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The Effect of Different Concentrations of Activated Carbon with Bleaching Earth During Bleaching Process on Physicochemical Properties of Soybean Oil

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ABSTRACT: Considering the importance of bleaching earth with activated carbon in reduction of impurities in vegetable oils, this study was conducted to investigate the effects of bleaching process on physicochemical properties of the bleached soybean oil. The bleaching process was carried out with bleaching earth (1 % w/w) containing different concentrations of activated carbon (0.1 % up to 0.5 % w/w). The results indicated that bleaching process did not affect the structure of the oil while the percentage of free fatty acids (FFA) of the oil samples after bleaching was increased as compared to the blank. During bleaching, peroxide values of the samples and color were decreased. Since the vegetable oils have been shown to be the major sources in the diet therefore the refining particularly the bleaching process plays a decisive role in the production of high quality oil. Therefore this step is one of the critical control points (CCP) in ensuring the quality of the final product.

Keywords: *Activated Carbon, Bleaching Earth, Free Fatty Acids (FFA), Peroxide, Soybean Oil.*

Introduction

Soybean oil is the most important vegetable oil in the world, because it is abundant, inexpensive, and liquid due to its unsaturation. The color of crude soybean oil is amber yellow. Tocopherols are present in respective decreasing order, γ followed by δ, α and β tocopherols.

The presence of α linolenic acid in the oil affects both the stability and its flavor.

In comparison with other vegetable oils, the amount of phosphates is considerable, that is reduced by the degumming process. The main factors in the extraction unit include the capacity of oiling, the reduction of hexane solvent consumption, the energy, and the quality of extracted oil in terms of free fatty acid, the amount of hexane remaining in oil and the color of the pulp. Finally, the quality of the pulp based on moisture, oil residue and protein content are examined. (Camargo *et al*., 2011; Johnson *et al*., 2015; Yang *et al*., 2018)

The refining involves adjusting the operating set that is used to create an edible product that includes the steps of degumming (to remove phospholipids and mucilages), neutralizing or purifying the alkali (to remove free fatty acids), bleaching (to remove unwanted pigments that accelerate fat oxidation and lead to undesirable colors in the final product and

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removal of oxidation products namely hydroperoxides), this process is performed with bleaching earth or activated carbon, and the deodorisation step (removing volatile compounds and decomposed peroxides in order to improve the quality, taste and stability of the final product). (Camargo *et al.*, 2012; Dijkstra *et al.*, 2013; Jiang *et al.*, 2015).

The bleaching step is a subset of adsorption processes: This stage in fact is called oil purification by surface absorption method and a supplementary process for separating pigments and other natural impurities residues or created during maintenance procedures, preparation, extraction or early purification steps. The bleaching process occurs by a number of different mechanisms, including various chemical interactions; most often they improve the quality of the oil. These mechanisms include:

1. Adsorption, the mechanism that traps impurities occur in three different ways: Physical (with the involvement of Vander Waals forces): Chemical, (by chemical absorption, with an electrochemical bond to the surface of bleaching earth): By molecular sieve, (which during the filtration, traps the impurities under pressure in the bleaching earth).

2. Filtration, the mechanism removes suspended contamination or removes them physically. The physical action of removing suspended bleaching earth by simultaneous filtration, removes minor contamination absorbed by bleaching earth particles. Filter used in bleaching process include: Process filters (vertical and horizontal plate filters) and polisher filters (cartridges, plates).

3. Catalysis, the mechanism by which the contamination decompose through interaction with the surface of the bleaching earth. For example, peroxides are effectively reduced by interaction between bleaching earth and oil. (polymerized or decompose into volatile sub-oxidation products). By excessive heat and oxidation, pigments can form colored compounds that are hardly removed. If the color is stabled, the removal of the red color is very difficult only with the bleaching earth, and it is resistant to bleaching process, which results in a higher red color after deodorant step. (Kaynak *et al.*, 2004; Shahidi, 2005; Dijkstra., 2009)

In this way, the bleaching process plays a decisive role in the production of high quality oil. Due to the fact that it is not possible to isolate most of these components in the remaining steps, this step is one of the critical control points (CCP) in ensuring the safety and quality of the final product. Factors that affect the bleaching process include: oxygen, bleaching earth acidity, soap remaining in the oil, process temperature and contact time. In order to use a bleaching agent in the oil industry, there are three important factors: coast of color absorbent material, bleaching activity and the amount of oil is stored by the bleaching earth. (Kaynak *et al.*, 2004; Kuuluvainen *et al.*, 2015).

