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Scientific paper

Optimization of The Bleaching Process of Sunflower Oil

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Abstract

Bleaching is a crucial step in oil refining that removes unwanted pigments and oxidative products, which degrade oil quality. This study evaluated the effects of temperature (75–115 °C), adsorbent concentration (1–3 wt%), and time (20–40 minutes) on sunflower oil bleaching using a Box-Behnken factorial design in Minitab 21. Responses were divided into two groups: (I) peroxide value and spectroradiometric-based chroma and hue-angle, and (II) peroxide value and spectrophotometric-based bleaching efficiency. Under optimal conditions (approximately 78 °C, 2.95 wt% adsorbent, and 20 minutes), peroxide value decreased from 1.8 meq/kg to 0.398 meq/kg, chroma reduced from 45.89 to 9.61, hue-angle increased from 92.69 to 105.55, and bleaching efficiency reached 71.86%. Composite desirability was higher in the first group (0.97 vs 0.89), primarily due to the more objective nature of spectroradiometric parameters (hue-angle and chroma).

Keywords: Oil bleaching, Optimization, Response Surface Method, Sunflower oil

1. Introduction

Global consumption of sunflower oil exceeds 20 million tons per year; therefore, there is a growing demand for high-quality oil products.[1] Bleaching is a crucial step in the edible oil refining process, as it removes unwanted pigments and oxidation products that deteriorate the oil's quality.[2,3] Crude sunflower oil contains various components such as phospholipids, metals, phytosterols, carotene, tocopherols, and tocotrienols. As a result,, a refining process is necessary to transform crude oil into an edible product that is odorless, tasteless, and oxidatively stable, which is preferred by consumers.[5,6,7]

Adsorption processes are particularly important in refining because they effectively remove most of the contaminants found in crude edible oil.[8,9] Suitable adsorbents can sequester oxidation products, pigments, the trace metals, and residues of phospholipids and soaps, which greatly improve the oxidation stability and sensory quality of the oil.[10] The color improvement results from the removal of organic compounds such as carotenoids, particularly β-carotene and their derivatives, xanthophylls, chlorophylls, pheophytins, tocopherols, gossypol, and their degradation products. These compounds can contribute to an undesirable color to oil.[11]

In the process of removing unwanted pigments and oxidative compounds from neutralized oil, various adsorbents-such as bleaching earth, activated carbon, zeolites, silica gel, and activated alumina-are used due to their neutral effect on the beneficial properties of the oil.[12,13,14] Among these, bleaching earth is preferred because of its high adsorption capacity for color pigments and low initial cost.[15] In industrial conditions, edible oil is most often bleached with commercial bentonite or montmorillonite adsorbents (bleaching earth) in concentrations of 0.5–3.0 wt% of the oil mass, at temperatures between 90 °C and 120 °C and at a contact time of 15–60 min, depending on whether it is a batch or continuous flow.[12,15]

Bleaching earth physically adsorbs some pigments, while others are chemically bound via covalent or ionic bonds. The acidity of the bleaching earth correlates with its pigment adsorption capability. Acid-activated bleaching earth serves multiple functions, such as a solid catalyst, adsorbent, cation exchanger, and filter, whereas neutral bleaching earth primarily serves as an adsorbent.[16] In addition to the adsorption characteristic of the bleaching earth, temperature and contact time are also critical process parameters throughout the bleaching process.

Recently, Response Surface Methodology (RSM) has emerged as an effective tool for process optimization.[5,17] The main objective of the RSM is to identify optimal process conditions. The application of statistical design techniques can increase efficiency and bring output closer to nominal values, minimizing variation, modification time, and overall cost.[12,18] Digital tools such as real-time process monitoring and machine learning algorithms for predictive control are also being used to optimize processes.[19,20,21] For the bleaching of edible oils, ultrasonically assisted bleaching and microwave activation of adsorbents have been explored. In addition, nanostructured or modified bioadsorbents are used in continuous process, enabling faster diffusion of undesirable compounds and reducing adsorbent consumption while maintaining high oil quality.[22,23] Recently, waste shells, rich in calcium carbonate, have emerged as a promising low-cost biomaterial for neutralizing and potentially bleaching vegetable oils.[24]

The aim of this research is to optimize the process parameters in the sunflower oil bleaching process using a Box-Behnken factorial design. The effect of temperature, adsorbent concentration, and contact time on the oil's chroma, hue-angle, bleaching efficiency, and peroxide value will be evaluated. The responses are divided into two main assessment groups: (I) hue-angle, chroma and peroxide value, and (II) bleaching efficiency and peroxide value. Response variables in previous oil-bleaching studies have included chroma, hue angle and peroxide value, or bleaching efficiency and peroxide value, but none have directly compared these two groups. The primary goal of the study is to determine which group of responses give more reliable and objective results. The findings of this research have a practical application in the industry because they enable more rational use of adsorbents and energy, ultimately reducing production costs and improving the quality of the final product.

2. Experimental

2. 1. Materials

In this work, degummed and neutralized unrefined sunflower oil was used, which was obtained from the oil refinery "Bimal" d.d. Brčko (Bosnia and Herzegovina). To prevent unwanted oxidation prior, the oil was stored in a plastic bottle wrapped with aluminum foil and kept in a dark place. Commercial bleaching earth (Bimal, Brčko, Bosnia and Herzegovina) was used as an adsorbent for oil bleaching.

To enhance the adsorption capacity of the bleaching earth, a two-step pretreatment process was employed:

- **Sieving**: The bleaching earth was sieved using a 36 μm sieve to eliminate dust particles, which could form stable emulsions with oil.
- **Acid activation**: The sieved earth was treated with sulfuric acid (Gram-mol, Zagreb, Croatia) in a 1:3 ratio (w/w) for 3 hours at 85 °C under continuous stirring. The activation process was performed in a digester, which was a closed system, without evaporation, line with laboratory safety protocols. The goal of the acid activation was to increase the specific surface area and porosity of the adsorbent, thereby improving its adsorption properties.[15,25]

2. 2. Sunflower Oil Bleaching

The neutralized sunflower oil was initially heated to the desired temperature (Factor A, Table 1). The temperature was maintained at a constant level throughout the process using a magnetic stirrer (uniSTIRRER 3) with an integrated heater and automatic temperature controller (LLG Labware, Meckenheim, Germany). Following this, the acid-activated bleaching earth (Factor B) was added. A mixing speed of 450 min^{-1} was chosen based on preliminary tests and literature, as it ensured sufficient dispersion of the bleaching earth particles within the oil matrix without the formation of emulsions or phase separation.[26] Bleaching was carried out for the predeterminated contant time (Factor C), after which the mixture was cooled to room temperature. The oil was then separated from the residual bleaching earth using a Centric 322A centrifuge (Domel, Železniki, Slovenia) at 5000 rpm for 10 minutes. To further minimize oxidative degradation, the bleached oil was packaged in tightly sealed bottles lined with aluminum foil to reduce exposure to air (oxygen). To further limit oxidative degradation, storage was maintained at room temperature and sources of heat and light were avoided.

2. 3. Methods

The following methods were used to characterize the oil: determination of peroxide value, spectrophotometric determination of bleaching efficiency and spectroradiometric determination of hue-angle and chroma.

