Proximate, mineral, amino acid composition, phenolic profile, antioxidant and functional properties of oilseed cakes

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Summary Proximate, minerals, amino acids (AAs), phenolic composition, antioxidant and functional properties of flaxseed (FSC), sesame (SSC), mustard (MSC), nigella (NSC) and groundnut (GSC) seedcakes were studied. SSC exhibited higher ash and fat contents while GSC and FSC had more protein and mineral contents (P, Mg, Mn and Cu), respectively. The total flavonoids, phenolics and antioxidant activity were higher in NSC (18.60 mg QE g^{-1}), MSC (5.12 mg GAE g^{-1}) and FSC (12.28 µmol TE g^{-1}), respectively. NSC exhibited the highest oil absorption capacity (2.10 g g^{-1}) and emulsifying activity index (89.52 m² g⁻¹). Twenty-one AAs including citrulline and y-aminobutyric acid were detected in oilseed cakes (OSC). MSC had more essential AAs (methionine, isoleucine, tryptophan, threonine and lysine) while GSC had more total hydrophobic, acidic and basic AAs. Syringic acid and rutin were identified as major phenolic compounds in GSC and MSC, respectively. The total free phenolic acids were more in GSC while FSC had high total bound phenolic acids and flavonoids. OSC can be utilised in food products as a supplement to improve nutritional properties.

Keywords Amino acids, antioxidant activity, functional properties, minerals, oilseed cake, phenolics.

Introduction

Oilseeds are the major crops grown primarily for the supply of fats and proteins in the human diet. Oilseed cakes (OSC) are solid residues obtained after the extraction of oil from oilseeds. The production of oil from oilseeds generates tonnes of residues as agroindustrial by-products (Mannucci et al., 2019). India is native to a wide range of oilseed crops and is known for the production of OSC as major by-products (Sunil et al., 2015). The residual OSC is generated in large amounts (317 million tons in 2016) globally and is further forecasted to rise to 386 million tonnes by 2025 (Duodu et al., 2018). OSC is a precious source of protein, carbohydrates, minerals, dietary fibre, phenolic compounds and lipids (Sahin & Elhussein, 2018). They are generally consumed as valuable feedstuffs by ruminants, fish and poultry or used as an organic fertiliser

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in their production areas (Ramachandran et al., 2007; Sahin & Elhussein, 2018).

OSC is an excellent raw material for the development of food products rich in antioxidants, vitamins, proteins, minerals and dietary fibre (Sunil et al., 2015). They have potential applications as nutraceuticals and functional food ingredients (Sahin & Elhussein, 2018). The value-added products such as organic acids, vitamins, industrial enzymes, bio-pesticides and bioactive compounds can be developed using OSC (Ramachandran et al., 2007). The phytochemicals derived from OSC have potential applications in food, pharmaceutical and cosmetic industries as they exhibit antioxidant, antimicrobial and health-promoting properties (Sahin & Elhussein, 2018). Valorisation of OSC in high added value products has been an emerging trend owing to their bioactive constituents with many medical properties (Sahin & Elhussein, 2018). Efficient ultrasonic extraction technique has been developed to enhance polyphenol extraction form OSC (Teh & Birch, 2014). Being rich in nutrients and antioxidants, if processed suitably, OSC can be utilised for human food supplementation (Sunil *et al.*, 2015).

OSC is the cheap and attractive raw material available throughout the year for extraction of proteins (Ramachandran et al., 2007). Effective utilisation of OSC as sources of nutrients and antioxidants can reduce the environmental and sanitation problems of oilseed industries. Phytonutrients present in OSC can be utilised for value-addition in processed foods and low-cost healthy foods to meet the demand of increasing human population (Mekky et al., 2019). The previous studies reported proximate composition and mineral content of flaxseed cake (Ogunronbi et al., 2011), AAs profile of nigella seed cake (Al-Gaby (1998), antioxidant potential of rapeseed, white mustard, camelina and linseed pressing residues (Terpinc et al., 2012), proximate and AAs composition of cottonseed meal and groundnut meal (Duodu et al., 2018). As far as we know, there is no comparative and detailed study available on nutritional, functional and antioxidant properties of OSC. Therefore, the present study evaluated proximate, minerals, AAs, phenolic profile, functional and antioxidant properties of five OSC.

Materials and methods

Materials

Five oilseeds (flaxseed, sesame, mustard, nigella and groundnut) purchased from the local Amritsar market (Punjab, India) were cleaned to remove the particles of dust, stones and other impurities. The moisture contents of flaxseed, sesame, mustard, nigella and groundnut seeds were 5.67%, 3.32%, 6.21%, 5.84% and 5.53%, respectively. The seeds were passed through a mechanical screw press oil expeller (Rajkumar Agro-Engineers Pvt Ltd, India) at 60° C to obtain oil and seedcake. The oilseed cakes (OSC) obtained were finely ground, passed through a mesh sieve (35 µm) and stored in airtight polyethylene bags at -18 °C till analysed.

Proximate composition

The moisture (MC), ash (AC), fat (FC) and protein (PC) contents of OSC were determined using AACC method 44-19, 08-01, 30-20 and 46-12, respectively (AACC, 2000).

