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Investigation of activated carbon obtained from the liquid products of pyrolysis in sunflower oil bleaching process

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Abstract

Decolourization process of neutralized sunflower oil was carried out with activated carbon products obtained from pyrolysis liquid products. The effect of parameters such as time, temperature, moisture and dosage on bleaching sunflower oil was investigated. It was found that activated carbon samples are an effective adsorbent for decolourization process of sunflower oil. It was determined that the ability of adsorption of the activated carbon samples at 20 °C temperature is higher than that of the bleaching earth which is used for this purpose. The analysis results directly prove that activated carbons remove the unwanted pigments and residues from sunflower oil. Moreover, activated carbon sample A2 has a more positive effect on pigment removal than other activated carbon samples (A1 and A3). 1% of activated carbon sample A2, 0.01% sunflower oil moisture, 20 °C temperature and 30 min contact time are optimal values in a sunflower oil bleaching process. Therefore, these activated carbon samples can be used to bleach sunflower oil instead of bleaching earth.

Keywords Sunflower oil · Neutralization · Bleaching · Bleaching earth · Activated carbon

Introduction

Vegetable oils and fats are important constituents of human foodstuffs. Approximately 70% of all oils produced in the world are of vegetable origin. In modern society, the consumers do not like to use crude oils directly without proper processing due to unpleasant colour and odour. Generally, vegetable oils contain minor components, which affect the end-product quality by alteration of its taste, colour and process efficiency. Water, free fatty acids (FFA), pigments, phosphatides, partial glycerides, oxidation products, and some trace elements such as iron, copper, sulphur and halogens are undesirable substances of oil. These impurities can be removed at various steps in the conventional chemical refining, which includes degumming, neutralization, washing, drying, bleaching, filtration and deodorization. Therefore, efficient industrial processing involves removing these impurities with the least possible effect on the desired components and the least possible loss of neutral oil [1–4].

Bleaching is a critical step in the physical and chemical refining process of edible oils and selecting the optimal condition for the bleaching process depends on the quality and type of crude oil. The bleaching process is always misunderstood as just a mere process of decolourization of oils. In fact, bleaching is a process of selective removal of pigments and impurities by the physical and chemical interaction of an adsorbent with an oil to improve its quality. This process refers to the art of removing not only the colour pigments but also trace metals and various organic impurities that promote oxidation [5–7].

The bleaching process is applied after degumming and neutralization in the chemical refining and it is more appropriately referred to as adsorption treatment. Crude edible oil still contains undesirable substances such as FFA, gummy materials and colouring matters. Colouring matters are due to the presence of pigments in the crude edible oil. These pigments are carotenoids, chlorophyll, gossypol and related compounds. These impurities from crude oils are removed by using the materials with a strong adsorption power. The usual method of bleaching is the adsorption of the colouring substances on an adsorbent material. Refiners have a wide choice of adsorbents for using and their main criteria are cost and performance of the adsorbent materials. Examples of bleaching agents



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are natural bleaching earth, acid-activated bleaching earth, synthetic silicates, synthetic resins and activated carbons. Bleaching agents normally possess a large surface that has a more or less specific affinity for pigment-type molecules, thus removing them from oil without damaging the oil itself. Acid-activated bleaching earth is sometimes called bentonite; it is the adsorbent material that has been used most extensively. This substance consists primarily of hydrated aluminium silicate. Usually, bleaching earth does not remove all the colour-producing materials; the majority of them are actually removed by thermal destruction during the deodorization process. Anhydrous silica gel and activated carbon are also used as bleaching adsorbents [8–12].

