PAPER

EFFECT OF INFRARED THERMAL PRE-TREATMENT OF SESAME SEEDS (*SESAMUM INDICUM* L.) ON OIL YIELD AND QUALITY

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ABSTRACT

The aim of the study was to determine the effect of thermal infrared treatment of Indian sesame seeds (*Sesamum indicum* L.) on the yield and quality of extracted oil. The infrared radiation treatment of the seeds was applied for 30 s, 60 s, 90 s, 120 s, and 150 s at a temperature of 180ºC. The mean moisture and fat content in the seeds was 7,46 and 45,61%, respectively. The extracted oil was assessed in terms of the hydrolysis degree (AV), primary oxidation degree (PV), oxidative stability in the Rancimat test, and content of carotenoid and chlorophyll pigments.
The study has shown that the infrared thermal treatment of sesame seeds contributed to an increase in the oil extraction efficiency, compared to the control sample. The highest increase in the oil yield was found in the case of seeds heated for 120 s. Simultaneously, there was a reduction of the quality of these oils accompanied by increased oxidative stability. The induction time in oils extracted from infrared-treated seeds increased gradually together with the increase in the length of the heating process. The longest induction time (9,53 h) was noted in sesame oil obtained from seeds heated for 150 s.

Keywords: antioxidant additives, acid value, peroxide value, thermal infrared treatment, oxidative stability.

1. INTRODUCTION

Freshly pressed and non-refined vegetable oil is characterised by a delicate flavour and aroma typical for the seeds used in the process. It should not have an unpleasant bitter taste, as this may imply that the oil is not fresh. Such oils are biologically active, because they are pressed at low temperatures. Hence, they are also referred to as 'virgin', which means that they have the same chemical composition after extrusion as that in the seeds of plants from which they originate. These oils are not supplemented with pigments, antioxidants, and preservatives and are not devoid of phospholipids or tocopherols (vitamin E) as is the case of the common refined edible oils (PALA, 2001).
Cold-pressed oils contain mainly highly active antioxidants (tocopherols, polyphenols, carotenoids, feldspar), polyunsaturated fatty acids from the n-3 and n-6 groups, and bioactive sterols (ROSZKOWSKA *et al*., 2014).
The method of oil extraction with the use of a screw press is quite common. The method has been widely popularised as a simple and ecologically friendly technology that does not require excessive energy costs and investment expenditures. Unfortunately, there are some limitations to the method, e.g. low yields of the process (considerable oil content left in the pomace) and difficulties in achieving a stable quality of the product. Investigations have evidenced that the process of fragmentation or heating the seeds prior to oil extraction increases the efficiency of the pressing process (GUPTA, 2011).
A very important step in the oil extraction process is the hydrothermal pre-treatment of oil-bearing plants. In the oil industry, pre-treatment (T 30 - 40°C) is applied to achieve a homogenous temperature and humidity of the seed mass and to ensure a proper fragmentation process. In turn, the basic conditioning (roasting) of the seed pulp (T 80 - 100°C) is applied to facilitate fat extraction and to increase the oil yield (GUPTA, 2011).
Currently, investigations are being carried out to assess the potential of other alternative seed pre-treatment methods that are more time and energy efficient. An example of such methods is the infrared thermal treatment technique, which provides a higher temperature (over 160°C) within a shorter time than the conventional convection heating (AZADMARD-DAMIRCHI *et al.,* 2011).
Infrared radiation penetrates and heats the product inside, thereby leading to partial evaporation of water from the seed interior accompanied by an increase in intracellular pressure. The disintegration of the cellular structure expands the surface area of the oil release thus contributing to enhancement of extraction yields (AZADMARD-DAMIRCHI *et al*., 2011, YANG *et al.,* 2013).
The infrared thermal treatment of seeds applied prior to the pressing process not only increases the yield but also shortens the heating time, which is accompanied by reduced energy consumption (AZADMARD-DAMIRCHI *et al.*, 2011).
As shown by literature data, the pressing method and seed pre-treatment exert a significant effect on the quality, nutritional value, and oxidative stability of extracted oil (NIU *et al.*, 2013, YANG *et al.*, 2013).
The range of qualitative changes in such oils depends both on the pre-treatment and extraction parameters and on the species of pressed seeds. Therefore, the aim of the study was to determine the effect of infrared thermal treatment of Indian sesame seeds (*Sesamum indicum* L.) on the yield and quality of extracted oil.

