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Optimization of Decolorization of *Azadirachta indica.* A. Juss Oil by Activated Carbon Based on the Date Palm Seeds (*Phoenix dactylifera*)

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Abstract The decolorization of edible plant oils involves the adsorption of pigments like chlorophylls and carotenes, along with removal of impurities such as soaps, metals, and phosphorus. This study aimed to optimize the decolorization of *Azadirachta indica A. Juss* oil using activated carbon (AC) derived from *Phoenix dactylifera* kernels. The activation of the coal obtained from *Phoenix dactylifera* kernels was carried out using phosphoric acid. The AC used had an iodine index of 427.95 ±0.02 mg/g, a methylene blue index of 10.58 ±0.01 mg/g, a specific surface area of 521.52 ±0.02 m^2/g and a pH at the point of zero charge (pH_{PZC}) of 5.5. *Azadirachta indica* A. Juss oil was extracted using both the traditional and Soxhlet methods. The optimization of the decolorization of *Azadirachta indica* A. Juss oils was carried out a Central Composite Design (CCD), which is a second-degree mathematical model that involves the variables, mass of AC (X_1), temperature (X_2) and time (X_3). Regarding carotene content, the optimal temperatures were 72 ℃ and 44 ℃ for the oil extracted by the traditional method and for the oil extracted by the Soxhlet method, respectively. For chlorophyll content, the optimal temperature was 55 ℃ for both extraction methods. The optimal reaction times were 40 min and 20 min for carotene content, respectively, for traditional method and for Soxhlet method. Decolorization yields were 41.20% and 4.81% for carotene content and 49.75% and 75% for chlorophyll content, for traditional method and for Soxhlet method respectively. Optimal decolorization of *Azadirachta indica* oil is best for Soxhlet extraction method with minimal time (20 min) and minimum temperature (44 ℃).

Keywords *Azadirachta indica* A. Juss oil, Activated carbon, Optimization of decolorization, Date palm, Oil extraction

1. Introduction

Vegetable oils are essential components of the human nutrition. They provide energy, essential fatty acids, fat-soluble vitamins, and perform important organoleptic functions in food [1]. They also serve as raw materials in various industries, including the food and cosmetics sectors. These oils are mainly derived from oilseeds, including soybean, rapeseed, sunflower, palm, and peanuts. These oils are mainly composed of triglycerides (95-98%), fatty acids, and other minor constituents [2]. Diversification of vegetable oil uses worldwide (oleo chemicals, biofuels, cosmetics, and pharmaceuticals) and population growth have boosted global vegetable oil production in recent years [3], [4], [5]. Vegetable oils contain coloring pigments that reduce their shelf life and aesthetic value. Therefore, it is imperative to separate these coloring pigments to increase their shelf life and improve their aesthetic value. These pigments are composed primarily of chlorophyll, carotenoids, and related compounds. In the presence of light, chlorophyll promotes oxidation and significantly reduces the oxidative stability of oils. Chlorophyll pigments act by blocking nickel's active sites and altering the hydrogenation process [6].

Azadirachta indica A. Juss seeds are very rich in oil, often exceeding 50% of the dry weight of the seeds and contains more than 300 bioactive compounds [7] including limonoids or tetranortriterpenes, protolimonoids, flavonoids, and other constituents such as tannins, beta-sitosterol, and vilasinin [8]. Neem oil is a vegetable oil extracted from *Azadirachta indica* A. Juss seeds. In Cameroon, Neem oil production is carried out artisanally and represents a source of income for

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many households, including seed collectors and processors. The extraction activity is considered profitable, with benefits realized at different levels of the production chain. Neem oil is very bitter, contains vitamin E and essential amino acids. It also has an almost opaque brown color and a distinctive spicy scent and is particularly valued for its medicinal properties, including anti-inflammatory, anti-fungal, antioxidant, cosmetics and biocidal activities [9], but its inherent odor and color pose challenges during consumer use. Therefore, decolorization processes will be essential and necessary to improve its palatability and user acceptance. Decolorization of vegetable oils involves the adsorption of pigments (chlorophylls and carotenes), as well as the removal of impurities (soaps, metals, and phosphorus) [10], [11].

Among the decolorization methods, we have common methods using activated carbon (AC) as an adsorbent, bleaching earths or biosorbents in continuous or batch or processes are used for purification, decolorization, and contaminant removal in applications like wastewater treatment and oil refining. The efficiency of oil decolorization depends on the oil extraction method. This study focuses on the optimization of the decolorization process of *Azadirachta indica* oils extracted by the traditional method and the Soxlet method using AC derived from *Phoenix dactylifera* seeds. AC derived from *Phoenix dactylifera* seeds is particularly promising due to its efficiency and availability as a waste product in date-producing regions [12].

The main objective of this work was to determine the optimal conditions (time, temperature and amount of AC) to achieve maximum decolorization efficiency by using response surface methodology (RSM). To achieve this, the centered composite design (CCD) was selected for its ability to efficiently explore a large experimental space [13], [14], [15].

2. Experimental

2.1. Materials

The fruits of date palm (*Phoenix dactylifera)* were collected and harvested in N'Djamena, Chad. They were manually pitted, washed thoroughly with tap water to remove kernel impurities, and subsequently rinsed with distilled water. Seeds of *Azadirachta indica* A. Juss were collected from the far-north region of Cameroon (Maroua) and stored at room temperature. These seeds underwent various processes to obtain the fine powder required for oil extraction. Chemicals utilized in this study were of analytical grade: Sodium chloride NaCl (99%) and sodium hydroxide NaOH (99%) were supplied by Prolabo, while sulfuric acid H_2SO_4 (96%) was purchased from Sigma-Aldrich (Saint-Quentin Fallavier, France). All test solutions were prepared using ultra-pure Milli-Q water (Millipore, resistivity > 18 MΩ·cm).

