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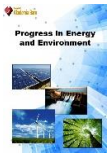

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Original Article

Physical and chemical characterization of oil extracted from *Citrofortunella microcarpa*, *Hibiscus sabdariffa* and *Artocarpus heterophyllus* seeds

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Soon Zheng Fai, Cheok Choon Yoong*

Department of Chemical and Petroleum Engineering, Faculty of Engineering, Technology and Built Environment, UCSI University, Kuala Lumpur, 56000, Malaysia

Abstract

Consumable plant oils had been related with highly nutritional content and antioxidant source that provide crucial health benefits. Hence, in this study a series of experimental work and analysis were conducted to evaluate the practicability and the properties of oil extraction from seeds of calamansi (*Citrofortunella microcarpa*), roselle (*Hibiscus sabdariffa*), and jackfruit (*Artocarpus heterophyllus*). The yield, antioxidant activities, iodine value, saponification value, and peroxide value of extracted seed oils were determined. Results obtained shown that among the three seeds species, calamansi seeds resulted in the highest yield of 32.7%, followed by roselle seeds with a yield of 11.00% and lastly, jackfruit seeds with the highest yield obtained was only 0.60%. Antioxidant activities conducted has shown that roselle with EC_{50} of 49.04 mg/mL has greater antioxidant properties as compared with calamansi with EC_{50} of 109.74 mg/mL. Iodine value determined for unheated oil has resulted in a value of 100.83 g I_2/100g oil and 110.60 g I2/100g oil that has classified roselle and calamansi seeds oil under the non-drying oil categories and heating effect shown a slight decrease in the iodine value due to loss of unsaturation within the oil. Both calamansi and roselle seed oils did not show significance reduction in saponification value upon heating. Peroxide value for roselle and calamansi seeds oil under unheated condition was determined to be 10.36 meq/kg and 24.86 meq/kg respectively.

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1 Introduction

Pulps, fruits, seeds, and plumules of various plants have been the main source of edible plant oil. The plant is one of the main sources of edible plant oil from their seed and in the early phase of production, sesame oil and olive oil were the majorly produced edible plant oil. Being a major source of energy towards human activities, edible plant oil has been heavily used in cooking and another portion of it is being utilized in the cosmetics and health industry. It has been reported that the market size of edible plant oil was reaching nearly 203 million tons in 2019 [1]. With the growth in the global population, the demand for edible plant oil has been rising co-currently [1]. As reported, the global demand for edible oil has been increasing drastically and the forecasted global production of edible oil that is mainly represented by soybean oil is expected to achieve 632 million tons in the year 2022 [1]. Therefore, there has been increasing interest and attention to discovering and producing oils from plant/fruit seeds [2].

* **Corresponding author** cheokcy@ucsiuniversity.edu.my ✉

Oils extracted from seeds of pomegranate [3] and safflower [4] have been discovered having great antioxidant properties.

Tropical fruits such as calamansi, roselle and jackfruit which contain substantial amount of seeds could be considered as a potential source of oil. Calamansi is an important citrus hybrid that belongs to the family of *Rutaceae* [5]. Calamansi fruit or pulp generally has 6 to 10 segments that are in yellow to orange colour with high juice contents with 1 to 5 small elliptical green seeds within the pulp [6]. The juice, which is typically characterized by its high acidity because of the high citric acid content, are widely used in food processing industries. As a result, the pressed pulp, seeds, and the rind were considered as residues and citrus waste. However, these citrus wastes have been discovered containing phytochemicals, include coumarins, flavonoids, lignin, phenolic acids and tannins, with health-promoting properties [7,8] and natural antioxidants [9]. Roselle is a species of flowering plant that is catogerized to the family of *Malvaceae*. The useful part of roselle calyx and bracts are often collected at the fructification phase in the production of refreshing drink. Roselle seeds are often classified as waste and disposed of upon processing of roselle for roselle-related products [10]. Seeds of roselle are reported to contain a high amount of phytosterol that is linked to reducing total cholesterol and serves as an anti-carcinogenic effect on humans [10]. Jackfruit is a species of tree that belongs to the family of *Moraceae*. The fruits of jackfruit are known to be of vital importance and contribution to the dietary due to their high nutritional value. Jackfruit is reported to contain phytonutrients such as lignans, saponins, and isoflavones that possess a wide range of health benefits for humans. These phytonutrients that exist in jackfruit possess antihypertensive, anticancer, antiulcer, and antiaging properties [11]. Seeds of jackfruit, which taken 8-11% of the whole fruit [12], are oblong ellipsoidal to oval round shape and commonly consumed upon boiling, steaming and roasting. Besides, its seeds are processed canned in brine and sauce of tomato upon boiling [13]. Study has shown that jackfruit including pulp, seed, and peel contain several bioactive and functional compounds such as carotenoids that serve as antioxidants in demonstrating the health-benefiting effect that prevents chronic diseases like cancer and cardiovascular diseases [14].

