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RESEARCH ARTICLE

COMPOSITION AND STABILITY OF TRADITIONAL PROCESSED SESAME OIL

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Abstract: This study aimed to investigate the physicochemical properties, oxidative stability and sensory quality of Walad (W) and Normal (N) sesame oils. 20 samples were collected from traditional mills (Assara) from five states of Sudan. Significant differences ($P \leq 0.05$) in physicochemical characteristics of N and W sesame oils were found. During storage for three days at 70ºC the N oil was less oxidized, producing fewer amounts of peroxides than W sesame oil through the five states. Using FTIR spectroscopy, the peak intensities of N and W oils of all states at frequency 723, 1163, 1745, 2852 and 2923 cm^{-1} was changed as time of storage increased. The overall acceptability of N and W oils showed significant differences ($P < 0.05$). Normal oils from the five states showed higher scores than that of Walad oils indicating their preference by the panelists.

Keywords: Sesame, Walad, Assara, Physicochemical, Oxidative Stability, FT-IR.

Introduction

Sesame (*Sesamum indicum l.)* is an old crop known to human, belonging to *Pedaliaccae* family. The seed contains 54% oil, 20% protein, 13.4% carbohydrate, 3.2% crude fiber and 3.7% ash (Nzikou *et al.*, 2009). China, India, Myanmar and Sudan are considered the world largest producers. The oil has industrial, cosmetics, insecticides, paints and varnishes uses (Warra, 2011). The sesame seeds are used for oil extraction and food purposes (Elleuch *et al.*, 2007). The high stability of sesame oil towards oxidation could be attributed to its high natural antioxidants (sesamol, sesamolin and sesamin) together with tocopherols (Ali *et al.*, 2007). The oil composition varies and depends on climatic conditions, soil type, plant maturity, variety and method of processing. The physicochemical properties of oils are directly related to their lipids and glyceride composition (Rahman *et al.*, 2007). One of the older methods of mechanical oil extraction is using to squeeze oil out of the seeds of oil crops, which may or may not be filtered afterwards. While the use of heat in combination with pressure has the potential to extract relatively more oil, it also has the potential to degrade the oil's quality and alter other desirable characteristics, like taste, odor, colour and texture (www.ehow.com). Kamal-Eldin *et al.* (1992) described the traditional camel-powered Ghani method as grinding sesame seeds (12 kg, oil content 53.1%) in a camel-powered Ghani with 0.5 L of water. Oil release was observed after 30 min, when the temperature of the mass was 41°C. After 40 min., 2.0 L of oil previously extracted (temperature 46°C) were added to assist extraction. Extraction was complete in 55-60 min., giving approximately 5 L of oil (temperature 50°C). The crushing system that presses oilseeds which developed in the form of a mortar-and-pestle arrangement powered by animals is commonly called ghani in India, and Assara in Sudan (Achaya, 1993). The device is widely used in the Sudan to crush sesame seeds, though its antiquity needs to be documented. Two types of sesame oil are obtained using Assara method, which are locally known as Normal and Walad sesame oil.

The applications of Fourier transform infrared (FT-IR) spectroscopy were expanded in food research and particularly in the study of edible oils and fats (Vlachos *et al.*, 2006). Many authors reported the application of FT-IR to determine stability of sunflower oil (Mariod *et al.*, 2012), soy lecithin-based emulsions (Whittinghill *et al.*, 2000) and it has been successfully used to analyze vegetable oil phospholipids (Nazi and Proctor, 1998).

This study aims to investigate the physicochemical properties, sensory quality and oxidative stability of Walad and Normal sesame oils processed using Assara traditional method and collected from different areas of Sudan.

Materials and methods

Materials

Source of samples

Twenty (Walad and Normal) sesame oil samples (approximately 500 ml each) were collected from five major sesame oil producing states in Sudan, four samples from each state. The states are Khartoum, Gadarif, Kordofan, Blue Nile and Sennar. The samples were collected from traditional mills (Assara) and packed in plastic bottles and stored at refrigerator temperature prior to analysis.

