



NUTRITIONAL VALUE OF KARIYA (*HILDEGARDIA BARTERI* MAST. KOSTERM) OIL AS BASIS FOR ITS CONSERVATION

AREMO AE* • OLUWADARE AO

Department of Forest Resources Management, University of Ibadan, Ibadan, Oyo state, Nigeria.

*Corresponding Author: evelynadeniyi2004@yahoo.com

ABSTRACT: Extracted oil samples were obtained from the sun dried and oven dried kernels of *Hildegardia barteri*, using two different extraction methods (mechanical and solvent) methods and were analyzed for their nutritive value. Physicochemical properties, proximate and mineral analyses were determined using ASTM and AOAC standard analytical methods. Oil yields ranged from 23.15-30.53% for both mechanical and solvent extraction methods. Specific gravity, Acid value, Free fatty acid, Iodine value and Calorific value were 0.73-0.91, 0.032-0.034 mgKOH/g, 0.016-0.017, 57.95-61.85 and 33.21-36.14 MJ/kg, respectively; Moisture content, crude protein and ash contents ranged between 1.03-2.0%, 1.02-1.79% and 0.05-0.12% respectively while estimate of percentage crude fat obtained by calculation was 11.37-14.26%. C, O, H, N and S ranged between 71.29-72.12%, 17.7-18.57%, 8.3-9.57%, 0.16-0.27% and 0.02-0.06% respectively. Heavy metals analysed were Pb, Cd, Ni, and Co and their values ranged between 0.007-0.02%, 0.003-0.007%, 0.022-0.034% and 0.01-0.017%, respectively. Results obtained from this study, indicate the suitability of kariya oil for consumption purpose. Hence, the need for the conservation of lesser-used species such as *Hildegardia barteri*.

Keywords: Kariya oil, extraction methods, physicochemical properties, proximate and mineral analyses.

1. INTRODUCTION

Kariya (*Hildegardia barteri*) is a flowering plant species which belongs to the family Malvaceae. The species is commonly found in dry tropical forest in West Africa from Ivory Coast to south eastern Nigeria (Master 1868). Alongside aesthetic value and other environmental services that could be derived from the tree species. This tree species is reported to be among the 95% of the productive hardwoods that are categorized as lesser-known or underutilized (Yeom 1984 and Omolodun *et al.* 1991); this buttress the fact that vast tonnage of seeds are simply left to waste on the forest floors (Science news 2013). However, exploitation of its edible seeds for food or oil contributes to the livelihood of man. Kariya seeds are consumed either raw or roasted by only few parts in West Africa) and are known to be source of condiments. According to Inglett *et al.* (1973), who carried out studies on seed composition of *H. barteri* kernels, reported that thirty seeds generated 39% oil. Burkill 1985, also confirmed that Kariya seeds have oil content. Recent study by Ishola, *et al.*, 2015 also revealed the bioassay animal experiment of *Hildegardia barteri* seed with a view to provide useful data on the consumption mode and sensitize the populace on the need for further processing to avert food poisoning.

This study, in corroboration with the findings of Ogunsina *et al.* (2010) on nutritional and physical properties of kariya seeds and Obasi *et al.* (2012) who also carried out similar studies on seeds of *Cocos nucifera* and *Colocynthis citrullus* was carried out to explore the nutritional values and maximize potentials that could be obtained from lesser-used species as the demand for oil, both for human consumption and industrial applications increases. The establishment of substantial values of under-utilized species serves as basis to ensure conservation of these species. Thus, this study examines the proximate, mineral composition and some of physicochemical properties of kariya oil in order to provide basis for its nutritional value.

2. MATERIALS AND METHODS

2.1 Sample preparation

Matured ripped Fruits of *Hildegardia barteri* were collected from the tree stands at the Department of Agronomy, University of Ibadan, Nigeria. The fruits were de-hulled, dried using two methods-sun-drying (SD) and oven drying (OD) methods, weighed and milled. The samples were then prepared for oil extraction using soxhlet extraction (SE) with *n*-hexane solvent and

mechanical method (ME). Therefore, a total of four oil samples which include OD+SE, SD+SE, OD+ME and SD+ME were and analysed for their properties.

