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Variety of Watermelon and Method of Drying Affect the Chemical and Functional Characteristics of Oils Extracted from Watermelon Seeds

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ABSTRACT

Utilization of watermelon seeds as a source of oil production can help reduce the waste generated from its consumption and also provide an alternative source of domestic or industrial oil to peanut and palm nut oil in Ghana. The objective of this study was to investigate the effect of the variety and the mode of drying on some chemical and functional properties of oils extracted from the dried seeds of two varieties of watermelon (Crimson sweet and Black seeded ice cream). Seeds of the two varieties were subjected to different drying conditions (sun-drying and oven-drying) after which oils were extracted from the dried seeds using the Soxhlet apparatus. Chemical and functional properties investigated in the extracted oils were acid value/free fatty acid, saponification value, iodine value, ester value, peroxide value, water absorption capacity, emulsion stability and ability. Results of the study showed that the oil extracted from oven-dried crimson sweet had the lowest acid value, free fatty acid and saponification value. It was observed that the acid value, free fatty acid, peroxide value and saponification value are significantly affected by the drying method and to a lesser extent the variety of watermelon used. Figures obtained for the acid value, free fatty acid, saponification value and ester value of the crude oils were within the standard range for edible oils.

Keywords: *chemical properties, functional properties, edible oils, sun-drying, oven-drying, watermelons, variety.*

INTRODUCTION

Watermelon (*Citrullus lanatus*), which belongs to the family Cucurbiticeae, is a major fruit widely distributed in the tropics [1]. The fruit serves as a thirst-quencher due to its high moisture content(92%)[2]. Watermelon is an herbaceous creeping plant and it is mainly propagated by seeds. Its propagation requires a temperature of over 25 °C and thrives best in fairly acidic and drained fertile soil [3]. In Ghana, several watermelon varieties can be grown along the coastal areas, forest zones and along river beds in the Northern Savannah areas. There are several varieties of watermelons characterised by their round, oval or oblong fruit shape. Rind colour may range from light green to very dark green which may or may not be patterned or striped; yellow, red or orange flesh; and varying sizes of white, brown, black, red, green or mottled seeds which are flat and smooth [4].The seeds of watermelon contain phyto chemicals such as saponins, alkaloids, phenols, flavonoids and tannins [5]which have antioxidant, anti-inflammatory, antimicrobial, anti-infection and anticancer properties [6].

Watermelon seeds are also known to be rich sources of proteins, vitamins, minerals (such as magnesium, potassium, phosphorous, sodium, iron, zinc, manganese and copper) and essential fatty acids and oils[5]. The seeds are sometimes milled and used for making sauces or snacks. Watermelon seeds and rinds can serve as food source yet they are discarded adding up to the agricultural wastes in the environment[7, 8]. The seeds contain oils which may have comparable or even better physical and functional properties than oils sourced from nuts which are predominantly used domestically and industrially [9]. The qualities of oils are ascertained using their functional and chemical properties such as iodine value, saponification value, free fatty acid, peroxide value, emulsion

stability & activity and water holding capacity [10]. These functional and chemical properties (quality) of the oils are affected by temperature, light and oxygen during their processing [11]. The quality of vegetable oil has also been reported to be dictated by the source of the oil and storage conditions [12]. Several researchers have confirmed oxidation of oils as a major contributing factor to the reduction of quality caused by; processing procedures, temperature, light and oxygen [11, 13].

With respect to edible oils, the type of raw oil, its colour, free fatty acid content, taste, other physical and chemical properties and other parameters need specific attention in order to obtain the much needed quality of the finished product [14]. Majority of the major edible oils on the Ghanaian markets are obtained from either palm nut fruits or groundnuts/peanut. However, several foodstuffs like watermelons become abundant and most of them go waste during their peak seasons since they are perishable and some parts are also not consumed. There is therefore the need to look for good and novel alternative sources of oils that would be useful domestically and perhaps industrially and at the same time address the issue of waste generated from unconsumed portions of certain foodstuffs.

This study therefore sort to investigate the effect variety and processing method (drying) had on the functional and chemical properties of oils extracted from dried watermelon seeds and also evaluate the viability of the extracted oils as an alternative source of oils to that obtained from peanut and palm nut fruit. This was done by drying (in the sun and oven) seeds from these two watermelon varieties (crimson sweet and black seeded ice cream), extracting oils from the dried seeds and then evaluating the functional (emulsion activity & stability and water absorption capacity) and chemical properties (saponification value, free fatty acids/acid value, peroxide value and iodine value) of the extracted oils.

