DOI: 10.1002/aocs.12932

ORIGINAL ARTICLE

AOCS * WILEY

Raphia sese unconventional oil from the Congo Basin: Comparison between chemical and screw press extraction

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Funding information

AMES (Appui à la Modernisation de l'Enseignement Supérieur) program; Marien Ngouabi University (Brazzaville, Congo)

Abstract

This study highlights the importance of adding value to Raphia sese (raffia) from the Congo Basin, by using a screw press to extract its pulp oil that can be used in agri-food industry. A 2^k factorial experiment was used to study the influence of the intrinsic parameters of the press (rotation speed and diameter of the output die) on the oil extraction efficiency. Screw press extraction efficiency yield was of 43.3 ± 1.7%, representing an extraction rate of 80.8 ± 6.2%. Mechanical extracted raffia oil was chemically characterized measuring acidity and peroxide values; fatty acid (FA), tocopherol, and sterol compositions, and carotenoids content. These results were compared to those obtained from the analysis of a solvent-extracted oil. The screw press extracted raffia oil exhibited good quality (richer in monounsaturated FA and total sterols; similar total contents in unsaturated FA, tocopherols, and carotenoids; lower acidity and peroxide values), surpassing the solvent-extracted one. Raffia oil's composition would offer a promising alternative to palm oil (obtained from the pulp of Elaeis guineensis fruit). Compared to palm oil, the press- and solvent-extracted raffia oils presented a similar content in saturated FA, \sim 3.3-fold less monosaturated FA, \sim 3.4-fold more polyunsaturated FA, 3.5-fold more linoleic acid, and between 2.4 and 2.8-fold more linolenic acid. Raffia oil's use in food products as a replacement for palm oil would thus allow to diversify edible oil sources, stimulate the local economy, and promote healthier and more sustainable diets.

KEYWORDS

 2^k factorial experiment, Raphia sese, screw press extraction, solvent extraction, unconventional oil

INTRODUCTION

The use of fats is of great importance in the fields of food, pharmacopeia, and cosmetology. In the Congo Basin, particularly in Congo Brazzaville, the available edible oils are mainly imported. The main oils on the market, generally used for cooking (peanut, olive, palm, and sunflower oils) and the manufacture of cosmetic products (cocoa and shea butters), have seen their price double or even triple in recent years, as a result of disruptions in agricultural production and the logistics of distribution chains,

due to factors such as the impact of geopolitical tensions, climate disasters, and increased food demand in some countries. This trend, which is impacting the whole world, is influencing people's eating habits, thus increasing the research of alternative sources known as unconventional fats. The work of Silou (2014) highlighted the potential of the Congo Basin in terms of unconventional oilseeds and oleaginous fruits, as for example Raphia sese (raffia) pulp, which could substitute palm oil (refined oil extracted from the pulp of Elaeis guineensis) on the market. Raffia oil is already consumed as a substitute of red palm oil

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(crude oil extracted from the pulp of E. guineensis fruits) in some regions of Congo Brazzaville. Solvent extracted raffia oil contains: 43.8%–44.9% of total saturated fatty acids (SFA) (being C16:0 (35.7%–37.4%) the major one) and 55.1%–55.4% of total unsaturated fatty acids (being C18:2 (32.9%–38.8%) and C18:1 (15.7–20.8%) the major ones); bioactive compounds of interest such as carotenoids (113.0 mg/kg), tocopherols (493.0 mg/kg), and sterols (155.9 mg/100 g), and a SFA fraction melting point of 23.1°C (similar to that of the palm oil SFA fraction) (Dzondo-Gadet et al., 2004; Goteni et al., 2011).

In general, small producers traditionally extract raffia oil using the hot water flotation method after crushing the pulp and macerating the shredded material (Goteni et al., 2011; Head et al., 1995). Usually, extraction parameters as time and temperature are not defined, and differ from one producer to another, generating hydrolysis and oxidation reactions in the oils. As a result, the oils' quality varies a lot from one producer to another. Raffia oil has already been the subject of some researches, mainly focused on the composition of the traditional and laboratory solvent extracted oil (Dzondo-Gadet et al., 2004; Goteni et al., 2011; Silou et al., 2000). As far as we know, there is no reported work on the optimization of screw press extraction conditions for the production of raffia oil.

Mechanical press extraction is used for extracting oils for food use. Screw presses are the main presses used in oil mills. Among these presses, Komet-type screw presses are the ones that are currently the most used on a small scale thanks to their affordable costs and their versatility (i.e., adapted to several types of plant matrices: seeds, pulps, kernels) (Head et al., 1995). The extraction of oils with a press is generally influenced by its intrinsic parameters (screw's speed of rotation, diameter of the cake exit die), but also by the operations (drying, shelling, grinding, and heat treatment) carried out on the plant matrices before passing through the press.

Among the factors influencing the oil extraction yields, some characteristics of the plant material can be cited: (i) its water content (i.e. for a good yield of coconut oil extraction, it is recommend an optimal moisture content of 4.5%–7%, 10%–12% or 3% if extracted with a screw press, a low-pressure bridge press or a ram press, respectively (Head et al., 1995)); for this, drying is necessary and farmers often apply a sun drying (i. e. from 10 to 20 days for shea nuts depending on weather conditions (Kapseu, 2009)); (ii) its particle size, particularly when large dies are used; (iii) its temperature, as sometimes a thermal pre-treatment (ranging from 60 to 100 $^{\circ}$ C) (Head et al., 1995) is necessary to facilitate the extraction of oil from the plant matrix, especially for those containing oils rich in SFA with high melting points such as palm kernel oil or shea butter; however, it is important to control the temperature to avoid oil degradation.