2. MATERIALS AND METHODS

The research was conducted on Indian sesame seeds (*Sesamum indicum* L.) harvested in 2016 and oil extracted from the seeds. To balance the humidity of the seeds, the material was stored in a glass jar without a lid at room temperature (20±2˚C) for 14 days.
The infrared thermal treatment of the seeds was applied for 30, 60, 90, 120, and 150 s at a temperature of 180ºC. The choice of the temperature range was based on analysis of previous investigations conducted by ANDREJKO *et al.* (2011).
The seeds were heated with the use of an original laboratory device for thermal treatment of bulk plant materials. The device is equipped with two heating heads with four 400 W ECS-1 radiators manufactured by ELCER. These temperature radiators supplied by electricity (230 V) have a small fraction of visible radiation (dark radiators) in the spectrum and heat all plane points uniformly (plane radiators). The average temperature of the filament is approx. 500°C and the wavelength is λ = 2,5-3,0 μm. The belt conveyor carrying the research material is powered by a DC motor equipped with a voltage regulator facilitating smooth adjustment of the belt travel speed in the range from $5 \cdot 10^{-3}$ m·s^{-1} to $7 \cdot 10^{-2}$ m·s^{-1} (the time that the seeds stay in the heating zone ranges from 15 to 200 s, respectively).
After cooling, the seeds were pressed in a DUO screw press manufactured by Farmet (Czech Republic) with a capacity of 18-25 kg·h^{-1}, motor power 2,2 kW, and a screw speed of 1500 r·min^{-1}. A 0,4 kg seed batch was pressed with the use of a 10-mm diameter nozzle. Before the process, the screw press was heated to a temperature of 50°C, which was measured with an Ama-digit thermometer. After the pressing process, the oil was left for natural sedimentation for 5 days in refrigeration conditions (T 10±1ºC). Next, the oil was analysed.
The oil extraction efficiency was calculated based on the weight of extracted oil, the weight of the seed sample, and the percentage content of oil determined in the seeds. The extraction efficiency "W" was calculated with formula (1) (ROTKIEWICZ *et al.*, 2002):

$$W = \frac{m_{ol}}{Z_{ol}} \cdot 100 \ [\%]$$

Where: m_{ol} - mass of extruded oil, kg, Z_{ol} - mass of oil masa contained in seeds during pressing, kg.
The seed moisture was determined with the oven-drying method using a Radwag max 50/1/WH moisture analyser at a temperature of 120°C in accordance with standard PN-EN ISO 665:2004.
The fat content in the seeds and pomace was determined with the Soxtec 8000 device following the Soxhlet method based on application ASN 310 and in accordance with standard PN-EN ISO 659:201020.
The acid value (AV) defines the amount of free fatty acids in cold-pressed oils. The test was carried out with the AOCS Official Method Cd 3d-63 (Official Method, 2000).
The peroxide value (PV) defines the amount of primary oxidation products in cold-pressed oils (MALHEIRO *et al.*, 2013). The test was carried out with the AOCS Official Method 965.33 (OFFICIAL METHOD, 1999).
The oxidative stability was determined with the Rancimat accelerated oxidation test, which measures the induction time by detection of volatile acids formed during oil oxidation (MATHÄUS, 1996). The test was carried out with the AOCS method (OFFICIAL METHOD, 1989) with the use of the 893 Professional Biodiesel Rancimat device manufactured by Metrohm.
Oil samples (2,50 ± 0,01g) were accurately weighed, placed in a measurement vessel, and subjected to air flow of 20 l/h at a temperature of 120˚C. The results were expressed as the

