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EFFECT OF DIFFERENT PROCESSING TECHNIQUES ON INDONESIAN ROSELLE (*HIBISCUS RADIATES*) SEED CONSTITUENTS

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ABSTRACT

Background. Roselle seeds are waste that is left behind during processing of roselle for juices or other roselle related products. Disposing of waste is highly undesirable both economically and environmentally. Roselle seeds contain high amount of protein, crude fibers, fats, and carbohydrates. The objective of this study was to determine the effect of different processing techniques (roasting using oven/microwave, and boiling) on Indonesian roselle (*Hibiscus radiates*) seed constituents.

Methods. Three treatments were carried out to prepare the samples: The seeds were roasted at 130 and 150°C for 30 min (HOR), microwave roasting for 10, 20 and 30 min HMR10, HMR20, HMR30 of roselle seeds. Hibiscus seeds (3×200 g) were put into boiling tap water (100°C) (HB) in a 500 ml beaker on magnetic stirred hot plate at a ratio of 1:4 seed: water for 40 min until the pieces were well cooked and tender. Proximate chemical analysis was determined following the standard methods of the Association of Official Analytical Chemists. Hibiscus different samples were analysed in triplicate and the results were reported as means. Total carbohydrate content was calculated from the difference. The fatty acids of the oil samples were analysed using gas chromatography (Shimadzu, GC-2010A series, Shimadzu, Tokyo, Japan) equipped with a flame ionization detector and a BPX70 capillary column of 30 m × 0.32 mm i.d. (SGE, Melbourne, Australia). The tocopherol content of the oil samples was measured by HPLC (Cecil Instruments Ltd., Cambridge, England).

Results. Proximate composition of untreated, roasted and boiled hibiscus seeds showed that, roasting and boiling temperatures can increase fat and fiber content, microwave and boiling showed higher fat content when compared with oven roasting treatment. Protein content of HU was significantly lower ($p < 0.05$) than roasted and boiled Rossle seeds. Statistical results indicated that the protein of Roselle seeds increased in the order of HB > HMR > HOR. Protein content of HB, HMR and HOR increased significantly as compared with HU. The carbohydrate values of Indonesian Rossele treated seeds were significantly ($p < 0.05$) different from each other. The main fatty acids in all samples were palmitic and linolenic. These fatty acids did not change with roasting and boiling temperatures. Tocopherol concentration decreased during processing techniques as a result of heating temperature.

Key words: *Hibiscus radiates*, roasting, boiling, fatty acid, tocopherols

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INTRODUCTION

Hibiscus belongs to the Malvaceae family. It is quite large, containing several hundred species that are native to warm-temperate, subtropical and tropical regions throughout the world. Roselle seeds are the waste that is left behind during processing of roselle products. Roselle seeds contain about 14.9% protein, 21.2% crude fiber, 14.6% fats and oils, 35.6% carbohydrate by weight. Roselle seed oil contains phytosterols and tocopherols [Nyam et al. 2009].

The nutritional usefulness of the Roselle seeds has been studied as compared with the calyces [Abu-Tarboush et al. 1997, Rao 1996]. However, no nutritional data have been reported on *Hibiscus radiates* seeds grown in Indonesia. Also, nutritional compositions of seeds vary depending on the variety, location and environmental conditions where the seeds were grown.

Significant differences in protein, ash and fat contents of products prepared from potato flour were observed due to their compositional differences connected with frying and fermentation techniques [Lakra and Sehgal 2011]. Roasting is the key step for making condiment oil, since the colors, flavor, composition and quality of the oil are affected by the processing conditions used [Lee et al. 2004, Ozdemir and Devres 2000]. The crude protein and total ash contents of flour from the Ivorian taro (*Colocasia esculenta* cv. fouê) corm were not affected significantly ($p < 0.05$) by the change in boiling time [Amon et al. 2011]. Mariod et al. [2012] studied the effects of roasting and boiling on compositional and oil stability of safflower seeds, they found that moisture, carbohydrate and fiber contents were decreased because of roasting and boiling treatments while fat and protein contents increased. Lipid oxidation strongly affects shelf-life and sensory characteristics of oilseeds, and depends on many factors such as the concentrations of unsaturated fatty acids and presence of natural antioxidants [Ozdemir and Devres 2000]. The present study investigated the effects of microwave, oven roasting and boiling on chemical, fatty acids and tocopherol composition of Indonesian Roselle seeds.

