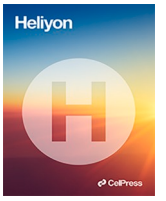
Heliyon 10 (2024) e30777

Contents lists available at ScienceDirect

Heliyon

journal homepage: www.cell.com/heliyon



Review article

Effects of extraction parameters on the yield of oils from non-edible seeds of *Bauhinia variegata* and *Pachira glabra*

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ARTICLE INFO

Keywords:
Bauhinia variegata
Pachira glabra
Modeling
Optimization
Response surface methodology

ABSTRACT

The influence of extraction temperature, seed age, and extraction time and their interactions on the *Bauhinia variegata* seed oil (BVSO) and *Pachira glabra* seed oil (PGSO) yield was studied using the response surface methodology (RSM). The BVSO and PGSO obtained were characterized to determine their suitability for use as biofuel. Numerically predicted optimum values for the extraction process using the RSM model proved to be a one-week-old seeds, a 10 h extraction time and a 60 °C temperature with a 47.05 % PGSO yield, and a one-week-old seed, a 6.5 h extraction period and 60 °C temperature, with a 23.1 % BVSO yield. Performance evaluation of the models by coefficient of determination (R^2), Adjusted R^2, and absolute average deviation (AAD) showed that the RSM model (R^2 = 0.99, Adjusted R^2 = 0.99, AAD = 0.07 % for BVSO yield, and R^2 = 0.99, Adjusted R^2 = 0.99, AAD = 0.01 % for PGSO yield) was satisfactory, reliable, and flexible. The physicochemical properties of BVSO and PGSO, i.e. acidity index (4.63 mg KOH/g and 27.21 mg KOH/g) and kinematic viscosity (3.24 mm^2/s and 12.45 mm^2/s), reveal the need for post-treatment of oils for use as additives to conventional fuels. Other physicochemical properties obtained, such as oxidative stability, higher heating value, cetane number, flash point, iodine value, and saponification value, demonstrate that these oils are excellent potential sources for biodiesel production.

1. Introduction

The growth in energy needs is mainly based on the exploitation of fossil fuels, dominating at 80 % [1]. Although fossil fuels play a crucial role in industrial, transportation, and agricultural development, they, as a major source of energy, are non-renewable sources and could therefore be on the verge of depletion [2,3]. And this is three decades from now, as British Petroleum announced in 2016, in its World Energy Statistical Review [4]. Moreover, the use of fossil fuels contributes to various concerns, including the progression of global warming, air pollution, environmental problems [5], and rising crude oil prices [6]. Therefore, it is necessary to propose more suitable alternative fuels [5], renewable, non-polluting, and sustainable [3]. These challenges have led to the valorization of vegetable oils for biodiesel production [7], thus making it a promising future as an alternative energy source [8,9]. Currently, edible oils

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https://doi.org/10.1016/j.heliyon.2024.e30777
Received 24 July 2023; Received in revised form 17 April 2024; Accepted 5 May 2024
Available online 7 May 2024

Nomenclature

AAD	Absolute average deviation
ANOVA	Analysis of variance
BVSO	*Bauhinia variegata* seed oil
BV	*Bauhinia variegata*
PG	*Pachira glabra*
PGSO	*Pachira glabra* seed oil
R^2	Coefficient of determination
RSM	Response surface methodology

dominate 95 % of the global biodiesel production [9], a matter of great debate. To address this concern, efforts are directed toward identifying and valorizing non-edible vegetable oils.

Cameroon, because of its geographical location, has a wide variety of oilseeds, both indigenous and exotic, cultivated or grown in the wild [10]. These non-edible seeds can be used as raw materials to produce vegetable oils. Among the poorly studied and neglected oleaginous seeds in Cameroon, the seeds of *Bauhinia variegata* L. (BV) and *Pachira glabra Pasq.* (PG) are used as inputs in this study.

Bauhinia variegata is a species of Leguminosae (Caesalpinioideae), commonly known as mountain ebony, orchid tree, and kachnar in Hindi [11–13]. This species has already been identified in the western, southwestern, and northwestern regions of Cameroon and has potential for adaptation in other parts of the country. Its ability to grow on all soil types and its low water requirement make it relatively easy to maintain [14]. BV is used for ornamental purposes [15] and as fuel with a calorific value ranging from 20.08 to 20.54 MJ/kg [16–18]. The chemical compounds of the seed oil obtained after extraction contain oleic acid, linoleic acid, palmitic acid, and stearic acid [19].

Pachira glabra, also known as *Bombacopsis glabra*, belongs to the family Malvaceae, in the Bombacaceae subfamily [20,21]. It is also known as the French peanut but often referred to as Malabar chestnut, money tree, or lucky tree [21]. PG is well adapted to various soil types, grows both in full sun and in partial shade [22], and it is resistant to both drought and flooding [21]. It is used as a shade and ornamental tree in public spaces, in the hotel landscapes, gardens, and private courtyards for its attractive flowers and seeds [20–23]. This fuel has a calorific value of 22.57 MJ/kg [24]. The oil obtained after extraction generally contains chemical compounds such as palmitic acid, stearic acid, oleic acid, linoleic acid, linolenic acid, and sterculic acid [22,25].

In general, oil extraction from seeds is carried out by various methods [1]. Of these, solvent extraction is one of the most used techniques. In addition to the choice of oil extraction method, several parameters, namely extraction temperature, the nature of the solvent, seed age, and extraction time, influence both the yield and the quality of the produced oils [26].

From the literature, Govindhan et al. [3] evaluated the influence of extraction time and the nature of the solvent on the BVSO yield by solvent extraction method. In another study, Yatish et al. [17] extracted BVSO using *n*-hexane for an extraction time of 4 hours. Concerning PGSO, Kibazohi and Sangwan [27] as well as Araújo et al. [22] extracted the oil using hexane for extraction times of 7 hours and 24 hours, respectively.

As mentioned earlier, many factors influence the yield and quality of oils. Therefore, optimization of the extraction process is necessary to maximize the yield and quality of oils, save time, and reduce production costs [7,26,28]. According to the literature, the conventional optimization method, also known as one-factor-at-a-time (OFAT) [7,26], and the response Surface Methodology (RSM) [29,30] are commonly used. The OFAT allows for the variation of a factor on the response variable while maintaining the other factors in process constant [31], and repeating this for all the factors considered [32]. For processes with multiple parameters to evaluate, this method is both time-consuming and exhausting. In addition, this approach can lead to errors, not considering the interactions between the parameters, thereby determining the optimal conditions of the extraction process [31]. Unlike the OFAT method, RSM describes the interactions between the input variables and the output response [26,33]. It aims to determine the optimal operating conditions of a system [34], either to maximize or minimize the response or to a specific value [35]. In addition, it also aims to establish an adequate approximation of the relationship between the input variables and the output variable through a polynomial model [35]. Thus, RSM offers several advantages including the reduction in the number of experimental trials and the ability to detail interactions between different variables with expected accuracy [36]. However, RSM is limited, as it adjusts data to a second-order polynomial model, while all systems with curvature may not be compatible with this type of model [31].