induction time, which was automatically determined from the curve inflection point with the use of StabNet1.0 software provided by the company.
The overall colour was determined with a spectrophotometric method in accordance with standard PN-A-86934:1995. Measurements of the absorbance of the oil samples were performed after dilution at two wavelengths in the visible range: λ=442nm for the carotenoid pigment group and λ=668nm for the chlorophyll pigment group. The absorbance values were pooled and expressed as an integer
The determination was carried out in triplicate. The arithmetic mean of the repetitions was taken as the result. The statistical analysis of the results was carried out in the Statistica 10 package. The analysis of variance ANOVA was employed to determine the significance of the differences between the values. Inference was carried out at a significance level of 0,05. The mean confidence intervals were analysed in detail with Tukey's test.

3. RESULTS AND DISCUSSION

3.1. Seed moisture and fat content

The initial mean moisture and fat levels in the sesame seeds were 7,46 and 45,61%, respectively (Table 1). The seed moisture content was higher than the value reported by GHARBY *et al*. (2017) (6%), whereas the fat content was lower than that determined by NZIKOU *et al.* (2009) (54%).

3.2. Chemical determinations of sesame oil

Selected chemical determinations that are indicators of the quality of the extracted sesame oil assessed immediately after pressing are shown in Table 1. The acid value (AV) was 1,52 mg KOH/g and the peroxide value (PV) was 0,58 meq O_2/kg. This indicates that the analysed oil fulfilled the quality requirements for cold pressed oils specified in the Codex Alimentarius in terms of the acid value and peroxide value (AV ≤ 4 mg KOH/g, PV ≤ 10 meq O_2/kg) (2009). The acid value of the analysed oil was lower than that reported by RAO *et al.* (1955), i.e. 2,5 mg KOH/g. Similarly, the peroxide value determined in this study was significantly different from the results described by YOSHIDA AND TAKAGI (1997) (1,42 meq O_2/kg), OGBONNA AND UKAAN (2013) (3,95 meq O_2/kg), and GHARBY *et al.* (2017) (2,7 meq O_2/kg) for cold-pressed sesame oil. The differences between the results of this study and those reported by other authors may be associated with the quality of the seeds used. The raw material, e.g. the degree of damage, has a significant effect on the yield and quality of pressed oils (KRYGIER *et al*. 2000).

Table 1. Mean values of moisture and fat content in sesame seeds as well as the acid value (AV), peroxide value (PV), and induction time in the extracted oil.

Sesame seeds		Sesame oil	
Moisture (± SD) (%)	7,46±0,05	AV (± SD) (mg KOH/g)	1,52±0,02
Fat content (± SD) (%)	45,61±0,63	PV (± SD) (meq O_2/kg)	0,58±0,02
		Induction time (± SD) (h)	3,91±0,05

Oxidation is primarily responsible for deterioration of the quality of fats, disagreeable odour and flavour of fat products, and reduction of their nutritional value. Therefore,

oxidative stability is one of the most important indicators of the quality of oils, especially those extracted at low temperatures, which contain natural antioxidants (e.g. tocopherols, carotenoids, sterols, phospholipids, and phenolic compounds). Concurrently, they contain undesirable compounds with prooxidant activity (e.g. metals, chlorophylls), which are removed in the refining process (KOSKI *et al*., 2002; ZINE *et al*., 2013).
The Rancimat test revealed (Tab. 1) that the induction time of the analysed oil was 3,91 h, and this value differed from results reported by other authors. In their investigations, KAMAL-ELDIN AND APPELQVIST (1995) as well as GHARBY *et al*. (2017) demonstrated higher induction times of sesame oil, i.e. 6,1 and 28,5h, respectively. These differences may be associated with the different sesame species and Rancimat test parameters (T 100°C) used in the investigations.

3.3. Effect of seed thermal treatment on oil extraction efficiency and oil content in pomace

The thermal treatment contributed to an increase in the oil yield (Table 2) and exerted an effect on the traits of the sesame oil (Figs. 1-4).
The extraction efficiency of the sesame seed oil gradually increased together with the seed heating time until it reached 120 s. The highest extraction yield (56,25%) was achieved after the 120 s thermal treatment. In comparison with the control sample, the increase in the extraction efficiency was 12,68% ($p \leq 0,05$). The longer heating time (150 s) also contributed to an increase in the oil yield, in comparison with the control sample; however, the difference over two-fold lower (5,24%) than in the case of seeds treated thermally for 90 and 120 s (Table 2).

