and 'ALPC' are associated with elevated levels of Na, Mg, P, and Mn. The observed variability in the distribution of data samples underscores significant differences in mineral profiling, which can be attributed to inherent genetic distinctions among the samples. Moreover, strong correlations between various mineral elements are noticeable. Figure 1-c presents the distribution of press cakes based on their antioxidant activity to construct a data matrix with a PCA score plot for the first two PCs of the samples. As can be seen in Figure 1-c, press cakes are separated according to PC1 (87.72%) and PC2 (7.69%), the first two principal components were retained as they explain more than 95% of the total variance in our data. The application of the PCA algorithm reveals that all analysed parameters exhibit positive correlations with PC1. Notably, "SFPC" appears as the only positively oriented variable along this axis, revealing its higher representation of target parameters (located in the right area of the plot) relative to other variables located in the left area of the plot. This strategic alignment emphasizes its distinctive composition within the dataset. A conspicuous clustering of samples, specifically 'ALPC,' 'SBPC,' and 'SEPC,' is evident in the center-left area of PC1. This dense grouping clearly indicates their moderate similarity and proximity in terms of antioxidant activity profiles. Another PCA model was constructed using the analysed press cakes. In this process, the parameter peak areas of the targeted proximate composition in the samples were used to construct a data matrix with a PCA score plot for the first two PCs of the samples. As depicted in Figure 1-d, the press cakes exhibit discernible separation along PC1 (44.81%) and PC2 (24.94%). In the context of this

initial exploratory model, noteworthy differentiation was observed among three distinct groups. The first group, characterised by the 'SFPC' sample, displayed variance within the group attributed to specific compounds, namely Carbohydrates, ADL, ADF, and NDF, positioned on the positive side of PC1. The second group, comprising 'ALPC' and 'SEPC,' is distinguished by elevated values of the variables CF and Residual oil, positioned on the negative side of PC1. The third group, consisting of 'SBPC' and 'ARPC' on the positive side of PC2, exhibited the highest recorded values for parameters, ASH and Proteins. These outcomes, unveiled through PCA, align with the findings derived from mean values comparisons as reported in Tables 2.

### Correlation Matrix

The correlation matrix results depicting the relationship among the analysed press cake components (ash, oil, protein, carbohydrate content, moisture, micro and macro-elements, TPC, TFC, and Antioxydant Activity) are shown in Figure 2. Significant correlations, both positive and negative, were observed between the different variables studied. In particular, the DPPH test revealed a significant positive correlation with FRAP ( $r=0.91$ ), Cu ( $r=0.89$ ) and moisture ( $r=0.9$ ). In contrast, DPPH showed a negative correlation with Ca ( $r=-0.76$ ) and Cu ( $r=-0.79$ ). Ca also showed a negative correlation with humidity ( $r=-0.9$ ). On the other hand, FRAP showed robust correlations with TFC ( $r=0.94$ ), TPC ( $r=0.88$ ) and Cu ( $r=0.89$ ). TPC was positively correlated with NDF ( $r=0.96$ ) and ADF ( $r=0.92$ ), while ADF was positively correlated with NDF ( $r=0.98$ ). In addition, phosphorus (P) was positively correlated with magnesium (Mg) ( $r=0.93$ ), while boron (B) was negatively correlated with manganese (Mn) ( $r=-0.81$ ), and protein was negatively correlated with TFC ( $r=-0.78$ ). The coefficient of correlation between fatty acids and sterols in the various cakes analysed are shown in Figure 3. The results show significant correlations between these variables. For example, C16:0 is positively associated with C20:1 ( $r=0.78$ ), C18:0 is positively correlated with C20:0 ( $r=0.75$ ), C18:1 is positively related to C16:1 ( $r=0.7$ ), and C16:1 is positively correlated with  $\beta$ -sitosterol ( $r=0.66$ ) and  $\Delta$ -5-avenasterol ( $r=0.7$ ). In addition, a positive correlation was observed between C18:2 and campesterol ( $r=0.63$ ). On the other hand, negative correlations were observed,

such as that between C18:1 and C18:2 ( $r=-0.97$ ), as well as between  $\beta$ -sitosterol and spensterol ( $r=-0.95$ ) and schotenol ( $-0.95$ ). In addition,  $\Delta$ -7-stigmasterol showed a negative correlation with C20:1 ( $r=-0.97$ ).