To the knowledge of the authors, no study has so far been published on the optimization of the solvent extraction process for oil from the seeds of *BV* and *PG* using the response surface methodology (RSM) optimization tool. This study aims to extract oil from the eeds of *BV* and *PG* by the solvent method, while evaluating the influence of the extraction parameters by the OFAT approach. These parameters include extraction temperature, seed age, and extraction time. The RSM then served as a tool to evaluate the interactions between the parameters studied and to obtain the optimal parameters inducing maximum oil yield. The different optimal parameters were subsequently validated by the coefficient of determination (R^2), Adjusted R^2, and absolute average deviation (AAD). The physicochemical properties of the obtained oils were used for characterization to assess their potential as biofuels for industrial applications.

2. Materials and methods

2.1. Raw materials and sampling

One kilogram (1 kg) of *PG* seeds were collected in the district of Djemgheu-Baham (located at 5.33602 N and 10.39365 E) and that of BV seeds (1 kg) within the campus of the University of Buea of Cameroon (located at 4.14910 N and 9.28840 E). The seeds were dried at 105 °C for 12 h, to reduce their water content, until a constant weight was obtained [26]. After drying, the seeds were ground into fine particles using an electric moulinex (SILVER CREST, Model SC-1589). The powders obtained were carefully stored in dry paper bags to avoid any moisture absorption.

2.2. Oil extraction process from Bauhinia variegata and Pachira glabra seeds

The methodology described by Suganya and Renganathan [37] and Khan et al. [38] was adopted as part of the extraction process. A Soxhlet apparatus equipped with a 250 ml round-bottom, containing 99 % pure normal hexane as the solvent (selected based on the work of [3,25]), was used for extraction (Fig. 1A). The top of the Soxhlet is equipped with a bulb condenser, operating with cooling water. The solvent contained in the balloon was heated to a boil, then evaporated, condensed, and collected in the extraction chamber. A sample mass (M_1) of ~35 g of seed powder, placed in the extraction chamber, is covered with the condensed solvent.

When the solvent level reaches its maximum height in the chamber, the enriched solvent with extracted oils falls back into the balloon until the room is empty. This process was repeated several times until the oil was completely isolated. Extraction was carried out at different time intervals and at specific temperatures, as detailed in section 2.3. The volume of the solvent in the balloon was fixed to 200 ml. Once the solvent was boiled, the oil extraction process began and continued until a clear liquid appeared in the extraction chamber. At the end of the extraction time, the filter paper used to pack the sample was removed. Subsequently, a final extraction cycle was carried out to ensure a complete distillation of the oil-solvent mixture. The purpose of this cycle was to bring the solvent mixed with oil back into the extraction chamber, as illustrated in Fig. 1B. The balloon containing the oil was placed in an oven at 100 °C for 12 h to remove solvent residues. After cooling in a desiccator, the balloon was weighed [3] and the extraction yield (%) was calculated based on Equation (1).

$$\text{Oil yield } (\%) = \frac{M_2}{M_1} \times 100 \tag{1}$$

Where: M_1 is the mass of the sample (g), M_2 is the mass of oil produced (g).

2.3. Experimental design of extraction

In this study, three variables were examined: seed age (A), extraction temperature (B), and extraction time (C), with oil yield as the response variable. The influence of each factor on BVSO and PGSO yields was evaluated using experimental trials. To this end, an experimental device based on a fully randomized three-factor experimental design was implemented.

According to the studies [3,17,22,27], extreme extraction time and temperature values were selected. For seed age, the selected

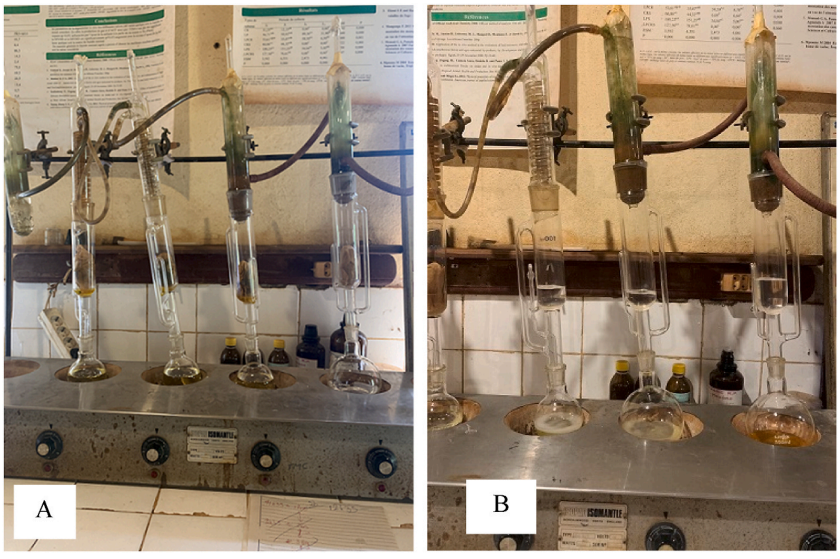

Fig. 1. Extraction process (A) and oil solvent distillation process (B).

durations ranged from one week after harvest to a maximum storage time of 52 weeks.

The experimental trials of PG seeds included a total of 24 trials. These tests assessed the influence of extraction time (5, 6, 7, and 10 h), temperature (60 and 75 °C), and seed age (1, 26 and 52 weeks) on oil yield. In the case of BV seeds, 18 trials were carried out and these trials also allowed us to assess the influence of extraction time (4,5, 5.5, and 6.5 h), temperature (60 and 75 °C) and seed age (1, 26, and 52 weeks) on oil yield. Each of these trials was repeated three times to ensure the reproducibility of the results.

2.4. Modeling, statistical analysis of RSM, and optimization

The experimental design for the modeling and optimization of the extraction process were made using the Design Expert software (version 13.0.5 Stat-Ease Inc. Minneapolis, MN, USA) [17]. The RSM, based on the Box-Behnken Design (BBD), evaluated the response variable and determined the optimal conditions to induce high yield in PGSO and BVSO. The BBD is distinguished by its efficient design in terms of number of runs and its ability to produce a rotating plan [39] combining 2 k factorials and incomplete block designs [35]. The number of runs required (N) were defined by N = 2 k (k-1) + Co, (where k is the number of factors and Co is the number of central points) [35]. As mentioned in section 2.3, the independent variables were seed age (A), extraction temperature (B), and extraction time (C). Each factor was coded at three levels: 1, 0, and +1, corresponding to the minimum, mean, and maximum values, respectively (see Table 1 et Table 2). These values were selected by the experimental trials presented in section 2.3. The total number of experiments was 19, including 7 central points and 12 factorial points. The dependent variable was oil yield (Y). The experimental data obtained were adjusted to a quadratic regression model using a second-order polynomial equation described in Equation (2).

$$Y = \beta_1 A + \beta_2 B + \beta_3 C + \beta_{12} AB + \beta_{13} AC + \beta_{23} BC + \beta_{11} A^2 + \beta_{22} B^2 + \beta_{33} C^2 \quad (2)$$

Where: Y is the predicted response variable, β1 −β3 are the linear coefficients, β12, β13, and β23 are the interaction coefficients, β11, β22, and β33 are the quadratic coefficients, and A, B, and C are the coded independent variables representing seed age (weeks), temperature (°C), and extraction time (h) respectively. AB represents the interaction between seed age and temperature, AC is the interaction between seed age and time, and BC is the interaction between temperature and time.