Therefore, this study aimed to characterize and evaluate the physical and chemical properties of oils obtained from calamansi, roselle, and jackfruit seeds if they are feasible for human consumption.

2 Materials and methods

2.1 Calamansi, roselle, and jackfruit seed preparation

Seeds of calamansi, roselle, and jackfruit were collected from a self-planting garden located in Negeri Sembilan, Malaysia. The seeds were washed with distilled water and then dried with tissue paper to remove the excess water on the surface. The cleaned seeds were dried in oven at 75°C for 24 hours. The dried seeds (Fig. 1a) were crushed mechanically through pestle and motor. Grinded seeds were sieved through a 0.5mm sieves (Fig. 1b).

2.2 Oil extraction and oil yield determination

Oil was extracted from seed using Soxhlet method [10]. Fifteen grams of seeds were placed into a cellulose thumbed and extracted using 150 mL of n-hexane (99%, Systerm Chemicals) for 4 hours. The oil was then recovered using rotary evaporator (R-200, BUCHI, Malaysia) and the residual solvent was removed through drying in an oven at 80 °C for 1 hour. Oils obtained from calamansi, roselle and jackfruit seeds were kept in respective glass vial (Fig. 2). The extraction experiment was repeated using low-boiling point petroleum ether (Systerm Chemicals) as solvent. The experiment was conducted in duplicate for each solvent and result was expressed in mean value ± standard deviation (n=2).

To determine the percentage of oil yield, the mass of oil extracted from seed is divided by mass of seed used and multiplied with 100% as Eq. (1) [10,15]:

$$\text{Oil Yield (\%)} = \frac{W_{oil}}{W_{seed}} \times 100\% \quad (1)$$

where, W_{oil} (g) is mass of oil extracted and W_{seed} (g) is mass of seed.

Fig. 1 (a) Dried and (b) grinded seeds of calamansi, roselle and jackfruit.

Fig. 2 Extracted seed oils of calamansi, roselle and jackfruit.

2.3 FTIR analysis of seed oil

The Fourier Transform Infrared Spectra (FTIR) through attenuated total reflected (ATR) method was recorded using a Spectrum Two FT-IR Spectrometer (PerkinElmer, USA) with scanning resolution of 16 cm^{-1}. A sample quantity of seeds oil sample (approx. 8 microliters) was deposited on the diamond ATR crystal plate that was cleaned with pure chloroform to eliminate the presence of impurities between measurements and dried using nitrogen gas after each measurement. Spectra were recorded for all oil samples, scanned in the absorbance mode from 4000 to 400 cm^{-1}.

2.4 Heating effect evaluation

Oil sample was distributed into four glass vials and heated in an oven at a frying temperature of 185°C with heating durations of 20 min, 40 min, and 60 min. After the heating process, seeds oils were stored in dark for subsequent analyses of antioxidant activity, iodine value, saponification value, and peroxide

value. Due to extremely low oil yield of jackfruit seeds, hence only oils obtained from calamansi and roselle seeds were subjected to the analyses.