The chemicals and reagents

Glacial acetic acid, 0.1N KI, 0.1N sodium thiosulphate, starch solution, 95% ethanol, petroleum ether, 0.1N KOH, phenolphthalein, conc. Hydrochloric acid (sp.gr.1.19), acetonitrile, methanol, benzene, hexane, Tri-Floro-Acetic acid, cupric carbonate, NaCl, HCl, dichloromethane, and chloroform were supplied by Merck (Darmstadt, Germany). HPLC-grade water was obtained from Prime for Scientific Services, Khartoum, Sudan. All other inorganic chemicals and organic solvents were of reagent grade or higher.

Methods

Processing of Normal and Walad sesame oils

All sesame oil samples were obtained from camel traditional oil processing mills (Assara) in Al gadarif, Sennar, Blue Nile, Kordofan and Khartoum State. The camel tradition mill was composed of wood bowl (Assara) approximately 75 cm diameter and 100 cm length, woody stick (Walad), woody connector (Hawam) and a camel, milling was started with putting enough cleaned sesame seeds (approximately 50Kg) in the woody bowl then was pressed by the woody stick (Walad) which was driven by a blindfolded camel plodding round and round and used to *squeeze oil out* of *sesame seeds*, pressed seeds squeeze out oil into two layers the clean, light colour upper one which is known as Normal sesame oil and the lower layer that produced cloudy, dark colour and strong flavour known as Walad sesame oil (Umarabi, 2011). The physicochemical characteristics of sesame oil were determined; these include Refractive Index (RI), Viscosity, Colour, Specific Gravity, Peroxide Value (PV), and Acid Value (AV).

Physical characteristics of sesame oil

Refractive index

The Refractive index (RI) was determined by an Abbe 60 refractometer according to AOAC method (2000) at 30°C.

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Viscosity

The viscosity of the oil samples was recorded using an Ostwald-U-tube viscometer according to AOAC method (2000).

Colour

The colour intensity of oils was recorded using a Lovibond Tintometer as units of red, yellow and blue according to the AOCS method (1996).

Specific Gravity

The AOCS official method (1996) was followed for determination of the specific gravity of the oils at 25°C.

Chemical characteristics of sesame oil

Acid value (AV)

The AV of the oil samples was determined according to the AOCS method (1996).

Oxidative stability of sesame oil

To study the oxidative stability of sesame oil, 80 ml of oil were transferred in duplicate in 100 ml glass beakers (Lee *et al.*, 2004). The samples were stored in forced-draft air oven at 70°C for three days. The oxidative stability of oils was studied by the increase in peroxide values, differences in Fourier transformation infrared (FT-IR) between samples and sensory evaluation.

Peroxide value

The peroxide value in all samples was determined according to AOCS official methods (1996). In brief: Samples of 5.00g ± 0.05 g were weighed into a 250 ml Erlenmeyer flask with glass stopper and 30ml of acetic acid and chloroform solution (3:2) were added then swirled to dissolve the samples. Then 0.5ml of saturated KI solution was added using a suitable volumetric pipette. Allowed the solution to stand with occasional shaking for exactly 1min and then immediately 30 ml of distilled water added. Titration with 0.1N sodium thiosulfate was performed, with gradual addition and constant agitation. The titration has continued until the yellow iodine colour has almost disappeared. The titration has continued with constant agitation, especially near the end point, to liberate all of the iodine from the solvent layer. The thiosulfate solution was added until the blue color just disappeared. It was Conducted a blank titration which has not exceeded 0.1 ml of the 0.1 N sodium thiosulfate solutions.

Fourier transformation infrared (FT-IR) spectroscopy

The IR spectra were collected using a Vector 22 FT-IR spectrometer (8400S, Shimadzu, Japan) with OPUS software (version 3.1). A film of a small amount of oil (five drops) was placed on the ATR device, which was equipped with a Zn Se crystal (Van de Voort *et al.*, 1994). The spectra were obtained using 128 scans against the spectrum of the clean crystal. The range from 4,000 to 400 cm^{-1}, with a resolution of 4 cm^{-1}, was used to obtain spectral information. After each measurement, the ATR plate was carefully cleaned by wiping it with analytical grade acetone and dried with a soft tissue before it was filled with the next sample. Three spectra replicates were obtained for each sample.

Sensory evaluation test of sesame oil samples

Organoleptic assessment for overall acceptability of N and W sesame oils was done using a taste panel consisting of 20 semi-trained panelists.