Activated carbon in its broadest sense includes a wide range of processed amorphous carbon-based materials. It is not truly an amorphous material but has a microcrystalline structure. Activated carbons have highly developed porosity and an extended inter particulate surface area. Their preparation involves two main steps: the carbonization of the carbonaceous raw material at temperatures around 800 °C in an inert atmosphere and the activation of the carbonized product. Thus, all carbonaceous materials can be converted into activated carbon, although the properties of the final product will be different, depending on the nature of the raw material used, the nature of the activating agent and the conditions of the carbonization and activation processes. The bleaching action seems to be due to the large adsorbent surface of the carbon. This large surface held in a small volume would influence the surface tension of the compounds with which it will come into contact, thus causing adsorption. Activated carbon possesses extremely high surface area, often in excess of 1000 m²/g. Much of that surface area is, however, associated with micropores—that is, pores < 20 Å (< 2 nm) in diameter. The surface area associated with mesopores–pores 20 to 500 Å (2 to 50 nm) in diameter—is considerably lower (typically in the range 10-100 m²/g). Most liquid-based applications (including fats and oils purification) involve the adsorption of high-molecular-weight contaminants whose molecular dimensions prevent penetration into micropores; therefore, activated carbon containing significant mesoporosity is most desirable in these applications. There are a large number of commercial grades of activated carbon that are used for adsorbing gasses and vapours, odours and colouring materials [13–17].

Studying high adsorption capacity of bleaching materials in the bleaching of sunflower oil is a very critical and necessary step for developing the bleaching process. The purpose of this study was to determine bleaching performance of carbon materials, especially activated carbons which were produced from pyrolysis of the liquid products in chemical vapour deposition (CVD) unit and activated acids and also to determine the effects of parameters such as temperature,

time, moisture and dosage on the sunflower oil's bleaching process.

Chemical vapour deposition is a deposition method used to produce high-quality solid materials, typically under vacuum. CVD is the most popular method for obtaining pure carbon nowadays. Thermal decomposition of hydrocarbons is achieved in the presence of metal catalysts in this unit. Hydrocarbon vapours transfer through a tubular reactor at sufficiently high temperature, so hydrocarbons decompose and carbon grows on the catalyst bed in the reactor. Volatile by-products are removed by gas flow through the reaction chamber. Control physical conditions such as pressure, temperature, carrier gas and chamber material in reactor play a big role in the production of pure carbon. Carrier gases are also enhancing surface reaction and improving reaction rate, thereby increasing deposition of C₅ hydrocarbon fractions onto the substrate. Advantages of pyrolysis in CVD have more than conventional pyrolysis techniques. Especially, short process time, rapid and energy-efficient heating process increase production rates and help to obtain more pure carbon [22, 23].

Materials and methods

Materials

Activated carbon sample A1 obtained from C₅ hydrocarbon fraction of pyrolysis in the presence of a manganocene catalyst and on the surface of MgO at 850 °C, for 20 min and Argon atmosphere in CVD unit. Activated carbon sample A2 obtained from C₅ hydrocarbon fraction of pyrolysis in the presence of a ferrocene catalyst and on the surface of Al₂O₃ in 920 °C, for 20 min and Argon atmosphere in CVD unit. The chemical agents (Mg, Al, and other ions) are removed by washing with sulphuric acid, which creates a highly porous structure (1.3 cc/g for A2) and then dried under vacuum. A commercial activated carbon sample A3 and bleaching earth sample A4 were supplied from different companies. The sunflower oil (SF) was obtained from Baku Oil Factory in the Azerbaijan Republic. All chemicals and solvents were of HPLC grade and obtained from the commercial source (Merck, Germany) [18–20].

Methods

The bleaching of sunflower oil was carried out according to AOCS Cc 8f-91 [21]. Initially, 100 ml of neutralized sunflower oil was poured into a 500 ml Erlenmeyer flask. Then 0.3 g of activated carbon and bleaching earth samples were added to the oil and mixed. The temperature was maintained at 20 °C for 10 min. After 10 min, the mixture was filtered through a Whatman No 1 filter paper. This process





was repeated with the dosage of 0.6, 0.9, 1.2 and 1.5 g to determine the effect of dosage. The effect of temperature was studied at 40, 60, 80 and 100 °C. The effect of time was done by varying the time (15, 20, 25, 30, 35 and 35 min).

Analysis of activated carbon and oil quality

The characterization of the adsorbents includes estimation of various parameters such as proximate analysis (moisture content, volatile matter content and fixed carbon content), bulk density, BET surface area, SEM, porosity, and pH. The surface area of adsorbents was found by BET surface area analyzer. The pore structure of activated carbon was observed through SEM analysis. The porosity and pore volume was estimated using mercury porosimeter.

