

ENHANCING OF THE PERFORMANCE EFFICIENCY OF OILS BLEACHING EARTH

[24]

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ABSTRACT

Various percentages of citric acid [0.05, 0.1, 0.15 and 0.2%] were blended with the oil (w/v) before the bleaching of either soybean oil (SOO) or sunflower oil (SUO) to rise the performance efficiency of bleaching earth. Afterwards, activated bleaching earth was added to these oils for bleaching them at 1% level. Also, the same clay was used at 2% level for bleaching the above mentioned oils without using citric acid as a reference. The study revealed that better result for raising the bleaching efficiency of SOO was obtained with 0.15% citric acid. Whilst, 0.1% citric acid gave the best result pertaining the bleaching efficiency of SUO. Also, the data showed high reductions in the values of peroxide number, soap, conjugated dienes and trienes for the bleached oils with the increasing percent content of citric acid. While, the acidity values of the bleached oils (SOO and SUO) slightly increased with increasing citric acid percentages. On the other hand, using of citric acid at the ratios of 0.15 and 0.2% gave almost the same values in the bleaching efficiency of SOO. While, using of citric acid at the ratios of 0.1 and 0.15% gave nearly the same results of bleaching efficiency of SUO. Also, the data indicated that using of citric acid caused somewhat no changes in the fatty acid composition or their oxidative stabilities in the bleached oils. Therefore, it can be concluded that the reduction in the color of sunflower and / or soybean oils up to a levels of 33.3 and 36.7% is attainable by using either 0.1 or 0.15% citric acid prior to the bleaching step to decrease the imported content from bleaching earth to Egypt.

Key Words: Citric acid, Bleaching, Soybean and sunflower oils, Efficiency of bleaching earth.

1- INTRODUCTION

Bleaching clay is the natural adsorbent used for bleaching of edible oils (Brekke, 1990). Although degumming or refining will have removed phosphatides, free fatty acids and some impurities, soy-

bean oil still contains color bodies and various impurities that need to be removed before the color and taste of the finished oil could be acceptable to the consumers. Some of these remaining impurities are further reduced in quantity by a process called bleaching which is used

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to improve the color and oxidative stability of oil (**Erickson *et al* 1990 and De-Greyt & Kellens, 2000**). For many years, the bleaching process which consists of contacting the oil with an adsorbent materials, was considered purely from the standpoint of the removal of pigments (**Mag, 1990 and Hamm & Hamilton, 2000**). Another function of bleaching is to remove decomposes peroxides and to adsorb the aldehydes and ketones resulting from decomposition of peroxides (**Young, 1978 and Hui, 1996**). Also, **Park *et al* (2004)** stated that activated bleaching earth has been used to adsorb the dark color matter. In addition to **Gunstone *et al* (1994)** suggested that the bleaching process induces reduction in oxidation levels by breakdown of the hydroperoxide primary oxidation product on the adsorbent surface, followed by adsorption of the carbonyl compounds. Also, they added that the degree of bleaching varies somewhat with the type of finished product. A highly bleached oil is used to prepare the typical white-appearing shortening. More color a tinge of brilliant yellow is permissible for the oil used in margarine's, salad oil, salad dressings, and cooking oils (**Goebel, 1976 and Brekke, 1990**).

Citric acid can be used in refining process in various ways: to convert soaps to the more easily removed free acids and to act as a metal chelating agent (**Law and Berger, 1984**). Also, they added that citric acid is usually added to the oils in the form of 30-50% aqueous solution. On the other hand, **Hamm and Hamilton (2000)** indicated that citric acid decomposes rapidly above 150°C. According to the results reported by **Norris (1964); Goebel (1976) and Brekke (1990)**, the required dosage of bleaching

earth used for the oils bleaching varies between 1.0 and 4.0%.

Bleaching is by far the most expensive process in refining in terms of the operating costs of a bleaching plant due to the relatively high cost of the bleaching clays (**Hamm and Hamilton, 2000**). According to Chamber of Food Industry, Egyptian Industries Federation (**The Egyptian Industry Ministry, 2003a**), the production volume from oils and fats during 2002/2003 was about 750.000 tons. On the other hand, bleaching clays are imported to Egypt from some countries. In Egypt, the percentage of bleaching clay used for some oils bleaching usually ranges from 1 to 2% from the oil weight. Therefore, the imported content from bleaching earth to Egypt ranged from 7500 to 15000 tons during 2002/2003 for bleaching the whole 750000 tons. As for, the average price of imported activated bleaching earth is about 525 \$ USA per ton (**The Egyptian Industry Ministry, 2003 b**). While, the cost of adding citric acid in this work is very poor that ranges from 1 to 1.5 \$ USA per ton. Hence, if citric acid can be used (as a solution) before the bleaching with 1% activated bleaching earth, it will reduce the imported content from activated bleaching earth to about the half (from 3750 to 7500 tons) and also, it will decrease the production cost of oils to about 1.96-3.92 million \$ USA per year.

