Journal of Food Science and Technology (Iran)

Homepage: www.fsct.modares.ir

Scientific Research

Best combination of bleaching parameters on quantitative and qualitative characteristics of sunflower and corn oils using response surface methodology

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ARTICLE INFO

Article History:

Received: 2023/10/3
Accepted: 2023/11/14

Keywords:

Sunflower oil;
Corn oil;
Optimization;
Bleaching parameters;
Response surface methodology;
Bleaching efficiency;
Bioactive compounds

DOI: 10.22034/FSCT.20.140.169
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ABSTRACT

The bleaching process is important in the refining operation of edible oils. In this study, bleaching parameters were changed to evaluate the best condition for optimizing of bleaching in corn and sunflower oils. For this purpose, the effect of time (15, 25, 35, 45, and 55 min), temperature (80, 90, 100, 110, and 120 °C), and bleaching clay concentration (0.4, 0.6, 0.8, 1, and 1.2%) was evaluated using response surface methodology as statistical design in 5 levels by Central composite design with the aim of reducing the consumption of bleaching clay. The studied factors were including carotenoids, sterols, tocopherols, pigment and cations. After optimization, the best conditions for bleaching of corn and sunflower oils was different. For corn oil, the best conditions was time 39.59 min, temperature 103.61 °C, and bleaching clay concentration 1%, which meet 67% of our expectations. For sunflower oil with the aim of reducing the consumption of bleaching clay, the best conditions were time 37.49 min, temperature 97.53 °C and bleaching clay concentration 1%, which satisfied 70% of our expectations. The practical results did not show any significant differences with obtained theory values using response surface methodology and confirmed these results.

1- Introduction

Edible oils and fats are essential nutrients. When fats and oils are consumed as part of a balanced diet, they have many health properties. Lipids and fat-related compounds play a key role in growth and preventing disease. Fats and oils are one of the most important source of energy supply which produce about 9 kcal/g compared to protein and carbohydrates that have about 4 kcal/g. Lipids effect on the texture, taste, and palatability of food products (Dunford and Dunford, 2004). Vegetable oils contain some compounds as impurities that cause an unpleasant taste and odor and adverse effect on health properties. Therefore, the refining process is necessary to remove impurities and improve its quality (Didi and Makhoukhi, 2007). The four basic steps used to refine oil include gumming, neutralizing, bleaching, and deodorizing (Ssebuwufu et al., 2014).

Bleaching is an adsorption process to remove undesirable components (Nwabanne and Ekwu, 2013), Bleaching involves the removal of pigments, impurities, trace metals, and the molecular oxidative component of fats and oils (Okolo and Adejumo, 2014; Nwabanne and Ekwu, 2013). Removal of these substances is essential in refined oil as it improves the stability, appearance and sensory quality of the oil (Okolo and Adejumo, 2014). In bleaching step, undesirable compounds are primarily removed such as oxidation products, dyes, soaps, contaminants and metal elements (especially copper and iron). Their presence in oil can be due to the transfer of equipment containing copper and iron, although due to the modern use of stainless steel equipment in food processing plants is less common than before. However, metal elements must be removed from the oil during bleaching in order to improve the oxidative stability of oils. However valuable compounds such as tocopherols and sterols may also be eliminated, a significant loss of oxidative stability may occur, and the amount of free fatty acid may increase (Skevin et al., 2012).

Therefore, the aim of this study was to evaluate the simultaneous effect of changes in bleaching parameters at 5 levels including temperature (80, 90, 100, 110 and 120 ° C), time (15, 25, 35, 45 and 55 minutes), and bleaching clay concentration (0.4, 0.6, 0.8, 1 and 1.2 percent) on the quality of sunflower and corn oils by response surface methodology (RSM). The analysis included bleaching efficiency (by measuring carotenoid, color, and metals contamination), the content and composition of bioactive compounds (tocopherol and sterol).

2. Materials and methods

Degummed and neutralized sunflower and corn oils were prepared from Varamin 2 oil factory (Varamin 2, Tehran, Iran) and Glucosane factory (Glucosane, Ghazvin, Iran) respectively. Sulfuric acid-activated bleaching clay was provided from Kanysazejam Company (Lushan, Iran). All chemical material prepared from Merck Company.

2.1. Oil bleaching under laboratory conditions

Bleaching of sunflower and corn oils was performed in laboratory condition at 30 rpm in a 2000 ml flask equipped with a thermometer and connected to a vacuum pump. Bleaching was performed by an electromagnetic mixer with an adjustable heater equipped with a thermocouple and a temperature switching system. To accurately adjust the temperature using a thermometer, apply the command to disconnect and connect to the heater and adjust the temperature to the desired temperatures. The industrially neutralized oils were weighed into a round bottom flask and heated by vacuum mixing. Acid-activated bleaching clay with concentrations of 0.4, 0.6, 0.8, 1 and 1.2 ٪ was added to preheated oil at 60 °C. The oil was then gradually reached the desired bleaching temperature (80, 90, 100, 110 and 120 °C) by constant stirring under vacuum to completely disperse the bleaching clay. After reaching the desired temperatures, the heating was gradually cut off and the intended bleaching time (15, 25, 35, 45 and 55 min) was applied. At the end of the bleaching, the heating and vacuum were turned off. After bleaching, the mixture of bleaching clay and hot oil from the flask was filtered through a millipore in vaccum. The bleached oil sample was held at -18 °C until be evaluated (Skevin et al., 2012).

2.2. Fatty acid composition

In order to analyze the fatty acids of oil samples, GC 6890 Agilent device was equipped with auto sampler and FID detector was used. Preparation of methyl ester of fatty acids was done according to ISO 12966-2 and ISO 12966-4. Approximately 10 g of the sample was transferred to a ballon. 10 ml of hexane and 5 ml of 2 M methanol KOH were added to it. The content of the ballon was refluxed for 15 min at 70 °C and then was cooled. 10 ml of saturated NaCl solution was added to it. The mixture was stirred well and then allowed to form a two-phase mixture. The organic layer was separated and transferred to a vial then about 1 g of sodium hydrogen phosphate was added and stirred well. The vial was centrifuged and the top layer was used for injection into the GC. Fatty acid levels were expressed as sub-peak percentages (ISO, 2011).

[DOI: 10.22034/FSCT.20.140.169]

Experimental conditions:
Column: 5 m× 250 µm × 0.2 µm Cpsil88
Oven temperature: 180 °C
Detector temperature: 280 °C
Injector temperature: 260 °C
Carrier gas: Nitrogen
Injection volume: 1 mL
Injection ratio: 1/100

2.3. Bioactive compounds

2.3.1. Sterols

Sterols content and composition were determined using by gas chromatography and according to ISO 12228: 1999 method. The prepared sterol fraction (1 µl) was injected into a gas chromatograph equipped with a DB-17 capillary tube column (30 m × 0.32 mm × 0.25 mm) containing 50% of the stable phase of phenyl-methylpolysiloxane. Helium was used as a carrier gas at a constant rate of 0.36 ml/min. The injection and detector temperature was 280 °C and 290 °C, respectively. The oven column temperature was programmed to rise 6 °C from the initial temperature of 180 °C to 270 °C and then held for 30 min. Peaks were identified by comparing sterol retention times with standards. Quantitative determination of all sterols was based on internal standard by α-cholestenol (ISO, 1999).

2.3.2. Tocopherols

Tocopherols were determined according to the standard ISO 9936: 2006 method. Analysis was performed by Knauer high-performance liquid chromatography (HPLC) consist of a K-1001 pump, a 20 µl injection loop, Eurosphere C_{18} 5 µm (250 × 4.6 mm) column, and a RF-10AXL fluorescence detector controlled by Eurochrom software (all from Knauer, Germany). The sample was prepared by dissolving 0.1 g of oil in 10 ml of *n*-hexane, and 20 µl of solution was injected into the column. Then an excitation wavelength at 295 nm and an emission wavelength at 330 nm were used. Isocratic chromatography was used at room temperature with a mobile phase of %0.7 propan-2-ol in *n*-hexane at a flow rate of 0.6 ml/min. Quantitative determination of tocopherols was performed using standard α and-γ tocopherol calibration curves covering the mass fraction range of 5-750 mg / kg (ISO, 2006).