Table 2. Mean extraction efficiency of the sesame oil relative to the fat content in the pomace.

Heating time of seeds in T 180°C (s)	The fat content in the pomace (± SD) (%)	The oil yield (± SD) (%)
0	$26,31^{e}$ ±0,21	$43,57^{a}$ ±0,04
30	$25,73^{e}$ ±0,18	$45,12^{b}$ ±0,04
60	$24,65^{d}$ ±0,23	$47,31^{c}$ ±0,04
90	$22,05^{b}$ ±0,37	$55,6^{e}$ ±0,04
120	$21,25^{a}$ ±0,24	$56,25^{f}$ ±0,03
150	$23,77^{c}$ ±0,23	$48,81^{d}$ ±0,04

Mean values in the column denoted by different letters differ significantly statistically at $p \leq 0,05$.

The highest fat content, i.e. 26,31%, was determined in the pomace from the thermally non-treated seeds (Table 2). At the longer 120 s thermal treatment of the seeds, the pomace fat content declined from the value of 25,73% noted in the case of seeds heated for 30 s to 21,25% obtained at the heat treatment lasting 120 s. At the 150 s heating time, the fat content in the pomace increased to 23,77% (Table 2). Analogous changes in "Bakara" cultivar rapeseed were reported in the investigations conducted by RĘKAS *et al*. (2015). The oil yield from these seeds was in the range from 53,9% in non-heated samples to 65,3% in the case of seeds heated with microwaves for 3 minutes. The oil extraction efficiency in seeds heated for 7 minutes was reduced to 59,8%. The pomace fat content was 25,5% in non-heated seeds, 20,2% in the case of seeds heated for 3 minutes, and 22,2% in seeds heated for 7 minutes.

The higher fat content in pomace and the lower differences in the oil pressing yields in the case of the 150 s heating time, compared to the control sample, can be attributed to the insufficient moisture level caused by the excessive time of the thermal treatment. Similar conclusions were formulated in the study conducted by KACHEL-JAKUBOWSKA (2008).

3.4. Effect of seed thermal treatment on the physicochemical traits of the extracted oils

To assess the effect of the infrared seed treatment applied prior to the pressing process on the physicochemical traits of the oils, the basic quality parameters were evaluated (Figs. 1-4).
The acid value of the analysed oils ranged from 1,52 mg KOH/g in the oil extracted from the non-heated seeds to 2,02 mg KOH/g in the oil from seeds treated with heat for 150 s (Fig. 1).

