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INTEGRATED VALORIZATION OF NEEM SEEDS (*AZADIRACHTA INDICA*): OIL YIELD AND QUALITY, AND POTENTIAL FOR SOAP, BIODIESEL, AND AGROMATERIALS

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ABSTRACT

This study aims to establish an integrated valorization pathway for neem seeds (*Azadirachta indica*) harvested at the INP-HB (Yamoussoukro, Côte d'Ivoire), addressing the local underutilization of this abundant oleaginous resource. Three oil extraction methods (hot pressing, cold pressing, and Soxhlet hexane extraction) were compared, followed by comprehensive physicochemical characterization of the oils (acid, saponification, iodine, and peroxide values; cetane index; and lower heating value). Additionally, solid residues (shells and press cakes) were processed via thermocompression into eleven agromaterial formulations, with selected samples subjected to water-immersion testing. Solvent extraction achieved the highest yield (65.01% ± 2.4, on ground kernels). Cold-pressed oil exhibited the highest saponification value (212.46 mg KOH g^{-1}, single determination), favourable for soap production, while all oils showed lower heating values (39.47–41.81 MJ kg^{-1}) comparable to diesel fuel, indicating biodiesel potential, pending deacidification of the high-acid-value oils (27.6–41.76 mg KOH g^{-1}). Furthermore, thermocompressed agromaterials derived from solid residues demonstrated superior water-absorption resistance compared to conventional hemp shiv, with a maximum mass gain of only 50.83% (shell-only plate, at the onset of its disintegration). This work demonstrates the technical feasibility of an integrated valorization chain, transforming neem seed waste into high-value cosmetic, energy, and construction materials within a circular-economy framework tailored to the West African agro-industrial context.

Keywords: *Azadirachta indica*; Neem oil; Extraction processes; Soap making; Biodiesel; Agromaterials

1 INTRODUCTION

Agriculture remains a structural pillar of the Ivorian economy: it employs close to half of the labour force [1] and contributes decisively to the country's export earnings, notably through cocoa, cashew, rubber, oil palm, and cotton [2, 3]. This productive dynamic is nevertheless accompanied by a considerable generation of harvest and processing residues, estimated at 15 to 17 million tonnes per year nationwide [4]. A substantial share of this residual biomass remains unvalorized today, even as the scarcity of fossil resources and the imperatives of the circular economy call for these flows to be reconceived as deposits of secondary raw materials rather than as simple waste.

Beyond the food and cash-crop sectors, the Ivorian agricultural landscape includes numerous woody species not intended for consumption, planted for ornamental purposes, windbreak hedges, or traditional pharmacopoeia. The neem tree, *Azadirachta indica* A. Juss (Meliaceae), is an emblematic example. Native to the Indian subcontinent and the dry forests of South and Southeast Asia [5], it was introduced to West Africa during the twentieth century and became widely naturalized there owing to its remarkable tolerance to hot, dry climates [6, 7]. On the INP-HB campus in Yamoussoukro, as in many Ivorian cities, the tree is today ubiquitous as an element of urban landscaping, even though its seeds, produced abundantly (30 to 40 kg of fruit per tree per year at full maturity), fall to the ground and are regarded as waste with no recognized economic use.

This situation contrasts sharply with the documented potential of the neem seed, whose kernel contains approximately 45% oil, and whose biopesticidal, antibacterial, and hypoglycaemic properties have long been known and exploited in traditional medicine [7, 8]. Several studies conducted on West and Central African neem populations have characterized cold- or solvent-extracted oil and have shown saponification values and heating values compatible with soap-making and biofuel applications [9-12]. These studies, however, most often address a single extraction process and rarely cover, within the same work, the entire valorization chain, that is, the oil itself together with the two solid co-products of extraction, the shells, and the press cakes. The fate of these residues, when studied, is most often examined from an agronomic angle, as a soil amendment or biopesticide [13], or from an energy angle, as a solid fuel [14] or a pyrolysis feedstock [15], and, more recently, as biochar for thermal energy storage [16] or as filler particles in polymer composites [17], but rarely from the angle of a construction agromaterial.

Consequently, a scientific gap remains on two complementary fronts. First, the effect of the extraction process, hot pressing, cold pressing, or solvent extraction, on the yield and physicochemical quality of Ivorian neem oil remains insufficiently documented in a comparative manner, even though this choice directly conditions the oil's subsequent uses. Second, the joint material valorization of shells and press cakes as bio-based agromaterials for the building sector remains scarcely documented for this species, even as neighbouring value chains, based on other oleaginous or lignocellulosic residues (pongamia press cake, coconut fibre, hemp shiv), demonstrate the technical feasibility of such valorizations [18-20].

The present study addresses this dual perspective and pursues one overall objective: to achieve an integrated valorization of the products derived from processing neem seeds collected in Côte d'Ivoire. Three specific objectives follow: to evaluate the influence of three extraction processes, hot, cold, and solvent, on the yield and quality of the resulting oil; to characterize the physicochemical properties of this oil with respect to its potential applications in soap making and biodiesel; and to develop and preliminarily characterize agromaterial formulations incorporating the shells and press cakes generated during extraction. The article follows the IMRAD structure: Section 2 details the plant material, the extraction processes, and the analytical methods used; Section 3 presents and discusses the results obtained, systematically set against published work on neem and on comparable raw materials; Section 4 concludes with the contributions, limitations, and outlook of this work.

2 MATERIAL AND METHODS

2.1 Plant material and study area

The seeds used in this study were collected from neem trees (*Azadirachta indica*) growing on the central campus of the Institut National Polytechnique Félix Houphouët-Boigny (INP-HB), in Yamoussoukro, Côte d'Ivoire. Fruits that had reached physiological maturity and fallen naturally to the ground were collected by hand and then sorted to remove impurities (plant debris, unripe or deteriorated fruit). The resulting seeds constituted the starting material for the complete set of trials.

2.2 Biomass pretreatment

The collected seeds were dried for five days at 50 °C in an electric dehydrator, then hulled by hand with a mortar and pestle, which separated the kernels, intended for oil extraction, from the shells, intended for agromaterial formulation. The kernels then underwent a second five-day drying step at 50 °C before extraction.