Colour, FFA, peroxide value (PV), anisidine value, chlorophyll and some trace metals were analysed in the filtered oil samples. Free fatty acids were determined by titration according to the AOCS standard method Ca 5a–40. Colour measurements were made by means of Lovibond Tintometer Model F and Lovibond PFX-i 995 (Wilts, England) by using 5 1/4 cuvette (AOCS Official Method Cc 13e–92). The concentration of chlorophyll pigment was determined spectrophotometrically, using AOCS Official Method Cc 13d–55. The anisidine value was determined with Bruker MPA according to the AOCS Cd1e–01. The moisture determination is made in an oven, calculating the moisture and volatile materials by weight difference (AOCS, Ca 2d–25). The concentration of soap was determined according to AOCS, Cc 17–79.

Results and discussion

The pyrolysis product analysis had been conducted in "TruSpec Micro" and it was found that the elemental composition of A1 and A2 samples consists of 97.2-98% C, 0.5-1% H, 0.4% N and 0.5% O. After CVD, these samples were activated with sulphuric acid which removed residual matters from carbon and created porous structure. Then there is the combination of treating, heating and extensive washing to remove any remaining chemicals. Activated samples' elemental composition consists of more than 99.2% carbon. As is known, the adsorptive properties of activated carbons are settled not only by its pore structure but also by its chemical composition. The length and diameter of carbon particles were measured by scanning electron microscope (SEM) and it was determined that in 500 times enlarged description (Fig. 1a) activated carbon materials are visible in porous tubular structure. These tubes and their pore volume are visible clearly in 1000 times magnified micrograph (Fig. 1b). And it is determined that the diameter of macropores of activated carbons is larger than about 50 nm. Their volume in the activated carbon is 0.4 cm³/g and their surface area is 1.8 m²/g. The effective diameters of transitional pores are 40 nm. Their specific surface area is around 6% of the total surface area of the activated carbons. The micropore volume is around 0.3 cm³/g and the specific surface area of microspores is over 90% of the total specific surface area [14].

Chemical and physical properties of activated carbons are determined in respect of their adsorptive properties. The results are shown in Table 1.

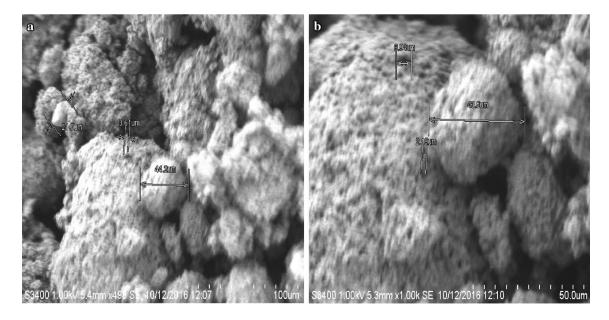


Fig. 1 a, b Scanning electron micrographs of the activated carbon at 500–1000 magnification



 Table 1
 Typical properties of activated carbons and commercial powdered activated carbon

Property	A1	A2	A3
Moisture (%)	0.7	0.6	7.2
pH (water extract)	4.2	3.9	7.45
Surface Area (m ² /g)	1210	1260	1200
Pore Volume (cc/g)	1.1	1.3	1.1
Bulk Density (kg/m ³)	498	513	504
Wt% passing 100 mesh	97	98	95
Wt% passing 325 mesh	82	86	83

Adsorptive purification, in its most general sense, involves the use of adsorbents to remove undesirable constituents and contaminants from fats and oils by adsorptive mechanisms. The major factors that affect the bleaching process, aside from the actual quality of incoming oil, are the conditions applied during the contact between oil and adsorbent. Assuming good quality oil (i.e., low in phospholipids, soaps and free fatty acids) and the proper choice of adsorbent, other important factors include moisture of oil, absence of air during bleaching and bleaching temperature and time. From the standpoint of traditional bleaching processes, the major objectives of water degumming and alkali refining/ water washing are to reduce phosphatides and soaps to low levels; these constituents are easily adsorbed by bleaching materials and interfere with the adsorption of pigments and other bleaching functions. For water degummed/alkali refined oils, the desired levels are as follows: phosphorous <3 ppm, soaps <40 ppm and free fatty acids <0.05%. The levels of FFA, soaps, phosphorous before and after bleaching are shown in Table 3.