2.5 DPPH antioxidant activity

The antioxidant property of seed oils were assessed using 1,1-diphenyl-2-picrylhydrazyl (DPPH) radical scavenging assay. Results were reported as mean value ± standard deviation from triplicate determination. The DPPH free radical-scavenging assay was conducted according to method described by Parry et al. [16]. Seed oil of 0.5 g was added into 10 mL of methanol. Then the mixture was vortexed and centrifuged at 4300 rpm for 15 min to obtain the supernatant portion. The supernatant portion was further diluted to obtain concentrations of 5, 10, 15, and 20 mg/mL. 1 mL of each diluted concentration was mixed with 3 mL of 0.2mM of DPPH which has dissolved in methanol. The mixture was kept in dark for 30 min before measuring absorbance at 517 nm. The scavenging activity was calculated using Eq. (2). Solution of DPPH with no samples was used as the control. A supernatant portion with concentration of 5 mg/mL was used in determination of antioxidant activity of seed oils after heating duration of 20 min, 40 min, and 60 min at 185 °C.

$$\text{Scavenging Activity (\%)} = \left(\frac{A_c - A_s}{A_c}\right) \times 100\% \quad (2)$$

where A_c is the absorbance of the control, A_s is the absorbance of the sample.

2.6 Physicochemical properties determination

Seed oils obtained were characterized for iodine value, saponification value and peroxide value according to AOAC [17] standard procedure. Iodine value was determined by addition of solution of iodine monochloride into glacial acetic acid to induce uptake by unsaturated in oil followed by titration of excess halogen with sodium thiosulfate. Saponification value was determined by completely saponifying the oil sample under excess alkali ethanolic potassium hydroxide followed by titration of excess alkali with 0.5 M hydrochloric acid (HCL). Peroxide value was determined by addition of potassium iodide in chloroform-acetic mixtures to oil sample followed by titration of excess with sodium thiosulfate.

2.7 Seed oil fatty acids composition determination

The determination of fatty acids composition was performed using gas chromatography-flame ionization detection (Model No. 6820, Agilent Technologies, USA) attached with BPX-70 capillary column. Sample injection to be 1 µL, temperature of column oven was at 210°C with flow of nitrogen carrier gas to be 0.4 mL/min and flow of hydrogen to be 30 mL/min.

3 Results and Discussion

3.1 Percentage of oil yields extracted from calamansi, roselle and jackfruit seeds

The percentage of oil yield from seeds did not show significant difference using both solvents of n-hexane and petroleum ether (Table 1). Among the three types of seeds, calamansi seeds gave the highest oil yield, followed by roselle seed and small amount from jackfruit seeds.

The percentage of oil yields obtained in the present study using petroleum ether were compared to previous studies (Table 1). Manaf et al. [9] reported oil yield of 338.0 ± 11.3 g/kg from calamansi seed after extraction duration of 8 hours. Meanwhile, both Peng et al. [15] and Bouanga Kalou et al. [18] reported the percentage of oil yield of 23.8% and 23.27% obtained from roselle seed extracted after 8 hours. The difference in oil yields obtained between present study and Peng et al. [15] was probably due to the longer extraction duration and finer seeds sample of 0.3 mm used. On the other hand, Naeem et al. [10] reported 18.98% oil yield obtained from roselle seed but did not mention the extraction duration. The percentage of oil yield obtained from jackfruit seed in the present study was lesser than Madrigal-Aldana et al. [19], where they studied the jackfruit seed in two stages of ripeness, i.e., physiological mature and consumption ripeness. Seed oil yields obtained from the present study (Table 1) were also compared with oil yields from seeds of sacha inchi [20], fig [21], *Gmelina arborea* [22], *Maesa lanceolata* [23] and *Croton macrostachyus* [24] as they similarly performed oil extraction using Soxhlet apparatus and hexane as solvent but with different extraction time.

Table 1 Oil yields obtained from calamansi, roselle and jackfruit seeds via Soxhlet method using n-hexane and petroleum ether.

Seed Type	*n-Hexane*	*Petroleum Ether*	*Reference*
Calamansi	32.70 ± 0.63%a	31.67 ± 0.07%a	Present study
	-	338.0 ± 11.3 g/kg	[9]
Roselle	11.00 ± 0.20%b	10.30 ± 0.63%b	Present study
	18.98%	-	[10]
	-	23.8%	[15]
	-	23.27%	[18]
Jackfruit	0.60 ± 0.07%c	0.53 ± 0.00%c	Present study
	-	0.81% (physiological mature) 0.70% (consumption ripeness)	[19]
Sacha inchi	~45%	-	[20]
Fig	26.22 ± 1.42%	-	[21]
Gmelina arborea	35.1%	-	[22]
Maesa lanceolata	35.5%	-	[23]
Croton macrostachyus	45.89%	-	[24]