2.3 Physicochemical characterization of the biomass

The moisture content of the seeds, kernels, press cakes, and shells was determined by oven-drying, following the principle of the AOAC method [21]: a test portion of approximately 1 g, previously weighed in a tared crucible, was placed in an oven at 105 °C for 24 hours, cooled in a desiccator, and reweighed. The moisture content (MC, in %) was calculated from the mass of the empty crucible (m_0), the mass of the sample before oven-drying (m_1), and the mass after oven-drying (m_2), according to Equation 1:

$$MC\ (\%) = [(m_1 - (m_2 - m_0)) / m_1] \times 100 \qquad (1)$$

Dry matter (DM, in %) was deduced by difference:

$$DM\ (\%) = 100 - MC \qquad (2)$$

Ash content was determined following the principle of the AOAC method [22], by incinerating a test portion of approximately 1 g in a muffle furnace at 550 °C for 6 hours, followed by cooling in the furnace and then in a desiccator before weighing. The ash content (AC, in %) was calculated from the mass of the empty crucible (m_0), the mass of the sample (m_1), and the mass after incineration (m_2), according to Equation 3:

$$AC\ (\%) = [(m_2 - m_0) / m_1] \times 100 \qquad (3)$$

Each determination was conducted in triplicate, and results are expressed as the mean ± standard deviation.

2.4 Oil extraction

Oil was extracted using three distinct processes, each applied separately to kernel batches derived from the same pretreatment.

Hot-press extraction was performed with a screw-type oil press (VEVOR), into which the kernels were introduced without any thermal pretreatment other than that generated by the mechanical friction of the screw; the oil was collected at the lower outlet, while the press cake was discharged laterally.

Cold-press extraction was performed with a thermo-press used without external heat input, as the available screw press had no dedicated cold-pressing function.

Solvent extraction was conducted following the principle of the Soxhlet method [23], using hexane as a non-polar solvent. A 10 g portion of raw material (whole or ground kernels, depending on the trial) was placed in a cellulose cartridge and extracted under reflux with 150 mL of hexane for 6 hours at 250 °C on the hot plate. The solvent was then removed by rotary evaporation, and the residual oil was dried in an oven (105 °C, 15 minutes) before weighing. The fat content (FC, in g per 100 g of dry matter) was calculated from the mass of the empty flask (EF), the mass of the flask containing the oil after extraction (OF), and the test portion (W_p), according to Equation 4:

$$FC\ (g/100\ g\ DM) = [(OF - EF) / W_p] \times 100 \qquad (4)$$

For all processes, the extraction yield (Y, in %) was calculated from the initial mass of kernels used (m_0) and the mass of oil actually recovered (m), according to Equation 5:

$$Y\ (\%) = (m / m_0) \times 100 \qquad (5)$$

2.5 Physicochemical characterization of the oil

The pH of the oils was measured directly with a pH meter. Water and volatile matter content was determined according to ISO 662:2020 [24], by oven-drying (105 °C, 1 hour) a 5 g test portion, following a calculation principle analogous to that used for biomass moisture content (Equation 1).

Acid value and acidity were determined by acid-base titration after dissolving 0.405 g of oil in a diethyl ether/0.2 N alcoholic potash mixture, in the presence of phenolphthalein, following a protocol adapted from the literature [25]. The

acid value (AV, in mg KOH per gram of oil) and the acidity (expressed as oleic acid equivalent) were calculated from the normality of the titrant HCl solution, the equivalence volumes recorded for the sample and the blank, the molar mass of KOH (56.1 g mol^{-1}), and that of oleic acid (282 g mol^{-1}).

Peroxide value was determined by iodometry, following the principle described by Dahamna and Chergui [25]: the oil is dissolved in a chloroform/glacial acetic acid mixture, treated with potassium iodide, and the liberated iodine is titrated with a 0.01 N sodium thiosulfate solution in the presence of starch as a colour indicator. The result is expressed in milliequivalents of active oxygen per kilogram of fat (meq O_2 kg^{-1}).

Iodine value was determined by the Wijs method, by adding Wijs reagent to a solution of oil in chloroform, followed, after one hour in the dark, by titration of the liberated iodine with 0.1 N sodium thiosulfate in the presence of potassium iodide and starch. The result is expressed in grams of iodine fixed per 100 g of oil.

Saponification value was determined by reflux saponification of a 2 g portion of oil in the presence of 0.5 N ethanolic potash, followed by back-titration with 0.5 N hydrochloric acid in the presence of phenolphthalein. The result is expressed in mg of KOH required to saponify 1 g of fat.

Cetane index was estimated following the principle of the ASTM D976 method, from the saponification value (SV) and the iodine value (IV), using the empirical relation 46.3 + (5458 / SV) − (0.225 × IV) [10].

Lower heating value (LHV) was estimated, in the absence of an available bomb calorimeter, from an empirical relation linking LHV (in kcal kg^{-1}) to the saponification value and the iodine value [26], as cited by Diedhiou [10], with results converted to MJ kg^{-1} (1 kcal = 0.004184 MJ).

2.6 Formulation and characterization of the agromaterials

The shells and press cakes obtained from the hulling and extraction steps were ground separately, then subjected to particle-size analysis by successive sieving on a column of four sieves (1 mm, 400 µm, 200 µm, and 150 µm).

Eleven agromaterial formulations were developed by varying the mass proportion of shells (matrix) from 100 to 0%, in 10-point increments, the complementary fraction consisting of press cake (binder). Each formulation used a total mass of 30 g of raw material, shaped by thermocompression (thermo-press and dedicated mould) at a temperature of 140 to 160 °C for 5 minutes, under a pressure of 10 to 30 tonnes depending on the formulation.

The resulting plates were subjected to morphological characterization (length, thickness, visual appearance) and a water-absorption test. The latter was performed on three representative formulations, namely 0% shells (sample designated Plate 1), 50% shells (Plate 2), and 100% shells (Plate 3), immersed in 1 L of distilled water for 5 minutes, with each sample weighed at one-minute intervals after surface wiping. Mass gain (in %) was calculated from the wet sample mass (M_{abs}) and its initial dry mass (M_d), according to the relation adapted from [27]:

$$Mass\ gain\ (\%) = [(M_{abs} - M_d) / M_d] \times 100 \quad (6)$$

2.7 Statistical analysis

Physicochemical measurements were performed in triplicate (except for the determinations flagged in Table 2), and results are expressed as the arithmetic mean ± standard deviation. Data processing (calculation of means and standard deviations, and chart construction) was carried out in Microsoft Excel. No formal inferential statistical test (analysis of variance, comparison of means) was applied in this exploratory work; this point is discussed among the study's limitations (Section 3.7).