The panelists were asked to evaluate the sesame oil samples for overall acceptability (appearance, colour and flavour). Five point hedonic scale, with a score of 5 for ‘like very much’, 4 for “like slightly”, 3 for “neither like nor dislike” 2 for “dislike slightly and 1 for ‘dislike very much’ was used to evaluate the degree of liking, for a particular quality attribute. A score of 3.0 or higher denotes that the attribute is generally acceptable (Yalegama *et al.*, 2007). Analysis of variance ($P< 0.05$) was performed on the sensory evaluation scores using the MINITAB statistical package (MINITAB Inc., State Collage, PA).

Statistical analysis

All the analyses were performed in duplicate, and the results were expressed as the mean ± standard deviation. The results were submitted to analysis of variance (ANOVA), and mean separations were performed using Tukey’s multiple range test ($P\leq 0.05$).

Results and discussion

Physical characteristics of Walad and Normal sesame oil

Refractive index. The refractive index (RI) which related to the molecular structure and degree of the oil unsaturation was determined in the studied samples that were collected from five states and the results are shown in Table (1). The lowest values of RI (1.471 and 1.472) were recorded in Walad samples from Algadarif and Kordofan states, respectively, the values for Normal samples ranged between (1.472-1.474), while that for Walad samples ranged between (1.471-1.474). A significant difference *($P \leq 0.05$)* for W and N oils from different states was observed, which might be due to variation in sesame type used. The RI of sesame oil at 40ºC was 1.465-1.469, according to Codex standards (internet website) which seems to be less than the present results due to type of oil.

Viscosity. Viscosity is a measure of resistance of a fluid to deform under shear stress. It is commonly perceived as thickness, or resistance to pouring. Viscosity describes a fluid's internal resistance to flow and may be thought of as a measure of fluid friction (Nzikou *et al*., 2009). Viscosity is an important thermo-physical property of industrial oil processes. It is also an important factor affecting the stability of oils (Nichols and Sanderson, 2003). Table (1) shows viscosity of two types (W and N) crude sesame at ambient temperature (34±5ºC). The highest levels recorded (24.71 and 24.68cp) in Khartoum state for samples N and samples W, respectively, while the lowest levels were recorded 17.98, and 17.95cp in Sennar state samples N and samples W, respectively. These results were in agreement with Murwan, (1994) who reported that the viscosity of sesame seed oil ranges between 18.90 - 26.43 centipoises at 32ºC. Generally W oil showed lower viscosity than N oil, the viscosity readings of N oil differ significantly *($P \leq 0.05$)* from state to other the values of viscosity ranges from 17.98 cp in Sennar state to 24.71cp in Khartoum state, in the same manner the viscosity values of W oil differ significantly *($P \leq 0.05$)* from 17.75 cp in Kordofan state to 24.68 cp in Khartoum state. From Table 1 it was clear that the same type of sesame oil differ significantly ($P \leq 0.05$) within the states. These results indicate that the viscosity of sesame oil depend on various factors such as location, soil type, and variety. There is a relationship between viscosity and temperatures, Mariod *et al*. (2009) studied viscosity of four Sudanese conventional oils including sesame oil and compared them with three unconventional ones and found that viscosity decreased with increase in temperature in all vegetable oils studied.

Specific Gravity. The specific gravity of W & N sesame oil samples from five states is shown in table (1), the specific gravity of N oil ranged between 0.9176 $g \cdot cm^{-3}$ in Kordofan samples and 0.9533 in Sennar there was significant difference in specific gravity of N oil in the five states and oil samples from Sennar state had the highest specific gravity value (0.9533) followed by Blue Nile (0.9192) and Algadarif (0.9185), respectively, the lowest specific gravity value was recorded by Kordofan samples (0.9176) followed by Khartoum samples (0.9183).

