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Laboratory-scale extraction of *Hildegardia barteri* (Mast.) Kosterm. seed oil with different solvents, purified with membrane filtration

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Abstract. The shortcomings encountered from the use of conventional methods of refining draw considerable attention when it comes to the processing of edible oils. The present invention to this effect is the usage of membrane technology, which stands out as a suitable alternative, as it offers significant advantages such as minimal chemical additions, low energy consumption, and the retention of nutrients and other desired components. This paper seeks to ascertain the quality of edible oil extracted by different solvents, refined via membrane filtration. Amounts of 25 mL of crude oil sample were extracted with three different solvents (n-hexane, ethanol, and n-hexane-ethanol blend) from seeds of *Hildegardia barteri* (Mast.) Kosterm. and were refined using a micro-filter of pore size of 0.45 µm and an ultrafiltration membrane with 50 kDa cut off before bleaching and deodorizing to obtain table oil. Proximate composition and elemental analyses were carried out on the table oil samples produced and compared with food-grade standards. Results obtained showed 0.133–0.53% moisture content, 0.04% ash, 98.90–99.67% ether extract, 0.23% carbohydrate, and elemental compositions of 51.60–55.00% C, 6.12–6.30% H, 6.21–6.28% O, 0.01–0.02% N, and 13.0–15.0% P for edible oil samples. The findings of this study indicate that edible oil produced from the seeds of *H. barteri* via membrane technology yields good-quality oil for commercial production, except for the need to enhance further reduction of phosphorus content.

Keywords and phrases: *Hildegardia barteri*, edible oils, elemental analysis, solvent extraction, membrane filtration

1. Introduction

The processing of edible oils, which begins with the extraction process, has drawn considerable attention. It has long been a standard practice to extract such oils with an organic solvent to obtain miscella, which is subsequently removed, leaving a crude oil composition. Hexane is commonly used to extract oil because of its lower boiling point and easy separation after extraction and its non-polarity (high oil solubility). On the other hand, hexane is designated by the US Environmental Protection Agency as a hazardous air pollutant (HAP) and has been added to the list of other toxic chemicals (*NIOSA*, 2007). This problem has grabbed the curiosity of scientists seeking an alternative to solvents. Several solvents and mixtures, including n-hexane, petroleum ether, and alcohols, have been tested to obtain oil from *H. barteri* oilseeds (*Aremu et al.*, 2015; *Ochigbo & Paiko*, 2011), and their interfering effects on the nutritional value of the edible oil need to be investigated.

Secondly, after extraction, impurities, such as phospholipids, waxes, organic sulphur compounds, dye compounds, and other contaminants, can make up to 10% of the crude oils obtained. To remove these unwanted compounds, a refining process is needed to make the product useful and marketable.

Membrane technology has been observed to offer remarkable advantages – features include operation at room temperature, minimal chemical additions, low energy consumption, and the retention of all nutrients and other desired components in the oil (*Cheryan*, 1998). These advantages make the separation process stand out as a suitable alternative to the conventional refining method for the food, chemical, biotechnological, and pharmaceutical industries, as it is also known for increased final product quality and separation efficiency (*Fatima et al.*, 2021). Present inventions that relate to one of the methods for refining domestic edible oils is the usage of membrane technology for separation (*Lam et al.*, 2016; *Piacentini et al.*, 2014). However, it has been discovered that various types of membranes and make-up materials have varying degrees of success and inherent constraints. The characteristics of the membrane used impede the complete removal of phospholipids from unrefined oil, needing several different processing stages, such as degumming, refining, bleaching, and deodorizing, to get rid of free fatty acids, phosphatides, particulates, chlorine materials, and the like (*Vaisali et al.*, 2015). One of the goals of these inventions and a significant benefit would be the development of a process for purifying edible oils, wherein most of the hydrous and non-hydrated phospholipids, most of the colour compounds and free fatty acids are separated in the retentate, while neutral oils and the n-hexane pass through the membrane (*Ramos-Andrés et al.*, 2019).

Inferences from different studies carried out suggest that the membrane system technology has the potential for the processing (degumming and deacidification) of fatty oils because it is simple in operation, eco-friendly, and energy-efficient

(*Desai et al.*, 2002; *Chew & Nyam*, 2020). Therefore, this paper seeks to ascertain the quality of edible oil produced from seeds of *H. barteri* by two solvents (n-hexane and ethanol) and their mixture, refined via membrane filtration.