2.4. Efficiency of bleaching

The bleaching efficiency was investigated by measuring the reduction in color and metal contaminants.

2.4.1. Fe and Cu

Fe and Cu content were measured according to AOCS method No. 75- Ca15. In this method, first the samples were ashed, then acid was added to it. After preparing Fe and Cu standards, the concentration of these metals was determined by atomic absorption device (Varian company model 220Z) along with the flame atomizer and acetylene fuel with the air flow of 1.5 to 3 and a holocathode of a multi-element lamp (AOCS, 2007).

2.4.2. Carotenoids

The concentration of carotenoids, such as the amount of β-carotene, was determined by spectrophotometer (SL 160, India). Specific absorption measurements were performed with an UV visible spectrophotometer. According to this method, 1 g of oil in 25 ml flasks was dissolved in isooctane and filled to the line by isooctane. The maximum adsorption was recorded at 440-455 nm and the concentration of β-carotene was calculated from the following equation (AOCS, 1998):

$$C_{\beta\,\text{carotene}} = \frac{A \times v}{W \times 0.261}$$

Where A: adsorption, V: volume of solution (ml), W: sample weight (g).

2.4.3. Color measurement by Lovibond method

The evaluation of color for both samples of sunflower and corn oils were done according to ISO 15305: 1998 method using Lovibond Colorscan (AOCS, 1998).

2.4.4. Experimental optimization design and statistical analysis

RSM with 3 parameters including temperature (80-120 ℃), time (15-55 min), and bleaching clay concentration (0.4-1.2%) at 5 levels (-2, -1, 0, 1, and 2) and six replications at the central point (17, 16, 15, 12, 11, and 6 Nos) was used based on the central composite design (CCD). By considering these parameters and their levels (20 runs processes), the statistical design table was developed (Table 1), which X_1 (time), X_2 (temperature), and X_3 (bleaching clay concentration) were three independent parameters and measuring of sterol, tocopherol, cations, carotenoids and color were dependent variables. In RSM, a model is designed for each dependent variable that expresses the main and variable effects of parameters on each variable separately. The multivariate model is in the form of equation (2):

$$Y = \beta_0 + \sum_{i=1}^{3} \text{Bi Xi} + \sum_{i=1}^{3} \text{Bii Xi Xi} + \sum_{i=1}^{3} \text{Bij Xi Xj} \quad (2)$$

Where B_i is factor regression coefficient i; Bij is the regression coefficient of the interaction of two factors i and j; Xi is time, temperature, and bleaching clay concentration.

Table 1. Design of response surface methodology for the present study using design expert software.

3. Results and Discussion

[DOI: 10.22034/FSCT.20.140.169]

3.1. The fatty acid composition

Table (2) shows the fatty acid profile for sunflower and corn oils, which is consistent with Codex Stan 210-1999.

Table 2. Fatty acids composition of sunflower oil and corn oil.

3.2. Sterols

Sterols are very important compounds in edible oil. Bleaching step may cause considerable changes in this compound. The result of this study shows that the only effective parameters on sterols in sunflower oil were time and bleaching clay concentration. Unexpectedly no significant effect of temperature parameter was observed that can be due to the low amount of primary sterols in sunflower oil. As was shown in Fig. 1, the model for all sterols is a linear model that time and bleaching clay concentration is significant. The interaction of these two parameters has become very small and is not significant due to low coefficients of influence of these two parameters and therefore the software has proposed a linear model. Changes in sitosterol as the main sterol in sunflower oil is very important. According to Fig. 1, the only significant parameter in β-sitosterol is the bleaching clay concentration. As observed in other sterols, the effect of bleaching clay concentration on the reduction of sterols is much higher than the effect of time, which convinced the reduction of β-sitosterol. The effect of time and bleaching clay concentration on the weight percentage of sunflower oil sterols showed at Fig. 1.

Fig. 1. Effect of time and bleaching clay concentration on weight percentage of sunflower oil sterols a: Campesterol; b: Cholesterol; c: Stigmasterol; d: Δ^5-Avenasterol; eΔ^7-Stigmasterol; f: β- Sitosterol; g: Avenasterol; h: Other sterols; i: Total sterols.

In corn oil, for campesterol has been proposed a linear model due to its small amount compared to other sterols that this model is a suitable one for predicting the behavior of campesterol during oil bleaching step. In this model, the effect of temperature was no significant (such as most sterols behavior in sunflower oil). Bleaching clay concentration was more effective parameters in reducing of cholesterol rather than time in corn oil. In the case of cholesterol, due to its little amount in vegetable oils, the coefficient of these parameters is very small and the proposed linear model is a suitable model for changes in cholesterol levels during bleaching of oils.

About β-sitosterol, due to its higher amount than other sterols and also the higher amount of sterols in corn oil compared to sunflower oil, model 2FI[1] has been suggested in this model. According to the proposed model, in addition to the effect of each of parameters, their interactions were investigated, the interaction of temperature and bleaching clay concentration has become significant. Adj R-squared and adeq precision showed a suitable fit of this model to predict the behavior of β-sitosterol in oil bleaching operations. In the case of avenasterol and other sterols that have not been studied, a linear model has been proposed due to their small amount.

About total sterol similar to the topics mentioned for β-sitosterol, the effect of temperature have been seen more due to the higher amount. Adj R-squared and adeq precision indicated the appropriate fit of the proposed linear model for changes in the total amount of sterols in these oils. The effect of these parameters in terms of importance are bleaching clay concentration, time, and temperature, respectively. Effect of time and bleaching clay concentration on the weight percentage of corn oil sterols have been shown in Fig. 2.

Ayerdi and Rhazi (2016) stated that no study has so far evaluated the effect of the bleaching step on sunflower oil sterols. In other oils, the amount of total sterol in cottonseed oil decreased slightly. Decreases of 1.3, 8, and 18.5% in corn, soybean and rapeseed oils were reported for total sterol content, respectively. Due to the in-depth analysis of sterol evaluation during the purification process, different behavior depending on the oil matrix was shown to evaluate free and esterified sterols. Corn oil showed an increase of more than 3% of free sterol and a reduction of 5% of esterified sterol.

Fig. 2. Effect of time and bleaching clay concentration on weight percentage of corn oil sterols a: Campesterol; b: Stigmasterol; c: Cholesterol; d: Δ^5-Avenasterol; e: Delta-7-Stigmasterol; f: β-sitosterol; g: Avenasterol; h: Other sterols; i: Total sterol.

3.3. Tocopherol

Tocopherols are sensitive to light, heat, alkalis and metal contaminants. The amount of tocopherols in oils is specific and depends on the plant genotype, climatic conditions of growth and harvest, the amount of polyunsaturated fatty acids in the oil and storage, and process conditions. Industrial process and oxidation during storage may affect the level of tocopherols in the oil (Ortega-García et al., 2006).

In sunflower and corn oils, the effect of three parameters including temperature, time, and bleaching clay concentration on tocopherols at bleaching step has followed the quadratic equation

[1]. Two- Factor interactions

according to equations (3), and (4). All three parameters were effective in reducing tocopherols over time and had negative effect.

