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Quality characteristics of sesame seeds and by-products

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Abstract

The chemical composition, of raw sesame seed (RS); Sesame coats 1 (SC1) and sesame coats 2 (SC2) obtained as a by-product respectively after dehulling and roasting processes during preparation of sesame paste (tehineh) for the manufacturing of Halaweh (sweetened tehineh), was determined along with the physicochemical characteristics of the oil fraction. Compared to RS, SC1 and SC2 showed higher amounts of dietary fibre, ash and polyphenol and lower amounts of oil and protein. Oil from SC1 and SC2, had a higher content of free fatty acids, chlorophylls, polyphenols and sesamol than RS oil. SC2 oil showed more intense colour, more absorbance in UV-A, UV-B and UV-C ranges and a significant higher viscosity ($P < 0.05$). No differences ($P > 0.05$) were observed for refractive index, iodine value and fatty acids composition. This latter was essentially dominated by oleic and linoleic acids. Oxidative stability of oil was investigated using a Rancimat system and in an oven test at 65 °C over 60 days. RS oil was more resistant to the thermal treatment during a long period than SC1 and SC2 oils.
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Keywords: Chemical composition; Raw sesame seed; By-products; Oil; Oxidative stability

1. Introduction

Sesame (*Sesamum indicum* L.) is cultivated in several countries such as India, Sudan, China and Burma which are considered as the major producers (60% of its total world production) (Abou-Gharbia, Shehata, & Shahidi, 2000). In Tunisia, 80% of the needed sesame seed is imported from Sudan and 20% from Egypt (Institut National de la Statistique, 2005). The imported quantity rose from about 3400 tonnes in 1990 to 10 600 tonnes in 2005 (Institut National de la Statistique, 1990–2005).

Sesame plays an important role in human nutrition. Its seeds are used essentially for the production of oil, but also in the production of the paste (tehineh) and in food formulations such as Halaweh (sweetened tehineh), java beans and salads (Abou-Gharbia et al., 2000; Abu-Jdayil, Al-Malah, & Asoud, 2002; Namiki, 1995).

The chemical composition of sesame shows that the seed is an important source of oil (44–58%), protein (18–25%), carbohydrate (~13.5%) and ash (~5%) (Kahyaoglu & Kaya, 2006; Kamel-Eldin & Appelpvist, 1994a; Mohamed & Awatif, 1998; Shyu & Hwang, 2002; Yoshida, 1994). The oil fraction shows a remarkable stability to oxidation (Abou-Gharbia et al., 2000; Yoshida, Shigezaki, Takagi, & Kajimoto, 1995). This could be attributed to endogenous antioxidants (lignans) together with tocopherols (Yoshida et al., 1995). Mohamed and Awatif (1998) showed that the addition of unsaponifiable matter extracted from sesame seed increases the stability of sunflower oil. This stability is more pronounced in the case of unsaponifiable matter extracted from roasted sesame seeds due to a synergistic role. This latter effect rendered by sesamol formed following the decomposition of sesamolin during roasting and other minor compounds such as squalene and anti-polymerisation components, mainly sterols (Mohamed & Awa-

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tif, 1998). Oxidative stability of sesame oil is high in case of the oil extracted from coated seeds than in those extracted from dehulled seeds (Abou-Gharbia, Shahidi, Shehata, & Youssef, 1997). Chang, Yen, Huang, and Duh (2002) reported that the ethanolic extract of sesame coat shows an antioxidant activity similar to the tocopherol using a model system linoleic acid. In line with this, Shahidi, Liyana-Pathirana, and Wall (2006) have shown that white and black sesame seeds and their coat fractions possess considerable antioxidant activity, especially black sesame coats.

In Tunisia, the major part of the imported sesame is essentially transformed to Halaweh. This food product is obtained after mixing the white tehineh (white sesame seed dehulled, roasted and grinded), saponin (*Saponaria officinalis*) and Nougat (heat-treated sucrose) (Abu-Jdayil et al., 2002). The sesame coat is a by-product of Halaweh manufacture which could be recovered and used as a value added product. However, in some sesame processing countries, this by-product is generally discarded, or used in animal feeding. The aim of this work was to study the chemical composition of the raw sesame seeds as well as the by-products of the Halaweh manufacture (SC1 and SC2), and to determine fatty acid profiles, sensorial profiles and oxidative stability of their lipid fraction.

2. Materials and methods

2.1. Samples

White sesame seed (*S. indicum* L.) from Sudan and by-products of Halaweh manufacture were obtained after dehulling (sesame coat 1: SC1) and after roasting (sesame coat 2: SC2) during the preparation of the sesame paste for the manufacture of Halaweh (Fig. 1). SC1 and SC2 consisted of the sesame coat and dehulled seed with a predominance of the coat fraction for SC1.

The raw sesame seed (RS) and these by-products were supplied by the Moulin-Triki Industry (Sfax, Tunisia). The relative percentage weight (on dry matter basis) of by-products, compared to the raw sesame seed (RS) weight, was about 13.6% and 0.7% for SC1 and SC2, respectively (Fig. 1). RS was sieved to discard impurity and SC1 was dried for 24 h at 40 °C. Then, RS, SC1 and SC2 were milled and preserved at −20 °C until analysis and oil extraction.

2.2. Oil extraction and preservation

RS, SC1 and SC2 (50 g) were placed in dark flasks (capacity = 1 l) and homogenized with 250 ml of hexane. The mixture was agitated in a shaker (Rotabit, Selecta, Spain) at 180 min^{-1} during 4 h at ambient temperature (~20 °C). The hexane was separated using centrifugation (1000 g for 15 min at 20 °C) and filtration. The extraction procedure was repeated twice and the extracts were evaporated using a rotary evaporator at 40 °C. The obtained oil was drained under a stream of nitrogen and then stored in a freezer (−20 °C) for subsequent physico-chemical analyses.