The most important color absorbent is bleaching earth. The natural bleaching earth, known as the Fuller's earth, contains hydrated aluminum silicate. The most common natural bleaching earth is Montmorillonite. Today instead of natural bleaching earth, bleaching earth activated with acids (sulfuric acid and chloridric acid) is employed. The crude materials for activated bleaching earth production are consisted of bentonite and Montmorillonite. Due to the action of sulfuric acid or chloridric acid, the surface of the bleaching earth develops, therefore their bleaching capacity is much higher than the natural bleaching earth, at the same time the absorbed oil increases at their special surface. The amount of oil absorbed by the natural bleaching earth is 20-25% w/w, while for activated bleaching earth is 35-40%w/w and for activated carbon is more than 40%w/w. Activated bleaching earth is

known as solid acids and have adsorption, catalytic and ionic exchange properties. The biggest advantage of this earth is to bleach from dark oils. On the other hand, some of the colors are destroyed only by activated bleaching earth, for example soybean chlorophyll which is an undesirable green color, due to its unstable acidic condition, it only disappears with activated bleaching earth. (Jahouach *et al.*, 2007; Kuuluvainen *et al.*, 2015; Vaisali *et al.*, 2015).

Activated bleaching earth has an imperfection that is hydrolysis of the neutralized oil and increase in the amount of free fatty acid in the oil. The most important catalytic property of bleaching earth is the decomposition of hydroperoxides from oxidation of oils by reaction of dehydration. Therefore, aldehydes, ketones and conjugated compounds are formed. Bleaching earth can catalyze the formation of a small amount of trans fatty acids. The amount of bleaching earth used varies according to the bleaching capacity and the type of oil. Usually for soybean oil with good quality, the amount of bleaching earth is 1%w/w. In general, the required amount of bleaching earth is between 3-15%w/w. (Naji *et al.*, 2010; Haji Hoseini *et al.*, 2014; Vaisali *et al.*, 2015).

Activated carbon that is very effective in the separation of green pigments and polycyclic aromatic hydrocarbons (PAHs). However, despite its high bleaching capacity it is not used alone because of its high cost, but usually 10-20 parts by weight of bleaching earth with one part of activated carbon are combined. Activated carbon in addition to its high bleaching capacity does not have a specific smell, which itself is an important advantage. On the other hand, the amount of oil stored by activated carbon is also high, and it is a good absorbent material for the remaining soap in refined oils. (Kuuluvainen *et al.*, 2015; Vaisali *et al.*, 2015).

The oldest bleaching method is the discontinuous bleaching of oil. Advantage of this method is simplicity of operation.

Continuous bleaching under vacuum makes the oil quality even better than the discontinuous under vacuum. On the other hand, in this method the bleaching earth becomes better mixed with the oil, and the time of contact between the bleaching earth and oil is reduced. As a result, the oil has a better quality, therefore even when activated bleaching earth is used the minimum amount of free fatty acid is formed. (Vaisali *et al.*, 2015)

Many previous studies on bleaching process have been carried out on physicochemical properties of vegetable oils. Ghavami *et al.* (2003) concluded that free fatty acids, peroxides, phospholipids, induction period, color, metals, non-saponifiable matters and tocopherols were reduced in the refining operations.

Haji Hoseini *et al.* (2014) indicated that there were significant reductions in peroxides and red and yellow colors of the oils. The induction period representing the oil resistance to oxidation was improved however the acidity was increased that might be due to the nature of the acid activated earth that has caused slight hydrolysis.

Abbasi *et al.* (2017) compared the conventional and ultrasonic waves in bleaching. By application of conventional bleaching a considerable reduction in chlorophyll and carotenoid contents of olive and sunflower oils were observed.