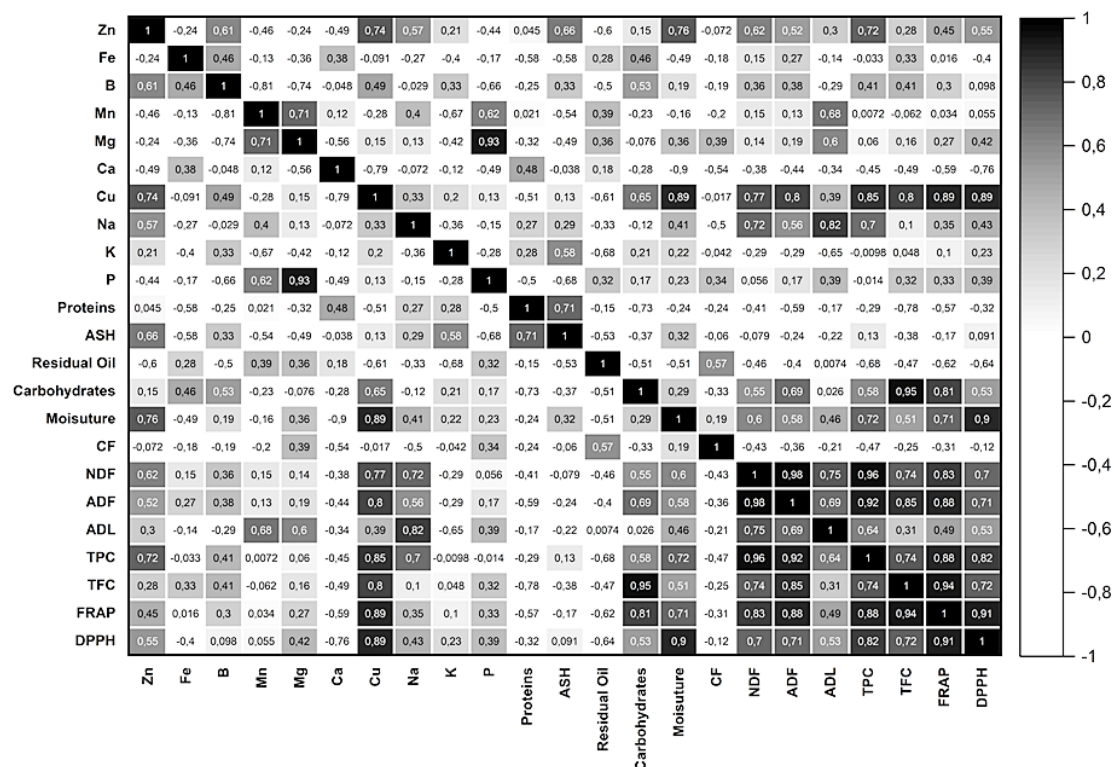


Fig.2 : Coefficients of correlation between proximate composition, minerals and antioxidant activity

**Discussion**

**Biogenic Elements**

The biogenic elements also predominate in many other oil cakes in several additional case studies, including rapeseed cake, flaxseed cake hempseed cake, camelina cake, and pumpkin cake.<sup>33-36</sup> Among the studied press cakes, SBPC demonstrated the highest potassium content. ARPC, NIPC, and SEPC were identified as the richest in calcium. However, SFPC and SEPC exhibited the highest magnesium levels. These findings align with those reported by<sup>6,18</sup> for ARPC and NIPC, surpassing the results reported by<sup>37</sup> for SEPC and by<sup>38</sup> for SFPC.

**Ash Content**

The results obtained for ARPC and NIPC align with those from a study conducted by<sup>18</sup> and similarly, the results for SFPC closely match those reported by<sup>38</sup> However, the values found in SEPC and ALPC were lower than those reported by<sup>41</sup> and<sup>9</sup>, respectively.

