ISSN: 1648-6897 / eISSN: 1822-4199

JOURNAL of ENVIRONMENTAL ENGINEERING & LANDSCAPE MANAGEMENT

2025
Volume 33
Issue 2
Pages 207–214
https://doi.org/10.3846/jeelm.2025.23565

REGENERATION AND CHARACTERIZATION OF SPENT BLEACHING EARTH: RECYCLING IN THE CORN OIL BLEACHING PROCESS

Ikhlas BENKHOUD✉, Lassaad BEN MANSOUR

Laboratory of Applied Fluid Mechanics, Process Engineering and Environment, Faculty of Sciences of Sfax, University of Sfax, Sfax, Tunisia

Highlights:
- maceration as solid-extraction technique for oil extraction from SBE;
- optimization of SBE regeneration process by maceration using experimental planning methodology (EPM);
- analysis and characterization of SBE before and after regeneration;
- successful recycling of RSBEH in the edible oil bleaching process.

Article History:
- received 02 April 2024
- accepted 13 January 2025

Abstract. The spent bleaching earth (SBE) is a solid waste from the edible oil refining industry which generates soil contamination was successfully recycled after deoiling through an extraction process using different organic solvents, followed by heat treatment. In the current study, the effects of factors, such as solvent to (SBE) ratio [1:1–5:1], temperature [20–40 °C], and stirring time [30–60 min] on the efficiency of extracted oil were investigated by maceration method. Characterization analyses (SEM, XRD, XRFA and TGA) were carried out to compare the characteristics of samples. The best oil extraction efficiency was obtained at the highest level of solvent to (SBE) ratio (M_R = 5) at 30 °C temperature and at the 45 minutes stirring time this condition led to 72.82% oil extraction yield. The corn oil bleaching efficiency using the SBE treated at optimal condition and heated at 400 °C was improved to 84.75%.

Keywords: bleaching process, spent bleaching earth (SBE), soil contamination, regeneration.

✉Corresponding author. E-mail: *ikhlasbkd@gmail.com*

Notations

A_{445} – absorbance of sample at 445 nm;
E – error (%);
CCO – crude corn oil;
E_B – bleaching efficiency;
E_{EM} – efficiency of oil extraction by maceration (%);
EPM – experience planning methodology;
FCBE – fresh commercial bleaching earth;
M_R – mass ratio (L/S);
O_c – oil bleached by FCBE;
O_n – neutralized corn oil;
O_{op} – oil bleached by RSBE at optimal maceration condition;
O_{op1} – oil bleached by RSBEH at 300 °C;
O_{op2} – oil bleached by RSBEH at 400 °C;
O_{op3} – oil bleached by RSBEH at 500 °C;
RSBE – regenerated spent bleaching earth by hexane extraction;
RSBEH – regenerated spent bleaching earth by hexane extraction followed by heat treatment;
SBE – spent bleaching earth;
SEM – scanning electron microscopy;
T_{ca} – carotene content;
TGA – thermogravimetry analysis;
XRD – X-ray diffraction;
XRFA – X-ray fluorescence analysis.

1. Introduction

Vegetable oils are a highly edible source of food (Food and Agriculture Organization, 1994). In 2019, the crude vegetable oils world consumption reached 174 million tons (Lettre de L'ONAGRI, 2020). The crude corn oil (CCO) is generally processed by the refining (Barrera-Arellano et al., 2018). Four major steps, namely, degumming, neutralization, bleaching and deodorization, are involved in chemical refining of CCO (De Greyt, 2013; Chew & Nyam, 2019). Bleaching is an important step in the refinery process (Zio et al., 2020). The purpose of this step is to adsorb impurities which include pigments (β-carotenoids and chlorophyll) (Sabah et al., 2007), phosphatides, gums and metals trace (Gharby, 2022). This enables for producing a light colored and stable oil acceptable to consumers. In the industry the bleaching step is realized by using fresh commercial bleaching earth (FCBE). It is an activated clay belongs to smectite group that mostly clay mineral. After bleaching, the process of oil separation performed by filtration of the spent bleaching earth (SBE), the quantities of SBE in Tunisia are estimated at 1000 tons per year. This solid waste is currently disposed in landfills without any appropriate treatment and causes severe environmental problem, its storage in the open air could cause spontaneous combustion (Low et al., 2022). Currently, many studies are devoted to regenerate SBE (Abdelbasir et al., 2023). Among these ways, solvent extraction can be pointed out as one of the process carried out in this for removing the oil content (Foletto et al., 2002; Hatami et al., 2018; Merikhy et al., 2019; Shahi et al., 2015), acid activation treatment was also studied (Eko Saputro et al., 2020; Sariğolan et al., 2010).

In addition to these methods, others used the solvent extraction followed by thermal treatment (Abd Wafti et al., 2011; Pujiastuti et al., 2022), thermal activation (Bachmann et al., 2020; Soetaredjo et al., 2021), pyrolysis (Naser et al., 2021).