The most effective absorbent material is activated carbon in bleaching process. Due to the lack of proper information for the amount of activated carbon in bleaching process especially in the industry and studies have shown that almost none of the oil refineries in Iran use activated carbon due to its high cost, therefore the main goal in this research is to use activated carbon and bleaching earth and investigate the effect on physicochemical properties of soybean oil.

Materials and Methods

- *Materials*

Isooctane, methanolic solution of potassium hydroxide, sodium chloride, sodium hydrogen sulphate, acetic acid, potassium iodide, distilled water, standard thiosulfate solution, starch solution, ethanol, phenolphthalein, potassium hydroxide, sodium hydroxide were purchased from Merck (Darmstadt, Germany).

The neutralized soybean oil was obtained from Behshahr Company of Iran. The samples were stored at room temperature until required for the analysis.

A bleaching earth type (Tonsil 210 optimum) was purchased from Kanisaz-Jam Company, Iran, with the following characteristics (chemical mixture: Bentonite activated with acid, Color: white, Appearance: powder, Solubility: insoluble, Moisture percentage: 9, Free acidity in sulfuric acid: 0.66, Density: 500, pH: 3)

Activated carbon was purchased from Merck (Darmstadt,Germany), with the following characteristics (chemical mixture: Bituminous carbon, Size in millimeters: 0.5-2, Diameter of the particle in millimeters: 1.25, Moisture percentage: 2, the actual density of the particle in kilograms per liter: 0.81, Special surface area per square meter per kilogram: 1.2, Volume of the pore in milliliters per gram: 0.73, Pore diameter in nanometers: 2).

- *Bleaching Procedure*

In this research, sample a (blank) was not bleached. Bleaching of the other neutralized soybean oil samples was carried out according to the method described by Azadmard: Dutta with slight modification (Azadmard and Dutta, 2007), about 10 g of the neutralized soybean oil with 1 % w/w of bleaching earth and different levels of activated carbon (0.1 %, 0.2 %, 0.27 %, 0.3 %, 0.4 % and 0.5 % w/w) were added to a 250 ml round bottom flasks. Then, the flasks were attached to the rotary evaporator, mixed and heated to 85 ° C. The bleaching process was carried out for 1 h at the mentioned temperature under vacuum (1000 mbar) with the vigorous stirring of the mixture by the device (100 rpm). After the elapsed time, the mixture was cooled down and immediately filtered on a Buchner funnel under vacuum to remove the bleaching earth and activated carbon. The obtained samples consisted of (b[1], c[2], d[3], e[4], f[5], g[6] and h[7]).

- *Determination of fatty acid compositions of the oil samples*

Fatty acid methyl esters were formed according to the Iranian National Standard number 13126 and 2011: (ISO 12966-2).

- *Determination of free fatty acids (FFA) of the oil samples*

Percent free fatty acid indicating the degree of hydrolysis was determined according to the Iranian National Standard number 4178 and 2009: (ISO 660).

- *Determination of peroxide value of the oil samples*

Peroxide value presents the formation of peroxides that are the primary oxidation products and was determined according to the Iranian National Standard number 4179 and (ISO 3960: 2007).

[1] Bleached soybean oil with 1 % w/w of bleaching earth

[2] Bleached soybean oil with 1 % w/w of bleaching earth ,0.1 % w/w of activated carbon

[3] Bleached soybean oil with 1 % w/w of bleaching earth ,0.2 % w/w of activated carbon

[4] Bleached soybean oil with 1 % w/w of bleaching earth, 0.27 % w/w of activated carbon

[5] Bleached soybean oil with 1 % w/w of bleaching earth ,0.3 % w/w of activated carbon

[6] Bleached soybean oil with 1 % w/w of bleaching earth ,0.4 % w/w of activated carbon

[7] Bleached soybean oil with 1 % w/w of bleaching earth ,0.5 % w/w of activated carbon

- *Determination of the color of the oil samples*

The color, indicating the yellow, red, blue and white units was measured according to the Iranian National Standard number5110 and (ISO 15305: 1998).