MATERIAL AND METHODS

Samples solvents and reagents

All solvents used were of analytical grade n-hexane, petroleum-ether, and 2-mercaptoethanol, besides HCL, Ca (OH), and Tris-HCl/glycine buffer were obtained from Merck, Darmstadt, Germany.

Roselle seeds (*Hibiscus radiates*) originated from Indonesia, and were provided by Dr. Mohammed Hanafi (Indonesian Institute of Sciences, LIPI, Bandong, Indonesia).

Roselle seeds were collected from the Bandung local market, cleaned under tap water and dried at 40°C in an oven then ground to obtain homogeneous samples, before representative samples were taken for chemical analysis. For determination of the oven-dry weight, samples was dried at 105°C for 24 h.

Preparation of Hibiscus (Roselle) seeds

Oven roasting. Whole roselle seeds were arranged in a single layer in aluminium foil dishes (12×8 cm), then placed in an electric forced air oven (FN400, NO.03822, Turkey). The seeds were roasted at 130 and 150°C for 30 min (HOR). Three dishes were treated once at each of the different roasting times to prepare sufficient sample material for analysis and testing. The hibiscus oven roasted seeds (HOR) were allowed to cool to ambient temperature, then ground in an electric grinder (Panasonic, Japan) and stored at room temperature for further analysis [Lee et al. 2004].

Microwave roasting. Microwave roasting for 10, 20 and 30 min HMR10, HMR20, HMR30 of roselle seeds, 200 g of seeds were placed in an even layer in Pyrex petri dishes (26 cm diameter) inside the microwave (Model: MW2300 GF, 800 W). Samples were microwave treated at a frequency of 2450 MHz for 10, 20 and 30 min. Three sets of 200 g roselle seeds were used at each radiation time [Azadmard-Damirchi et al. 2010].

Boiling. Hibiscus (Roselle) seeds (3×200 g) were put into boiling tap water (100°C) (HB) in a 500 ml beaker on magnetic stirred hot plate at a ratio of 1:4 seed: water for 40 min until the pieces were well cooked and tender. The treated samples were dried and ground in a grinder (Panasonic, Japan) and used in proximate chemical analysis and oil extraction. Control samples were not treated with either of the two

techniques described above but also ground at the same methods mentioned above. Hibiscus untreated seeds (HU) sample was used as a control sample.

Proximate chemical analysis

Proximate chemical analysis was determined following the standard methods of the Association of Official Analytical Chemists [1995]. Hibiscus different samples were analysed in triplicate and the results were reported as means. Total carbohydrate content was calculated from the difference.

Fatty acid composition

All oil samples were derivatized to methyl esters following the International Organization of Standards (ISO) draft standard (ISO, 2000). Methyl ester sample (1 µL) was injected into the gas chromatography (Shimadzu, GC-2010A series, Shimadzu, Tokyo, Japan) equipped with a flame ionization detector and a BPX70 capillary column of 30 m × 0.32 mm i.d. (SGE, Melbourne, Australia). The initial temperature of 140°C was held for 2 min, which was then increased at 8°C/min to 220°C where it was held for another 5 min. The oven and the injector and the detector ports were set at 140, 240 and 260°C, respectively. The carrier gas was helium with column flow rate of 1.10 ml·min^{-1} in a 50:1 split ratio. The fatty acid peaks were identified by comparing the retention times with those of a mixture of standard FAMEs (Sigma Chemicals, Deisenhofen, Germany).

Determination of tocopherols by high-performance liquid chromatography

The tocopherol content of the oil samples was measured by HPLC (Cecil Instruments Ltd., Cambridge, England) according to the method described by Azadmard-Damirchi et al. [2010]. Approximately, 10 mg of the extracted oil were dissolved in 1 ml n-heptane, and 10 µl were directly injected. The column used was LiChro CART 250-4. Tocopherols were detected by a fluorescence detector at wavelengths of 294 and 320 NM for excitation and emission, respectively. According to the retention times of reference samples of tocopherols in the chromatogram, each tocopherol in the analysed oil samples was identified. Quantification was carried out using an external standard method with reference samples of tocopherols. All samples were analysed in triplicate and the results report is the means of these.

STATISTICAL ANALYSIS

Each reported value is the mean of determinations for triplicate samples (except fatty acids) prepared from replicating roasting and boiling process technique, and the data were analysed and the discussion was based on one-way analysis of variance (ANOVA) using SPSS (version 16). Statistical significance was accepted at a level of $P < 0.05$ (Statgraphics 1985-1989).