2. Materials and methods

Extraction of *H. barteri* crude oil samples

Crude oil samples were extracted from the seeds of *H. barteri* with three different solvents: n-hexane, ethanol, and hexane-ethanol blend, using a Soxhlet apparatus, and an amount of 25 mL of each sample was collected for further refining.

Refining by membrane filtration

The membrane filtration procedures for the production of edible oil, described by *Van Reis and Zydney* (2007) were employed. The initial step involved pre-filtering crude oil samples using syringe micro-filter with a pore size of 0.45 µm (Millipore Millex-HN, Merck), facilitated by a simple vacuum pump. Subsequently, an ultra-filtration membrane with a 50 kDa molecular weight cut-off and a 15 mL capacity, contained within a centrifuge tube, were utilized to filter the oil samples.

The filtration process was carried out using a fixed-angle rotor centrifuge operating at 5,000 rpm with a duration of 20 minutes. Following this, the filtered oil samples underwent bleaching and deodorization.

Bleaching

The neutralized oil obtained was transferred into a beaker and heated to 90°C. One percent of activated clay (by weight of oil) was added to absorb colouring components. The mix was agitated continuously for half an hour. An increase in temperature to 110°C was allowed for another half an hour. The oil content was then filtrated at the temperature of 70°C by employing a Büchner funnel, filter paper, and vacuum, according to the AOAC technique (*Nkpa et al.*, 1989).

Deodorization

Deodorization was carried out using a modified procedure (*Zulkurnain et al.*, 2013). A rotavapour (Büchi R-210) was used for vacuum distillation process (at $T_{bath} = 95$°C, $p = 4$ mbar), but for a longer period of $t \geq 4$ hours, to get rid of volatile compounds that would cause off-odours and tastes of the oil.

Proximate and elemental properties

The proximate and elemental analysis of table oil samples was analytically determined by using standard laboratory methods (*Helrich*, 2006; *Latimer*, 2016) in three replicates. The oil samples were evaluated for moisture, ash, and crude protein content.

Proximate composition

Determination of moisture content (MC)

Moisture content in the samples was evaluated in line with *Helrich*'s (2006) procedure by dehydrating to constant weight in an oven. The MC was determined by oven dehydration method at 105 ± 2°C till a constant weight.

Determination of ash

The ash proportion in the samples was estimated by following the outlined procedures of *Helrich*'s (2006) method using a muffle furnace at 600°C.

Determination of crude protein

The Kjeldahl method was the procedure adopted in evaluating crude protein in the samples (*Helrich*, 2006). The total Kjeldahl nitrogen content (N_K%) and thereafter the percentage crude protein content (P_C%) of the sample was calculated using the following formulae, (1) and (2):

$$N_K\% = 100 \cdot \frac{(S - B) \cdot N_{HCl} \cdot E_N \cdot D}{m_s \cdot V}, \tag{1}$$

where: S – sample titration reading, mL; B – blank titration reading, mL; N_{HCl} – normality of hydrochloric acid, N; E_N – equivalent weight of nitrogen, with value of E_N = 0.014 mEq; D – dilution of the sample after digestion; m_S – weight of sample, g; V – volume of distillate, mL; and

$$P_C\% = k \cdot N_K\%, \tag{2}$$

where k is the conversion factor of Kjeldahl nitrogen to crude protein, in this case: k = 6.25.

Elemental analysis

The elements such as Carbon (C), Hydrogen (H), Oxygen (O), Phosphorus (P), and Nitrogen (N) of the oil samples were assessed following the methods of ASTM 3174-76 (*AOCS,* 1993).