So, the aim of this work was to produce a good quality raffia oil at room temperature using a Komettype screw press. For this, a 2^k factorial experiment was applied to study the different extraction parameters (screw rotation speed, diameter of the output die) influencing the screw press extraction efficiency yield and extraction rate. The quality of raffia oils obtained using the Komet screw press versus those extracted with solvent using a Soxhlet device, was also compared by evaluating their overall quality (peroxide value (PV) and acid value (AV)) and their chemical composition (fatty acid (FA), tocopherols, and sterols composition; carotenoid content). This work contributes to providing valuable information for R. sese edible oil's process extraction.

MATERIALS AND METHODS

Materials

Plant material

R. sese (raffia) seeds were collected in Cuvette, department of the Republic of the Congo $(1^{\circ} 13^{\circ})$ $27''$ S, 16 $^{\circ}$ 47' 38" E), harvest date: September 13th, 2022.

Chemicals

The chemicals used (analytical grade or equivalent) were purchased from Sigma-Aldrich Co. (Saint-Quentin Fallavier, France): sodium methylate, methanol, acetyl chloride, phenolphthalein, hexane, potassium hydroxide, absolute ethanol, diethyl ether, potassium hydrophthalate, chloroform, acetic acid, potassium iodide, iodine indicator, petroleum ether, copper acetate, phosphoric acid, fatty acid methyl ester standard Supelco 37 component FAME mix, 0.1 N sodium thiosulfate standard solution, soy phytosterols (containing cholesterol, campesterol, stigmasterol, β -sitosterol, and lanosterol standards), betulin standard, β -carotene standard, and tocopherols standard (containing a mix of α -, β -, δ -, and $v-$).

Methods

Sample preparation and water content analysis

The raffia fruits were manually sorted to remove impurities and damaged fruits, and dried for 1 day (at room temperature (28 $^{\circ}$ C) in the dark at the harvest site to reduce their water content). The raffia pulp was then manually separated, before its transportation to France (Figure 1). Water content of raffia pulp was determined in triplicate, by gravimetric analysis, drying 5 g of raffia

FIGURE 1 Dried raffia pulp.

pulp until it reached a constant weight in an oven at $105 \pm 2^{\circ}$ C during 4 h.

Oil extraction

Solvent extraction

The solvent extraction of raffia oil was carried out using a Soxhlet device according to the ISO 659:2009 standard method (ISO, 2009). Raffia pulp was dried in an oven at 40° C for 4 h, and ground with a coffee mill (Moulinex, Ecully, France). Then 15 g were weighed into an extraction cartridge that was placed in the Soxhlet device. The oil was extracted with petroleum ether for 6 h. Then the solvent was evaporated under vacuum using a rotary evaporator $(40^{\circ}C, 90$ rpm). The resulting oils were placed in amber bottles, inerted with nitrogen, and stored at 4° C until use.

Mechanical extraction

The mechanical extraction was carried out at room temperature (18 $^{\circ}$ C), using a Komet CA 59 G press (IBG Monforts Oekotec GmbH & Co KG, Mönchengladbach, Germany) with a capacity of 3–5 kg/h and a power of 1.1 kW (Figure 2). The raffia dried pulp was coarsely ground by hand to fit into the press feed hopper of the Komet press and to be compressed by the auger. The extracted oil was decanted and transferred into amber bottles, then inerted with nitrogen, and stored at 4° C until use. The extraction yield (Y, Equation (1)) and extraction rate $(R, Equation (2))$ were determined as follows:

$$
Y(\%) = \frac{Amount\ of\ extractedoil(g)}{Test\ sample(g)} \times 100\tag{1}
$$

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FIGURE 2 Screw-press Komet CA 59G.

A 2^k factorial experiment was used to model the oil extraction efficiency. Two parameters were considered (X1, X2). The diameter of the output die (X1) (7 and 8 mm) and the rotation speed (X2) (2.2 and 2.4 Ua (arbitrary units)) of the press are essential aspects that modulate the pressure exerted on the raffia pulp, thus influencing the yield and quality of the extracted oil.

Oil analysis

Fatty acid composition analysis

Fatty acid methyl ester (FAME) preparation and gas chromatography (GC) analysis for FA composition were performed according to the NF EN ISO 12966-2 (AFNOR, 2017). FAME were analyzed by GC (FOCUS GC chromatograph (Thermo Fisher Scientific, Massachusetts, USA)) equipped with a CP-Sil 88 column (50 m \times 0.25 mm \times 0.2 µm; Agilent, Santa Clara, USA) and a split injector (1:20 ratio). Carrier gas: helium. Flow rate: 1.0 mL/min. Injector temperature: 250°C. FID detector temperature: 270°C. Oven heated from 185 to 225 \degree C at a speed of 5 \degree C/min and maintained at 225° C for 10 min. FAME were identified by comparing the retention times of each peak to those of the FAME mix standard and quantified as a relative percentage of total FA using Chromcard 2.3.32005 software (Thermo Fisher Scientific, Massachusetts, USA).

Tocopherols' composition analysis

The quantification of tocopherols $(\alpha, \beta, \delta, \text{ and } \gamma)$ was performed according to the ISO 9936:2016 standard

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method (ISO, 2016). Ten milligrams of oil were diluted with 1 mL of hexane. The high-performance liquid chromatography (HPLC) analysis was performed with an Ultimate 3000 chromatograph (Dionex, Massachusetts, USA) equipped with an hypersil silica column (250 mm, 4.6 mm, and 5 μ m; Delaware, USA) and a fluorescence detector (FL 3000, Thermo Fisher Scientific, Massachusetts, USA). The mobile phase consisted of a hexane/1,4-dioxane mixture (97:3, v/v). The column temperature was maintained at 25° C and the flow rate was 1.3 mL/min. Fluorescence detection was set to 290 and 330 nm for excitation and emission, respectively. The injection volume was 100 μL and calibration curves were performed with standard solutions of each tocopherol isomer. Tocopherols were quantified using Chromeleon 7.2.6 software (Thermo Fisher Scientific, Massachusetts, USA).