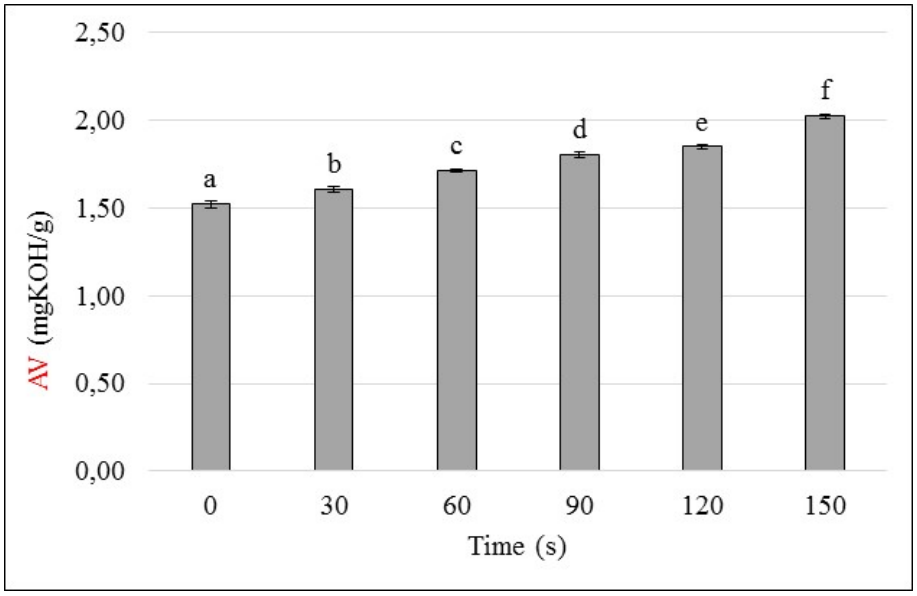

Figure 1. Acid values of oils pressed from sesame seeds relative to the time of seed thermal pre-treatment "t"; different letters above the bars indicate statistically significant differences.

The highest increase in the hydrolysis degree was observed in the oil extracted from seeds treated with 150 s heating.
Furthermore, the oils were characterised by a low peroxide value (Fig. 2), which determines the primary products of oil oxidation.
The peroxide value ranged from 0,58 meq O_2/kg in the non-heated seeds to 1,75 meq O_2/kg in the oil from seeds treated thermally for 150 s. All the analysed oils met the requirements for the acid value and peroxide value in cold-pressed oils specified in the CODEX ALIMENTARIUS (2009). A similar effect of seed thermal treatment on changes in AV and PV of extracted oils was observed by other authors. In investigations of sesame oil from seeds heated at 160°C for 10, 20, and 25 minutes, YOSHIDA AND TAKAGI (1997) demonstrated a PV range from 1,42 (non-heated seeds) to 5,38 meq O_2/kg (seeds heated for 25 minutes). KRALJIĆ *et al*. (2013) evidenced that PV of oil from different rapeseed species heated at a temperature of 80°C for 30 minutes ranged from 1,6 (non-heated seeds) to 2,6 mg KOH/g (heated seeds -Toccata cultivar), from 1,5 (non-heated seeds) to 2,3 mg KOH/g (heated seeds - Oase cultivar), and from 1,3 (non-heated seeds) to 2,3 mg KOH/g (heated seeds - Remy cultivar). In turn, the AV of these oils was in the range from 0,2 meq

O_2/kg (non-heated seeds) to 0,25 meq O_2/kg (heated seeds -Toccata cultivar), from 0,29 (non-heated seeds) to 0,36 meq O_2/kg (heated seeds - Oase cultivar), and from 0,43 (non-heated seeds) to 0,44 meq O_2/kg (heated seeds - Remy cultivar). In investigations of oil from sesame seeds heated with microwaves at a temperature of 100°C for 15 minutes, ABOU-GHARBIA *et al.* (2000) showed a PV range from 0,46 (non-heated seeds) to 0,98 mg KOH/g (heated seeds).
The analysis of the oxidative stability of the extracted oils (Fig. 3) proved that the parameters of the seed thermal treatment applied before the extraction process extended the oil induction time.

