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Original Research Article

Effect of Roasting Time and Temperature on the Biochemical Contents, Phytochemical Properties and Antioxidant Activity of *Sesamum indicum* L. Seeds

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ABSTRACT

Roasting is a dry heat processing technique used to develop sensory properties of food which has caught the interest of customers. Roasted seeds of sesame is one of the most recommended oilseeds in the world thanks to its sensorial, medicinal and nutritional benefits. However, the choice of effective roasting conditions is important for the sensorial parameters and for the biological potentials of sesame seeds, as well. In this study, the impact of oven temperature and roasting time on sesame quality was investigated. A total of 16 different roasting conditions were used. Sesame seeds were roasted at the following temperatures: 130°C, 140°C, 150°C and 160°C. During this process, samples were taken and analyzed at different time intervals (40 min, 60 min, 90 min, and 120 min). Both the roasting temperature and time significantly ($P<0.05$) affected the quality characteristics of sesame seeds. The amounts of oil yield, total proteins, total sugars, total phenolic and flavonoids content, total antioxidant activity and DPPH radical scavenging were increased by increasing the treatment. Roasting sesame seeds at 140°C/40min, gave a significantly higher content of oil yield (52.2%), proteins (20.95%), sugars (29.62%), flavonoids content (0.12 mg/g) and total phenolic content (1.2 mg/g) in comparison to the other roasting conditions. However, beyond this treatment, these contents decreased significantly. Based on these results, time and temperature are undoubtedly important factors to consider in the roasting process of sesame.

Keywords: *Sesamum indicum*, Roasting, Phytochemical content, Antioxidant activity

Introduction

Sesamum indicum L. (Sesame) is an annual plant belonging to *Pedaliaceae* family. Sesame seeds have an important place in human food and used as ingredients in several food products.[1] Archaeological studies showed that sesame culture dates back to 5.500 BC.[2] Nowadays, it represents one of the most economically important crops. In 2010, the world area of this crop was 10 245 246 ha, and Asia and Africa were the main producers with 2 489 518 and 1316 690 tons respectively. [3]

Chemical composition of sesame seeds showed the presence of proteins (20-27%)with a predominance of Globulin (67.3%), and oil (25-32%) with a good percentage of unsaturated fatty acids (80%) as oleic and linoleic acids.[4]

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Several researchers highlighted the interesting biological activities of sesame, especially, antimutagenic property,[5] analgesic effect,[6] antioxidant power,[7] anti-inflammatory activity,[8] and ability to reduce the plasma cholesterol,[9] Many authors related the biological potential of sesame seeds to their phenolic compounds.[10,11]

In the oil industry, roasting is a crucial procedure that causes significant physical and chemical modifications.[12] Primarily, roasting is a dry heat treatment which is employed for cooking and preparing food materials to improve their digestibility and sensory aspect.[13] This technique depends on heating (90–260 °C) to cook or gelatinize, expand/pop/puff the products evenly and transform them into a more pleasant, appetizing and attractive form.[14,15] Historically, roasting is known as a widely applied mechanism in the food industry. This process is widely used in food industry, particularly with coffee.[16] The roasting process is divided into three distinct stages: drying, roasting, color aroma and flavor generation.[17]

Many studies have been conducted to evaluate the roasting effects on the quality and stability of various foods.[18,19] According to earlier research, roasting sets off a series of metabolic processes in food, [20] that result in a variety of chemicals that can have both positive and negative consequences.[21,22] Generally, two major factors have a significant effect on seeds during roasting: time and temperature.[23]

Several studies have highlighted the importance of determining the best duration, and temperature for roasting in order to improve the organoleptic properties of seeds without altering their nutritional quality.[24,25] Therefore, understanding the roasting process and how it affects foods is necessary for planning particular roasting operations.

To date, a comprehensive evaluation of roasting and the resulting changes in oilseeds, especially sesame, has not been reported. However, the aim of our study was to evaluate the effect of roasting temperature and time on the biochemical composition and antioxidant activity of Moroccan sesame seeds.