Sterols' composition analysis

The determination of phytosterols was carried out according to the NF EN ISO 12228-1 standard method (AFNOR, 2014). The oil sample (500 mg) was saponified with 5 mL of alcoholic potassium hydroxide (0.5 N) in the presence of 1 mL of betulin internal standard at 1 mg/mL in chloroform, for 15 min. The unsaponifiable components were separated from the soaps on an alumina column (10 g) and washed with diethyl ether (30 mL). The solvent was evaporated, and the residue was solubilized in 1 mL of chloroform. Finally, sterols isolation was performed by thin-layer chromatography (TLC). TLC plates (TLC silica gel 60 F254, Merck, Darmstardt, Germany) were developed with a chloroform/diethyl ether solution (9:1, v/v). In order to locate the band corresponding to the sterols, a small part of the plate was cut, revealed and visualized with the 85% copper acetate/phosphoric acid solution (5:5, v/v), then placed in the oven at 180° C for 10 min. The corresponding sterol strip of the non-revealed plate was scraped off and transferred to a tube. Then, sterols were desorbed from the silica in 5 mL of chloroform, then centrifuged. The supernatant was filtered (0.45 μm) and evaporated under nitrogen at room temperature. Chloroform (50 μL) was added to the dry extract and 1 $μ$ L of this solution was injected into GC-FID (Thermo Fisher Scientific, Massachusetts, USA) equipped with an Equity-1 column (15 m \times 0.32 mm \times 0.25 µm; Supelco, Saint-Quentin Fallavier, France), using helium as carrier gas at a flow rate of 2.0 mL/min. The temperatures of the injector and detector were 280 and 360° C, respectively. The oven was heated from 180 to 350° C at a rate of 15 $^{\circ}$ C/ min, and maintained at 350° C during 2 min. The area for each peak was obtained by integration in function of the area of the internal standard, using Chromcard 2.4.12007 software (Thermo Fisher Scientific, Massachusetts, USA).

Total carotenoids analysis

Carotenoids were analyzed by colorimetry using the UV spectrometer (Perkin Elmer Lamba, Apeldoorn, The Netherlands), using the UV software WinLab software Lambda 2–40 version 2.8. (Perkin Elmer Lamba, Apeldoorn, Netherlands). One milligram of raffia oil was diluted in 1 mL of hexane and the absorbance was determined at 453 nm. The carotenoid content was determined using the β -carotene calibration curve (0 to 1 mg/mL of hexane).

Quality indexes

The acid value (AV) and peroxide value (PV) were determined according to the ISO 660/2020 (ISO, 2020) and ISO 3960:2017 (ISO, 2017) standard methods, respectively.

Statistical analysis

All samples were analyzed in triplicate, and the results are expressed as means ± standard deviation (SD). The factorial experiment was generated and analyzed using XLSTAT 2022.5.1 software.

RESULTS AND DISCUSSION

Factorial experiment analysis

The factorial experiment analysis aims to identify the crucial parameters influencing the extraction yield (Y) (Equation (1)) of raffia oil using a Komet-type screw press. The detailed analysis of the normalized coefficients, presented in Table 1 and Figure 3, provides valuable insights into these parameters. The normalized coefficient for the output die diameter (X1) is 2.7 (Figure 3), suggesting that optimizing the output die diameter is crucial for maximizing Y, as an increase in the output die diameter significantly enhances Y. In contrast, the rotational speed (X2) has a coefficient of 0.6 (Figure 3), showing a positive but less pronounced effect on Y than the output diameter. This implies that even if increasing the rotational speed can improve Y, its impact is less substantial compared to that of the output die diameter.

The interaction between the output die diameter and the rotational speed (X1*X2) shows a coefficient of -2.8 (Figure 3), indicating a negative influence on Y. This negative interaction suggests that there is an optimal balance between the output die diameter and the rotational speed that must be maintained to avoid diminishing returns on Y.

Based on the factorial experiment analysis, the results presented in Table 1 show that increasing the output die diameter, from 7 to 8 mm, allows to increase Y of 2.4% and 1.5% for a screw rotation speed

TABLE 1 Raffia oil screw-press extraction yields (Y) resulted from the factorial experiment analysis.

Observations	Output die diameter (mm)	Screw rotation speed (Ua)	Y (%)
Obs1		22	43.3
Obs2		24	39.5
Obs3	8	22	45.7
Obs4	8	24	41.0

Abbreviation: Ua, Arbitrary units (press adjustment).

FIGURE 3 Normalized coefficients (extraction yields) of raffia oil. X1: Output die diameter. X2: Rotational speed.

of 2.2 and 2.4 Ua, respectively. When conserving the same output die diameter, the increase of the screw rotation speed from 2.2 to 2.4 Ua decreased Y of 3.8% and 4.7% for an output die diameter of 7 mm and 8 mm, respectively. The best Y (45.7%) was obtained using an output die diameter of 8 mm and a screw rotation speed of 2.2 Ua. These optimized conditions were used for the rest of the work to extract raffia oil with the press. Therefore, to achieve the best extraction performance, it is recommended to focus on optimizing the output die diameter while maintaining an appropriate rotational speed to prevent any negative interactions.

Raffia oil's extraction yield (Y) and extraction rate (R)

Y and R results are presented on Table 4. The obtained Y using the Soxhlet extraction $(53.6 \pm 2.4\%)$ was similar to that reported by Head et al. (1995) (51%–54%) and Dzondo-Gadet et al. (2004) $(52 \pm 0.9\%)$. A new

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batch of raffia oils was obtained using the previously obtained optimized conditions (Table 1): output die diameter of 8 mm and screw rotation speed of 2.2 Ua. Results show that the press extraction was efficient with a Y of $43.3 \pm 1.7\%$ and a R of $80.8 \pm 6.2\%$. These results suggest that press extraction of raffia oil offers significant performance, with a competitive Y, compared to the solvent extraction method.