The main objectives of this study are the regeneration and the recycling of SBE in the corn oil bleaching step to minimize the environmental pollution impacts of these wastes. Regenerated (SBE) must meet the quality requirements of FCBE used in industry for the bleaching of the neutralized corn oil.

2. Materials and methods

In this study, a FCBE, is a fine white powder it was used as standard sample (FCBE), the SBE obtained after bleaching of corn oil as (SBE) is dark brown oily paste. The SBE samples were collected at Oil Refinery in Tunisia and regenerated by solvent extraction followed by heating process (RSBE). Samples were characterized by X-ray diffraction (XRD), gravimetric thermal analysis (TGA), X-ray fluorescence analysis (XRFA) and scanning electron microscopy (SEM). X-ray diffractogram was operated at 40 kV and 20 mA using radiation Cu-Kα (λ = 1.5405Å), at a scanning speed of 1° (2θ)/min. Chemical composition was determined by X-ray fluorescence with a spectrometer. The thermogravimetric analysis (TGA) of FCBE, RSBE and RSBEH was performed from room temperature to 900 °C with a heating rate of 10 °C/min, under an atmospheric air flow of 35 ml/min. The surface morphology and elemental analysis of SBE and RSBEH were examined by SEM (FEI Q250/EDAX thermos Fisher, at 10kx) with acceleration tension of 25 kv. The samples were sputter coated with a conductive layer of gold using a sample metallizer (Ion SPUTTER JFC-1100) before SEM analysis.

Step 1: Extraction by Soxhlet

In order to evaluate the percent of total oil content (PEO) in SBE and select the best solvent different samples of dry SBE rich on corn oil were extracted using Soxhlet method by testing different organics solvents (Hexane, Methyl isobutyl ketone (MIBK), methyl ethyl ketone (MEK) and Xylene). Typically, 20 g of dry SBE was weighed into the thimble to be extracted by solvents. The System was placed on heating balloon for 6 hours. Boiling is stabilized by glass beads. Then, the oil was separated from the solvent by a rotary vacuum evaporator, the temperature of the water bath increased to 70 °C to remove any possible moisture. Finally, the cooled samples were weighed, and the results were compared. The percent of total oil content (*PEO*) for each sample was calculated by the following Equation (1).

$$PEO = \frac{W_s}{W_a} \times 100, \qquad (1)$$

where W_s is the weight of oil extracted by the Soxhlet extractor (g), W_a is the weight of the dry bleaching earth sample (g), *PEO* is considered as the percentage of total oil content in SBE.

Step 2: Extraction by maceration

The maceration method to be extrapolated as reliable industrial process is carried out with dry SBE by adopting the experience planning methodology (EPM), the dry SBE sample was mixed with hexane as the best solvent, the liquid- solid ratio was varied from 1:1 to 5:1, the stirring time was varied in the interval 30 to 60 min at the temperature varying from 20 to 40 °C. Then the solution was filtered (Figure 1), air-dried. Then, the deoiled SBE was treated at different temperatures 300 °C, 400 °C and 500 °C for 30 min (Figure 2).

Figure 1. Extracted oil

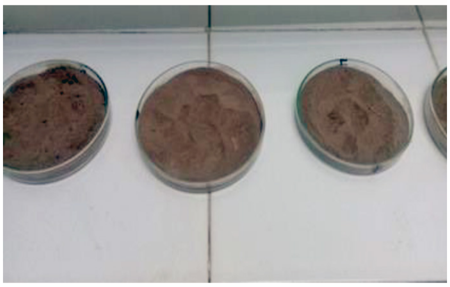

Figure 2. Regenerated bleaching earth (RSBE)

Step 3: Bleaching test

The samples of regenerated bleaching earth (RSBE) produced in this study were tested for their efficacy in bleaching neutralized corn oil. The first series of tests was made by adding 1 g of RSBE to 100 g of neutralized corn oil, the second series of bleaching test was made using thermal treated (RSBEH) at different temperature. Then the mixture was stirred mechanically for 40 min heated to 100 °C (Naser et al., 2021). Eventually, after mixing the samples and oil the separation was made by filtration. A sample of FCBE was also used for a comparison.

3. Results and discussion

3.1. Physicals and chemicals characterization of samples

3.1.1. X-ray diffraction (XRD)

The mineralogical composition evolution of FCBE, RSBE and RSBEH sample is illustrated by Figure 3. XRD analysis indicates that the samples have almost the same composition. They are composed of Quartz (SiO_2) and Calcite (Ca (CO_3)) which are the basic elements of montmorillonite ($Al_2O_3.4SiO_2.H_2O$). XRD of commercial sample presents the 001 reflection with low intensity. The diffractogram of RSBE show the presence of a more intense peak, corresponding to the Quartz. The clay fraction of our materials consists of Quartz and Calcite as a major compound in our samples, this confirms the results of Fluorescence X which show high proportions of SiO_2 (Quartz).