Material and Methods

Plant material and roasting conditions

Moroccan sesame samples were collected in October 2015 from Taounate, an agricultural province situated in North Morocco at 34°33' North, 4°39' West. The plant was identified by Rabat Scientific Institute under voucher number 33336. 16 roasting conditions were used. The sesame seeds were roasted at 130°C, 140°C, 150°C and 160°C, and in a time of 40 min, 60 min, 90 min, and 120 min for each temperature. After roasting, sesame seeds were chilled at room temperature (37°C), and stored in polyethylene plastic bags at 4°C.

Biochemical composition

Total oil content

Sesame oil was extracted using unroasted and roasted seeds. Thus, two grams of each samples were subjected to 7h of extraction using hexane solvent in Soxhlet apparatus.[26] The solvent was removed under reduced pressure, and the resulting oil was stored at 4°C in obscurity prior to use.

Iodine value (IV)

Iodine value can be used to measure the degree of unsaturation of oils and fats. In this study, the iodine value of sesame seeds oil was determined according to methods and recommended practices of the AOCS.[27] Firstly, the oil was diluted with chloroform and iodine bromide solution. After incubation (Model = BINDER GmbH, made in Morocco) in obscurity and at temperature for 30 min, potassium iodide and water were added. Finally, the mixture was titrated with sodium thiosulphate.

Peroxide value (PV)

Peroxide value (PV) states the milliequivalents of peroxide oxygen combined in a kilogram of oil and able, under testing, to liberate iodine from potassium iodide; the iodine is next estimated using a standard sodium–thiosulfate solution in this way, five grams of sesame oil was added to 30 mL of acetic acid and isooctane. After that, 0.5 mL of KI was added too. Finally, our solution was titrated with a standard volumetric thiosulfate solution.[27]

Total protein content

Total proteins were determined using the method described by Khalid et al. [28] , and based on the nitrogen concentration determined by Kjeldahl method described by McKenzie and Wallace.[29] Briefly, 1g of oil were mixed with 8 mL of H_2SO_4 into Kjeldahl flask. In the presence of a catalyst (potassium sulfate, copper sulfate) until the color of the mixture changed to a greenish color. Then, to distill the sample, a volume of 15 mL of NaOH 30% was added using a semi-automatic distillation system (Model = Orto Alresa , made in Espagne). Then, boric solution (4%) was used to collect the produced nitrogen NH_3. The titration was conducted with H_2SO_4 in the presence of mixed indicator solution (bromocresol green and methyl red). The following equation (1) was used to estimate the total Nitrogen concentration:

$$\text{Total protein content (\%)} = 6.25 \times [V\ (H_2SO_4) \times N\ (H_2SO_4) \times 0.014 \times SW] \quad (1)$$

With:

V (H_2SO_4): volume of H_2SO_4 used for titration

N (H_2SO_4): the normality of H_2SO_4 used for titration

SW: sample dry weight

Total soluble sugars

To extract the soluble sugars, 100 mg of unroasted and roasted seeds were mixed with 4 mL of Water : Ethanol (2:8) solvent. The mixture was then, placed in a water bath (Model = JB Academy, made in Morroco) at 80°C for 30 min**.** Next, the mixture was centrifuged (Model = TDL- 40B, made in Morroco), and the supernatant was collected for sugars content using Anthrone reagent 0.2 % (Anthrone : Sulfuric acid (w/v)).

The absorbance was determined at 625 nm by UV visible spectrophotometer (Model = BK-V1600, made in Morocco). Soluble sugars were calculated using a calibration curve based on the standard glucose. Results were expressed as mg of glucose equivalent (GAE)/g (GAE: Gallic acid equivalent while GE: glucose equivalent. Check and revise) of extract.[30]

Phytochemical and antioxidant activity

Extracts preparation

Extraction of sesame seeds were carried out using maceration method. Methanol : water (70:30 v/v) was added, with constant shaking for 8 hours, to ground seeds of each treatment. The extracts were then filtered using Whatman filter paper and concentrated under reduced pressure. The extracts were then stored at 4 °C for further experiments.