Materials and Methods

Acquisition and Preparation of Samples

The two watermelon varieties, crimson sweet and black seeded ice-cream were purchased from Winneba in the Central Region of Ghana. The fruits were washed and sliced open using clean stainless steel laboratory knife. The seeds were removed from the flesh and washed severally with clean water. The seeds from each variety were divided into two groups and afterwards weighed; one group from each variety was dried in the sun for 7 days while, the other group from each variety was dried in an oven at 70°C for 72 hours. The dried seeds from each treatment group were milled using an industrial blender and kept in desiccators for further analysis.

Extraction of Crude Oil by Soxhlet Method

An amount of 80g of the powdered seeds were weighed into a medium sized thimble and carefully loaded into the main chamber of a soxhlet extractor. A volume of 645mL of n-hexane was measured and poured into a round bottom flask mounted beneath the soxhlet main chamber. The flask was fixed onto a heating mantle and heated for 5 hours at 68 ^{0}C until no visible trace of oil was seen in the main chamber. The round bottom flask was carefully removed and the content transferred into a quick-fit bottom flask for rotary evaporation. The heating pan of the rotary evaporator was filled with cold water and set at 60 ^{0}C and the quick-fit receiver flask was mounted at the receiver end of the rotary evaporator. As the heating pan and rotary evaporator were switched on, the solvent evaporated and condensed back into the quick-fit receiver. The process was carefully monitored until all the solvent condensed into the quick-fit receiver. The crude oil left in the quick-fit bottom flask was transferred into an Erlenmeyer flask and allowed to stand uncorked for about 30 minutes before corking. The weights of the crude oils extracted from the four samples were recorded.

Determination of Chemical Parameters of the Sample Oils

Acid Value/Free Fatty Acids

A total volume of 75mL of petroleum ether was mixed with 75mL of ethanol in a 250mL conical flask. A volume of 10mL of the mixture was measured and transferred into three different 100mL conical flasks. To each of the three conical flasks, 0.4g of oil sample was added and mixed thoroughly. One drop of phenolphthalein was added as an indicator, and the solutions were titrated against aqueous 0.1M KOH by dissolving 2.2442g of KOH in 400mL of distilled water, until a pink colour persisted for 15 seconds. This procedure was repeated for the other three oil samples. The acid value in mg KOH/g was determined using the expression;

$$\frac{Titre\ value(L) \times Concentration\ of\ KOH(M) \times 56.1}{sample\ weight\ (g)}$$

The free fatty acid was calculated by dividing the acid value by two (2).

Saponification Value

An amount of 0.2g of the oil sample was weighed into a 25mL conical flask and a volume of 5mL of ethanoic potassium hydroxide was added and stirred thoroughly. The resulting mixture was then heated on a hot plate for 5min at 100 ^{0}C until the oil dissolved. One drop of phenolphthalein indicator was added to the solution and titrated against 0.5M HCl solution while shaking constantly until a faint pink colour persisted for 30seconds and the end points recorded. This procedure was repeated for the other oil samples. The saponification value in mg KOH/g of the oil samples was found by using the expression;

$$\frac{\left[\frac{Blank - titre\ volume}{L} \times Conc(KOH) \times Mr\ (KOH) \times Reagent\ factor(1.006) \times 1000\right]}{\text{sample weight}}$$

Peroxide Value

Peroxide value was analysed using American Oil Chemists' Society (AOCS) Official Methods [15]with slight modifications. To a mass of 1g of the watermelon seed oil in a flask,a total volume of 6mL of acetic acid/chloroform solution (3:2, v/v) was added. This was followed by the addition of 0.1ml saturated potassium iodide (KI) and the solution was allowed to stand for 1 minute while occasionally shaking the flask. A total volume of 6mL of distilled water was added, and the solution titrated against 0.012N sodium thiosulfate until the yellow colour almost disappeared. A volume of 0.1mL of starch indicator was added and the titration continued until the blue colour disappeared and the end-points noted. A blank was prepared with all the reagents except the oil sample. This procedure was repeated for the other oil samples and the peroxide value in meq/kg calculated using the expression;

$$\frac{(titre\ volume(ml) \times Concentrationof\ thiosulfate(M) \times 1000)}{Weight\ of\ sample\ (g)}$$