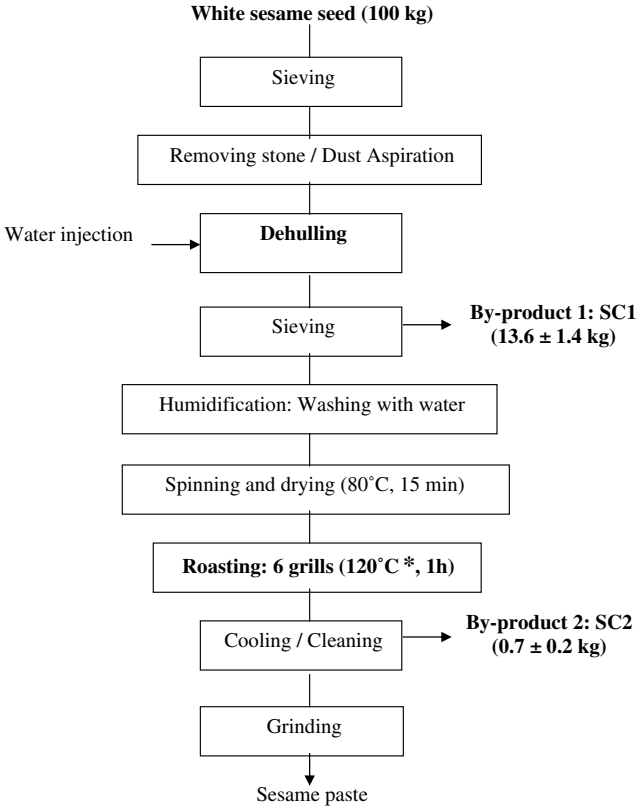

Fig. 1. Stages of the by-product elimination during the preparation of the sesame paste for the manufacturing of Halaweh. SC1: sesame coat 1; SC2: sesame coat 2. *Temperature at the end of the roasting.

2.3. Analytical methods

2.3.1. Chemical analysis of RS, SC1 and SC2

2.3.1.1. Dry matter. This was determined by oven drying at 105 °C to constant weight (AOAC, 1990).

2.3.1.2. Fat content. This was determined by Soxhlet extraction with hexane for 8 h at boiling point of the solvent (68–70 °C) (Abaza, Msallem, Daoud, & Zarrouk, 2002). The extraction procedure was previously described by Manirakiza, Covaci, and Scepens (2001). This extraction (Soxhlet method) was carried out to estimate the content of oil (Abaza et al., 2002).

2.3.1.3. Protein. Total nitrogen was determined by the Kjeldahl method as described by Pearson (1970). Protein was calculated using the general factor (6.25) (Khalid, Babiker, & EL Tinay, 2003).

2.3.1.4. Dietary fibre. Insoluble and soluble dietary fibres were determined according to the AOAC enzymatic-gravimetric method of Prosky, Asp, Schweizer, De Vries, and Furda (1988). Briefly, the defatted samples were gelatinized

with heat stable alpha amylase (A-3306, sigma chemical Co., St. Louis, MO, USA) (100 °C, pH 6, 15 min) and then enzymatically digested with protease (P-5380, sigma Chemical Co., St. Louis, MO) (60 °C, pH 7.5, 30 min) followed by incubation with amyloglucosidase (A-9268, Sigma Chemical Co., Poole, Dorset, UK) (60 °C, pH 4.5, 30 min) to remove protein and starch. Then, the samples were filtered, washed (with water, 95% ethanol and acetone), dried and weighted to determine insoluble fibre. Four volumes of 95% ethanol (preheated to 60 °C) were added to the filtrate and to the water washings. Then, the precipitates were filtered and washed with 78% ethanol, 95% ethanol and acetone. After that, the residues (soluble fibre) were dried and weighted. The obtained values were corrected for ash and protein. Total dietary fibre was determined by summing insoluble dietary fibre and soluble dietary fibre.

2.3.1.5. Starch. After removing sugars with ethanol (80%), starch was isolated by extraction with perchloric acid reagent (52%) twice, from a sugar-free residue according to the method described by McCready, Guggolz, Silviera, and Owens (1950). Starch in the extract was determined using the anthrone reagent and colorimetric measurement at 630 nm (McCready et al., 1950).

2.3.1.6. Soluble sugars. Sugars were extracted with ethanol (960 ml/l) by shaking at 50 °C for 30 min (Larrauri, Rupérez, Borroto, & Saura-Calixto, 1996). After centrifugation, the supernatant was collected and the sugar content was analysed with phenol/sulphuric acid reagent (Dubois, Gilles, Hamilton, Rebers, & Smith, 1956).

2.3.1.7. Ash and mineral content. Ash was determined by combustion of the sample in a muffle furnace at 550 °C for 12 h (Bryant & McClements, 2000). The residue was dissolved in HNO_3 with 50 g/l of $LaCl_3$ (Larrauri et al., 1996) and the mineral constituents (Ca, K, Mg, Na, Fe, Cu, Zn and Mn) were analysed separately, using an atomic absorption spectrophotometer (Hitachi Z6100, Tokyo, Japan). Phosphorus content (P) was determined by the phosphomolybdate method (AOAC, 1990).

2.3.1.8. Polyphenol determination. RS and by-products (0.5 g) were extracted three times with methanol:water (70:30, v/v): 10 ml for 1 h, 10 ml for 30 min and 5 ml for 30 min in a shaker (Rotabit, Selecta, Spain, 200 min^{-1}, ambient temperature (~20 °C)). After centrifugation (1000 g, 10 min, 20 °C), the supernatants were combined to a final volume of 25 ml. Total polyphenols were determined using the Folin-Ciocalteu colorimetric method (Singleton & Rossi, 1965). Results were expressed as milligrams of gallic acid equivalents.