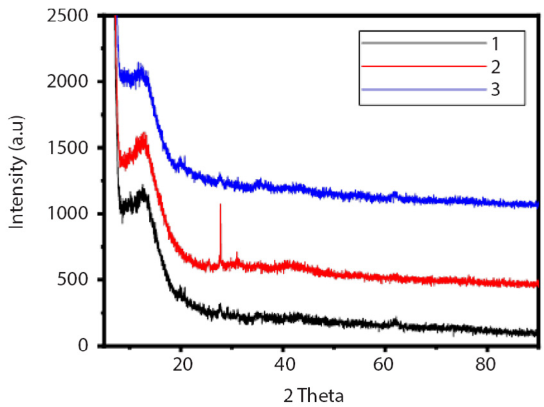

Figure 3. XRD diffractograms of (1: FCBE), (2: RSBE) and (3: RSBEH)

3.1.2. TGA analysis

The results of the TGA of FCBE, RSBE and RSBEH are shown in Figure 4, performed under oxygen atmosphere. The TGA thermograms of three samples showed two zones of weight loss. The first mass loss between [40–200 °C] can attributed to the loss of adsorbed water in the interlamellar clay layer which occurs throughout the process and any volatile matter. The second weight loss can also be associated to the removal of structural water and start of the carbonized matter decomposition.

3.1.3. X-ray fluorescence analysis (XRFA)

Table 1 shows the chemical composition by XRFA of FCBE, RSBE and RSBEH. The main chemical component of these samples is Silicon dioxide (SiO_2) followed by Al_2O_3, igniting loss MgO, Fe_2O_3 and CaO.

Table 1. The chemical element of FCBE, RSBE and RSBEH

Characteristics	FCBE (%)	RSBE (%)	RSBEH (%)
SiO_2	73.78	70.79	72.42
Al_2O_3	7.97	7.37	7.64
Fe_2O_3	2,48	2.37	2.55
MnO	0.012	0.010	0.014
MgO	1.7	1.13	1.27
K_2O	1.02	1.01	0.82
CaO	2.09	2.08	2.28
Na_2O	0.97	0.96	0.98
TiO_2	0.45	0.42	0.46
P_2O_5	0.71	1.04	0.66
Loss on ignition	8.82	12.82	10.9

These results show that the chemical composition is relative to the typical smectite clay. The solvent extraction process doesn't have significant effects on the major chemical element composition. Indeed, there is an increase of the loss on ignition in the case of RSBE.

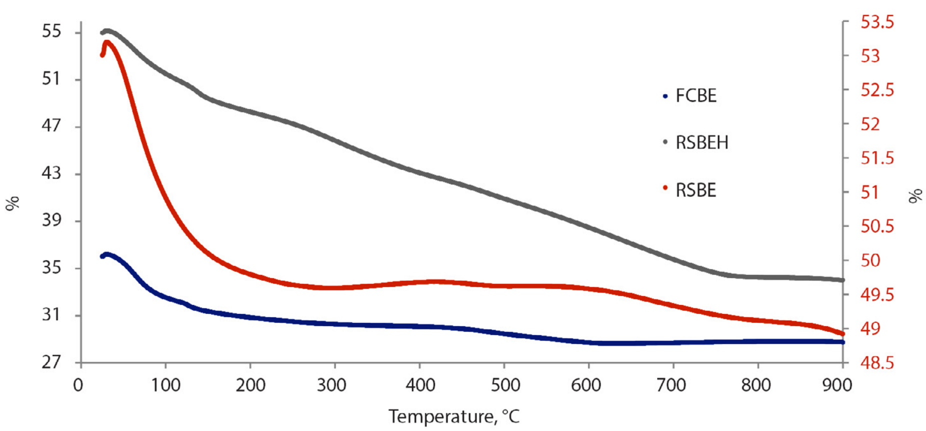

Figure 4. TGA analysis of FCBE, RSBE and RSBEH

3.1.4. Surface morphology and elemental content (SEM)

The SEM results for SBE and RSBE are shown in Figure 5, the surface of SBE particles is covered with oil and organic compounds. The SEM micrographs of SBE and RSBE show almost similar morphological characteristics with exfoliated or flake-like appearance and massive agglomerate of irregular shapes associated with montmorillonite clay.

The results of EDX study (Figure 6) indicated that all the samples are Ca-montmorillonite type and contain a small quantity of impurities. High amounts of carbon in SBE are related to the adsorbed organic compounds and oil on the BE during the bleaching process. After the solvent extraction and thermal treatment, oil and the other adsorbed materials were removed.

The EDX spectra (Figure 6) and the elemental composition (Table 2) confirm the presence of major chemical