- *Statistical analysis method*

The analysis of the data obtained from the physicochemical tests in soybean oil samples was performed based on a factorial experiment and a completely randomized design. Significant differences between the main effects in the investigated methods was considered by Duncan-test at the significant level of α=1 %. For the data analysis, MSTAT-C software (version 2.18, Michigan State University) was employed.

Results and Discussion

- *Fatty acid composition*

Changes in fatty acid composition of neutralized and bleached soybean oil samples were obtained using gas chromatography device in three replicates as presented in Table 1.

Regarding the average of three replications obtained from the data for each treatment, there was no significant change in the structure of fatty acids in bleached soybean oil as compared to the neutralized soybean oil (control) however slight changes in fatty acid composition was observed that was due to the oxidation of poly unsaturated fatty acids namely linoleic acid in oil samples.

Other previous studied have obtained similar observation(Ghavami *et al.*, 2003; Naji *et al.*, 2010; Haji Hoseini *et al.*, 2014; Abbasi *et al.*, 2017).

- *Percent free fatty acid (FFA) contents of the oil samples*

The results of three replications of free fatty acid (FFA) contents of soybean oil samples of soybean oil before and after bleaching are shown in Table 2.

As it can be observed except treatment 1, the percentages of free fatty acids (FFA) have been increased as compared to the blank sample.

Table 1. Fatty acid composition of neutralized and bleached soybean oil samples

	C_{14}:0	C_{16}:0	C_{18}:0	$C_{18:1}$	$C_{18:}$2c	C_{18}:3	C_{20}:0	C_{22}:0	C_{22}:1
Sample a[1]	0.06±0	11.81±0.08	4.53±0.15	22.88±0.24	52.53±0.49	7.31±0.28	0.02±0	0.35±0.18	0.02±0
Sample b	0.06±0	11.06±0.12	4.55±0.14	22.71±0.07	53.30±0.06	7.58±0.06	0.02±0	0.31±0	0.02±0
Sample c	0.06±0	11.56±0.14	4.44±0.09	22.88±0.24	53.04±0.27	7.20±0.10	0.03±0	0.32±0.01	0.02±0
Sample d	0.06±0.01	12.10±0.03	4.56±0.13	22.61±0.33	52.80±0.34	7.16±0.08	0.03±0	0.27±0.02	0.02±0
Sample e	0.06±0	10.08±0.44	4.56±0.24	22.78±0.19	52.87±0.47	6.72±0.39	0.02±0	0.39±0.01	0.02±0
Sample f	0.06±0	11.13±0.02	4.50±0.26	22.86±0.45	52.92±0.27	7.29±0.40	0.02±0	0.43±0.08	0.02±0
Sample g	0.06±0.01	11.14±0.01	4.54±0.23	22.90±0.45	52.76±0.39	7.38±0.24	0.02±0	0.36±0.34	0.02±0
Sample h	0.06±0	11.20±0.01	4.49±0.18	22.34±0.10	52.83±0.34	7.46±0.19	0.03±0	0.36±0.02	0.02±0

[1] Sample a is blank

The numbers are the mean of 3 repetitions ± standard deviations

Table 2. Changes in the percentage of free fatty acids in neutralized and bleached soybean oil samples

	Blank sample (a)	Sample (b)	Sample (c)	Sample (d)	Sample (e)	Sample (f)	Sample (g)	Sample (h)
Repeat 1	0.49	0.15	0.053	0.051	0.050	0.050	0.052	0.055
Repeat 2	0.051	0.16	0.051	0.048	0.051	0.051	0.052	0.050
Repeat 3	0.050	0.14	0.049	0.055	0.054	0.052	0.054	0.051
Total	0.15	0.45	0.153	0.154	0.155	0.153	0.158	0.156
Average±SD[1]	0.050±0.25	0.15±0.01	0.051±0.002	0.051±0.003	0.051±0.002	0.051±0.001	0.052±0.001	0.052±0.002

[1]SD= Standard Deviation

There is no significant difference between the percentages of free fatty acids of the blank sample and other treatments ($P < 0.01$).