2.3.2. Analysis of oil extract

2.3.2.1. Index determination. AOCS (1997) official methods were used for the determination of the acidity (method Cd 3d-63) and iodine value (method Cd 1-25). The refractive index was determined using an Abbe refractometer (Bellinghan, & Stanley Ltd, United Kingdom) at 40 °C (AOAC, 1990).

2.3.2.2. Fatty acid composition. Fatty acid composition was determined by gas–liquid chromatography after derivatization to fatty acid methyl esters (FAMEs) with 2 M KOH in methanol at room temperature according to the IUPAC (1992) standard method. GC analyses were achieved according to the method described by Besbes, Blecker, Deroanne, Drira, and Attia (2004).

2.3.2.3. Colour and UV–visible profile. The CieLab coordinates (L^*, a^*, b^*) were directly read with a spectrophotocolorimeter (Tintometre, Lovibond PFX 195 V 3.2, Amesbury, UK). In this coordinate system, the L^* value is a measure of lightness, ranging from 0 (black) to 100 (white), the a^* value ranges from −100 (greenness) to +100 (redness) and the b^* value ranges from −100 (blueness) to +100 (yellowness).

UV–visible profiles of oil solution in hexane were measured with a spectrophotometer (SECOMAN, Type: ANTHELIE 70 MI 0291, No: 344, Domont, France) with light of wave lengths between 205 and 800 nm.

2.3.2.4. Chlorophyll content. Chlorophyll (mg/kg) was quantified by spectrophotometry according to AOCS (1997) method Cd 13d-55.

2.3.2.5. Viscosity determination. Viscosity was followed at 25 °C with a Stress Tech Rheologica Rheometer (Rheologica Instruments AB, Lund, Sweden) using a steel cone-plate (C40/4) under a constant shear rate of 100 s^{-1}.

2.3.2.6. Polyphenols and sesamol determination. Polyphenols were isolated by extraction with methanol:water (60:40, v/v) three times, from an oil-in-hexane (5 g:10 ml) solution with a separatory funnel, according to the method described by Satue, Huang, and Frankel (1995). The Folin-Cicalteau reagent was added to a suitable aliquot of the combined extract, and the absorption of the solution at 727 nm was measured. The results were expressed as gallic acid (mg/kg of oil) (Gutfinger, 1981).

Sesamol was isolated by extraction with potassium hydroxide solution (potassium hydroxide:ethanol:water, 10:20:80, w/v/v) three times, from an oil-in-isooctane using a separatory funnel according to the method described by Budowski, O'conner, and Field (1950). Sesamol was determined in the extract using a furfural solution and colorimetric measurement at 518 nm (Budowski et al., 1950).

2.3.2.7. Oxidative stability. Two methods were used to evaluate the oxidative stability for the oils fraction (Oven test and Rancimat method).

2.3.2.7.1. Oven test. Oil samples (70 g) were kept in equal portions in open flasks (30 ml capacity, 30 mm diameter

and 70 mm height) in the dark in an oven (Binder, No: 970465, Tuttlinger, Germany) at 65 °C over 60 days. Samples were removed each 5 days. The stability to the oxidation was evaluated by the peroxide value (PV): 1 ± 0.1 g of each oil sample was weighed and subjected to iodometric determination according to Cd 1-25 method (AOCS, 1997). Specific absorptivity at 232 and 270 (absorption of 1% solution in cyclohexane at 232 and 270 nm with 1 cm of pass length) were also determined using a UV spectrophotometer (SECOMAN, Type: ANTHELIE 70 MI 0291, No: 344, Domont, France).

2.3.2.7.2. Rancimat method. Oxidative stability was also evaluated by the Rancimat method. Stability was expressed as the oxidation induction period (h), measured with the Rancimat 679 apparatus (Metrohm AG, Herison, Switzerland) using an oil sample of 5 g, warmed to 100 °C and a purified air flow rate of 20 l/h. In the rancimat method, the volatile degradation products were trapped in distilled water and measured conductmetrically. The induction period was defined as the necessary time to reach the inflection point of the conductivity curve (Halbault, Barbé, Arroztegui, & De La Torre, 1997).

2.4. Statistical analysis

All analytical determinations were performed at least in triplicate. Values of different parameters were expressed as the mean ± standard deviation ($\bar{x} \pm SD$). Significant differences between mean ($P < 0.05$) were determined by Fisher's test.

3. Results and discussion

3.1. Chemical composition

Table 1 presents the approximate chemical composition of raw sesame seed (RS) (*S. indicum* L.) and by-product of Halaweh manufacture obtained after dehulling SC1 and roasting SC2 (Fig. 1). SC1 shows higher moisture content than RS and SC2 (16.20 against 3.88% and 2.98%). This difference is likely due to the humidification and roasting processes realised before obtaining SC1 and SC2, respectively, during the preparation of sesame paste (Fig. 1).

Compared to the RS, SC1 and SC2 contained higher amounts of dietary fibre in the range of 32.34–42.03% (Table1). The by-products of Halaweh could be considered as a potential fibre source that could be used in food formulations. Insoluble dietary fibre was the major fraction which ranged between 13.96% and 33.41% (Table1). The soluble dietary fibre contents in SC1 and SC2 are relatively high compared to cereal derivatives (corn bran, wheat bran, oat bran, rice bran) which have a low soluble dietary fibre (between 0.4% and 4.1%) (Abdul-Hamid & Luan, 2000; Grigelmo-Miguel & Martina-Belleso, 1999a; Prosky et al., 1988).