The removal of the main colour pigments in neutralized sunflower oils with three types of activated carbons and bleaching earth has been examined at different temperatures, moisture, time and dosage and the most appropriate bleaching condition has been chosen.

The properties of sunflower oil which is crude, neutralized and bleached with earth and activated carbons

are shown in Table 2. 1% concentration, 30 min contact time, 20 °C bleaching temperature for A1 and A2 and 1.5% concentration, 30 min contact time, 100 °C temperature for A3 and A4 as bleaching parameters are selected for Table 2. Colour, FFA content and PV and other parameters of bleached oil were determined to see oil quality after the bleaching process. As is known, anisidine value increases and peroxide value decreases with increasing temperature for bleaching with earth. Table 2 shows that FFA, chlorophyll, colour and peroxide values decreased while using A1 and A2 activated carbons. Furthermore, it can be seen that FFA and colour values have decreased more using activated carbon A2 compared to bleaching earth A4.

The presence of high levels of chlorophyll in sunflower oil creates very serious processing problems for the refiner. As is known, pigments are removed by increasing the amount of bleaching earth used; this is expensive and not always completely effective. However, comparison of the chlorophyll levels after bleaching with activated carbons and earth sample indicated that activated carbon A2 can be used instead of bleaching earth A4.

Many researchers also reported decreased peroxide values but increased anisidine values as bleaching temperature was increased. This conforms to expectation because the anisidine test is diagnostic for shorter chain aldehydes (including alkenals and dienals), which are secondary oxidation products of peroxide decomposition. Based on the discussions thus far, it might be concluded that higher bleaching temperatures are desirable because increased pigment removal and peroxide decomposition could be achieved with the lesser amounts of bleaching adsorbents. The problem, of course, is the increased levels of anisidine generated by higher bleach temperatures. It is clear that bleaching with activated carbons A1 and A2 has been conducted at 20 °C; that is why the anisidine number is increased less compared to A3 and A4. Moreover, in American trading rules, the maximum red colour of bleached sunflower oil is 2.5 and the maximum yellow colour is 25. In this study, all adsorbents showed red and yellow colour values below this figure. Moreover, it can

Table 2 Crude, neutralized, and bleached sunflower oil's quality parameters

Samples of sunflower oil	Crude sunflower oil	Neutralized sunflower oil	Oil bleached with A1	Oil bleached with A2	Oil bleached with A3	Oil bleached with A4
Peroxide value, (mmolO ₂ /kg)	7.84	1.8	1.34	1.1	1.56	0.98
FFA, (%)	1.2	0.11	0.08	0.09	0.12	0.11
Anisidine number	2.26	3.6	4.19	4.3	5.53	5.27
Color, (red/yellow)	3.9/30	3.8/30	1.5/10	1.4/8	1.4/12	1.8/20
Chlorophyll, (mg/kg)	0.46	0.31	0.02	0.01	0.03	0.01
Soaps, (ppm)	N/A	26	N/A	N/A	N/A	N/A
Phosphorous, (ppm)	124	8	N/A	N/A	N/A	N/A





be seen that all adsorbents have absorbed soap and phosphorous from oil totally [4].

Effect of moisture

Understanding the effect of oil moisture on bleaching is somewhat problematic because both bleaching adsorbents and incoming oils can have different levels, to begin with. Many workers have reported that some amount of moisture can have a beneficial effect in bleaching of oils. Their works show Lovibond red and yellow, and chlorophyll levels improve with increasing moisture up to 0.6%. Beyond that level, however, additional moisture is deleterious from the standpoint of colour reduction.

As is known, pigments in the vegetable oil give it a special colour. This colour is measured by Lovibond method. The basic plan of the system is that each glass is marked with the number of unit glasses to which it is equivalent colourimetrically; that is, the number of unit glasses through which light from the source must be passed to produce the same colour. Furthermore, the unit glasses of the red, yellow and blue scales are related so that combinations of all three kinds of glasses of equal numbers on the Lovibond scales give a nearly neutral filter, that is, produce little or no change in chromaticity from that of the source.