2.2. Preparation of Activated Carbon

To produce the adsorbent material, we followed the method outlined by [16]. Initially, the absorbent material (date seeds or kernels of *Phoenix dactylifera*) was thoroughly washed with water, then dried in an oven and subsequently dried at 105 ℃ for 24 hours. Once dried, the date seeds were crushed using a muffle mill (brand RAF) and sieved to achieve particles with a diameter of less than 200 µm. The resulting ground material was stored in tightly closed polyethylene bottles to prevent exposure to air. The powders obtained from this process were then subjected to pyrolysis at 600 ℃ using a programmable NABERTHERM muffle furnace 30-3000 ℃ with a heating rate of 2.5 ℃/min for 3 hours. After calcination, the carbons produced were allowed to cool and stored in a flask.

Next, 300 g of the powdered material (**powder obtained after calcination**) date seeds or kernels was combined with 5.10 mL of a 1N phosphoric acid solution, maintaining a ratio of 1:10. The mixture was stirred for 24 hours on a magnetic stirrer (Mivar Magnetic Stirrer) at room temperature, then filtered through filter paper (**whatman flat qualitative filter paper, grade 1, 11 µm)**. The filtered material was subsequently dried in an oven at 105 ℃ for another 24 hours and cooled in a desiccator. The powders obtained after impregnation were placed back into the programmable NABERTHERM muffle furnace 30-3000 ℃ and heated at 600 ℃ for 3 hours. After cooling in a desiccator for 24 hours, the AC produced underwent a washing process with distilled water for 48 hours, ensuring the pH of the wash water reached 6.5. Following this, the AC was dried for 24 hours in an oven at 105 ℃. Upon removal from the oven, the sample was cooled for 30 minutes in a desiccator before further use.

2.2.1. Physico-Chemical Characterization of Activated Carbon

After the preparation of AC, several physicochemical characteristics were determined, including pyrolysis yield, moisture content, ash content, iodine number, methylene blue index, specific surface area, and pH at the point of zero charge.

The yield reflects the mass loss during chemical decomposition under heat and indicates the activation degree of activated carbon and was calculated using Equation 1.

$$\text{Yield}(\%) = \frac{\mathbf{M'}}{\mathbf{M}} \times \mathbf{100} \qquad (1)$$

- M′ is the final mass (g);
- M is the initial mass (g).

Moisture content (MC) was determined using the [17] method, involving drying a known mass of the sample at 105 ℃ until a constant mass is achieved and is calculated using Equation 2.

$$\boldsymbol{MC}(\%) = \frac{\mathbf{m1-m2}}{\mathbf{m1-m0}} \times \mathbf{100} \qquad (2)$$

- m_0 is the mass (g) of the empty crucible;
- m_1 is the mass (g) of the crucible plus wet sample;
- m_2 is the mass (g) of the crucible plus sample after drying.

The ash content represents the residue after organic material is incinerated at 550 ℃. It was obtained according to the [17] method described by [18] and it is calculated using Equation 3.

$$\textbf{Ash content}(\%) = \frac{\mathbf{m3-m1}}{\mathbf{m2}} \times \mathbf{100} \qquad (3)$$

- m_1 is the empty crucible mass (g);
- m_2 is the mass (g) after drying;
- m_3 is the mass (g) after incineration.

The volatile matter content (VM) was determined by drying powdered carbon at 105 ℃ until constant weight, followed by calcination at 550 ℃ and it is calculated using Equation 4.

$$\text{VM} = \left(\frac{M1-M2}{M1}\right)*100 \qquad (4)$$

- M_1 is the mass (g) of the powder dried at 105 ℃;
- M_2 is the mass (g) of ash after calcination at 550 ℃.

The iodine number was determined using a known mass of AC mixed with 0.1N iodine solution and it is calculated using Equation 5.

$$\text{Iodine index} = \left(\frac{(Vb-Vs)X\ N\ X\ M\ X\ 1{,}5}{mA}\right)*100 \qquad (5)$$

- Vb and Vs are volumes (mL) of sodium thiosulfate solution used for blank and iodine solution;
- N is the normality of sodium thiosulfate solution (0.1 mol/L);
- M is the molar mass of iodine;
- mA is the mass (g) of the adsorbent.

The specific surface (SS) area was determined using methylene blue adsorption (IBM). IBM represents the amount of methylene blue (mg/g) needed to cover the monomolecular layer of the external and internal surfaces of the fine particles of a solid suspended in water. It was determined by the method used by [19] and [20] with some modifications and it is calculated using Equation 6.

$$\mathbf{IBM}\left(\frac{\mathbf{mg}}{\mathbf{g}}\right) = \frac{\mathbf{(Ci-Cf)V}}{\mathbf{mAC}} \qquad (6)$$

- Ci and Cf are initial and final methylene blue concentrations (mg/L);
- V is the solution volume (L);
- mAC is the mass of AC.

The specific surface is given by Equation 7.

$$\text{SS} = \text{Qm} \times N_A \times S_{BM} \qquad (7)$$

- SS: Specific surface of Langmuir (m^2/g);
- Qm: Maximum adsorption capacity (mg/g);
- N_A: Number of Avogadro ($6{,}022.10^{23} mol^{-1}$);
- S_{BM}: Area occupied by a molecule of methylene blue ($175.10^{-20} m^2$).

The pH at zero point of charge (pH_{ZPC}) is defined as the pH at which there is neither a positive nor negative charge on the surface of activated carbon. A mass (g) of activated carbon was brought into contact with 20 mL of 0.1M NaCl solution, and the pH was varied from 2 to 10 under magnetic stirring for 24 hours. After filtration, the new pH was measured, and the curve pHf – pHi = f(pHi) was plotted. The point of intersection between this curve and the pHi axis gives the pH at zero point of charge (pH_{ZPC}).

2.3. Oil Extraction Method

Extraction of *Azadirachta indica* A. Juss oils was conducted using Soxhlet and traditional methods.

For the Soxhlet method, 300g of *A. indica* A. Juss powder was initially placed in a capsule and introduced into a 500 mL flask containing a refrigerant. Then, 250 mL of hexane was added to the flask with pumice stones to control boiling temperature. The apparatus was connected to tap water for cooling during oil extraction. The entire setup was placed in a heated chamber at 68 ℃ for 8 hours. After heating, the oil-hexane mixture was recovered and subjected to rotary evaporation (Heidolph) to separate the oil from hexane. The residual solvent was removed by placing the mixture in an oven at 105 ℃ for 1 hour. The flask containing the oil was then stored in a desiccator to prevent moisture absorption.