The peroxide value represents the primary oxidation status of the oil, and the standard method ISO 3960:2017 was used for its determination.[27] The method is based on the titration of oil sample, which was previously diluted with a mixture of acetic acid (Lachner, Neratovice, Czech Republic) and chloroform (Macron Fine Chemicals, Radnor, Pennsylvania, US), followed by the addition of potassium iodide (Gram-mol, Zagreb, Croatia). The liberated iodine was then titrated with a standardized sodium thiosulfate solution (Semikem, Sarajevo, Bosnia and Herzegovina). The results are expressed as milliequivalents per kg of oil (meq/kg).

The bleaching efficiency was determined following the method by Nwabanne and Ekwu.[12] The oil samples were first poured into a 10 mm cuvette and the absorbance was measured at a wavelength of 450 nm on a Shimadzu 1800 spectrophotometer (Agilient Technologies, Santa Clara, California, United States) with hexane as a blank. The sensitivity of the device was ±0.001 AU (absorbance unit), and the calibration was performed using certified reference standards in the UV-VIS range (Starna Scientific, Ilford, UK). The efficiency of bleaching was calculated according to the following formula:

$$Bleaching\ efficiency\,(\%) = \frac{(A_0 - A)}{A_0}\cdot 100 \quad (1)$$

where:
A_0 – absorbance of the raw, neutralized sunflower oil and
A – absorbance of bleached sunflower oil.

Spectroradiometry was used to measure the color of the samples. Compared to spectrophotometry, spectroradiometry captures the entire visible spectrum, making it more efficient for the analysis of cloudier samples and provides non-subjective measurements.[28] Oil analysis was performed in cuvettes (10 mm·10 mm·50 mm) using a Conica Minolta CM-5 (Conica Minolta, Tokyo, Japan), which measured the entire visible color spectrum. From the spectral measurements, the CIELAB color coordinates: a* and b*, and the psychometric light index L were derived. The parameter a* has a positive value for reddish samples and a negative value for greenish samples, while b* has a positive value for yellowish samples and a negative value for bluish samples. L is an estimate of relative brightness (luminosity) and according to this parameter, each color can be considered equivalent to a member of the grayscale, ranging from black (L = 0) to white (L = 100). The hue-angle (h) and chroma (C*) parameters can be derived from the values of a* and b* via the following equations, respectively:

$$h = \tan^{-1}\left(\frac{b^*}{a^*}\right) \quad (2)$$

$$C^* = \sqrt{\left(a^*\right)^2 + \left(b^*\right)^2} \quad (3)$$

Hue-angle is the value according to which colors are traditionally categorized into red, green, yellow and others. The chroma value is related to a quantitative attribute of a color and allows for each hue-angle to determine the degree of difference compared to gray color with the same lightness.

2. 4. Experimental Design

Experimental design and statistical analysis were performed in MINITAB 21 (software version 21.1.1.0) with the Response Surface Method (RSM).

A Box-Behnken experimental design (BBD), a form of Response Surface Method, was applied to determine the influence of three experimental factors: temperature (A), bleaching earth concentration (B), and contact time (C) on the output variables (Responses) (Table 1).

Table 1. Coded and actual levels of independent variables used in the RSM design for bleaching sunflower oil.

Symbol	Independent variables	Levels −1	Levels 0	Levels 1
A	Temperature [°C]	75	95	115
B	Bleaching earth concentration [wt%]	1	2	3
C	Time [min]	20	30	40

The Box-Behnken design was selected because it avoids extreme experimental conditions, unlike the Central Composite Design (CCD). The selected process parameters were chosen to resemble industrial conditions, so there was no need to include extremes as in CCD. Additionally, the BBD factorial design uses fewer experiments than the CCD, while still providing enough data to develop a reliable second-order polynomial model.[29] A second-order polynomial model was employed due to its ability to be validated using ANOVA analysis, allowing for an assessment of the data's reliability.[30] The main disadvantage of BBD compared to CCD is the existence of only a mid-level without axial points. This can lead to a high fitted R^2 but a low Predicted R^2 indicating that the model "remember" the sample but not the general curvature. Due to the limited number of experiments, extrapolating the results beyond the specific process parameters becomes problematic.[31] In this study, there were three experimental factors, each with three levels, resulting in 13 experimental runs. Two two replicates were carried out at the design center point to estimate pure error and to calculate the repeatability of the method. This allows validation of the model's adequacy of the model and separation of lack-of-fit from experimental error. As a results, a total of 15 experimental runs were performed.

The responses in this study were chroma, hue value, bleaching efficiency and peroxide value of the bleached oil.

The experimental data were fitted to a second-order polynomial model to obtain the regression coefficients. The generalized second-order polynomial model used in the response surface method is as follows:

$$Y = a_0 + \sum a_i X_i + \sum a_{ii} X_i^2 + \sum a_{ij} X_i X_j \quad (4)$$

where Y represents the experimental response, a_0 is a constant, a_i, a_{ii} and a_{ij} are coefficients of linear, quadratic and interactive regression models, and X_i and X_j are independent variables in coded values.

Lack of fit, coefficient of determination (R^2) and p-value obtained ANOVA were used to assess the adequacy of the developed model. Regression analysis and contour plots were generated to explain the effects of independent variables on responses. Finally, parameters were optimized using the RSM optimization procedure.

3. Results and Discusion

According to the Box-Behnken factorial design with three factors, 15 experimental runs were performed, and the output variables are shown in Table 2. The bleaching process not only affects the color removal but also contributes to the decomposition of unwanted oxidation products. Therefore, in addition to the chroma value, the hue-angle and bleaching efficiency (which are direct indicators of the degree of removal of colored pigments) and the peroxide number were used as the responses in this study.

The L, a*, and b* values of the starting, unbleached oil were 91.49, –2.15, and 45.84, respectively. These values indicate that the oil is yellowish in color, with a high concentration of carotenoids.[32] The value of L increased after bleaching, which resulted in the oil samples becoming lighter and slightly more transparent. As mentioned, more positive b* values meant the presence of yellow color in the sample. The parameter b* exhibited the greatest change, decreasing from starting value of 45.84 to a range of 8.94–14.22, depending on the process conditions. This decrease in the b* value of the samples with the addition of bleaching earth indicated that most of the carotenoid pigments (which are mostly yellow-brown in color) have been removed. The negative value of a* value for each sample indicated that the greenish color predominates in the samples.

To determine the influence of process parameters on product quality, ANOVA analysis and evaluation of the obtained models were performed.

The experimental data of each measured variable were fitted into a complete quadratic model. Polynomial coefficients for the response surface model were calculated through multiple regressions. An F-value and a p-value were calculated for each term in the regression model. The F-value represents the ratio between the variance explained by the model and the error variance – the higher it is, the better the model explains the data. On the other hand, the p-value shows how much chance the F-value of that size would have if there was no real effect. A confidence level of 95% was chosen and a p-value greater than 0.05 were not considered statistically significant. The Adjusted R^2 and Predicted R^2 were evaluated to determine whether the model was adequate after eliminating non-significant parameters, i.e. whether the model can accurately predict responses under different process conditions. Table 3 shows the ANOVA results for the response surface quadratic model for responses of bleached sunflower oil.

The R^2 values for chroma, hue-angle, bleaching efficiency and peroxide value in bleached oil were 0.993, 0.9789, 0.952 and 0.9701, respectively. These values indicate that the response variability was well explained in the generated model. The models could explain 99.3% of the variation in chroma, 97.89% of the variation in hue-angle, 95.52% of the variation in bleaching efficiency and 97.01% of the variation in peroxide value. The R^2 value for all four responses were close to unity, indicating a good correlation between the independent variables and the responses.