3 RESULTS AND DISCUSSION

3.1 Physicochemical characteristics of the biomass

Table 1 summarizes the moisture and ash contents determined for neem seeds, kernels, press cakes, and shells.

After drying, the seeds and kernels show moisture contents of 3.7% ± 0.15 and 3.71% ± 0.07, respectively, well below the 10% threshold set by ISO 659:2009 [28] for oleaginous seeds at the time of extraction. This compliance is practically important: excessive residual moisture would promote enzymatic hydrolysis of the triglycerides during storage and extraction, resulting in an increase in the free acidity of the oil. These values also fall within the 3 to 9% range reported for oleaginous seeds in general [29].

The moisture content of the press cake (9.96% ± 0.22) is, by contrast, noticeably higher than that reported by Djenontin [9] for Beninese neem press cake (4.6%), which is most likely explained by the absence of a post-extraction drying step in the protocol followed here. The ash content of the press cake (4.34% ± 0.15) is, in contrast, of the same order as the 5.3% reported by the same author and lies just below the 4.6 to 6.3% range recorded for rubber-seed press cake [30], suggesting a comparable mineral content among tropical oleaginous co-products of different botanical origin. The shells show a moisture content of 9.85% ± 0.35 and an ash content of 2.00% ± 0.33, the latter being close to the 2.3% reported by Djenontin [9], which reflects a low mineral matter content consistent with the essentially lignocellulosic nature of this co-product.

Table 1: Moisture content and ash content of the raw materials derived from neem seeds.

Raw material	Moisture content (%)	Ash content (%)	Comparison reference
Seeds (before drying)	10.08 ± 0.07	-	-
Seeds (after drying)	3.7 ± 0.15	3.97 ± 0.63	ISO 659:2009 [28]
Kernels (before drying)	6.63 ± 0.24	-	-
Kernels (after drying)	3.71 ± 0.07	5.93 ± 0.49	-
Press cake	9.96 ± 0.22	4.34 ± 0.15	Djenontin (2006) [9]; Enderlin & Le Bras (1937) [30]
Shells	9.85 ± 0.35	2.00 ± 0.33	Djenontin (2006) [9]

Values are mean ± standard deviation (n = 3). A dash indicates a determination not applicable at that processing stage.

3.2 Extraction yields by process

Figure 1 illustrates the yields obtained for the three extraction processes tested alongside their standard deviations.

Hot-press mechanical extraction gave a yield of 36.15% ± 4.36, a value close to that reported by Louni [29] for the hot extraction of *Moringa oleifera* seed oil (41.33%) and markedly higher than the oil contents generally accepted for soybean (15 to 25%) and olive (17 to 30%), although lower than that of groundnut (approximately 50%). Neem thus ranks among the seeds with high oleaginous potential, which strengthens the case for a simple mechanical extraction chain that is transposable to a village or artisanal scale.

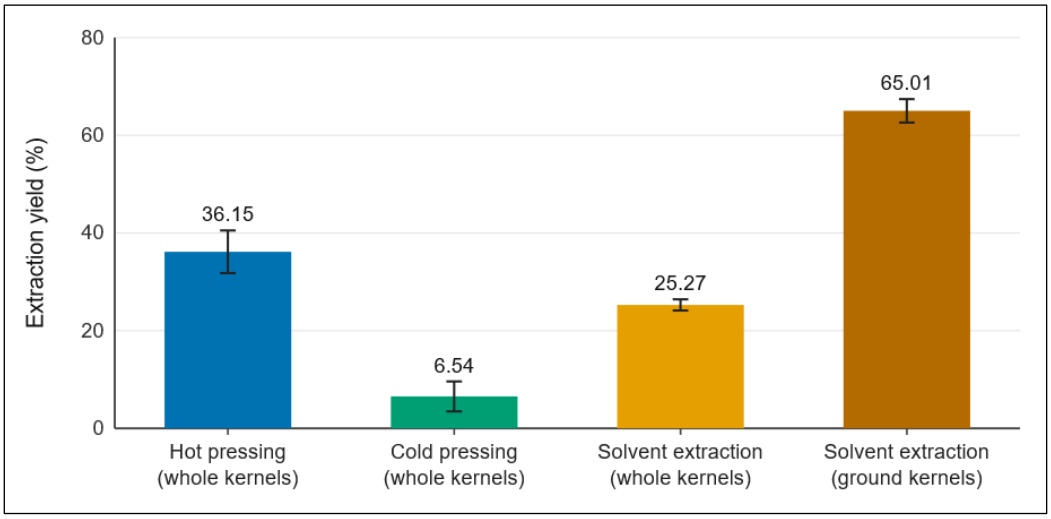


Figure 1: Oil extraction yield according to the process and the kernel particle size. Error bars represent the standard deviation (n = 3)

Solvent extraction revealed a marked effect of the particle size of the raw material: the yield obtained on whole kernels (25.27 g per 100 g DM ± 1.14) rose to 65.01 g per 100 g DM ± 2.4 after grinding, a relative increase of 157%. This result is explained by the increase in the contact surface area between the solvent and the oleaginous matrix following grinding, which facilitates the diffusion of hexane within the cellular tissue and the migration of triglycerides into the solvent phase. A comparable trend was reported by Dahamna and Chergui [25], who obtained a yield of 44.21% for the

solvent extraction of ground *Peganum harmala* seeds. The same effect was reported for neem itself by Agu et al. [31], whose hexane yields rose as particle size decreased.