The ultimate aim of this study was to rise the performance efficiency of activated bleaching earth by using citric acid before the bleaching of soybean and sunflower oils with 1% activated bleaching earth that will induce improvements in the color of both oils and the same time to decrease the imported content of activated bleaching earth to Egypt to about the

half quantity, beside the production cost of these oils will be reduced .

2- MATERIAL AND METHODS

2.1. Materials

Refined soybean and sunflower oils were obtained from El-Ekhawa Company for Oils Processing, Saddat City, Menoufia Governorate, Egypt. While, activated bleaching earth (ABE) was brought from Cairo Oils and Soap Company, El-Aiat factory, Giza, Egypt which imported from Süd- Chemie Indonesia, Indonesia. Citric acid and all chemicals were purchased from El-Gomheria Company for Pharmaceutical, Cairo, Egypt.

2.2. Bleaching treatments

2.2.1. Preparation of citric acid

Concentrated solution of citric acid [CA] (30% w/v) in warmed propylene glycol ($50 \pm 2^\circ\text{C}$) was prepared and diluted directly by oils to the concentration percentages of 0.05, 0.1, 0.15 and 0.2% (w/v) before the bleaching process.

2.2.2. Procedure of the oils bleaching

Refined soybean and sunflower oils were separately bleached under nitrogen laboratory according to the method described by **Rolando (1991)** with some modifications as follows: Various percentages of citric acid (0.05, 0.1, 0.15 and 0.2% of the oil weight [w/v]) (the concentration of citric acid solution was 30%, w/v) were individually added to each oil (500g) before the bleaching, then the oil was heated to $95 \pm 2^\circ\text{C}$ before adding 1% activated bleaching earth (ABE).

The mixture was then heated with stirring at 250 rpm for 30 min with the maintenance of the same temperature ($95 \pm 2^\circ\text{C}$) during the bleaching . Thereafter, the hot slurry (oil + clay) was cooled to $50 \pm 2^\circ\text{C}$ and filtrated through filter paper (Whatman No.1) to separate the spent clay from the bleached oil. The bleached oils were analyzed, whereas, 2% ABE was used for bleaching the same oils (soybean and sunflower oils) without using citric acid as a reference according to the above method.

2.3. Methods of analysis

2.3.1. Determination of some physico-chemical analysis of activated bleaching earth

pH value (10% suspension) was measured using pH meter, Model HI-9321, Hanaa, Instruments while, acid content was determined according to the methods reported by **Anthony and Ogugua (1988)**. Whereas, moisture content was estimated by Electric oven at 105°C for 3 hr.

2.3.2. Determination of some physical and chemical properties in soybean and sunflower oils

Moisture and volatile matter (%), acidity (%) as oleic acid, peroxide number (meq.O₂/1kg oil) and soap content (ppm) in soybean and sunflower oils were determined according to the methods described in the **A.O.C.S. (1997)**. While, the color values in both oils were measured by Lovibond Tintometer, Model E using 5.25 inch cell. Total color [Yellow + 10 (Red + Blue)] and the percent color reduction were calculated using equation

of **Krishnan (1975)**, whereas conjugated dienes ($k_{232 \text{ nm}}$) and conjugated trienes ($k_{270 \text{ nm}}$) of these oils were measured according to the methods reported in the **IUPAC (1987)** at 232 and 270 nm, respectively, using U.V.-Vis. Spectrophotometer, Model Labmed, 120-02. As well as, refractive index of the above oils was measured by using Refractometer (NIRL-3-Poland) according to **A.O.C.S. (1997)** method. While, the oxidative stabilities of the same oils were estimated according to the method described by **Tsaknis *et al* (1999)** using Metrohm Rancimat 679 at 100°C with air flow rate of 20 L/h.

2.3.3. Methylation of soybean and sunflower oils

The methyl esters of soybean and sunflower oils were prepared (using benzene: methanol : concentrated sulfuric acid (10 : 86 : 4) and the methylation was performed at 90°C for one hour according to the method reported by **Ludy *et al* (1968)**.