RS, SC1 and SC2 contained a low percentage of starch (0.84–1.33%) and low soluble sugars contents (0.97–2.48%). Thus, the carbohydrate was dominated by the non-starch polysaccharide (Dietary fibre).

Table 1
Chemical composition of raw sesame seeds and by-products of Halaweh manufacturing

Component	RS	By-products	
		SC1	SC2
Dry matter (%)	$95.29^{b} \pm 0.19$	$83.79^{a} \pm 0.04$	$97.02^{c} \pm 0{,}90$
Oil[A]	$52.24^{c} \pm 0.34$	$12.21^{a} \pm 0.02$	$32.84^{b} \pm 2.32$
Protein[A]	$25.77^{c} \pm 1.02$	$10.23^{a} \pm 0.32$	$18.35^{b} \pm 0.16$
Total fibers[A]	$19.33^{a} \pm 1.97$	$42.03^{c} \pm 0.60$	$32.34^{b} \pm 1.36$
Insoluble fibers[A]	$13.96^{a} \pm 1.62$	$33.41^{c} \pm 0.32$	$26.08^{b} \pm 0.52$
Soluble fibers[A]	$5.37^{a} \pm 0.28$	$8.61^{b} \pm 0.32$	$6.26^{a} \pm 0.84$
Ash[A]	$4.68^{a} \pm 0.20$	$23.90^{c} \pm 1.04$	$13.7^{b} \pm 0.844$
Calcium[A]	$1.03^{a} \pm 0.04$	$10.54^{b} \pm 0.13$	$7.94^{c} \pm 0.58$
Potassium[B]	$525.9^{b} \pm 17.90$	$441.4^{a} \pm 23.3$	$451^{a} \pm 36.8$
Magnesium[B]	$349.9^{a} \pm 39.32$	$455.9^{b} \pm 23.06$	$449.6^{b} \pm 34.7$
Phosphorus[B]	$516^{c} \pm 26.89$	$158.0^{a} \pm 0.93$	$437.2^{b} \pm 37.6$
Sodium[B]	$15.28^{a} \pm 1.63$	$39.80^{b} \pm 0.83$	$117.4^{c} \pm 20.9$
Iron[B]	$11.39^{a} \pm 0.27$	$47.13^{c} \pm 2.55$	$29.70^{b} \pm 0.08$
Copper[B]	$2.15^{a} \pm 0.06$	$3.48^{b} \pm 0.57$	$3.48^{b} \pm 0.51$
Zinc[B]	$8.87^{b} \pm 0.26$	$6.73^{a} \pm 0.45$	$7.98^{a,b} \pm 0.74$
Manganese[B]	$3.46^{a} \pm 0.43$	$5.10^{b} \pm 0.46$	$3.66^{a} \pm 0.09$
Soluble sugars[A]	$2.48^{c} \pm 0.09$	$0.97^{a} \pm 0.09$	$1.34^{b} \pm 0.05$
Starch[A]	$0.88^{b} \pm 0.01$	$1.33^{c} \pm 0.01$	$0.84^{a} \pm 0.01$
Polyphenols[B]	$87.77^{c} \pm 3.15$	$598.2^{a} \pm 4.47$	$260.6^{b} \pm 3.99$

All the given values are means of tree determinations ± standard deviation. Mean in a row followed by the same letters are not significantly different ($P > 0.05$).
RS: raw sesame seed, SC1: sesame coat 1, SC2: sesame coat 2.
[A] In % dry matter.
[B] In mg/100 g dry matter.

Regarding the ash, SC1 and SC2 contained higher amounts than RS in the range of 13.7–23.9%. The by-products could be considered as potential source that could be used to meet part the nutritional requirement of animal feeds. The ash content in SC1 and SC2 was high compared to the other by-products (wheat bran, rice bran, oat bran, washed peach bagasse, washed orange bagasse) which have a relatively low ash content (between 2.6% and 8%) (Abdul-Hamid & Luan, 2000; Grigelmo-Miguel, Gorinstein, & Martina-Belleso, 1999; Grigelmo-Miguel & Martina-Belleso, 1999a, 1999b). The mineral composition of RS and by-products shows that calcium was the predominant mineral followed by potassium, magnesium and phosphorus. All other elements were present in comparatively low concentrations (Table 1). The mineral elements contents varied significantly ($P < 0.05$) between RS and SC1. This could be due to the recuperation or elimination of the mineral elements during the dehulling of sesame seed. For instance, calcium content of 10.54% in SC1 was considerably higher than 1.03% for RS. This can be explained by the fact that most of the calcium was recuperated in sesame coat during the dehulling processes. Indeed, El-Adawy and Mansour (2000) reported a very low content of calcium for dehulled sesame seed (~0.064%).

RS and SC2 were a good source of protein, yielding 18.35–25.77% total dry mass (Table 1). However, SC1 contained a lower percentage of protein (~10%) than SC2 and

other by-products such as rice bran and wheat bran as was shown by Claye, Idouraine, and Weber (1996) and Grigelmo-Miguel and Martina-Belleso (1999a).

The RS contained 52.24% of oil, while SC2 had 32.84% and SC1 had only 12.21%. This indicates that the oil was mainly localised in the dehulled seed (endosperm layers). This confirms previous findings by El-Adawy and Mansour (2000) who reported a high content of oil for the dehulled sesame seeds (~58.9%).