A multiple regression analysis was then carried out to evaluate the Analysis of Variance (ANOVA) of the experimental data. This analysis helps verify the statistical significance of the models. The optimal conditions for the studied variables were identified by solving the regression equation. The adjusted polynomial equation was expressed in graphical form to assess the influence and interactions between independent variables and the dependent variable. The coefficient of determination (R^2) and adjusted coefficient of determination (adjusted R^2) were calculated to measure the performance of the regression equation.

2.5. Verification of estimated data

The evaluation of the estimation capability of the developed RSM models was determined. For this purpose, the model output error between experimental and predicted values was estimated. The precision of the models was assessed by determining and appreciating the values of R^2, Adjusted R^2, adequacy precision, and absolute average deviation (AAD). R^2 shows the relationship between predicted and experimental values, while adjusted R^2 describes the degree of fit of the mathematical model. Adequate accuracy compares the range of predicted values at design points to the average prediction error [40]. The AAD defines the level of accuracy of a model prediction.

AAD is defined by Equation (3):

$$AAD\ (\%) = \left(\frac{1}{n} \sum_{i=1}^{n} \left(\frac{y_{pred} - y_{exp}}{y_{exp}} \right) \right) \times 100 \quad (3)$$

Where: ypred and yexp are the predicted and experimental responses, respectively, and n is the number of experimental data points. A model must have an adequate accuracy greater than 4 [40], the lowest possible AAD [7], and an R^2 greater than 70 % to be considered better [26].

2.6. Determination of physicochemical properties of bio-oils

The following physicochemical characteristics of oils obtained after extraction were determined.

Table 1
Factors and levels used for the RSM of BV.

Factors	Units	Symbols	Coded Factor Levels		
			−1	0	1
Seed age	Weeks	A	1	26	52
Temperature	°C	B	60	60	75
Extraction time	hr	C	4.5	5.5	6.5

Table 2
Factors and levels used for RSM of *PG*.

Factors	Units	Symbols	Coded Factor Levels		
			−1	0	1
Seed age	Weeks	A	1	26	52
Temperature	°C	B	60	60	75
Extraction time	hr	C	5	7	10

2.6.1. Acid value (A_v)

The acid value was determined by applying the ASTMD 664 method. A quantity of 0.5 ± 0.01 g of oil was added to 100 ml of 95 % ethanol and 2 to 3 drops of phenolphthalein indicator. It was then titrated with 0.1 N KOH until a pale pink color was obtained. The acid value (A_V) was calculated using Equation (4) [41].

$$A_v = \frac{V}{m} \times 56.1 \times N \tag{4}$$

Where: A_v is the acid value in mg/g, V (ml) is the volume of the KOH solution for the sample, N is the concentration of ethanolic KOH used, m (g) is the mass of the sample taken.

2.6.2. Free fatty acid content (%)

The free fatty acid content was calculated from the acid value using Equation (5) [41,42]:

$$FFA\ (\%) = \frac{A_v}{2} \tag{5}$$

Where: A_v is the acid value (%), FFA is the free fatty acid content (%)

2.6.3. Acidity (%)

The acidity in terms of oleic acid percentage was calculated using Equation (6) [43]:

$$Oleic\ Acid\ (\%) = \frac{M \times V \times T}{10m} \tag{6}$$

Where: V is the volume in ml of standard KOH used (ml), T is the normality of the standard KOH (0.1), m is the weight in grams of the sample, M is the molecular weight of the dominant fatty acid (expressed as oleic acid).

2.6.4. Iodine value (I_v)

The method specified by ISO 3961 (1989) was used. A mass of 0.2 ± 0.01 g of oil was introduced into flasks containing 7.5 ml of carbon tetrachloride (CCl4), 3.5 ml of Wijs' reagent, 3 ml of mercuric acetate, and 4.5 ml of potassium iodide. Then, 5 drops of starch paste were added to each flask, and each flask was titrated with 0.1 N sodium thiosulfate ($Na_2S_2O_3$, $5H_2O$) solution until the volume V of sodium thiosulfate was reached each time [41]. The iodine value was then calculated using Equation (7):

$$I_v = \frac{V_0 - V}{m} \times 12.69 \times T \tag{7}$$

Where: V_0 (ml) is the volume of the thiosulfate solution for the blank, V (ml) is the volume of the thiosulfate solution for the sample, T is the titer of the sodium thiosulfate solution used, m (g) is the sample taken.

2.6.5. Saponification value (S_v)

The specified method as indicated in ISO 3657 (1988) was used. A quantity of 1.0 g of oil was added to 25 ml of 0.5 N KOH, heated under reflux for 1 h. After the sample cooled, the solution was added to 0.5 ml of phenolphthalein indicator and titrated with 0.5 N HCl solution until the pink color disappeared. The saponification value is calculated using Equation (8) [41,42,33,44]:

$$Sv = \frac{56.1 \times N \times (V_b - V_a)}{W} \tag{8}$$

Where: Sv is the saponification value, W is the weight of the oil sample taken in grams, N is the normality of the HCl solution, Va is the volume of the HCl solution used in the test in ml, Vb is the volume of the HCl solution used in the blank in ml.

2.6.6. Peroxide value (P_v)

A quantity of 2 g of oil was added by mixing 10 ml of chloroform, 15 ml of glacial acetic acid, and 25 ml of 95 % ethanol. Then, 1 ml of KI was added to the solution and kept in the dark for 30 min. The next step involved adding 3 to 4 drops of starch paste to the mixture. The excess iodine in the solution was titrated with 0.02 N sodium thiosulfate ($Na_2S_2O_3$) using a starch solution as an indicator. The peroxide value is calculated using Equation (9) [42]:

$$Pv = \frac{(V - V_0) \times N}{m} \times 1000 \quad (9)$$

Where: P_v is the peroxide value in milliequivalents per kilogram (meq/kg), V_0 (ml) is the volume of $Na_2S_2O_3$ (0.01 N) solution needed to titrate the blank, V (ml) is the volume of $Na_2S_2O_3$ (0.01 N) solution needed to titrate the sample, m (g) is the sample weight in grams.

2.6.7. Density at 20 °C

The density at 20 °C was measured using the standard ASTM D4052-91 method with a standard pycnometer as described by Ref. [18]. It was then determined using Equation (10) [41]:

$$D = \frac{W_1 - W_0}{W_2 - W_0} \quad (10)$$

Where: W_1 is the mass of the pycnometer filled with oil, W_2 is the mass of the pycnometer filled with water, W_0 is the empty pycnometer mass.

2.6.8. Viscosity

Empirical viscometers were used for determining dynamic viscosity. This involves measuring the terminal velocity of a sphere with radius r falling through a fluid with density ρ. The dynamic viscosity was determined according to Pierron [45] and is given by Equation (11):

$$\eta = \frac{g \times r^2 (\rho_{ball} - \rho_{fluid}) \times t}{18 \times d} \quad (11)$$

Where: η is dynamic viscosity in Pa.s (Pascal-seconds), ρball is the density of the sphere, ρfluid is the density of the fluid (kg/m^3), d is the distance traveled by the sphere in meters (m), t is the time taken by the sphere in seconds (s), r is the radius of the sphere in meters (m), g is the acceleration due to gravity (gravitational constant ≈ 9.81 m/s^2).