Total Phenolic Content

Total phenolic content of each hydro-methanolic extract was determined according to the method described by Mssillou et al. [31], with minor changes. Briefly, the appropriate dilution of each extract was mixed with 1 mL of the diluted Folin–Ciocalteu reagent. Then, 2 mL of 5% sodium carbonate solution was added. After incubation for 2 hours at room temperature, the absorbance was measured at 750 nm. Results are expressed as Gallic Acid Equivalents (GAE).

Total Flavonoids Content

To evaluate the total flavonoids content, 0.1ml of each hydro-methanolic extracts were mixed with aluminum chloride methanolic solution (10%) and 0.1ml of sodium acetate. After incubation, the absorbance was measured at 415nm. Results are expressed as Quercetin equivalents.[32]

DPPH scavenging activity

The effect of each extract on the 1,1-diphenyl-2-picrylhydrazyl (DPPH) radical scavenging was estimated according to the method of Molyneux,[33] with minor modifications. Different concentrations of hydro-methanolic extracts were added to the DPPH solution (0.5 mM), and then the mixture was incubated at room temperature for 30 min. The absorbance of the solution was measured at 517 nm. Ascorbic acid was used as a positive control. The proportion of the DPPH radical scavenging is calculated using equation (2):

$$\text{\% inhibition of DPPH radical} = [(Ac - Ae)/Ac)] * 100 \quad (2)$$

With:

Ac: Absorbance of the control

Ae: Absorbance of the extract.

The % inhibition of DPPH radical was then used to calculate IC_{50}, which is the anti-radical concentration required to cause 50% of inhibition.

Total Antioxidant Capacity (TAC)

Total antioxidant capacity was carried out using the phosphomolybdenum method as described by Prieto et *al*., 1999 [34]. The tubes, containing a mixture of each hydro-methanolic extract and reaction solution (0.6 M sulfuric acid, 28mM sodium, and 4 mM of ammonium molybdate), were incubated at 95°C for 90 min. After the cooling process, the solution absorbance was measured at 695 nm. The antioxidant activity was expressed as ascorbic acid equivalents.

Statistical analysis

Statistical analysis was performed using SPSS statistical software (version 20, IBM SPSS Statistics 20, New York, NY, USA). Roasting treatments were done in triplicate and values expressed were means ± standard deviation. All analyses were carried out using a completely randomized design. At $P<0.05$, Duncan test was used for means

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separation, when the effect of roasting treatments was revealed to be significant

Results and Discussion

Total oil content

The total oil content of unroasted and roasted seeds is shown in Figure 1. The yield of unroasted seeds was 47.2%. This value was slightly lower than the oil yield (52-2.5 %) reported by Gharby et al. [35]. The observed difference in oil content could be due to the extraction conditions (temperature, time and solvent used) [36].

The oil yield was significantly influenced ($p < 0.5$) by roasting temperature and time. The highest value (52.2%) occurred at a temperature of 140°C and a time of 90 min. Similar results were reported in the apricot kernel [37]. The increase in the oil content of roasted oilseeds may be explained by a change in the porosity of the cell wall after the roasting process, allowing the oil to be released [38,39].

Histograms represent mean value of oil content. Error bars represent standard error and letters (a, b, c, d, e, f and g) denote significant differences according to Duncan's test, ($p < 0.05$) is the significance level.

Peroxide value

Peroxide value (PV) of oil from unroasted and roasted sesame seeds is shown in Figure 2. The PV of unroasted oil seeds was determined as 2.48 meq O_2/kg. Under the 150°C/0min condition , the PV was reported lower than the recommended PV in edible oils (10 meqO_2/kg) [38]. These results suggest that the oil stability observed in roasted sesame seeds is probably due to the presence of a large range of antioxidants like sesamol and sesamolin [38,40]. However, the PV increased gradually with high temperature; in fact, the highest value was recorded at 160°C/120min with 20.77 meq O_2/kg. Warra et *al*., 2011 [39] showed that the PV is correlated with oil oxidation during storage. Similar results have also been reported by Mazaheri et *al*., 2019 [41].