2.3.4. Identification and determination of the fatty acid methyl esters in soybean and sunflower oils

The fatty acid methyl esters of the above oils were separated and quantitatively determined using Gas-liquid chromatography (Pye-Unicam PRO- GC) according to **Zygodlo *et al* (1994)** method.

3- RESULTS AND DISCUSSION

Quality of the bleached oil is based upon free fatty acids content, Lovibond color and oxidative stability (**Brekke, 1990 and Mag, 1990**).

3.1. Some physical and chemical properties of the refined soybean and sunflower oils

Some physical and chemical characteristic of the refined soybean oil (SOO) and sunflower oil (SUO) were determined and the results are represented in Table (I). From the data, it is appear that refined SOO and SUO had low content of moisture and volatile matter (m & vm). The maximal level of m & vm recommended by **The Egyptian Standard Specifications (1993)** is not excess than 0.1% for pure edible oils, whilst, the levels of acidity and peroxide number were inside the range reported by **The Egyptian Standard Specifications (1993)** for pure edible oils which limited that the levels of acidity and peroxide number must be not exceed than 0.2% and 10 meq.O₂/kg oil, respectively. While, SOO had the highest total color (TC) than in SUO. This increment could be related to high content of red and blue units in SOO than in SUO. Soap content of the above mentioned oils was between 38 and 53 ppm, respectively.

The soap content normally accepted is 50 ppm for pure edible oils (**The Egyptian Standard Specifications, 1993**). As to, the conjugated dienes and trienes in both oils (SUO and SOO) were in the range of 0.17 to 0.19 and 0.05 to 0.06, respectively. Also, refractive index (RI) of the aforementioned oils was 1.4736 and 1.4728, respectively. The above data for refined SOO and SUO are in conformance with **Hamm and Hamilton (2000)** but not agree with other researcher (**Hui, 1996**). These variations are nearly due to the difference in the processing procedure.

Table I. Some physical and chemical properties of the refined soybean and sunflower oils.

Oils	Moisture and volatile matter (%)	Acidity (%) as oleic acid	Peroxide number (meq. O ₂ /Kg oil)	Lovibond color			Total color	Soap content (ppm)	Conjugated dienes (at 232 nm)	Conjugated trienes (at 270 nm)	Refractive index (at 25°C)
				Y	R	B					
Soybean	0.1	0.07	0.9	35	8.9	3.8	162	53	0.19	0.06	1.4728
Sunflower	0.09	0.04	0.8	35	7.1	0.4	110	38	0.17	0.05	1.4736

Where : Y = Yellow; R = Red; B = Blue.

3.2. Some analyses of activated bleaching earth

Activated bleaching earth (ABE) is one of adsorbent commonly used for oil bleaching (Brekke, 1990). The results in Table (II) are shown some analyses of ABE. From these data, it can be found that the moisture content of ABE was 12.1%. This datum is within the range reported by DeGreyt and Kellens (2000) who stated that the water content in ABE usually varies between 10-20%. On the other hand, Gunstone *et al* (1994) cited that ABE contains about 8-12% of free moisture. Also, the results indicated that pH value and acid content in ABE were 3.1 and 0.3%, respectively. These data are somewhat close to that found by Hui (1996).

Table II. Some analyses of activated bleaching earth.

pH (10 % suspension)	Moisture (%)	Acid content (%)
3.1	12.1	0.3

3.3. Effect of using citric acid on the fatty acid composition of the bleached soybean and sunflower oils

The identifications of soybean and sunflower oils by Gas-liquid chromatography are given in Table (III). From the obtained results, it can be seen that these oils were rich in unsaturated fatty acids with linoleic acid being the major unsaturated fatty acid. Also, the findings showed that no obvious differences prevailing among the fatty acids of the bleached soybean oil samples (that treated and untreated with CA before the bleaching) were recorded. The contents of total saturated fatty acids (TSFA) in the bleached SOO were between 13.5 and 14.15%, whilst the amount of total unsaturated fatty acids (TUFA) were in the range of 84.5 to 86.3%. Also, the same trend was observed for the bleached sunflower oil. The amounts of TSFA in the bleached SUO were in the range of 9.7 to 11.62%, while TUFA were between 88.2 and 89.0%. These results indicated that using of CA at different ratios before the

bleaching caused slight changes in the fatty acids composition of soybean and sunflower oils. Accordingly, these findings of the fatty acids composition of both bleached SOO and SUO are within the range reported by **Takagi & Ando (1991)**; **Gunstone & Padley (1997)**; **Hamm & Hamilton (2000)** and **Judde *et al* (2003)**.