Based on the other studies the increase in the percentage of free fatty acids might be attributed to the high temperature of the bleaching process (85 ° C) and the presence of activated bleaching earth. Because the activated bleaching earth with acid causes hydrolysis of the neutralized oil, therefore increasing the concentration of free fatty acid in the oil samples (Ghavami *et al.*, 2003; Naji *et al.*, 2010; Haji Hoseini *et al.*, 2014; Abbasi *et al.*, 2017).

The percentage of free fatty acids (FFA) of soybean oil samples are shown in Figure 1 before and after bleaching.

- *Peroxide values of oil samples*

The results of three replications showed the amount of peroxide in soybean oil samples before and after bleaching according to Table 3.

According to the findings shown in Table 3, during the bleaching process a decrease in the concentration of peroxide as compared to the blank sample is observed.

Based on Duncan's multiple range test at 5% probability level, it can be concluded that there is no significant difference in peroxide value between treatments. ($p>0.01$)

On the other hand, during the bleaching of samples, with different amounts of bleaching earth and activated carbon, a decreasing trend is observed in the peroxide values of the samples, which expresses the absorption of peroxide by the activated bleaching earth and activated carbon or the breakdown of peroxides at high temperature of the bleaching.

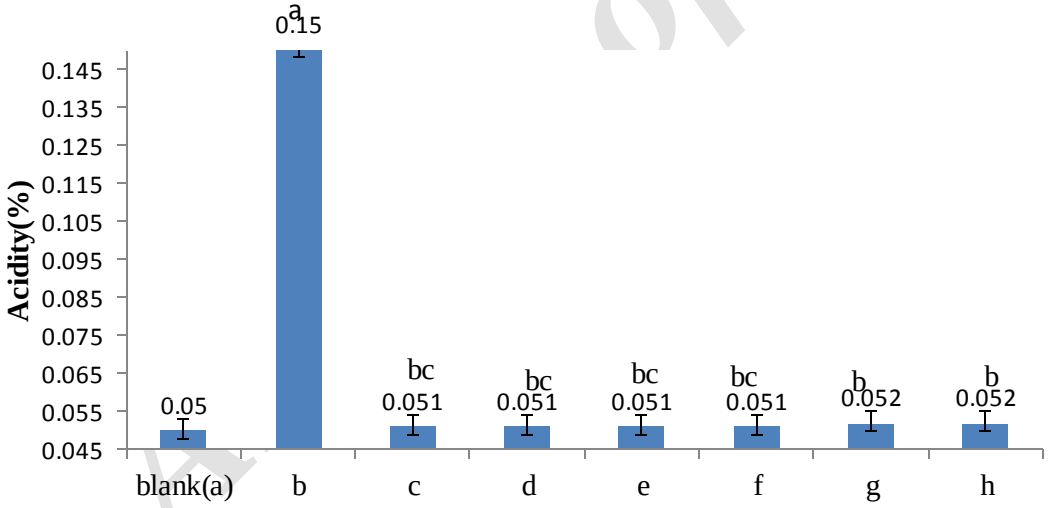

Fig. 1. The acidity of neutralized (control) and bleached soybean oils.

Table 3. Peroxide value (meq/Kg) changes of the neutralized and bleached soybean oil samples

	Blank sample (a)	Sample (b)	Sample (c)	Sample (d)	Sample (e)	Sample (f)	Sample (g)	Sample (h)
Repeat 1	2.71	2.64	2.45	2.40	2.35	2.29	2.12	2.09
Repeat 2	2.82	2.58	2.48	2.31	2.32	2.24	2.15	2.08
Repeat 3	3	2.63	2.40	2.38	2.30	2.28	2.19	2.05
Total	8.53	7.85	7.33	7.09	6.97	6.81	6.46	6.22
Average±SD[1]	2.84±0.14	2.61±0.03	2.44±0.04	2.36±0.04	2.32±0.02	2.27±0.02	2.15±0.03	2.07±0.02

[1]SD= Standard Deviation

As the amounts of activated carbon with the bleaching earth increases, a decrease in the peroxide value of the samples is observed, therefore the treatment containing 1%w/w of bleaching earth and 0.5%w/w of activated carbon had the lowest peroxide value as compared to the blank and other treatments. These results are consistent with the results of previous studies. (Ghavami *et al.*, 2003; Naji *et al.*, 2010; Haji Hoseini *et al.*, 2014; Abbasi *et al.*, 2017).