Mean value ± standard deviation (n=2)

3.2 Functional groups of calamansi, roselle and jackfruit seed oils

The major functional groups oils extracted from calamansi, roselle and jackfruit seeds were identified through FTIR spectrum (Fig. 3). Calamansi (Fig. 3a) and roselle (Fig. 3b) seed oils have shown similar peak characteristics. In the spectra obtained for calamansi and roselle seed oils in the diagnostic region, the peaks at 3008.32 cm^{-1} resulted from the vibrations of the cis olefinic CH double bonds (=C–H) [20]. While, 2923.86 cm^{-1} and 2855.53 cm^{-1} suggested the methylene (–C–H) asymmetrical and symmetrical stretchings. The proportion of polyunsaturated fatty acids in edible oil could be indicated by these vibrations. The degree of unsaturation was determined from the ratio of absorptions of peaks at 3008.32 cm^{-1} and 2855.53 cm^{-1} [20]. Therefore, a ratio of ~0.15 was calculated for both calamansi and roselle oil. The degree of unsaturation was lower than seed oil of sacha inchi (~0.60) [20], fig (~0.36) [21] and *Lepidium sativum* (~0.24) [25]. For peak at 1744.93 cm^{-1}, shows presence of carbonyl (C=O) stretching of esters. In the fingerprint region, peaks of 1460.41 cm^{-1} falls between region of 1465 cm^{-1} and 1450 cm^{-1} might be due to the presence of C-H bending of methyl or methylene group. As for peaks of 1375.29 cm^{-1}, it might be attributed by the presence of O-H bending of alcohol. Peaks identified at 1234.81 cm^{-1}, 1160.10 cm^{-1}, and 1096.4 cm^{-1} suggest the potential presence of C-N stretching of amine. Peak at 721.76 cm^{-1} might be due to C=C bending of alkene of di-substituted (cis).

Fig. 3(c) shows the FTIR spectra of jackfruit seed oil, the peak that lie on 3451.40 cm^{-1} indicates presence of alcohols and phenols of the –OH group detected [23]. This finding was in agreement with *Maesa lanceolata* seed oil by Bayisa & Bultum [23]. While for peaks of 2924.11 cm^{-1} and 2856.10 cm^{-1} suggested the presence of C-H stretching of alkane. Peaks of 1738.54 cm^{-1} refers to the presence of C=O stretching of ester. As for peak 1458.73 cm^{-1}, it is similar as to roselle oil as the peak fall between 1465 cm^{-1} and 1450 cm^{-1}, it was suggested that it may be due to presence of C-H bending of methyl or methylene group. While peaks from 1375.47 cm^{-1}, 1244.44 cm^{-1}, 1168.03 cm^{-1}, 1097.11 cm^{-1}, and 1029.51 cm^{-1} probably due to presence of C-N stretching which showed the presence of aromatic hydrocarbons and amines [21]. While for peak 889.78 cm^{-1}, 829.87 cm^{-1}, and 722.11 cm^{-1} it was suggested that it could potentially be contributed by C=C bending.