**Proteins Content**

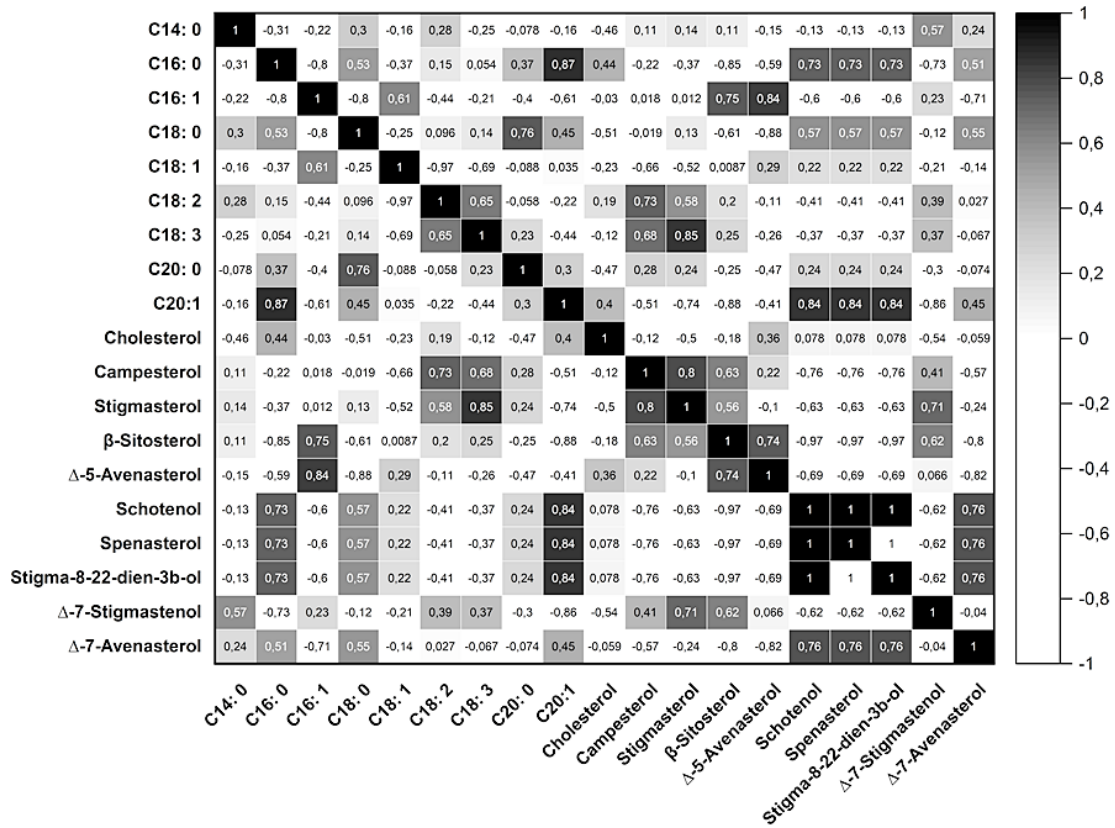
Compared to published results, the proteins content in ARPC surpasses that reported by<sup>45</sup> but aligns with findings from.<sup>46</sup> Conversely, the outcomes for ALPC are lower than those reported in the study of.<sup>9</sup> However, the values obtained for NIPC and SBPC are consistent with those reported by<sup>22</sup> and<sup>47,48</sup> respectively.

**Residual Oil Content**

The ALPC, SEPC, and NIPC results align with values reported by,<sup>51-53</sup> respectively, positioning these cakes as potentially lucrative sources of oil with notably higher levels than others. This opens up promising avenues for valorizing these agri-food by-products, positioning them as potential raw materials for high quality residual oil production. Given their substantial oil content, these cakes can be considered valuable resources in the circular economy framework, contributing to maximizing the added value

throughout the production process. On the other hand, SBPC and SFPC exhibit minimal residual oil ( $1.25 \pm 0.35$  and  $1.05 \text{ g}/100 \text{ g}$ , respectively), and are not significantly different from each other. These

outcomes align with those reported by other authors for Soybean oil press cake and sunflower oil press cake, respectively.<sup>54,55</sup>



**Fig.3 : Coefficients of correlation between fatty acids and sterols**

**Carbohydrates**

All three types of cake are rich in carbohydrates, giving them a high energy value. However, our findings showed higher quantities compared to previous studies<sup>38,58,59</sup> SEPC and ALPC have intermediate carbohydrate contents, with average values of ( $39.40 \pm 0.85 \text{ g}/100 \text{ g}$ ) and ( $34.15 \pm 0.83 \text{ g}/100 \text{ g}$ ) respectively. ARPC has a slightly lower carbohydrate content than ALPC, with an average value of ( $29.80 \pm 0.38 \text{ g}/100 \text{ g}$ ). Nevertheless, this value is lower than that reported by.<sup>18</sup>

**Energy Value**

The variations in their protein, fat, and carbohydrate content typically account for these variations in nutritional value.<sup>21</sup> The energy value of oilcakes

can have an impact on their use as a source of energy for animals. For this reason, energy-rich oilcakes may be suitable for animals with high energy requirements, such as dairy cattle or pigs. Low-energy feeds may be suitable for animals with lower energy requirements, such as poultry or fish.