2.2 Physicochemical Properties

2.2.1 Percentage (%) oil yield

Oil was extracted from 50g of each oven dried and sun dried samples of milled seed. Weight of oil was obtained by subtracting the weight of sample bottle used for collection from the total weight of bottle and oil. Average of triplicate results were determined and used for the computation of the percentage yield per 50g of unextracted milled samples (Julius *et al.* 2013).

$$\% Y = \frac{\text{Weight of oil}}{\text{Weight of unextracted milled seed}} \times 100 \dots (1)$$

Where, Y = Oil yield

2.2.2 Specific gravity

This was determined by the standard method of A.O.A.C (2000). Specific Gravity of the oil is determined by using the formula:

$$SG = \frac{W_2 - W_1}{W_3 - W_1} \dots \dots \dots (2)$$

Where W_1 = Weight of specific gravity bottle

W_2 = Weight of oil and specific gravity bottle

W_3 = Weight of equal volume of water + specific gravity bottle.

2.2.3 Acid value and Free Fatty Acid (FFA)

This was determined according to the procedures outlined by AOAC (1984). This was determined by first preparing a mixed neutral solvent containing a mixture of 25ml diethylether with 25ml alcohol and 1ml of phenolphthalein solution (1%). The mixed solution was then neutralized with 0.1M NaOH.

1g of the oil will be dissolved in a 250ml conical flask containing 25ml of the mixed neutral solvent. The mixture was heated to boiling point and shaken vigorously. The solution was cooled and then titrated with 0.1M alcoholic caustic potash solution with constant shaking using phenolphthalein as indicator until the pink colour persisted for 15secs. The acid value was calculated as:

$$AV \text{ (mgKOH/g)} = \frac{\text{Titre value of alkali} \times 0.1M \text{ alkali} \times 56.1}{\text{Wt of sample used (g)}} \dots (3)$$

Where, AV = Acid value

%FFA calculated as 0.503 x acid value (Ajiwe *et al.* 1997)

2.2.4 Determination of Iodine value

The iodine value was determined according to the method outlined by AOCS (1978). It was calculated as:

$$\text{Iodine value} = \frac{(b-a) \times 1.269}{\text{Wt of sample used (g)}} \dots (4)$$

Where a = End point titration of mixture of Wijs' solution with potassium iodide solution (10%) and 100ml water and titrated with 0.1M thiosulphate solution.

b = titration of blank solution, commencing with 10ml of carbon tetrachloride

2.2.5 Determination of calorific value

The gross calorific/energy value of oil was determined using a CAL 2K-ECO bomb calorimeter. 0.5gm of each sample depending on bulkiness was weighed into the steel capsule. A 10cm cotton thread was attached to the thermocouple to touch the capsule. The bomb was closed and charged in with oxygen up to 3000kpa. The Bomb was fixed up by depression into the measuring chamber. The next step was the firing phase when the sample was ignited to burn in excess oxygen. After a successful firing phase, the main phase sets in where it takes duration of 10minutes before the final reading was taken. The maximum temperature rise in the bomb was measured with the thermocouple and galvanometer system and reading was recorded as the calorific value.

2.3 Proximate Analysis

The proximate composition of the oil was determined using standard laboratory methods (AOAC) in three replicates.

