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Some Physicochemical Properties of the Petroleum Ether-Extracted Watermelon (*Citrullus lanatus*) Seed Oil

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ABSTRACT

Increasing the dietary and industrial utilization of food wastes could ultimately improve the environment. Thus, watermelon (*Citrullus lanatus*) seed oil, extracted with petroleum ether (boiling range 60-80°C for 8 h) using Soxhlet extractor and separated from the solvent with a rotary evaporator was studied, using standard protocols. Result of the study showed these properties of the seed oil viz: specific gravity (0.87), color (light yellow), pH (4.9) and texture at 37°C (liquid). The peroxide value (2.80 meq kg^{-1}) of the watermelon seed oil was lower than 10 meq kg^{-1} required for the standard oils. The saponification value (205.3 mg KOH g^{-1}) of the watermelon seed oil compared with the range for the standard oils (except that for coconut oil). The iodine value (28.51 g/100 g) was lower than the values for standard oils (except that for coconut oil). However, the acid value (6.10 mg KOH g^{-1}) and the free fatty acid value (4.61 mg KOH g^{-1}) of the oil sample were higher than the corresponding value for the standard oils. The watermelon seed oil properties compared fairly with that of the standard edible and industrial oil sources, suggesting its stability, edibility and industrial potentials. However, the comparatively high acid and free fatty acid values, supported by the low pH value could compromise the watermelon seed oil qualities and benefits. Further studies aimed at reducing the high acid and free fatty acid values of the oil sample could enhance the potential uses of the seed oil and reduce the attendant environment burden of the watermelon seed, hence are warranted.

Key words: Iodine value, acid value, saponification value, peroxide value, free fatty acid value

INTRODUCTION

Reports of search for plants and plant parts with pharmacologic (Anderson *et al*., 2001; Egbuonu and Nwankwo, 2012), nutrient and industrial (Egbuonu, 2015) potentials abound. Vegetable oils account for 80% of the world's natural oils and fat supply (FAO., 2007) with increasing importance in nutrition and commerce owing to their dietary energy, antioxidant, bio-fuels and raw materials potentials (Fasina and Colley, 2008) and the attendant increasing global demand. Thus, there is need for further search in this and related areas, including hitherto food wastes.

In Nigeria, the pulp of various fruits but not the rind and seed, is usually consumed. This contributes to food wastes in the environment as is the case with watermelon (family Cucurbitaceae and specie *Citrullus lanatus*), a major fruit widely distributed in the tropics that serves as a thirst-quencher owing to its high (92%) water content (Yamaguchi, 2006; Ensminger and

Ensminger, 1986). To prevent solid waste related hazards to the environment, effort should be made to increase the utilization of food wastes which requires studies on their properties for scientific basis. Studies on watermelon fruits were reported but mainly on the juice/pulp (Johnson *et al.*, 2012; Oseni and Okoye, 2013) and a little on the peel/rind (Fila *et al.*, 2013; Gin *et al.,* 2014). Studies on the seed were essentially on the flour (Egbuonu, 2015).

Knowledge of the physicochemical properties of the watermelon seed oil may offer insight into the nature and potential benefits of the oil. For instance, pH value, an index of the concentration of hydrogen ion [H^+] in water aside indicating the acidic or basic nature of an oil sample suggests that the oil contains materials, including water that could compromise its purity, qualities and benefits. This is so because oil in a pure state is not an ionizing solvent, does neither contain water nor supply hydrogen ions and hence does not have a pH. These warranted the present study aimed at assessing some physicochemical properties of watermelon (*Citrullus lanatus*) seed oil extract.

MATERIALS AND METHODS

Collection and preparation of samples: Watermelon fruits were bought from Onuimo market, in Imo State border/boundary with Abia State, Nigeria. It was identified as Charleston gray variety in the Department of Plant Science and Biotechnology, Michael Okpara University of Agriculture, Umudike, Nigeria. The watermelon was thoroughly washed to remove sand particles after which it was sliced using a home choice European knife. The seeds were handpicked and washed off the pulp particles using clean water.

The seeds were weighed, using Satorious Digital Weighing Balance, Model BP210S, Germany. The seeds (wet weight = 1016.9 g) were separately spread on a foil and sundried to obtain the corresponding dry weight (468.5 g). The dry weight sample was milled into powder using Arthur Thomas Laboratory Mill, Crypto model, USA, covered in a labeled white nylon and kept in the desiccators until used.

Chemicals and reagents: All chemicals used, including those used in the preparation of reagents, were of analytical grade and products of reputable companies.

Extraction of the seed oil: The oil sample was extracted from the seed flour with petroleum ether (boiling range 60-80°C for 8 h) using Soxhlet extractor. After complete extraction (when the extracting solution became clear), the oil was separated from the solvent using a rotary evaporator, poured into a beaker and left for 5 days for the remaining solvent to evaporate. The oil sample was stored in a refrigerator using tight-stoppered brown bottles.

