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Microwave pretreatment effects on the changes in seeds microstructure, chemical composition and oxidative stability of rapeseed oil

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ABSTRACT

The purpose of the present study was to investigate the effect of microwave (MV) irradiation on the microstructure of rape seeds, recovery of oil, and to examine changes in oxidative stability, composition of FA, and contents of tocochromanols and canolol of rapeseed oil. Rapeseeds (*Brassica napus*) of two varieties, Kana and Bakara, adjusted to moisture contents of 7 and 9%, undergone MV pretreatment under 800 W for 0, 3, and 7 min, prior to oil extraction by cold-press. Tocochromanol contents were differently affected by the MV pretreatment of rapeseed prior to pressing. Depending on the adjusted seed moisture content both individual and total tocopherol contents increased to the maximum at different microwave times. The amount of canolol detected in cold-pressed oil samples ranged from 16.34 to 16.97 μg/g of oil; this was increased to 926.42 and 821.86 μg/g by MV pretreatment for 7 min (at 7% moisture level). The oxidative stability prolongation from 3.64 to 4.09 h up to 11.52–12.75 h was observed. Regarding the FA composition of the oils, no significant changes ($P > 0.05$) in the fatty acid composition occurred during seeds MV pretreatment.

1. Introduction

Along with soybean and palm oil, rapeseed is one of the most important oil crops in the world and a major source of edible vegetable oil in Poland. In the years 2013/2014 production acreage increased to 1 million ha, and rapeseed production amounted to 2.8 million tons, while worldwide production of rapeseed oil reached 26.98 million tons (www.faostat.fao.org). Rapeseed oil is considered one of the healthiest cooking oil, particularly due to its beneficial balance of fatty acids. Moreover, it contains nutritionally favourable linoleic (ω-6) to α-linolenic (ω-3) essential fatty acids ratio of 2:1 (Przybylski, Mag, Eskin, & McDonald, 2005).

Recently, the extraction of oil using cold-pressed method is re-blooming (Febrianto & Yang, 2011). As a result, in the past 20 years, a new interest in virgin oils beyond virgin olive oil arose steadily (Matthaüs, 2008). Despite the many advantages of mechanical pressing, low oil extraction yield hindering the development of this technique to become commercially viable. However, this may be improved by oilseeds heat pretreatment by roasting or microwave irradiation. In reference to microwave irradiation, both initial seeds moisture content and microwave exposure time exert a significant effect on oil extraction yield rather than the level of potency. Azadmard-Damirchi, Habibi-Nodeh, Hesari, Nemati, and Achachlouei (2010) found that rapeseed microwave pretreated for 2 and 4 min can increase oil yield by 10%. Nonetheless, no information was given regarding seeds moisture content prior exposure to microwave irradiation. Yang et al. (2013) treated rapeseeds, adjusted to moisture content of 9, 11, 13, 15%, with microwaves for 1–7 min (1 min interval) and reported the lowest residual oil content in cake, when seeds, adjusted to 9% moisture content, were MV-treated for 7 min. However, no specific data was given regarding oil yield after such seeds heat treatment with microwaves.

The oils produced through cold-pressing retain all of their

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bioactive components, due to strict pressing temperature regimen. However, recent studies suggest that through thermal pretreatment of seeds via conventional oven heating (Kraljić et al. 2013; Siger, Kaczmarek, & Rudzińska, 2015) or by microwave pretreatment (Azadmard-Damirchi et al., 2010; Yang et al., 2013) greater amount of bioactive compounds may penetrate into the produced oil. The effects of rapeseed roasting prior to pressing on oil compositional changes have been well investigated. Kraljić et al. (2013) found that conditioning rapeseeds at 80 °C for 30 min increased the amount of tocopherols, phytosterols and phenolic in the oil. Similar results were presented by Rękas, Wroniak, and Rusinek (2015), who reported that along with rapeseed roasting temperature elevation (80–140 °C for 1 h), a significant increase in the content of total tocopherols and a slight increase of total sterols concentration occurred. Siger et al. (2015), who studied the effects of high roasting temperatures, namely 140, 160, 180 °C on the contents of the native antioxidants (tocopherols, PC-8, phenolic compounds, canolol, and phytosterols), found higher content of these compounds in oils pressed from seeds that had previously undergone roasting. On the other hand, researchers studying microwave irradiation as a rapeseed heat pretreatment prior to cold-pressing have so far focused on the content of tocopherols and phytosterols in the resulting oil and their impact on the oxidative stability (Azadmard-Damirchi et al., 2010; Yang et al., 2013), but there is insufficient data on the effect of microwave pretreatment on the canolol content and fatty acid composition of rapeseed oil. Canolol, formed by thermally initiated CO_2 splitting off from sinapic acid, constitute a major phenolic compound found in rapeseed oil obtained from seeds that had previously undergone thermal treatment (Pudel et al., 2014). Spielmeyer, Wagner, and Jahreis (2009) reported that the maximum canolol formation in rapeseed was achieved after rapeseeds 7.5-min microwave exposure (800 W), with a final temperature of 160 °C (increase from 0.58 to 72 mg/100 g). However, extraction with organic solvent (petroleum ether) was used to recover the oil from seeds.