Adjusted R^2 is the corrected value for R^2 after eliminating non-significant terms in the model. Adjusted R^2 values for chroma, hue angle and peroxide value in bleached oil were 0.981, 0.941, and 0.916, respectively. These values

Table 2. Measured values for the response variables.

Number of rafination	Process condition Temp [°C]	Bleaching earth conc. [wt%]	Time [min]	L	Color a*	b*	Chroma (C*)	Hue-angle (h)	Bleaching efficiency [%]	Peroxide value [meq/kg]
Int. sample	–	–	–	91.49	–2.15	45.84	45.89	92.69	–	1.80
1	75	1	30	93.37	–2.46	10.62	10.90	103.03	68.27	1.30
2	115	1	30	94.02	–3.30	14.22	14.60	103.06	74.18	1.70
3	75	3	30	91.49	–2.72	9.90	10.27	105.37	61.71	0.50
4	115	3	30	91.22	–3.40	13.30	13.73	104.32	59.52	0.59
5	75	2	20	93.14	–2.32	8.94	9.24	104.55	72.43	0.60
6	115	2	20	92.51	–2.98	11.65	12.02	104.37	69.15	0.70
7	75	2	40	92.32	–2.50	9.58	9.90	104.62	68.27	0.50
8	115	2	40	91.64	–3.43	13.91	14.33	103.85	67.30	0.70
9	95	1	20	94.00	–2.68	11.36	11.67	103.26	69.37	1.30
10	95	3	20	92.67	–2.90	10.37	10.77	105.61	68.71	0.40
11	95	1	40	93.85	–2.75	11.84	12.16	103.05	76.81	0.90
12	95	3	40	90.37	–3.12	11.80	12.21	104.82	53.39	0.50
13	95	2	30	91.84	–2.81	10.94	11.29	104.41	64.99	0.52
14	95	2	30	90.16	–2.78	11.21	11.55	104.93	64.55	0.59
15	95	2	30	92.42	–2.93	11.16	11.53	104.69	65.86	0.60

Table 3. ANOVA results for the response surface quadratic model for all responses of bleached sunflower oil.

Source	DF[a]	Chroma		Hue angle		Bleaching efficiency		Peroxide value	
		F-Value[b]	P-Value[c]	F-Value	P-Value	F-Value	P-Value	F-Value	P-Value
Model	9	79.22	<0.0001	25.76	0.0011	11.01	0.0083	18.01	0.0027
Linear	3	213.62	<0.0001	65.78	0.0002	21.30	0.0028	38.70	0.0007
Temperature (A)	1	560.67	<0.0001	11.68	0.0189	0.73	0.4327	6.58	0.0503
Adsorbent conc. (B)	1	14.99	0.0117	179.35	<0.0001	54.07	0.0007	107.81	<0.0001
Time (C)	1	65.19	0.0005	6.33	0.0535	9.12	0.0294	1.70	0.2490
Square	3	17.37	0.0045	7.79	0.0248	1.38	0.3510	12.83	0.0088
AA	1	6.94	0.0463	7.20	0.0437	0.76	0.4246	7.12	0.0444
BB	1	31.24	0.0025	17.77	0.0084	0.00	0.9643	28.12	0.0032
CC	1	11.48	0.0195	0.18	0.6917	3.54	0.1184	2.90	0.1494
2-Way Interaction	3	6.67	0.0337	3.71	0.0959	10.36	0.0138	2.49	0.1749
AB	1	0.31	0.6001	7.02	0.0455	3.45	0.1222	1.90	0.2265
AC	1	14.78	0.0121	2.10	0.2074	0.31	0.6042	0.20	0.6706
BC	1	4.90	0.0777	2.02	0.2140	27.31	0.0034	5.37	0.0684
Lack-of-Fit	3	3.00	0.2600	0.36	0.7949	9.61	0.0957	8.05	0.1126
Coefficients of determinations		$R^2 = 0.993$; Adjusted $R^2 = 0.981$; Predicted $R^2 = 0.906$		$R^2 = 0.9789$; Adjusted $R^2 = 0.941$; Predicted $R^2 = 0.852$		$R^2 = 0.952$; Adjusted $R^2 = 0.866$; Predicted $R^2 = 0.274$		$R^2 = 0.970$; Adjusted $R^2 = 0.916$; Predicted $R^2 = 0.553$	

[a] – Degree of Freedom [b] – ANOVA test statistic (variance ratio) [c] – $p < 0.05$ indicates statistical significance.

were very close to the R^2 values, which meant that the proposed models remain valid after eliminating members whose p-value is greater than 0.05. The Adjusted R^2 value for bleaching efficiency was 0.866, slightly lower but still reliable enough that the abbreviated regression model can validly represent the responses.

Predicted R^2 is used to determine how well a regression model makes predictions. It is extremely high for chroma and hue angle (0.906 and 0.852), confirming valid predictions for new data. On the other hand, Predicted R^2 is significantly lower (0.274 and 0.553) for bleaching efficiency and peroxide value, which means that the model fits the original data, but the predictions are not accurate enough. This indicates that the model is complicated and begins to model noise in the data (a condition known as "overfitting the model").[33] This may be due to a small number of samples, an overly complex model, or the inclusion of variables that have no real impact.

Lack of fit can be used to confirm the validity of the model. By ANOVA analysis for lack-of-fit values of all responses, it was determined that the p-value was significantly higher than 0.05, which indicated that the models were adequately adapted to the experimental data.

3. 1. The Influence of Process Parameters on the Value of Chroma

Chroma refers to the degree of color purity. A color with a high chroma value does not have a significant presence of black, white or gray. A high chroma value means that the color contains little to no presence of black, white or gray, distinctly different from neutral gray at the given brightness.

Table 4 shows the coefficients of the regression equation and p-values for the terms in the proposed quadratic

Table 4. Regression coefficients and p-values for all responses.

Variables	Chroma		Hue angle		Bleaching efficiency		Peroxide value	
	Regression coeff.	p-Value	Regression coeff.	p-Value	Regression coeff.	p-Value	Regression coeff.	p-Value
Constant	14.14	<0.0001	90.31	<0.0001	65.70	<0.0001	5.20	0.0003
Temperature (A)	−0.1058	<0.0001	0.1720	0.0189	−0.208	0.4327	−0.0632	0.0503
Adsorbent conc. (B)	−3.218	0.0117	4.471	<0.0001	21.24	0.0007	−1.627	<0.0001
Time (C)	0.0448	0.0005	0.1077	0.0535	−0.089	0.0294	0.016	0.2490
AA	0.000735	0.0463	−0.000711	0.0437	0.00246	0.4246	0.000379	0.0444
BB	0.624	0.0025	−0.447	0.0084	−0.05	0.9643	0.3012	0.0032
CC	−0.00378	0.0195	−0.00045	0.6917	0.0213	0.1184	−0.000967	0.1494
AB	−0.003	0.6001	−0.0135	0.0455	−0.1012	0.1222	−0.00376	0.2265
AC	0.002063	0.0121	−0.000738	0.2074	−0.00301	0.6042	0.000123	0.6706
BC	0.0238	0.0777	−0.0145	0.2140	−0.569	0.0034	0.01264	0.0684