**Moisture**

The values found in ARPC, ALPC, NIPC and SBPC are lower than those reported by <sup>45,61,58,59,61</sup> respectively. However, the value found in SFPC is higher than that obtained by.<sup>38</sup> In summary, moisture management is crucial for ensuring the quality and durability of oilcakes, and the results highlight significant differences between the types of oilcakes studied.

### Fatty Acids Composition

Fatty acids play an essential role as primary constituents of biological substances in humans and organisms.<sup>62,63</sup> They can be classified into different groups, such as saturated fatty acids (SFAs), monounsaturated fatty acids (MUFAs) and polyunsaturated fatty acids (PUFAs), depending on the saturation state of their hydrocarbon chains.<sup>64</sup> SFAs are important as components of cell membranes and as sources of energy.<sup>65</sup> However, studies have found that a reasonable quantity of SFAs is beneficial to fat metabolism, while an excess of SFAs can cause fat deposits in the organism.<sup>66,67</sup> Oleic acid is recognized for its many health benefits. In fact, its ability to improve the blood lipid profile has been demonstrated.<sup>70</sup> Moreover, studies indicate that adequate consumption of oleic acid is associated with significant cardiovascular benefits, in particular reducing the risk of cardiovascular disease.<sup>71</sup> Additionally, oleic acid demonstrates anti-inflammatory properties, which are pivotal in mitigating inflammation within the body.<sup>72</sup>

### Phytosterols Composition

The study of<sup>79</sup> found also  $\beta$ -sitosterol as a major sterols identified in NIPC oil. Our results are in agreement with those found in black cumin oil extracted by cold press ( $49.4 \pm 1.5\%$ ) by<sup>22</sup> In the case of SFPC oil, our results are similar to those obtained by soxhlet and by press ( $61.7 \pm 0.98$  and  $61.2 \pm 0.4$ , respectively) in the study of.<sup>80</sup> Moreover, another work on sunflower found the same results ( $59.7 \pm 1.8\%$ ).<sup>81</sup> Velasco<sup>82</sup> mentioned the phytosterol content of the world's main vegetable oils, we found a very high degree of similarity between SEPC oil and SBPC oil (61.8% and 54.3%, respectively). Almost all sterol levels in SEPC, SFPC and SBPC oils have met the requirements set out by Codex Alimentarius.

### Fibers Content

Our findings are in line with those reported by<sup>7,51</sup> in ALPC and SFPC respectively. However, they are lower than the values reported by<sup>45,84</sup> in ARPC and SBPC respectively. Whereas for NIPC and SEPC our results are higher than those published by<sup>53,39</sup> respectively. Various publications have highlighted the beneficial impact of fiber in the diets of non-ruminant animals. Fiber has been associated with functions such as digestion, antimicrobial action, immune support and regulation of harmful lipids.<sup>85,86</sup> However it is important to be careful when

incorporating high-fiber dietary components, as a high concentration of fiber in the diet of monogastric animals can influence the animal's performance, as well as digestion processes and hepatic lipid metabolism.<sup>7</sup> The results of Neutral detergent fiber (NDF), which is defined as the residue left over after extraction with the neutral detergent solution, measure the amount of cellulose, hemicellulose, lignin, cutine, and insoluble minerals in the cell wall,<sup>83</sup> and ADF, which is the insoluble fraction of dietary fiber, measured as acid detergent fiber,<sup>87</sup> reveal that NDF and ADF content are in the range of 16.40–54.40 % and 16.60–47.50 %, respectively, whereas SFPC has higher average NDF as well as ADF content compared to other cakes, followed by NIPC and ARPC, while low NDF and ADF values were recorded in ALPC and SBPC respectively. However, like NDF, the highest ADL results were recorded in SFPC with an average of 19 % and the lowest values were recorded in SBPC with an average of 2.20%. Our outcome for NDF, ADF and ADL found in SFPC and SBPC are higher than those reported by da Silva Oliverira.<sup>88</sup> It is also relevant to note that our results for the NDF and ADF fractions are also higher than those obtained by<sup>89</sup> in the analysis of ARPC. These findings demonstrate a significant divergence from earlier data, highlighting the significance of our study in understanding oilcake composition.