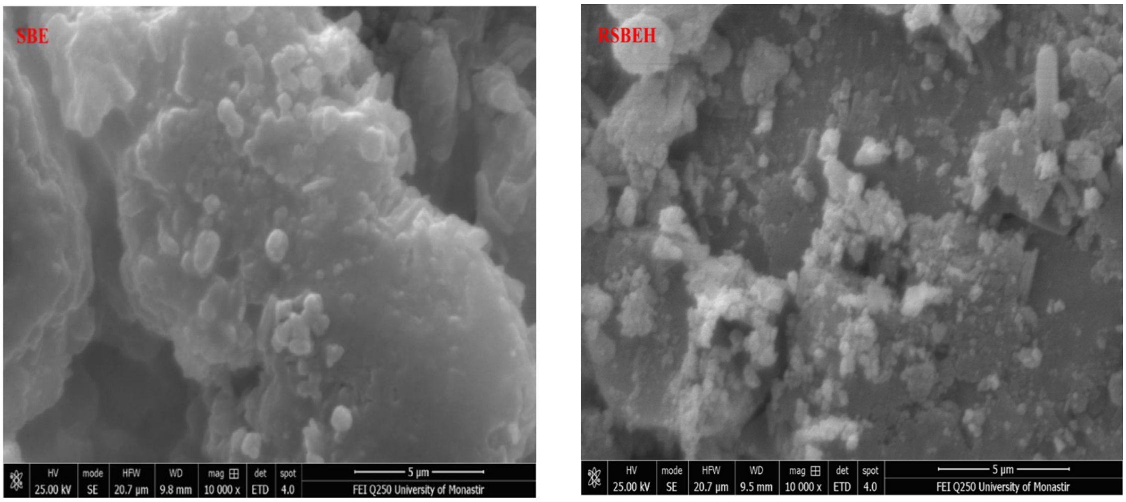

Figure 5. SEM images of SBE and RSBEH

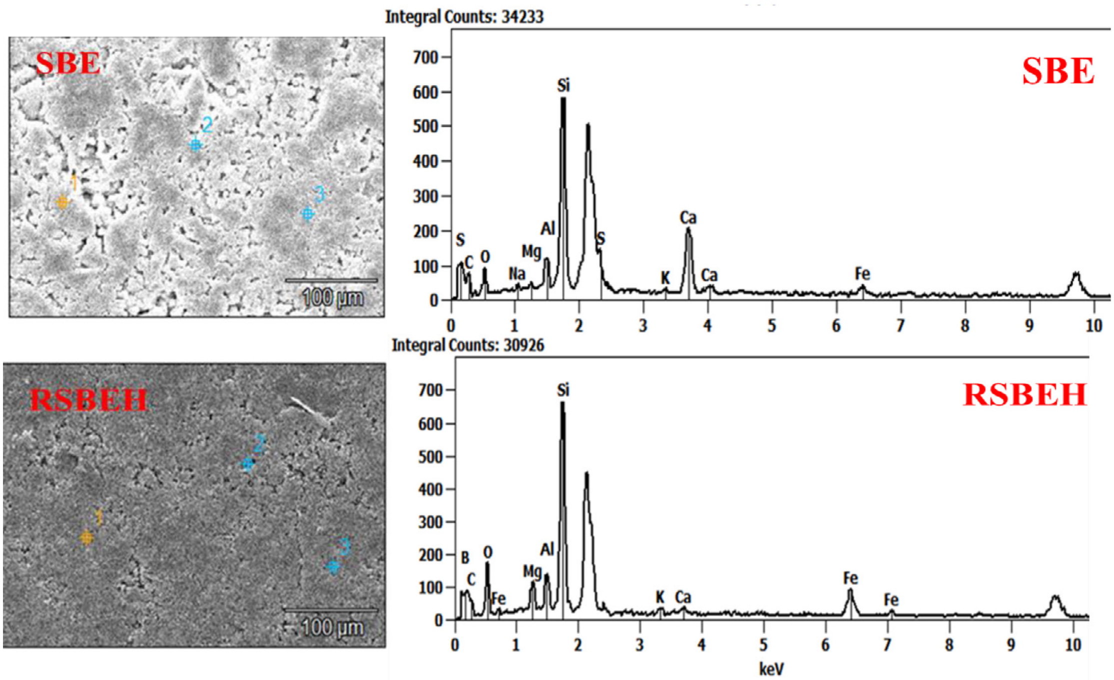

Figure 6. EDX analysis of SBE and RSBEH

Table 2. Elemental composition (%) using EDX analysis of SBE and RSBEH

	B	C	O	Na	Mg	Al	Si	S	K	Ca	Fe	Au
SBE	–	15.65	15.57	0.70	0.60	2.47	16.16	5.20	0.45	9.33	2.22	31.64
RSBEH	47.00	8.78	22.46	–	1.93	1.93	12.69	–	0.43	0.44	4.34	–

elements common to the structure of SBE and RSBEH. Thereby, the high amounts of Si, Al, Fe, C and O in SBE and RSBEH, respectively were related to the presence of SiO_2, Al_2O_3 and Fe_2O_3. The presence of C element in SBE indicates that there is an organic compound as residual oil which is adsorbed during bleaching process.

3.2. Effect of Solvent Type on the extraction efficiency

The results of comparative of the total PEO by the different solvents are Hexane (36%), MIBK (28.9%), MEK (30.1%) and Xylene (32.1%).

These results shows that the maximum PEO it is observed in the case of the samples treated by hexane as solvent, so the highest efficiency is reached with apolar solvent.