Traditional extraction methods involve manual techniques. *A. indica* A. Juss seeds were ground into a fine powder using a mortar. The powder was mixed with water to form a paste, which was then boiled in a pot until oil appeared on the surface. The oil was collected, cooled, and reheated until all water evaporated. The resulting oil was stored in a bottle to prevent contamination.

2.4. Decolorization of *Azadirachta indica* Oil

Nearly all oils undergo a decolorization process during production or processing. This step aims not only to reduce oil color but also to remove or convert undesired products from fats and oils. Decolorization is a batch process. The experiment on the bleaching of *Azadirachta indica* oil was carried out in a 250 mL Erlenmeyer flask. 10 mL of *Azadirachta indica* oil was introduced into the Erlenmeyer, then the required amount of activated charcoal (X_1) obtained from *Phoenix dactylifera* seeds was added, and the mixture was brought to the experimental temperature (X_2) for a duration (X_3) under stirring at 250 rpm using the Mivar Magnetic Stirrer. Subsequently, the bleached oil was separated from the AC using a centrifuge (Centrifuga MKE T4-S) and the carotene and chlorophyll contents were determined.

Carotenoid content 0.2 g of oil was weighed into a 20 mL flask and diluted to 1% with cyclohexane. Using a spectrophotometer (Model ZUZI 4211/50), absorbance was measured between 405 and 500 nm, with the control filled with cyclohexane [21], [22]. Carotenoid concentration is expressed in ppm of fat and was calculated using Equation 8.

$$\textbf{Carotenoid content } (\mathbf{ppm}) = \frac{\mathbf{A\ x\ V}}{\mathbf{PS\ x\ 0.261}} \qquad (8)$$

Where: A: Absorbance at 401 nm; - V: Volume (mL); - PS: Sample mass (mg).

Chlorophyll content: 0.2 g of oil was weighed into a 20 mL vial and diluted to 1% with cyclohexane. Absorbance measurements were taken at wavelengths 630, 670, and 710 nm using a spectrophotometer (Model ZUZI 4211/50) with a 1 cm path length, compared to a control filled with cyclohexane [21]. Chlorophyll content in oil, expressed in ppm of fat and was calculated using Equation 9.

$$\textbf{Chlorophyll content (ppm)} = \frac{[\mathbf{A670-((A630+A710)/2)}]}{\mathbf{K\,x\,L}} \quad (9)$$

Where: A_{630}, A_{670}, and A_{710} respectively absorbance at 630, 670, 710 nm; - L: Tank thickness (1 cm); - K: Extinction coefficient of chlorophyll in oil (k = 0.0964).

2.5. Optimization of Decolorization of *Azadirachta indica* A. Juss Oil

Response surface methodology (RSM) was employed to optimize the decolorization process of activated carbon-based Neem oil derived from *Phoenix dactylifera* kernels. Initial experiments guided the selection of temperature, time, and mass of activated carbon as variables. For the optimization of oil decolorization, a centered composite design was chosen. With three factors, this design involves $2^3 + 2 \times 3 + 3 = 17$ tests to be conducted. Thus, variables were designated as X_1 for activated carbon (AC) mass (g), X_2 for temperature (℃), and X_3 for time (min) (Table 1).

Table 1. Parameter values with their variation levels

Variable parameters	-α	-1	0	+1	+α
Mass AC X_1 (g)	0.02	0.01	0.03	0.05	0.45
Temperature X_2 (℃)	27.00	35.00	57.00	80.00	87.00
Time X_3 (min)	9.00	15.00	30.00	45.00	50.00

Variable levels and their characteristics. High levels (+1), lower levels (-1), and α = 1.682

The design of the different experiments and the analysis of the results were done by the design expert. The experimental response associated with the CCD was characterized by a quadratic polynomial model:

$$\boldsymbol{Y = \beta_0 + \sum\beta_i x_i + \sum\beta_{ii} x_i^2 + \sum\beta_{ij} x_i x_j + \beta_{ijk} x_i x_j x_k + \varepsilon} \quad (10)$$

Where: Y: predicted response; β_0: constant coefficient; βi, βij: coefficients of linear, interaction, and quadratic terms, respectively; X_1, X_2 and X_3: coded variables; ε: error term.

In order to assess the accuracy of the fitted model, the analysis of variance, the lack of fit, and the coefficient of determination R^2 were carefully exploited (**statistical analysis)**. After that, 3D graphs were plotted to show the response surface, the interaction between factors, and their combined effect on the efficiency of the decolorization.

3. Results and Discussion

3.1 Characterisation of the Actived Carbon (AC) Derived from *Phoenix dactylifera*

The yield of AC derived from *Phoenix dactylifera* was 77.01% (Table 2). It is an important measure of the performance of the AC preparation process. This value is higher than that obtained for AC derived from coconut shells (38%) and close to that obtained for AC derived from date palm pits (73%) [23], [24]. This difference would be due to the nature of the constituents of the different plants.

The moisture content is 0.39%, relatively lower than that reported by [25] for AC from *Bos indicus* bones, which is 1.50%. This difference may be attributed to the nature of the materials used.

The ash content provides insight into the activated carbon's ability to retain very small molecules. It is observed to be 3.76% (Table 2), a value similar to that reported by [26], for AC from date pits (4%). This low ash content indicates that activated carbon derived from *Phoenix dactylifera* pits could have excellent adsorption capacity and hence potential for good microporosity. The ash content can significantly influence the properties of AC as it represents the inert content that reduces overall activity by potentially blocking pores and occupying adsorption surfaces. Thus, activated carbons with lower ash content are generally more effective.

The volatile matter content (99.61%) indicates that a large portion of the material's constituents, such as lignin, cellulose, and hemicellulose, volatilized during calcination. The iodine number provides insights into the specific surface area evolution of AC and is commonly used to measure porosity. The iodine adsorption capacity on AC derived from *Phoenix dactylifera* pits is 427.95 mg/g, indicating the presence of micropores on its surface. Micropores largely determine the adsorption capacity of AC, as they represent nearly all of the available surface area and volume for adsorption [25]. This value is similar to that obtained by [24] for activated carbons derived from coconut shells (432 mg/g).