Mineral composition

The mineral composition of OSC was determined using an atomic absorption spectrophotometer (iCE^{TM}) 3400, Agilent Tech) as described by Bhinder et al. (2021). Briefly, OSC (1 g) charred in a porcelain crucible at 550 °C was mixed in 2.5 mL of 1N nitric acid solution, filtered and diluted to 100 mL with Milli-Q water. The solution obtained was analysed for mineral contents and concentration expressed as $mg 100 g^{-1}$.

Antioxidant properties

Extract preparation

OSC was defatted with hexane, dried overnight at 40 \degree C, ground and passed through a sieve (250 µm) for subsequent analysis. The free and bound phenolics were extracted from OSC using the method given by Bhinder et al. (2021) with minor modification. OSC (500 mg) was ultrasonicated for 1 h in 80% methanol (8 mL), centrifuged and the supernatant was collected. The residue was again suspended in methanol, ultrasonicated and centrifuged. The supernatants were pooled and labelled as free phenolic extract after adjusting pH to 4.0–4.5 with 6N HCl. The leftover residue was mixed with 0.4 M NaOH (25 mL) for 2 h under continuous agitation. The pH of the mixture was adjusted to 4.0- 4.5, centrifuged, supernatant collected and labelled as a bound phenolic extract. The free and bound phenolic extracts were used for estimation of total phenolic and flavonoid contents, radical scavenging capacity, level of phenolic acids and flavonoids.

Total phenolic and flavonoid content

The content of free, bound and total phenolics and flavonoids was quantified using the method given by Bhinder *et al.* (2019). In brief, OSC extract (100 μ L), deionised water (4.8 mL) and Folin-Ciocalteu reagent $(300 \mu L)$ were mixed and incubated for 8 min in an amber glass tube. Then, 20% Na₂CO₃ (900 μ L) added, vortex-mixed and kept at 40 °C for 30 min. The absorbance of the mixture was recorded at 765 nm and phenolic content reported in mg gallic acid equivalents (GAE) g⁻¹ dry weight (DW) of OSC extract. For estimation of flavonoid content, OSC extract $(250 \mu L)$, deionised water (1.25 mL) and sodium nitrite $(5\%, 75 \mu L)$ were mixed and allowed to react in an amber glass tube for 5 min. Then, 150 μ L of AlCl₃H₂O (10%) was added and incubated for 5 min before adding NaOH (1 M, 0.5 mL) and ethanol (275 μ L). The absorbance of the solution was recorded at 510 nm and flavonoid content reported in mg quercetin equivalent (QE) g^{-1} DW.

Radical scavenging capacity (RSC)

The RSC of free and bound phenolic extract of OSC was estimated using DPPH assay (Bhinder et al., 2019). Briefly, $100 \mu L$ of OSC extract was reacted with DPPH solution (3.9 mL, 6×10^{-9} M) at 25 °C for 30 min. Then the decrease in absorbance of DPPH solution was measured at 515 nm and the free, bound and total RSC were expressed in terms of µmol Trolox equivalents (TE) g^{-1} DW.

Phenolic composition

The free and bound extracts of OSC (described in antioxidant properties section) were used for phenolic

profiling with the HPLC system (Agilent 1260 Infinity) equipped with a binary pump, a C18 column, a DAD-PDA detector and an auto-sampler as described by Kaur et al. (2021). The phenolics were estimated using mobile phase A (0.1% trifluoroacetic acid in water) and B (acetonitrile, water and trifluoroacetic acid in the ratio of 50: 49.8: 0.2) with column maintained at 35 °C and the flow rate adjusted to 1 mL min⁻¹. The gradient conditions of mobile phase established were 95% A and 5% B (0–5 min); 95–75% A and 5%–25% B (5–40 min); 75%–62% A and 25%–38% B (40– 47 min); 62%–55% A and 38%–45% B (47–49 min); 55% A and 45% B (49–51 min); 55%–20% A and 45%–80% B (51–70 min); 20%–5% A and 80%–95% B (70–75 min); 5%–95% A and 95%–5% B (75– 77 min); and 95%–5% A and 5%–95% B (77– 90 min). The standards used for counting of phenolic compounds include gallic (GAC), protocatechuic (PCA), syringic (SYA), vanillic (VLA), ferulic (FRA), caffeic (CFA), chlorogenic (CGA), sinapic (SPA), p-coumaric (p-CA), cinnamic (CMA) acids, catechin, epicatechin, rutin, quercetin and resveratrol. The peaks of phenolics were detected at 367, 350, 320 and 280 nm. Quantification of phenolic compounds carried out through area normalisation of peaks and results reported as mg 100 g^{-1} DW.

Amino acid composition

The AAs content of OSC was determined by the Shimadzu LC-30 AD high-performance liquid chromatography (HPLC) system as described by Kaur *et al.* (2021). Briefly, 100 mg of OSC flour was acid hydrolysed using 6 N HCl under vacuum conditions in a closed digestion bottle for 24 h and extracted with 1 mL of 0.1 N HCl. AAs were dervatised using o-phthaladehyde, 9-fluorenylmethyl chloroformate and mercaptopropionic acid. The mobile phase consisted of methanol:acetonitrile:water (40:45:15, $v/v/v$) and phosphate buffer (20 mM L^{-1}). The flow rate and column temperature were adjusted to 1 mL min⁻¹ and 40 °C, respectively. The peaks detected at 254 nm were analysed using the 5.54SP 5 LAB Solutions software. The AAs were quantified by comparing the area ratio of the sample with the working standard AA (Thermo Scientific, NCI0180) and content reported as mg 100 g^{-1} .