3.3. Design of experiments by maceration method

Hexane was selected as solvent to compose the matrix of extraction experiments by maceration method. **Experiment Planning Methodology (EPM)** is an analytic systematic approach based on mathematical modeling used to solve and analyze experimental results with the goal of optimizing processes. It allows for the identification of significant variables and their interactions with fewer experimental trials, thus saving time and resources. EPM is particularly useful when dealing with multiple variables, as it helps to find the optimal conditions by modeling the relationship between variables. This EPM was used in order to reduce the number of experiments and generate a regression equation which links all optimized variable parameters such as mass ratio (M_R), stirring time (t) and temperature (T) with extraction yield (Y_{th}%). The total number of the experiment is determined by the Equation (2) (Kafarov, 1974).

$$N = 2^K + 2 \times K + n_0. \tag{2}$$

In which: n_0 is the number of tests to be carried in the center of plan, K is the independent variable number. The adopted experimental method is an orthogonal central composite planning which requires in this case 14 experiments in addition to 4 tests in center. The variable parameters to be optimized are the liquid to mass ratio varying in the intervals of [1:1–5:1], stirring time [30–60 min] and temperature [20–40 °C].

The results obtained from 18 experiments were used as yields responses in EPM analysis to achieve the optimization process. The response was defined as a second-order polynomial Equation (3):

$$Y_{th} = a_0 + \sum_{i=1}^{k} a_i X_i + \sum_{i=1}^{k} a_{ii} X_i^2 + \sum_{i}^{k}\sum_{j}^{k} a_{ij} X_i X_j, \tag{3}$$

where a_0 is the constant term of the regression equation, a_i ($i \in \{1,2,3\}$), a_{ii} ($i \in \{1,2,3\}$) and a_{ij} ($i, j \in \{1,2,3\}$) are the coefficients of linear, quadratic and interaction effects, respectively. X_i is the coded value of the variable i and k is the number of variables. The matrix values, experimental variables and their responses are presented in Table 3.

The Equation (3) was developed for 3 variables:

$$Y_{th} = a_0 + \left[a_1 X_1 + a_2 X_2 + a_3 X_3\right] + \left[a_{11} X_1^2 + a_{22} X_2^2 + a_{33} X_3^2\right] + \left[a_{12} X_1 X_2 + a_{13} X_1 X_3 + a_{23} X_2 X_3\right]. \tag{4}$$

Y_{exp} is the response from the experiment was calculated by the following Equation (5):

$$Y_{exp} = E_{EM}(\%) = \frac{W_e \times 100}{W_s}, \tag{5}$$

where W_s is the weights of the oil extracted from the Soxhlet extractor in th case of maximum (*PEO*), and W_e are the weight of the oil extracted from the maceration extraction experiment.

All coefficient (a_0, a_i, a_{ij} and a_{ii}) in the mathematical model (Equation (3)) with (k) independent variables undergo validation by Student's tests (Kafarov, 1974; Goupy, 2006). the coefficients are determined by the following matrix calculation:

$$a_i \frac{\sum_{j=1}^{N} X_{ij} \times Y_{exp}^{j}}{\sum_{j=1}^{N} X_{ij}^2}; \quad a_{ij} = \frac{\sum_{k=1}^{N} \left(X_i \times X_j\right) \times Y_{exp}^{k}}{\sum_{k=1}^{N} (X_i^2 \times X_j^2)_k};$$

$$a_{ii} = \frac{\sum_{j=1}^{N} X_{ij}^2 \times Y_{exp}^{j}}{\sum_{j=1}^{N} \left(X_{ij}\right)^2}. \tag{6}$$

Testing and validation

Once we determine all the regression coefficients of the mathematical model, we then carry out tests to check whether the model is adequate.

Student test

After calculating the different coefficients of the regression equation, we calculate their respective variances. We then note:

$$S_{rep}^2 = \frac{\sum_{i=1}^{4} \left(Y_i^0 - Y_m^0\right)^2}{f_{rep}} : \text{Reproducibility variance.} \tag{7}$$

With Y_i^0: i value of test at the center of the Excperimental plan

$$Y_m^0 = \frac{\sum_{i=1}^{4} Y_i^0}{4} : \text{Average value of experimental test at the center of the plan.} \tag{8}$$

And $f_{rep} = 4 - 1 = 3$: The experimental test number at the center of the plan – 1.

Variance of each coefficient

$$S_{ai}^2 = \frac{S_{rep}^2}{\sum_{j=1}^{N}\left(X_{ij}\right)^2};\; S_{aij}^2 = \frac{S_{rep}^2}{\sum_{k=1}^{N}\left(X_i^2 X_j^2\right)_k};$$
$$S_{ajj}^2 = \frac{S_{rep}^2}{\sum_{j=1}^{N}\left(X_{ij}^{'}\right)^2}. \qquad (9)$$

Calculation of Student test

$$t_i = \frac{|a_i|}{S_{ai}};\quad t_{ij} = \frac{|a_{ij}|}{S_{aij}};\quad t_{jj} = \frac{|a_{jj}|}{S_{ajj}}. \qquad (10)$$

Student tests are carried out to verify each coefficient. The calculated Student test values must be greater than the tabulated values for these coefficients to be significant. Otherwise, when we obtain for (*a*) coefficient $ts_{caculated} < ts_{tab}$, it will be eliminated from the regression equation and taken as being equal to zero (Goupy, 2006).