The polyphenols content of SC1 (598 mg/100 g) was higher than those of SC2 (260 mg/100 g) and RS (87.33 mg/100 g). Table 1 shows that the high value of polyphenols is related to the high dietary fibre content. This can be explained by the fact that the polyphenols are compound associated with dietary fibre (Larrauri et al., 1996). Other studies on the vegetable seeds, the cereal grains and the fruits correlate with these findings. Indeed, it was established that coats of vegetable seeds, coats of cereal grains and peels of fruits (characterized by the high dietary fibre content) contain higher amounts of polyphenols than the cotyledon, the endosperm and the pulp fractions respectively (Dueñas, Hernández, & Estrella, 2002; Gorinstein et al., 2001; Kahkonen et al., 1999; Shahidi et al., 2006).

The chemical composition of SC1 and SC2 revealed that these by-products of Halaweh manufacture could be valuable. In order to justify the extraction of oil, it is necessary to study its functional properties.

3.2. Physicochemical profiles of the RS, SC1 and SC2 oils

3.2.1. Extraction yield and characteristics index

The Soxhlet extraction (hot extraction, Section 2.3.1.2) showed a higher oil yield than the method described in Section 2.2 (cold extraction) (Tables 1 and 2). (In the protocol described in Section 2.2, the conditions of extraction have been chosen at random.) The difference in the yield at each extraction can be due to the effect of heat on oil extraction and to the progressive depletion by the successive and multiple washing cycles by the Soxhlet extraction (the sample is always in contact with fresh and hot solvent) (Manirakiza et al., 2001).

Based on the fact that the temperature of extraction can affect the initial quality of oil (Besbes, Blecker, Deroanne, Lognay, et al., 2004), the cold extract was used in the following study.

SC1 and SC2 oils had a relatively high content in the free fatty acid (FFA) ranging between 6.68% and 10.73%. These values were higher than 2.37% for RS oil. This can be explained by the injection of the water during dehulling of sesame seed as well as the moisture content in SC1 (16.20%). These conditions are favourable to a lipasic activity before drying in the oven. Indeed, FFA were partly formed by hydrolysis of triacylglycerols, which was promoted by the presence of food moisture (Al-Harbi & Al-Kabtani, 1993).

Regarding the refractive index, there was no significant difference ($P > 0.05$) between RS and by-product oils (Table 2). Thus, the dehulling and roasting processes did not cause a significant modification ($P > 0.05$) in the short-medium hydrocarbon contents. The refractive index of RS and by-products oils was higher compared to that of other oils (date seed oil, *Xylopia aethiopica* seed oil, *Moringa oleifera* seed oil, Virgin olive oil) (Barminas, James, & Abubakar, 1999; Besbes, Blecker, Deroanne, Lognay, et al., 2004; Lalas & Tsaknis, 2002).

Table 2
Physicochemical characterisation of raw sesame seeds and by-products of Halaweh manufacturing oils

Determination	RS	By-products	
		SC 1	SC 2
Oil (%)[A]	$41.46^{c} \pm 1.52$	$7.03^{a} \pm 0.08$	$25.65^{b} \pm 0.54$
Free fatty acids (as oleic acid %)	$2.37^{a} \pm 0.41$	$10.73^{c} \pm 0.16$	$6.68^{b} \pm 0.12$
Iodine value (g of I_2/100 g of oil)	$99.08^{a} \pm 0.73$	$98.76^{a} \pm 0.99$	$97.87^{a} \pm 0.33$
Chlorophyll (mg/kg)	$0.18^{a} \pm 0.04$	$2.45^{b} \pm 0.09$	$4.30^{c} \pm 0.05$
Viscosity (mPa s)	$12.93^{a} \pm 0.05$	$13.00^{a} \pm 0.10$	$14.53^{b} \pm 0.05$
Refractive index (at 40 °C)	$1.470^{a} \pm 0.001$	$1.4705^{a} \pm 0.0$	$1.4703^{a} \pm 0.001$
Polyphenols (as mg gallic acid/kg of oil)	$23.06^{a} \pm 4.41$	$79.32^{c} \pm 3.35$	$71.45^{b} \pm 3.05$
Sesamol (mg/kg of oil)	$8.11^{a} \pm 0.70$	$22.00^{b} \pm 2.32$	$54.86^{c} \pm 2.21$
Induction period (h)	$28.23^{c} \pm 0.73$	$25.76^{b} \pm 0.72$	$20.60^{a} \pm 0.60$

All the given values are means of tree determinations ± standard deviation. Mean in a row followed by the same letters are not significantly different ($P > 0.05$).
RS: raw sesame seed, SC1: sesame coat 1, SC2: sesame coat 2.
[A] Extraction with the method described in Section 2.2.

SC1 and SC2 oils show high iodine values (about 98 g of I_2/100 g of oil) comparable to those found in RS (Table 2). This result indicates that these oils are non-drying, highly unsaturated and suggests that they contain high levels of oleic and linoleic acids (Ajayi, Oderinde, Kajogbola, & Uponi, 2006; Norris, 1965) (see also fatty acid composition, Table 3). Abou-Gharbia et al. (1997) reported a higher iodine value in fresh oils prepared from raw sesame seeds of an Egyptian variety (111.7 against 99.08 g of I_2/100 g). Roasting and dehulling processes did not cause a significant change ($P > 0.05$) in the iodine value (Table 2). These results are further supported by the fatty acids composition.

3.2.2. Fatty acid composition

Fatty acid composition of the extracted oils from RS, SC1 and SC2 is presented in Table 3. The most abundant fatty acids were oleic (~ 43%), linoleic (~35%), palmitic (~11%) and stearic (~7%) acids, which together comprised about 96% of the total fatty acids (Table 3). Compared to the Japanese variety (White species) studied by Yoshida (1994) and Yoshida et al. (2000), this white RS from Sudan (present study) was higher in oleic acid (~45 against ~38%) and lower in linoleic aid (~35.5 against ~48%).