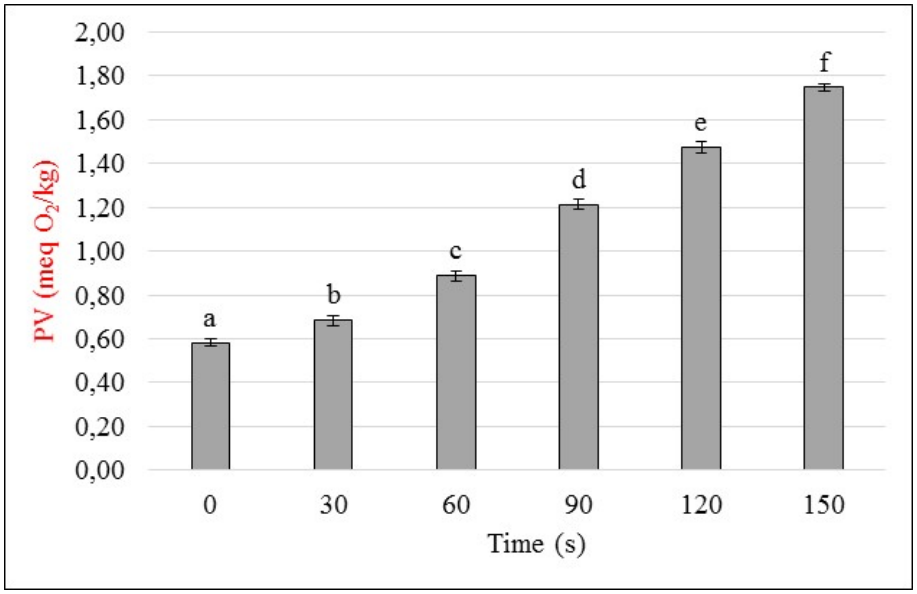

Figure 2. Peroxide values of oils pressed from sesame seeds relative to the time of seed thermal pre-treatment "t"; different letters above the bars indicate statistically significant differences.

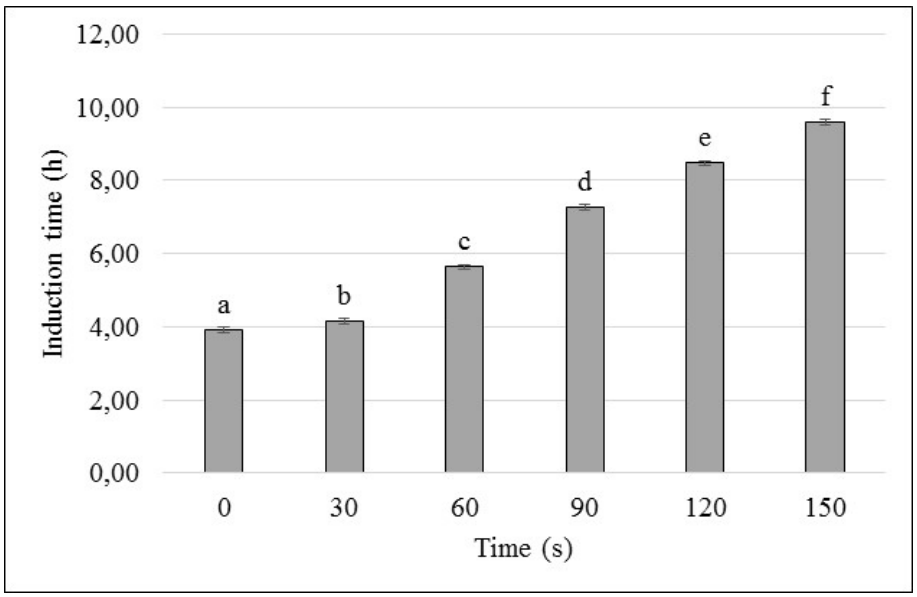

Figure 3. Induction time of oils pressed from sesame seeds relative to the time of seed thermal pre-treatment "t"; different letters above the bars indicate statistically significant differences.

The value of this parameter increased with the longer seed heating time and ranged from 3,91 h for the oil from the non-heated seeds to 9,6 h for the oil extracted from seeds heated for 150 s. The 150 s thermal pre-treatment of the seeds contributed to an almost three-fold increase in the oil induction time, i.e. by 5,69 h. The literature provides reports of the increase in the oxidative stability of oil extracted from thermally treated seeds. In their analyses of oil extracted from sesame seeds heated with microwaves at 100°C for 15 minutes, ABOU-GHARBIA *et al.* (2000) noted an induction time in the range from 9,49 (oil from non-heated seeds) to 18,68 h (oil from heated seeds).
Although there was a statistically significant increase in the LN value as a result of heating, this value was low (1,8 meqO_2/kg) even for oil extracted from seeds heated for 150 s. This did not influence the oxidative stability of the analysed oils. Similarly, FLACZYK *et al*. (2004) did not find a direct relationship between the content peroxides in oils and their oxidative stability. The higher content of natural antioxidants in the oils obtained from the heated seeds may have been a determinant of the long induction time in the analysed oil.