Iodine index

The iodine index is another indicator that provides information on the degree of instability of oils. It is used to determine the rate of unsaturated fatty acid. As shown in Figure 3, the iodine value of raw sesame seed oil is found at 101.45 mg/100 g. This value is lower than that of sunflower oil (130 mg/100 g), but higher than that of olive oil (90.2 mg/100 g) [42]. Excessive roasting caused an important diminution of the iodine value recorded in roasted oil seeds at 160°C/120min with 97.8 mg/100 g. Similar results were reported by Rizki et al. [43].

Total protein content

The effect of different roasting conditions on the protein content of sesame seeds is given in Figure 4. Total protein content was significantly increased with the increase in roasting temperature and time. The highest value was recorded at 140°C/40min with 20.95%. Hossain et *al*., 2012[44] explained that as the roasting temperature increase, molecular bonds are degraded and the solubility of the molecules in the cell matrix increases, resulting in higher yields.

Results showed also that the protein rate decreased when sesame seeds were roasted at higher temperatures and for longer periods (until 17.03% for 160°C/120min.). Several previous studies showed that temperature is responsible for the denaturation of proteins; furthermore many amino acids like methionine are degradable.[45] Aspartic acid, glutamic acid and phenylalanine act directly to give volatile aromatic compounds like pyrazine, which is retained by the oily fraction.[46]

Total soluble sugars

The sugars yield of roasted and unroasted sesame seeds is shown in Figure 5. Total soluble sugars of roasted sesame seeds were remarkably increased by increasing the time and temperature up to 140°C/40min, with a total of 29.62%. Then it reduced after increasing both roasting time and temperature to 160°C/120 min, with a significant reduction of 67.32%.

This decrease can be explained by the degradation of sugar molecules in Maillard reactions during the high-temperature roasting process to produce flavor components and a yellow-brown color .[47]

Phenolic and flavonoids content

The phenolic and flavonoids contents as affected by roasting are given in Figure 6 and Figure 7. As shown, the values were increased significantly ($P < 0.05$) after the sesame seeds were roasted. The value in unroasted samples was 0.1 mg/g and 1.13mg/g for flavonoids and phenolic content respectively, and it was increased by 16.66% and 7.89% when sesame seeds were roasted for 160°C/120min and 140°C/40min respectively. However, the flavonoid and polyphenol content fell to 30% and 35.39% respectively when heated for prolonged periods at 160°C for 120 minutes.

The high rate of phenolic and flavonoids content during roasting may be due to the production of other phenolic compounds.[48] These results agree with Wang et *al*., 2009 [49] who found that the polyphenols rate is high when extracted from roasted seeds at moderate temperatures; but according to the same study, roasting at high temperatures for a long time can cause a reduction in phenolic compounds. These results suggest that the phenolic content present in sesame seeds may be thermolabile.[50]

Total antioxidant activity (TAC)

Many studies have revealed the total antioxidant capacity of plant extracts in the prevention of oxidative stress. [51,52] The TAC is based on green phosphomolybdenum complex formation. In fact, at acidic pH, Mo (V) is reduced to Mo (VI). Figure 8 shows the effect of roasting at different times and temperatures on total antioxidant activity. Analysis of the results revealed a low antioxidant potential for all treatments compared with temperatures of 130°C and 140°C, which showed an interesting total antioxidant activity of 12.95% and 11.82% compared with unroasted seeds.

Several previous studies showed a strong and positive correlation between the quantity of phenolic compounds and antioxidant activity.[53,54] Thus, a reduction in phenolic compounds could reduce the plant antioxidant capacity, as clearly shown in this study. On the other hand, Woffenden et al. [55] explained the increase in antioxidant activity by the formation of neo-formed antioxidant and non-enzymatic products like melanoid.