Physico-chemical analysis of the watermelon seed oil sample

Determination of specific gravity (at 27±0.5°C), pH, color and texture: The specific gravity was determined at 27±0.5°C using a 50 mL bottle pycnometer that was washed thoroughly with detergent, water and petroleum ether according to method described in AOAC (2005). The color was identified by simple observation. The pH was determined with a pH meter while the texture was determined by measuring the temperature, using a thermometer, at which the oil (brought out from the refrigerator and placed in a glass beaker) changed from solid to liquid.

Determination of acid value and free fatty acid value: The acid value of the oil sample was determined using ASTM method (ASTM., 1998) and the free fatty acid calculated.

Twenty five milli liter diethyl ether with 25 mL alcohol and 1 mL phenolphthalein solution (1%) was mixed and carefully neutralized with 0.1 M NaOH. One to ten gram of the oil was dissolved in the mixed neutral solvent and titrated with aqueous 0.1 M NaOH, shaken constantly until a pink color which persists for 15 sec was obtained. The acid value was calculated using the relation:

$$\text{Acid value} = \frac{\text{Titre (mL)} \times 5.61}{\text{Weight of sample}}$$

The free fatty acid value was calculated from the relation:

$$\text{Free fatty acid} = \text{Acid value} \times 0.503$$

Determination of specific gravity, saponification, peroxide and iodine values: The specific gravity, peroxide and saponification values of the watermelon seed oil extract were determined as described by methods in AOAC (2005). The iodine value of the seed oil was measured using Wijis solution as described in AOAC (1984).

Data analysis: Data was expressed as simple mean of duplicate test values.

RESULTS AND DISCUSSION

Result of the analysis showed these properties of the seed oil viz: specific gravity (0.87), color (light yellow), pH (4.9) and texture (liquid at 37°C) (Table 1).

The saponification value (205.3 mg KOH g^{-1}) of the watermelon seed oil compared with the range for the standard oils (except that for coconut oil). The iodine value (28.51 g/100 g) was lower than the values for standard oils (except that for coconut oil) while the peroxide value (2.8 meq kg^{-1}) was less than 10 meq kg^{-1} required for the standard oils. However, the acid value (6.10 mg KOH g^{-1}) and the free fatty acid value (4.61 mg KOH g^{-1}) of the oil sample were higher than the corresponding value for the standard oils (Table 2).

Table 1: Some physicochemical characteristics of the watermelon (*Citrullus lanatus*) seed oil

Parameters	Seed oil sample characteristic/value
Specific gravity (25°C)	0.87
pH	4.9
Color	Light yellow
Texture (at 37°C)	Liquid

Table 2: Other physicochemical characteristics of the watermelon (*Citrullus lanatus*) seed oil [Compared with some recommended (standard) oils by FAO/WHO and Tanzanian Bureau of Standards, TBS

Oil types	Saponification value (mg KOH g^{-1})	Iodine value (Wijis or g/100 g)	Free fatty acid value, as % oleic acid (mg KOH g^{-1})	Acid value (mg KOH g^{-1})	Peroxide value (meq kg^{-1})
Sunflower	188-194 (188-194)	110-143 (110-143)	0.085 (0.085)	≤0.6 (≤0.5)	≤10 (-)
Palm	190-209 (190-202)	50-55 (50.6-55.1)	1.376 (1.376)	≤0.6 (≤0.5)	≤10 (≤3)
Cottonseed	189-198 (190-198)	90-119 (99-119)	0.225 (0.225)	≤0.6 (≤0.3)	≤10 (-)
Soya bean	189-195 (189-195)	120-143 (120-143)	0.176 (0.176)	≤0.6 (≤0.5)	≤10 (-)
Coconut	248-265 (250-265)	6-11 (7.5-10)	2.540 (2.540)	≤0.6 (≤0.6)	≤10 (-)
Watermelon seed	205.3*	28.51*	4.61*	6.10*	2.80*

*Watermelon seed oil obtained in this study and are simple average of two determinations, Values enclosed in a bracket are that of Tanzanian Bureau of Standards, TBS, (Ngassapa and Othman, 2001), values not enclosed in a bracket are that of FAO/WHO (Anonymous, 1993)

Increasing the dietary and industrial utilization of food wastes could ultimately improve the environment. The Charleston gray variety of watermelon (*Citrullus lanatus*) seeds were discarded as wastes, thus its oil, extracted with petroleum ether (boiling range 60-80°C for 8 h) using Soxhlet extractor and separated from the solvent with a rotary evaporator was studied. The extracted oil was light yellow and a liquid at 37°C which are characteristic color and texture for oils. Its pH (4.9) suggested high acidic content of the oil due to the presence of other materials, including water. Oil in a pure state is not an ionizing solvent, does neither contain water nor supply hydrogen ions and hence does not have a pH. The specific gravity (0.87) of the watermelon seed oil showed that the oil is less dense than water hence could easily flow and spread on surfaces, including the skin. The specific gravity of the watermelon seed oil compared with the range 0.89-0.93 reported for *Afzelia africana* seed oil (Ejikeme *et al.,* 2010) and the values 0.740, 0.9235, 0.926 and 0.964 reported, respectively for the oil of custard seed (Amoo *et al.,* 2008), *Nicotiana tabacum* L. (Ali *et al.,* 2008), tropical almond seed (Agatemor, 2006) and cashew nut seed (Aremu *et al.,* 2006a).