Compared with earlier work on neem, Djenontin [9] reported a fat content of 44% at 9% moisture, while Diedhiou [10] determined a value of 49%; when our own results are adjusted to a comparable moisture content of 9%, the fat content obtained in the present study amounts to 61.43%, a value higher than both references. This high yield, although consistent with the literature in terms of trend, exceeds the upper bound generally accepted for the oil content of the neem kernel (on the order of 40 to 50%) and the 54.14% obtained by Agu et al. [31] under optimized hexane-extraction conditions (0.5 mm particles, 55 °C, 150 min); it cannot therefore be excluded that a fraction of the mass quantified by the Soxhlet method corresponds to non-triglyceride lipophilic compounds (waxes, pigments, terpenoid compounds) co-extracted by hexane, which calls for a cautious reading of this result and for its confirmation by complementary assay methods, such as gas chromatography of fatty acids.

Cold-press extraction, by contrast, gave an exceptionally low yield of 6.54% ± 3.07, far below the 40.1% ± 1.1 reported by Nitièma-Yefanova et al. [11] under comparable operating conditions (whole kernels), a yield about six times lower. This discrepancy is mainly explained by the limitations of the equipment used: the thermo-press, used in the absence of a dedicated cold-pressing function, most likely did not reach the optimal working pressure for this type of extraction, unlike hydraulic presses or screw presses specifically designed for cold trituration. This observation underscores the importance of matching available equipment to the intended process, particularly for cold pressing, which is more demanding in terms of mechanical pressure than hot pressing.

3.3 Physicochemical properties of neem oil

Table 2 summarizes the physicochemical characteristics measured for the hot-pressed (oil C), solvent-extracted (oil S), and cold-pressed (oil F) oils.

Table 2: Physicochemical characteristics of neem oils according to the extraction process

Parameter	Oil C (hot)	Oil S (solvent)	Oil F (cold)
Water and volatile matter content (%)	0.09 ± 0.01	29.51 ± 0.05	Not determined
pH	4.34 ± 0.04	4.17 ± 0.15	Not determined
Acid value (mg KOH g^{-1})	41.76 ± 4.82	27.60 ± 2.96	36.79 ± 5.81
Acidity (%, oleic acid eq.)	3.41 ± 0.39	2.29 ± 0.21	3.05 ± 0.47
Peroxide value (meq O_2 kg^{-1})	0.10[a]	0.33 ± 0.06	0.73 ± 0.06
Iodine value (g I_2/100 g oil)	0.61 ± 0.13	0.44 ± 0.19	0.66 ± 0.10
Saponification value (mg KOH g^{-1})	183.26 ± 34.78	151.45 ± 9.18	212.46[a]
Cetane index (dimensionless)	30.06	36.37	25.93
Lower heating value (MJ kg^{-1})	40.59	41.81	39.47

Values are mean ± standard deviation (n = 3) unless noted. [a] Single determination, no standard deviation available.

3.3.1 Water and volatile matter content, and pH

The water and volatile matter content of the hot-pressed oil is exceptionally low (0.09% ± 0.01), reflecting effective prior drying of the raw material and the absence of water input during the process. Conversely, the solvent-extracted oil shows a markedly higher water content (29.51% ± 0.05), a result that cannot be attributed with certainty: residual water in the solvent or condensation during rotary evaporation are possible causes, but residual hexane, which is volatile at 105 °C and therefore counted as volatile matter, is at least as likely. This elevated content is a point of attention for the subsequent valorization of the solvent-extracted oil, since water promotes triglyceride hydrolysis and microbial proliferation during storage; a complementary drying step, or even decantation, therefore appears necessary before any industrial use of this fraction.

The pH of the oils obtained (4.34 ± 0.04 for oil C; 4.17 ± 0.15 for oil S) falls within the range usually reported for commercial vegetable oils, such as coconut or sunflower oil (pH between 4 and 7). This parameter nonetheless remains of limited informative value regarding the intrinsic quality of a fat, since oils are non-aqueous media for which the strict notion of pH applies poorly; the acid, peroxide, iodine, and saponification values discussed below provide far more relevant information on the quality and fitness for use of these oils.

3.3.2 Acid value and acidity

The acid values of the hot- and cold-extracted oils (Figure 2a) reach 41.76 ± 4.82 and 36.79 ± 5.81 mg KOH g^{-1}, respectively, while the solvent-extracted oil shows a markedly lower value (27.6 ± 2.96 mg KOH g^{-1}). All three values remain elevated relative to the 10.2 mg KOH g^{-1} reported by Diedhiou [10] and the 8.97 to 9.16 mg KOH g^{-1} reported by Hamadou et al. [12] for Cameroonian neem oils and reflect a more advanced degree of hydrolysis of the oils studied here. The corresponding acidity (3.41% ± 0.39 for oil C, 2.29% ± 0.21 for oil S, and 3.05% ± 0.47 for oil F) nonetheless remains below the 5.6% reported by Djenontin [9], placing the oils studied here in an intermediate position relative to the available references. These relatively high acid values indicate that neem oil, under the harvesting, drying, and extraction conditions applied here, is not perfectly stable at room temperature, and that partial refining, whether by alkaline neutralization (deacidification) or by prior esterification of free fatty acids, would be necessary before any energy valorization demanding in terms of quality, such as biodiesel production by conventional alkaline transesterification, which is sensitive to free-fatty-acid content.

3.3.3 Peroxide value

The peroxide value measured for the three oils (0.1 to 0.73 meq O_2 kg^{-1}) is markedly lower than the values reported by Diedhiou [10] (1.49 meq O_2 kg^{-1}) and Faye [7] (6.76 meq O_2 kg^{-1} on average). A low peroxide value reflects a limited primary oxidation state of the unsaturated fatty acids, which is a favourable indicator of freshness at the time of analysis, including for the solvent-extracted oil. This result, in apparent contradiction with the elevated acid value discussed in the preceding paragraph, is explained by the fact that these two parameters report on distinct degradation mechanisms: acid value essentially reflects triglyceride hydrolysis, whereas peroxide value reports on the radical oxidation of double bonds. The oils studied therefore appear partially hydrolysed but still weakly oxidized at the time of characterization, which justifies prompt storage after extraction to limit the subsequent evolution of these two parameters.