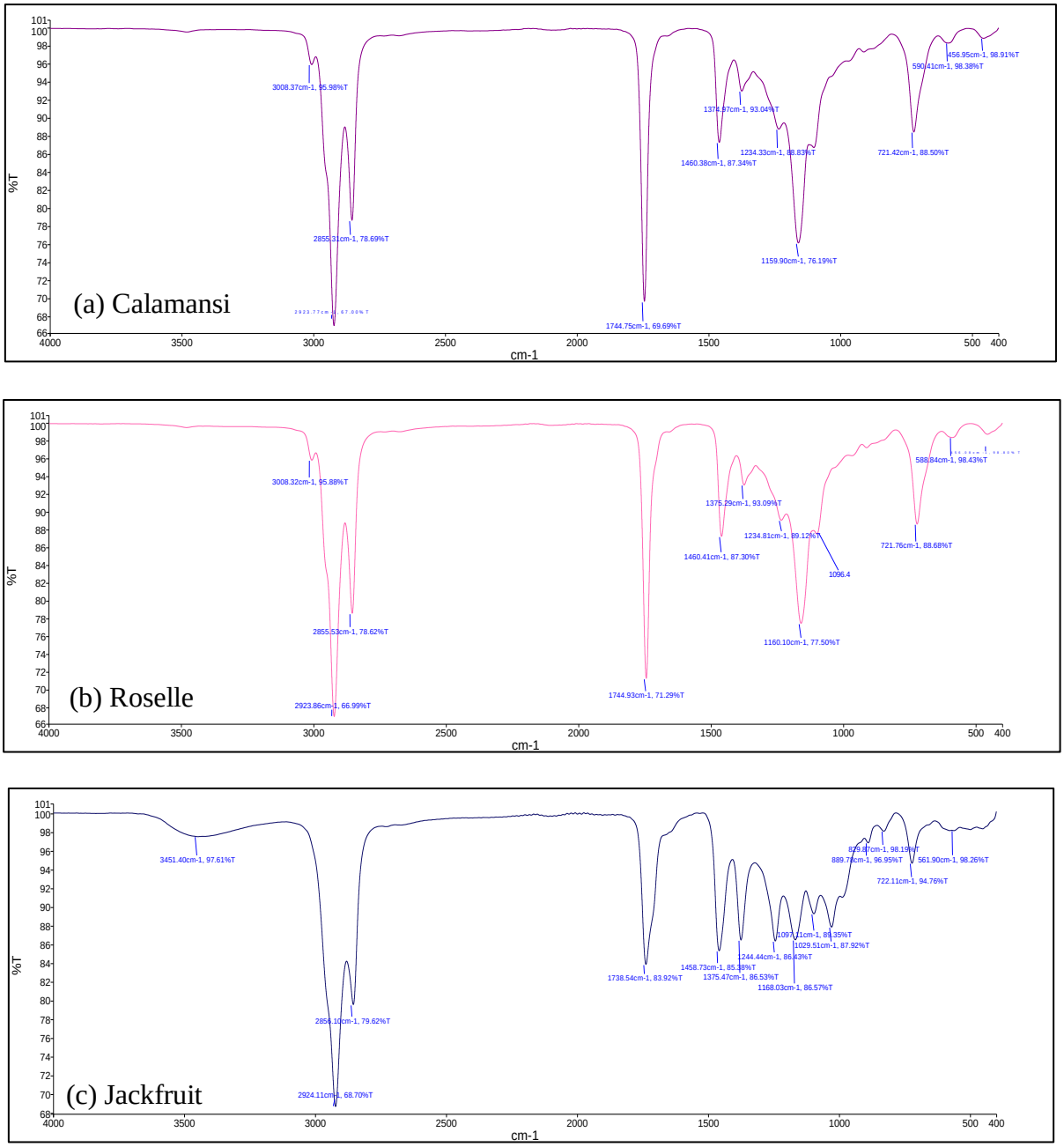

Fig. 3 FTIR spectroscopy of oils obtained from seeds of (a) calamansi, (b) roselle, and (c) jackfruit.

3.2 Antioxidant activity

The antioxidant activity of unheated calamansi and roselle seed oils both shows an increasing trend with increasing supernatant portion concentrations of 5, 10, 15 and 20 mg/mL (Fig. 4). This linear relationship was evidenced by high R^2 values of 0.9207 and 0.9618 for calamansi and roselle, respectively. Effective concentration (EC_{50}) was determined by substituting scavenging activity of y = 50% into the linear equations obtained in Fig. 4. EC_{50} indicates the effective concentration that would results in 50% scavenging activity. The calculated EC_{50} for calamansi and roselle seed oils were 109.74 mg/mL and 49.04 mg/mL, respectively. From the value of EC_{50}, it has clearly shown that roselle seed oil has greater antioxidant properties as compared to calamansi seed oil.

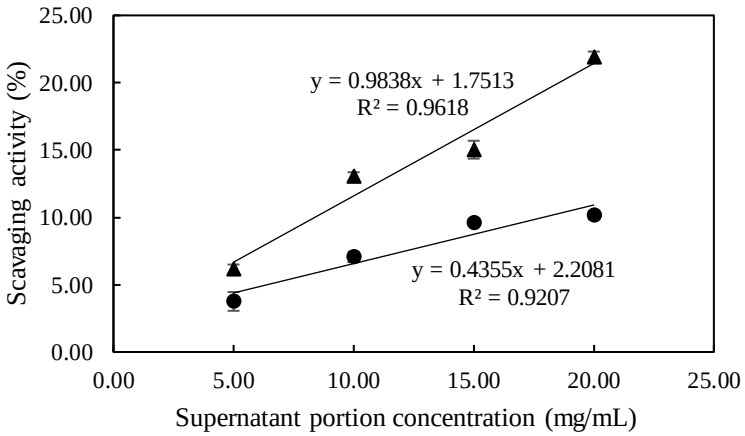

Fig. 4 Scavaging activity of unheated seed oils obtained from (●) calamansi and (▲) roselle.