DPPH scavenging activity

DPPH scavenging activity is the most commonly used method for the estimation of antioxidant activities. It is indicated by the neutralization of free radicals (DPPH), which are purple-colored [56]. In this study, all sesame extracts were able to reduce the stable DPPH radical, with IC50 values ranging from 1.63 to 2.44 µg/mL (Fig. 9). DPPH scavenging activity was gradually increased with the increase in roasting conditions. Besides, the hydro-methanolic extract of roasted sesame seeds treated at 140°C/40min and 150°C/40 min showed the highest antioxidant activity (IC50 =2.44 µg/mL).

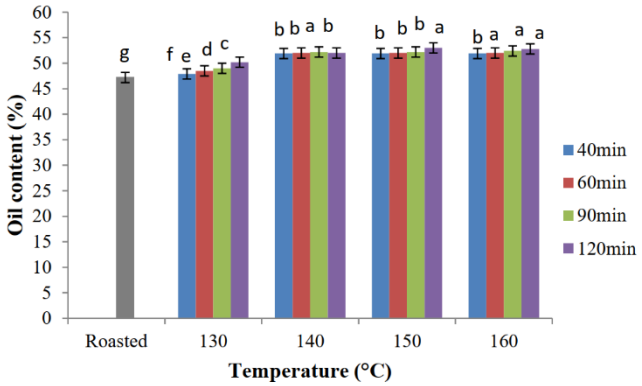

Figure 1: Effect of roasting temperature and time on oil content of sesame seeds

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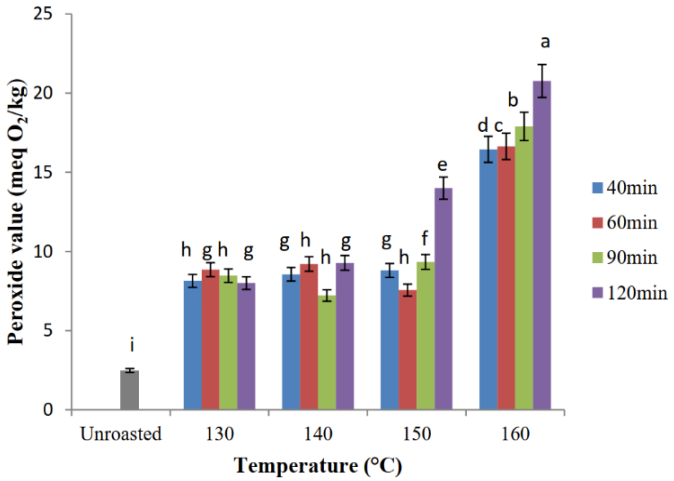

Figure 2: Effect of roasting temperature and time on peroxide value of sesame seeds oil

Histograms represent mean value of oil content. Error bars represent standard error and letters (a, b, c, d, e, f and g) denote significant differences according to Duncan's test, ($p < 0.05$) is the significance level.

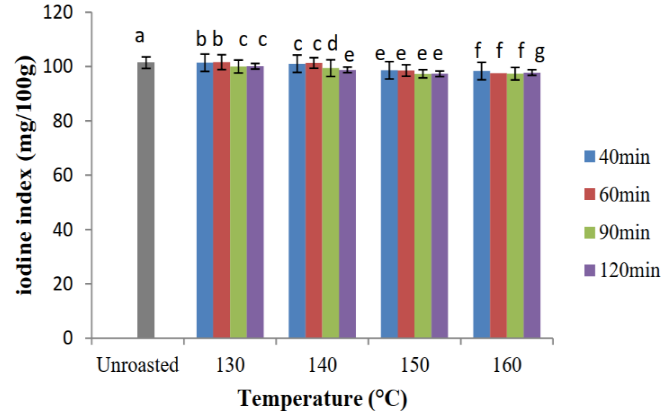

Figure 3: Effect of roasting temperature and time on iodine index of sesame seeds oil

Histograms represent mean value of oil content. Error bars represent standard error and letters (a, b, c, d, e, f and g) denote significant differences according to Duncan's test, ($p < 0.05$) is the significance level.