3.3.4 Iodine value

The average iodine value measured in this study (0.44 to 0.66 g iodine per 100 g of oil) is very much lower than the values reported in the literature for neem oil, whether from the work of Diedhiou [10], Djenontin [9], and Faye [7], or from the more recent work of Hamadou et al. [12], who determined iodine values between 73.8 and 74.4 g/100 g for Cameroonian neem oils. This discrepancy, of more than two orders of magnitude, far exceeds the biological variability expected among provenances and also exceeds the standard deviation of the other indices measured in this study. It therefore seems more likely to reflect a methodological difficulty, for instance a unit-reporting error, a test mass poorly matched to the concentration of the Wijs reagent, or a failure to detect the colorimetric endpoint, rather than a genuinely near-zero unsaturation of the fatty acids of the neem studied, which would contradict the unsaturated fatty acid composition (oleic, linoleic) classically reported for this oil. This result is therefore treated here with caution and explicitly flagged as a methodological limitation requiring a repeat determination of the iodine value in future work, rather than as a confirmed intrinsic characteristic of the oil studied.

3.3.5 Saponification value and soap-making potential

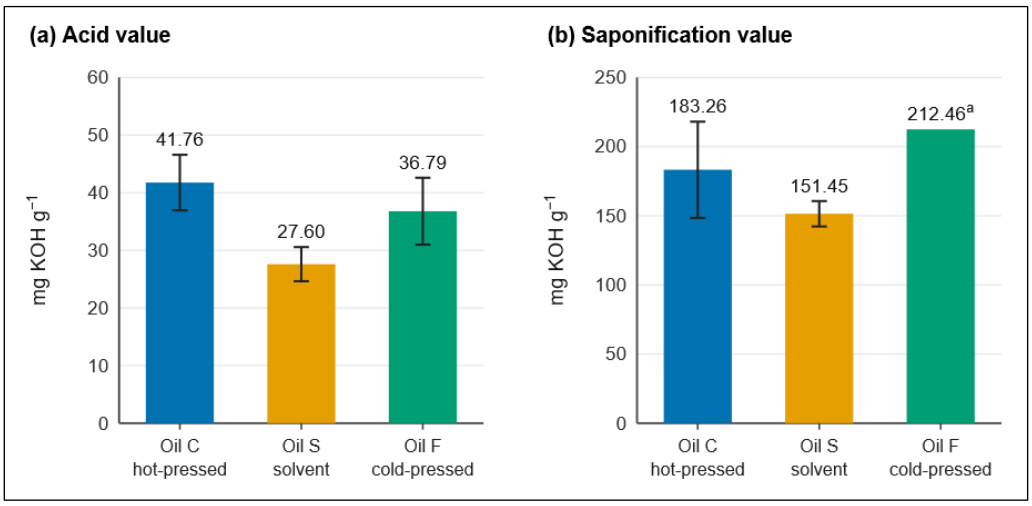


Figure 2: (a) Acid value and (b) saponification value of neem oils by extraction process. Error bars represent the standard deviation (n = 3; a: the saponification value of oil F is a single determination)

The saponification value is the parameter most linked to a fat's aptitude for soap manufacture, a high value reflecting a substantial proportion of low-molecular-weight triglycerides favourable to the saponification reaction. The values obtained in this study are high for all three extraction processes, with a maximum of 212.46 mg KOH g^{-1} for the cold-

pressed oil (Figure 2b), against 183.26 ± 34.78 mg KOH g^{-1} for the hot-pressed oil and 151.45 ± 9.18 mg KOH g^{-1} for the solvent-extracted oil. The higher index obtained for cold extraction may be related to the absence of heating of the raw material, which could better preserve the integrity of the native triglycerides, although this difference must be interpreted cautiously given the single determination for oil F and the large standard deviation for oil C. These values are consistent with those reported by Diedhiou [10], Djenontin [9], and Faye [7], and fall within the same range as the 200.45 mg KOH g^{-1} measured by Owoicho [32] on a neem oil used for soap manufacture in Nigeria, whose finished product moreover met the usual specifications for pH (10.03), total fat content, and foaming power. This convergence strengthens the case for the aptitude of neem oil, particularly when cold-pressed, for soap manufacture, a valorization already documented for this raw material [33], as cited by Diedhiou [10], and consistent with the dermo-cosmetic properties traditionally attributed to neem in West Africa.

3.3.6 Cetane index and lower heating value: biodiesel potential

The average cetane index of the oils studied (Figure 3a; 30.79, with individual values of 30.06 for oil C, 36.37 for oil S, and 25.93 for oil F) remains below the range of 45 to 55 recommended for diesel engines, and well below the value of 82.22 reported by Diedhiou [10]. This difference, which might appear concerning for the direct use of the crude oil as a fuel, must be put into perspective: the cetane index of a crude vegetable oil is structurally lower than that of the biodiesel obtained after transesterification, which reduces viscosity and improves the ignition quality of the product. Improvement routes such as methyl or ethyl transesterification, already successfully implemented on neem oil by Radha and Manikandan [34] and more recently optimized via heterogeneous catalysis [12] (with a biodiesel yield of 89%), or the addition of ignition-improving additives, would likely bring the cetane index of neem biodiesel closer to the thresholds required for diesel-engine use, as has also been demonstrated for blends of non-conventional oils combining notably *Azadirachta indica*, *Hevea brasiliensis*, *Madhuca longifolia*, and *Gossypium hirsutum*.

The lower heating value of the three oils (Figure 3b), ranging from 39.47 to 41.81 MJ kg^{-1}, is consistent with the values reported by Diedhiou [10] for Senegalese neem oil, and is particularly close to the 40.997 to 41.008 MJ kg^{-1} measured by Hamadou et al. [12] on Cameroonian neem oils, which strengthens confidence in the reproducibility of this energy parameter across the continent. These values also remain close to the heating value of diesel fuel (43.8 MJ kg^{-1}) [35], given that the lower heating value accounts for approximately 80% of the aptitude of a vegetable oil or animal fat for biofuel use [36, 37], as cited by Diedhiou [10]. Taken together, these elements confirm the potential of neem oil as a feedstock for biodiesel production, a potential already exploited experimentally by several research teams [12, 34], subject to a deacidification pretreatment that considers the elevated acid value discussed in Section 3.3.2.