Peroxide values of soybean oil samples are shown in Figure 2 before and after bleaching.

- *Color of oil samples*

The results of three replications of red color in soybean oil samples before and after bleaching measured by the loviband method are shown in Table 4.

Based on the results of three replications, the red color of the treatments during bleaching process with bleaching earth and activated carbon decreased as compared to the blank sample.

Considering the one-way ANOVA and the significance of the effects of bleaching agents on the redness of the samples, the average of treatments were calculated using Duncan's method at 1% probability level.

According to the results in Figure 3, there was no significant difference between the treatments b, c, d and e in comparison to the control (P>0.01)

On the other hand, blank samples and treatments b, c and d were significantly different with treatments f, g and h. (P<0.01)

Also, there was no significant difference between treatment e and treatments f and g (P >0.01).

Significant differences were observed between treatment e and h in terms of red color (P<0.01).

Finally, there was no significant difference between treatments f, g and h in terms of redness (P >0.01).

The red color decreases during the bleaching process with activated bleaching earth and activated carbon, especially by increasing the activated carbon. Other previous studies have shown the red color has decreased in refining process especially in bleaching oils (Ghavami *et al.*, 2003; Naji *et al.*, 2010; Haji Hoseini *et al.*, 2014).

Red color values of soybean oil samples are shown in Figure 3 before and after bleaching.

The results of three replications of yellow color in soybean oil samples before and after bleaching by the loviband method are shown in Table 5.

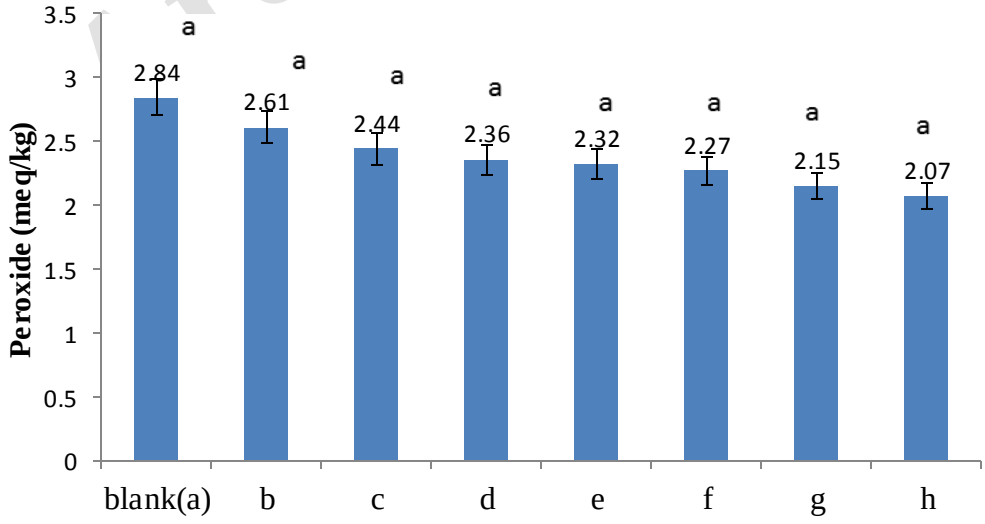

Fig. 2. Peroxide value of neutralized (control) and bleached soybean oils.