Functional properties

Water (WAC) and oil absorption (OAC) capacities, emulsifying activity (EAI) and emulsion stability (ESI) indexes were determined as described by Bhinder et al. (2020). For OAC, 100 mg of OSC was vortex-mixed with sunflower oil (1 mL), maintained at room temperature for 30 min and centrifuged at 13 600 \times g for 20 min. For WAC, 100 mg of OSC was vortex-mixed with deionised water (1 mL) and centrifuged at $1800 \times g$ for 20 min. The separated oil and water were decanted and tubes were drained at 45° angle for 10 min and reweighed for the calculation of WAC and OAC as grams of water or oil absorbed per gram of OSC. For EAI and ESI, OSC (100 mg) was mixed with deionised water (10 mL) and sunflower oil (10 mL) and the mixture was homogenised at 20 000 rpm for 1 min. Emulsion (100 μ L) taken from the bottom of the tube at 0 and 10 min was mixed with 10 mL of sodium dodecyl sulphate (0.1%) solution. The absorbance of the solution was measured at 500 nm for the calculation of EAI $(m^2 g^{-1})$ and ESI (min).

Statistical analysis

Experiments were conducted in triplicates and results reported as average values \pm standard deviation. The difference between the average values was determined by performing one-way ANOVA and multivariate analysis was applied to establish the relation among different parameters.

Results and discussion

Proximate composition

The proximate composition of five OSC is given in Table 1. The MC, AC, FC and PC of OSC ranged from 5.02–7.64 g 100 g^{-1} , 3.89–5.89 g 100 g^{-1} , 15.45– 23.64 g 100 g^{-1} and 22.55–36.78 g 100 g^{-1} , respectively. The highest MC was observed for FSC $(7.64 \text{ g }^{100} \text{ g}^{-1})$ while SSC had more AC $(5.89 \text{ g } 100 \text{ g}^{-1})$ and FC $(23.64 \text{ g } 100 \text{ g}^{-1})$. Ogunronbi et al. (2011) also recorded higher MC $(8.8-10.4 \text{ g } 100 \text{ g}^{-1})$ and comparable AC $(3.9-5.4 \text{ g } 100 \text{ g}^{-1})$ and FC $(12.4-22.5 \text{ g } 100 \text{ g}^{-1})$ in four batches of FSC. The highest PC was observed for GSC $(45.47 \text{ g } 100 \text{ g}^{-1})$, followed by MSC $(36.78 \text{ g } 100 \text{ g}^{-1})$, SSC (35.41 g 100 g^{-1}), NSC (22.55 g 100 g^{-1}) and FSC $(17.85 \text{ g } 100 \text{ g}^{-1})$. These results are comparable to PC reported in NSC (Al-Gaby, 1998), SSC (Sunil et al., 2015) and groundnut meal (Duodu et al., 2018). However, Ogunronbi et al. (2011) and Mannucci et al. (2019) reported higher PC in FSC (29.8–35.4 g 100 g^{-1} and 30.4 g 100 g^{-1} , respectively). GSC exhibited lower FC $(15.45 \text{ g } 100 \text{ g}^{-1})$ while Duodu *et al.* (2018) reported higher FC (27.6 g 100 g⁻¹) in unprocessed groundnut meal. The FC was lower and PC was higher in SSC compared to Mohdaly et al. (2010).

Mineral composition

The mineral composition of five OSC is given in Table 1. The most abundant minerals were potassium (272.48– 349.48 mg 100 g^{-1}), phosphorous (154.20–602.52 mg) 100 g^{-1}), magnesium (112.76–142.33 mg 100 g⁻¹) and

calcium (98.0–297.71 mg 100 g^{-1}) in OSC. Olaofe *et al.* (1994) also reported more potassium, magnesium and phosphorous in flour samples of different oilseeds. The OSC had low copper among the studied minerals. FSC had high contents of potassium, magnesium, manganese and copper (349.48, 142.33, 3.44 and 0.87 mg 100 g^{-1} , respectively) while SSC contains more phosphorous $(602.52 \text{ mg } 100 \text{ g}^{-1})$ and iron $(89.35 \text{ mg } 100 \text{ g}^{-1})$ than other OSC. NSC presented higher contents of calcium $(297.71 \text{ mg } 100 \text{ g}^{-1})$ and zinc $(11.12 \text{ mg } 100 \text{ g}^{-1})$ while GSC had more sodium content $(82.09 \text{ mg } 100 \text{ g}^{-1})$. The levels of phosphorous, magnesium, calcium, iron, zinc, manganese and copper were low in GSC while MSC had low contents of potassium and sodium. The level of calcium, magnesium, phosphorous and potassium in FSC concurs with the previous study (Ogunronbi et al., 2011).