2.3.1 Determination of moisture content

Moisture content was determined according to AOAC (1990) method by drying to constant weight in an oven. The moisture content was determined by oven dehydration method between 95-100°C for 5hr using this formula:

$$MC(\%) = \frac{W_1 - W_2}{\text{Weight of oil sample}} \times 100 \dots \dots \dots (5)$$

Where W_1 = Initial weight of oil sample, W_2 = Final weight of oil sample

2.3.2 Determination of ash

The ash content in the samples was determined by following the outlined procedures of AOAC (1990) method; using a muffle furnace at 600°C. Weight of ash was calculated as

Difference in wt. of Ash= $W_3 - W_1$ and percentage obtained as follows:

$$\text{Ash}(\%) = \frac{W_3 - W_1}{\text{Weight of sample}} \times 100 \dots \dots \dots (6)$$

Where W_1 = weight of empty crucible, W_2 = weight of crucible + sample, W_3 = weight of crucible + sample after heating.

2.3.3 Determination of crude protein

Crude protein was determined using the kjeldahl method. 2g of samples was taken in digestion flask. 10-15 ml of concentrated H_2SO_4 and 8 g of digestion mixture i.e. K_2SO_4 $CuSO$ (8: 1) was added. The flask was swirled in order to mix the contents thoroughly then placed on heater to start digestion till the mixture become clear (blue green in color). This was done for 2 hrs. The digest was cooled and transferred to 100 ml volumetric flask and volume was made up to mark by the addition of distilled water. Distillation of the digest was performed in Markam Still Distillation Apparatus (Khalil and Manan 1990). Ten milliliters of digest was introduced in the distillation tube then 10 ml of 0.5 N NaOH was gradually added through the same way. Distillation was continued for at least 10 min and NH_3 produced was collected as NH_4OH in a conical flask containing 20 ml of 4% boric acid solution with few drops of modified methyl red indicator. During distillation yellowish color appears due to $NHOH$. The distillate was then titrated against standard 0.1 N HCl solution till the appearance of pink color. A blank was also run through all steps as above. Percentage crude protein content of the sample was calculated by using the following formulae:

$$\%N = \frac{(S-B) \times N \times 0.014 \times D}{\text{Weight of milled seed} \times V} \times 100 \dots \dots \dots (7)$$

$$\% \text{ Crude Protein} = 6.25 \times \%N \text{ (*CF)} \dots \dots (8)$$

Where S = Sample titration reading, B = Blank titration reading, N = Normality of HCl, D = Dilution of sample after digestion, V = Volume taken for distillation, 0.014 = Milli equivalent weight of Nitrogen, CF = Correction factor.

2.4. Elemental Analysis of Seed Oil

The mineral nutrients and heavy metal analyses of the oil was determined according to the methods of ASTM 3174-76.

2.4.1 Determination of carbon, hydrogen and oxygen

These were determined according to the procedures of ASTM 3174-76. 2g of each sample was weighed into platinum crucible and placed in a Leibig-pregle chamber containing Magnesium percolate and sodium hydroxide. The sample was burnt off to produce carbon-dioxide and water. The CO_2 was absorbed by Sodium hydroxide while the water was absorbed by magnesium percolate. The amount of water and CO_2 were calculated by difference. The elements were calculated as follow:

$$\% C = \frac{a \times 0.2727}{\text{Weight of oil sample}} \times 100 \dots \dots \dots (9)$$

$$\% H = \frac{b \times 0.117}{\text{Weight of oil sample}} \times 100 \dots \dots \dots (10)$$

$$\% O = 100 - (\%C + \%H) \dots \dots \dots (11)$$

Where a = Quantity of CO_2 b = Quantity of H_2O

2.4.2 Determination of nitrogen

Nitrogen content was determined using kjeldahl method, as described for determination of crude protein.

2.4.3 Sulphur content determination

This was determined using the formula below (Tabatabai 1974). 5ml of the digest was pipette into a 100ml beaker and 10ml of distilled water was added and mixed thoroughly to a complete homogenized solution. Then 1ml of 1% Barium gelatin solution was added to precipitate the sulphur. Working standard of sulphur was prepared from range 0-50mg/l and treated similarly like sample.