Table 4. The red color of the neutralized and bleached soybean oil samples

	Blank sample (a)	Sample (b)	Sample (c)	Sample (d)	Sample (e)	Sample (f)	Sample (g)	Sample (h)
Repeat 1	7.2	7	7	6.5	6.5	6	5	5
Repeat 2	7	7	6.8	6.5	6.5	6	5	5
Repeat 3	7	6.8	6.8	6.8	6.3	6.5	6.5	6
Total	21.2	20.8	20.6	19.8	19.3	18.5	16.5	16
Average±SD[1]	7.06±0.11	6.93±0.11	6.86±0.11	6.6±0.17	6.43±0.11	6.16±0.28	5.5±0.86	5.3±0.57

[1]SD= Standard Deviation

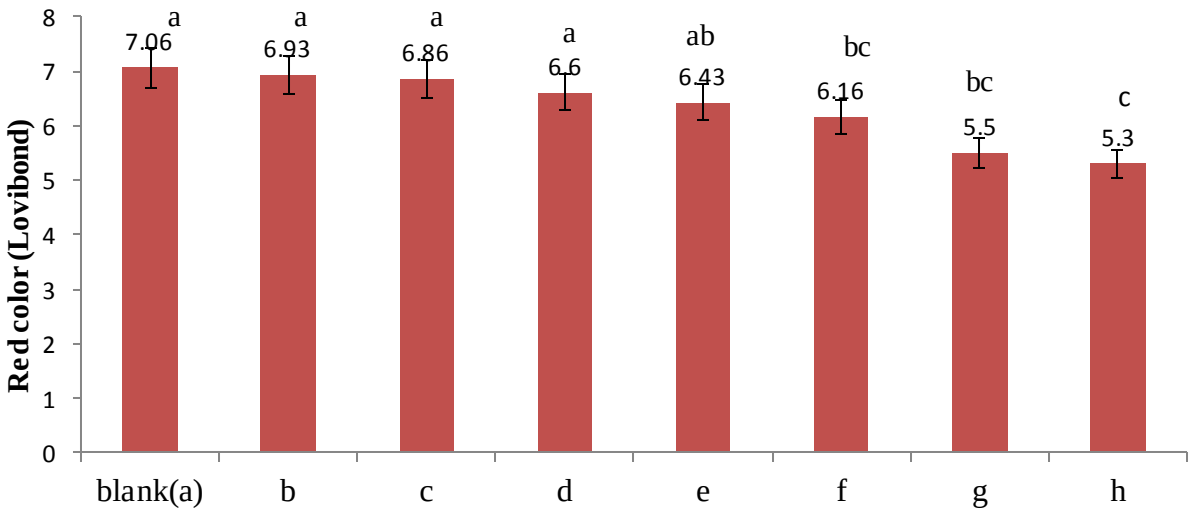

Fig. 3. The redness of neutralized (control) and bleached soybean oils.

Table 5. The yellow color of the neutralized and bleached soybean oil samples

	Blank sample (a)	Sample (b)	Sample (c)	Sample (d)	Sample (e)	Sample (f)	Sample (g)	Sample (h)
Repeat 1	70	68	64	63	63	60	56	51
Repeat 2	68	66	63	61	60	55	51	51
Repeat 3	70	68	66	63	60	60	60	56
Total	208	202	193	187	183	175	167	158
Average±SD[1]	69.33±1.15	67.33±1.15	64.33±1.52	62.33±1.54	61±1.73	58.33±2.88	55.66±4.50	52.66±2.88

[1]SD= Standard Deviation

According to the results, yellow color decreases as compared to the blank sample. This decrease is partly due to the increasing of activated carbon.

Regarding the one-way ANOVA and the significance of the effects of bleaching agents on the yellow color of the samples, the average of treatments were calculated using Duncan's method at 1% probability level.

According to the results of Table 5, the yellow color of blank sample had no significant difference with b and c treatments ($P > 0.01$) but with other treatments there was a significant difference ($P < 0.01$).

There was no significant difference between treatments b and c in terms of yellow color with treatments d, e and f ($P > 0.01$). However, there was a significant difference between treatments g and h at 1% level ($P < 0.01$).

Treatments d and e showed no significant difference in yellow color with treatments b, c and f ($P > 0.01$), but there was a significant difference between the blank and g, h treatments at 1% level ($P < 0.01$)

There was no significant difference between treatments f and d, e and g, h treatments ($P > 0.01$). However, this difference

was significant with the blank and treatment b and c (P <0.01).