As for heating effect both roselle and calamansi seed oil, a decrease in antioxidant activity was observed with the increasing heat duration under the temperature of 185 °C (Fig.5). Calamansi oil with 5mg/mL supernatant portion concentration, the scavenging activity was found decreasing to 2.74%, 2.18% and 2.13% after heating duration of 20 min, 40 min, and 60 min, respectively, in comparison to unheated oil of 3.76%. While for roselle oil, the scavenging activity was found decreasing to 5.43%, 5.33% and 4.37% after heating duration of 20 min, 40 min, and 60 min, respectively, as compared to unheated oil of 6.19%.

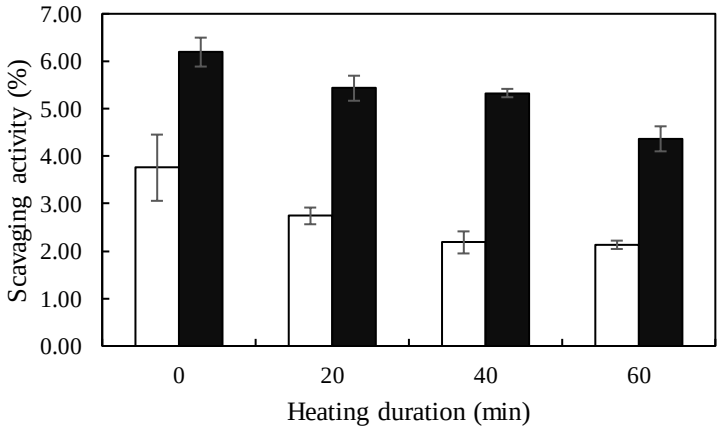

Fig. 5 Effect of heating duration on antioxidant activity of seed oils of (□) calamansi and (■) roselle, at 185°C.

Antioxidant capacity is varied with seed oil obtained from different extraction method and plant materials. Pu et al. [26] reported that *Majia* pomelo seed oil obtained via hot pressing, solvent extraction, and aqueous enzymatic extraction gave antioxidant activity EC_{50} of 4.06 mg/mL, 9.40 mg/mL, and 14.06 mg/mL, respectively. Boyapati et al. [27] discovered that the antioxidant activity of the optimized microwave-assisted extracted oil from the dragon fruit seed is found to be greater than that of the seed oil obtained from solvent extraction.

3.3 Physicochemical properties

Iodine value was expressed in term of gram iodine (I_2) absorbed per 100 grams of oil (g I_2/100g oil). Results showed that iodine value for unheated calamansi seed oil was 110.60 g I_2/100g oil and roselle seed oil was 100.83 g I_2/100g oil (Fig. 6). The lower iodine value indicates a lower degree of unsaturation in oil and thus, indicating a greater resistance against oxidation [28]. According to the classification of oils based on iodine value, both calamansi and roselle seed oils are belong to non-drying oil category [28] and have high unsaturated fatty acid contents [23]. However, comparing

between the iodine values obtained for calamansi and roselle seed oils, a high iodine value was observed for calamansi seed oil. Iodine values of calamansi and roselle seed oil obtained in this study were slightly lower than previous studies done by Maaf et al. [9] (118.1 g I_2/100g oil) and Bamgbove and Adejumo [29] (111.2 g I_2/100g oil).