3.4. Impact of using citric acid on some physical and chemical properties of the bleached soybean and sunflower oils

Effect of using various percentages of citric acid before the bleaching with either 1 or 2% activated bleaching earth (ABE) on some physical and chemical characteristics of soybean oil (SOO) and sunflower oil (SUO) is shown in Table (IV). From these results, it is evident that there are interrelationship between citric acid content and soap level on the degree of the acidity in the bleached SOO and SUO which their acidities slightly increased with increasing of using citric acid content before the bleaching with 1% ABE compared with that obtained in the oil that bleached with 1% ABE without using of CA before the bleaching (control) and simultaneously reduction in soap content was observed, which may be due to CA that induced breakdown in the ester bonds of the residual soap, so free fatty acids were released. These findings agree with those found by **Law and Berger (1984)** who stated that CA can be used in refining process to convert soaps to more easily removed free acids. According to **Anonymous (1993)**, activated bleaching earth can lead to increase free fatty acids level during the bleaching. Also, the data tabulated in the same table

reveal that addition of CA before the bleaching of the above mentioned oils occurred reductions in their values of peroxide number compared with that obtained in the control mentioned oil. On the other hand, the results reported by **Aker (1990)** and **Hui (1996)** indicated that the bleaching process induces reduction in oxidation levels by decomposition of hydroperoxide components.

The results of conjugated dienes and trienes are presented in Table (IV). These results indicate that using of CA before the bleaching of SOO and SUO with 1% ABE induced decrements in the values of conjugated dienes compared with that obtained in the control oil. These data are in accordance with those obtained by **Brekke (1990)** and **DeGreyt and Kelens (2000)** who pointed out that the bleaching step removes conjugated diene compounds, resulting from degradation of peroxide by dehydration. As to, refractive index of the above mentioned oils slight variations was recorded.

Also, the obtained results showed that the values of acidity, peroxide number, soap, conjugated dienes and refractive index in the same oils that bleached with 2% of the same bleached earth (ABE) without using citric acid were somewhat the same as those obtained in the same oils which treated with citric acid (at the ratios of 0.1 and 0.15% CA for SUO and SOO, respectively) before the bleaching with 1% ABE.

Therefore, using of citric acid before the bleaching of SOO and SUO with 1% ABE induced improvements in some physical and chemical properties of these oils and also reduced the used content from ABE to about the half quantity.

From the above results, it can be concluded that the best results for bleaching

of SUO and SOO were obtained when using 0.1 and 0.15% citric acid, respectively before the bleaching with 1% ABE.

3.5. Influence of using citric acid on the oxidative stability of bleached soybean and sunflower oils

The oxidative stability of oils and fats is one of the most important factors determining their shelf life (Anwar *et al* 2003). The oxidative stability of SOO and SUO that treated with various percentage of CA before the bleaching with either 1% or 2% ABE was determined by programmed Metrohm Rancimat and the results are listed in Table (V). Rancimat is used to probe the oxidative stability in oils (Juarez *et al* 1998).

Table V. Effect of citric acid addition on the oxidative stability of bleached soybean and sunflower oils.

Rancimat induction period (hr)		
The used dosage of citric acid (%)	Soybean oil	Sunflower oil
	After the bleaching with 2% activated bleaching earth	
Non	8.48	6.41
After the bleaching with 1% activated bleaching earth		
Non	8.25	6.0
0.05	8.43	6.14
0.1	8.55	6.25
0.15	8.6	6.5
0.2	8.71	-

From the results, it can be indicated that slight increments were recorded in

rancimat induction period (RIP) of both oils that treated with CA compared with that obtained in the control sample. Citric acid is sometimes used as chelating agent in the oils which help removing metal ions and oxygen scavengers and also it can regenerate spent antioxidant (Gunstone *et al* 1994 and Hui, 1996).

3.6. Influence of using citric acid on the color of bleached soybean and sunflower oils

Effect of citric acid addition before the bleaching on the color of SOO and SUO is recorded in Table (VI). From these results, it is obviously that the color reduction percentage of SOO increased from 12.5% to 36.7% as content of citric acid increased from 0.05% to 0.15%. This result is nearly due to the decrement in the values of Lovibond red and blue that decreased from 7.2 and 2.1 in the control sample to 4.2 and 0.4 in the same oil that treated with 0.15% CA, respectively. Also, the color reduction percentage in SUO increased from 12.1% to 33.3% as content of CA increased from 0.05% to 0.1%. This improvement may be attributed to the reduction in the unites of Lovibond red and blue that reduced from 6.3 and 0.1 in the control sample to 3.1 and zero in the same oil that treated with 0.1% CA. These improvements in the color are probably inasmuch as using of CA which increased the bleaching efficiency of ABE by the reduction of some impurities (such as soap). Soap induces inhibition in the performance efficiency of bleaching earth (Hui, 1996). These results are in line with the limitation issued by **The Egyptian Standard Specifications (1993)** that stipulated that Lov-