Tocopherol = 693.201-0.025295A-3.13290B-46.97045C + $0.014399B^2$ (3)

Tocopherol=0.711-3.62729*10-3A-0.0333B-1.03233C+6.37407*10-5A^2-1.51656*10-4B^2-0.56465C (4)

The effect of time can be expressed according to Fick's law (equation 5) which according to Fick's law, the speed of mass transfer increases with increasing time. According to Stoke-Einstein's (equation 6), the effect of temperature on the reduction of viscosity and further increase of the adsorption rate can be explained. The increasing of bleaching clay concentration in addition to the direct effect, by increasing the mass transfer in Fick law also is effective.

$$\dot{m}B = DA\frac{\Delta C}{\Delta x} \quad (5)$$

Where: m_B is the mass velocity of the particle flow B (kg/hr), C is the concentration B (kg/m3) or the number of moles per unit volume (kg-mol/m3), D is the propagation factor (m2/s), A is the area (m2), and X is diffusion distance (m).

$$\mathrm{D} = \frac{\mathrm{k}B\mathrm{T}}{6\pi\eta r} \quad (6)$$

Where: k_B is Boltzmann’s constant; T is the absolute temperature; η is the dynamic viscosity; r is the radius of the particle. In bleaching step, tocopherols are absorbed into the bleaching clay and oxidized. This increases the lack of tocopherols and thus affects the stability of the oil (Ergönül and Köseoğlu, 2014; Wu et al., 2019). The results were similar to the findings of Ortega-Garcia et al. (2006). Exposure of oil to air or heat may reduce the amount of tocopherols by oxidation or polymerization. The effect of bleaching parameters on the tocopherols in sunflower and corn oils is shown in Figs. 3 and 4.

Fig. 3. Effect of temperature and bleaching clay concentration on tocopherols in sunflower oil.

Fig. 4. Effect of bleaching parameters on tocopherols in corn oil a: Temperature and time; b: Bleaching clay concentration and temperature.

3.4. Cations

In the present study, the results in corn oil were similar to sunflower oil. The results showed that the bleaching clay concentration was the only significant parameter for changing the amount of Fe during bleaching step in sunflower and corn oils which had a negative effect (Fig. 5 and Fig. 6). The main reason for this could be related to the adsorption mechanism of cations into the bleaching clay which depends on ionic attraction. Therefore, normal diffusion equations have no effect on it and the effect of time and temperature on the adsorption rate is reduced. Linear model was used for the significant effect of bleaching clay concentration on the amount of Fe in sunflower oil (equation 7) and corn oil (equation 8), respectively.

Fe = 0.0627-0.020C

Fe = 0.3.572 - 0.12656C

Fig. 5. Effect of time and bleaching clay concentration on the amount of iron in bleaching operation in a: Sunflower oil; b: Corn oil.

According to equation (9) and (10), Cu content in sunflower oil and corn oil had been influenced by bleaching clay concentration.

Cu (ppm) = 0.050931-0.046563C (9)

Cu (ppm) = 0.039925-0.024375C (10)

Mukasa-Tebandeke et al. (2014) stated that the reduction in the amount of Fe was the highest in all samples in bleached oils and the amount of Cu showed the smallest change.

Fig. 6. The effect of time and bleaching clay concentration on the amount of Cu in sunflower oil; b: the effect of temperature and concentration of bleaching clay on the amount of Cu in corn oil in bleaching operations.

3.5. Carotenoids

The results of statistical analysis showed that the linear equation was significant to investigate the effect of bleaching parameters on the carotenoid content in sunflower oil and its lack of fit was not significant, which indicates optimal fit of this equation with experimental results. Adj R-squared 0.98 and adeq precison 64.62 indicate the appropriate fitting of the model in predicting the behavior of the studied parameters in sunflower oil (equation 11 and Fig. 7). In the proposed equation, the effect of bleaching clay concentration as the only effective parameter on changes in carotenoid content is significant.

Carotenoids(ppm) = 0.28125C-0.41450 (11)

As was shown in equation 10, amount of carotenoids in corn oil had followed the quadratic equation. In this quadratic equation, all factors became significant in form of independent, quadratic, and in interaction with each other (equation 12 and Fig. 8). Adj R-squared 0.99 and adeq precision 32.14 shows a very high relationship between the proposed equation and experimental results.

The difference between the equations of sunflower and corn oils can be due to the low initial amount of carotenoids in sunflower oil. Therefore, the rate of carotenoid changes was less under the influence of parameters and their effects were not significant.

Carotenoids(ppm)=41.93168-0.39670A-0.48160B-16.00455C+2.5*10^4AB+0.06AC+0.037500BC+4.40114*$10^{-3}A^2$+2.10114*$10^{-3}B^2$+5.34659C^2 (12)

[DOI: 10.22034/FSCT.20.140.169]

The results of Sanei et al. showed that bleaching clay concentration is the most important factor in β-carotene uptake and then temperature had the greatest effect. The results of these studies were in agreement with the obtained results. At low temperatures, β-carotene is adsorbed on the exterior of the bleaching clay, while at higher temperatures, more adsorption is done indoors. According to this result, reduction of β-carotene due to the increasing in temperature can be explained. The effect of temperature on the removal of carotenoids is intensifying, which is not much and only facilitates and accelerates the absorption on the surface of the bleaching clay (Sanaee et al., 2014).

Fig. 7. Effect of time and bleaching clay concentration on the amount of carotenoids in sunflower oil.

Fig. 8. Effect of bleaching step on the amount of carotenoids in corn oil a: effect of temperature and time; b: effect of bleaching clay concentration and temperature.

3.6. Color by Loviband method

3.6.1. Red factor (R)

The amount of R factor in sunflower oil is less than corn oil, which can be attributed to the lower amount of carotenoids in this oil. The change rate of R factor can be indicated by the quadratic equation**.** The significance of the equation and the insignificance of the lack of fit indicate the optimal fit of the equation with the experimental results (Table 3). The results of variance analysis showed that among the studied parameters, time, temperature, bleaching clay concentration and the second power of time and bleaching clay concentration were significant parameters in this equation. Adj R-squared 0.85 and adeq precision 16.75 indicates the optimal accuracy of this equation in predicting experimental results.

In corn oil, the effect of bleaching parameters on the amount of R factor followed the linear equation (Table 3). Significant parameters in this equation were time and bleaching clay concentration and also had a negative effect on the R factor. The intensity of the effect of bleaching clay concentration is greater than time. The effect of the time can be expressed by Fick's law. The effect of bleaching clay concentration can also be explained and interpreted by increasing the surface of bleaching clay with Fick equation. The linear equation was significant and the lack of fit of the model was insignificant. Also adj R-squared 0.76 and adeq precisor 12.16 indicated the appropriate fitting of the model with the experimental results.

3.6.2. Yellow factor (Y)

In sunflower oil, variance analysis of the equation indicated that the equation was significant and its lack of fit was insignificant. According to Table 3, among the parameters used, time, bleaching clay concentration, and their quadratic power were significant. According to Table 3, the value of adj R-squared 0.95 and adeq precision 16.61 indicates the appropriate accuracy of the proposed equation in predicting experimental results.

In corn oil, the effect of bleaching parameters on the Y factor can be explained with the 2FI equation. The independent effect of all variables was significant and all of them reduced Y factor (Table 3). In addition, the interaction of time and bleaching clay concentration was also significant.

Among the studied factors, the concentration of bleaching clay had the greatest effect on reducing Y factor. Adj R-squared 0.76 and adeq precison 12.16 showed a very high correlation between the studied parameters and experimental results.

3.6.3. Blue factor (b)

The bleaching parameters did not have significant effect on b in sunflower oil. Its reason was the very low amount of this factor in oil samples (maximum 0.3). According to the accuracy of the experiment, the effect of the studied parameters on its changes has not been very obvious. In corn oil, two parameters of time and bleaching clay concentration were the most effective in reducing b. (Table 3). Although temperature also showed a decreasing effect, but this effect was not significant. Factor b is important because it can be an indicator of burns and the presence of undesirable compounds in the oil (Sedaghat Boroujeni et al., 2020). Although its value is lower than other factors, its presence is important in terms of oil quality. Its low initial value (0.9) may obscure the interaction effects of the parameters and the value of the adj R-squared and adeq precison showed a very good correlation between the proposed equation and the experimental results in Table 3.

Table 3. The effect of bleaching parameters on the color of corn and sunflower oils.