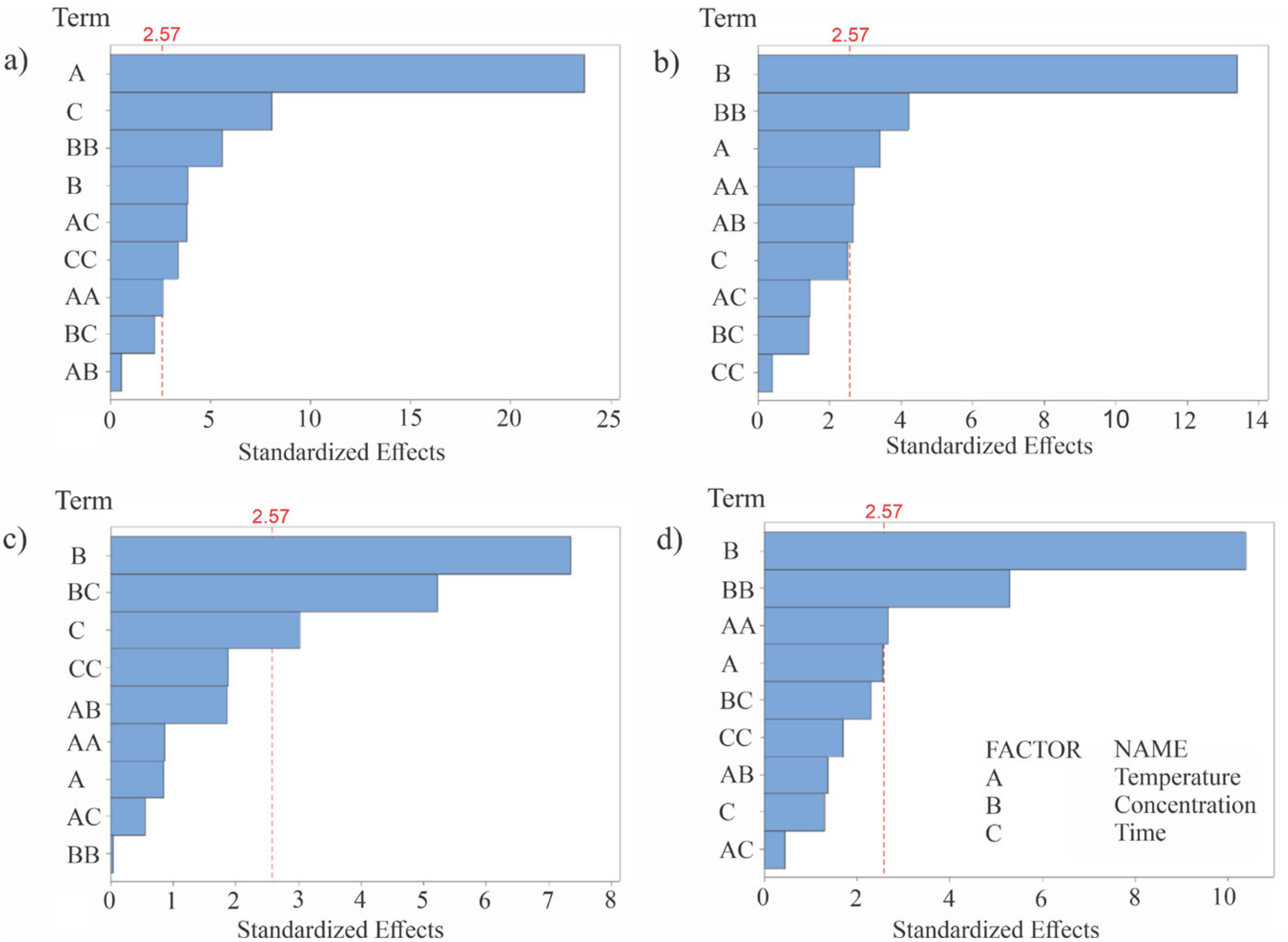

Figure 1. Pareto Chart of the Standardized Effect for a) chroma, b) hue angle, c) bleaching efficiency and d) peroxide value.

model for chroma in bleached oils, while Figure 1a shows the corresponding Pareto diagram. Among the parameters, temperature (A) has a very significant effect ($p < 0.0001$) on the chroma value.

The following parameters have an influence on chroma ($p < 0.05$): time (C), square of concentration (BB), concentration (B), cross product of temperature and time (AC), square of time (CC) and square of temperature (AA). The mutual interaction of concentration and time (BC) and temperature and concentration (AB) did not have significant effects ($p > 0.05$), and can be excluded from the regression model.

By discarding members that do not have a large impact, the regression equation in uncoded units has the following form:

$$C^* = 14.14 - 0.106 \cdot A - 3.218 \cdot B + 0.0448 \cdot C + 0.000735 \cdot \cdot AA + 0.624 \cdot BB - 0.00378 \cdot CC + 0.002063 \cdot AC \quad (5)$$

In order to assess the influence of bleaching earth concentration (B) and temperature (A) on the chroma value, a contour diagram was constructed based on the mean level (0) of time (30 min). Based on Figure 2a, it is observed that the chroma value decreased with decreasing temperature. Also, it was noticed that the lowest value of chroma was at the addition of 2–2.5 wt% of bleaching earth, at a temperature of about 75 °C.

Figure 2b shows the influence of time and temperature at a constant concentration of added earth (2 wt%). The chroma value was low for the entire covered time interval of 20 to 40 minutes at temperatures of 75–80 °C. As the temperature increased, the response value gradually increased. The contour diagram showing the effect of time and concentration on the chroma value of the bleached oil that was not processed, because based on the number of degrees of freedom, it is dependent on the previous two diagrams.

The lowest chroma values (9.24–9.90) were achieved at the lowest temperature in the study (75 °C) and moderate adsorbent concentration (2 wt%) regardless of time. At 75 °C, molecular diffusion in the pores of the adsorbent is fast enough, while further increase in temperature, although leading to even faster diffusion, also leads to thermal damage, which causes the chroma value to deteriorate. A dose of 2 wt% of adsorbent provides an optimal surface area for adsorption, while further increase in dose leads to the occurrence of particle agglomeration and pore block-

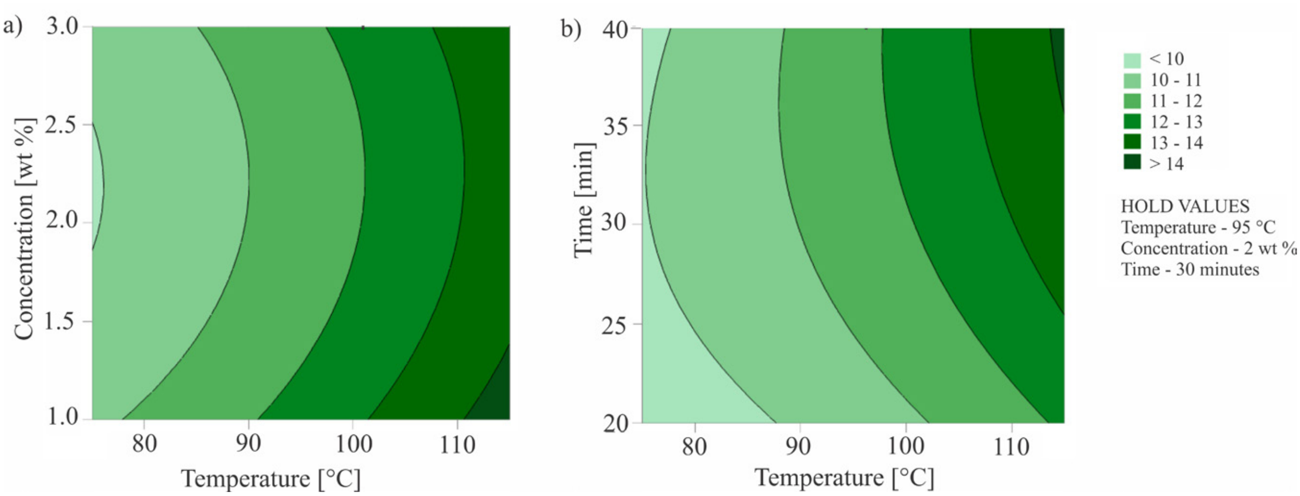

Figure 2. Contour diagrams to show the influence of a) concentration and temperature, b) time and temperature on the chroma value of bleached oil.

ing.[34] Similar trends are present in the work of Marrakchi et. al.[35]

3. 2. Influence of Process Parameters on the Value of Hue-angle

At the beginning of the interpretation of the results, an individual explanation of the CIELAB parameters a* and b* was provided. These parameters can be combined via Equation 2 to obtain the value of the hue-angle, which indicates the dominant color type of the sample (e.g., yellow, green, red...) and its shades. The hue-angle is crucial for assessing the purity and freshness of the oil, as it helps identity the presence of residual yellow and green pigments. Conceptually, the hue angle is graphically represented as an RGB CMY circle or a hue hexagon (Figure 3).