The kinematic viscosity was deduced from this using Equation (12) [46]:

$$\upsilon = \frac{\eta}{\rho_{fluid}} \quad (12)$$

Where: ν is kinematic viscosity in m^2/s, η is dynamic viscosity in Pa.s.

2.6.9. Cetane number

The cetane number was determined using the ASTM D613 method, expressed by Equation (13), which is a function of the Saponification Value (Sv) and the Iodine Value (Iv) of the bio-oil [41]:

$$CN = 46.3 + \left(\frac{5458}{Sv}\right) - 0.225(I_v) \quad (13)$$

Where: CN is the cetane number, Sv is the Saponification Index (mg of KOH per gram of oil), Iv is the Iodine Value (g of iodine/100 g of fat).

2.6.10. Calorific value

The calorific value was determined using the oxygen bomb calorimeter IKA C20, according to ASTM D240 standard as described by Ref. [18].

2.6.11. Flash point

The flash point (FP) was determined following the ASTM D93 method using Equation (14) with a regression coefficient of 0.964 [47]:

$$FP = 12.36 \times \eta + 176.3 \quad (14)$$

Where: η is the kinematic viscosity at 40 °C in mm^2/s, FP is the flash point in °C.

2.6.12. Oxidation stability test

The oxidation stability of the oils, measured by the induction period (IP) in hours, was determined in accordance with ISO 6886 using a Rancimat apparatus (model 873, Metrohm, Brazil) at 110 °C as described by Ref. [25].

3. Results and discussion

3.1. Modeling of variables by RSM

Table 3 and Table 4 present the Box-Behnken design and the results of the 19 experiments carried out for *PG* and *BV* seeds, respectively. The regression models in terms of actual values describe the extraction process are presented by Equations (15) and (16).

$$Y1 - 3,56A + 0,004B + 5,08C - 0,11AB + 1,05AC - 0.08BC + 0.63A^2 + 40.92B^2 - 2.02C^2 \quad (15)$$

$$Y2 = -2.83A + 0.062B + 1.16C - 0.04AB - 0.11AC - 0.013BC + 1.19A^2 + 18.20B^2 - 0.2C^2 \quad (16)$$

Where Y1 and Y2 are the predicted oil yield (%) for *Pachira glabra* and *Bauhinia variegata*, respectively, A, B, and C are the independent variables coded for seed age (weeks), temperature (°C), and extraction time (hours) respectively. AB is the interaction between the age of seeds and temperature, AC is the interaction between seed age and time, BC is the reaction between temperature and weather, and A^2, B^2, and C^2 are quadratic terms.

The positive and negative signs before the terms indicate synergistic and antagonistic effects, respectively. The results of the significant tests for each regression coefficient and the ANOVA of the quadratic response models are presented in Table 5 and Table 6, for PG and BV, respectively. The quality of the developed models is appreciated by the determination of the R^2 and Adjusted R^2 coefficients. The R^2 values (0.99 and 0.99) of the models show an excellent correlation between the predicted and experimental values of PGSO and BVSO yields. The Adjusted R^2 (0.99 and 0.99) are also an indication of the accuracy of the established model. It is suggested that R^2 should be at least 80 % for a good model fit [48]. Furthermore, the low values of the coefficient of variation (C.V. = 0.99 % and 3.08 %) and absolute average deviation (AAD = 0.01 % and 0.07 %) for PG and BV, respectively, imply that the results obtained by the fitted models are satisfactory, reliable, and flexible. The F-values of model 21782.37 and 2273.19, and the p-value <0.0001 imply that the models are significant. The calculated probability values (*P*-value) less than 0.05 indicate that the terms of the regression model are significant at a confidence level of 95 %, except for terms greater than 0.05. The "lack of fit F values" of 70.07 and 94.79 imply that the lack of fit is not significant relative to the pure error, which is desirable (tableau 5 et 6). In our study, it was found that A, C, AC, A^2, B^2, and C^2 were significant terms in the model, and that the terms B, AB, and BC were non-significant in the case of *PG*. For the *BV*, A, C, A^2, and B^2 were significant terms, and terms B, AB, AC, BC, and C^2 were nonsignificant in the model.

3.2. Effects of Individual variables

3.2.1. Effect of extraction time on oil yield

The extraction time has a significant influence ($p < 0.05$) on PGSO and BVSO yield as shown in Tables 5 and 6 above. Fig. 2A **and** Fig. 2**B** illustrate the effect of extraction time on PGSO and BVSO yield, respectively. From the results obtained, it was observed that the diffusion of oil from the samples was rapid at the beginning of the extraction, before decreasing to a stable state [49]. This could be due to a higher oil concentration on the particle surface during the initial period of the extraction process, which gradually decreases over time inside the particles until it reaches a threshold [18]. The maximum yield of 47.08 % for PGSO was obtained for 10 h extraction time, and 22.90 % for BVSO at 6.5 h. So, an extended extraction time has a significant affect on oil yield. This observation has also been reported by Yusuff et al. [32] for *Leucaena leucocephala* seeds and by Mas'ud et al. [50] for mango kernels.

Table 3
Box-Behnken experiment design, actual and predicted values for PGSO by RSM.

Run	Seed age (weeks)	Temperature (°C)	Time (hr)	Actual oil yield (wt%)	Predicted oil yield (wt%)	Résiduel
1	52	75	7	36.92	36.59	0.33
2	26	75	7	39.87	39.92	−0.05
3	52	75	10	41.52	41.92	−0.40
4	1	60	7	43.83	44.1	−0.27
5	52	60	5	29.56	29.87	−0.31
6	26	60	7	39.65	39.88	−0.23
7	1	75	7	44.01	44.36	−0.35
8	52	60	10	42.09	42.29	−0.20
9	1	60	10	46.95	47.1	−0.15
10	26	60	5	33.61	33.83	−0.22
11	26	75	5	33.9	34	−0.10
12	1	75	5	39.37	39.27	0.10
13	1	60	5	39.37	38.88	0.49
14	52	60	7	37.36	36.78	0.58
15	1	75	10	47.32	47.17	0.15
16	26	60	10	44.43	44.11	0.32
17	26	75	10	44.26	43.97	0.29
18	52	75	5	29.83	29.82	0.01
19	1	75	5	39.3	39.27	0.03

Table 4
Box-Behnken experiment design, actual and predicted values for BVSO by RSM.