4. Conclusion

The results generally showed that the bleaching operation can be performed for other purposes except color reducing. For example, by changing the parameters which affect the bleaching process, useful compounds such as sterols, tocopherols, etc. can be preserved in oil. According to the obtained results in corn oil, it was found bleaching clay concentration is the most important parameters in the oil bleaching process and its effect on all factors measured was greater than other parameters. The best bleaching clay concentration obtained was 1%, which can improve the quality of the bleached oil by reducing the bleaching time. In this situation, the bleaching process meets about 67.8% of our needs (Table 4).

[DOI: 10.22034/FSCT.20.140.169]

In sunflower oil due to low initial amount, the bleaching operation is more desirable. The most important parameter in achieving optimal conditions of sunflower oil is bleaching clay concentration. The amount of desirability under optimal conditions is about 71% which the maximum desirability is obtained if 1% of bleaching clay is used.

Table 4. Optimal values of evaluated parameters and theoretical and practical results of tests in sunflower and corn oils processing.

Declaration of Competing Interest

The authors report no declarations of interest.

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[DOI: 10.22034/FSCT.20.140.169]

Figure captions

Fig. 1. Effect of time and bleaching clay concentration on weight percentage of sunflower oil sterols a: Campesterol; b: Cholesterol; c: Stigmasterol; d: Δ^5-Avenasterol; eΔ^7-Stigmasterol; f: β- Sitosterol; g: Avenasterol; h: Other sterols; i: Total sterols.

Fig. 2. Effect of time and bleaching clay concentration on weight percentage of corn oil sterols a: Campesterol; b: Stigmasterol; c: Cholesterol; d: Δ^5-Avenasterol; e: Delta-7-Stigmasterol; f: β-sitosterol; g: Avenasterol; h: Other sterols; i: Total sterol.

Fig. 3. Effect of temperature and bleaching clay concentration on tocopherols in sunflower oil.

Fig. 4. Effect of bleaching parameters on tocopherols in corn oil a: Temperature and time; b: Bleaching clay concentration and temperature.

Fig. 5. Effect of time and bleaching clay concentration on the amount of iron in bleaching operation in a: Sunflower oil; b: Corn oil.

Fig. 6. The effect of time and bleaching clay concentration on the amount of Cu in sunflower oil; b: the effect of temperature and concentration of bleaching clay on the amount of Cu in corn oil in bleaching operations.

Fig. 7. Effect of time and bleaching clay concentration on the amount of carotenoids in sunflower oil.

Fig. 8. Effect of bleaching step on the amount of carotenoids in corn oil a: effect of temperature and time; b: effect of bleaching clay concentration and temperature.

[DOI: 10.22034/FSCT.20.140.169]

Tables

Table 1. Design of response surface methodology for the present study using design expert software.

Run no.	Time (min)	Temperature (˚C)	Concentration (%)
1	35(0)	120(+2)	0.8(0)
2	35(0)	80(-2)	0.8(0)
3	35(0)	100(0)	1.2(+2)
4	45(+1)	110(+1)	1(+1)
5	55(+2)	100(0)	0.8(0)
6	35(0)	100(0)	0.8(0)
7	45(+1)	110(+1)	0.6(-1)
8	35(0)	100(0)	0.4(-2)
9	45(+1)	90(-1)	1(+1)
10	25(-1)	90(-1)	1(+1)
11	35(0)	100(0)	0.8(0)
12	35(0)	100(0)	0.8(0)
13	25(-1)	110(+1)	1(+1)
14	45(+1)	90(-1)	0.6(-1)
15	35(0)	100(0)	0.8(0)
16	35(0)	100(0)	0.8(0)
17	35(0)	100(0)	0.8(0)
18	15(-2)	100(0)	0.8(0)
19	25(-1)	110(+1)	0.6(-1)
20	25(-1)	90(-1)	0.6(-1)

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Table 2. Fatty acids composition of sunflower oil and corn oil.

Percent		Fatty acid
Corn oil	Sunflower oil	
0.21	0.53	C14
-	0.1	C14:1
13.8	8.15	C16
0.13	0.17	C16:1
3.28	3.82	C18
44.76	-	C18:2
1.45	-	C18:3
-	0.15	C18:1t
35.87	-	C18:1c
-	29.27	C18:1c9
-	0.5	C18:1c11
-	56.86	C18:2c
0.5	0.25	C20

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Table 3. The effect of bleaching parameters on the color of corn and sunflower oils.

	Corn oil			Sunflower oil		
	R[1]	**Y**[2]	**b**[3]	**R**	**y**	**b**
Equation	linear	2FI[4]	linear	quadratic	quadratic	ns
B_0	8.42	49.61	0.91	12.36	91.75	
B_1	-0.032	-0.17	$6.87*10^{-3}$	-0.047	-0.17	
B_2	ns	-0.095	ns	-0.079	ns	
B_3	-1.84	1.62	-0.41	-9.81	-29.94	
B_{12}	ns	ns	-	Ns[5]	ns	
B_{13}	ns	-0.106	-	ns	ns	
B_{23}	ns	ns	-	ns	ns	
B_{11}	ns	-	-	$1.26*10^{-3}$	$7.25*10^{-3}$	
B_{22}	ns	-	-	ns	ns	
B_{33}	ns	-	-	3.15	-21.56	
R^2	0.76	0.76	0.99	0.81	0.95	0.92
adeq precisior	12.16	12.16	123.21	16.75	16.61	13.16

1- Red factor

2- Yellow factor

3- Blue factor

4-Two- Factor interactions

5- not significant

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Table 4. Optimal values of evaluated parameters and theoretical and practical results of tests in sunflower and corn oils processing.

Corn oil		Sunflower oil		Factors
Practical value	**predicted value**	**Practical value**	**predicted value**	
	0.67		0.706	Desirability
	39.59	37.5	37.49	Time(min)
	103.61	97	97.53	Temperature(˚C)
	1	1	1	Concentration(%)
0.02±0.005	0.02	0.02±0.01	0.02	Campesterol
ns	ns	ns	ns	cholesterol
0.016±0.003	0.018	0.016±0.002	0.017	Stigmasterol
0.01±0.005	0.01	0.009±0.001	0.009	Δ_5- avenasterol
0.014±0.003	0.015	0.015±0.002	0.015	Δ_5 -stigmasterol
0.14±0.02	0.13	0.12±0.02	0.13	β-sitosterol
0.007±0.002	0.008	0.008±0.001	0.008	Avenasterol
0.02±0.002	0.019	0.018±0.003	0.019	Other sterols
0.226±0.008	0.222	0.213±0.01	0.218	Total sterols
453.65±44.67	486.33	479.32±23.2	485.02	Tocopherol(ppm)
0.14±0.03	0.13	0.11±0.02	0.12	Carotenoid(ppm)
0.02±0.01	0.02	0.02±0.01	0.02	Cu(ppm)
0.05±0.01	0.04	0.04±0.01	0.04	Fe(ppm)
1.64±0.22	1.75	1.73±0.04	1.72	R
18.02±1.49	17.55	17.22±1.48	17.37	Y
0.16±0.03	0.17	0.17±0.02	0.17	B