Iodine Value

Iodine value was analysed using the American Oil Chemists' Society (AOCS) Official Method [15] with slight modifications. To a conical flasks containing 0.1g of the oil sample, a total volume of 3.75mL of chloroform solution was added. This was followed by the addition of 6.25mL of WIJ's reagent and stored in the dark for 30 minutes. Once the flask was removed from storage, 5mL of KI solution was added, followed by 25 mL of distilled water. The solution was titrated against 0.IM sodium thiosulfate until the yellow colour almost disappeared. A volume of 1mL of starch indicator was added to discontinue the titration when a blue colour just disappeared, and the end point recorded.

This procedure was repeated for the other oil samples and on a blank sample, (contained all the stated reagents and solutions except the oil sample). The iodine value in gI_2/100g of oil samples was found using the expression;

$$\frac{[(ml\ Blank - titre\ volume) \times Conc\ of\ thiosulfate(M) \times 12.69]}{Weight\ of\ sample\ (g)}$$

Analysis of Functional Properties of the Oil Samples

Emulsion Activity and Stability

The emulsion activity and stability of the oil samples were determined by the method of [16]with some slight modifications. Solutions consisting; 0.5g of each oil sample, 10mL of distilled water and 10mL of soybean oil were prepared in separate calibrated centrifuge tubes. The solution in each tube was centrifuged at 3000 rpm (2000× g) for 5 min. The ratio of the height of the emulsion layer to the total height of the mixture was calculated as emulsion activity in percentage, thus using the expression;

$$\frac{Height\ of\ emulsion\ layer}{Total\ height\ of\ the\ mixture} \times 100$$

For emulsion stability, solution in each tube was heated at 80 ^{0}C for 30 min in a water bath. After cooling for 15 min in a cold water bath, the content was centrifuged at 3,000 rpm (2000 × g) for 15 min. The emulsion stability expressed as a percentage was calculated as the ratio of the height of the emulsion layer to the total height of the mixture, thus using the expression;

$$\frac{Height\ of\ emulsion\ layer}{Total\ height\ of\ the\ mixture} \times 100$$

Water Absorption Capacity

Water Absorption Capacity of the oil samples were determined by the method of Sosulki *et al.*,[17] with some slight modifications. An amount of 0.5g of each of the oil samples was mixed separately with 5mL distilled water and weighed in pre-weighed plastic centrifuge tubes. The contents were allowed to stand at an ambient temperature (30^{0}C) for 30min, and centrifuged at 3,000 rpm (2000 × g) for 30 min. Just after centrifugation, the supernatant of each tube was carefully decanted and the new mass of the oil samples were recorded by weighing the plastic centrifuge tube after decantation. The water absorption capacity of the oil samples expressed as a percentage was found using the expression;

$$\frac{Mass\ of\ oil\ after\ centrifugation - Mass\ of\ oil\ before\ centrifugation}{Mass\ of\ oil\ before\ centrifugation} \times 100$$

Data Analysis

Data obtained from the test analysis were analyzed statistically. Statistical Package for the Social Scientist (SPSS) version 20.0 was used for the analysis. The laboratory data were submitted to analysis of variance using one-way ANOVA and was applied to distinguish between means that were statistically different (P<0.05)

Table1. *Chemical properties of oils extracted from seeds of crimson sweet and black seeded ice cream watermelon subjected to sun and oven drying*

Chemical Property	Sample			
	CSs	CSo	BSICs	BSICo
Acid value (mgKOH/g)	8.42±0.24^{abc}	2.62±0.39^{a}	4.07±0.42^{b}	2.79±0.14^{c}
Free fatty acid (mgKOH/g)	4.21±0.12^{abc}	1.31±0.20^{a}	2.04±0.21^{b}	1.40±0.07^{c}
Saponification value (mgKOH/g)	28.04±2.14^{ab}	17.76±1.87^{a}	24.37±1.64^{c}	17.78±1.33^{bc}
Iodine value (gI2/100g)	7.74±0.90	14.03±2.00	7.13±0.85	9.70±0.80
Peroxide value (meq./Kg)	11.61±1.03^{ab}	6.60±1.12^{ac}	12.13±0.35^{cd}	5.40±0.30^{bd}
Ester value (mgKOH/g)	19.60±1.81	15.17±1.81	20.30±2.05	14.99±0.37

Values are means ± standard deviations of the means of triplicate determinations. Means with the same superscript within a particular row are significantly different (P<0.05).