In our studies, we use neutralized oils because their moisture level was very low. Initially, we use dry oil, whose moisture level was 0.01% and its colour 3.8 red. This process

repeated at 0.1, 0.2, 0.3 and 0.4% levels for the determination of the effect of moisture in the bleaching process. It can be seen from the Fig. 2 that the best Lovibond red level is shown in the lowest moisture level, at 0.01%, so 0.8 red colour is obtained after bleaching. The increase of moisture impacts bleaching process negatively and the worst result is shown in the 0.4% moisture level. That is, when the moisture content is increased to 0.4%, the bleaching process begins to deteriorate and red colour decreases from 3.8 to 1.5 for A1, A2, and A3. From Fig. 2, it is clear that bleaching earth A4 bleaching performance was more affected by high moisture level. So, firstly adsorbents had adsorbed moisture from oil and began to loss of its special adsorption properties. These studies show that too much moisture is clearly detrimental and optimal moisture for the adsorption process in the range 0.01 - 0.1%.

Moreover, it can be seen from Table 3 that the activated carbon sample A2 adsorbed colour pigments from the dry sunflower oil more and 1% of adsorbent and 0.01% moisture are optimal values in the sunflower oil bleaching process.

Effect of temperature

The effect of temperature on the bleaching process is shown in Fig. 3a. The bleaching process was performed within 30 min, using 1% A1 and A2 and 1.5% A3 and A4 at different temperatures ranging from 20 to 100 °C. The result obtained by bleaching using 1% sample A2 was

Fig. 2 Effect of moisture on the bleaching process (30 min contact time, 1% dosage of samples)

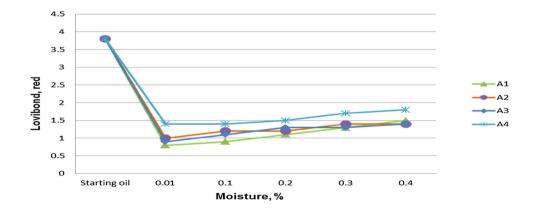
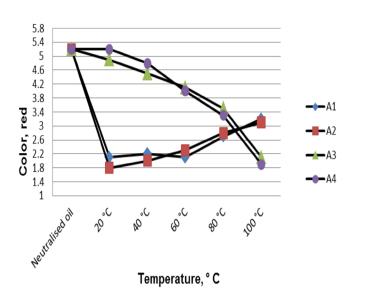
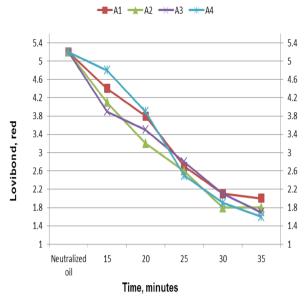


Table 3 The changes in Lovibond red and yellow value at different moisture level

Moisture, (%)	Lovibond, red			Lovibond, yellow		
	0.5% dosage of A2	1% dosage of A2	1.5% dosage of A2	0.5% dosage of A2	1% dosage of A2	1.5% dosage of A2
0.01	2.3	1.8	1.8	12	8	7
0.1	2.6	2.1	1.9	16	11	9
0.2	2.7	2.3	2.1	18	12	9
0.3	2.9	2.4	2.2	21	12	10
0.4	3.3	3.0	2.6	22	13	11







- (a) The dependence of colour from temperature
- (b) The dependence of colour from time

Fig. 3 Effect of temperature (a) and time (b) on the bleaching process (1% dosage for A1, A2 and 1.5% dosage for A3 and A4)

obtained at 1.5% using A4. Therefore, 1.5% of A3 and A4 was compared with 1% of A1 and A2 in the temperature chart. The red colour value was obtained 2.6 while using 1% of A4. According to the literature, bleaching temperature typically ranges from 90 to 125 °C. Because, oil viscosity decreases with increasing temperature resulting in the better dispersion of particles, improved adsorbent oil interactions and flowability [4, 5]. It can be seen from Fig. 3a that colour reduction was found to be increased with increasing temperature from 20 to 100 °C for the commercial activated carbon A3 and earth A4 samples, so increasing temperature increased activation of A3 and A4. But contrary to expectations, the highest bleaching was obtained at 20 °C temperature for A1 and A2 activated carbon samples.