Table 3. The coded values, experimental variables and their responses

Essai	Variables						Response
	X_1	M_R	X_2	t	X_3	T	
	Coded	Value	Coded	Value	Coded	Value	Y_{exp}%
1	1	5	1	60	1	40	71.71
2	–1	1	1	60	1	40	51.69
3	1	5	–1	30	1	40	58.93
4	–1	1	–1	30	1	40	47.51
5	1	5	1	60	–1	20	78.2
6	–1	1	1	60	–1	20	47.38
7	1	5	–1	30	–1	20	65.4
8	–1	1	–1	30	–1	20	44.58
9	0	3	0	45	0	30	55.23
10	1.215	5.43	0	45	0	30	68.51
11	–1.215	0.57	0	45	0	30	39.89
12	0	3	1.215	63.225	0	30	57.33
13	0	3	–1.215	26.775	0	30	47.69
14	0	3	0	45	1.215	42.15	78.85
15	0	3	0	45	–1.215	17.85	58.32
16	0	3	0	45	0	30	57.06
17	0	3	0	45	0	30	54.1
18	0	3	0	45	0	30	58.54

The matrix of experiments is the principal support of study. Indeed, 18 experiments were conducted, the regression Equation (11) obtained is below:

$$Y_{th} = 26.52 + 18.14\,X_1 + 6.81\,X_2 + 2.96\,X_3 + 3.83\,X_2^2 + 7.75\,X_3^2 + 3.35\,X_1X_2 + 3.64\,X_1X_3. \qquad (11)$$

In order to maximize the extraction efficiency (Y_{th}%) the method of Newton was applied (Collombier, 1995). This method generates the optimal conditions of extraction by maceration process. The values of each optimized operating parameters (M_R, t, T) are M_R = 5, t = 45 min and T = 30 °C. In these optimal conditions the extraction efficiency calculated by the regression Equation (10) is Y_{th} = 72.82% and the value obtained experimentally at optimized parameters is Y_{exp} = 71.02%. These two values are considered similar and comparable.

The deviation or Error between experimental and theoretical results at the optimal condition (Y_{th} = 72.82%, Y_{exp} = 71.02%) has been calculated by the following Equation (12) (E = 2.47%) this value show that the model feet the experimental results very well.

$$E = \left|\frac{Y_{exp} - Y_{th}}{Y_{th}}\right| \times 100. \qquad (12)$$

This allows us to conclude that the mathematical model of equation 10 simulates our experiments very well, the experimental data were fitted to a second-order polynomial model (Equation (10)), as linear, quadratic and interaction components have a significant effect on the response (validated by tabulated Student's tests) (Goupy, 2006).

So, we note that: all the first order terms exist, this confirms that mass ratio, stirring time and Temperature act directly on the extraction efficiency.

The terms of the second order (X_2^2, X_3^2) of the regression equations exist; this explains that the optimum is in the fixed intervals. Other interactions coefficients exist (X_1X_2) and (X_1X_3): These is explained there is an interaction between the mass ratio and stirring time; mass ratio and temperature.

3.4. Bleaching efficiency of neutralized corn oils by different samples

The RSBE produced must meet the FCBE standard in terms of bleaching efficiency. To assess this, a bleaching test was conducted on neutralized corn oil and oil samples treated under optimal conditions using RSBE, RSBEH and FCBE samples.

3.5. Bleaching efficiency

The results in Table 4 show the bleaching efficiency which was evaluated by the retention of carotene content in neutralized and bleached corn oil using different bleaching earth simples.

The carotene content was determinate by spectrophotometry UV method at 445 nm wavelength (Franke et al., 2010). Carotenoid content was calculated according to the following equation:

$$T_{ca}\ (\text{mg/100g}) = \frac{A.y(\text{ml}).10^6}{A_{1cm}^{\%}.1000.g}, \qquad (13)$$

where T_{ca} is the carotenoid content (mg/100 g), A the highest absorbency value at 445 nm, y the quantity of extracting solution (ml), g the weight of sample (g), $A_{1cm}^{\%}$ is the average absorption coefficient 2500 of carotenoid molecule.

The bleaching efficiency was calculated as:

$$E_B\left(\%\right)=\frac{\left(T_{ca}\ \textit{neutralized oil}-T_{ca}\ \textit{bleached oil}\right)\times 100}{T_{ca}\ \textit{neutralized oil}}. \quad (14)$$

Table 4. Result of carotene content and bleaching efficiency

Reference of bleached oils	A_{445} (nm)	T_{ca} (mg/100g)	E_B (%)
O_n	1.02	1.68	
O_c	0.29	0.49	70.80
O_{op}	0.68	1.12	34.01
O_{op1}	0.27	0.46	72.52
O_{op2}	0.16	0.24	84.75
O_{op3}	0.17	0.28	83.56

Based on result shows that the bleaching efficiency was maximum in the case of oil simple (O_{op2}) bleached by (RSBE) solvent extracted followed by thermal treatment at 400 °C.