RESULTS

Chemical Analysis of Oil Extracted from Seeds of Crimson Sweet and Black Seeded Ice Cream Watermelon Subjected to Sun-Drying and Oven Drying

The chemical parameters obtained for the oil extracted from sundried crimson sweet seeds (CSs) and sundried black seeded ice cream (BSICs) were all greater than the values obtained for oil extracted from oven dried crimson sweet seeds (CSo) and oven dried black seeded ice cream (BSICo) except the iodine values for CSs and BSICs which were lower than those of CSo and BSICo respectively (Table 1). However, the saponification values and peroxide values of CSs and BSICs were significantly higher than the corresponding values of CSo and BSICo (P<0.05) (Table 1). The acid value and free fatty acid value of CSs were also significantly higher than the corresponding values of CSo and the respective values of BSICs (P<0.05) (Table 1).

Functional Properties of Oil Extracted from Crimson Sweet and Black Seeded Ice Cream Watermelon Subjected to Sun-Drying and Oven Drying

The water absorption capacity (WAC), emulsion activity (EA) and emulsion stability (ES) of CSs were higher than those of CSo (Table 2). The water absorption capacity (WAC) and emulsion activity (EA) of BSICs were also higher than those of BSICo, whereas the emulsion stability (ES) of BSICs was lower than that of BSICo (Table 2).

Table2. *Functional Properties of oil extracted from crimson sweet and black seeded ice cream watermelon subjected to sun and oven drying*

Functional Properties	Sample			
	CSs	CSo	BSICs	BSICo
WAC (%)	41.07±2.06	34.53±1.03	37.76±0.84	35.47±0.88
EA (%)	38.20±0.56	37.07±0.95	38.23±0.85	37.07±1.11
ES (%)	38.90±0.60	36.10±1.25	37.83±1.10	37.93±1.17

Values are means ± standard deviations of the means of triplicate determinations.

DISCUSSION

Most often the seeds of watermelon (*Citrullus lanatus*) are discarded as food waste while they can serve as sources of food or oils which may be domestically and industrially useful. This study investigated the effect of the method of drying (oven and sun) and variety of watermelon on the functional and chemical properties of oils extracted from the dried seeds of crimson sweet and black seeded ice cream watermelon. The quality of oil is very important as it is the major determinant of its desirable use. Shadihi & Zhong, [18] reported that the quality of vegetable oil is dictated by several physical and chemical parameters that are dependent on the source of oil, processing, and storage conditions. The drying conditions had significant effects on the acid value, free fatty acid value, saponification and peroxide value of oil extracted from the two varieties of watermelon.

The acid values, free fatty acid values and peroxide values obtained in this study for sundried crimson sweet seed oil (8.42 mg KOH/g, 4.21 mg KOH/g & 11.61meq/Kg) and sundried black seeded ice cream seed oil (4.07 mg KOH/g, 2.04 mg KOH/g & 12.13meq/Kg) were higher than the values obtained for oven dried crimson sweet seed oil (2.62 mg KOH/g, 1.31 mg KOH/g & 6.60meq/Kg) and oven dried black seeded ice cream seed oil (2.79 mg KOH/g, 1.40 mg KOH/g & 5.4meq/Kg) respectively.

The peroxide values of the sundried seed oils of both varieties and the acid value and free fatty acid value of sundried crimson sweet seed oil were significantly higher than those obtained for their respective oven dried seed oils ($P<0.05$). The acid values of the sundried crimson sweet seed oil were also significantly higher as compared to the values obtained for the sundried black seeded ice cream seed oil ($P<0.05$). The acid values and peroxide values of the oven dried crimson sweet seed oil were closer to those of the oven dried black seeded ice cream seed oil, however, the peroxide value of the oven dried crimson sweet seed oil was a little higher than that of the oven dried black seeded ice cream oil. Taiwo *et al.*,[19] obtained an acid value and peroxide value of 8.89mgKOH/g and 18.75% for sundried melon seed oil and 13.40mgKOH/g and 18.75% for oven dried melon seed oil respectively. The values from *al.*,[19] show a lower acid value for sundried melon seed oil as compared to the oven dried and equal peroxide values for sundried and oven dried melon seed oils and these were contrary to what was obtained in this study.