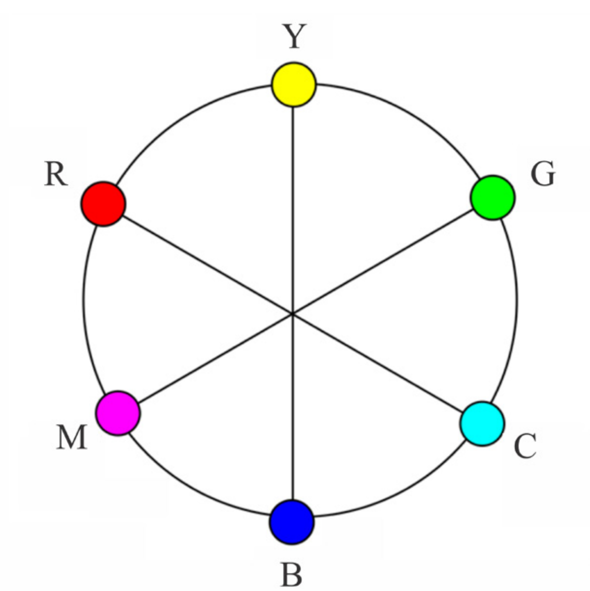

Figure 3. RGB CMY circle.[36]

The three primary screen colors-R (scarlet or orange-red), G (yellowish green), and B (deep violet-blue)- are arbitrarily placed at 120° angles to each other. Their additive complementaries Y (yellow), M (magenta or red-violet), and C (cyan or blue green)-are positioned opposite them.[36]

Table 4 shows the coded coefficients of the regression equation and p-values for members in the proposed quadratic model for the hue angle of bleached oils, while Figure 1b shows the corresponding Pareto diagram. Among the parameters, the concentration (B) has a very significant effect ($p < 0.0001$) on the hue angle value. Coefficient B has a high positive value, which indicates a favorable influence of concentration on the response.

The following parameters have a significant influence ($p < 0.05$): square of concentration (BB), temperature (A), square of temperature (AA) and mutual interaction of temperature and concentration (AB). The coded coefficient with these parameters has a negative value, which meant that increasing them decreased the value of the hue angle. The p-value of time (C) was 0.0535, slightly higher than allowed, but this term was kept in the reduced regression model to maintain the hierarchy of the model. The interactions of temperature and time (AC), concentration and time (BC) and time squared (CC) were dropped from the regression model because the p-value was far higher than 0.05.

By discarding terms that do not have a large impact, the regression equation in uncoded units for hue angle has the following form:

$$h = 90.31 + 0.172 \cdot A + 4.471 \cdot B + 0.108 \cdot C0 - 0.000711 \cdot AA - 0.447 \cdot BB - 0.01350 \cdot AB \quad (6)$$

In order to assess the influence of temperature (A), bleaching earth concentration (B) and time (C) on the hue angle value, contour diagrams were constructed showing the interaction of concentration and temperature (Figure 4a) and time and concentration (Figure 4b).

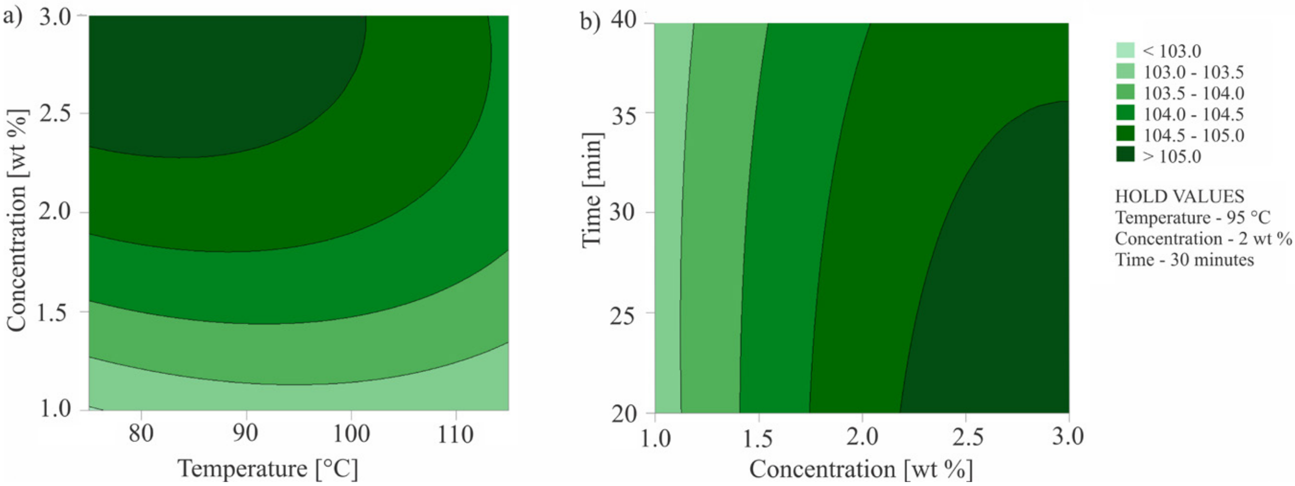

Figure 4. Contour diagrams to show the influence of a) concentration and temperature and b) time and concentration of adsorbent on the hue angle value of bleached oil.

By observing Figure 4, it is clear that concentration is the dominant factor, thus confirming the results of the ANOVA analysis and p-value. Addition of 2.4–3% of adsorbent, at temperatures of 75–102 °C lead to the maximum response value (h > 105) (Figure 4a). Hue-angle values are extremely high even when adding 1.8 wt% adsorbent in the entire temperature interval. A further decrease in the amount of added adsorbent led to a decrease in the value of hue angle which was independent of temperature.

A similar interaction between the variables can be seen in Figure 4b. The value of hue – angle was the highest in the first thirty minutes with the addition of 2.2–3% adsorbent, and with further extension of the process it decreased slightly. A sufficiently high response value was also achieved with the addition of 1.8% adsorbent, while further reduction of the added bleaching earth reduced the response value in the entire temperature interval.

By comparing chroma and hue-angle, it is observed that both the reduction of chroma and the increase of hue-angle are favored by lower temperatures, because further increase in temperature can lead to thermal decomposition. Higher doses of adsorbent (2.5–3 wt%) favor the increase of hue-angle, because for the shift of tone-angle to higher values (from 92.69 to 105.61) (less pure yellow, more yellow-green), carotenoids (yellow, brown colors) that bind more weakly at lower doses should be removed.[34,35]

3. 3. Influence of Process Parameters on Bleaching Efficiency Value

Table 4 and Figure 1c show the coded coefficients of the regression equation and p-values, i.e. the Pareto diagram for the proposed quadratic model for bleaching efficiency of bleached oils. As for the value of the hue angle, the concentration (B) has a very significant effect ($p < 0.001$) on the response.