Also, there was no significant difference between the yellow color of treatments g, h and treatment f (P >0.01), but between these treatments and the blank sample and treatments b, c, d and e there were significant differences in the amount of yellow color (P<0.01).

The amount of yellow color also decreased during bleaching process, and this decrease is due to the increase in the amount of activated carbon. The lowest amount of yellow color is related to treatment h (containing 1%w/w of bleaching earth and 0.5%w/w of activated carbon). Other previous studies have shown the red color has decreased in refining process especially in bleaching oils (Ghavami *et al.*, 2003; Naji *et al.*, 2010; Haji Hoseini *et al.*, 2014).

Yellow color values of soybean oil samples before and after bleaching are shown in Figure 4.

Conclusion

The bleaching step in vegetable oils refining is known as an adsorption process. Among the main factors of the bleaching process, it is possible to refer to bleaching earth with activated carbon. In this study, the effects of bleaching process with different amounts of activated carbon and bleaching earth on physicochemical properties of the bleached soybean oil were conducted. Based on the results of physicochemical tests, the fatty acid composition of the oil samples did not change as compared to the blank sample, while the percentage of free fatty acids (FFA) of the oil samples after bleaching increased. During bleaching of samples a decreasing trend was observed in the peroxide values of the samples and color levels were decreased during the bleaching process. Since vegetable oils have been shown to have a major role in the diet therefore the refining oils especially bleaching process plays a decisive role in the production of high quality oil.

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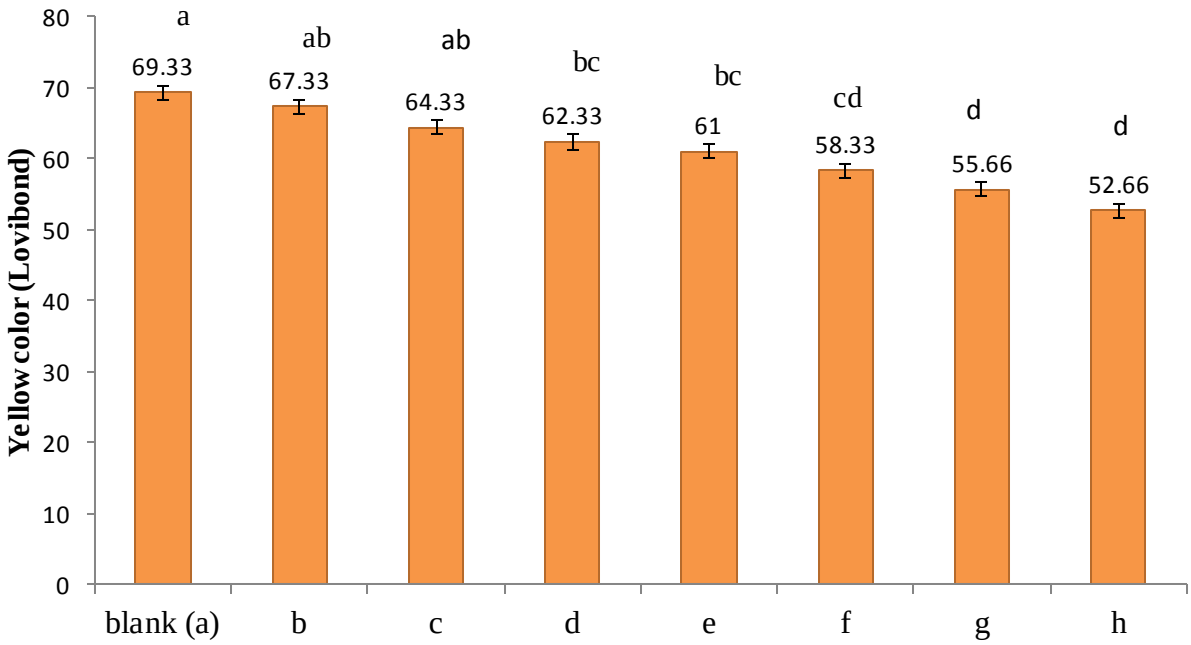

Fig. 4. The yellow color of neutralized (control) and bleached soybean oils.

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