Acid values, free fatty acid values and peroxide values are used to determine the quality of oils since they determine the extent of deterioration of the oils. The results obtained in this study suggests that sun drying has detrimental effects to the oils since the oils from sundried seeds showed higher acid values and peroxide values than the oils from the oven dried seeds. Exposing the sundried seeds to sunlight and atmospheric oxygen which are prerequisite elements for oxidation of oil could have resulted in the high acid and peroxide values [20]. However, the acid values reported in this study were within the accepted range (≤10.00 mg KOH/g) for edible oil as reported by Oyekele *et al.*,[21]. The results obtained in this study also suggests that the different varieties of melon seed oils have different stabilities against rancidity with black seeded ice cream seed oil having the highest stability against rancidity. Razik *et al.*,[22] also observed significant differences between peroxide values for different varieties of melon seed oils.

The saponification values and ester values of sundried crimson sweet seed oil (28.04 mg KOH/g &19.60 mg KOH/g) and sundried black seeded ice cream seed oil (24.37 mg KOH/g & 20.30 mg KOH/g) were higher than the values obtained for oven dried crimson sweet seed oil

(17.76 mg KOH/g & 15.17 mg KOH/g) and oven dried black seeded ice cream seed oil (17.78 mgKOH/g, 14.99 mg KOH/g) respectively with the saponification values of the sundried seed oils being significantly higher than those of their respective oven dried variety ($P<0.05$). However, iodine values for sundried crimson sweet seed oil (7.74 $gI_2/100g$) and sundried black seeded ice cream seed oil (7.10 $gI_2/100g$) were lower than the values obtained for oven dried crimson sweet seed oil (14.03 $gI_2/100g$) and oven dried black seeded ice cream seed oil (9.70 $gI_2/100g$). According to Jones & King, [23] and Osagie *et al.*,[24], iodine value is an index of unsaturation, the ability of oil to go rancid and the molecular size of fatty acid contents of oils, thus, marks the degree of stability of oils. The reduction of the iodine values in the sundried crimson sweet seed oil as compared to their oven dried variety may be due to the fact that upon exposure to atmospheric oxygen, the unsaturated bonds in the oil underwent oxidation. The saponification values obtained in the study were much lower than those reported in other works on watermelon seed oils [19, 25, 26]however, the values obtained in this study were closer to 21.09, 19.41 and 21.54mg KOH/g reported for sunflower oil, corn oil and olive oil respectively by Alajtal *et al.*,[27]. Akintayo & Bayer, [28] reported that low saponification values indicate the preponderance of long chain fatty acids in the oil and vice versa. Adebanjo & Kehinde, [29]also reported that saponification values less than 100 indicates fatty acids with low foaming capacities hence not suitable for making detergents or foaming agents.

Water absorption capacity (WAC), emulsification activity (EA) and emulsification stability (ES) of sundried crimson sweet seed oil (41.07%, 38.20% & 38.90%) were higher than those obtained for oven dried crimson sweet seed oil (34.53%, 37.07% & 36.10%) respectively. The WAC, EA and ES of the sundried black seeded ice cream seed oil (37.76%, 38.23% & 37.83%) were also higher than the values of oven dried black seeded ice cream seed oil (35.47%, 37.07% & 37.93%) except that of the ES. Chandra *et al.*,[30] stated that an appreciable increase in amounts of emulsion activity and stability are primary functional properties in comminute meat products, salad dressing and frozen desserts. Chandra *et al.*,[30], also reported that variation in water absorption capacities of different flours could be due to different concentrations, degree of interaction with water and conformational characteristics of proteins in the flour. Kuntz, [31]reported that the lower water holding capacity of some flours may be as a result of unavailability of polar amino acids. Therefore variations in water absorption capacities of oils could also be attributed to nature of fatty acids in the oil and this could account for the higher water absorption capacities obtained for the sundried melon seed oils in this study since the oxidation and/or hydrolysis of esterified fatty acids in sundried seed oils could have resulted in the production of polar fatty acids and/or derivatives that are more polar.

CONCLUSION

The present study observed that the acid value, free fatty acid, peroxide value and saponification value were significantly affected by the drying method and to a lesser extent the variety of watermelon used. Figures obtained for the acid value, free fatty acid, saponification value and ester value of the crude oils were within the standard range for edible oils.

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