Determination of carbon, hydrogen, and oxygen

This was determined by weighing 2 g of each oil sample and placing it in a Liebig fragile chamber with sodium hydroxide and magnesium perchlorate. The contents are burned to obtain carbon dioxide and water. The CO_2 is absorbed by sodium hydroxide, while the water is absorbed by magnesium perchlorate. The total amount of *C*(%) and *H*(%) were calculated using the formulae (3) and (4).

$$C(\%) = 100 \cdot \frac{0.2727 \cdot m_{CO_2}}{m_S} \tag{3}$$

$$H(\%) = 100 \cdot \frac{0.117 \cdot m_{H_2O}}{m_S} \tag{4}$$

The mass of the formed carbon dioxide (m_{CO_2}) is determined by calculating the mass increase of the absorbent material ($\Delta m_{a,c}$), i.e. sodium hydroxide ($m^0_{a,c}$), after absorbing the carbon dioxide produced during combustion ($m_{a,c}$), as shown in formula (5):

$$m_{CO_2} = \Delta m_{a,c} = m_{a,c} - m^0_{a,c} \tag{5}$$

The mass of the produced water (m_{H_2O}) is determined by measuring the weight increase of the absorbent material ($\Delta m_{a,w}$), i.e. magnesium perchlorate ($m^0_{a,w}$), after absorbing the water vapour produced ($m_{a,w}$) during combustion, as shown in formula (6):

$$m_{H_2O} = \Delta m_{a,w} = m_{a,w} - m^0_{a,w} \tag{6}$$

Determination of the nitrogen content

Kjeldahl's procedure was used again in assessing nitrogen content in the oil samples, as described for the determination of crude protein (equation (1)).

Determining phosphorus – Tri-acid digestion

The digestion is made using a mix of HNO_3:H_2SO_4:$HClO_4$ in the proportion of 9:4:1. The detailed procedure is as follows: 1 mL of the sample was added to 9 mL HNO_3 and heated for 1 hour at 150°C, and then the solution was allowed to cool. Then, 4 mL H_2SO_4 and 1 mL $HClO_4$ were added, the mixture was heated for 1 hour at 150°C, and the solution was cooled. Deionized water was added to the final volume of 100 mL (*Tandon*, 2001).

Estimation of the phosphorous content of the sample digest was obtained by transferring the sample to a volumetric flask:

$$P(\%) = C \cdot \frac{1}{m_S} \cdot \frac{100}{m_{alq}} \cdot \frac{V_f}{1000}, \tag{7}$$

where C – concentration of (P) in the digested sample, mg/L; m_S – sample weight, g; m_{alq} – aliquot weight, g; V_f – final volume, mL.

Colour analysis

The colour analysis of oil samples was performed using the Color Tec-PCM colorimeter (Color Tec, Clinton, NJ). The analysis is based on the CIE L*, a*, b* (CIELab) colour scheme established by the *Commission Internationale de l'Eclairage* (*CIE*, 2020). The measurements obtained are displayed in CIELab format, as described in *Belbin* (1993). The L^* value represents the lightness-darkness dimension, the a^* value represents the red-green dimension, and the b^* value represents the yellow-blue dimension. The hue of the colour (h°) is calculated as:

$$h^\circ = \arctan\left(\frac{a^*}{b^*}\right), \tag{8}$$

where $h^\circ = 0^\circ$ for red, and $h^\circ = 90^\circ$ for yellow.

Additionally, chromaticity (C^*) is calculated by formula (9):

$$C^* = \sqrt{a^{*2} + b^{*2}} \tag{9}$$

Statistical analysis

The significant differences among oil parameters were evaluated using one-way ANOVA (*SAS*, 2002) and laid in a completely randomized design, which was run on SAS statistical software.

3. Results and discussion

Proximate composition

The values of proximate composition obtained in *Table 1* reflect the nutritional value of a refined food-grade level of *H. barteri* oil samples that could be recommended for consumption.

Table 1. Proximate composition of the oil samples extracted with different solvents

Component	Extraction solvent			p-value
	n-Hexane	Blend	Ethanol	
	m/m%			
Moisture	0.53 ± 0.06^{a}	0.53^{a} ± 0.21^{a}	0.13 ± 0.06^{b}	0.014
Protein	0.20 ± 0.10^{a}	0.10 ± 0.10^{ab}	0.00 ± 0.00^{b}	0.064
Ash	0.04 ± 0.06^{a}	0.00 ± 0.00^{b}	0.00 ± 0.00^{b}	0.000
Ether extract	98.90 ± 0.10^{b}	99.37 ± 0.3^{a}	99.67^{a} ± 0.21^{ab}	0.015
Carbohydrate	0.23 ± 0.058^{a}	0.00 ± 0.00^{b}	0.00 ± 0.00^{b}	0.000

Notes: Blend: n-hexane:ethanol 1:1 (V/V); means ± SD of triplicate values with similar notations are not significantly different; significant at $p < 0.05$.