Fatty acid composition

The results of the FA composition analysis of raffia oil (in relative percent of total FA) presented in Table 2 reveal that the majority FA in this oil are the saturated FA (SFA) followed by the polyunsaturated FA (PUFA). Press extracted oil presented significantly less total SFA than solvent extracted oil $(51.8 \pm 0.08\%$ for press, 52.1 ± 0.12% for Soxhlet), being C16:0 (41.1 ± 0.18% for press, 41.6 ± 0.08% for Soxhlet) and C18:0 (10.2 \pm 0.10% for press, 9.2 \pm 0.05% for Soxhlet) the predominant SFA. With the press it was extracted less C12:0 and C14:0, and more C16:0, C18:0 and C20:0, than with the Soxhlet. Both of our raffia oils presented a higher SFA content than that of the Sohxlet extracted ones reported by Goteni et al. (2011) oil (44.9%) and Dzondo-Galet et al. (2004) (43.8%).

Both raffia oils contained lower levels of monounsaturated FA (MUFA) compared to total SFA or PUFA, representing $12.0 \pm 0.09\%$ for the press (C16:1 (0.14) \pm 0.01%), C18:1 (11.9 \pm 0.08%)) and 11.7 \pm 0.07% (as C18:1) for the Soxhlet extracted oils, respectively. Total MUFA in the press extracted raffia oil is significantly higher than that of solvent extracted one. Both oils presented a lower MUFA content that the ones reported by Goteni et al. (2011) (21.1%) and Dzondo-Gadet et al. (2004) (15.7%) for Soxhlet extracted raffia oil.

Total unsaturated FA in press-extracted oil (48.2 ± 0.08%) was significantly higher than that of the Soxhlet extracted one (47.9 ± 0.12%). Total PUFA content in press-extracted raffia oil was 36.2 ± 0.01% (C18:2 $(35.5 \pm 0.05\%)$, C18:3 $(0.71 \pm 0.04\%)$, and in Soxhlet extracted oil it was 36.2 ± 0.05% (C18:2 (35.3 ± 0.05%), C18:3 (0.85 ± 0.00%)). Both raffia oils presented a higher PUFA content that the one reported by Goteni et al. (2011) (34.1%), but lower than that reported by Dzondo-Gadet et al. (2004) (39.7%) for Soxhlet extracted raffia oil. Even if the press extracted raffia oil contained significantly more C18:2, and less C18:3 than the solvent extracted oil, the total PUFA in both oils was not significantly different. But the ratio n-6/n-3 are significantly different, being 49.8 ± 3.2 and 41.7 ± 0.2 for the press- and the Soxhlet-extracted raffia oil, respectively. Even if the total PUFA content in the press-extracted oil was higher than the Soxhlet extracted one, this last has a lower n-6/n-3 ratio, which

Note: Results are the mean of triplicates (mean values ± standard deviations values). Values in the same row with the same superscript letters are not significantly different at $p \le 0.05$ (ANOVA; $n = 3$).

Abbreviations: MUFA, monounsaturated fatty acids; N.D., not detected; PUFA, polyunsaturated fatty acids; SFA, saturated fatty acids.

could make it more interesting from a nutritional point of view. But it is worth considering that the pressextracted raffia oil belongs to the "cold pressed fats and oils" category, which according to the Codex Alimentarius (FAO & OMS, 1981) they are "edible vegetable fats and oils obtained, without modification of the oil, by mechanical processes, for example expulsion or pressure, and without the use of thermal processes". The solvent extracted raffia oil does not belong to this category and it should be analyzed to verify that residual solvent in raffia oil is inferior to 1 mg/kg, as recommended by the JORF (1998) relating to extraction solvents used in the manufacture of foodstuffs or their ingredients. Besides this, it is worth remembering that industrially, hexane is the solvent used to extract vegetable oils and solvent-extracted oils undergo a refining process. It would be interesting to compare the chemical composition of the press-extracted raffia oil with unrefined and a refined raffia oil extracted with hexane, as the oil yield extraction and composition is influenced by the extracting solvent and the extraction process (Almazan & Aldeyeye, 1998; Hamm et al., 2013; Tir et al., 2012).

According to the classification of oils proposed by Dubois et al. (2008), raffia, palm (E. guineensis), and coconut (Cocos nucifera) oils, and cocoa (Theobroma cacao L.) butter belong to the group of "SFA" and are solid at room temperature (group of "solid fats"). Raffia and palm oils belong to the sub-group of "palmitic acid (C16:0)", coconut oil to that of "lauric acid (C12:0)