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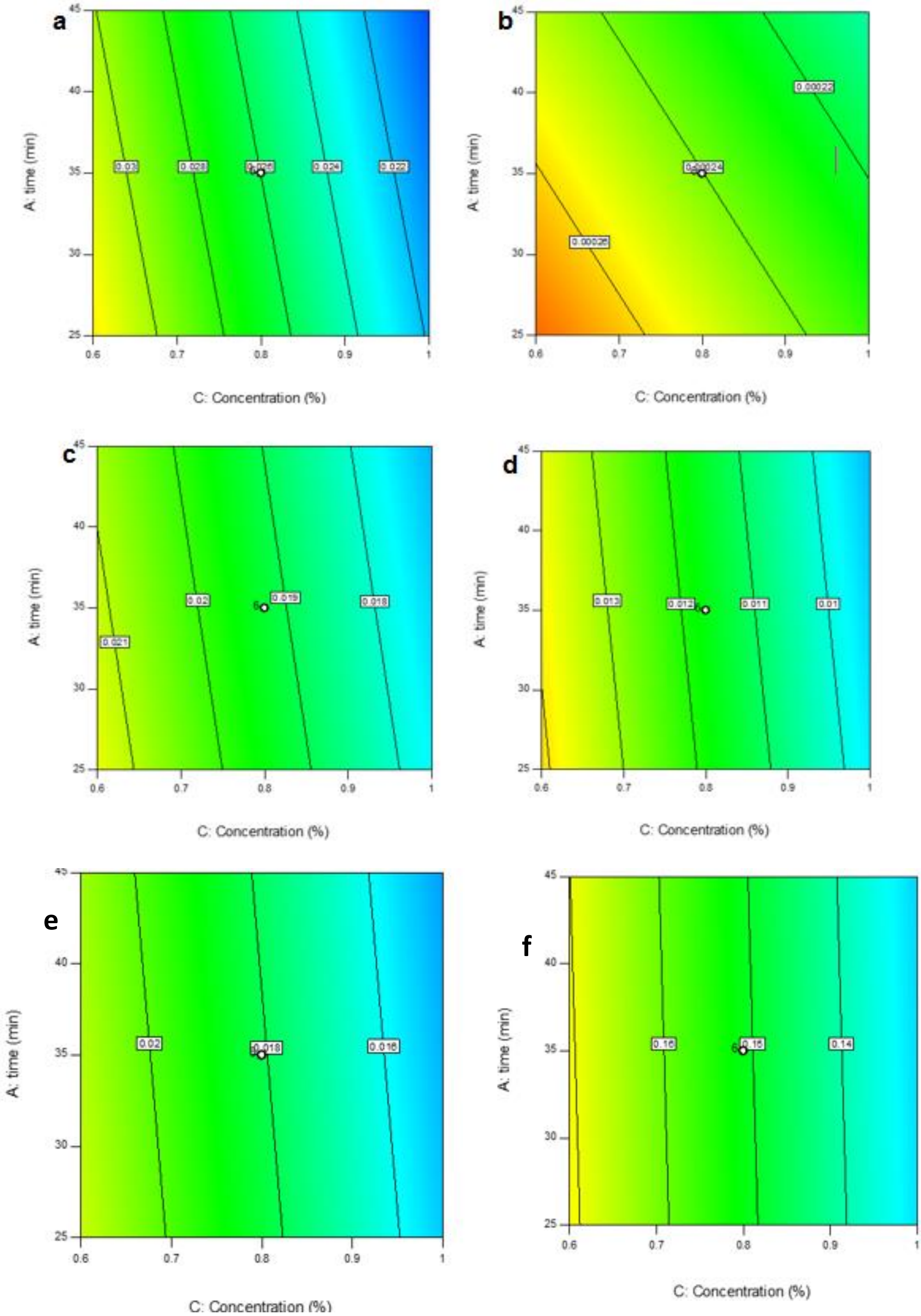
a
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0.028
0.024
0.022
b
0.00022
0.00024
0.00026
c
0.021
0.02
0.019
0.018
d
0.013
0.012
0.011
0.01
e
0.02
0.018
0.016
f
0.16
0.15
0.14
A: time (min)
C: Concentration (%)

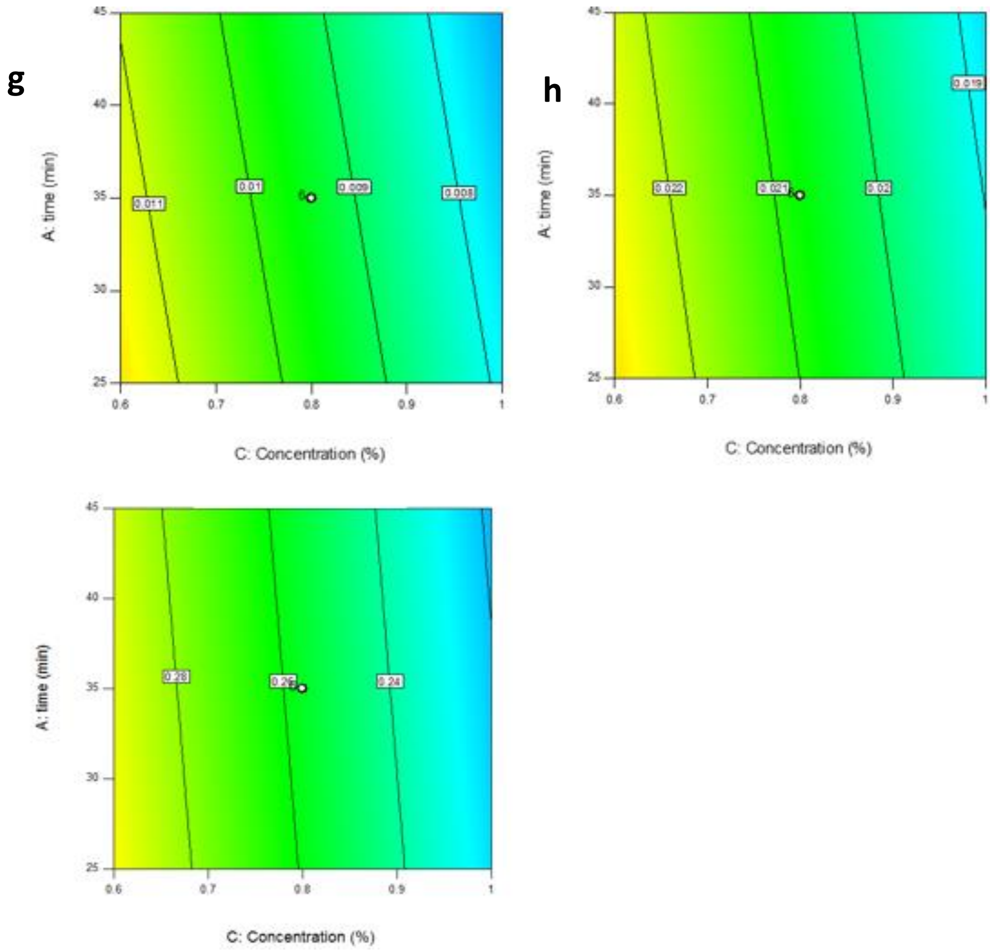

Fig. 1. Effect of time and bleaching clay concentration on weight percentage of sunflower oil sterols a: Campesterol; b: Cholesterol; c: Stigmasterol; d: Δ^5-Avenasterol; eΔ^7-Stigmasterol; f: β- Sitosterol; g: Avenasterol; h: Other sterols; i: Total sterols.