The figure shows that temperature does not influence the activation and future increase in temperature tends to darken the colour of the bleached oil. The reason for that may be that temperature does not affect activated carbon (A1 and A2) surface properties. Activated carbon releases adsorbed particles to oil when increasing temperature from 20 to 100 °C and then these particles burn and give the dark colour to oil. On the other hand, these activated carbons can interact with oil very well at low temperatures. Furthermore, it is deduced from Fig. 3a that sample A2 shows much higher bleaching capacity when compared to the other activated carbons and bleaching earth. A2 was reduced red colour from 5.3 to 1.8 at 20 °C temperature, but A4 obtained this value at 100 °C, so A2 is more energy efficient than A4.

Effect of contact time

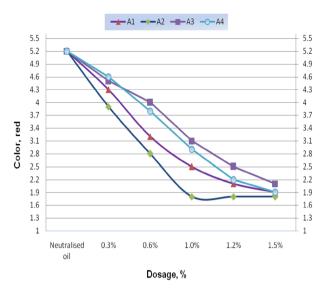
The effect of bleaching contact time for activated carbon and bleaching earth samples also was investigated. To study the effect of contact time on bleaching performance of the activated carbons, the bleaching process was performed at different times ranging from 15 to 35 min, with 1% A1, A2 and 1.5% A3, A4 adsorbents. The results are shown in Fig. 3b. The variation of bleaching performance showed an increase in the first 20 min and a slight increase up to 30 min when the increase of time had no significant effect on the adsorbent modification and thus the bleaching process. Further increase in contact time tends to darken the colour of the bleached oil. Thus, the optimum activation time was about 30 min for all adsorbents. This result is in agreement with the literature, which states that the contact time for effective bleaching typically ranges from 15 to 45 min, with 20 to 30 min being most common. From Fig. 3b, it is seen that the bleaching curve of activated carbon samples A1 and A2 is similar to that of commercial bleaching earth. However, A1 and A2 can be considered more effective adsorbent as it is used 0.5% less than other adsorbents. Thus, it is clear that activated carbons can be used to bleach vegetable oil in place of commercially available earth.

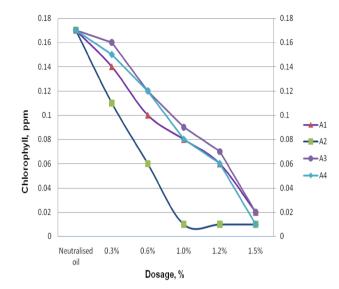
Effect of dosage

The amount of adsorbent is the most important factor in the bleaching process. The effect of adsorbent dosage for both bleaching earth and activated carbons was illustrated









- (a) The dependence of colour from dosage
- (b) The dependence of chlorophyll from the dosage

Fig. 4 Effect of dosage on the bleaching process (30 min contact time, 0.01% moisture and 20 °C for A1, A2 and 100 °C for A3 and A4)

in Fig. 4a. The results clearly indicate that activated carbon sample A2 reach an optimum value at 1% adsorbent dosage; a further increase in adsorbent dosage has no significant effect on it. This could be explained by the fact that the adsorption equilibrium has reached the maximum between the adsorbent/oil mixtures. However, the colour absorption increased by increasing the number of other adsorbents A1, A3 and A4 in the bleaching process and 1.5% earth sample A4 showed the same result with the 1% activated carbon sample A2. Figure 4b shows the effect of dosage on chlorophyll reduction when using bleaching earth and activated carbons to bleach sunflower oil. This graph also makes the point that adsorbent A2 has a more positive effect on pigment removal than others.

Conclusion

Thus, the adsorption of the color pigments in neutralized sunflower oil with different types of activated carbon and commercial earth material has been examined at different temperatures, contact times, moisture and bleaching material dosages. The results indicated that activated carbon A1 and A2 have great potential in the removal of colour pigments and residues from sunflower oil. Another advantage for the use of activated carbons A1 and A2 in the processing of sunflower oil is the decrease in free fatty acids and the effective overall performance of sample A2 was found higher than bleaching earth and other activated carbon samples. According to the results of oil analysis, 1% concentration, 30 min contact time, less than 0.1% moisture and 20 °C bleaching

temperature were optimal bleaching parameters for activated carbon A1 and A2. These activated carbon samples can be used as an adsorbent in bleaching of sunflower oil besides bleaching earth in the domestic market.

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