The methylene blue index is 10.58 mg/g, similar to that obtained by [27] for AC from sugarcane bagasse, but higher than that obtained by [23] for AC from coconut shells (10.47 mg/g and 3.32 mg/g, respectively). A low methylene blue index is generally indicative of smaller particle size of AC, with larger sizes typically exhibiting reduced adsorption capacity. Furthermore, the specific surface area is 521.52 m^2/g, indicating that AC derived from *Phoenix dactylifera* *p*its possesses a high surface area and good adsorption capacity for both small and large molecules. This value is lower than that obtained by [18], for AC from cocoa pod husk (621.45 m^2/g), possibly due to differences in carbonization temperature. The specific surface area plays a crucial role in pollutant removal via adsorption in aqueous environments.

Table 2. Physicochemical characteristics of activated carbon

Yield (%)	77.01 ± 0.02
Humidity (%)	0.39 ± 0.01
Ash (%)	3.76 ± 0.10
Volatile matter (%)	99.61 ± 0.02
Iodine index (mg/g)	427.95 ± 0.02
Methylene blue (mg/g)	10.58 ± 0.01
Specific surface (m^2/g)	521.52 ± 0.02

The pH at the point of zero charge (pH_{PZC}) (Figure 1) provides information on the surface charges developed by the absorbent material at its surface. The equilibrium pH_{PZC} of activated carbon derived from *Phoenix dactylifera* pits is 5.5. This value is similar to that obtained for AC based on shea and cotton cake [28].

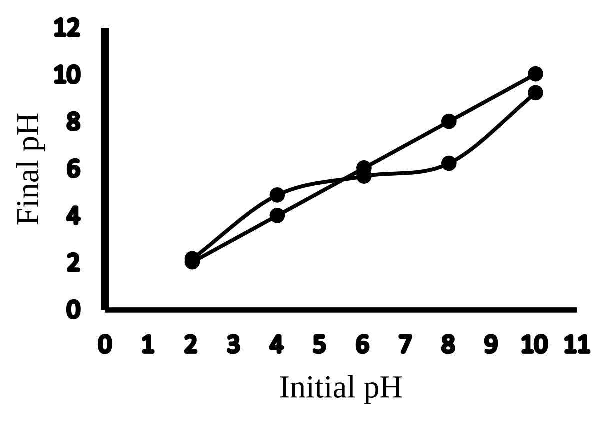

Figure 1. pH at the point of zero charge

3.2. Optimization by Response Surface Methodology

3.2.1. Experimental Results

The study of the influence of variable parameters, namely mass AC (X_1), temperature (X_2), and time (X_3), was conducted. The responses obtained were the content of carotenes (Y_{CB}) and chlorophyll (Y_{CHB}) in the oil extracted by the traditional method, and the content of carotenes (Y_{CS}) and chlorophyll (Y_{CHS}) in the oil extracted by Soxhlet extraction. The experimental design chosen was a centered composite design. Thus, with three (3) factors, 17 experiments were conducted, and the results are presented in Table 3. It is noted that the experiments were conducted randomly to avoid any influence of the experimenter on the response. In practice, we assumed that interactions of order three or higher are negligible as these interactions are often not significant in the analysis of variance and are difficult to interpret.

Table 3. Experiment plan with the obtained responses

	Real variables			Carotene content (calculated and adjusted)				Chlorophyll content (calculated and adjusted)			
N °exp	X_1	X_2	X_3	Y_{CBC}	Y_{CBA}	Y_{CSC}	Y_{CSA}	Y_{CHBC}	Y_{CHBA}	Y_{CHSC}	Y_{CHSA}
1	-1	-1	-1	2.00	2.01	3.15	3.15	0.07	0.06	0.08	0.08
2	+1	-1	-1	1.94	1.93	2.57	2.53	0.04	0.03	0.14	0.13
3	-1	+1	-1	2.64	2.59	2.28	2.21	0.24	0.22	0.02	0.02
4	+1	+1	-1	2.22	2.21	2.71	2.72	0.16	0.15	0.16	0.14
5	-1	-1	+1	2.53	2.50	2.94	2.91	0.24	0.24	0.20	0.21
6	+1	-1	+1	2.53	2.54	2.16	2.21	0.27	0.27	0.25	0.24
7	-1	+1	+1	2.20	2.17	2.38	2.40	0.02	0.02	0.04	0.04
8	+1	+1	+1	1.95	1.90	2.85	2.83	0.01	0.01	0.15	0.14
9	-α	0	0	2.15	2.19	2.56	2.60	0.22	0.22	0.16	0.13
10	+α	0	0	1.93	1.95	2.49	2.47	0.19	0.19	0.22	0.24
11	0	-α	0	2.25	2.24	2.66	2.65	0.17	0.19	0.17	0.17
12	0	+α	0	2.13	2.20	2.04	2.43	0.09	0.1	0.07	0.06
13	0	0	-α	2.24	2.25	2.67	2.72	0.12	0.14	0.09	0.09
14	0	0	+α	2.33	2.38	2.68	2.64	0.19	0.17	0.19	0.18
15	0	0	0	2.13	2.20	2.58	2.53	0.24	0.22	0.19	0.17
16	0	0	0	2.21	2.15	2.52	2.53	0.23	0.22	0.17	0.16
17	0	0	0	2.21	2.15	2.52	2.53	0.23	0.22	0.17	0.17

Y_{CBC} and Y_{CBA}: Carotene content respectively calculated and adjusted from oil extracted by the traditional method;
Y_{CSC} and Y_{CSA}: Carotene content respectively calculated and adjusted from oil extracted by Soxhlet;
Y_{CHBC} and Y_{CHBA}: Chlorophyll content respectively calculated and adjusted from oil extracted by the traditional method;
Y_{CHSC} and Y_{CHSA}: Chlorophyll content respectively calculated and adjusted from oil extracted by Soxhlet.