In addition to concentration (B), the efficiency of bleached oil removal was significantly influenced ($p < 0.05$) by two other factors: time (C) and the interaction of concentration and time (BC). The coded coefficients with these parameters had negative values, which meant that increasing them decreased the value of bleaching efficiency.

ANOVA analysis also found that all other parameters have a p-value greater than 0.05, and can be discarded from the regression model while still maintaining a high degree of precision.

The abbreviated regression equation for the bleaching efficiency response has the following form:

$$Bleaching\ efficiency = 65.7 + 21.24 \cdot B - 0.089 \cdot C - 0.569 \cdot BC \quad (7)$$

By observing Figure 5a, it can be figured out that the bleaching efficiency did not change significantly with temperature, thus confirming the results of the ANOVA analysis. On the other hand, looking at the same figure, it was clear that the degree of bleaching depended on the amount of added adsorbent. Efficiency is highest with the addition of only 1 wt% of adsorbent for the entire temperature range, and the same efficiency can be achieved with the addition of up to 1.4 wt% of adsorbent at temperatures of 105–115 °C (bleaching efficiency >75%). By increasing the amount of added adsorbent, the degree of bleaching decreases almost linearly, and the lowest value of bleaching efficiency (bleaching efficiency <55%) is achieved with 3 wt%of adsorbent and temperatures higher than 90 °C.

It is noticed from the contour diagram 5b that at a time between 20–25 minutes, regardless of the amount of added adsorbent, almost the same bleaching efficiency is achieved. By prolonging the adsorption time, significant differences in bleaching efficiency are observed with the change in concentration. Thus, with smaller amounts of added adsorbent and time between 35 and 40 minutes, the highest degree of removal of pigments from the un-

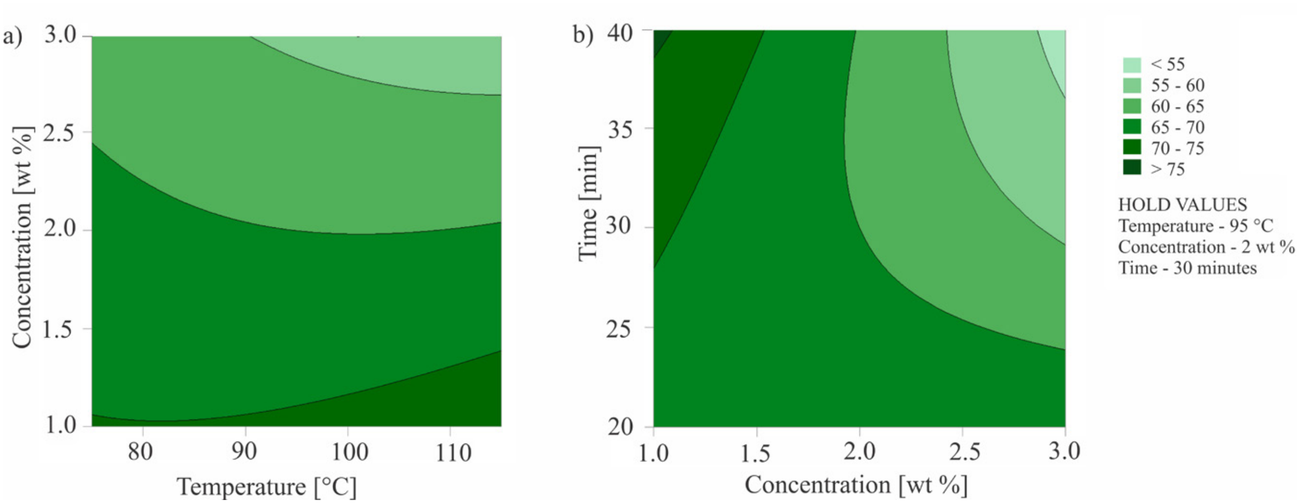

Figure 5. Contour diagrams to show the influence of a) concentration and temperature and b) time and concentration on the value of bleaching efficiency of bleached oil.

bleached oil (>70%) was achieved. On the contrary, at the same time interval and with the addition of larger amounts of adsorbent, a drastic decrease in the value of the degree of bleaching was observed.

Thus, the key influence of adsorbent concentration (factor B) and its interaction with contact time (BC). The highest efficiency (> 75%) was achieved at a low dose of 1 wt% adsorbent because at that concentration the ratio of active sites on the surface of the adsorbent and pigment distribution in the oil is the best, while the interaction of BC is most instructive at longer times (35–40 minutes), because at low doses of adsorbent additional time enhances the binding of pigments.[37] A similar trend is present in other works.[38,39]

3. 4. Influence of Process Parameters on Peroxide Value

Although some studies use the TOTOX index (peroxide value combined with anisidine value) to determine the content of primary and secondary oxidation products, in our research only the peroxide value was analyzed. Preliminary analysis found that the PV values were low for unrefined oil, and it was considered unnecessary to include the anisidine value. However, this approach has certain limitations because it is possible that certain secondary oxidation products were formed during refining that cannot be detected using the peroxide value.

Table 4 shows the coded coefficients of the regression equation and p-values for the members in the proposed quadratic model for peroxide value of bleached oils, while Figure 1d shows the corresponding Pareto diagram. Among the parameters, the concentration of the added adsorbent (B) has a very significant effect ($p < 0.0001$) on the peroxide value. A negative regression coefficient indicates that increasing the concentration decreases the value of the responses.

The following parameters have an influence on the peroxide value ($p < 0.05$): the square of the concentration (BB) and the square of the temperature (AA). The p-value of temperature (A) is slightly higher than the allowed value (0.0503), but since it is a linear variable, it will not be removed from the regression model. Other factors do not greatly affect the value of the responses and can be excluded from the regression model.

A shortened regression model for the peroxide value is given by the relation (8):

$$\textit{Peroxide value} = 5.20 - 0.0632 \cdot A - 1.627 \cdot B + 0.000379 \cdot AA + 0.3012 \cdot BB \tag{8}$$

Although the previous shortened regression model does not have many terms, the same predictive accuracy is still maintained. At the same time, it enables a clearer interpretation, reduces the risk of over-adjustment, and facilitates practical application.

Figures 6a and 6b show that the amount of added adsorbent is the key variable that affects the value of the peroxide value, which confirms the results of the ANOVA analysis. The response value is the highest at a temperature of 110 °C, with the addition of 1 wt% adsorbent in the initial 30 minutes of adsorption (>1.5 meq/kg). Also, with the addition of the same amount of adsorbent, but at lower temperatures and longer adsorption time, the value of response decreases (1.25–1.5 meq/kg). By increasing the concentration, almost independently of temperature and time, the value of the peroxide value decreases.