The MC obtained for all oil samples ranges between 0.13 and 0.53%, as the ethanol oil extract had the lowest value (0.13%) and was significantly different from both hexane-extracted (0.53%) and blend-extracted (0.53%) oils. The obtained result was much lower than the results reported from other studies such as for *Coco nucifera* oil: MC = 8.43% (*Evbuomwan & Emmanuel*, 2019) or *Moringa oleifera* seed oil: MC = 10.5% (*Adegbe et al.*, 2016). However, values were higher than the maximum limit recommended by the ASTM standard (0.05%). Consequently, having low MC will allow for the longer shelf life and stability of the products, as they will not be easily susceptible to rancidity.

The protein content obtained in all food-grade oil samples ranged between 0.1 and 0.2% and was low and insignificant. No noteworthy differences were observed

in the protein content among the samples. The low values obtained show loss in protein content as compared to the range (1.29–1.66%) obtained from the different extracted crude oil samples of *H. barteri* (*Adeniyi & Oluwadare*, 2016) and serve as a proof that protein denatures as it is exposed to higher temperatures during the process of refining.

Ether extracts, which are synonymous to crude fat, ranged between 98.90 and 99.67% for all edible oils produced, and the statistical analysis indicated no significant difference among them. The result indicates not less than 98% pure oil content and ≤ 2% of other components, which could be attributed to impurities incurred during handling or processing, and this also correlates and affirms the result obtained for ash content in the oil samples.

Results for ash content and carbohydrate show that only the hexane oil sample was observed to have a low proportion of 0.04% and 0.23%, respectively, and it was not detected in other samples. The value of ash content, which indicates the inorganic or mineral content left in a food sample after it has been heated to a very high temperature (*Alinnor & Oze*, 2011), was only detected in the n-hexane-extract sample (0.04%). Though a little higher than the ASTM permissible amount (0.02%), it can be said to be within the range and compares well with the standard. The presence or absence of ash indicates a link to the processing and the interference of the different solvents used in extracting the oil from the seed oil. The absence of ash content in ethanol and hexane-ethanol blend extracts indicates the purity of the samples. The determination of ash content ensures that no toxic minerals are present and hence the safety of the oil sample.

The carbohydrate content obtained in just one of the samples (hexane extract) proves that the inherent energy content of oils is not based on carbohydrates (polymer of simple sugars) but on the fatty acids, which are the carboxylic group of lipids that yields a large quantity of adenosine triphosphate (ATP) when metabolized. This also suggests that there could be little influence of the different solvents used for the extraction of crude oils on the proximate composition.

Elemental analyses

Results in *Table 2* summarize the mean values ± SD of the elements analysed on all table oil samples.

For all oil samples, it was observed that the elements were of low values in all table oil samples except for phosphorus (*Table 2*). This, therefore, suggests that the phospholipid component, which has P as the major element, was not efficiently removed by the membrane-refining method. This was corroborated by *Koris and Vatai* (2002), who reported that the complete removal of phospholipids from the crude oil is impeded by the characteristics of the membrane employed, thus calling for a variety of processing steps.

Table 2. Elemental composition of the oil samples

Element	Extraction solvent			p-value
	n-Hexane	Blend	Ethanol	
	m/m%			
Hydrogen (H)	6.14 ± 0.02[b]	6.30 ± 0.02[a]	6.12 ± 0.02[b]	0.000
Carbon (C)	55.00 ± 0.01	51.60 ± 0.02	54.30 ± 0.02	0.296 (NS)
Oxygen (O)	6.24 ± 0.02[ab]	6.21 ± 0.02[b]	6.28 ± 0.03[a]	0.014
Nitrogen(N)	0.01 ± 0.00	0.02 ± 0.00	0.01 ± 0.00	0.054 (NS)
Phosphorus (P)	15.00 ± 1.00[a]	13.67 ± 0.58[ab]	13.00 ± 1.00[b]	0.079 (NS)
Total	82.39	77.80	79.71	

The nitrogen content (N%) obtained for the set of samples (0.01–0.02%) was not significant as compared to 1.94% obtained in grape seed oil as reported by *Luque-García and Luque de Castro* (2004). This elemental test was carried out to document the quantities of elements obtained after adopting the membrane-refining process and to know the extent of reduction of phospholipids in the seed oil of *H. barteri*. The removal or reduction of phospholipids during refining reduces the final content of phosphorus, which is a pro-oxidant that could accelerate the rancidification of the oils (*Choe & Min*, 2006).