It can be also seen that the optimal regeneration temperature was 400 °C for RSBE. In addition, the RSBEH used showed the following order in term of the oil bleaching efficiency:

$E_{B\ (O_{op2})} > E_{B\ (O_{op3})} > E_{B\ (O_{op1})} > E_{B\ (O_c)} > E_{B\ (O_{op})}$.

According to the results above, the heat treatment after extraction with hexane has improved the bleaching efficiency from 34% to 84.75%. This indicates that the thermal treatment at 400 °C was enhance the bleaching efficiency on 50%. Moreover, RSBEH become more effective than the FCBE.

3.6. Analysis of the color by Lovibond

The color of neutralized corn oil before and after bleaching using different Bleaching simples was made according to the AGRIS method (Organisation Internationale de Normalisation, 2010). The results shown in Table 5 demonstrates the link between the regeneration efficiency of RSBE and the oil color.

Table 5. Mean color of bleached and neutralized corn oil

Color	O_n	O_c	O_{op}	O_{op2}
Red (R)	1.4	5.2	12.2	9.2
Yellow (Y)	0.7	68.5	57	69

These results show also a significantly higher Lovibond color (Y = 69) in the case of (O_{op2}) oil simple. This oil simple bleached by (RSBE) thermal treatment at 400 °C show the value of the yellow color index of the bleached oil increased as well from 0.7 to 69. These results agree with those obtained in Table 4.

4. Conclusions

In view of the results and analysis, the following conclusions were drawn:

- The regeneration process doesn't have significant effects on the structure and chemical composition of treated bleaching earth this was shown by results of different analysis (XRD. TGA. XRFA and SEM).
- The total (PEO) by soxhlet for different solvents used showed the following order:
- PEO (Hexane) > PEO(Xylene) >PEO(MEK) > PEO (MIBK).
- The corn oil bleaching efficiency using only RSBE is not satisfied, so to enhance the oil bleaching efficiency the regenerated SBE by maceration extraction at optimal condition was heat treated at 400 °C.
- The corn oil bleaching efficiency using (RSBEH) thermal treated at optimum condition has become comparable as in the case of FCBE. These results agree with those obtained in the case of Lovibond color analysis.

Acknowledgements

The authors are grateful to the Research Center of Technology and Energy (CRTEn) Borj Cedria – Tunisia for the assistance to complete the XRD Analysis. This work was supported by the Ministry of Higher Education and Scientific Research, Tunisia.

Funding

The authors declare that no funds. Grants were received during the preparation of this manuscript.

Authors' contributions

Lassaad Ben Mansour contributed to conceptualization and supervision. Ikhlas Benkhoud contributed to experimental work, writing original draft preparation and writing review and editing. All authors have read and agreed to the published version of the manuscript.

Conflict of interest

The authors declare no conflict of interest.

References

Abd Wafti, N. S., Kien Yoo, C., Wai Lin, S., Choong, T., & Luqman Chuah, A. (2011). Regeneration and characterization of spent bleaching clay. *Journal of Oil Palm Research*, *23*(1), 999–1004. https://www.researchgate.net/publication/269826084

Abdelbasir, S. M., Shehab, A. I., & Khalek, M. A. A. (2023). Spent bleaching earth; recycling and utilization techniques: A review. *Resources, Conservation & Recycling Advances*, *17*, Article 200124. https://doi.org/10.1016/j.rcradv.2022.200124

Bachmann, S. A. L., Valle, R. D. C. S. C., Vegini, A. A., & Tavares, L. B. B. (2020). Determination of optimum conditions for thermal regeneration and characterization of a spent bleaching earth. *Journal of Environmental Chemical Engineering*, *8*(2), Article 103503. https://doi.org/10.1016/j.jece.2019.103503

Barrera-Arellano, D., Badan-Ribeiro, A. P., & Serna-Saldivar, S. O. (2018). Corn oil: Composition, processing, and utilization. In *Corn: Chemistry and technology* (3rd ed., pp. 593–613). Elsevier. https://doi.org/10.1016/B978-0-12-811971-6.00021-8

Chew, S. C., & Nyam, K. L. (2019). Refining of edible oils. In *Lipids and edible oils: Properties, processing, and applications* (pp. 213–241). Elsevier. https://doi.org/10.1016/B978-0-12-817105-9.00006-9

Collombier, D. (1995). *Plans d'expérience factoriels: construction et propriétés des fractions de plans* [Factorial experimental designs: Construction and properties of design fractions] (Vol. 21). Springer Science & Business Media.

De Greyt, W. (2013). *Edible oil refining: Current and future technologies*. Springer Nature. https://doi.org/10.1002/9781118535202.ch5

Eko Saputro, K., Siswanti, P., Wahyu Nugroho, D., Ikono, R., Noviyanto, A., & Taufiqu Rochman, N. (2020). Reactivating adsorption capacities of spent bleaching earth for using in crude palm oil industry. *IOP Conference Series: Materials Science and Engineering*, *924*(1), Article 012014. https://doi.org/10.1088/1757-899X/924/1/012014

Foletto, E. L., Alves, C. C. A., Sganzerla, L. R., & Porto, L. M. (2002). Regeneration and utilization of spent bleaching clay. *Latin American Applied Research*, *32*(2), 205–208.