Absorbance of standard as well as sample was read on a spectrophotometer at a wavelength of 420nm.

$$S = A \times GF \times DF \dots \dots \dots (12)$$

Where: A = Absorbance, GF = Gradient factor and DF = Dilution factor

2.4.4 Heavy metals Analysis

Heavy metal analysis of the oil was determined according to the methods of ASTM d3174-76 with the use of the atomic absorption spectrophotometer (AAS) to determine Pb, Cd, Ni and Co. 0.5g of each sample was weighed into a beaker and 10ml of an acid solution (Nitric/perchloric acid) in ratio 1:2 was added to the sample in the beaker. Then it was allowed to undergo heating on a hot plate at 105°C for an hour. The colour change while heating was observed to be from brown to

clear colourless. The digest at this point was allowed to cool. The digest was then read on the atomic absorption spectrophotometer to determine the following metals.

3. RESULTS AND DISCUSSION

3.1 Physico-chemical properties

Percentage Oil Yield

The oil yields from SD+SE was observed to be significantly higher ($30.5\pm 3.02\%$) and different from other samples, while SD+ME, OD+SE and OD+ME oil samples were not significantly different from each other. The SD+ME sample had the least value ($19.5\pm 2.1\%$). The observed oil yield values obtained ($19.52\text{--}30.53\%$) fall within the reported range for commercial vegetable oils such as cotton seed (19.50%), soybeans seed (19.0%), palm oil (48.65%) and groundnut (49.0%) as reported by Dawodu (2009). However, according to Matchet (1963), oil-bearing seed that yield up to 30% oil are considered suitable for commercial and industrial applications.

Specific gravity

Specific gravity is the heaviness of a substance compared to that of water, and it is expressed without units, the specific gravity values ranged between $0.73\pm 0.09\text{--}0.91\pm 0.06$ and no significant difference among the oil samples was observed. However, the result falls within the range (0.88 to 0.94) for common edible oils reported by Toolbox (2005).

Table 1: Physico-chemical properties of *H. barteri* Seed oil

SV	% oil yield	SG	AV	FFA	IV	CV
OD+ME	$21.96\pm 1.35^{*a}$	0.88 ± 0.05^a	0.03185 ± 0.001^a	0.016 ± 0.0003^a	57.95 ± 0.65^a	33.210 ± 1.01^a
OD+SE	23.15 ± 1.51^a	0.91 ± 0.06^a	0.03395 ± 0.001^a	0.0171 ± 0.0003^a	61.25 ± 0.05^b	35.690 ± 0.01^b
SD+ME	19.52 ± 2.10^a	0.73 ± 0.09^a	0.03365 ± 0.002^a	0.0169 ± 0.001^a	58.6 ± 0.2^a	34.180 ± 0.01^a
SD+SE	30.53 ± 3.02^b	0.86 ± 0.0001^a	0.0336 ± 0.0001^a	0.0169 ± 0.00001^a	61.85 ± 0.05^b	36.140 ± 0.01^b

* Means \pm SE. Values with the same alphabet in each column are not significantly different at $\alpha = 0.05$.

3.2 Proximate analysis

The results obtained for the proximate composition of *Hildegardia barteri* seed oil (Table 2) compares favorably well with the proximate composition of the kernel carried out by Inglett *et al.* (1973). This substantiates the findings that the oil of *H. barteri* is a basis for the exploitation of the species for food or oil.

Moisture content (MC)

Moisture content observed in the oil samples ranged between $1.05\pm 0.01\text{--}1.98\pm 0.01\%$ with the SD+ME oil sample had the least value while OD+SE sample had the

Acid value (AV)/ Free-fatty acid (FFA)

No significant difference was observed between the Acid and Free fatty acid values of the oil samples; range between $0.032\text{--}0.034\text{mgKOH/g}$ and $0.016\text{--}0.017\text{mgKOH/g}$. In general, this gives an indication about edibility of the lipid (Analytical methods of fats and oils 2011) as well as their suitability for industrial use, as it depicts the free fatty acid level in the oil due to hydrolytic or lypolytic activity. The acid value of the seed oils studied fell within allowable limits (Not more than 0.8mgKOH/g) for edible oils given by Codex Alimentarius Commission (1983). Therefore, the FFA content of kariya oil which is lower than that of soya oil (0.7mgKOH/g) and maize oil (0.03mgKOH/g) (Salunke *et al.* 1992) attests to its edibility.