Table 3
Fatty acid composition (%) of raw sesame seeds and by-products of Halaweh manufacturing oils

Fatty acid	RS	By-products	
		SC1	SC2
Palmitic C16:0	$11.18^{a} \pm 0.76$	$10.93^{a} \pm 0.21$	$11.39^{a} \pm 0.36$
Palmitoleic C16:1	$0.21^{a} \pm 0.005$	$0.21^{a} \pm 0.02$	$0.24^{a} \pm 0.02$
Stearic C18:0	$6.40^{a} \pm 0.17$	$6.86^{b} \pm 0.07$	$6.92^{b} \pm 0.02$
Oleic C18:1(n-9)	$44.06^{a} \pm 0.58$	$43.15^{a} \pm 0.18$	$43.07^{a} \pm 0.30$
cis-Vaccenic C18:1(n-11)	$0.97^{a} \pm 0.002$	$0.97^{a} \pm 0.005$	$0.96^{a} \pm 0.03$
Linoleic C18:2	$35.56^{a} \pm 0.07$	$36.07^{a} \pm 0.27$	$35.61^{a} \pm 0.12$
Linolenic C18:3	$0.50^{a} \pm 0.03$	$0.51^{a} \pm 0.04$	$0.51^{a} \pm 0.04$
Arachidic C20:0	$0.70^{a} \pm 0.04$	$0.74^{b} \pm 0.02$	$0.83^{c} \pm 0.003$
Eicosenoic C20:1	$0.18^{a} \pm 0.01$	$0.20^{b} \pm 0.01$	$0.22^{c} \pm 0.005$
Lignoceric C24:0	$0.20^{a} \pm 0.005$	$0.19^{a} \pm 0.01$	$0.22^{a} \pm 0.012$
SAFA	18.49 ± 0.98	18.85 ± 0.50	19.37 ± 0.40
MUFA	45.44 ± 0.60	44.64 ± 0.11	44.50 ± 0.36
PUFA	36.06 ± 0.11	36.59 ± 0.04	36.12 ± 0.16

All the given values are means of tree determinations ± standard deviation. Mean in a row followed by the same letters are not significantly different ($P > 0.05$).
SC1: sesame coat 1, SC2: sesame coat 2, SAFA: saturated fatty acids, MUFA: monounsaturated fatty acids, PUFA: polyunsaturated fatty acid, RS: raw sesame seed.

The dehulling and roasting processes did not cause a significant ($P > 0.05$) modification in the major fatty acid composition (Table 3). Yoshida (1994) found that no significant difference ($P > 0.05$) occurred in fatty acid composition between unroasted and roasted sesame seed even after roasting at 250 °C during 30 min.

3.2.3. Phenol and sesamol content

SC1 and SC2 oils showed a high total phenol content compared to RS oil (71.45–79.32 against 23.06 mg as allic acid/kg of oil). These total phenol contents are higher than those for other oil such as coriander seed oil and niger seed oil as was shown by Ramadan and Mörsel (2004) (between 5 and 11 mg/kg oil).

Sesamol is a potent phenolic antioxidant (Budowski et al., 1950; Yoshida et al., 1995; Yoshida & Takagi, 1997). It was detected in low amounts in RS oil (8.11 mg/kg oil). This amount was much higher than that reported by Mohamed and Awatif (1998) (0.2 mg/kg oil), but lower than that reported by Budowski et al. (1950) (10 mg/kg oil) using the same colorimetric method. This difference may be due to extraction techniques of oil, environmental and ecological characteristics of the particular growing area. Yoshida and Takagi (1997) reported a sesamol content of 5.1 mg/kg using chromatographic method. The sesamol increased after dehulling to 22 mg/kg and considerably to 54.86 mg/kg after roasting. This was probably due to the conversion of sesamolin to sesamol during roasting as was explained by Yoshida et al. (1995), Yoshida and Takagi (1997) and Mohamed and Awatif (1998). Yoshida and Takagi (1997) reported that sesamol was most effective in stabilizing substrate oil than tocopherols (δ, γ and α) when added individually at 800 mg/kg levels. Fukuda, Nagata, Osawa, and Namiki (1986) and Yoshida and Takagi (1999) found that sesamol has a synergitic action with γ-tocopherol.

3.2.4. Viscosity

RS, SC1 and SC2 oils showed a lower viscosity (between 12 and 15 mPa s) than most vegetable oils (mean value ~50–100 mPa s) as was reported by Besbes et al. (2005). This could be explained by the high content of monounsaturated and polyunsaturated fatty acids as previously shown in Table 3 (Besbes, Blecker, Deroanne, Drira, et al., 2004; Geller & Goodrum, 2000). The roasting processes caused a significant increase ($P < 0.05$) in the oil viscosity (Table 2). This was attributed to polymerisation and formation of high-molecular-weight compounds including carbon–carbon bonds and carbon–oxygen–carbon bond between fatty acids (Guillén & Ruiz, 2004; Stevenson, Vaisey-Genser, & Eskin, 1984).