RESULTS AND DISCUSSION

Proximate composition

Proximate composition of untreated, roasted and boiled hibiscus (*Hibiscus radiates*) seeds powder is presented in Table 1. Results showed that, roasting and boiling temperatures can increase fat and fiber content, microwave and boiling showed higher fat content when compared with oven roasting treatment. The seeds, regarded as byproduct of Roselle processing had 5.0% moisture content. However, after microwave roasting for 5, 10 and 15 min, the remaining moisture content was 3.9, 2.8 and 1.4%, respectively. Protein content of HU was significantly lower ($p < 0.05$) than that of roasetd and boiled Rossle seeds. Statistical results indicated that the protein of Roselle seeds increased in the order of HB > HMR > HOR. Protein content of HB, HMR and HOR increased significantly as compared with HU.

The fat content of hibiscus untreated seeds was 14.7%, the statistical analysis revealed a significant difference ($p < 0.05$) in fat content of the microwave, oven roasting and boiling seeds studied. Fat content of seeds roasted using microwave was significantly ($p < 0.05$) increased with time and reached up to 19.3% in HMR 200 W/15. Total available carbohydrate was found to be higher in HU than HMR, HOR and HB. The carbohydrate values of Indonesian Rossele treated seeds were significantly ($p < 0.05$) different from each other.

Fatty acid and tocopherol composition of Roselle seeds oil

Vegetable oils undergo changes in terms of chemical and physical properties when they interact with

Table 1. Proximate analysis of Roselle seeds powder

Constituent	HU	HMR 200W/5	HMR 200W/10	HMR 200W/15	HOR 130C/30	HOR 150C/30	HB
Moisture	5.01 ±0.0^{a}	3.94 ±0.3^{b}	2.82 ±0.1^{c}	1.45 ±0.1^{d}	3.92 ±0.1^{e}	2.56 ±0.1^{f}	4.91 ±0.1^{g}
Protein	20.12 ±0.8^{a}	20.84 ±1.4^{a}	22.54 ±0.2^{c}	21.45 ±0.8^{a}	21.11 ±0.1^{a}	20.20 ±1.9^{a}	22.10 ±0.5^{c}
Fat	15.75 ±0.5^{a}	18.33 ±1.2^{b}	17.64 ±0.6^{c}	19.33 ±1.8^{d}	15.74 ±0.9^{e}	16.15 ±0.2^{f}	17.90 ±0.3^{c}
Fiber	25.30 ±02^{a}	38.12 ±0.3^{b}	31.90 ±0.3^{c}	32.14 ±0.2^{d}	28.55 ±0.2^{e}	27.54 ±0.2^{f}	24.55 ±0.2^{g}
Ash	6.15 ±0.1^{a}	6.52 ±0.1^{b}	7.75 ±0.1^{c}	8.50 ±0.1^{d}	6.92 ±0.1^{b}	7.25 ±0.1^{c}	7.92 ±0.1^{c}
Carbohydrate	27.80 ±0.3^{a}	12.75 ±0.2^{b}	17.55 ±0.1^{c}	17.37 ±0.1^{c}	23.95 ±0.2^{d}	26.55 ±0.2^{e}	22.74 ±0.2^{f}

HU – Hibiscus untreated sample, HMR 200W/5 – Hibiscus microwave roasted at 200°C for 5 min, HMR 200W/10 – Hibiscus microwave roasted at 200°C for 10 min, HMR 200W/15 – Hibiscus microwave roasted at 200°C for 15 min, HOR 130C/30 – Hibiscus oven roasted at 130°C for 30 min, HOR 150C/30 – Hibiscus oven roasted at 150°C for 30 min, HB – Hibiscus boiled.
Values are expressed as mean ±S.E.M. of three measurements. Data were statistically analysed using one-way ANOVA. Values with different letter are significantly different at $p < 0.05$ within the same row.