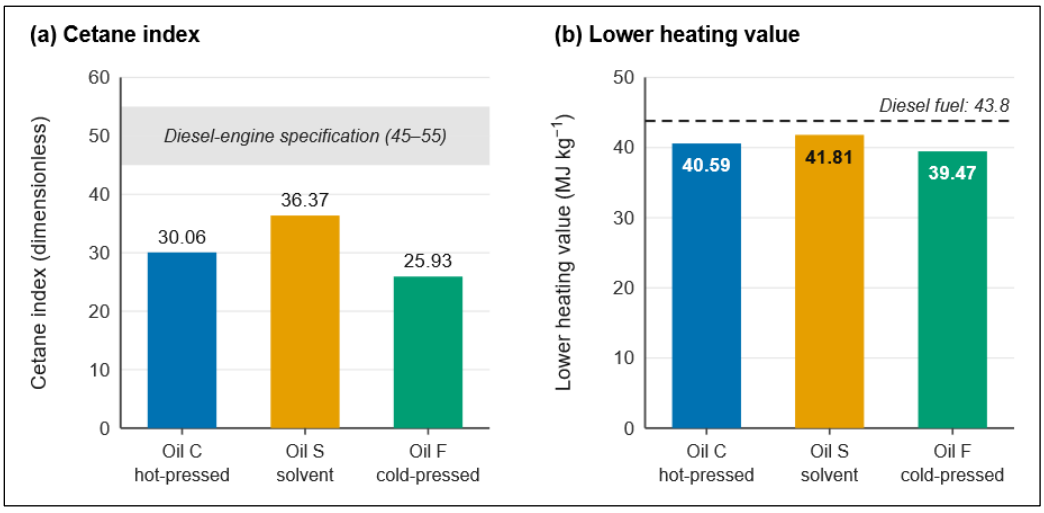


Figure 3: (a) Cetane index and (b) lower heating value of the three neem oils, benchmarked against the diesel-engine cetane specification and diesel fuel heating value, respectively

Considered together, these physicochemical characteristics indicate that the choice of extraction process should be guided by the intended end use: solvent-extracted oil, despite its higher water and residual-solvent content, offers the best compromise between yield and hydrolytic stability (lowest acid value) for energy valorization, whereas cold-pressed oil, despite its low yield under the experimental conditions of this study, offers the highest saponification potential and is therefore preferentially suited to soap-making or cosmetic use.

3.4 Valorization of by-products into agromaterials

3.4.1 Particle size of shell and press-cake powders

Particle-size analysis (Figure 4) shows that most of the shell powder is retained between the 1 mm and 400 µm sieves, placing the dominant particle size of this powder between approximately 400 µm and 1 mm.

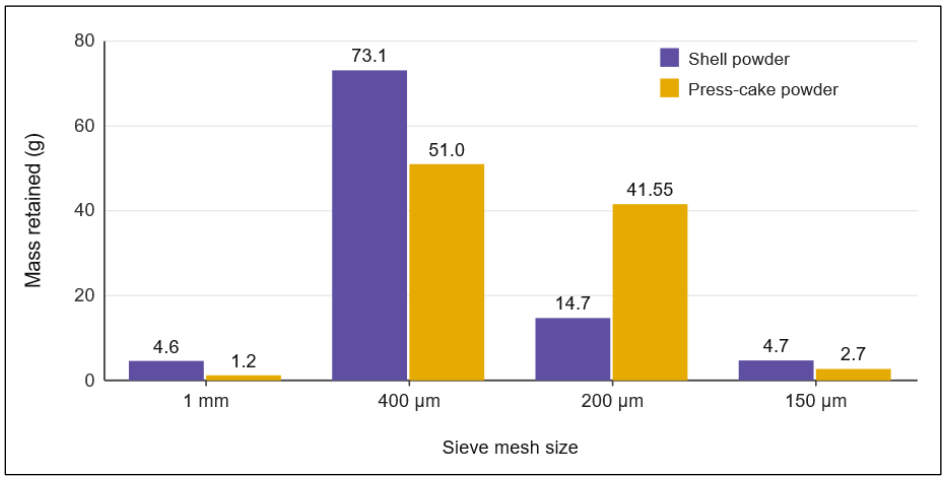


Figure 4: Particle-size distribution of shell and press-cake powders

The press-cake powder shows a finer distribution, with a substantial fraction (41.55 g) retained between the 400 and 200 µm sieves, placing its dominant particle size between 200 and 400 µm. This difference in fineness between the two co-products, consistent with their respective origins (rigid lignocellulosic matrix for the shells, partially delipidated and more friable residue for the press cake), directly influences the compactness and cohesion of the agromaterials obtained by thermocompression, with the finer press-cake particles acting as a binder between the coarser shell particles, a role consistent with the thermoplastic behaviour of the protein fraction reported for other oilseed press cakes during thermopressing [38].

3.4.2 Morphological characteristics of the plates

The eleven formulations produced yielded square plates with an average weight of 25.72 g, an average thickness of 4 mm, and an average side length of 6.9 cm. These dimensions, obtained at laboratory scale with a mould of limited format, remain consistent with those reported in preliminary work on agro-composites for the building sector [39], and are directly transposable to a pilot scale by adapting the mould and the press.

3.4.3 Water-absorption behaviour

The water-absorption test (Figure 5) was performed on three representative formulations: Plate 1 (0% shells, 100% press cake), Plate 2 (50% shells, 50% press cake), and Plate 3 (100% shells, 0% press cake).

The mass gain after 5 minutes of immersion increases markedly with the proportion of shells in the formulation: 9.09% for Plate 1, 14.15% for Plate 2, and 39.61% for Plate 3. Plate 3, composed exclusively of shells, shows the most atypical behaviour: its mass gain reaches a maximum of 50.83% at t = 3 minutes, before decreasing to 45.56% and then to 39.61% in the following minutes, this decline reflecting a progressive disintegration of the material observed visually from the third minute of immersion onward. This behaviour suggests that shells alone, in the absence of the binder provided by the press cake, do not develop sufficient cohesion to maintain the structural integrity of the material in a water-saturated environment, which underscores the functional role of the press cake as a binding agent within the formulation [38], consistent with the material's original design.