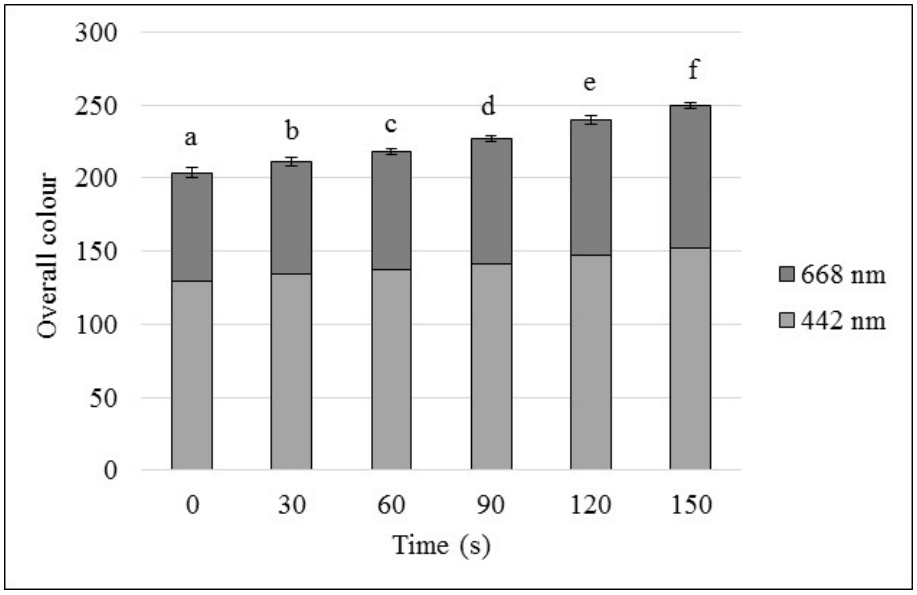

Figure 4. Colour of oils extracted from sesame seeds (1000 x (A442nm + A668nm)) relative to the time of thermal pre-treatment of the seeds "t"; different letters above the bars indicate statistically significant differences.

The analysis of the colour of the sesame oil (Fig. 4) revealed that the oil from the non-heated seeds was the lightest and clearest (overall colour 203). It contained the lowest content of carotenoid and chlorophyll pigments in comparison with the oils extracted from the heated seeds. The thermal treatment of the seeds caused significant darkening of the extracted oil and an increase in absorbance at the wavelengths of 442 and 668 nm. The colour of the oil from the heated seeds was in the range from 211 after the 30 s thermal treatment to 249,67 after heating the seeds for 150 s. Therefore, the oil extracted from seeds receiving the longest thermal pre-treatment was the darkest and the least clear. The same effect of thermal seed treatment on changes in the colour of extracted oils was reported by GHARBIA *et al.* (2000). In investigations of oil extracted from sesame seeds heated with microwaves at 100°C for 15 minutes, the authors demonstrated a three-fold higher content of total carotenoid and chlorophyll pigments in oil from heated seeds than that in oil

extracted from non-heated seeds. The darker colour of the oils extracted from pre-heated seeds can be explained by the presence of products of Maillard reaction and phospholipid and chlorophyll degradation products derived from the thermal seed treatment (AZADMARD-DAMIRCHI *et al.*, 2011; LAMORSKA AND TYS, 2011).

4. CONCLUSIONS

Based on the research results, the following conclusions were formulated:
Infrared thermal treatment of sesame seeds exerts a significant effect on the oil extraction efficiency. The highest increase in the oil yield was noted in the case of seeds heated for 120 s. The longer heating time of 150 s reduced the efficiency of the extraction process.
Infrared thermal treatment of sesame seeds causes a significant increase in the oxidative stability of oil. The longest induction time in the Rancimat test (9,53 h) was recorded for the oil extracted from seeds heated for 150 s.
Thermal seed pre-treatment contributes to an increase in the acid value and the peroxide value, a higher absorbance value at wavelengths of 442 and 668 nm, and a darker overall colour of oil. All the oils analysed in the study fulfil the quality standards in terms of the acid value (AV ≤ 4 mg KOH/g) and the peroxide value (PV ≤ 15 meq O_2/kg).

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