Table 2. Fatty acid (%) and tocopherol (%) composition of Roselle seeds oil

Fatty acid	HUT	HMR 200W/5	HMR 200W/10	HMR 200W/15	HOR 130C/30	HOR 150C/30	HB
C14:0	0.3 ±0.02	0.3 ±0.02	0.2 ±0.02	0.3 ±0.03	0.2 ±0.01	0.2 ±0.02	0.3 ±0.01
C16:0	20.21 ±0.4	19.85 ±0.3	20.18 ±0.3	20.68 ±0.4	20.15 ±0.3	20.09 ±0.3	20.54 ±0.3
C16:1	0.4 ±0.01	0.4 ±0.03	0.4 ±0.02	0.4 ±0.03	0.4 ±0.03	0.4 ±0.02	0.4 ±0.02
C18:0	4.58 ±0.2	4.5 ±0.2	4.53 ±0.2	4.47 ±0.3	4.33 ±0.3	4.46 ±0.2	4.42 ±0.3
C18:1	21.62 ±0.5	21.63 ±0.4	21.36 ±0.3	21.32 ±0.4	22.55 ±0.3	21.42 ±0.2	21.37 ±0.3
C18:2	51.39 ±0.7	52.02 ±0.7	52.09 ±0.6	51.40 ±0.5	51.50 ±0.6	51.89 ±0.5	51.77 ±0.4
C18:3	0.9 ±0.02	0.9 ±0.02	0.8 ±0.1	1.05 ±0.3	0.54 ±0.03	1.07 ±0.3	0.9 ±0.1
C20:0	0.5 ±0.02	0.4 ±0.01	0.4 ±0.01	0.4 ±0.02	0.3 ±0.01	0.4 ±0.02	0.3 ±0.01
Total SFA	25.57 ±0.6^{a}	25.08 ±0.6^{a}	25.38 ±0.6^{a}	25.53 ±0.5^{a}	25.02 ±0.5^{a}	25.22 ±0.4^{a}	25.53 ±0.3^{a}
Total PUFA	74.43 ±0.3^{b}	74.91 ±0.3^{b}	74.61 ±0.4^{b}	74.57 ±0.3^{b}	74.98 ±0.5^{b}	74.88 ±0.4^{b}	74.47 ±0.5^{b}
Tocopherol	0.05 ±0.01	0.03 ±0.01	0.02 ±0.3	0.01 ±0.2	0.04 ±0.3	0.01 ±0.2	0.02 ±0.4

For abbreviations see Table 1.
Values are expressed as mean ±S.E.M. of three measurements. Data were statistically analysed using one-way ANOVA. Values with different letter are significantly different at $p < 0.05$ within SFA and PUFA.

the food or the atmosphere. Some food processing techniques can affect fatty acid composition of oils when hardly subjected to successive heating [Lee et al. 2004]. Types of reaction that are known to lead to degradation of vegetable oils include polymerization, oxidation and hydrolysis. The fatty acid composition (FAC) of oil can be an indicator of its stability, physical properties, and nutritional value. The fatty acid composition determined by GC of Indonesian Roselle seed oils (HU, HMR, HOR and HB) is illustrated in Table 2. The result shows that the major fatty acids in Roselle seed oils were linoleic, oleic, and palmitic. Roselle oil from seeds roasted by microwave and oven and that boiled was not different from oil of untreated (HU) Roselle seeds. Fatty acid compositions of Roselle oils did not change with roasting and boiling temperatures.

The results in Table 2 indicate the main SFA and PUFA in all samples were palmitic and linolenic acid, respectively. These fatty acids did not change with roasting and boiling temperatures. Hiromi et al. [2005] mentioned that unsaturated fatty acids of peanut oil are significantly protected from oxidation during microwave roasting. Sachiko and Hiromi [1999] stated that the principle characteristics of fatty acids still remained after 20 minutes of microwave heating.

Tocopherols are important natural antioxidants present in vegetable oils. The contents of total tocopherols in Indonesian Roselle seeds oil prepared at various roasting and boiling temperatures are given in Table 2. As general saying the total tocopherol concentration decreased during processing techniques as a result of heating temperature. Tocopherol of untreated Roselle seed oil was 0.05% this amount affected by microwave temperatures and time and decreased to 0.03, 0.02 and 0.01% in seeds treated by microwave temperature for 5, 10 and 15 minutes, respectively. In the same manner total tocopherol concentration decreased to 0.04, 0.01 and 0.02% of samples roasted using an oven temperature of 130 and 150°C and boiling, respectively.

CONCLUSIONS

In conclusion, the results obtained in the present work have shown that the proximate chemical analysis, as well as tocopherols of *Hibiscus radiates* seeds, can be affected by roasting and boiling techniques. Roasting and boiling improve the nutritional value of Roselle seeds by increasing fat, protein and fiber content. Microwave and boiling showed higher fat and protein content when compared with oven roasting treatment. The main fatty acids in all samples were palmitic and linolenic. These fatty acids did not change with roasting and boiling temperatures. Tocopherol concentration decreased during processing techniques as a result of heating temperature.

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