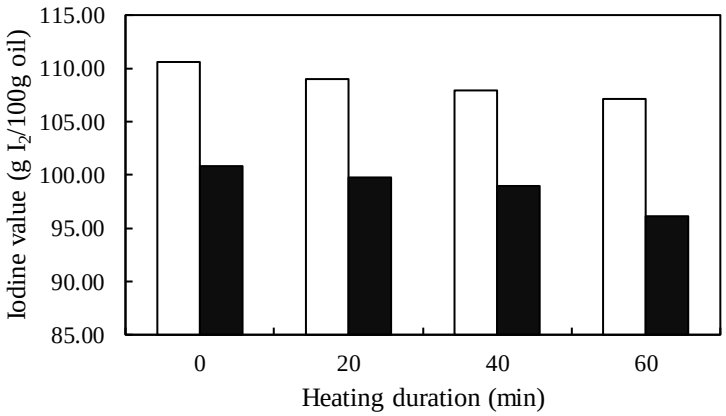

Fig. 6 Effect of heating duration on iodine value (IV) of seed oils of (□) calamansi and (■) roselle, at 185°C.

Both oils showed a decreasing trend of iodine value after heating for 20 min, 40 min, and 60 min at 185°C. This observation was in agreement with the study conducted by Bouanga-Kalou et al. [18]. They demonstrated that under a heating temperature of 200°C the iodine value of roselle seeds oil undergoes a decrease from 97.78 to 97.33 g I_2/100g oil upon 30 min of heating and further decreases to 96.85 g I_2/100g oil upon reaching 60 min of heating. They explained that the decrease in iodine value was contributed by the loss in unsaturation in the fatty acids of triacylglycerols upon heating.

Saponification value of seed oil was measured in order to understand alkali reactive groups in fats and oils and predicting the type of glycerides [23]. Oils of higher saponification value indicate that fatty acids exist in the glycerides of oil are of lower molecular weight with short-chain acids [30]. Saponification value is expressed in term of mg potassium (KOH) absorbed per gram of oil (mg KOH/g oil). The saponification value for both unheated calamansi and roselle seed oil were 191.50 and 194.19 mg KOH/g oil, respectively, and no significance reduction in saponification value was observed upon heating at 185 °C for 20, 40, and 60 min (Fig. 7). Saponification values obtained for both unheated calamansi and roselle seed oil from the present study were slightly different from previous studies done by Manaf et al. [9] and Bouanga-Kalou et al. [18] where they reported saponification values of calamansi and roselle seed oils of 192.6 and 198.45 mg KOH/g oil, respectively. Other seed oils such as sacha inchi [20] and fig [21] had saponification values of 190.2 and 201.6 mg KOH/g oil, respectively.

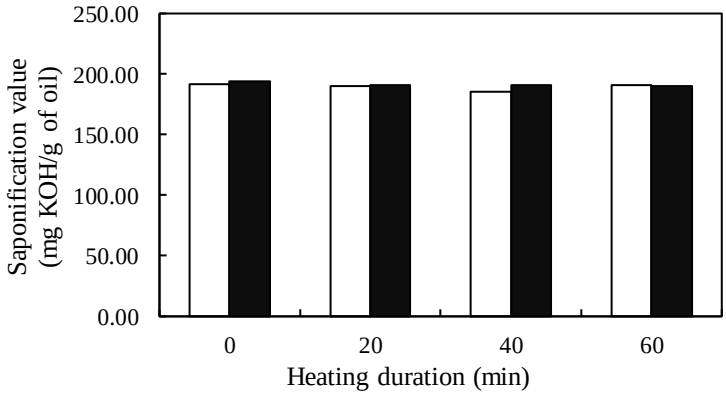

Fig. 7 Effect of heating duration on saponification value of seed oils of (□) calamansi and (■) roselle, at 185°C.