Food and Agriculture Organization. (1994). Processing and refining edible oils. In *Fats and oils in human nutrition* (pp. 33–43). https://www.fao.org/4/v4700e/v4700e00.htm

Franke, S., Fröhlich, K., Werner, S., Böhm, V., & Schöne, F. (2010). Analysis of carotenoids and vitamin E in selected oilseeds, press cakes and oils. *European Journal of Lipid Science and Technology*, *112*(10), 1122–1129. https://doi.org/10.1002/ejlt.200900251

Gharby, S. (2022). Refining vegetable oils: Chemical and physical refining. *The Scientific World Journal*, *2022*(1), Article 6627013. https://doi.org/10.1155/2022/6627013

Goupy, J. (2006). *Plans d'expériences* [Design of experiments]. Ed. Techniques Ingénieur.

Hatami, A. M., Sabour, M. R., & Amiri, A. (2018). Recycling process of spent bleaching clay: Optimization by response surface methodology. *Global Journal of Environmental Science and Management*, *4*(1), 9–18.

Kafarov, V. (1974). *Méthodes cybernétiques et technologie chimique* [Cybernetic methods and chemical technology]. Éditions Mir Moscou.

Lettre de L'ONAGRI. (2020). *Observatoire Nationale de l'Agriculture* [National Observatory of Agriculture]. http://www.onagri.nat.tn/uploads/lettre/lettre-tri-1-2020-M.pdf

Low, A., Shamsuddin, R., & Siyal, A. A. (2022). Economic analysis of waste minimisation and energy recovery from spent bleaching earth. *Cleaner Engineering and Technology*, *7*, Article 100418. https://doi.org/10.1016/j.clet.2022.100418

Merikhy, A., Heydari, A., Eskandari, H., & Nematollahzadeh, A. (2019). Revalorization of spent bleaching earth a waste from vegetable oil refinery plant by an efficient solvent extraction system. *Waste and Biomass Valorization*, *10*(10), 3045–3055. https://doi.org/10.1007/s12649-018-0311-0

Naser, J., Avbenake, O. P., Dabai, F. N., & Jibril, B. Y. (2021). Regeneration of spent bleaching earth and conversion of recovered oil to biodiesel. *Waste Management*, *126*, 258–265. https://doi.org/10.1016/j.wasman.2021.03.024

Organisation Internationale de Normalisation. (2010). *Corps gras d'origines animale et vegetale – Determination de la couleur Lovibond – Methode automatique* [Animal and vegetable fats and oils – Determination of Lovibond colour – Automatic method] (ISO Norme No. 27608:2010). https://www.iso.org/obp/ui/#iso:std:iso:27608:ed-1:v1:fr

Pujiastuti, C., Sumada, K., Armidianti, M., Rimarsya, A., & Ahmad, A. R. B. (2022). Bleaching earth recovery from waste to purify cooking oil by extraction-activation method. *Journal of Research and Technology*, *8*(2), 169–177. https://doi.org/10.55732/jrt.v8i2.401

Sabah, E., Çinar, M., & Çelik, M. S. (2007). Decolorization of vegetable oils: Adsorption mechanism of β-carotene on acid-activated sepiolite. *Food Chemistry*, *100*(4), 1661–1668. https://doi.org/10.1016/j.foodchem.2005.12.052

Sariğolan, Ş., Yuzer, H., & Koral, M. (2010). Acid activation and bleaching performance of Turkish (somas) bentonite in crude soybean oil. *Particulate Science and Technology*, *28*(4), 298–308. https://doi.org/10.1080/02726351.2010.496296

Shahi, M., Sabour, M. R. Amiri, A., & Ghasemnezhad, M. (2015). Cleaning spent bleaching clay using solvent extraction method and RSM statistical approach. *Cumhuriyet University Faculty of Science Journal (CSJ)*, *36*(7), 23–40. https://doi.org/10.17776/csj.66920

Soetaredjo, F. E., Laysandra, L., Putro, J. N., Santoso, S. P., Angkawijaya, A. E., Yuliana, M., Ju, Y.-H., Zhou, C.-H., & Ismadji, S. (2021). Ecological-safe and low-cost activated-bleaching earth: Preparation, characteristics, bleaching performance, and scale-up production. *Journal of Cleaner Production*, *279*, Article 123793. https://doi.org/10.1016/j.jclepro.2020.123793

Zio, S., Cisse, H., Zongo, O., Guira, F., Tapsoba, F., Somda, N. S., Hama-Ba, F., Songre-Ouattara, L. T., Zongo, C., Traore, Y., & Savadogo, A. (2020). The oils refining process and contaminants in edible oils: A review. *Journal of Food Technology Research*, *7*(1), 9–47. https://doi.org/10.18488/journal.58.2020.71.9.47