By observing Figure 6a, it can be concluded that at concentrations of 2.5–3 wt% adsorbent and temperatures of 80–105 °C, resulted in the lowest value of the peroxide number (< 0.5 meq/kg), and that range of conditions is considered optimal. The reason for the pronounced decrease in peroxide value with increasing adsorbent concentration is that its abundant porous structure and surface functional

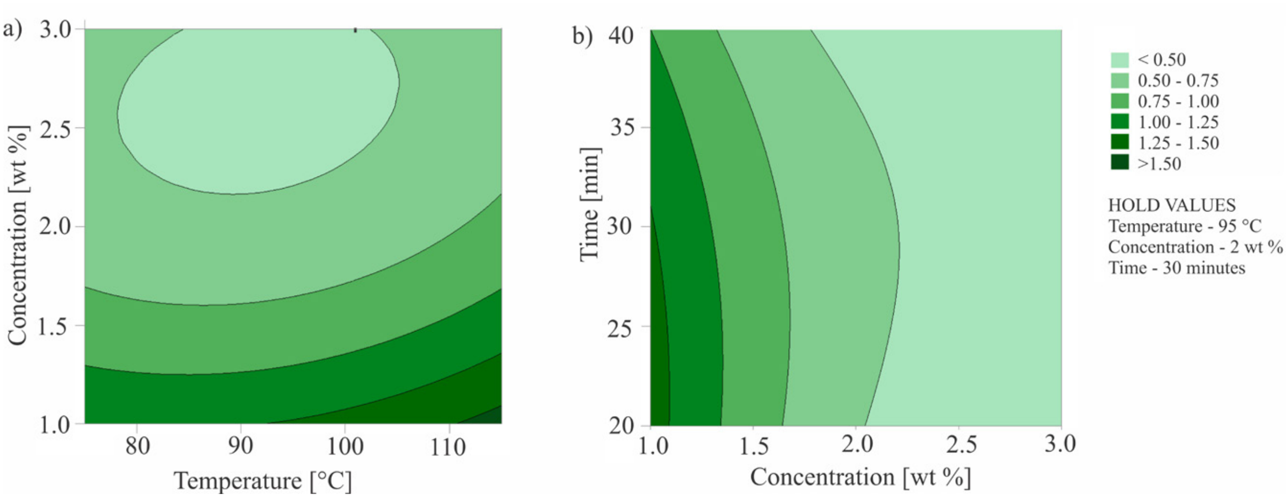

Figure 6. Contour diagrams to show the influence of a) concentration and temperature and b) time and concentration on the Peroxide value of bleached oil.

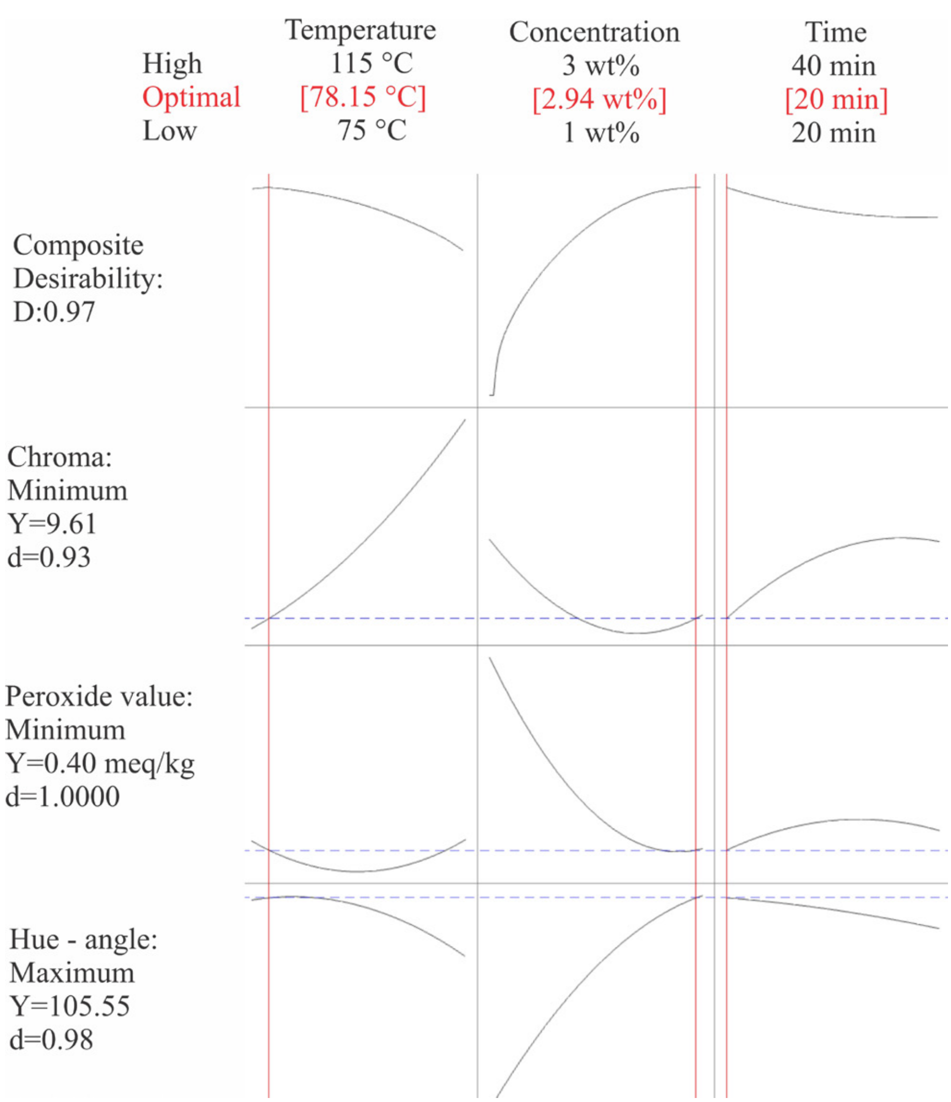

Figure 7. Optimization plot for three responses: chroma, peroxide value and hue-angle.

groups actively adsorb hydroperoxide species.[12] Also, these surface sites catalyze the decomposition of trapped hydroperoxides into non-oxidizing substances.[40] These effects are consistent with a similar study by Chew et al., where kenaf seed oil was bleached under different bleaching conditions.[5]

3. 5. Optimization

The input parameters (temperature, time and amount of added adsorbent) affect the output variables (chroma, hue angle, peroxide value and bleaching efficiency) differently. An optimal combination of input parameters must be found for each response. The goal of color quality optimization is to maximize the hue-angle to obtain lighter oils, and minimize the chroma value to reduce the intensity of the color.[34] Also, a minimum peroxide value is required, in order to obtain refined oil free from oxidative compounds while also maximizing bleaching efficiency.

Given the existence of four responses and the complex optimization, the responses were grouped into two categories.

In the first group, the process conditions were optimized for the following responses: chroma, hue angle and peroxide value. On one hand, there is chroma and hue angle serve asindicators of the color quality of refined oils, on the other hand, the peroxide value serves as an indicator of the presence of unwanted oxidative compounds. For this optimization, the goal is to minimize both chroma value and peroxide value, and to maximize the hue-angle value.

Figure 7 shows the optimization plot for the first group of responses. Since there is no specific target value, only a minimum or maximum values are considered for each response, leading to one optimal solution. The optimal process parameters are: temperature of 78.15 °C, adsorbent concentration of 2.94 wt% and time of 20 minutes, at which the value of chroma is minimized (9.61), the peroxide value is minimized (0.40 meq/kg), and the hue-angle is maximized (105.56). Composite desirability (0.97) is close to unity, indicating that the chosen settings achieve favorable results for all responses overall. Individual desirability indicates that the set settings most strongly affect the peroxide value (1.00), followed by the the hue-angle (0.98) and the least on the chroma (0.93).

In the second group, two responses were contrasted: peroxide value, as a measure of the chemical quality of the oil, and bleaching efficiency, as a value that shows the percentage of colored pigments removed. As in the first group, the goal is to minimize the value of the peroxide number, while on the other hand the value of bleaching efficiency is maximized.