3.2.5. Colour and UV–visible profile

CieLab coordinates (L^*, a^*, b^*) of the oil extracted from the raw sesame seed and by-products is shown in Fig. 2. Compared to the oil extracted from raw sesame, SC 1 and SC2 showed a lower L^* and a higher a^* and b^* values. This means that dehulling and roasting processes have caused an increase in the dark, red and yellow units of colour. Yoshida and Takagi (1997) showed that the roasting processes causes an increase in the red value which is more pronounced with roasting temperatures up to 200 °C. The colour formation in sesame oil during heating processes could be attributed to both non-enzymatic browning and phospholipids degradation during roasting (Husain, Terao, & Matsushita, 1986; Mohamed & Awatif, 1998; Yoshida, 1994; Yoshida & Takagi, 1997). The non-enzymatic browning is favoured by heat treatment and includes a wide number of reactions such as Maillard reaction, caramilisation and chemical oxidation of phenols (Manzocco, Calligaris, Mastrocola, Nicoli, & Lerici, 2001; Tomasik, Wiejak, & Palasinski, 1989). For instance, in the case of Maillard reaction, the sesame seed contains the required reaction, sugars and amine groups as found in protein molecules, to give the Maillard reaction products (MRPs)

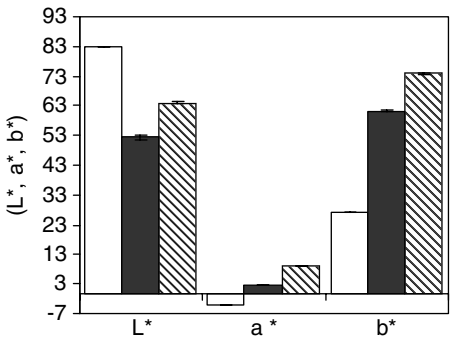

Fig. 2. CieLab coordinates (L^*, a^*, b^*) of seeds and sesame coats oils. (□) Raw sesame seed, (■) sesame coat 1, (▨) sesame coat 2.

(Horrobin, Landman, & Ryder, 2003). Thus, the colour formation during heat treatment is partly due to the formation of coloured MRPs. These latter correspond to compounds with a low-molecular-weight and melanoidins with high-molecular-weight (Ames, 1992, chap. 4). The MRPs in oil consisted of the non-polar compounds of roasted seeds solubilized in hexane during extraction of oil.

RS, SC1 and SC2 oils showed some absorbance in the UV-C (100–290 nm), UV-B (290–320 nm), UV-A (320–400 nm) and visible (400–800 nm) range. This absorbance was more accentuated for SC2 oil (Fig. 3). This could be explained by roasting processes which could contribute to the absorbent compounds formation in the UV and visible light.

In the UV-B and the UV-A ranges, the wavelengths of the ultraviolet light are responsible for most of the cellular damage (Oomah, Ladet, Godfrey, Liang, & Girard, 2000). RS, SC1 and SC2 oils shield against UV-B and UV-A radiation as indicated by the absorbance at 290–400 nm (Fig. 3). Thus RS, SC1 and SC2 oils may be used in the formulation of UV protectors which provide protection against both UV-A and UV-B.

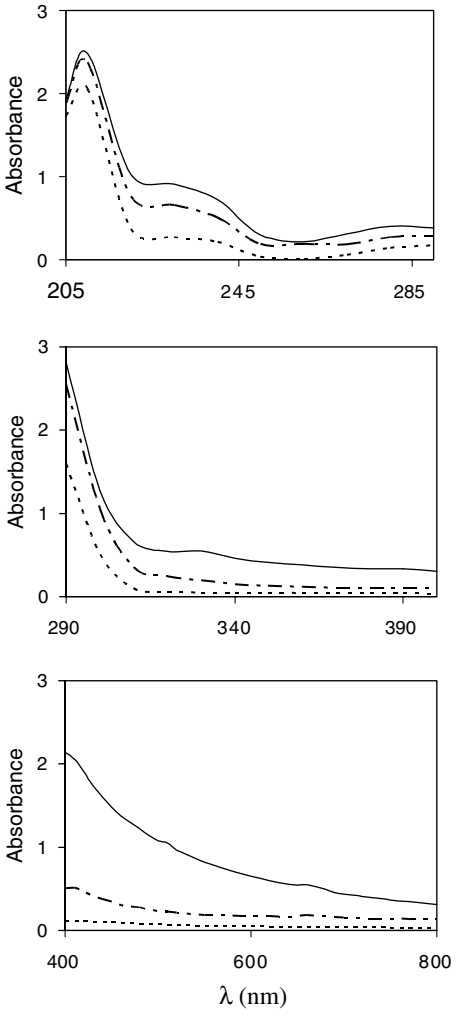

Fig. 3. Ultra-violet spectra of seeds and sesame coats oils. Figure derived from scan ($\lambda = 205$–290 nm) of oil diluted 1:800; from scan ($\lambda = 290$–400 nm) of oil diluted 1:100 and from scan ($\lambda = 400$–800 nm) of oil diluted 1:10, all in hexane. (---) Raw sesame seed, (– - –) sesame coat 1, (—) sesame coat 2.

SC2 oil showed a darker colour as indicated by the high absorbance at all wavelengths (Fig. 3). SC2 oil contained also more yellow colouring than SC1 and RS oils, as indicated by the absorbance at 440–460 nm for 10% oil in hexane (Fig. 3). This confirms the results obtained with the Lovibon instrument (Fig. 2). This yellow colour, which includes carotenoids, is beneficial, since it stimulates the appearance of butter without the use of primary colourants, such as carotenes and annattos, commonly used in the oil and fat industry (Oomah et al., 2000).

Roasting and dehulling treatment caused also an increase in green pigment and particularly in chlorophyll content (600–750 nm) (Table 2). Oomah, Busson, Godfrey, and Drover (2002) showed that microwave treatment of hempseed produced an increase in the oil chlorophyll pigment.

3.2.6. Oxidative stability of RS, SC1 and SC2 oils

Two methods were used to evaluate the oxidative stability of the oil fractions: thermal oxidation at 65 °C and Rancimat method.

Fig. 4 illustrates the change in peroxide value during heating at 65 °C for 60 days. The peroxide values of SC1 and SC2 oils at zero time of storage (fresh oil) were higher than those of RS oil (3.02, 8.06 and 0.64 mequiv. O_2/kg oil for SC1, SC2 and RS respectively). This could be explained by the effect of roasting and dehulling processes leading to higher content in primary oxidation products (Fig. 4).