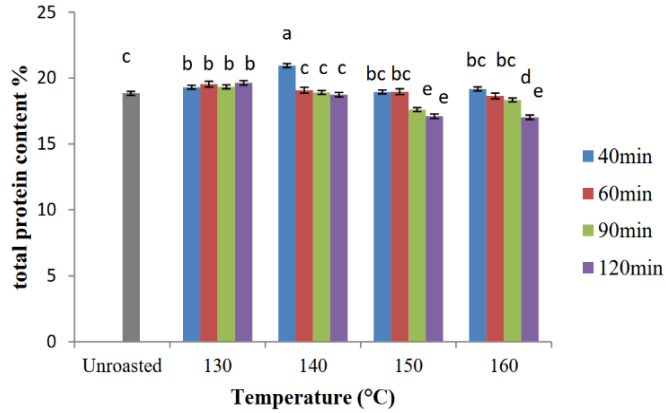

Figure 4: Effect of roasting temperature and time on total protein content of sesame seeds

Histograms represent mean value of oil content. Error bars represent standard error and letters (a, b, c, d, e, f and g) denote significant differences according to Duncan's test, ($p < 0.05$) is the significance level.

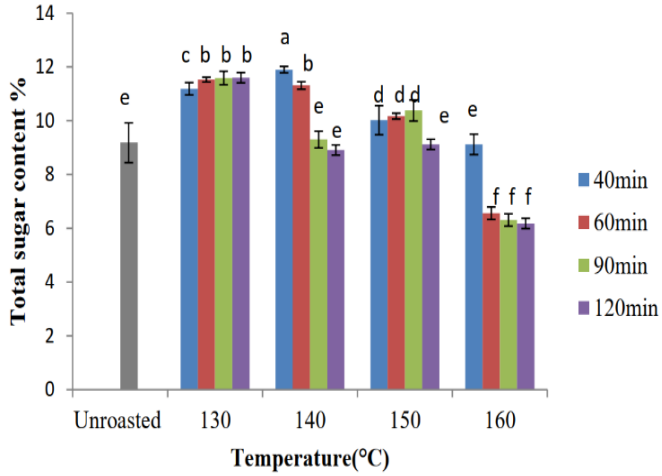

Figure 5: Effect of roasting temperature and time on total sugar content of sesame seeds

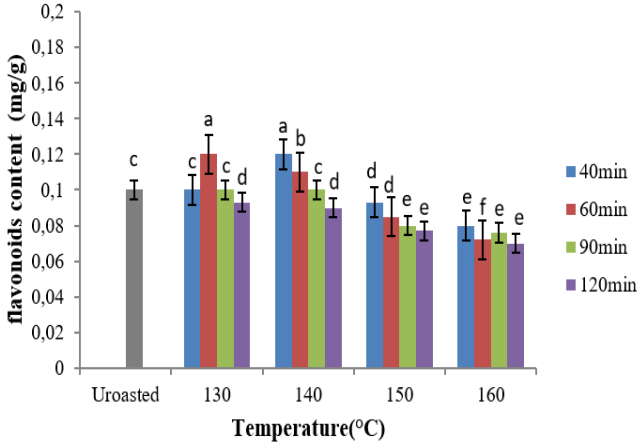

Figure 6: Effect of roasting temperature and time on flavonoids content of sesame seeds

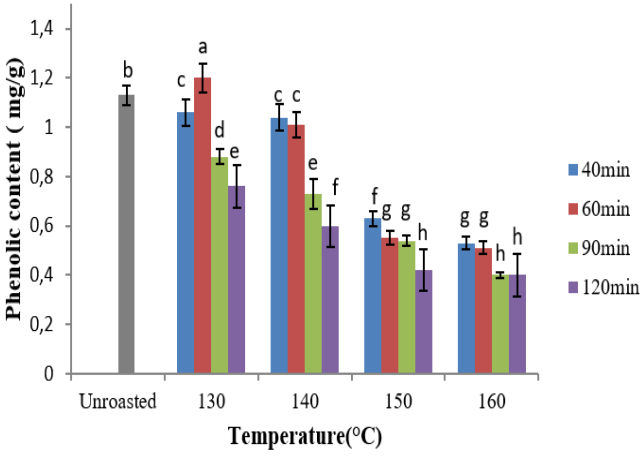

Figure 7: Effect of roasting temperature and time on phenolic content of sesame seeds