Compared with the water-absorption rates reported for hemp shiv aggregates after 5 minutes of immersion, namely 260% [40], 280% [18], 225% [41], and 225% [42], all values synthesized by Chabannes [27], the shell- and press-cake-based agromaterials developed in this study show markedly lower water absorption, including for the most hydrophilic formulation (50.83% at maximum against 225 to 280%). This gap, greater than a factor of four, could be explained by the residual presence of fat in the press cake and by the denser, less porous structure of the neem shell and press-cake particles compared with the woody hemp fragments (shiv), whose alveolar structure favours greater capillary absorption. A more limited water-absorption behaviour is, as a first approximation, a favourable indicator for use as a building cladding material, insofar as it suggests better dimensional stability upon contact with humidity; it must nonetheless be interpreted with caution, since it has not yet been related to mechanical properties (flexural strength,

compressive strength) nor to longer-term behaviour (humidification-drying cycles, resistance to mould), which remain to be documented. This behaviour nonetheless aligns with trends reported for other agromaterials based on tropical oleaginous or fibrous residues, such as particleboard obtained from coconut residues [19], for which the incorporation of particles rich in lipophilic compounds also improves dimensional stability and reduces water absorption relative to control panels.

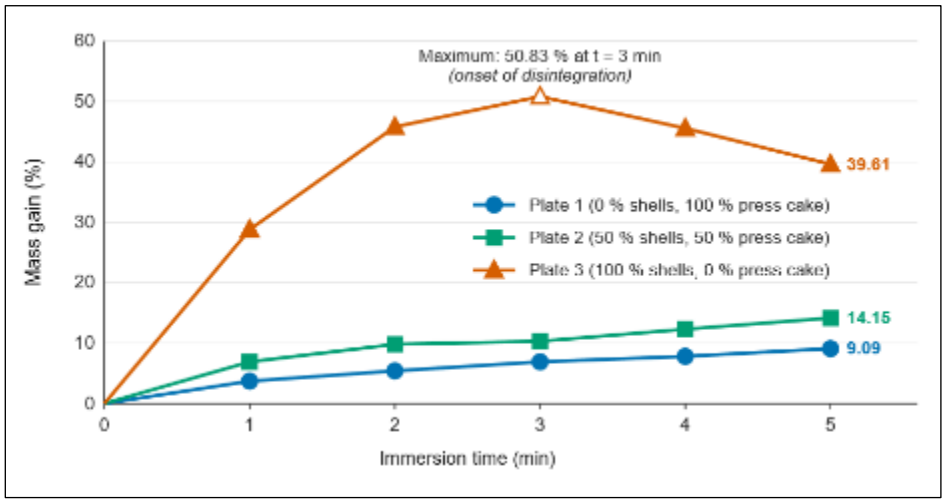


Figure 5: Water-absorption kinetics of the three agromaterial plates (0, 50, and 100% shells) as a function of immersion time. The curve highlights the increasing relationship between shell proportion and water absorption, as well as the downturn of Plate 3 after three minutes, associated with the onset of its disintegration

3.5 Comparative synthesis with literature

Table 3 brings together, for the main parameters discussed above, a synthetic comparison between the results obtained in this study and the values reported in earlier work on neem or on comparable raw materials. Figure 6 presents this benchmark graphically for five key parameters.

Table 3: Comparative synthesis of the main results with literature

Parameter	Present study	Reference(s) and compared value(s)
Hot-press extraction yield (%)	36.15 ± 4.36	*Moringa oleifera*: 41.33% [29]
Solvent extraction yield, ground kernels (%)	65.01 ± 2.4	Neem: 44% [9]; 49% [10]; *Peganum harmala*: 44.21% [25]
Cold-press extraction yield (%)	6.54 ± 3.07	Neem: 40.1 ± 1.1% [11]
Acid value (mg KOH g^{-1})	27.6 to 41.76	10.2 [10]; 8.97–9.16 [12]
Saponification value (mg KOH g^{-1})	151.45 to 212.46	200.45, neem soap [32]; comparably high values [7, 9, 10]
Lower heating value (MJ kg^{-1})	39.47 to 41.81	41.00–41.01 [12]; comparable values [10]; diesel: 43.8 [35]
Maximum water absorption during the 5-min test, most hydrophilic material (%)	50.83 (t = 3 min)	Hemp shiv aggregates at 5 min: 225 to 280% [18, 40-42]

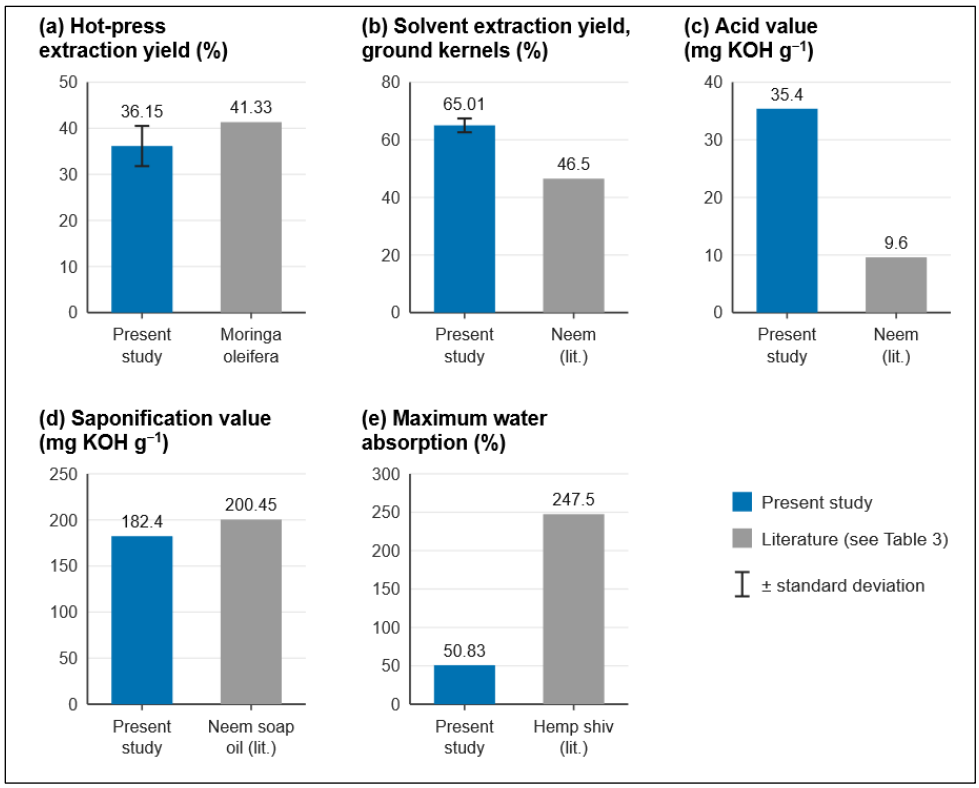


Figure 6: Benchmark of key present-study parameters against previously published values. Panels: (a) hot-press yield, (b) solvent-extraction yield on ground kernels, (c) acid value, (d) saponification value, (e) maximum water absorption. Present-study values in (c) and (d) are means of the three oils; error bars (present study) represent the standard deviation. See Table 3 for the full comparative synthesis and source attribution