The Design Expert software enabled the generation of second-degree polynomial mathematical models describing the decolorization of traditional (Equation 11 and 12) and Soxhlet-extracted oils (Equation 13 and 14).

$$\begin{aligned} Y_{CB} = {} & 1{,}342 + 9{,}313\ X_1 + 0{,}016\ X_2 + 0{,}015\ X_3 \\ & - 114{,}189\ X_1^2 - 0{,}169\ X_1{*}X_2 + 0{,}095\ X_1{*}X_3 \\ & + 0{,}00007\ X_2^2 - 0{,}0006\ X_2{*}X_3 + 0{,}0003\ X_3^2 \end{aligned} \quad (11)$$

$$\begin{aligned} Y_{CS} = {} & -\ 0,\ 710 + 0{,}865\ X_1 + 0{,}019\ X_2 + 0{,}025\ X_3 \\ & - 26{,}874\ X_1^2 - 0{,}025\ X_1{*}X_2 + 0{,}054\ X_1{*}X_3 \\ & - 0{,}0001\ X_2^2 - 0{,}0002\ X_2{*}X_3 - 0{,}0001\ X_3^2 \end{aligned} \quad (12)$$

$$\begin{aligned} Y_{CHB} = {} & 4{,}830 - 36{,}928\ X_1 - 0{,}033\ X_2 - 0{,}041\ X_3 \\ & + 7{,}545\ X_1^2 + 0{,}627\ X_1{*}X_2 - 0{,}066\ X_3{*}X_1 \\ & + 0{,}00001\ X_2^2 + 0{,}0003\ X_2{*}X_3 + 0{,}0003\ X_3^2 \end{aligned} \quad (13)$$

$$\begin{aligned} Y_{CHS} = {} & -\ 0{,}216 - 0{,}769\ X_1 + 0{,}006\ X_2 + 0{,}012\ X_3 \\ & + 15{,}420\ X_1^2 + 0{,}038\ X_1{*}X_2 - 0{,}016\ X_1{*}X_3 \\ & -0{,}00006\ X_2^2 - 0{,}00008\ X_2{*}X_3 - 0{,}00009\ X_3^2 \end{aligned} \quad (14)$$

Where X_1, X_2, and X_3 represent mass of AC, temperature, and time respectively.

The model validation parameters (Table 4) were determined from the equations. A model as valid if the responses (R^2) are greater than 90%. In our study, the R^2 values were 96.68%, 97.70%, 98.62%, and 97.31% for both oils. Additionally, all adjusted R^2 values were greater than 90%, confirming the high significance of the model. Similarly, a model is valid if the Accuracy Factor and Bias Factor vary between 0.75 and 1.25 [29] and if the Average Absolute Deviation of the Median (AADM) is between 0 and 0.3 [30]. Our results fit well with these model validation parameters.

Increasing temperature reduced the carotene and chlorophyll concentrations of the oils, likely due to thermal degradation of fatty substances as demonstrated in the decolorization of shea butter and palm oil using Cameroonian smectite [31]. Conversely, mass would have notable negative effect, darkening the oil with increasing quantity, due to increased energy dissipation and rapid environmental conditioning. Time accelerated oxidation reactions and pigment adsorption due to surface area exposure. Quadratic effects were insignificant, indicating that the mass effects of activated carbon were minimal regardless of the oil quantity. The interaction effects positively influenced chlorophyll and carotene contents in both oils. The interaction of AC mass and reaction temperature reduced chlorophyll and carotene contents. In Equation 11, there is a positive singular effect (9.31) but a negative quadratic effect (-114.189) of variable X_1 (mass). Conversely, Equation 13 shows a positive singular effect (0.016) and a positive, although negligible, quadratic effect (0.00007) of X_1. This difference could result from AC contact during stirring, which fixes small particles and facilitates the breaking of fatty acid bonds cleavage. Equation 13 also shows positive singular (0.016) and quadratic (0.0003) effects of X_3 (time), influencing oxidation and adsorption. Positive interactions (0.095) between X_2 (temperature) and X_3 were observed in Equation 13, increasing carotene content.

For chlorophyll models, Equation 12 shows a positive singular effect (0.865) of X_1 (mass) but a negative quadratic effect (-26.874), reducing chlorophyll content. In contrast, Equation 14 reveals a negative effect (-0.769) of X_1 and a positive quadratic effect (15.420). Other effects such as X_2, X_3, and their interactions were minimal.

Table 4. Model validation parameters

Validation criteria	Y_{CB}	Y_{CS}	Y_{CHB}	Y_{CHS}	Standad value
R^2 (%)	96.68	97.70	98.62	97.31	100
R^2ajusted (%)	92.41	94.76	96.85	93.86	100
AADM	0.00000003	0.00000001	0.00000003	0.000000007	0-0.03
Bias factor	0.999999468	0.999999773	1.00000000	0.999999879	1
Accuracy Factor (Af_1)	0.999999468	0.999999773	1.00000000	0.999999879	1
Accuracy Factor (Af_2)	0.99999984	0.999999932	1.00000000	0.999999963	1