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According to Xu *et al.*[57] , after roasting process, DPPH scavenging activity was increased by 82.2% in oats. Siddhuraju et al. [58] showed that higher antioxidant power of roasting process could be due to the formation of Maillard products such as pyrazines which could also act as antioxidants. However, in the case of exposure to high temperatures (especially 160°C), DPPH scavenging activity decreased significantly ($p < 0.5$) until 1.63 µg/mL. This result corroborates with previous researches, especially for pistachios and rapeseeds (citations needed) . Randhir et al. [61] reported that the decrease in antioxidant activity after high temperature roasting may be explained by the degradation of phenolic content.

Conclusion

This work describes changes in certain quality parameters of sesame seeds, depending on the roasting conditions. The total oil content, the total protein content, the total soluble sugars, the peroxide value, the iodine index and the antioxidant activity were choses as parameters that specifically determine seeds quality. The wide range of roasting conditions was applied to obtain more information about the dynamics of changes in the studied components, which recorded the best values at the moderate temperatures. However, the higher roasting time and temperature showed a damaging effect on the tested parameters of sesame seeds. Based on the findings of our study, sesame seeds which are used worldwide, can be beneficial in their health promoting effects when roasted at a moderate temperature and time.

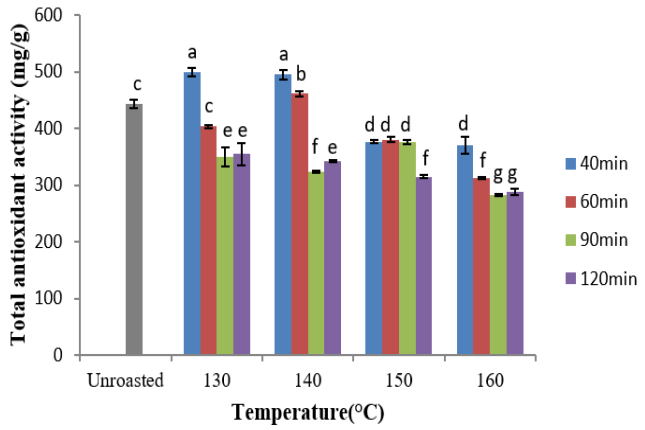

Figure 8: Effect of roasting temperature and time on total antioxidant activity of sesame seeds
Histograms represent mean value of oil content. Error bars represent standard error and letters (a, b, c, d, e, f and g) denote significant differences according to Duncan's test, ($p < 0.05$) is the significance level.

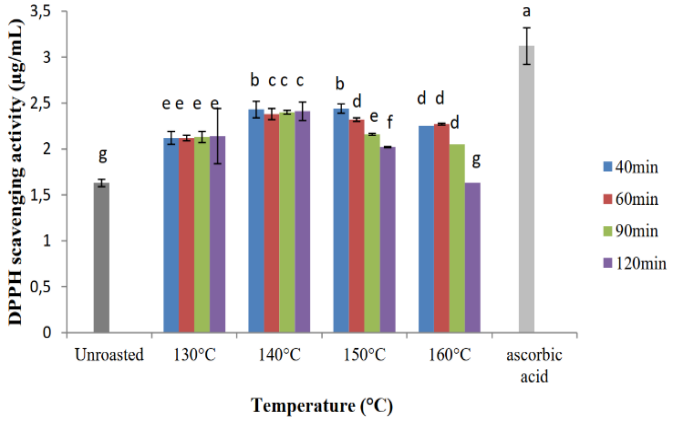

Figure 9: Effect of roasting temperature and time on DPPH scavenging activity of sesame seeds

Conflict of Interest

The authors declare no conflict of interest.

Authors' Declaration

The authors hereby declare that the work presented in this article is original and that any liability for claims relating to the content of this article will be borne by them.

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