Iodine value

Iodine value obtained in solvent extracted oil samples were significantly higher (61.3 ± 0.05 and 61.9 ± 0.05) than the mechanically extracted oil samples (57.95 ± 0.65 and 58.6 ± 0.2). Iodine value is a parameter that corresponds to the unsaturated fatty acid constituents. Generally, Iodine values of *Hildegardia bateri* seed oil obtained in this study were observed to be lower compared to a range of $110.93\text{--}111.46$ obtained in *Colocynthis citrullus* oil (Obasi *et al.* 2012) which was classified as non-drying or stable oil. A good drying oil should have Iodine value of 180 and above (Codex Alimentarius Commission 1982). The iodine value obtained, simply connotes that *H. barteri* seed oil will have good oxidative stability. In other words, its tendency to become rancid is very low.

highest value. Mechanically extracted (ME) oil samples had the least percentage moisture content ($1.1\pm 0.01\%$). The low moisture content obtained in the oil samples is said to be advantageous in terms of shelf life as this optimize the keeping quality of oil since oils with high moisture content are known to undergo rancidity (Fellows 1997).

Crude protein (%)

Crude Protein (CP) obtained ranged between $1.29\text{--}1.66\%$ with OD+ME sample having the highest value ($1.66\pm 0.07\%$) among the oil samples. Though this result varied from the crude protein obtained in the kernels of

H. barteri (33.2%) as reported by Inglett et al. (1973), it implies that the oil samples are not highly proteinous but edible to humans and can serve as a good supplement in animal feed formulation. This corroborates the findings and comparison made by Raji and Orelaja (2014), between oil characteristics of Cucumis melo seeds and high protein seeds and nuts like cowpea (22.7%) and soyabeans (35%)

Ash (%)

The values obtained in the oil samples) ranged from 0.05 ± 0.003 - $0.1 \pm 0.01\%$. Ash content indicates the inorganic matter or mineral elements with specific metabolic functions present in the biomass. This was observed to be lower to the values (1.02 and 2.94%) obtained for *Cocos nucifera* and *Colocynthis citrullus* respectively (Obasi et al. 2012) and some oil-bearing seeds such as cotton (4.56%), sunflower (6.0%), corn (1.5%) as cited by Muhammad et al. (2012).

Crude fat

Estimates of percentage crude fat of the oil samples was obtained by getting the difference between the mean percentage crude fat of the milled kernel and the oil-free kernel (based on the drying methods samples). Table 3

shows that crude fat for the whole seed meal (40.32 and 39.97%) varied from the report of Ogunsina et al. (2010) who obtained 37.5% crude fat in the kernels of *H. barteri*. The resultant crude fat for the oil samples shows an estimate of the lipid that tends to be solid at room temperature.

3.3 Elemental Analysis

Result obtained from the analysis of these essential elements C, H, O, N and S, shows that all elements except for carbon and hydrogen content are lower in the oils samples. Nitrogen and Carbon contents in Mechanical Extracted (ME) samples were observed to be significantly different from the Soxhlet Extracted (SE) samples. These analysed elements are the basic elemental components of amino acid (Amino acid 2016) which is responsible for tissue growth.

3.4 Heavy metals Analysis

Result obtained from this analysis shows that concentration of these heavy metals fall within the permitted range permitted by USEPA (1992). The lower values of heavy metals obtained in the oil sample, gives a basis for the exploitation of oil from *H. barteri* seed as a potential source for consumption purpose.