Antioxidant properties

Total phenolic and flavonoid content

The free (FPC), bound (BPC) and total phenolic (TPC) content of five OSC ranged from 0.51–3.89, 0.87–1.23 and 1.38–5.12 mg $GAE[g^{-1}]$, respectively (Table 2). MSC and SSC exhibited the highest and lowest FPC, BPC and TPC, respectively. Terpinc et al. (2012) recorded significantly higher TPC in methanol and ethanol extracts of white MSC compared to rapeseed cake and FSC. The low TPC observed in SSC was comparable with Mohdaly et al. (2010) and Mekky et al. (2019). TPC in FSC (3.50 mg GAE g^{-1}) concurs with the range $(3.09-4.84 \text{ mg }$ GAE g^{-1}) reported by Mannucci et al. (2019). NSC showed TPC of 2.72 mg GAE g^{-1} while Mariod et al. (2009) reported significantly higher TPC $(27.8 \text{ mg } \text{GAE g}^{-1})$ in crude methanolic extract of NSC. The free (FFC), bound (BFC) and total flavonoid

Table 1 Proximate and mineral composition of five oilseed cakes

(TFC) contents in OSC ranged from 1.90–8.98, 1.55– 12.12 and 8.29–18.60 mg \overline{QE} g⁻¹, respectively. GSC exhibited the highest FFC and lowest BFC (8.98 and 1.55 mg QE g^{-1}) while NSC had more BFC and TFC $(12.12 \text{ and } 18.60 \text{ mg} \text{ QE } g^{-1})$ than other OSC. TFC noted in SSC (12.05 mg $\overline{QE}g^{-1}$) was comparable to those reported by Kermani et al. (2019) in sesame seeds. Teh & Birch (2014) recorded higher TFC in FSC than our results (10.54 mg QE g^{-1}).

Radical scavenging capacity

RSA of free (FRSC) and bound (BRSC) phenolic extracts of OSC ranged from 1.74–7.39 and 3.42– 6.20 µmol TE g^{-1} , respectively (Table 2). MSC showed the highest FRSC $(7.39 \text{ }\mu\text{mol})$ TE g^{-1} while NSC exhibited more BRSC (6.20 μ mol TE g⁻¹). The lowest FRSC and total RSA (TRSC) was observed for phenolic extract of SSC (1.74 and 5.80 µmol TE g^{-1} , respectively) while GSC showed low BRSC (3.42 µmol TE g^{-1}). The difference in RSC relates with free, bound and total phenolic and flavonoid contents of OSC. MSC showed the highest TPC (5.12 mg GAE g^{-1}) and also exhibited higher FRSC. However, our results contradict with Terpinc et al. (2012) who reported the weakest scavenging potential for white MSC compared to camelina, FSC and rapeseed cakes. NSC exhibited a total antioxidant activity of 10.73 μ mol TE g^{-1} . The previous report by Mariod et al. (2009) noted DPPH IC_{50} values in the range of 1.89–2.65 mg mL⁻¹ for different extracts obtained from NSC.

Phenolic composition

The phenolic profile revealed the presence of ten phenolic acids, four flavonoids and one non-flavonoid

Values with similar superscripts in a row do not differ significantly $(P < 0.05)$.

AC, Ash content; Ca, Calcium; Cu, Copper; FC, Fat content; Fe, Iron; FSC, Flaxseed cake; GSC, Groundnut seed cake; K, Potassium; MC, Moisture content; Mg, Magnesium; Mn, Manganese; MSC, Mustard seed cake; Na, Sodium; NSC, Nigella seed cake; P, Phosphorous; PC, Protein content; SSC, Sesame seed cake; Zn, Zinc.

DW, Dry weight; FFC, BFC and TFC, Free, Bound and Total Flavonoid Contents, respectively; FPC, BPC and TPC, Free, Bound and Total Phenolic Contents, respectively; FRSC, BRSC and TRSC, Free, Bound and Total Radical Scavenging Capacities, respectively; FSC, Flaxseed cake; GAE, Gallic acid equivalents; GSC, Groundnut seed cake; MSC, Mustard seed cake; NSC, Nigella seed cake; QE, Quercetin equivalents; SSC, Sesame seed cake; TE, Trolox equivalents.