### TPC, TFC and Antioxidant Activities of Press Cakes Extract's

Plants contain many bioactive compounds with powerful antioxidant activity.<sup>97,98</sup> The antioxidant efficacy of an individual compound or a mixture of compounds is linked to its ability to neutralise free radicals, break down singlet oxygen, act as a metal chelator, or act in synergy with other compounds present in the plant.<sup>19</sup> However, various tests are available to assess the antioxidant activity of plant materials, each with its own mechanism for measuring these characteristics.<sup>99</sup> It is recommended to use at least two different tests to correctly assess the antioxidant activity of the product.<sup>99</sup> In our study, FRAP and DPPH assays were used to evaluate the antioxidant properties of the press cake extracts. The DPPH test is a free radical scavenging activity testing method. Regarding the antioxidant activity of the press cakes studied, using DPPH radical, it can be seen that SFPC presented high percentages of reduction of DPPH radical with values of 78.07



$\pm 1.92$  %. The second group includes the SBPC, ARPC, ALPC, and SEPC, for which the percentages of reduction did not vary significantly and were as follows:  $19.99 \pm 0.76$  %,  $19.03 \pm 0.19$  %,  $18.84 \pm 0.38$  %, and  $15.76 \pm 0.3$  %. However, the lowest value of DPPH was observed in the NIPC extract. With respect to the antioxidant activity quantified by FRAP in each of the study press cakes, the lowest value was obtained for ALPC ( $0.56 \pm 0.03$  AAE mg/g DM), followed by ARPC ( $1.17 \pm 0.01$  AAE mg/g DM). The press cakes with the highest values were NIPC and SFPC with ( $2.68 \pm 0.12$ ,  $1.6 \pm 0.2$ ; AAE mg/g DM) respectively. It is not possible to make a direct comparison between the results obtained and those in the literature, given that most studies reported DPPH and FRAP values using Trolox as the standard reference.<sup>8,96,100</sup>

### Conclusion

This study examines the proximal composition, mineral profile, lipid profile, fiber and antioxidant activity of argan press cake in comparison with almond, sesame, nigella, sunflower and soybean press cakes. The results reveal a significant positive correlation between phenolic compound content and the antioxidant activity of argan oilcake, highlighting the importance of bioactive compounds in improving antioxidant properties. In addition, correlations were also observed between protein content, fiber content and fatty acid composition, suggesting that these components jointly contribute to the overall nutritional value of the argan oilcake. More specifically, the argan cake analysed has high levels of protein, fiber and phenolic compounds, positioning it as a promising reservoir of these essential elements for human and animal nutrition. The argan press cake also has a balanced mineral profile, with notable concentrations of potassium, phosphorus, magnesium, calcium and sodium. The lipid profile of argan press cake reveals high levels of unsaturated fatty acids. These results indicate that argan oilcake could be exploited well beyond its traditional use, offering substantial benefits for human and animal nutrition. In terms of commercialization, the valorization of argan cake in various food and pharmaceutical products could present substantial commercial opportunities, due to its richness in nutrients and bioactive compounds. For future studies, it would be relevant to explore the mechanisms of interaction between bioactive

compounds and other nutritional components of argan oilcake, as well as the effect of various extraction processes on its nutritional and functional quality. Studies on the cost-benefit analysis of marketing argan oilcake and the development of cost-effective processes for extracting bioactive compounds could also open up new prospects for the agri-food industry.

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The authors do not have any conflict of interest.

### Data Availability Statement

This statement does not apply to this article.

### Ethics Statement

This research did not involve human participants, animal subjects, or any material that requires ethical approval.

### Informed Consent Statement

This study did not involve human participants, and therefore, informed consent was not required.

### Clinical Trial Registration

This research does not involve any clinical trials.

### Permission to Reproduce Material from other Sources

Not Applicable.

### Author Contributions

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- review & editing
- **Angelo Maria Giuffrè:** Formal analysis – Review & editing – Visualization – Resources
- **Said Gharby:** Conceptualization - Supervision – Methodology - Review & editing

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