The specific gravity of W oil ranged between 0.9172 and 0.9535, these values differ significantly ($P \leq 0.05$) within the states, and also differ significantly at ($P \leq 0.05$) from N sesame oil sample within the five states. The values are in agreement with Codex (1999) sesame oil standards. The increase in specific gravity determined the purity of edible oils during processing and storage (Murwan, 1994). It was concluded that the specific gravity of sesame oil varying with the source and seeds which reflect the quality of sesame oil. These may be lipochromes originated from tissues or artifacts causes by degradation by thermal processing and treatment

Acid Value. The acid value (AV) is a common parameter in the specification of fats and oils. It is defined as the weight of KOH in mg needed to neutralize the organic acids present in one gram of oil and it is a measure of the free fatty acids present in the oil. An increment in the amount of FFA in a sample of oil or fat indicates hydrolysis of triglycerides. Table (1) shows the changes in acid value (AV) of N and W sesame oil from five states, the acid value of N sample ranged from 1.367 to 2.783 in the five states, while that of W samples ranged from 1.580 to 3.680. These results revealed that the acid value of W samples was higher than N samples. These results indicated that there was significant difference ($P \leq 0.05$) observed between N

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and W types of sesame oil within states, probably due to source of sesame seeds. These values were comparable to the results of Borchani, *et al*. (2010) who reported 1.64 as acid value of raw sesame oil extracted using hexane. Generally saying acid value of W sesame oil seem to be higher than N sesame oil indicating that its lower in quality might be due to the collection of different constituents during processing passed to W oil (e.g. drops of water ect). It is well known that increase of acid value usually considered to be one of the main parameter to use in evaluating the quality of oil especially the state of storage and heat. These results are indicated that, they are significantly different in acid value of different samples at ($P \leq 0.05$).

Peroxide Value. As shown in table (1) the changes in peroxide value (PV) of N and W sesame oils collected from five states recorded the highest level (22.55 meq/Kg oil) observed in W sesame oil of Blue Nile state, and the lowest level (0.665 meq/Kg oil) was observed in Algadarif state. While in N sesame oil the highest PV (16.92 meq/Kg oil) was recorded in Blue Nile state and the lowest value (0.8643) was recorded in Algadarif state. Generally the peroxide values of W sesame oil were higher than that of N sesame oil, and these values differ significantly ($P \leq 0.05$) within the five states.

In these results the variation in PV due to the difference in location of sesame seeds may refers to storage period of sesame seed and oil as well as the observed high fluctuation in PV for same type of oil from different states and less fluctuation for two types from the same state. The values are in agreement with 1.43-29.55 meq/Kg oil reported by Fad Elseed (2001) for oil extracted from sesame seed cultivars stored for 12 months at ambient temperature. While SSMO (2002) standards showed PV of unrefined sesame oil as 15 meq/Kg oil and refined sesame oil as10 meq/Kg.oil.

The colour readings of N and W sesame oils. Colour is measured by the Lovibond tintometer, usually in red and yellow terms. Most finished edible oils are less than 10 yellow and 2.5 red, with high-grade shortenings being less than 1.0 red (Fengxia *et al*., 2001). Table 2 show Tintometer colour values of Normal and Walad sesame oils processed using Assara traditional method. Walad sesame oil exhibited higher yellow and red values than Normal one through the five states. This means that Walad sesame oil was lighter, more red and yellow-coloured than Normal sesame oil. Elleuch *et al.* (2007) studied colour of the oil extracted from raw sesame and its byproducts, and reported that dehulling as well as roasting causes an increase in the dark, red and yellow units of colour. The colour formation in sesame oil during heat treatment is probably due to non enzymatic browning (Maillard reaction) that occurs during roasting.

Oxidative stability of Normal and Walad oils processed using Assara traditional method during storage