Figure 8 shows the optimization plot for the second group of responses. The obtained results are similar to the first group; the minimum value of the peroxide value (0.41 meq/kg), i.e. the maximum value of the bleaching efficiency (71.87%) is achieved at a temperature of 77.83 °C, a concentration of the added adsorbent of 2.96 wt% and a time of 20 minutes.

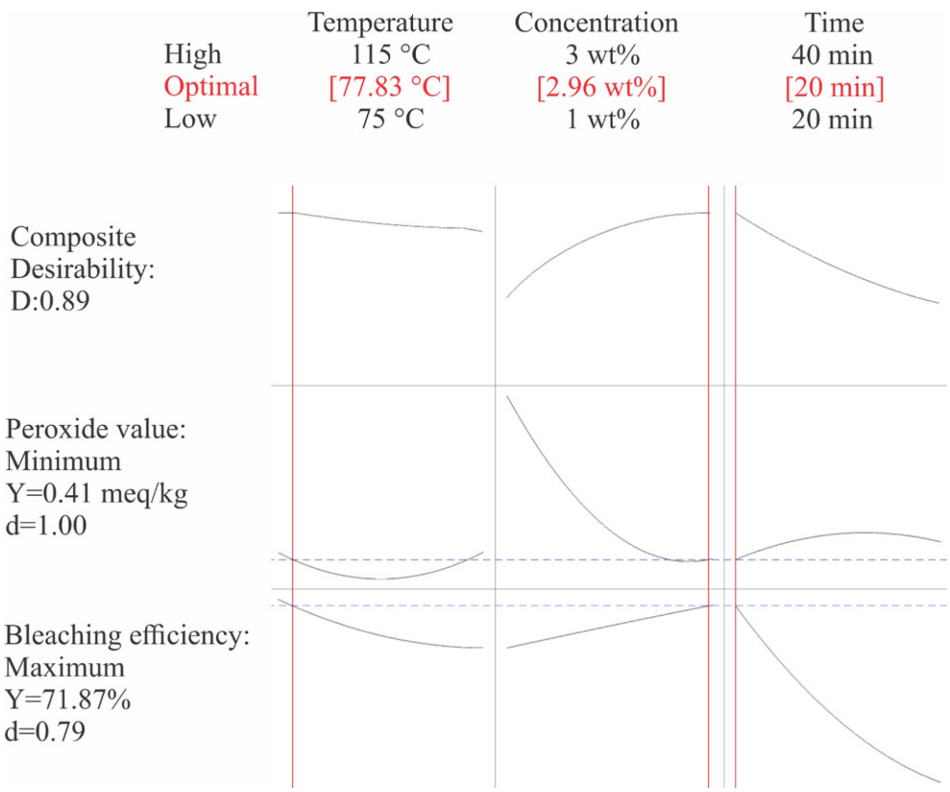

Figure 8. Optimization plot for two Responses: bleaching efficiency and peroxide value.

Composite desirability is slightly lower compared to the first optimization (0.89), which indicates that the settings achieve slightly weaker results when both responses are observed simultaneously. When the responses are considered separately, it is concluded that the settings have a far greater effect on the peroxide value (1.00) than on the bleaching efficiency (0.79).

Observing the optimal conditions for both groups of responses, it is clear that similar results are obtained: in both cases, the minimum time (20 min) is optimal for obtaining the best quality oil, the temperature ranges between 77.83–78.15 °C, and the concentration of the added adsorbent 2.94–2.96 wt%. When performing the optimization, either the first or the second group of responses can be examined because similar results are obtained. Nevertheless, the first group has a higher composite desirability (0.97) compared to the second (0.89), on the basis of which it was concluded that spectroradiometry, which was used to determine chroma and hue-angle, was far more reliable than spectrophotometry that was used to determine bleaching efficiency, because it covered the entire visible color spectrum, not just the 450 nm wavelength.

Minimum time (20 min) and lower temperatures (78 °C) have a favorable effect on the oxidative properties (peroxide value) and the color of the sample. By prolonging the adsorption time and increasing the temperature, the oxidative stability of edible oils decreases.[40] On the other hand, the unfavorable influence of increasing the time and increasing the temperature on the color of the oil is reflected in the destruction of the active sites on the adsorbent.[41] Moderate temperatures were required to obtain an optimum color for the bleached oil. This can be explained by the fact that working in this range of temperature, the following phenomena may occur: (I) a better activation, in terms of pigments removal, of the acid-activated earth employed, (II) a reduction in the oxidation of colorless components which cause alteration in the oil color, and (III) a less pronounced fixing of the existing color pigments.[42] In some studies, it was found that high temperatures (100–110 °C) have a favorable effect on the removal of colored pigment because the viscosity decreases with temperature, and therefore the dispersion of particles and the interaction of the oil with the adsorbent is better.[12] However, this is not the case in this paper. A possible reason for this is that the adsorbent particles were small enough (d50 = 45 μm), thus viscosity is not a limiting factor.

The addition of high concentrations of bleaching earth resulted in the removal of undesirable compounds in the oil. The catalytic properties of acid-activated bleaching earth lead to the decomposition of hydroperoxides into secondary oxidation products, which decrease the peroxide value with increasing concentration.[43] High adsorbent concentrations also have a favorable effect on color reduction, because there are more active sites where the adsorption of colored pigments takes place.[5]

4. Conclusion

The Box–Behnken factorial design proved to be an effective tool for optimizing temperature, adsorbent concentration, and time in the sunflower oil bleaching process. Significant p-values (<0.05), along with high R^2 (>0.95) and Adjusted R^2 confirmed that the model provides a strong fit to the experimental data. The composite desirability for the group containing chroma and hue-angle (determined by spectroradiometry) along with peroxide value is 0.97, compared to 0.89 for the group containing bleaching efficiency (determined by spectrophotometry) and peroxide value. This indicates that full-spectrum coverage by spectroradiometry provides a more reliable assessment of oil quality. The optimal conditions identified in the study can be to bleach bleaching sunflower oil in industrial conditions with minimal resource use. Future studies should explore the use of other adsorbents, such as bio-based or modified adsorbents, in oil bleaching. Additionally,the adsorbent used in this study could be applied to bleach other vegetable oils, such as soybean or palm oil.

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Povzetek

Beljenje je ključni korak v rafiniranju olja, ki odstranjuje nezaželene pigmente in oksidacijske produkte, ki poslabšajo kakovost olja. Ta študija je ocenila učinke temperature (75–115 °C), koncentracije adsorbenta (1–3 wt%) in časa (20–40 minut) na beljenje sončničnega olja z uporabo Box-Behnken faktorske analize v Minitab 21. Odgovori so bili razdeljeni v dve skupini: (I) vrednost peroksida in spektrometrična metoda ter barvni kot, in (II) vrednost peroksida in spektrofotometrično merjena učinkovitost beljenja. Pod optimalnimi pogoji (približno 78 °C, 2,95 wt% adsorbenta in 20 minut), se je vrednost peroksida zmanjšala z 1,8 meq/kg na 0,398 meq/kg, kromatska vrednost se je zmanjšala s 45,89 na 9,61, kot obarvanosti se je povečal s 92,69 na 105,55, učinkovitost beljenja pa je dosegla 71,86%. »Composite desirability« je bila višja v prvi skupini (0.97 proti 0.89), predvsem zaradi bolj objektivne narave spektrometričnih parametrov.