Run	Seed age (weeks)	Temperature (°C)	Time (hr)	Actual oil yield (wt%)	Predicted oil yield (wt%)	Résiduel
1	1	75	5.5	22.13	22.33	−0.20
2	26	75	4.5	16.06	16.98	−0.92
3	1	60	4.5	20.98	20.64	0.34
4	52	75	6.5	17.11	17.42	−0.31
5	26	60	4.5	16.06	16.83	−0.77
6	1	60	5.5	22.06	22.12	−0.06
7	52	75	5.5	16.53	16.58	−0.05
8	1	75	4.5	21.42	20.88	0.54
9	52	60	6.5	17.08	17.41	−0.33
10	26	75	6.5	19.9	19.27	0.63
11	52	75	4.5	15.78	15.36	0.42
12	26	60	6.5	19.8	19.17	0.63
13	52	60	4.5	15.68	15.3	0.38
14	1	75	6.5	23.12	23.4	−0.28
15	26	60	5.5	18.46	18.2	0.26
16	1	60	6.5	23.08	23.21	−0.13
17	26	75	5.5	18.49	18.32	0.17
18	52	60	5.5	16.43	16.55	−0.12
19	1	60	6.5	22.99	23.21	−0.22

Table 5
Test of significance for every regression coefficient and ANOVA for *PG*.

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	30335.88	9	3370.65	21782.37	**< 0.0001**
A-Seed age	158.19	1	158.19	1022.27	**< 0.0001**
B-Temperature	0.0003	1	0.0003	0.0022	0.9632
C-Time	326.62	1	326.62	2110.72	**< 0.0001**
AB	0.16	1	0.16	1.00	0.34
AC	9.57	1	9.57	61.85	**< 0.0001**
BC	0.08	1	0.08	0.4959	0.50
A^2	1.62	1	1.62	10.50	**0.009**
B^2	5734.45	1	5734.45	37058.05	**< 0.0001**
C^2	15.15	1	15.15	97.90	**< 0.0001**
Residual	1.55	10	0.16		
Lack of Fit	1.54	9	0.17	70.07	**0.09**
Pure Error	0.0025	1	0.0025		
Total	30337.43	19			
Standard deviation		0.39			
Mean		39.64			
Coef of variation (%)		0.99			

Table 6
Test of significance for every regression coefficient and ANOVA for *BV*.

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	7076.74	9	786.30	2273.19	**< 0.0001**
A-Seed age	101.33	1	101.33	292.94	**< 0.0001**
B-Temperature	0.07	1	0.0724	0.2092	0.66
C-Time	16.91	1	16.91	48.90	**< 0.0001**
AB	0.03	1	0.03	0.07	0.79
AC	0.11	1	0.11	0.32	0.58
BC	0.0021	1	0.0021	0.006	0.94
A^2	5.79	1	5.79	16.74	**0.0022**
B^2	1200.86	1	1200.86	3471.67	**< 0.0001**
C^2	0.16	1	0.16	0.45	0.52
Residual	3.46	10	0.35		
Lack of Fit	3.45	9	0.38	94.79	**0.08**
Pure Error	0.0040	1	0.004		
Total	7080.20	19			
Standard deviation		0.59			
Mean		19.11			
Coef of variation (%)		3.08			

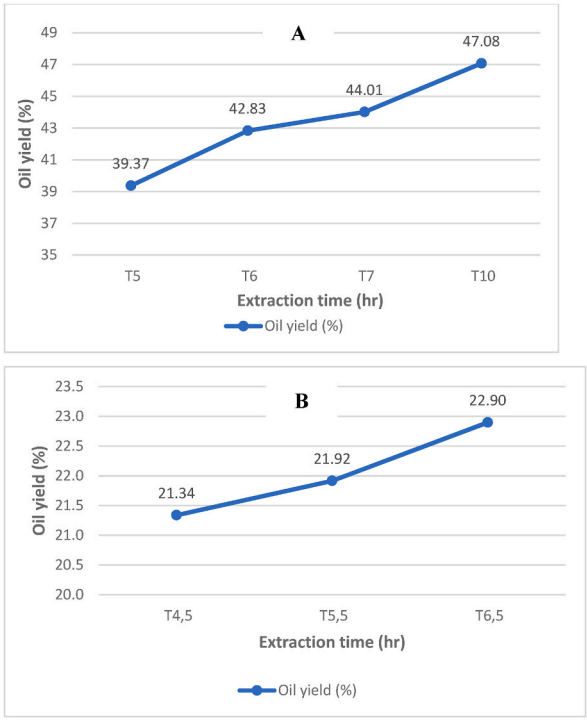

Fig. 2. Effect of extraction time on PGSO (A) and *BVSO* yield (B).

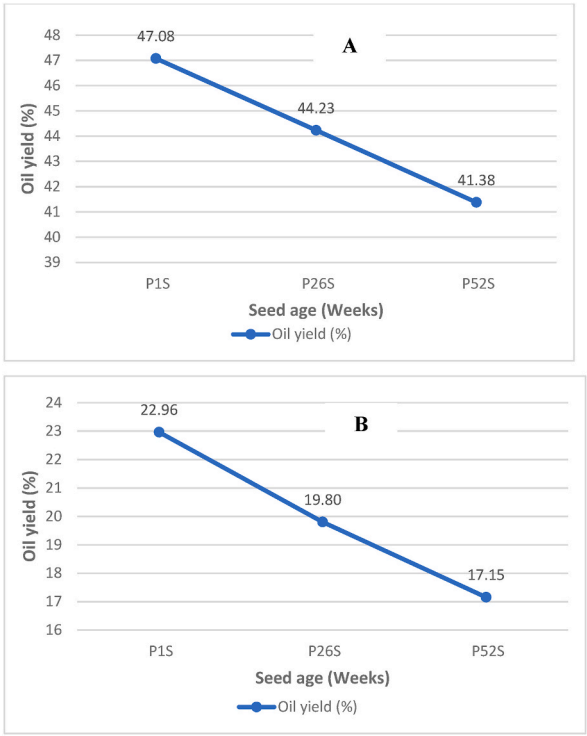

Fig. 3. Effect of seed age on PGSO (A) and *BVSO* yield (B).

3.2.2. Effect of seed age on oil yield

The age of seeds at the time of oil extraction has a significant affect ($p < 0.05$) on PGSO and BVSO yields, as shown in Tables 5 and 6 above. Fig. 3A and B illustrate the effect of seed age on PGSO and BVSO yield, respectively. It can be observed from the results obtained, that the seed storage time over a long duration (26 weeks and more) leads to a decrease in seed oil yield. This decrease may result from a physical, chemical, and biological alterations of the seed shell during storage. This alteration may be caused by environmental factors such as humidity, temperature, and the presence of oxygen and microorganisms [51]. With seeds one week old, the maximum oil yield was 47.08 % for *PG* and 22.96 % for *BV*. This conclusion aligns with the findings of Govindhan et al. [3].

3.2.3. Effect of temperature on oil yield

Regarding the influence of extraction temperature, this parameter has no significant effect ($p < 0.05$) on PGSO and BVSO yields as shown in Tables 5 and 6 above and in Fig. 4A and B below. The results obtained are attributed to the relative insolubility of the oil at the examined temperatures. Therefore, although the diffusion coefficient was improved, no significant variation in the extraction rate was observed in this temperature range. These observations are consistent with studies [18,32], who suggested that a high temperature could not result in a substantially different oil yield from that obtained at a lower temperature. Therefore, based on the results obtained in this study, the optimal extraction temperature is 60 °C, to limit the cost and demand for energy.