polyphenol in OSC (Table 3). The phenolic acids include four hydroxybenzoic (PCA, GAC, SYA and VLA) and six hydroxycinnamic (CMA, p-CA, SPA, CGA, CFA and FRA) acids. The free SYA was the major hydroxybenzoic acid in GSC (118.08 mg 100 g^{-1}) followed by MSC (56.33 mg 100 g^{-1}) and NSC (47.11 mg 100 g^{-1}). Mariod *et al.* (2009) also quantified SYA as the predominant phenolic acid in the water fraction of NSC. The free and bound VLA content was more in SSC $(42.53 \text{ mg } 100 \text{ g}^{-1})$ and FSC $(46.20 \text{ mg } 100 \text{ g}^{-1})$, respectively. SSC exhibited the highest content of free PCA $(16.55 \text{ mg } 100 \text{ g}^{-1})$ followed by MSC (9.05 mg 100 g⁻¹), NSC $(8.95 \text{ mg } 100 \text{ g}^{-1})$ and FSC $(1.67 \text{ mg } 100 \text{ g}^{-1})$ among the tested OSC. The free GAC was detected only in MSC (3.08 mg 100 g^{-1}) and FSC (1.85 mg 100 g^{-1}). Martinović et al. (2020) detected GAC along with PCA, SYA and VLA in white mustard seeds. Among hydroxycinnamic acids, the free p-CA and FRA were abundant in GSC $(80.13 \text{ and } 8.85 \text{ mg } 100 \text{ g}^{-1})$. However, de Camargo et al. (2017) quantified the low content of FRA and p-CA $(1.138$ and 1.191 mg 100 g⁻¹) in dry blanched groundnut meal. FSC had more bound CGA $(78.28 \text{ mg } 100 \text{ g}^{-1})$ compared to other OSC $(0.46 3.57$ mg 100 g⁻¹). Mariod *et al.* (2009) reported high p-CA $(3.83 \text{ mg } 100 \text{ g}^{-1})$ in the water fraction of NSC compared to our results $(2.38 \text{ mg } 100 \text{ g}^{-1})$. The free CMA, CGA and CFA contents were more in GSC $(22.43 \text{ mg } 100 \text{ g}^{-1})$, NSC $(15.32 \text{ mg } 100 \text{ g}^{-1})$ and SSC $(9.30 \text{ mg } 100 \text{ g}^{-1})$, respectively. Martinovic et al. (2020) reported free and bound SPA in white mustard seeds while in our study free SPA was detected only in NSC at a level of $1.45 \text{ mg } 100 \text{ g}^{-1}$. The total free phenolic acids were more in GSC (237.25 mg 100 g^{-1}) while total bound phenolic acids were high in FSC $(133.32 \text{ mg } 100 \text{ g}^{-1}).$

The flavonoids detected in OSC include flavan-3-ols (epicatechin and catechin) and flavonols (rutin and

quercetin). The flavan-3-ols were detected only in FSC, NSC and GSC. The bound catechin was predominant in FSC (386.04 mg 100 g^{-1}) while free catechin and epicatechin were more in GSC (38.24 mg 100 g^{-1}) and NSC $(23.87 \text{ mg } 100 \text{ g}^{-1})$, respectively. de Camargo et al. (2017) recorded high content of catechin and epicatechin in groundnut skin. Among flavonols, the free rutin content was more in MSC (1221.79 mg 100 g^{-1}), followed by GSC (72.48 mg 100 g^{-1}), NSC (72.04 mg $100~\text{g}^{-1}$) and SSC (6.74 mg $100~\text{g}^{-1}$). The rutin content in SSC was comparable to those reported by Kermani et al. (2019) in sesame seeds. The free quercetin was detected only in GSC, MSC and FSC (6.16, 2.18 and 0.91 mg 100 g^{-1} , respectively). The bound quercetin was not detected and bound rutin was quantified only in FSC $(5.14 \text{ mg } 100 \text{ g}^{-1})$. Martinovic *et al.* (2020) reported free epicatechin, rutin and quercetin in white mustard seeds. The total free flavonoids were high in MSC (1223.97 mg 100 g^{-1}) while total bound flavonoids were more in FSC $(391.18 \text{ mg } 100 \text{ g}^{-1})$. The high content of free resveratrol was quantified in MSC (17.88 mg 100 g^{-1}), followed by GSC (7.46 mg 100 g^{-1}), NSC $(0.99 \text{ mg } 100 \text{ g}^{-1})$ and FSC $(0.37 \text{ mg } 100 \text{ g}^{-1})$ while bound resveratrol was detected only in NSC and FSC at a level of 0.78 and 0.48 mg 100 g^{-1} , respectively.

Amino acid composition

Table 4 shows the AA profile of five OSC. The contents of AA differed significantly among the five OSC. The present results indicate that glutamic acid (2.96– 7.96 g 100 g⁻¹), arginine (1.72-4.75 g 100 g⁻¹), aspartic acid $(1.53-4.54 \text{ g } 100 \text{ g}^{-1})$ and glycine $(1.46-$ 2.55 g 100 g^{-1}) were the major AAs in the OSC. Olaofe et al. (1994) reported abundance of glutamic and aspartic acids in flour samples of different oilseeds. Among the hydrophobic AAs (HAAs), MSC