Table 2: Proximate Analysis of Seed oil

Source of variation	DF	MC	Crude protein	Ash
SD+ME	2	1.05 ± 0.01^b	1.32 ± 0.07^a	0.053 ± 0.003^a
SD+SE	2	1.25 ± 0.01^a	1.29 ± 0.07^a	0.11 ± 0.01^a
OD+SE	2	1.98 ± 0.01^c	1.53 ± 0.07^a	0.053 ± 0.003^a
OD+ME	2	1.18 ± 0.02^a	1.66 ± 0.07^b	0.063 ± 0.01^a

* Means \pm SE. Values with the same alphabet in each column are not significantly different at $\alpha = 0.05$.

Table 3: Mean Percentage Crude Fat

Samples	Kernel	Oil-free kernel	Oil*
SD	40.32	28.95	11.37
OD	39.97	25.7	14.26

* obtained by difference

Table 4 Elemental analysis of seed oil

Source of Variation	% N	% H	% O	% S	% C
OD+ME	0.16 ± 0.03^a	8.49 ± 0.01^b	17.99 ± 0.51^{ab}	0.02 ± 0.01^a	71.29 ± 0.01^a
OD+SE	0.26 ± 0.02^b	9.57 ± 0.01^d	18.57 ± 0.01^b	0.035 ± 0.01^a	72.05 ± 0.01^b
SD+ME	0.19 ± 0.01^a	8.3 ± 0.01^a	17.7 ± 0.01^a	0.035 ± 0.02^a	71.33 ± 0.02^a
SD+SE	0.27 ± 0.01^b	9.38 ± 0.01^c	18.42 ± 0.01^b	0.065 ± 0.02^a	72.12 ± 0.01^b

Table 5 Heavy metal analysis of seed oil

Source of Variation	% Pb	% Cd	% Ni	% Co
OD+ME	0.017±0.002 ^{bc}	0.0035±0.001 ^b	0.025±0.001 ^{ab}	0.012±0.001 ^{ab}
OD+SE	0.01±0.002 ^{a d}	0.007±0.001 ^c	0.029±0.001 ^a	0.014±0.001 ^{bc}
SD+ME	0.023±0.002 ^e	0.003±0.001 ^b	0.022±0.001 ^b	0.01±0.001 ^a
SD+SE	0.007±0.001 ^a	0.0045±0.001 ^b	0.034±0.002 ^c	0.017±0.002 ^c

* Means ±SE. Values with the same alphabet in each column are not significantly different at $\alpha = 0.05$.

Table 6: Maximum conc. Level for Heavy Metal conc. in Air, Soil and Water (UNEP)

Heavy Metal	Max Conc. in Air (mg/m ³)	Max Conc. in Sludge Soil (mg/kg)	Max Conc. In drinking water (mg/l)	Max Conc. In H ₂ O Supporting life (mg/l)
Cd	0.1-0.2	85	0.005	0.005
Pb		420	0.001	0.0058
Zn	1.5	7500	5.00	0.0766
Hg		<1	0.002	0.05
Ca	5	Tolerable	50	Tolerable>50
Ag	0.01	-	00	0.1
As	-	-	0.01	-

Adapted from USEPA, 1992; Washington Code, 1992.

4. CONCLUSION

1. The solvent extraction method yielded more oil than the mechanical extraction method. However, the yield obtained from both extraction methods attest that *H. barteri* is an oil-bearing tree species suitable for both domestic and industrial applications.
2. Results of proximate and elemental analyses which fall within permissible limit for edible oils reveal that kariya (*H. barteri*) oil will be a good source of supplement either for human consumption or animal feed. The oil is safe from heavy metal toxicity as the result of selected heavy metal analysed were below the permissible level.
3. The Iodine value result shows that the oil is non-drying or stable oil likewise its low free fatty acid value confirms stability to enzymatic hydrolysis.
4. The findings of this study, therefore provides a basis for the conservation of the species as this will supplement other sources of oil.

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