3.3. Effects of variable interactions on oil yield

According to the ANOVA analysis (Table 5), three interaction effect terms were identified and only one of these terms was significant. It's AC. Fig. 5A and B shows the 3D plot of the combined effect of seed age (A) and extraction time (C) on HGPG and HGBV yield (Y1 and Y2), while keeping the extraction temperature (B) is constant at 60 °C. It is observed that the maximum oil yield is obtained for an extraction time of 10 h and 6.5 h, respectively, for *PG* and *BV*, when the seeds are one-week-old.

These results highlight an increase in oil yield as the duration of seed storage decreases and the extraction time increases. The PGSO yield (47.32 %) obtained in our study exceeded that by Kibazohi and Sangwan (23.37 %) [27] and by Yoca et al. [24] (39.35 %). This is in line with the values reported by Araújo et al. [22], which ranged from 34 to 50 %. In comparison with the oil yield of other non-edible seeds, this yield is higher than that by Mustapha et al. [52] (40–42 %) for *Arachis hypogaea* L. seeds but remains lower than the yields of *Jatropha curcas* L. seeds (60 %) and *Ricinus communis* L. seeds (50 %) mentioned by Yadessa and Jorge [1]. The BVSO yield obtained in this study (i.e., 23.12 %), exceeds the yield reported by Sharma et al. [14] (18 %) and approaches the values obtained for *Azadirachta indica* seeds (20–30 %), although it remains below the yields of *Pongamia pinnata* seeds (30–40 %) as indicated by Yadessa and Jorge [1].

Similarly, oil yield tends to decrease when the temperature increases, and the extraction time is set at 10 h for *PG* and 6.5 h for *BV*. This observation is explained by the fact that maximum oil yield cannot be promoted by the combination of high temperatures and

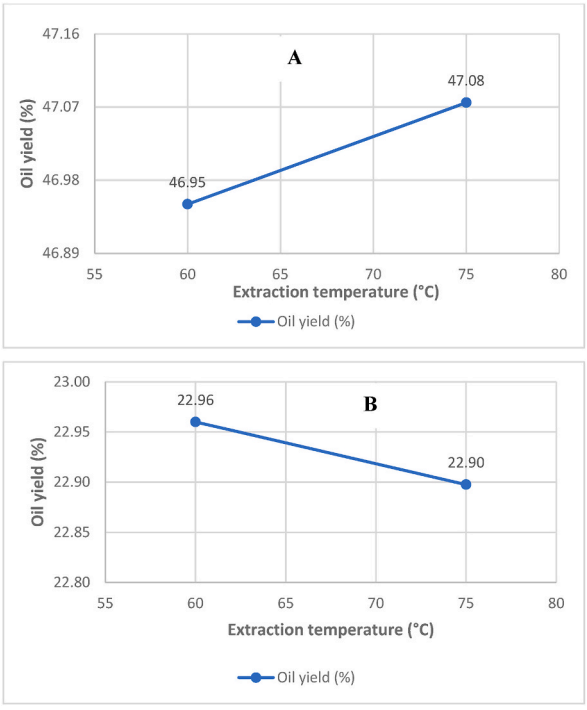

Fig. 4. Effect of extraction temperature on PGSO (A) and BVSO (B).

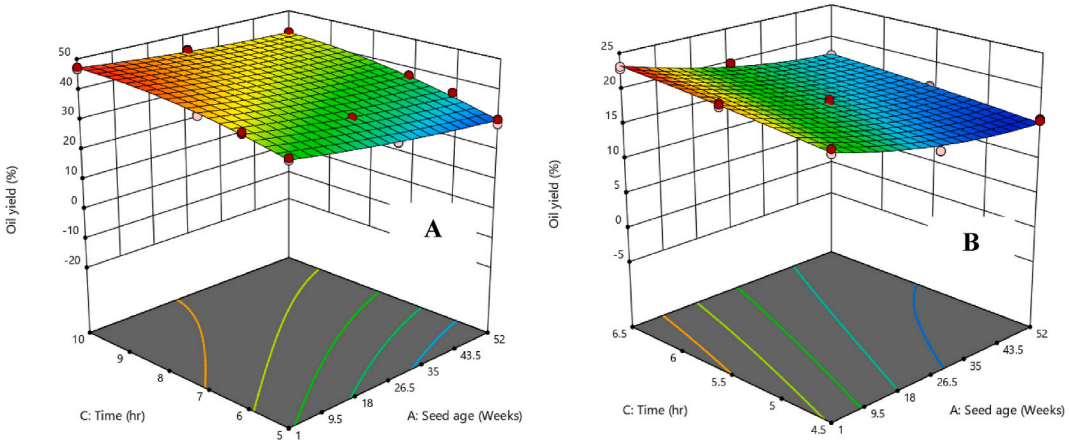

Fig. 5. 3D Diagram of the Combined Effect of Seed Age and Extraction Time on PGSO (A) and *BVSO* yield (B).

extraction time. Because this leads to a decrease in the diffusion coefficient and thus the extraction rate. In other words, low extraction times combined with high extraction temperature improve the oil yield. This is likely because a high level of temperature or duration is sufficient to enhance the diffusion coefficient, thereby increasing the extraction rate [32].

Furthermore, it has been observed that the aging of seeds has a greater influence on oil yield than extraction time. It is also important to note that when seeds reach a storage duration of 52 weeks, an extraction time of 10 h for *PG* and 6.5 h for *BV* is required to maximize oil production. This situation can be explained by the fact that seeds stored for an extended period may have undergone physical and biological alterations, affected their structure and reduced their content of available oil [51]. This yield could be further optimized by improving seed storage conditions, including having a low seed moisture content and storing them in dry plastic bags in a dry, well-maintained location, while would include a study on the impact of particle size on oil yields during the extraction process.

3.4. Optimization of oil yield

Among the many optimization methods available in Design Expert software version 13.0.5.0 (Stat-Ease Inc., Minneapolis, MN, USA), numerical optimization was used to optimize seed oil yield, as it is highly effective for continuous optimization [17]. Using the desirability function, the statistically predicted optimal extraction conditions are one-week-old seeds (Fig. 6A), an extraction time of 10 h (Fig. 6C), and a temperature of 60 °C (Fig. 6B), inducing a PGSO yield of 47.1 % (Fig. 6D), for a desirability of 0.99, as illustrated in Fig. 6. In the case of *BVSO*, the statistically predicted optimal conditions are one-week-old seeds (Fig. 7A), an extraction time of 6.5 h (Fig. 7C), and a temperature of 60 °C (Fig. 7B), inducing an oil yield of 23.21 % (Fig. 7D), with a desirability of 1.0, as shown in Fig. 7. Experiments were conducted to validate the optimal conditions predicted by the model. The experimental results obtained were an oil yield of 47.05 % and 23.1 % for *PG* and *BV*, respectively, with errors of 0.05 % and 0.11 %. The error between the predicted and actual yield was observed to be relatively low, confirming the effectiveness of the RSM models in the description the oil extraction process for both seeds.