Phenolic compounds		FSC	SSC	MSC	NSC	GSC			
Phenolic acids (Hydroxybenzoic acids)									
GAC	Free	1.85 ± 0.10^a	ND	$3.08 \pm 0.06^{\rm b}$	ND	ND			
	Bound	ND	ND	2.66 ± 0.12^a	ND	ND			
PCA	Free	$1.67 \pm 0.05^{\circ}$	16.55 ± 0.13^c	$9.05 \pm 0.09^{\rm b}$	$8.95 \pm 0.12^{\rm b}$	ND			
	Bound	ND	0.68 ± 0.04^a	$7.98 \pm 0.13^{\rm b}$	$0.79 \pm 0.09^{\rm a}$	ND			
SYA	Free	ND	ND	56.33 ± 0.20^a	47.11 \pm 0.39 ^a	118.08 ± 1.03^b			
	Bound	ND	ND	ND	ND	ND			
VLA	Free	1.87 ± 0.06^a	42.53 ± 0.42^c	$14.05 \pm 0.31^{\rm b}$	$21.65 \pm 0.20^{\rm b}$	ND			
	Bound	46.20 \pm 0.54 ^b	2.27 ± 0.11^a	ND	ND	6.16 ± 0.13^a			
	Phenolic acids (Hydroxycinnamic acids)								
FRA	Free	$0.76 \pm 0.05^{\circ}$	ND	7.74 ± 0.07^c	7.04 ± 0.14^b	8.85 ± 0.13^c			
	Bound	ND	ND	$2.45 \pm 0.11^{\rm b}$	0.25 ± 0.03^a	ND			
CFA	Free	0.46 ± 0.04^a	$9.30 \pm 0.14^{\rm b}$	ND	$0.59 \pm 0.03^{\rm a}$	ND			
	Bound	$7.17 \pm 0.11^{\rm b}$	0.35 ± 0.04^a	0.15 ± 0.04^a	$0.16 \pm 0.03^{\rm a}$	0.18 ± 0.01^a			
CGA	Free	ND	ND	ND	$15.32 \pm 0.24^{\rm b}$	7.76 ± 0.13^a			
	Bound	78.28 ± 0.77^c	$0.46 \pm 0.05^{\circ}$	$3.57\,\pm\,0.07^{\rm b}$	1.68 ± 0.07^a	$2.42 \pm 0.14^{\rm b}$			
SPA	Free	ND	ND	ND	1.45 ± 0.04^a	ND			
	Bound	ND	ND	ND	ND	ND			
p-CA	Free	1.13 ± 0.13^a	$0.49 \pm 0.03^{\rm a}$	ND	$2.38 \pm 0.03^{\rm a}$	$80.13 \pm 0.13^{\rm b}$			
	Bound	$1.67 \pm 0.04^{\rm b}$	ND	ND	0.56 ± 0.04^a	ND			
CMA	Free	ND	4.76 ± 0.04^a	ND	ND	22.43 \pm 0.24 ^b			
	Bound	ND	ND	ND	ND	ND			
Total free phenolic acids		7.74	73.63	90.25	104.49	237.25			
Total bound phenolic acids		133.32	3.76	16.81	3.44	8.76			
Flavonoids (Flavan-3-ols)									
Catechin	Free	13.04 ± 0.24^a	ND	ND	ND	38.34 ± 0.13^b			
	Bound	386.04 \pm 2.49 ^a	ND	ND	ND	ND			
Epicatechin	Free	ND	ND	ND	$23.87 \pm 0.23^{\rm b}$	$4.56 \pm 0.13^{\circ}$			
	Bound	ND	ND	ND	$3.74 \pm 0.09^{\rm b}$	$1.35 \pm 0.08^{\rm a}$			
Flavonoids (Flavonols)									
Rutin	Free	$0.66 \pm 0.08^{\rm a}$	6.74 ± 0.15^a	1221.79 ± 5.47^b	72.04 ± 0.51^a	72.48 ± 0.47^a			
	Bound	5.14 ± 0.06^a	ND	ND	ND	ND			
Quercetin	Free	0.91 ± 0.11^a	ND	$2.18 \pm 0.07^{\rm b}$	ND	6.16 \pm 0.11 ^c			
	Bound	ND	ND	ND	ND	ND			
Total free flavonoids		14.61	6.74	1223.97	95.91	121.54			
Total bound flavonoids		391.18	ND	ND	3.74	1.35			
Non-flavonoid polyphenol									
Resveratol	Free	0.37 ± 0.06^a	ND	17.88 ± 0.14^c	$0.99 \pm 0.02^{\rm a}$	$7.46 \pm 0.09^{\mathrm{b}}$			
	Bound	$0.48 \pm 0.07^{\rm a}$	ND	ND	$0.78 \pm 0.03^{\rm b}$	ND			

Table 3 Free and bound phenolic compounds (mg 100 g⁻¹) of five oilseed cakes

CFA, Caffeic acid; CGA, Chlorogenic acid; CMA, Cinnamic acid; FRA, Ferulic acid; FSC, Flaxseed cake; GAC, Gallic acid; GSC, Groundnut seed cake; MSC, Mustard seed cake; ND, Not detected; NSC, Nigella seed cake; p-CA, p-Coumaric acid; PCA, Protocatechuic acid; SPA, Sinapic acid; SSC, Sesame seed cake; SYA, Syringic acid; VLA, Vanillic acid.

had high contents of isoleucine, alanine, glycine, methionine and tryptophan (3.53, 2.97, 2.55, 2.24 and 1.26 g 100 g^{-1} , respectively) while it had low contents of leucine, phenylalanine and proline (0.87, 0.54 and 0.24 g 100 g^{-1} , respectively). SSC was characterised by high contents of valine and leucine (2.27 and $2.97 \text{ g } 100 \text{ g}^{-1}$, respectively) while GSC had high contents of proline and phenylalanine (2.85 and 2.54 g 100 g^{-1} , respectively). FSC had low contents of glycine, isoleucine and methionine (1.46, 0.72 and