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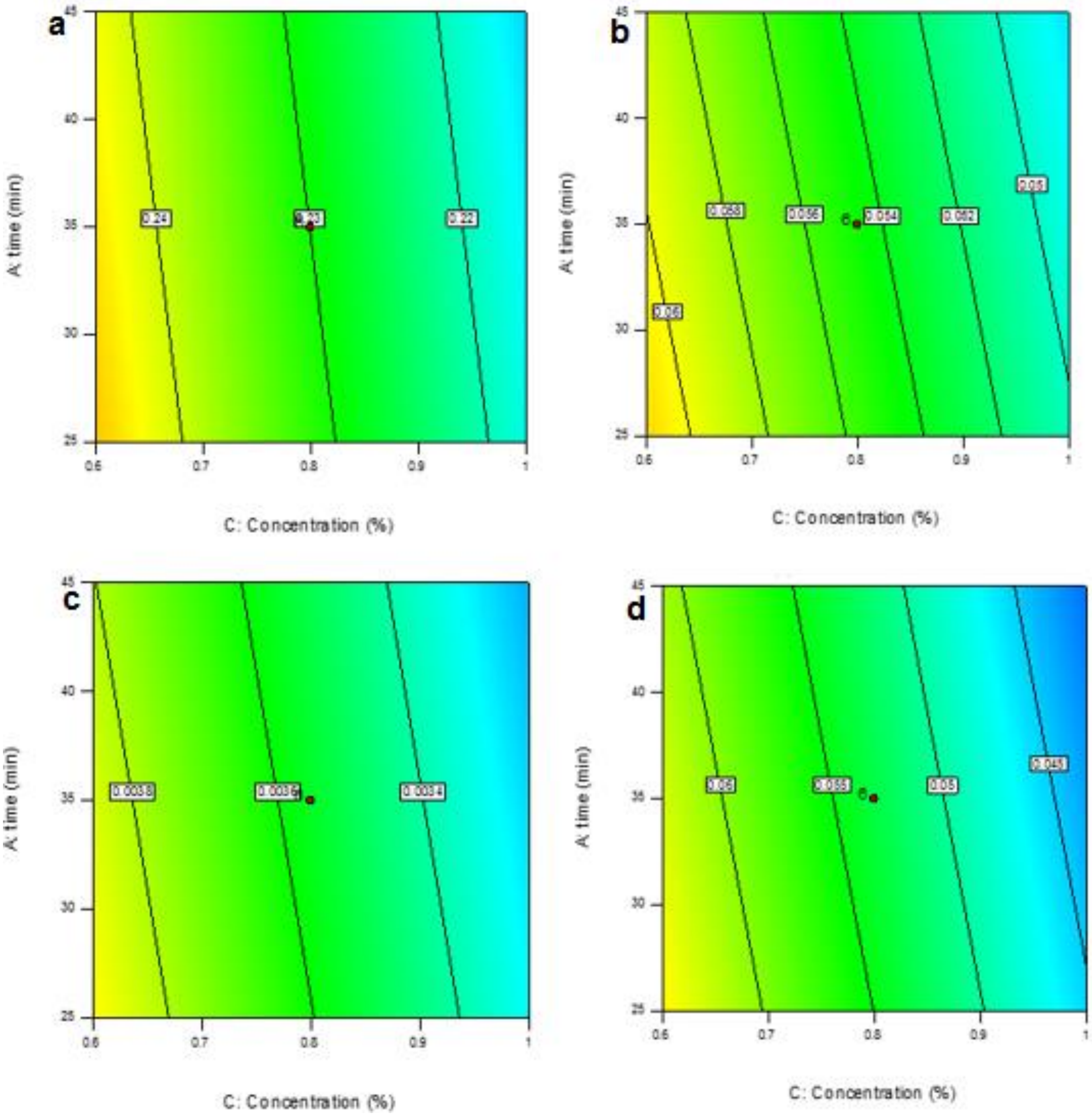

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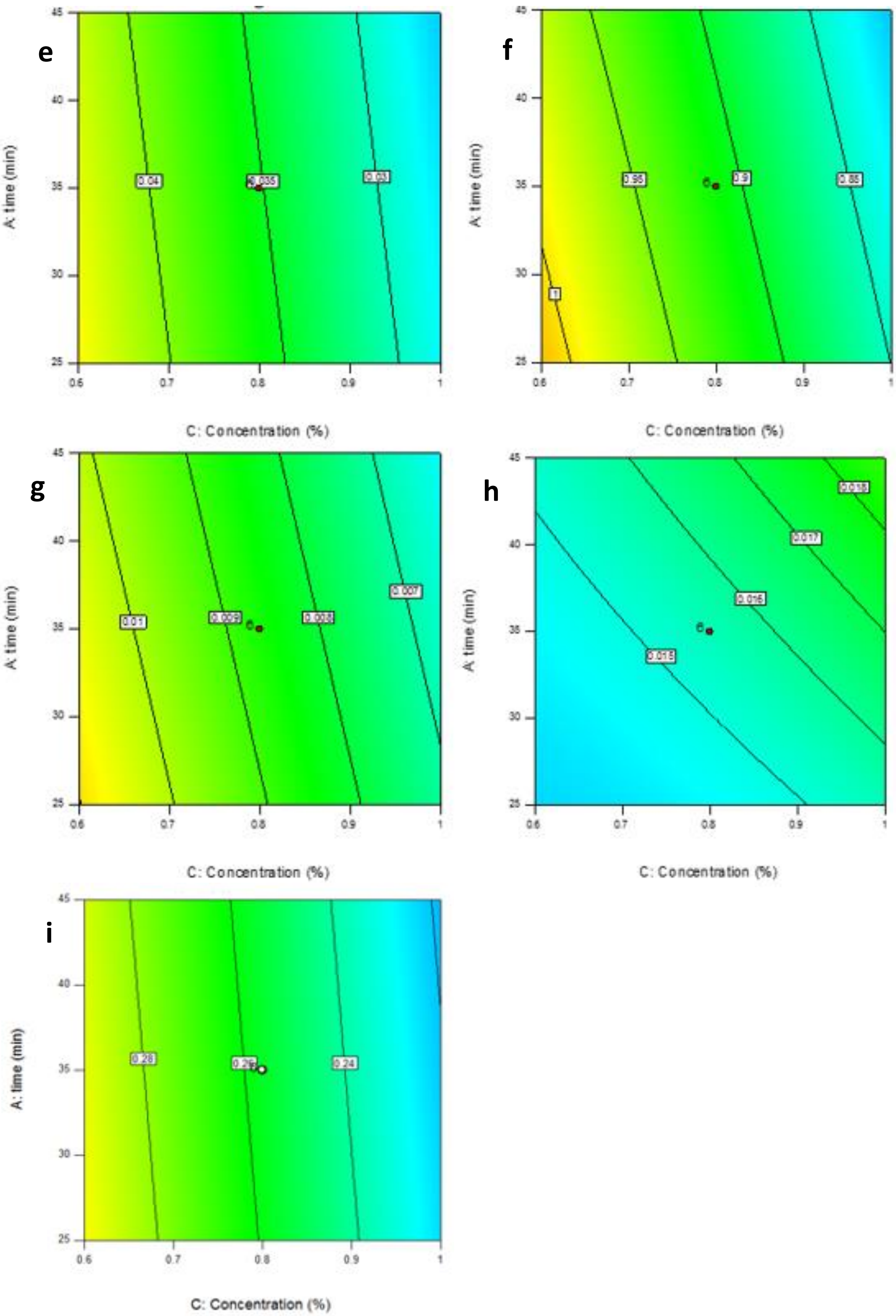

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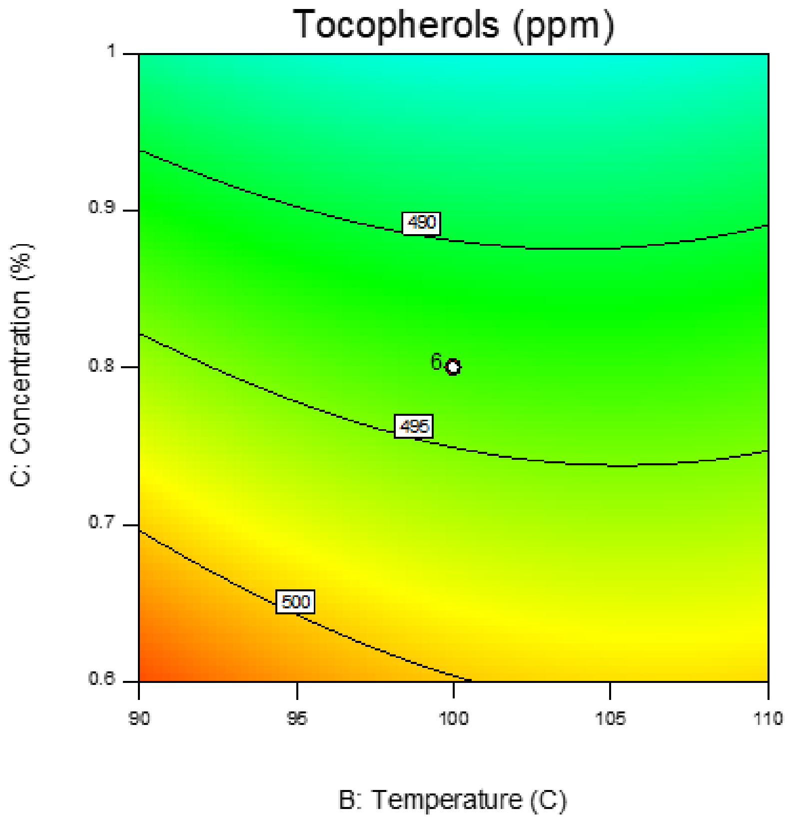

Fig. 3. Effect of temperature and bleaching clay concentration on tocopherols in sunflower oil.