High peroxide value (>10.00 meq kg^{-1}) indicated oil with a high susceptibility to auto-oxidation due to the presence of moisture or trace elements, especially copper (Adebisi and Olagunju, 2011) and high degree of unsaturation, Thus, the low peroxide value of the watermelon seed oil (2.80 mg KOH g^{-1}) suggested its high degree of stability and non-susceptibility to oxidative rancidity. The peroxide value (2.80 meq kg^{-1}) of the watermelon seed oil was lower than 10 meq kg^{-1} required for standard oils (Anonymous, 1993; Ngassapa and Othman, 2001) and could be suitable for human consumption. The watermelon seed oil saponification value (205.3 mg KOH g^{-1}) reported in this study was high, implying that the oil sample contains high proportion of short chain (or low proportion of long chain) fatty acids that favor stability. Low saponification value indicated the preponderance of long chain fatty acids in the oil and vice versa (Akintayo and Bayer, 2002). Apart from that of the coconut oil (248-265), the saponification value (205.3) of the watermelon seed oil was higher than the range 188-196 reported for the standard oils (Anonymous, 1993; Ngassapa and Othman, 2001) and the values 128.48 for avocado pear (Ikhuoria and Maliki, 2007) and 52.11 for custard seed (Amoo *et al.,* 2008). It however compared with the value (210.0 mg KOH g^{-1}) reported for baobab seed oil (Osman, 2004). The high saponification value as in the watermelon seed oil could be due high free fatty acid content also recorded in this study. The high saponification value and the low specific gravity value recorded in this study support the potential use of the watermelon seed oil in the production of such products as shoe polish, foaming agents, paints and creams requiring qualities as high saponification value as well as capacity to flow and spread easily on surfaces.

Generally, iodine value measures the degree of unsaturation, hence the degree of stability of oils. This is because saturated oils do not take up iodine hence have iodine value of zero, unlike the unsaturated oils. The watermelon seed oil iodine value (28.51 g/100 g) was within the range for the standard oils (but the value was, except that for coconut oil, lower than the value for the other standard oils) reported in Anonymous (1993) and Ngassapa and Othman (2001), this suggests the preponderance of low molecular weight saturated fatty acids (Osagie *et al*., 1986) and the edibility of the watermelon seed oil. This could further suggest lesser unsaturated (double) fatty acid bonds, lower reactivity, more stability and lower susceptibility to oxidation and rancid formation of the oil sample compared to most of the standard oils. Iodine value is an index of unsaturation, the ability of oil to go rancid and molecular size of fatty acid contents of oils (Osagie *et al.,* 1986). The iodine value of the watermelon seed oil (28.5 g/100 g) compared with the value 23.25 g/100 g reported by Oladimeji *et al.* (2001) for Hausa melon seed oil but lower than 38.50 g/100 g reported for pumpkin seed oil (Adebisi and Olagunju, 2011). Thus, the watermelon seed oil could be nutritionally beneficial and the low iodine value could confer it with greater oxidative stability during storage than most of the standard oils.

The acid value obtained (6.10 mg KOH g^{-1}) was higher than the value for the standard oils (Anonymous, 1993; Ngassapa and Othman, 2001) but lower in comparison to the values 11.5 mg KOH g^{-1} for *Plukenotia conophora* (Aremu *et al.,* 2006b), 47.6 mg KOH g^{-1} for benni seed oil (Aremu *et al.,* 2006b), 11.73 mg KOH g^{-1} for *Piliostigma thonningii* seed (Jimoh and Oladiji, 2005) and the limit value (10 mg KOH g^{-1}) for edible oils with stability against rancidity. The free fatty acid value (4.61 mg KOH/100 g) was higher than the value for the standard oils (Anonymous, 1993; Ngassapa and Othman, 2001), indicating its higher hydrolytic tendency since free fatty acids are the hydrolytic products of triacylglyceride. The higher acid and free fatty acid values of the watermelon seed oil as compared to the standard oils could account for its low pH (4.9) reported in this study, indicating the presence of other materials, including water that could compromise/lower its quality in terms of appeal, edibility and nutraceutical benefits. Low free fatty acid content of oil enhanced its appeal while high saturated fatty acid content of oil limited its nutraceutic role (Odoemena and Onyeneke, 1988). This study did not record the percentage oil yield from the watermelon seeds to enable the cost versus benefit analysis. This is a significant shortcoming that should be addressed in further and similar studies.

CONCLUSION

The watermelon seed oil properties compared fairly with that of the standard edible and industrial oil sources, suggesting its stability, edibility and industrial potentials. However, the comparatively high acid and free fatty acid values, supported by the low pH value could compromise the watermelon seed oil qualities and benefits. Nonetheless, the study provided basis for potential (domestic and industrial) uses of the watermelon seed oil. Further studies aimed at reducing the high acid and free fatty acid values of the oil sample could enhance the potential uses of the seed oil (and reduce the attendant environment burden of the watermelon seed), hence are warranted.

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