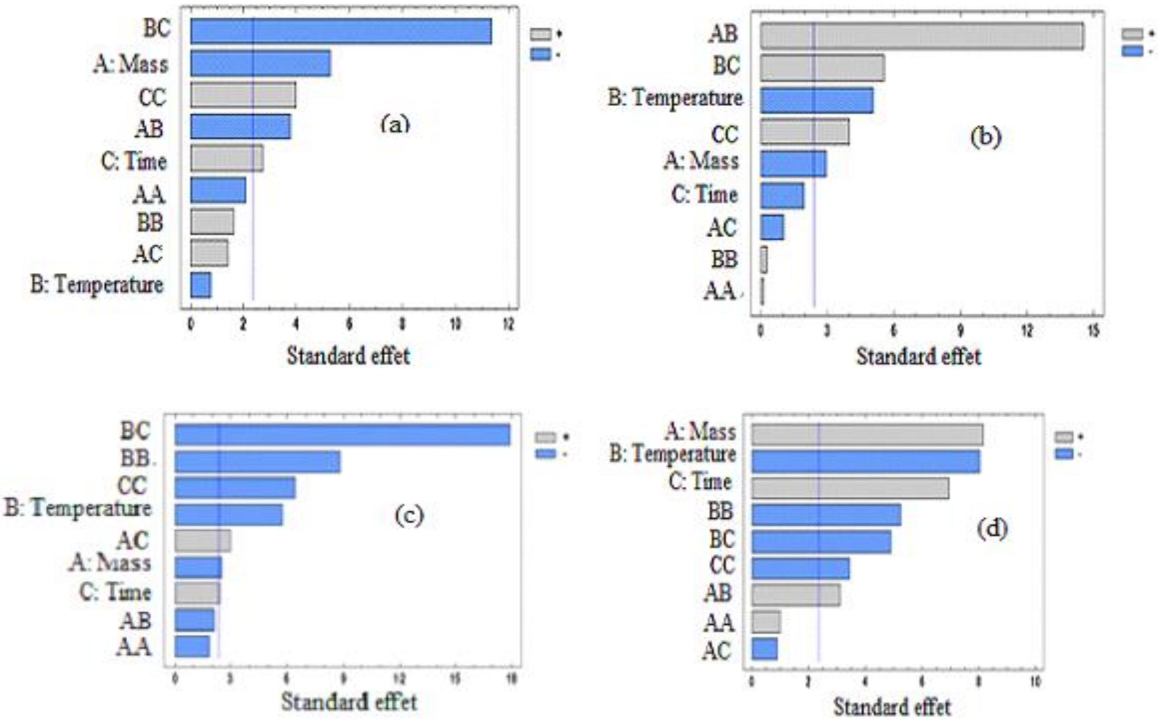

Figure 2. Pareto chart of carotenes of traditional oil (a) and solvent extracted oil (b); chlorophylls of traditional oil (c) and solvent extracted oil (d)

3.2.2. Pareto Analysis

Pareto analysis involves interpreting the influence of each parameter, as described by Equation 15.

$$pi = \frac{bi^2}{\sum bi^2} \times 100 \ (i \neq 0) \tag{15}$$

where bi represents the regression coefficient associated with the parameter under study.

The Pareto diagrams illustrate the significant effects of each variable and their interactions (Figure 2). Variables crossing the vertical line indicate significant (positive or negative) influences on the response. Positive influences contribute to increased decolorization, reducing carotene and chlorophyll content, while negative influences increase these responses. The Pareto chart analysis is detailed in Figures 2a, 2b, 2c, and 2d, illustrating the percentage effects of each variable independently and their interactions on the response.

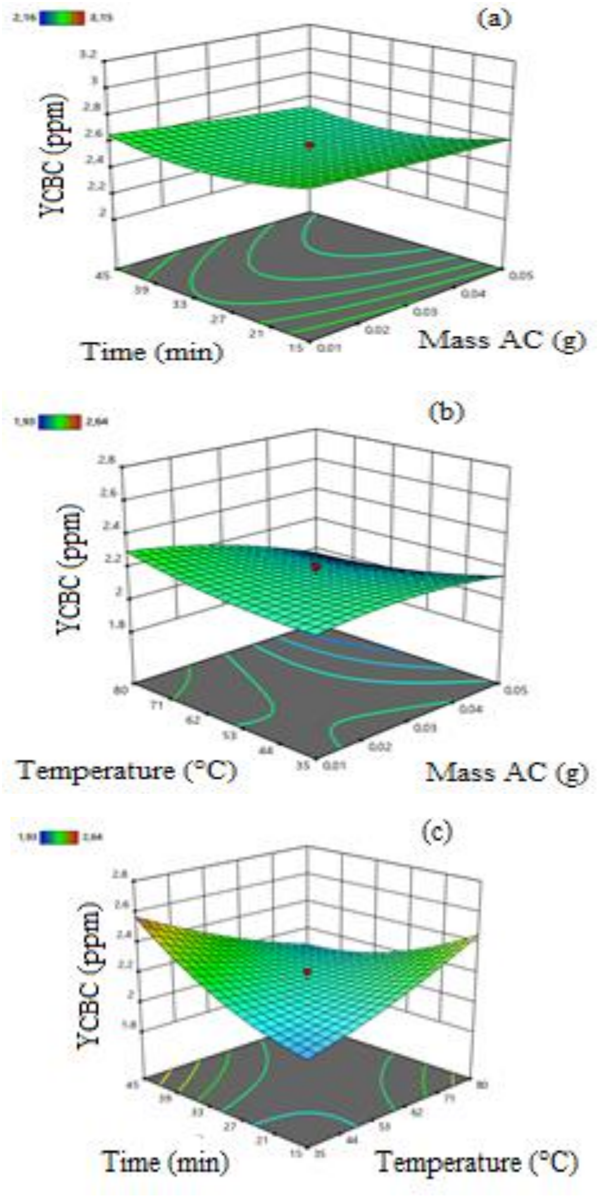

Figure 3a-c. Response Surface curves for traditional oil. (carotenes)