Peroxide value

The oxidative stability of N and W sesame oils after storage at 70ºC for three days was monitored using peroxide value and FTIR analysis. Table 3 shows the changes in peroxide values in N and W oils during storage at 70ºC. Peroxide value in W sesame oil in Khartoum state increased significantly ($P < 0.05$), from 7.0 in control to 14.1 meq O_2 per kg oil after three days of storage at 70ºC. Peroxide values of the oils obtained from N sesame oil in Khartoum state increased from 2.0 at zero time of storage (fresh oil) to 3.1, 4.2 and 5.4 meq O_2 per kg oil after 3 days of storage at 70ºC, respectively. The highest peroxide value (19.0±2.4) at zero time of storage (fresh oil) in W sesame oil through the five states was recorded in Blue Nile state while the lowest was recorded as 7.0 ±1.1 in Khartoum state. These figures increased to 43.0±2.8 meq O_2 per kg oil in Blue Nile state and 14.1± 1.4 meq O_2 per kg oil in Khartoum state. The highest peroxide value (13.3 ±1.3) at day zero in N sesame oil through the five states was recorded in Sennar state while the lowest was recorded as 2.0± 0.1 in Khartoum state. These figures increased to 28.2± 0.9 meq O_2 per kg oil in Blue Nile state and 5.4± 0.4 meq O_2 per kg oil in Khartoum state. From these results it is clear that N sesame oil was less oxidized producing less amount of peroxides than W sesame oil through the five states, this due to some aqueous, proteinous materials from the raw seeds. In fact, the peroxide values of N sesame oil reached approximately 5.4, 6.0, 16.3, 17.0 and 28. meq O_2 per kg oil for Khartoum, Algadarif, Kordofan, Blue Nile and Sennar states, respectively. Borchani *et al*., (2010) and Elleuch *et al*., (2007) reported 13.83, and

44.89 meq O_2 per kg oil for raw sesame oil and sesame paste.

Fourier Transformation Infrared (FTIR) Spectroscopy

To study the oxidative stability of W and N sesame oils, the two oils collected from five Sudanese states were stored in forced-drafted air oven at 70ºC for 3 days (72 h) then the changes in the FT-IR spectra were observed. FTIR has few advantages over classical parameters in the measurements of oil stability. With only a small drop of oil and a couple of minutes to prepare the sample, the infrared spectrum collected can give information on different functional groups present. Also, minimal errors in the determination of frequency and absorbency can be expected because it is fully computerized (Guillen & Cabo, 2004). The FTIR spectra of N and W sesame oils showed the typical characteristic absorption bands for other vegetable oils e.g. safflower oil (Mariod *et al.*, 2012), virgin coconut oil (Rohman *et al.*, 2011), who reported the bands of interest in safflower and virgin coconut oil are absorption peaks at 3100–2800 and 1800–700 cm)[1]. The band assignments and their respective mode of vibration of N and W sesame oils from Khartoum state are shown in Fig. 1A and 1B. From these figures it is clear that, there were six visible peaks at frequencies of 3006, 2923, 2852, 1745, 1163 and 723 cm^{-1} have been investigated. The intensity at frequency 723, 1163, 1745, 2852 and 2923 cm^{-1} decreased, while at frequency 3006 cm^{-1} remained constant. These bands showed change in absorbance with times of storage. In N and W sesame oils from Khartoum state the bands at 2923, 2854$cm^{-1}$and the shoulder at 3006 cm^{-1} their intensity and width increased as the times of storage increased from zero to day three, this increase was faster in W sesame oil than that of N oil. The Same results were observed in N and W sesame oils from the other four states (data not shown) as during heating storage, the prominent peak change observed was at frequency of 1747 cm^{-1}, which corresponded to the carbonylic compounds (esters, aldehydes, ketones, lactones) resulted from the hydroperoxide decompositions during oven heating. Carbonylic compounds are the major secondary products during hydroperoxide decomposition (Smith *et al.*, 2005). Intensities at frequencies of 2852, 1745, 1163 and 723 cm^{-1} were relatively changed for all samples of the five Sudanese states evaluated (data not shown).

Sensory evaluation of sesame oil samples using Hedonic test

The sensory evaluation carried out for the degree of liking of appearance, colour and flavour of N and W oil samples showed significant differences ($P< 0.05$) (Table 4). Normal sesame oils from the five states showed higher scores than that of Walad sesame oils indicating their preference by the panelists in overall acceptability. Again the Normal sesame oils had scores greater than 3 for the overall acceptability indicating their acceptance by the panelists (Table 4). Both N and W sesame oils showed scores greater than three up to the second day of storage and showed scores lower than 3 indicating that these oils were not accepted by the panelists in term of their colour and flavour. Between states Normal sesame oil collected from Khartoum state showed greater scores in overall acceptability than Normal oils collected from other states followed by Blue Nile and Sennar states. Oro *et al.* (2009) showed that the sensory characteristics of pecan nut oil were unaltered for up to 60 days of storage at room temperature.

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