0.38 g 100 g^{-1} , respectively) while NSC had low contents of valine and alanine (0.58 and 0.52 g 100g, respectively). However, Al-Gaby (1998) recorded high contents of valine $(4.21 \text{ g } 100 \text{ g}^{-1})$ and alanine $(5.10 \text{ g } 100 \text{ g}^{-1})$ in NSC. The total content of HAAs was high in SSC $(15.44 \text{ g } 100 \text{ g}^{-1})$ and low in FSC $(7.80 \text{ g } 100 \text{ g}^{-1})$. Among acidic AAs (AAAs), glutamic and aspartic acids were more in GSC (7.96 and 4.54 g 100 g^{-1} , respectively) and less in NSC (2.96 and 1.53 g 100 g^{-1} , respectively). Duodu et al. (2018) reported similar levels of glutamic

AAA, Acidic amino acids; Ala, Alanine; Arg, Arginine; Asp, Aspartic acid; BAA, Basic amino acids; Cit, Citrulline; Cys, Cysteine; GABA,

ɣ-aminobutyric acid; Glu, Glutamic acid; Glut, Glutamine; Gly, Glycine; HAA, Hydrophobic amino acids; His, Histidine; Ile, Isoleucine; Leu, Leucine; Lys, Lysine; Meth, Methionine; NAA, Neutral amino acids; ND, Not detected; Phe, Phenylalanine; Pro, Proline; Ser, Serine; Thre, Threonine; Tryp, Tryptophan; Tyro, Tyrosine; Val, Valine.

 $(7.42 \text{ g } 100 \text{ g}^{-1})$ and aspartic $(4.21 \text{ g } 100 \text{ g}^{-1})$ acids in unprocessed groundnut meal. MSC contains only aspartic acid $(3.47 \text{ g } 100 \text{ g}^{-1})$ as glutamic acid was not detected. The level of total AAAs was high in GSC $(12.50 \text{ g } 100 \text{ g}^{-1})$ and low in MSC $(3.47 \text{ g } 100 \text{ g}^{-1})$. GSC contains more arginine (4.75 g 100 g^{-1}) and MSC had a high content of lysine $(2.94 \text{ g } 100 \text{ g}^{-1})$ among basic AAs (BAAs). Duodu et al. (2018) recorded a similar level of arginine $(4.44 \text{ g } 100 \text{ g}^{-1})$ in unprocessed groundnut meal. Histidine was more abundant in SSC and GSC. The level of BAAs was high in GSC $(7.35 \text{ g } 100 \text{ g}^{-1})$ and low in FSC $(2.94 \text{ g } 100 \text{ g}^{-1})$.

Among neutral AAs (NAAs), MSC had high contents of threonine and serine $(5.17 \text{ and } 2.96 \text{ g } 100 \text{ g}^{-1})$, NSC was abundant in cysteine (2.75 g 100 g^{-1}) and GSC contains more tyrosine $(2.18 \text{ g } 100 \text{ g}^{-1})$. However, Duodu et al. (2018) reported low tyrosine $(1.70 \text{ g } 100 \text{ g}^{-1})$ in unprocessed groundnut meal. Glutamine was detected only in FSC $(2.84 \text{ g } 100 \text{ g}^{-1})$. The total content of NAAs was high in MSC $(10.07 \text{ g } 100 \text{ g}^{-1})$ and low in SSC $(3.50 \text{ g } 100 \text{ g}^{-1})$. Citrulline and GABA (nonproteinogenic AAs) were also detected in OSC. Citrulline was detected in SSC, MSC, NSC and GSC (0.27, 0.17,

0.16 and 0.13 g 100 g^{-1} , respectively). GABA was detected in SSC, FSC, NSC and GSC (0.55, 0.44, 0.44 and 0.25 g 100 g^{-1} , respectively). GABA has several physiological functions and beneficial effects on human health (Bhinder et al., 2020).

Functional properties

The OAC and WAC of five OSC ranged from 1.72– 2.10 and 3.32–4.51 g g^{-1} , respectively (Table 5). OAC was highest for NSC and lowest for GSC while WAC was highest for FSC and lowest for MSC. WAC and OAC might be related to lipophilic and hydrophilic constituents of OSC that interact with oil and water. OAC and WAC of GSC were comparable to those reported for defatted groundnut meal flour (Ihekoronye, 1986). However, Joshi et al. (2015) reported lower WAC (2.03 and 2.75 g g^{-1} , respectively) and higher OAC (2.17 and 2.33 $g g^{-1}$, respectively) for defatted groundnut and sesame seed flours. The ability of OSC to retain water and oil suggests their suitable use in various food formulations. OAC and WAC may help us to reduce moisture and fat losses, improve

				NSC	GSC
Parameters	FSC.	SSC	MSC		
OAC (g/g)	$1.97 + 0.07^{\circ}$	$1.87 + 0.10^{\rm b}$	$1.93 + 0.08^{\circ}$	$2.10 + 0.11^d$	$1.72 + 0.07$ ^a
WAC(g/g)	$4.51 + 0.02^d$	$3.43 + 0.13^a$	$3.32 + 0.12^a$	$4.14 + 0.14^c$	$3.83 + 0.12^b$
EAI (m^2/g)	$51.22 \pm 0.20^{\circ}$	27.00 ± 0.16^a	$52.91 + 0.38^c$	$89.52\,\pm\,0.13^{\rm d}$	$35.69 \pm 0.46^{\rm b}$
ESI (min)	$20.14 + 0.67^{\rm b}$	$43.67 + 1.01^c$	$12.42 + 0.79^a$	$16.62 \pm 1.19^{\rm a}$	$48.35 + 0.29^{\circ}$