 $+$ myristic acid (C14:0)", and cocoa butter to that of "stearic acid (C18:0)". Olive (Olea europea) and canola (Brassica napus L.) oils belong to the group of "MUFA" , and to the subgroup "MUFA $> 60\%$ " and "MUFA $+$ rich in C18:2", respectively. Soja (Glycine max), sunflower (Helianthus annuus L.), and linseed (Linum usitatissimum) oils belong to the group of "PUFA". "MUFA and PUFA oils" are liquid at room temperature of 20° C (group of "liquid oils"). "PUFA oils" are classified into the following subgroups: "C18:2 + MUFA+C18:3" for soya; $"C18:2 + MUFA"$ for sunflower; $"C18:3$ $+$ MUFA" for linseed. Figure 4 shows a comparison of the FA profile of these different vegetable oils (according to contents reported by Dubois et al. (2008) and the values of Table 2 for the press extracted raffia oil; detailed contents of the principal FA are presented and values for solvent extracted raffia oil were not included to not surcharge the figure). The screw-pressed raffia oil contains: (i) the highest PUFA $(36.2 \pm 0.01\%)$ and C18:3 $(0.71 \pm 0.04\%)$ contents, when comparing it, respectively, with coconut (1.9% and 0%), cocoa butter (3.0% and 0.2%), and palm (10.5% and 0.3%) "MUFA oils", and (ii) an interesting content of PUFA and C18:3 when comparing it, respectively, with sunflower (66.0% and 0.5%), olive (10.0% and 0.6%), soy (59.8% and 7.8%), and canola (31.5% and 9.9%) oils. Thus, raffia oil contains a PUFA content between that of canola and soya oils, and a C18:3 content similar to that of olive oil. "MUFA oils" are used to give texture to food products, such as in spreadable fats, but their ratio n-6/ n-3 is superior to 5. In order to tend to the recommended ratio n-6/n-3 < 5, to prevent cardiovascular diseases and other pathologies (AFSSA, 2006), it will be necessary to mix raffia oil with "PUFA oils" richer in C18:3, such as linseed oil (that is the richest in PUFA (71.8%) and C18:3 (55.0%)), canola or soya oils.

When comparing in more detail the FA composition of our press- and solvent-extracted raffia oils with that of E. guineensis palm oil (Dubois et al., 2008), it can be observed that in press- and solvent-extracted raffia oils: (i) total SFA content $(51.8 \pm 0.08$ and $52.1 \pm 0.12\%$, respectively) is similar to that of palm oil, which contains about 50.4% of SFA (C16:0 (43.8%), C18:0 (4.4%)), (ii) total MUFA content $(12.0 \pm 0.09$ and 11.7 \pm 0.07%, respectively) is \sim 3.3-fold lower than that of palm oil, which can contain up to 39.4% of MUFA (with 39.1% of C18:1), and (iii) total PUFA content (36.2 ± 0.01 and 36.2 ± 0.05 %, respectively) is 3.5-fold higher than that of palm oil, which contains only 10.5% PUFA (C18:2 (10.2%), C18:3 (0.3%)). Raffia oils contained 3.5-fold more C18:2 and between 2.4 and 2.8-fold more C18:3 FA than palm oil.

These results show that: (i) the FA global composition of the press extracted raffia oil is more interesting (less SFA, more MUFA, similar PUFA, more total unsaturated FA) than that of the solvent extracted one; (ii) our solvent extracted raffia oil is richer in SFA and

FIGURE 4 Comparison of different vegetable oils (adapted from Dubois et al., 2008) versus the press extracted raffia oil. Total saturated (SFA), monosaturated (MUFA), and polyunsaturated (PUFA) fatty acids (a, b) and main FA composition (c, d) of raffia oil with vegetable oils that are liquid (a, c) or solid (b, d) at room temperature (20 $^{\circ}$ C).

PUFA and poorer in MUFA than that reported by Goteni et al. (2011) and Dzondo-Gadet et al. (2004); (iii) raffia oil is richer in PUFA and C18:3 FA than coconut, cocoa butter, and palm oils, and contains a PUFA content between that of canola and sunflower oils, and a C18:3 content similar to that of olive oil; (iv) raffia oil is a better source of essential PUFA than palm oil, suggesting that raffia oil may be a good substitute for palm oil, that is commonly used to give texture to food products, and whose environmental impact is increasingly contested (Rival & Levang, 2013).

Tocopherols

Tocopherols are important antioxidants present in edible oils and provide vitamin E activity. Tocopherols can act as sacrificial agents, limiting oxidation through various reactions, including their reaction with radicals generated during oxidation, and the synergistic effects with other compounds such as phospholipids, vitamin C, and carotenoids that may further contribute to tocopherol regeneration. Additionally, tocopherols can form complexes with transition metals, inhibiting their prooxidant activity (Barouh et al., 2022; Choe & Min, 2006; Henry et al., 1998). The tocopherol composition of raffia oil is shown in Table 3. Raffia oil exhibited a high tocopherol content, with concentrations of 1654.3 ± 38.1 mg/kg of oil (press) and 1717.5 ± 76.2 mg/kg of oil (Soxhlet). The content of α -tocopherol (1612.0 ± 35.2 and 1672.3 ± 73.4 mg/kg of oil for press and Soxhlet, respectively) was much higher than that of β tocopherol (42.0 ± 3.4) and 45.2 ± 4.0 mg/kg of oil for press and Soxhlet, respectively). Quantitatively, our results differ from those reported by Goteni et al. (2011) who reported 493 mg/kg of oil of total tocopherols in a Soxhlet extracted raffia oil (containing α -, β -, and Δ-tocopherols at 466, 21, and 6 mg/kg of oil, respectively). Qualitatively, α -tocopherol was the major tocopherol in both, press and Soxhlet raffia oil, as in the

TABLE 3 Tocopherol and sterol composition, and total carotenoids content of raffia oil extracted using a press (screw-press Komet) and solvent (Soxhlet).