[DOI: 10.22034/FSCT.20.140.169]

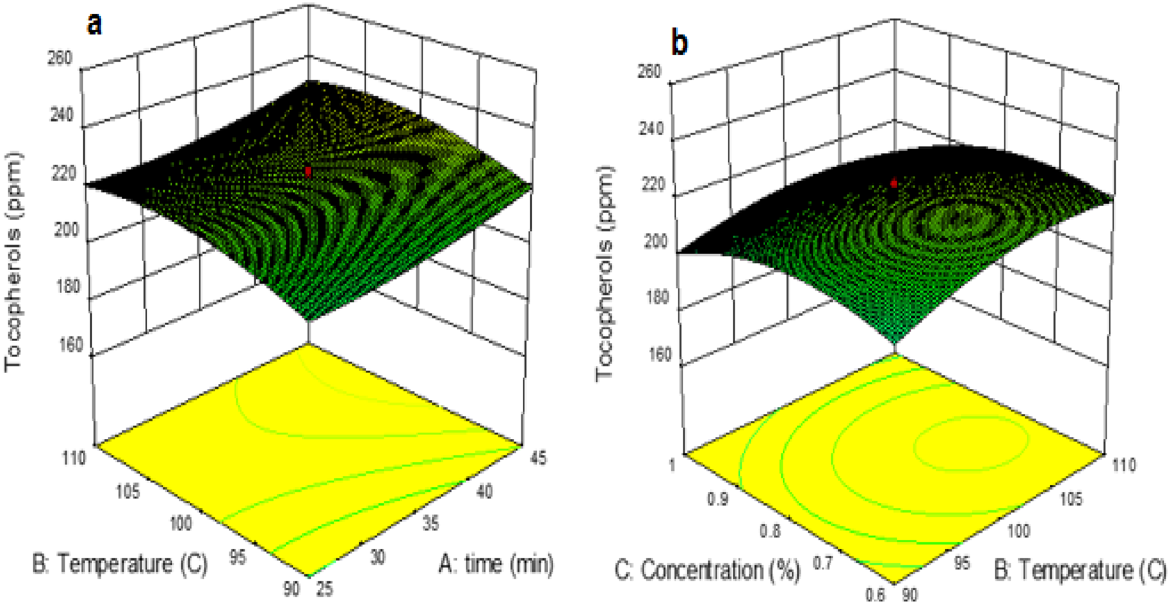

Fig. 4. Effect of bleaching parameters on tocopherols in corn oil a: Temperature and time; b: Bleaching clay concentration and temperature.

[DOI: 10.22034/FSCT.20.140.169]

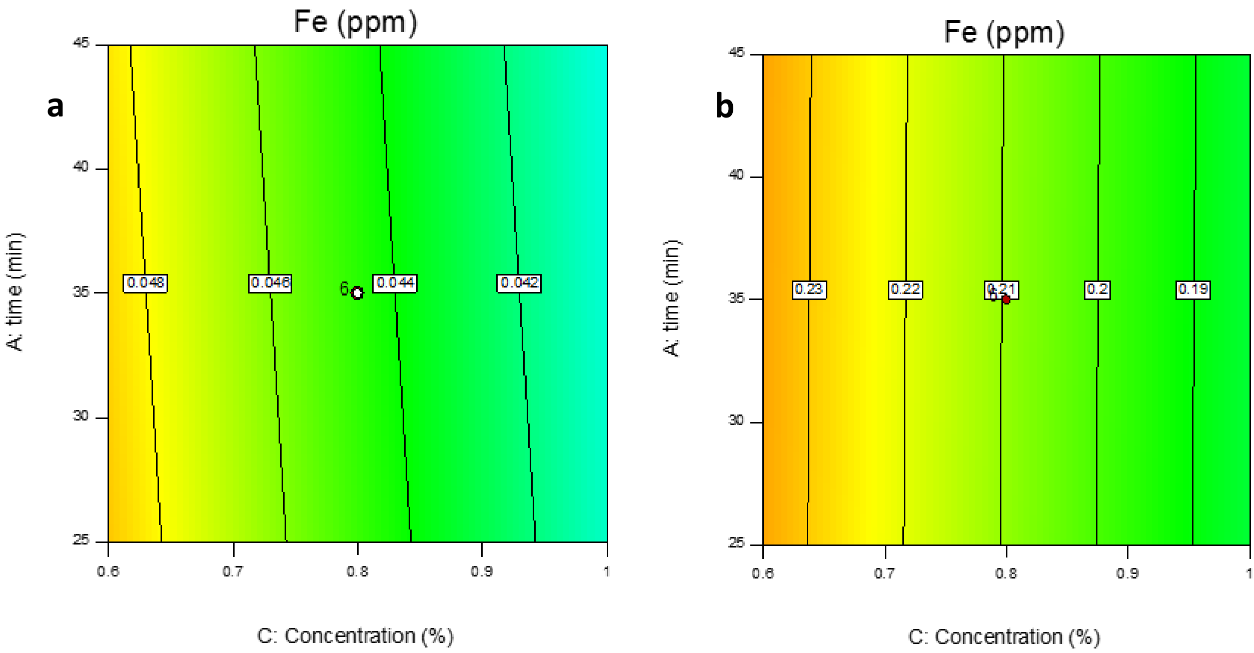

Fig. 5. Effect of time and bleaching clay concentration on the amount of iron in bleaching operation in a: Sunflower oil; b: Corn oil.

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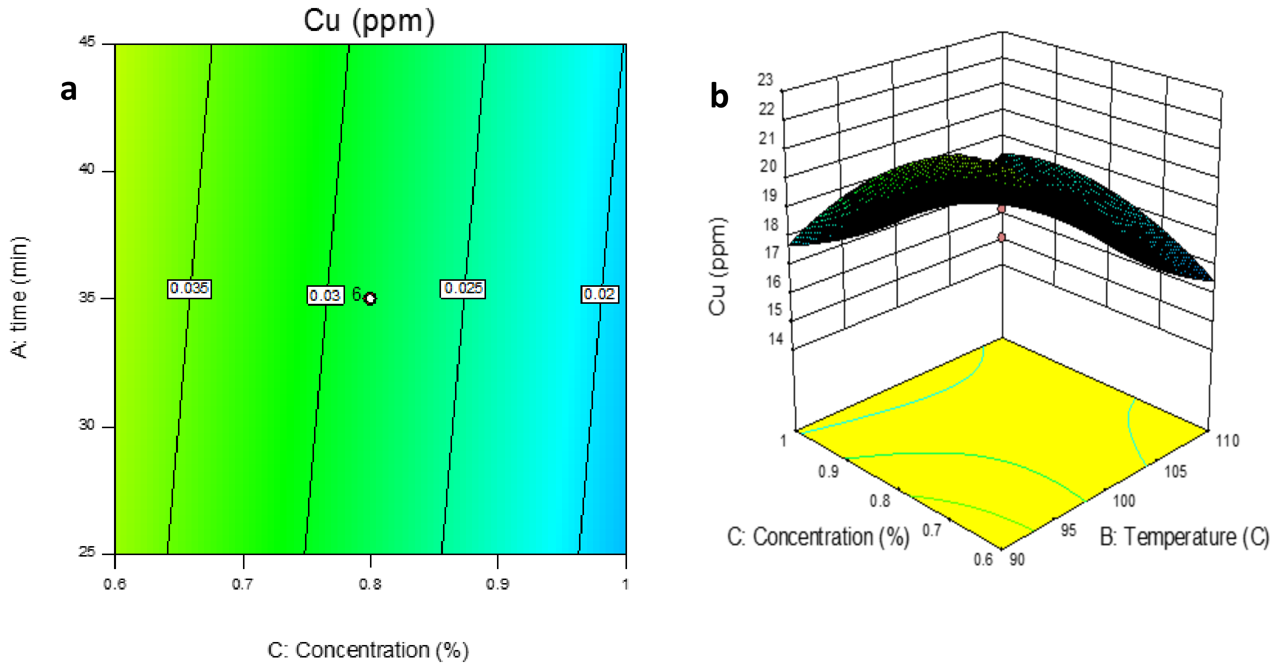

Fig. 6. The effect of time and bleaching clay concentration on the amount of Cu in sunflower oil; b: the effect of temperature and concentration of bleaching clay on the amount of Cu in corn oil in bleaching operations.