The peroxide values obtained for RS and SC2 oils proceeded at a lower rate initially. This period of time is called the induction period (IP) or induction time (IT) (Nissiotis & Tasioula-Margari, 2002). The induction periods are 50 and 15 days of storage at 65 °C for RS and SC2 oils, respectively, with the peroxide values reaching approximately 50 mequiv. O_2/kg oil. However, the peroxide values of SC1 oil increased gradually during the whole storage period and exceeded approximately 50 mequiv. O_2/kg oil after 25 days. Consequently, RS oil shows the highest stability followed by SC1 and SC2 oils. These results were confirmed by the induction periods determined by rancimat method (Table 2) in which RS oil had a higher IP (28.2 h) then followed by SC1 (25.7 h) and SC2 oils (20.6 h). Thus, the dehulling and roasting processes decrease the stability of the oils extracted from SC1 and SC2. This can be explained by the higher primary oxidation products found in the fresh SC1 and SC2 oils.

The higher oxidative stability of RS oil could be attributed to endogenous antioxidants (lignans) together with tocopherols (Yoshida et al., 1995). The total phenol contents in RS oil found in this study (23.06 mg/kg) could be partly attributed to the high oxidative stability of the oil. Kamel-Eldin and Appelpvist (1994b) and Yoshida and Takagi (1997) reported that RS (*S. indicum*) oil contains

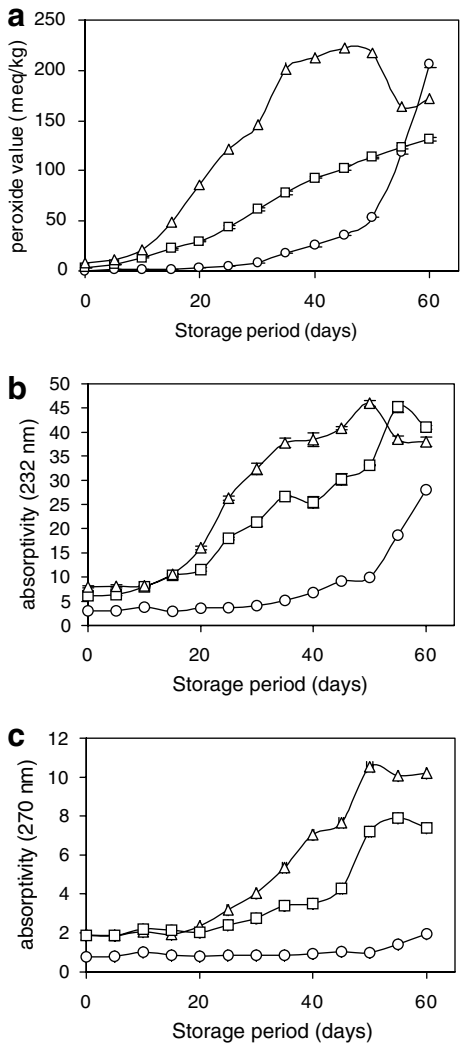

Fig. 4. Change in peroxide value (mequiv. O_2/kg oil) (a), in absorptivity at 232 (b) and at 270 (c) during 60 days at 65 °C of seeds and sesame coats oils. (○) Raw sesame seed, (□) sesame coat 1, (△) sesame coat 2.

between 392.6 and 663.68 mg/kg γ-tocopherol which is more potent antioxidant in oils.

The formation of hydroperoxides is accompanied by generation of conjugated diene measured by absorptivity at a wavelength of 232–234 nm. (Guillén & Ruiz, 2004; White, 1995). The hydroperoxide and the conjugated diene reflect the degree of formation of primary products of lipid oxidation (Guillén & Ruiz, 2004). Fig. 4b illustrates the evolution of the absorptivity at 232 during the storage period at 65 °C. The initial conjugated diene value of oil extracted from RS was lower than those of oils extracted from SC1 and SC2 (2.97, 6.15 and 8 for RS, SC1 and SC2, respectively). This confirms the effect of roasting and dehulling processes in generating the primary oxidation products. The coincidence in the changes in absorptivity at 232 nm with hydroperoxide formation was observed, and correlation coefficients of 0.997, 0.979 and 0.980 for RS, SC1 and SC2 respectively existed between peroxide value and absorptivity at 232 nm. Such correlations were observed in previous studies with a correlation coefficient of 0.944 in the case of sesame seed oil stored at 65 °C during 35 days (Abou-Gharbia et al., 1997).

The primary products of oxidation are not stable under heating and then they evolve to give secondary oxidation products that absorb at about 270 nm (Guillén & Ruiz, 2004; Vieira & Regitano d'Arce, 2001). The generation of these secondary products has as a consequence a role in the break-up of the acyl group chains as was suggested by Guillén and Ruiz (2004).

Fig. 4c shows the evolution of the absorptivity at 270 nm during accelerated condition at 65 °C. Absoptivity at 270 of RS, SC1 and SC2 oils presents a lower rate during 60, 45 and 20 days of accelerated storage respectively. This could also confirm that RS oil is the most stable oil during storage at 65 °C.

4. Conclusion

Considering the protein, fat, mineral and dietary fibre contents of the by-products of Halaweh manufacture, we can conclude they can be used as part of the nutritional requirement of animal feeds. These by-products of sesame seed processing industries could be considered as an excellent source of dietary fibre and may be used as a functional ingredient. This hypothesis will be supported by studies of the physical properties of these by-products.

The study of the oil fraction shows that the by-products, compared to the raw sesame seed, present a high content in free fatty acids, chlorophyll, polyphenols, sesamol, pigment and less oxidative stability.

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