Compound	Press	Soxhlet
Total tocopherols (mg/kg of oil)	1654.3 ± 38.1^a	$1717.5 + 76.2^a$
α-tocopherol	$1612.0 + 35.2a$	$1672.3 + 73.4^a$
β-tocopherol	$42.0 + 3.4^a$	$45.2 + 4.0^a$
Total sterols (mg/100 g of oil)	532.9 ± 48.1^a	$274.0 \pm 50.2^{\circ}$
Cholesterol	33.9 ± 2.1^a	14.6 ± 0.9^b
Brassicasterol	$5.7 + 1.1a$	8.2 ± 0.2^{b}
Campesterol	19.3 ± 2.6^a	$11.6 + 0.91b$
Stigmasterol	$53.9 + 2.8^a$	$23.2 + 6.5^{b}$
β-sitosterol	381.7 ± 31.7^a	$200.5 \pm 33.5^{\circ}$
Lanosterol	38.5 ± 11.4^a	$16.0 \pm 8.9^{\circ}$
Total carotenoids (mg eg β -carotene/kg of oil)	322.7 ± 0.00^a	322.6 ± 0.69^a

Note: Results are the mean of triplicates (mean values ± standard deviations values). Values in the same row with the same superscript letters are not significantly different at $p < 0.05$ (ANOVA; $n = 3$).

case of the Soxhlet oil reported by Goteni et al. (2011). It can be noted that our raffia oil contained more tocopherols than different palm oil varieties from Côte d'Ivoire $(864.1 \pm 24.5 \text{ to } 1124.4 \pm 14.5 \text{ mg/kg of oil};$ with α -tocopherol as the predominant), as reported by Monde et al. (2009).

Raffia oil belong to the family of α -tocopherol rich oils (>500 mg/kg), as sunflower, cotton, and safflower, according to FAO and OMS (1999). There was no significant difference in the tocopherol content between the press- and the Soxhlet-extracted raffia oils. It is therefore advisable to favor press extraction, which is more environmentally friendly and more economical, rather than solvent extraction to obtain raffia oil.

Sterols

The results of the sterol analysis of raffia oil are given in Table 3. These results show that the oils extracted by press and solvent (Soxhlet) have significantly different total sterol contents: 532.9 ± 48.1 and 274.0 ± 50.2 mg/100 g of press and Soxhlet extracted oils, respectively. The main sterols found in press and Soxhlet raffia oils are β -sistosterol (381.7 ± 31.7 and 200.5 ± 33.5, respectively) and stigmasterol (53.9 ± 2.8 and 23.2 ± 6.5, respectively). Ergosterol, fucosterol, Δ-5 and Δ-7-avenasterol were not detected. According to Phillips et al. (2002) total sterol content in plant oils ranges from 66 (coconut oil) to 1087 (evening primrose) mg/100 g, being sitosterol and campesterol the most abundant. Total sterol contents in our raffia oils were higher than those reported for a solvent-extracted raffia oil (155.9 mg/100 g of oil) (Goteni et al., 2011) and palm oil (66 mg/100 g of oil) (Phillips et al., 2002).

According to the literature, sitosterol represents between 38 and 91% of total sterols in oils and fats, 80.8% of total sterols in Soxhlet extracted raffia oil, and 59.8% of total sterols in palm oil (Goteni et al., 2011; Phillips et al., 2002). In press and Soxhlet extracted raffia oils, β -sitosterol represented, 71.6% and 73.2% of total sterols, respectively.

According to Goteni et al. (2011) a solvent extracted raffia oil contained β -sitosterol (126 mg/100 g), stigmasterol (16.7 mg/100 g), campesterol (11 mg/100 g), and Δ-5-avenasterol (2.2 mg/100 g). In contrast with these results, our raffia oils did not contain Δ-5-avenasterol, but contained in mg/100 g, for press and Soxhlet oils respectively: lanosterol $(38.5 \pm 11.4$ and 16.0 \pm 8.9), cholesterol (33.9 \pm 2.1 and 14.6 \pm 0.9), campesterol $(19.3 \pm 2.6$ and $11.6 \pm 0.91)$, and brassicasterol $(5.7 \pm 1.1$ and $8.2 \pm 0.2)$. Raffia oil obtained by press extraction was a better source of sterols than that extracted by solvent, which is in agreement with what was reported by Tir et al. (2012), namely that polar solvents can extract more sterols from sesame seeds than hexane.

Plant sterols are present as free sterols and conjugated as steryl esters (SE) (a FA is esterified with the OH group of the sterol moiety C3), steryl glycosides (SG) (a sugar is linked through a glycosidic bond with the OH group of the sterol moiety C3) and acyl steryl glycosides (ASG) (a FA is acylated with the C6's OH group of the SG's sugar moiety). The carbohydrate moiety in SG and AGS, render them more hydrophilic and thus more soluble in water than free sterols and SE (Nyström et al., 2012). It would be interesting to quantify the content in SG and AGS in raffia pulp. SG and AGS extraction and their glycosidic bond hydrolysis (by endogenous glycosylases) could have been favored by the raffia pulp water content $(6.2 \pm 0.45\%)$ during the oil press extraction, thereby increasing the total content of free sterols and SE in raffia pressextracted oil. Finally, for both, press- and solventextracted raffia oils, free sterols are liberated from SE during the saponification step during the saponification step in the sterol composition analysis. SGs are not soluble in petroleum ether, they are not extracted by the Soxhlet method, which could explain the lower sterol content in solvent extracted raffia oil.

Carotenoids

Carotenoids play the role of antioxidants like tocopherols in edible oils and are a vitamin A biological source (Choe & Min, 2006; Hamm et al., 2013). Results in Table 3 indicate that the carotenoid contents in the press $(322.7 \pm 0.00 \text{ mg} \text{ eq}$ β -carotene/kg) and in the Soxhlet $(322.6 \pm 0.69 \text{ mg})$ eq β -carotene/kg) extracted raffia oils were similar. These contents are lower than those reported for crude palm oil from

different varieties from Côte d'Ivoire: 831.9 ± 5.5 to 3574.9 ± 2.5 mg of total carotenoids/kg of oil with 580.0 ± 5.7 to 2390.0 \pm 6.4 mg eq β -carotene/kg of oil and the rest as α -carotenoids and non-identified compounds (Monde et al., 2009).