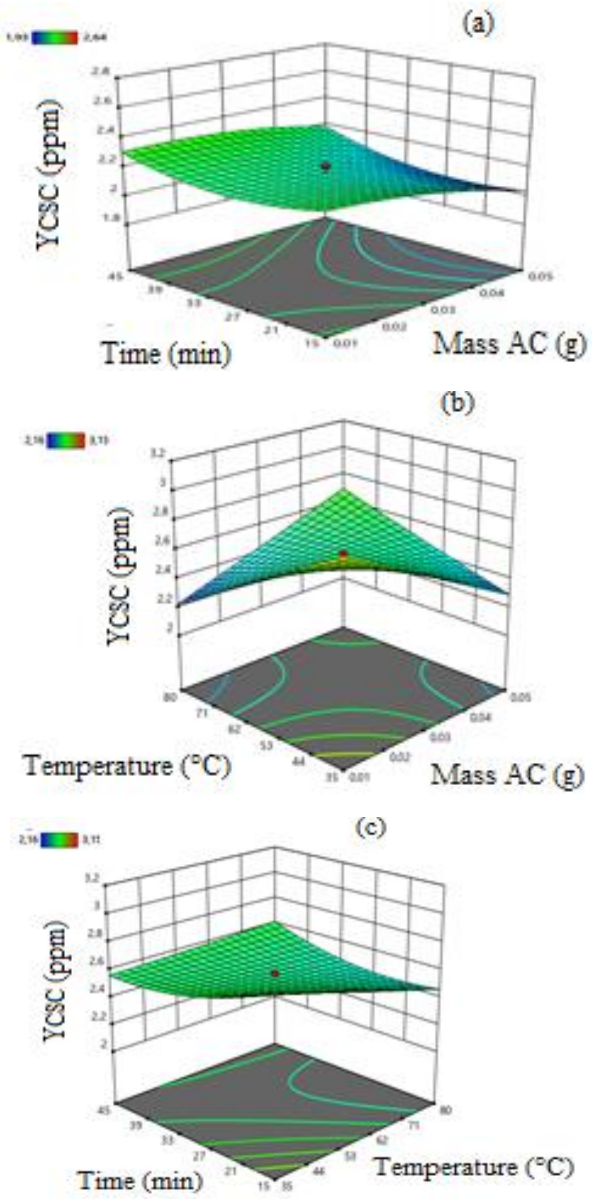

Figure 3a-c. Response Surface curves for oil extracted with soxhlet. (carotenes)

Figure 3. Response surface curve carotenes content

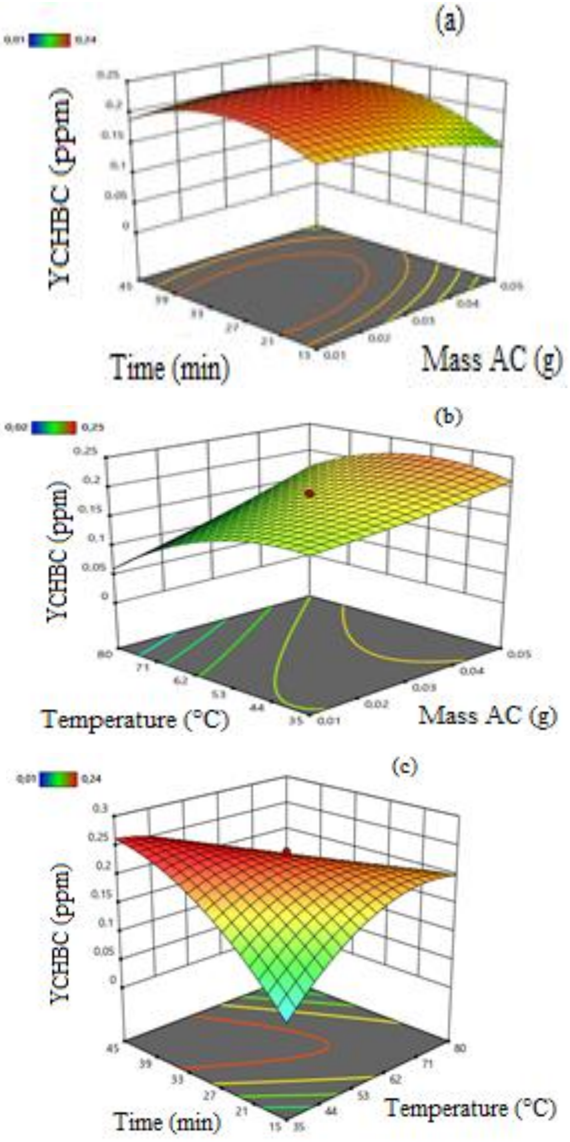

Figure 4a-c. Response Surface curves for traditional oil. (chorophylls)

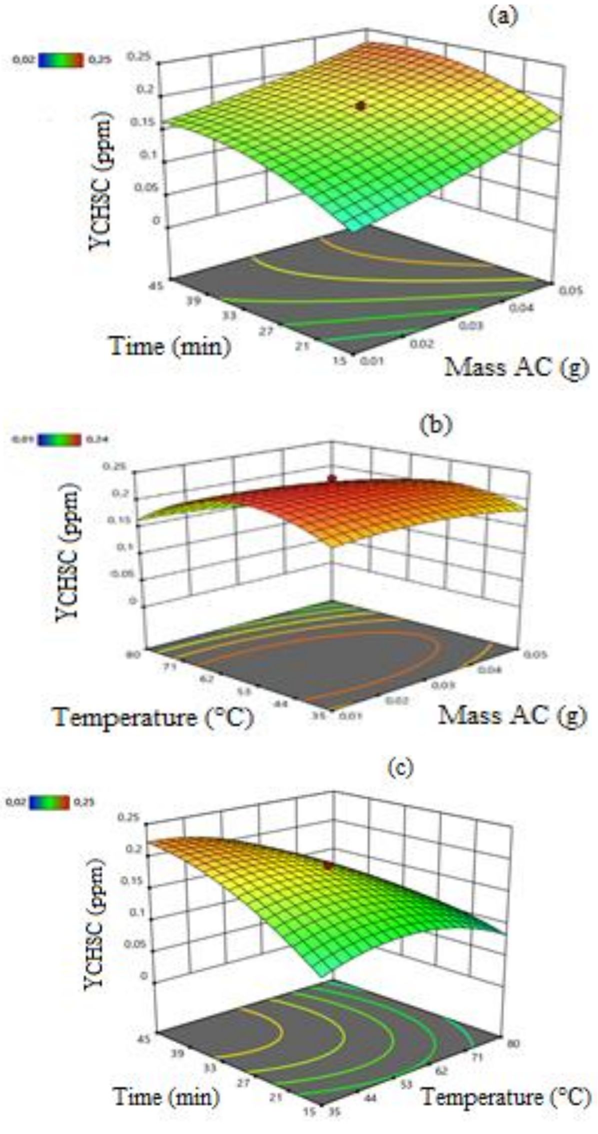

Figure 4a-c. Response Surface curves for oil extracted with soxhlet. (chorophylls)