CIELab oil colour

Results shown in *Table 3* reveal significant differences ($p < 0.05$) in L^* a^* b^* values of oil extracted using n-hexane, ethanol, and their blend. The L^* value was highest in ethanol-solvent-extracted oil followed by blend, the lowest being found in n-hexane. The n-hexane-extracted oil colour had the highest a^* value followed by blend and the lowest value in the ethanol-extracted oil. The b^* value was highest in hexane-extracted oil followed by blend, and ethanol-extracted oil had the lowest value.

The Chroma, which provides a quantitative measure of the colour saturation, had a highest value of 83.17 for hexane-extracted oil followed by 37.71 in blend-extracted oil and the lowest in ethanol-extracted oil, with a value 31.83. Colour is an important aesthetic quality parameter, which, according to the definition by the CIE, is the characteristic of visual perception that can be described by the attributes of hue, brightness or lightness, colourfulness, saturation, or chroma (*Fairchild*, 2013).

The *Hildegardia* seed oil has the highest value of yellowness, which suggests the higher concentration of highly lipophilic yellow pigment carotenoids such as carotenes (β-cryptoxanthin, α- and β-carotene) or xanthophylls with chemo-

preventive efficacy in animal models of skin carcinogenesis and buccal pouch carcinogenesis (*Nagao & Yanagita*, 2008). The higher value of b^* may be due to the higher solvation power of hexane in extracting carotenoids, of blend-extracted table oil, followed by a lower value of $b^* = 37.57$ with ethanol-extracted oil as the least significant $b^* = 31.83$; this may be due either to the lower solvency power of ethanol to extract carotenoid (*Ordóñez-Santos et al.*, 2017) or to its superior solvation power to extract chlorophyll, which is obvious in its negative colour coordinate $a^* = -0.41$, a result tending towards greenness, denoting the presence of chlorophyll, a green pigment, and also a lower $a^* = 3.2$ in 50% blend-ethanol-hexane-extracted oil, while hexane-extracted oil has a significantly higher $a^* = 10.25$; the psychometric indices of lightness for oil in this study varied significantly (*Figure 1*).

Table 3. CIELab colour analysis of the extracted oil samples

Colour parameters	**Extraction solvent**			**p-value**
	n-Hexane	**Blend**	**Ethanol**	
L^*	78.38 ± 0.34^b	81.88 ± 0.83^a	82.90 ± 0.49^a	0.0002
a^*	10.25 ± 0.46^a	3.20 ± 1.17^b	-0.41 ± 1.25^c	0.0001
b^*	82.54 ± 0.23^a	37.57 ± 1.57^b	31.83 ± 1.53^c	0.0001
Chroma	83.17	37.71	31.83	-
Hue ($h°$)	83.82	87.50	87.39	-
Chromaticity	Yellow-Red	Yellow-Red	Yellow-Reds	-

Figure 1. Oil samples extracted by different solvents

Lightness of blend- (81.88) and ethanol- (82.90) extracted oil compares well with the fresh variety of virgin olive oil L^* = 82.97, as reported in the literature (*Ceballos et al.*, 2003). Often, lighter colour has been associated with better-quality oils, especially for salad oils and shortenings (*Shahidi*, 2005).

4. Conclusions

According to the outcomes obtained from this study, the use of membrane technology and further refining applied for the production of edible oils from the seeds of *H. barteri* yielded lower moisture content in the oil samples. Consequently, a longer shelf life, stability of the products, and non-susceptibility to rancidity are attained. The solvents did not affect the ash content of the extracted oil, which was very low or undetectable. Different solvents used for oil extraction impact colour attributes, including lightness, yellowness, greenness, and colour saturation. The lightness values varied significantly, but both the blend- and ethanol-extracted oils had lightness comparable to high-quality virgin olive oil. Hexane extraction yielded a higher b^* value, indicating the greater concentration of yellow pigments, which could possibly be carotenoids. The n-hexane's superior solvating power for carotenoids could explain this outcome.

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