3.5. Evaluation of the performance of RSM models

The accuracy of the models obtained by RSM in the description of the oil extraction process was examined by evaluating R^2, AAD, and adeq precision. The results showed that the RSM models presented good predictions, based on high R^2 values (0.99 and 0.99 for PGSO and BVSO, respectively) of ~1.0, very low AAD values (respective of 0.01 % and 0.07 % for PGSO and BVSO, respectively), and adequacy precisions significantly greater than 4 (64.11 and 20.01 for PGSO and BVSO, respectively) (cf. Table 7). In addition, data matching to models was studied and both models showed good matching (Fig. 8A and B). These templates can be used to navigate the design space.

3.6. Physicochemical properties of extracted oils

The quality of BVSO and PGSO was assessed based on their physicochemical properties, as shown in Table 8 and Table 9. The respective densities of PGSO and BVSO were 877.32 and 885.47 kg/m^3, with kinematic viscosities of 12.45 and 3.24 mm^2/s. The kinematic viscosity values by Govindhan [18] and Yatish et al. [17] for BVSO ranged from 26.58 to 32.4 mm^2/s, while those by Araújo and al [22]. for PGSO were 48.63 mm^2/s, which is significantly higher than the values observed in this study. This difference could be explained by the low densities of the oils obtained in this work, compared to those by the same authors for HGBV, which vary from 910 to 960 kg/m^3. Also, the densities obtained in this study are very close to those of several edible oils listed in the literature, such as canola, coconut, corn, palm, colza, soy, and sunflower, with average densities ranging from 873 to 883 kg/m3 [53]. It is important to note that the kinematic viscosity of BVSO complies with the US (ASTM D445) and European standards (EN ISO 3104) [54], which

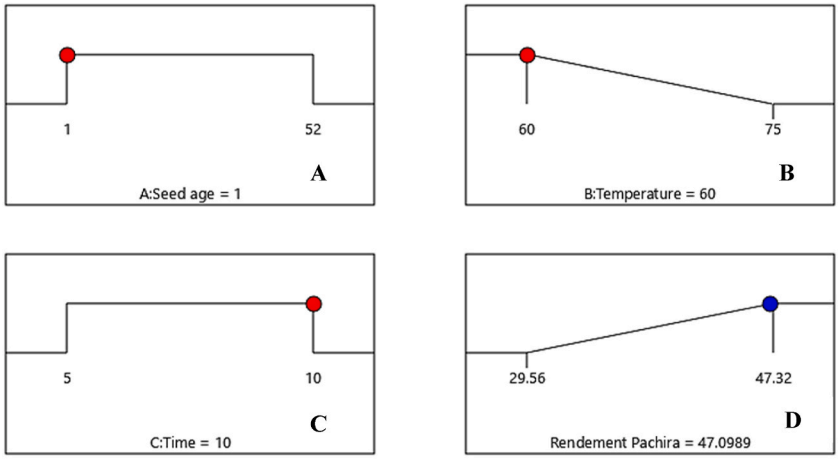

Fig. 6. Numerical optimization parameters for PGSO yield: (A) Seed age, (B) Temperature, (C) Time and (D) PGSO yield.

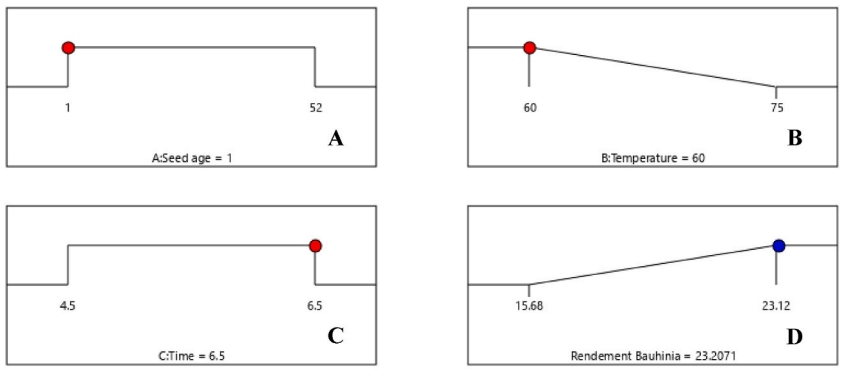

Fig. 7. Numerical optimization parameters for BVSO yield: (A) Seed age, (B) Temperature, (C) Time and (D) BVSO yield.

Table 7
Evaluation of RSM models.

Variables	PGSO	BVSO
R^2	0.99	0.99
Adjusted R^2	0.99	0.99
Adeq précision	64.11	20.01
AAD	0.01 %	0.07 %

define the requirements for the use of oil as a biofuel.

The acid indices of BVSO and PGSO in this study were 4.63 and 27.21 mg KOH/g, with free fatty acids levels of 2.31 and 13.6 %, respectively. These values correspond to the range given in the literature [17,18] for BVSO and are higher than those indicated in Refs. [22,55] for PGSO. In addition, according to ASTM D 445 (kinematic viscosity) and ASTMD 664 (acid index), PGSO and HGBV cannot be used directly to power a diesel engine and must therefore be processed to improve their quality [56,57].

The saponification indices of BVSO and PGSO were 207,57 and 183,03 mg KOH/g of oil, with peroxide indices of 10 and 8,06 meq O_2/kg of oil respectively. These values exceed those by Sharma et al. [14] for BVSO and are close to those previously mentioned by Ayodele and Badejo [55] and Araújo and al [22]. for PGSO. These saponification values indicate that the average molecular mass of the oils was 829.35 g/mol and 1080.21 g/mol for BVSO and PGSO, respectively, suggesting that these oils can be used as raw material for soap production. In addition, these saponification values meet the quality requirements of the U.S. standard for use as biodiesel (ASTM D5558-95). The peroxide values obtained are by the Codex Alimentarius [58], which sets a maximum value of 10 meq O_2/kg for

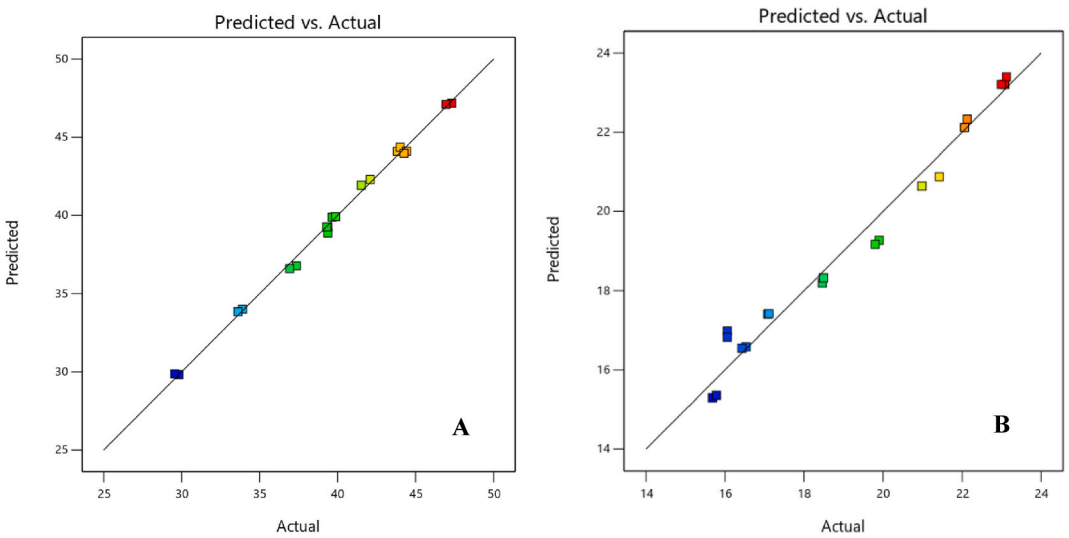

Fig. 8. Plots for predicted values against experimental values for PGSO **(A)** and BVSO **(B)** extraction process.