Table 5 Functional properties of five oilseed cakes

EAI, Emulsifying activity index; ESI, Emulsion stability index; FSC, Flaxseed cake; GSC, Groundnut seed cake; MSC, Mustard seed cake; NSC, Nigella seed cake; OAC, Oil absorption capacity; SSC, Sesame seed cake; WAC, Water absorption capacity.

mouth feel and enhance flavour retention in food products (Khattab & Arntfield, 2009). EAI and ESI of five OSC ranged from 27.00–89.52 m^2 g⁻¹ and 12.42– 48.35 min, respectively (Table 1). NSC and SSC exhibited the highest and lowest EAI while ESI was highest for GSC and lowest for MSC. EAI of MSC was comparable with defatted mustard seed meals (Aluko & McIntosh, 2004). However, EAI of FSC 2004). However, EAI of FSC $(51.22 \text{ m}^2 \text{ g}^{-1})$ was much higher than that of flaxseed protein isolate (40.1 m² g⁻¹) reported by Karaca et al. (2011). The emulsifying properties have been associated with structural flexibilities and surface-active properties of proteins (Bhinder et al., 2020).

Multivariate analysis

Multivariate analysis was performed to establish relationship between variables of OSC (Table S1 and Figure S1). The first four principal components (PC1-4) explained 100% variability (eigenvalues >1 considered) within the observations. PC1, PC2, PC3 and PC4 accounted for variability of 39.4%, 28.6%, 19.9% and 12.1%, respectively. The variables that correlated the most with PC1 were PC (-0.309) , Zn (0.244) , Cu (0.256) , HAA (-0.292) , BAA (-0.324) and OAC (0.266) while MC (-0.337) , Fe (0.283) , TRSC (-0.357) , TFF (-0.270) , NAA (-0.304) and ESI (0.304) contributed to building PC2. FSC and NSC exhibited similar characteristics and were positioned in far right side (quadrant II) of score plot due to high zinc, copper, OAC, WAC, TFC and TRSA (Figure S1a). However, MSC placed in quadrant I had the highest NAA and TPC while low zinc, copper, OAC and WAC compared to FSC and NSC. SSC placed in quadrant III had low TPC, TRSA, NAA, TFF and EAI but high FC. GSC placed in quadrant IV had particularly higher ESI, TFPA and PC with a noticeably higher HAA, AAA and BAA. OSC with higher HAA and BAA had higher ESI and can be incorporated into various aerated baked foods (cakes, souffles and breads) and potentially used to prepare pickering emulsions to deliver heat-sensitive bioactive materials. ESI correlated closely and positively with HAA, AAA and BAA (Figure S1b), indicating the positive impact of protein hydrophobicity on emulsification properties.

Also, TRSA correlated closely and positively with TPC and TFF indicating contribution of phenolics and flavonoids in the antioxidant activity of OSC.

Conclusion

The present work showed that OSC are a good source of protein, minerals, essential AAs and phenolic compounds. FSC had higher minerals (K, Mg, Mn and Cu), TRSC and WAC, NSC exhibited higher TFC, OAC and EAI while MSC had more TPC. GSC had higher PC, ESI, hydrophobic, acidic and basic AAs. SYA and p-CA were identified as major phenolic acids in GSC while rutin and catechin were the predominant flavonoids in MSC and FSC, respectively. The total free phenolic acids and flavonoids were more in GSC and MSC, respectively, while FSC exhibited higher total bound flavonoids and phenolic acids. OSC tested had sufficient amount of minerals and essential AAs to meet the requirement of human diet. The utilisation of tested OSC in different food products such as muffins, cakes and cookies as a potential source of natural antioxidants and nutrients has many advantages. GSC can be used in the preparation of baked foods and pickering emulsions for the deliver of bioactive constituents. Further investigation on incorporation of tested OSC in various food products is under process in our laboratory.

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Conflict of interest

The authors have no conflict of interest to declare.

Author contributions

Manpreet Kaur: Data curation (equal); Investigation (lead); Validation (equal); Writing-original draft (equal). Balwinder Singh: Conceptualization (equal); Formal analysis (equal); Investigation (equal);

Resources (equal); Writing-original draft (equal); Writing-review & editing (lead). Amritpal Kaur: Conceptualization (equal); Formal analysis (equal); Funding acquisition (lead); Investigation (equal); Project administration (lead); Resources (equal). Narpinder Singh: Formal analysis (equal); Resources (equal); Software (equal).

Ethical approval

Ethics approval was not required for this research.

Peer review

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Data availability statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Supporting Information

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

Table S1. Principal components for illustrating the interpretation in Fig S1.

Figure S1. Multivariate analysis score plot (a) and loading plot (b) describing relationship among different variables of OSC.