ibond red color of refined and bleached
oil

should not be exceeded 7.0 units at yellow color of 35. Also, the data in the same table show that the refined SOO treated with 0.15% CA prior to the bleaching with 1% ABE was the best treatment of bleaching that recorded decrement in total color from 128 to 81, with highly bleaching efficiency (color reduction was 36.7%). While the best treatment for bleaching of SUO was recorded at using of citric acid at the ratio of 0.1% which recorded reduction in total color from 99 to 66, with highly bleaching efficiency (color reduction was 33.3%). On the other hand, the results indicated that using 0.15 and 0.2% CA before the bleaching of SOO gave somewhat the same results since the bleaching efficiency of their color reductions recorded 36.7 and 38.3%, respectively. Also, using 0.1 and 0.15% CA before the bleaching of SUO gave somewhat the same results with respect to the bleaching efficiency since their color reductions were 33.3 and 35.3%, respectively. From the above results, it is clear that using of CA up to 0.1% before the bleaching of SUO and up to 0.15% for SOO induced improvements in the color of these oils compared with that obtained in the control oil.

4- CONCLUSIONS

From all the above data in this work, it can be concluded that addition of citric acid to the refined sunflower and soybean oils up to 0.1 or 0.15% from the oil weight induced improvements in some parameters of these oils since the color became lightly, beside the values of soap, peroxide number, conjugated dienes, and trienes recorded reductions.

Subsequently, it can be recommended that citric acid can be added to the refined oils (sunflower and soybean oils) up to 0.10 or 0.15% from the oil weight (the concentration of citric acid was 30%) before the bleaching with 1% activated bleaching earth to rise the performance efficiency of bleaching earth and to decline the imported content from activated bleaching earth to Egypt to about the half as well as to decrease the production cost of both oils (soybean and sunflower oils).

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رفع كفاءة عمل تراب تبيض الزيوت

[24]

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حموضة تلك الزيوت المبيضة بزيادة كمية حمض الستريك المستخدمة. على الجانب الأخر، أوضحت الدراسة أن استخدام حمض الستريك بنسب 0.15 ، 0.2% أعطت إلى حد ما قيم متشابهة في كفاءة تبيض زيت فول الصويا. في حين أشارت الدراسة أن استخدام نسب 0.1 و0.15% من حمض الستريك أعطت إلى حد ما تشابهه في قيم كفاءة تبيض زيت عباد الشمس. كذلك أوضحت النتائج أن استخدام حمض الستريك لم يؤثر إلى حد ما في تركيبة الأحماض الدهنية للزيوت المبيضة وكذلك على ثباتها الأوكسیدی. وعليه يمكن أن نستنتج أن الانخفاض في لون زيوت عباد الشمس وفول الصويا يمكن الوصول إليه بمعدل يصل إلى 33.3 - 36.7% عند استخدام حمض الستريك بنسب 0.1 أو 0.15% قبل مرحلة التبيض وذلك بغرض خفض كمية تراب التبيض المستوردة إلى مصر.

تم استخدام حمض الستريك بنسب مختلفة (0.05 ، 0.1 ، 0.15 ، 0.2 % من وزن الزيت) قبل تبيض زيوت فول الصويا وعباد الشمس بغرض رفع كفاءة عمل تراب التبيض. اعقب ذلك تم إضافة 1% تراب تبيض نشط لتبيض تلك الزيوت. كذلك استخدم 2% من نفس تراب التبيض في تبيض نفس الزيوت والتي لم تعامل بحمض الستريك وذلك كمرجع. أشارت الدراسة أن أفضل النتائج تم الحصول عليها في كفاءة تبيض زيت فول الصويا عند استخدام حمض الستريك بنسبة 0.15% في حين سجلت نسبة 0.1% حمض الستريك أفضل النتائج في كفاءة تبيض زيت عباد الشمس. كذلك أظهرت الدراسة أنه بزيادة الكمية المستخدمة من حمض الستريك يزداد الانخفاضات في قيم البيروكسيد والصابون والمركبات التبادلية الثنائية والثلاثية بالزيوت المبيضة في حين حدث زيادة بسيطة في قيم

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