3.6 Original scientific contributions

In light of the literature reviewed in this discussion, several elements of this work appear to carry scientific novelty. First, this study proposes, to our knowledge, one of the few direct and simultaneous comparisons of the three major extraction processes (hot pressing, cold pressing, solvent extraction) applied to a single batch of Ivorian neem seeds, allowing an integrated evaluation of the yield-quality trade-off specific to each process, rather than the isolated characterization of a single extraction mode, as prevails in most of the cited works. Second, it documents for the first time, to our knowledge, the joint valorization of the two solid co-products of extraction, shells and press cake, in the form of thermocompressed agromaterials intended for the building sector, whereas the existing literature on neem co-products very largely favours the agronomic (soil amendment, biopesticide) or energy (solid fuel, pyrolysis) uses of these residues [14, 15] taken separately. Third, the markedly more favourable water-absorption behaviour of the neem shell- and press-cake-based agromaterials, compared with the hemp shiv aggregates classically studied in the literature on plant-based concretes, constitutes a novel observation that deserves to be further investigated and confirmed by a complete mechanical characterization. Finally, the identification of an unexplained anomaly in the iodine value, together with a critical discussion of its probable methodological origin, constitutes in itself a useful contribution to the community working on the physicochemical characterization of neem oils, by flagging a point of analytical vigilance for future work.

3.7 Study limitations

This work presents several limitations that should be explicitly noted. On the analytical side, the iodine value obtained appears, as discussed in Section 3.3.4, markedly at odds with the literature and requires methodological revisiting before any quantitative use of this parameter. The lower heating value and the cetane index were estimated through indirect empirical relations rather than measured directly by bomb calorimetry or standardized engine testing, which introduces additional uncertainty in these two quantities. On the statistical side, although most measurements were repeated in triplicate (the saponification value of oil F and the peroxide value of oil C were single determinations) and expressed as mean ± standard deviation, no inferential statistical test was applied to establish the significance of the differences observed between extraction processes or between agromaterial formulations, which limits the comparisons to a descriptive level. Regarding the agromaterials, only the water-absorption property has been characterized at this stage; no mechanical property (compressive, flexural, or tensile strength), thermal property (conductivity), or acoustic property has been measured, which does not yet allow conclusions on the real fitness of these

materials for structural or semi-structural use in construction. Finally, sampling remains limited to a single collection site and a single harvest campaign, which does not allow the seasonal or geographic variability of the properties studied to be assessed, even though such variability is documented for other neem provenances.

4 CONCLUSION

This study characterized an integrated valorization chain for neem seeds (*Azadirachta indica*) collected in Côte d'Ivoire, combining, on the one hand, the extraction and characterization of the oil, and, on the other hand, the valorization of the solid co-products as agromaterials. The three extraction processes compared, hot pressing, cold pressing, and solvent extraction, proved to be strongly contrasted in terms of yield, from 6.54 to 65.01%, and of the physicochemical quality of the resulting oil. The neem oil studied shows promising potential for two major industrial applications: soap making, owing to a saponification value reaching 212.46 mg KOH g^{-1} for cold extraction, and biodiesel production, owing to a lower heating value (39.47 to 41.81 MJ kg^{-1}) close to that of diesel fuel, subject to a deacidification pretreatment imposed by the elevated acid values observed. Valorization of the shells and press cake as thermocompressed agromaterials furthermore showed markedly more favourable water-absorption behaviour than reference values from the literature on comparable plant-based aggregates, opening a promising avenue for the development of low-cost, bio-based cladding materials.

Beyond these results, this study makes an original contribution by simultaneously documenting the entire valorization chain of a raw material still underexploited in Côte d'Ivoire, and by critically identifying the methodological limitations (notably regarding the iodine value) and experimental limitations (absence of mechanical characterization of the agromaterials, absence of inferential statistical testing) that will need to be addressed to consolidate these preliminary results. Several research perspectives naturally emerge from this work: repeating and cross-validating the determination of the iodine value using an alternative reference method; conducting actual transesterification trials to convert neem oil into biodiesel, with standardized characterization of the resulting product (viscosity, density, flash point, measured cetane index); optimizing the cold-pressing process with dedicated equipment to improve its yield; carrying out a complete mechanical, thermal, and acoustic characterization of the shell- and press-cake-based agromaterials, ideally coupled with a design of experiments enabling statistical optimization of the formulation; and exploring complementary valorization routes for the press cake, such as organic amendment or biopesticide use, already demonstrated on maize [13], which could be combined with the agromaterial value chain in a multi-product village-scale biorefinery approach. Together, these perspectives would help consolidate the technical and economic feasibility of an Ivorian value chain for the integrated valorization of neem seeds, within a circular-economy approach applied to tropical agroresources.

STATEMENTS ON COMPLIANCE WITH ETHICAL STANDARDS

Disclosure of conflict of interest

The authors declare that they have no competing interests in relation to this work.

Author contributions

K.E.K.A.: conceptualization, investigation, formal analysis, writing of the original draft. O.L.Y.: conceptualization, methodology, validation, writing (review and editing), supervision, project administration. T.S.: investigation (physicochemical analyses), resources, validation. T.N.T.: investigation, data curation. S.B.: investigation, visualization. All authors read and approved the final manuscript.

Data availability

The data supporting the findings of this study are available from the corresponding author upon reasonable request.

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