Acid and peroxide values

The results of the acid value (AV) and peroxide value (PV) analysis are presented in Table 4. As seen before for the raffia oil composition, the oil extraction process also influences its quality and stability. AV and PV are important quality parameters which reflect the hydrolytic and oxidative stability of oils. AV (1.4 ± 0.07 mg KOH/g of oil) and PV (10.0 \pm 0.48 meq O₂/kg of oil) of press extracted oil were respectively 4- and 5-fold lower than those from the Soxhlet extracted oil (AV: 6.0 \pm 0.09 mg KOH/g of oil, PV: 50.7 \pm 4.8 meg O₂/kg of oil). The press-extraction method is thus more favorable in terms of maintaining a lower AV and PV, suggesting less oil hydrolytic and oxidative degradation.

Lipases present in mesocarp palm fruits are located contiguous to the external surface of the lipid droplets (Morcillo et al., 2013). During palm fruit ripening and/or when the fruit palm pulp cells are damaged (by incorrect handling after harvest and/or damaged during processing (crushing, grinding, etc.)), endogenous lipases enter in contact with lipids, catalyzing hydrolysis of acylglycerols and increasing free FA and AV (Hamm et al., 2013; Morcillo et al., 2013). These FA, if unsaturated, could be readily chemically and/or enzymatically oxidized (for PUFA, if endogenous lipoxygenases are present)), increasing PV. Before the Soxhlet extraction, raffia pulp was pre-treated (dried at 40° C for 4 h, and milled), and

TABLE 4 Comparison of the extraction yield (Y) and rate (R), acidic value (AV) and peroxide value (PV) of raffia oils extracted using a screw-press Komet (press) and solvent (Soxhlet).

	Press	Soxhlet	Literature data
Dried pulp water content (%)	6.2 ± 0.45		$7.0 - 12.0$ ¹
Oil extraction yield (Y) $(\%)$	$43.3 + 1.7^{a,*}$	$53.6 + 2.4^b$	$51.0 - 54.0$ **,2,3,4
Oil extraction rate (R) $(\%)$	$80.8 + 6.2$		
AV (mg KOH/g of oil)	1.4 ± 0.07 ^a	$6.0 + 0.09^{b}$	≤4.0 ^{5,6}
PV (meg d' $O2$ / kg of oil)	$10.0 + 0.48$ ^a	$50.7 \pm 4.8^{\rm b}$	≤10.0 ^{5,7} , ≤15.0 ^{5,6}

Note: Results are the mean of triplicates (mean values ± standard deviations values). Values in the same row with the same superscript letters are not significantly different at $p < 0.05$ (ANOVA; $n = 3$). *Conditions: output die diameter: 8 mm, screw rotation speed: 2.2 Ua. **Extraction by Soxhlet. ¹Head et al. (1995); ²Goteni et al. (2011); ³Silou (2014); ⁴Dzondo-Gadet et al. (2004); ⁵FAO and OMS (1981); ⁶for crude oils and cold pressed oils; ⁷for refined oils.

placed in a cartridge that underwent a prolonged exposure to solvent (6 h of extraction) at least a temperature around 40–60 \degree C (= petroleum ether boiling point). Thus, it can be supposed that during the pre-treatment and, during at least the first stages of the Soxhlet extraction in the cartridge, there were favorable conditions for lipolysis (still active endogenous raffia pulp lipases, residual water on raffia pulp (6.2 \pm 0.45% w/w, Table 4)), liberating FA and thus increasing AV (Kumar et al., 2016; Wang et al., 2022). Besides this, a more exhaustive compound extraction takes place, as Soxhlet extraction is done until total depletion of lipids in the matrix is achieved by the repeated condensed pure solvent extraction. Therefore, more FA, initially present in the raffia pulp, can be extracted during the 6 h that the solvent extraction lasts, compared to press extraction. In the case of the press extraction, the contact time between lipase and lipids in the raffia pulp is shorter than in the Soxhlet extraction, as neither heat nor milling pre-treatments of the pulp were made before oil extraction, resulting in a lower AV.

To explain the high PV in solvent-extracted raffia oil, it can also be considered that during this process there is: (i) an extended exposure to high temperatures and solvents that can lead to increased oxidation of lipids in oils, and/or (ii) a co-extraction of other oxidizing and/or colored compounds that can elevate the PV (Choe, 2008; Choe & Min, 2006; Hamm et al., 2013; Kim & Yoon, 1990; Wang et al., 2022). Pre-treatment conditions of raffia pulp have an important effect on the final PV's oil. Dzondo-Gadet et al. (2004) reported for a Soxhlet extracted raffia oil a PV of 4.5 ± 0.3 meg O₂/kg and an AV of 3.4 ± 0.9 mg KOH/g of oil, which are lower than ours (Table 4). These authors crushed the fresh raffia pulp, then dried it (105 \pm 2°C for 2 h + at room temperature to complete total drying) and ground it, and then extracted oil for 5 h (6 h for us) in Soxhlet with petroleum ether. Raffia pulp thermal pre-treatment at 105°C inactivates lipases, limiting lipids hydrolysis, and a shorter solvent extraction limits lipids oxidation in oil. We recommend heat treating the raffia pulp at least 80° C, before the solvent oil extraction, in order to inactivate lipases.