Table 8
Physicochemical characteristics of *BVSO*.

N°	Properties	Values
1	Acid value (mg KOH/g)	4.63 ± 0.60
2	Oleic acid (%)	9.32 ± 1.20
3	Free Fatty Acid (%)	2.31 ± 0.30
4	Iodine value (g of iodine/100 g)	17.26 ± 0.18
5	Saponification value (mg KOH/g)	207.57 ± 1.98
6	Peroxide value (meq O_2/kg)	10
7	Density at 20 °C (kg/m^3)	885.47 ± 0.05
8	Cetane number	68.71 ± 0.29
9	Higher Heating value (MJ/kg)	40.66 ± 0.06
10	Dynamic viscosity (Pa.s)	3.81 × 10^{-3}±0.0003
11	Kinematic viscosity at 40 °C (mm^2/s)	3.24 ± 0.25
12	Flash point (°C)	216.37 ± 3.14
13	Oxidative stability (h, 110 °C)	5.32
14	Impurity content (%)	2.23 ± 0.22
15	Ester value (mg KOH/g)	202.94 ± 1.82

Table 9
Physicochemical characteristics of *PGSO*.

N°	Properties	Values
1	Acid value (mg KOH/g)	27.21 ± 0.40
2	Oleic acid (%)	54.80 ± 0.80
3	Free fatty acid (%)	13.60 ± 0.20
4	Iodine value (g of iodine/100 g)	12.37 ± 0.27
5	Saponification value (mg KOH/g)	183.03 ± 2.98
6	Peroxide value (meq O_2/kg)	8.06 ± 2.02
7	Density at 20 °C (kg/m^3)	877.32 ± 0.001
8	Cetane number	73.34 ± 0.55
9	Higher Heating value (MJ/kg)	65.08 ± 0.13
10	Dynamic viscosity (Pa.s)	1.18 × 10^{-2}±0.0002
11	Kinematic viscosity at 40 °C (mm^2/s)	12.45 ± 0.98
12	Flash point (°C)	330.17 ± 12.13
13	Oxidative stability (h, 110 °C)	21.49
14	Impurity content (%)	14.87 ± 0.02
15	Ester value (mg KOH/g)	155.82 ± 1.82

refined oils and 15 Meq O_2/kg for crude oils. A high peroxide value can be associated with several factors such as the extraction process, the oxidation rate, and the types of fatty acids present in the oil [55]. The iodine values of BVSO and PGSO were 17.26 and 12.37 g of iodine per 100 g, respectively. Govindhan et al. [3] and Ayodele and Badejo [55] respectively an iodine value that was higher than the observed value for BVSO and slightly lower for PGSO. In addition, the European Standard [56] describing the requirements and test methods (EN 14214) suggests a maximum iodine value of 120 g I_2/100 g of oil for biodiesel. The values obtained from this work indicate that both oils are excellent fuels for producing biodiesel and other industrial chemicals. As for the cetane number, it is an indicator of the efficiency of combustion of fuel in a compression engine. The cetane number of BVSO and PGSO observed in this paper are by the limits suggested by ASTM D 6751 [57] and EN 14214 [56] for biodiesel, which further enhances their potential as raw materials for biodiesel. It can also be noted that the cetane indices obtained from HGSO and HGSO are higher than those of edible oils (canola, coconut, corn, palm, colza, soy, and sunflower) with mean values ranging between 52.3 and 61.5 [53]. BVSO and PGSO showed oxidative stability measured by the Rancimat induction period of 5.32 and 21.49 h respectively. The values observed in this study are by the limit suggested by ASTM D 6751 [57] for biodiesel. The oxidative stability of oil is influenced by the polyunsaturated fatty acids, linoleic (18:2) and linolenic (18:3), which give rise to minor oxidized compounds, contributing to the reduction of the stability at oxidation [25]. The Flash point of BVSO and PGSO are 216.37 °C and 330.17 °C respectively, thus guaranteeing safe storage, transportation, and handling than diesel. Furthermore, the Flash point obtained in this study are significantly higher than those of several edible oils mentioned in the literature, with mean values ranging from 113 to 175 °C [53]. About higher heat value (HHV), PGSO and BVSO have a high energy value. The HHV value of PGSO is higher than that of several other oils, such as maize (43.1 MJ/kg), camelina (45.2 MJ/kg), canola (41.3 MJ (kg), colza (41.1 MJ/kg) [53] and shea butter (42.2 MJ/kg) [59]. The HHV value of BVSO is comparable to that of Jatropha, palm, and sunflower with an average value of 40.6 MJ/kg. While this value is higher than that of soy and coconut with HHV less than 40 MJ/kg [53].

4. Conclusion

In this study, the application of the optimization technique by the response surface methodology (RSM), was implemented to model and optimize the process of extraction of HGPG and HGBV. Several important points were drawn.

- In optimal conditions, the maximum yield of 47.05 % for the PG was achieved with a weekly seed age, a 10-h extraction time, and a 60 °C temperature; for the BV, a 23.1 % yield was obtained under similar conditions, with an extraction period of 6.5 h. The evaluation of the effectiveness of the optimization tool has demonstrated that RSM method is a satisfactory, reliable, and flexible for both prediction and data adjustment.
- The physicochemical properties of HGBV and HGPG have comparatively higher energy properties than other oils, making these seeds excellent alternative and renewable sources to produce biodiesel.

However, for future work, this study could be further improved by examining the impact of particle size on oil yield, as well as comparing the RSM used with other modeling tools, such as the data-driven modeling technique, also known as SVM or the artificial neural network. The future study will focus on the valorization of these oils, to evaluate their effectiveness in industrial applications.

Data availability statement

The data will be made available upon request.

CRediT authorship contribution statement

Ulrich Cabrel Kenmegne Tebe: Writing – review & editing, Writing – original draft, Visualization, Validation, Resources, Project administration, Methodology, Formal analysis, Data curation, Conceptualization. **Julius Kewir Tangka:** Visualization, Validation, Supervision, Resources, Project administration, Conceptualization. **Henri Grisseur Djoukeng:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Resources, Methodology, Conceptualization. **Brice Martial Kamdem:** Writing – review & editing, Writing – original draft, Visualization, Validation, Resources, Formal analysis. **Esther Azemo Folepe:** Resources, Methodology.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

Authors thank A.L. Tapa and F.A. Kouame for their knowledge in data analysis, the Laboratory of Nutrition and Animal Feeding and Soil Sciences Laboratory of the University of Dschang and the Biochemistry Laboratory at Friedrich Alexander University in Erlangen-Nuremberg, Germany, for his assistance in the processing of seeds, extractions, and analysis of *Bauhinia variegata* and *Pachira glabra* oils.

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