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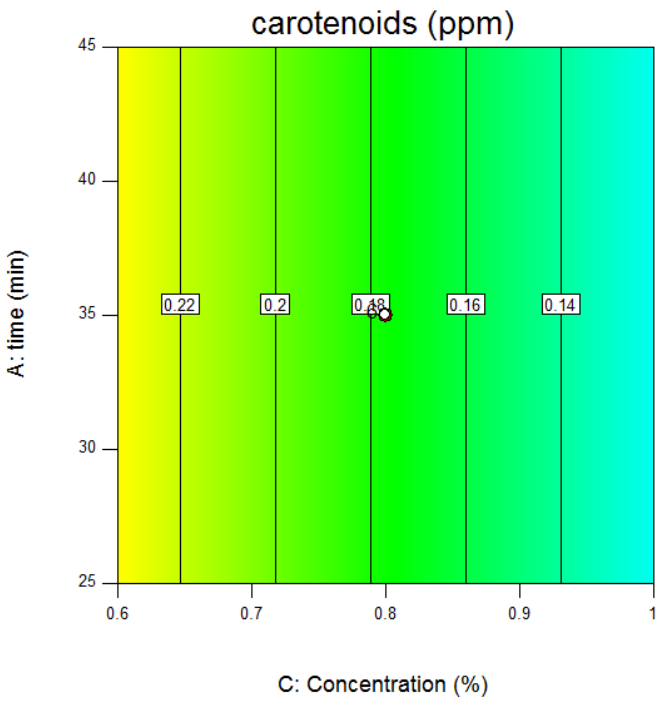

Fig. 7. Effect of time and bleaching clay concentration on the amount of carotenoids in sunflower oil.

[DOI: 10.22034/FSCT.20.140.169]

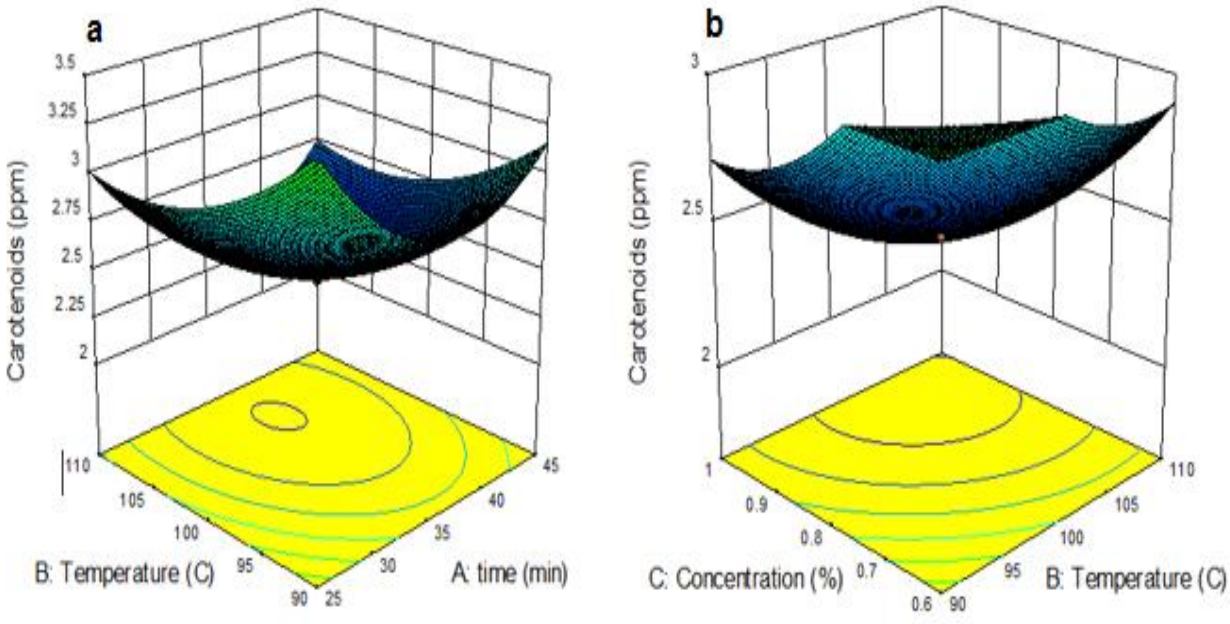

Fig. 8. Effect of bleaching step on the amount of carotenoids in corn oil a: effect of temperature and time; b: effect of bleaching clay concentration and temperature.

[DOI: 10.22034/FSCT.20.140.169]

مجله علوم و صنایع غذایی ایران

سایت مجله: www.fsct.modares.ac.ir

مقاله علمی_پژوهشی

بهترین ترکیب پارامترهای سفید کننده بر روی ویژگی های کمی و کیفی روغن های آفتابگردان و ذرت با استفاده از روش سطح پاسخ

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اطلاعات مقاله

تاریخ های مقاله :

تاریخ دریافت: ۱۴۰۲/۷/۱۱

تاریخ پذیرش: ۱۴۰۲/۸/۲۳

کلمات کلیدی:

روغن آفتابگردان;

روغن ذرت؛

بهینه سازی؛

پارامترهای بلیچینگ؛

روش سطح پاسخ.

راندمان بلیچینگ؛

ترکیبات زیست فعال

DOI: 10.22034/FSCT.20.140.169

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چکیده

فرآیند بلیچینگ در عملیات تصفیه روغن های خوراکی مهم است. در این مطالعه، پارامترهای بلیچینگ برای ارزیابی بهترین شرایط برای بهینه سازی بلیچینگ در روغن ذرت و آفتابگردان تغییر یافت. برای این منظور اثر زمان (۱۵، ۲۵، ۳۵، ۴۵ و ۵۵ دقیقه)، دما (۸۰، ۹۰، ۱۰۰، ۱۱۰ و ۱۲۰ درجه سانتیگراد) و غلظت رس سفیدکننده (۰,۴، ۰,۶، ۰,۸، ۱). و ۱/۲ درصد با استفاده از روش سطح پاسخ به عنوان طرح آماری در ۵ سطح با طرح مرکب مرکزی با هدف کاهش مصرف خاک سفیدکننده مورد ارزیابی قرار گرفت. فاکتورهای مورد مطالعه شامل کاروتنوئیدها، استرول ها، توکوفرول ها، رنگدانه و کاتیون ها بودند. پس از بهینه‌سازی، بهترین شرایط برای سفید کردن روغن‌های ذرت و آفتابگردان متفاوت بود. برای روغن ذرت، بهترین شرایط زمان ۳۹,۵۹ دقیقه، دمای ۱۰۳,۶۱ درجه سانتی گراد و غلظت خاک سفید کننده ۱٪ بود که ۶۷٪ از انتظارات ما را برآورده می کند. برای روغن آفتابگردان با هدف کاهش مصرف خاک رس سفیدکننده، بهترین شرایط زمان ۳۷,۴۹ دقیقه، دمای ۹۷,۵۳ درجه سانتی گراد و غلظت خاک سفید کننده ۱ درصد بود که ۷۰ درصد انتظارات ما را برآورده کرد. نتایج عملی با استفاده از روش سطح پاسخ تفاوت معنی داری با مقادیر تئوری به دست آمده نشان نداد و این نتایج را تایید کرد.