In summary, even if the oil extraction yield with the screw press is 1.2-fold lower than that of the Soxhlet extraction, the raffia press-extracted oil contained more MUFA, a similar total content in unsaturated FA (but more C16:1, C18:1, and C18:2, and less C18:3) (Table 2), tocopherols and carotenoids (Table 3), and a higher total sterol content than the solvent-extracted oil. Differences of composition between our raffia oils and data in the literature, can be due to different factors as for example, the degree of maturity of raffia fruits and the correct handle during harvesting, storage, transportation, and processing. In this study, the overall composition of raffia oils would influence the oils' oxidative stability, since antagonistic or synergistic effects may take place, causing or avoiding conservation problems. The content, type,

and ratio of tocopherols and sterols influence the oxidative stability of oils, as it was observed by Fang et al. (2017). According to these authors: (i) refining reduces the concentration of tocopherols and sterols in soybean and rapeseed oils increasing their oxidative instability, (ii) the addition of tocopherols and sterols to these oils improves their oxidative stability up to a certain concentration from which they have a prooxidant effect, and (iii) at certain concentrations, sterols can decrease the PV of these oils synergistically in the presence of tocopherols. In unrefined raffia oils, in addition to tocopherols, other compounds may be present that possess antioxidant (i.e., phenolic compounds, and carotenoids) or prooxidant (i.e., transition metals, chlorophyll, free FA, and peroxides) activities, and the extraction method has a direct influence on the content of these molecules in oils (Barouh et al., 2022; Choe, 2008; Hamm et al., 2013). Refining raffia oil would allow to obtain an oil with a high triacylglycerols content and a low AV, PV, pro-oxidant components and solvent residues. However, during refining some molecules possessing interesting biological and/or physicochemical properties (i.e., tocopherols, sterols, carotenoids, and phenolic compounds) and organoleptic properties, contributing to color and flavor, will be taken away (Choe, 2008; Hamm et al., 2013; Johnson, 2002). Solvent-extracted vegetable oils may contain traces of solvents and toxic molecules and must be further refined. It would be interesting to compare pressed raffia oil with refined solvent-extracted raffia oil.

Only the press extracted raffia oil AV and PV respected the recommended values by the Codex Alimentarius standard for edible oils: ≤4.0 mg KOH/g for crude oils or cold pressed oils and ≤10 or ≤15 meg O₂/ kg of oil for refined or crude and cold pressed oils, respectively (FAO & OMS, 1981). Therefore, the solvent-extracted raffia oil was of lower quality. Additionally, it already had a shelf-life issue, given its high AV and PV. This further reinforces the advantage of using press extraction for assuring a good raffia oil quality intended for food applications, where oxidative stability is crucial.

CONCLUSION

This study highlights the importance of adding value to raffia from the Congo Basin. R. sese can be used as a new local source to obtain an oil that can be used in artisanal and industrial food manufacturing, especially in a context where food prices are rising. It was shown that a better raffia quality oil is extracted using a screw press, than using a solvent extraction by Soxhlet. The press raffia oil presented: (i) a lower content of total SFA, (ii) a richer content of total MUFA, total unsaturated FA (with more C16:1, C18:1, and C18:2, and less C18:3), and sterols, (iii) similar contents of total PUFA, tocopherols, and

carotenoids, and (iv) lower AV and PV values respecting the Codex Alimentarius recommendations (FAO & OMS, 1981), than the Soxhlet extracted oil. The screw press extraction parameters influencing the extraction yield were studied and allowed to obtain a Y of $43.3 \pm 1.7\%$ and a R of $80.8 \pm 6.2\%$. This simpler, cheaper and more environmentally friendly process is a competitor to the solvent extraction method. It is already used both on a small scale (by local producers) and on a large scale (by the food industry) for the processing of various oilseeds. Additionally, extraction by pressing is much faster and offers better energy efficiency due to fewer processing steps, than the solvent one. There's no need for heating for 6–8 h, no solvents are used, and there's no evaporation step required. This method is also less hazardous to health, with reduced risks of fire and explosion due to the absence of solvents. In terms of parameters influencing the oil extraction, our study showed that the intrinsic parameters of the press (rotation speed, diameter of the output die) had a positive influence on the extraction of raffia oil. These results pave the way for the optimization of the screw-press extraction process of this oil. Research on the valorisation in food and non-food applications of the press residues should be done to complete the overall valorisation of this plant biomass.

Raffia oil offers a promising alternative to palm oil and represent an interesting source of unsaturated FA $(48.2 \pm 0.08\%)$, and minor compounds as tocopherols, sterols, and carotenoids, which possess interesting chemical and/or biological properties.

It is thus possible to add value to raffia through the extraction of its oil, to diversify the edible oils supply in the Congo basin region, and to stimulate the local economy by promoting the production and trading of this oleaginous fruit. In addition, the use of raffia oil in the agri-food industry could contribute to promote healthy and sustainable diets, offering alternatives to imported products and valuing the traditional knowledge and practices of local populations.

AUTHOR CONTRIBUTIONS

Mignon Prince Exaucé Taty: Formal analysis; investigation; methodology; validation; visualization; writing – original draft preparation; writing – review & editing. Bob Wilfrid Loumouamou and Jean Mathurin Nzikou: Formal analysis ; methodology; writing – review & editing. Michel Elenga and Maria Cruz Figueroa-Espinoza: Conceptualization; formal analysis; methodology; project administration; funding acquisition; supervision; writing – review & editing. Bruno Baréa: Conceptualization; investigation; methodology; validation; writing – review & editing. Nathalie Barouh and Jean-Paul Danflous: Investigation; methodology; writing – review & editing. All authors contributed to and approved the final draft of the manuscript.

ACKNOWLEDGMENTS

Authors thank the AMES (Appui à la Modernisation de l'Enseignement Supérieur) program between l'Institut Agro Montpellier (Montpellier, France) and Marien Ngouabi University (Brazzaville, Congo) for funding of this project and for the PhD fellowship grant of Mignon Prince Exaucé TATY.

CONFLICT OF INTEREST STATEMENT

The authors declare that they have no conflict of interest.

ETHICS STATEMENT

No humans or animals were used in this research.

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How to cite this article: Taty MPE, Loumouamou BW, Elenga M, Baréa B, Barouh N, Danflous J-P, et al. Raphia sese unconventional oil from the Congo Basin: Comparison between chemical and screw press extraction. J Am Oil Chem Soc. 2024. https://doi. org/10.1002/aocs.12932