Figure 4. Response Surface curve for chlorophylls content

Table 5. Optimal conditions for oil decolorization

Oils	Optimal variables			Experimental responses		Decoloration rate (%)
	Mass (X_1)	Temperature (X_2)	Time (X_3)	Carotenes	Chlorophylls	
Traditional method	0.02	72	40	2,2		41.20
	0.03	55	39		0.05	49.75
Soxhlet method	0.02	44	20	2,0		4.81
	0.03	55	39		0.15	75.00

3.2.3. Response Surface

Graphs are essential tools for interpreting results and drawing conclusions about factors influencing oil discoloration (raw oil and Soxhlet-based extract oil). Our experimental domain explores variations in three parameters mass of activated carbon, reaction temperature, and reaction time, which significantly influence our response surfaces. Response surfaces are three-dimensional (3D) graphs where the horizontal plane represents the variation domain of two factors, and the vertical axis depicts changes in the response surface (carotenes and chlorophylls) (Figure 3). From Figure 3a, interactions between temperature and mass detrimentally affect carotene discoloration. Increasing temperature decreases carotene content initially but may lead to oil denaturation beyond a threshold due to the presence of fatty acids inhibiting breakdown. Conversely, Figure 3b shows that increasing mass and temperature, as well as time and temperature, collectively favor reduced carotene concentrations in the solvent-extracted oil. This reduction is likely due to pigment adsorption on the activated carbon's specific surface area, characterized by macro pores. These findings align with prior studies on optimizing vegetable oil decolorization parameters [31], [32].

It appears from figure (3a) that the interaction of temperature and mass disadvantages the discoloration of the carotene content. However, an increase in temperature leads to a decrease in carotene content but at a certain threshold this leads to oil denaturation. This can be explained by the dense presence of fatty acids present in the oil. However, an increase in temperature hinders the breakdown between the fatty acids. This corroborates with the work of [31]. A mass disadvantages the discoloration of carotene. In our work, mass has a negative influence. The more the mass increases, the blacker the oil becomes. Our results are in contradiction with those of [32] who carried out his work on the optimization of the parameters of decolorization of vegetable oils with bleaching earth. As for the percentage of the earth, the more the discoloration increases. The same phenomena are observed in figure (3b). Figures (3a) and (3b) respectively show that the cumulative effects of mass and temperature as well as time and temperature favor the decrease in the concentration of carotene content from 3.2 to 2.2 ppm and 3, 2 to 2.5 ppm in the oil extracted by the solvent. This reduction can result in the adsorption of pigments on the specific surface of our activated carbon which has macro pores. These results are similar to the work of [21] on the optimization of decolorization parameters of vegetable oils. The same phenomena are observed in figure (3c).

The Figures 4a-c illustrate that the interactions between mass and time, as well as mass and temperature, contribute to reducing the chlorophyll concentration from 0.24 to 0.1 ppm in traditional oil. This decline is attributed to pigment adsorption on the pores of our activated carbon, which boasts a large specific surface area. These findings align with Kahina's work in 2017 on optimizing decolorization parameters for soybean and sunflower oils, indicating that higher temperatures, increased mass, and longer durations enhance pigment adsorption hena [32], [33], [34], [35]. Figures 4a-c demonstrate similar trends, where interactions between mass and time, along with mass and temperature, lead to decreased chlorophyll concentrations from 0.25 to 0.2 ppm in Soxhlet-extracted oil.

3.2.4. Optimum decolorization of *Azadirachta indica A. Juss* oil

Table 5 summarizes the optimal conditions for oil decolorization, encompassing the combined optima of all variables and the contents of carotenes and chlorophylls. Optimization significantly improved the decolorization of *Azadirachta indica A. Juss* oil using our activated carbon from *Phoenix dactylifera*. In traditional oil, the decoloration rates for carotenes and chlorophylls were 41.20% and 49.75%, respectively (table 5). For Soxhlet-extracted oil, these rates were 4.81% for carotenes and 75.00% for chlorophylls. This indicates that chlorophyll reduction is more pronounced compared to carotenes for both extraction methods. Additionally, the low decoloration rate of carotenes in Soxhlet-extracted oil may be due to denaturation caused by extraction temperatures [11].

4. Conclusions

The objective of this study was to decolorize *Azadirachta indica* A. Juss oil using activated carbon derived from *Phoenix dactylifera*. Thus, the characterization of AC prepared from *Phoenix dactylifera* seeds and activated with phosphoric acid allowed us to obtain an AC production yield of 77.01%, an iodine number of (427.95 ± 0.02) mg/g, a methylene blue number of (10.58 ± 0.01) mg/g, a specific surface area of (521.52 ± 0.02) m^2/g, and a pH_{PZC} of 5.5.

The extraction of *Azadirachta indica* oils was carried out using both the traditional method and the Soxhlet method. The optimization of the discoloration of the obtained oils was carried out using a central composite design with the variable parameters being the mass of AC, the reaction time, and the experiment temperature, and with the response being the chlorophyll content and the carotene content. It appears that the mathematical models that explain the discolorization of *Azadirachta indica* oils by AC obtained from *Phoenix dactylifera* activated with phosphoric acid are second-degree mathematical models with interactions between the different parameters (time, temperature, and mass of AC). The most influential parameter, whether for determining the carotene or chlorophyll content, for both oil extracted using the traditional method or the Soxhlet method, is the mass of AC.

Regardless of the extraction method of *Azadirachta indica A. Juss* oil, the optimal decolorization conditions are an AC mass of 0.02 g and 0.03 g for carotene and chlorophyll content, respectively, at a temperature of 55 ℃. Furthermore, the optimal decolorization temperature of *Azadirachta indica A. Juss* oil is 72 ℃ and 44 ℃ for carotene and chlorophyll content, respectively, for oil extracted using the traditional method and Soxhlet method, over a time of 40 and 20 minutes. Also, under optimal conditions, the decolorization rate are 40.20% and 4.81% for carotene content for oil extracted by the traditional method and Soxhlet method, respectively, and 49.75% and 75.00% for chlorophyll content. Optimal decolorization of *Azadirachta indica* oil is best for Soxhlet extraction method with minimal time (20 min) and minimum temperature (44 ℃).

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Conflick of Interest

The authors declare that they have no conflict of interest.

Funding Declaration

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