The peroxide value is another oil quality analysis and monitoring that gives a measurement that indicates the state of unsaturated oil. Unsaturated oil is susceptible to oxidation to form peroxides. Peroxide value could also be identified as a measurement of the rancidity and extent of oxidation that takes place on the lipids. Peroxide value is expressed in terms of milliequivalent of active oxygen per kilogram of oil (meq/kg). In this study, peroxide values obtained for unheated calamansi and roselle seed oil were 24.86 meq/kg and 10.36 meq/kg, respectively. The peroxide value is very much dependent on seed preparation and method employed in oil extraction, even though from the same seed species. Bamgboye and Adejumo [29] reported that the peroxide value of 6.0 to 9.3 meq/kg for roselle seed oil obtained from fine seed sample and 5.9 to 9 meq/kg obtained from coarse seed sample. However, the method utilised for the extraction of seeds oil was not mentioned. On the other hand, Eltayeib and Abd Elaziz [31] has reported a peroxide value of 4.6 meq/kg for roselle seeds oil extracted through solvent extraction using hexane. While, Naeem et al. [10] has reported the peroxide value of roselle seeds oil to be 1.01 meq/kg for oil extracted through supercritical CO_2, 1.59 meq/kg through hydraulic-press, 2.14 meq/kg through screw-press and 4.57 meq/kg through solvent extraction with hexane. Abdullahi et al. [32] who used solvent extraction with hexane has reported a peroxide value of 4.33 meq/kg. According to Codex Alimentarius Commission [33], the maximum peroxide value for virgin oils and cold pressed fats and oils is 15 meq/kg, and for other fats and oil is 10 meq/kg. As oil extraction was not by mechanical means or without application of heat, both roselle and calamansi seeds oil was classified under the other fats and oil categories. By referring to this peroxide value classification, both calamansi and roselle seed oils obtained in the present study were not within the limit to be classified as edible oil.

3.4 Fatty acids compositions

A spectrum of calamansi and roselle seeds oil were obtained from gas chromatography and fatty acid compositions were calculated from area of each peak over the total area of all peaks. A total of seven fatty acid compositions have been identified in calamansi seed oil as tabulated in Table 2. The fatty acid compositions identified of calamansi seed oil identified in this study were differed from previous study done by Manaf et al. [9]. They reported that the major fatty acids in calamansi seed oil were linoleic acid (31.8%), oleic acid (29.6%), palmitic acid (21.4%), and linolenic acid (7.8%). The difference fatty acid compositions detected was most probably due to different model of GC analyzer and column used. In the present study, gas chromatography-flame ionization detection (Model No. 6820, Agilent Technologies, USA) attached with BPX-70 capillary column was used. Meanwhile, Manaf et al. [9] used Ultrafast GC zNose 7100 analyzer with column (DB-5) where temperature was programmed from 40 to 200 °C and the surface acoustic wave quartz microbalance detector temperature was set at 60 °C. On the other hand, only three major fatty acid compositions have been detected in roselle seed oil. There were 46.93% of eicosapentainoic acid, 29.51% of linolelaidic acid, and 23.56% of tricosanoic acid as tabulated in Table 2. Results obtained from this study was totally different from previous work done by Naeem et al. [10] where they reported that linoleic acid, oleic acid, and palmitic acid were the major fatty acid compositions in roselle seed oil. Hence, in order to confirm the fatty acid compositions of calamansi and roselle seed oil, high performance liquid chromatography is recommended.

Table 2 Fatty acid composition (%) of seed oils.

Fatty Acids	*Calamansi*	*Roselle*
Linolelaidic (C18:2n6t)	25.59	29.51
Linoleic (C18:2n6c)	4.06	-
Arachidonic (C20:4n6)	4.55	-
Tricosanoic (C23:0)	18.29	23.56
Docosadienoic (C22:2)	9.16	-
Eicosapentaenoic(C20:5n3)	30.72	46.93
Nervonic (C24:1)	7.64	-

4 Conclusion

The results show that the extraction solvent affect the yield but both hexane and petroleum ether showed considerably close yield due to the similar in polarity index. Calamansi seed has the higher oil content followed by roselle and lastly, jackfruit. Comparing between calamansi and roselle seed oils, roselle seed oil was concluded to have greater antioxidant properties. Iodine value obtained revealed that both oils was classified under the non-drying oils categories. While, heating has caused a reduction in iodine value due to loss of unsaturation. For saponification value obtained that reflect the average molecular weight of oil, the saponification value for both roselle and calamansi seeds oil under unheated condition was 194.19 mg KOH/g oil. As for heating effect, both oils showed no significant changes upon heated on the designed temperature. As for peroxide value, roselle and calamansi seed oils under unheated condition was 10.36 meq/kg and 24.86 meq/kg. This indicated both calamansi and roselle seed oils were concluded to be not within the edible oil limit. Hence, their application such as biodiesel should be further explored.

Declaration of Conflict of Interest

The authors declared that there is no conflict of interest with any other party on the publication of the current work.

ORCID

Choon Yoong Cheok https://orcid.org/0000-0002-2947-9436

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