The objectives of this study were to investigate the changes in oxidative stability, composition of fatty acids, and contents of tocochromanols and canolol in rapeseed oil extracted by pressing after different microwave pretreatments as well as to examine the impact of pretreatment by microwave irradiation on the microstructure and recovery of oil.

2. Materials and methods

2.1. Rapeseed

Seeds of winter type rapeseed: Kana and Bakara, were provided by the Plant Breeding Strzelce Ltd. Co. – IHAR Group, Poland. Seeds were harvested in optimum maturity, and did not contain any impurities or broken seeds. They were stored in paper bags in atmospheric conditions at 15 ± 2 °C.

Oil content of seeds and cake was determined by automated Soxhlet extraction (Soxtec™ 2050 Auto Fat Extraction System, Foss North America, Eden Prairie, USA). The samples (5 g) were weighed into thimbles and inserted into the extraction unit. The 4-step extraction consisted of boiling, rinsing, evaporation/solvent recovery and pre-drying. The dried samples were accurately re-weighted and the oil content in seeds and cake was expressed as % of dry mass.

Moisture content of seeds and cake was determined according to ISO standard method (ISO 5509, 2000; ISO 665, 2000). The measured mass loss was expressed as percentage of water content in the sample.

2.2. Reagents

Analytical standards of γ-, α-, δ-, and β-tocopherols (purity > 95% by HPLC) were purchased from Calbiochem-Merck Biosciences (Darmstadt, Germany). HPLC-grade *n*-hexane, methanol, acetonitrile (ACN), orthophosphoric acid, and *1.4*-dioxane were obtained from Merck (Darmstadt, Germany). Potassium hydroxide (KOH), methanol (MeOH), and hexane were bought from POCH (Gliwice, Poland).

2.3. Microwave pretreatment

Whole seeds of two rape, Kana and Bakara, with initial moisture content of 5.5 and 5.7%, respectively, were equilibrated at refrigerated temperature (4 ± 2 °C) in closed containers for 24 h to 7 and 9% moisture contents. For each microwave (MV) pretreatment, 500 g of seeds were placed in glass beaker (16-cm diameter) inside the microwave (Model: NN-J155W, 800 W). Samples were microwave treated at a frequency of 2450 MHz for two times of radiation (3 and 7 min). Immediately after every heating run the temperature of the seeds was determined using handheld infrared thermometer (KC–180B, Tynaxtools, Poland). Rapeseed sample without microwave radiation (0 min radiation time) was used as a control sample. Each experiment was performed in triplicate for every rapeseed variety. Following each heating run, seeds were allowed to cool to ambient temperature and thoroughly mixed to obtain a homogenous sampling.

2.4. Oil extraction by cold-pressing

The oil was pressed with the use of screw press (Farmet, Czech Republic) at room temperature. The temperature inside the press was 60 ± 10 °C and the temperature of the outflowing oil was 39 ± 1 °C. After pressing oils were filtered to remove particles, and afterwards kept in dark glass bottles (vol. of 65 ml) under refrigerated temperature (4 ± 2 °C) until analysed.

Oil yield was calculated on the basis of the following formula (Swetman & Head, 1998):

$$Y = 100 \times \frac{R_s}{R_c}$$

where: Y – oil yield, R_s – the ratio of non-lipid components in seed to the oil content in seed, R_c – the ratio of non-lipid components in cake to the residual oil content in cake.

2.5. Seeds microstructure

Changes in the structure of rapeseeds were determined based on the analysis of sample images using an electron scanning microscope (FEI Quanta 200 ESEM, USA) equipped with an energy-dispersive spectrometer (EDS) and digital image recording. Rapeseed samples (raw and MV-treated), previously cut with razor blade, were microscopically examined to determine the visible effect of MV pretreatment on the kernel cotyledon/cell structure. Specimens were observed without earlier preparations of the samples at pressures of 100–133 Pa, under accelerating voltage of 25 or 30 kV. Graphical elaboration of seeds structure were performed using MultiScan v.18.03 software (Computer Scanning System).

2.6. Determination of tocochromanols and canolol

Tocochromanols (α-, β-, γ-, and δ-tocopherols and PC-8) and canolol were determined according to the method described by