Recovery of used frying sunflower oil with sugar cane industry waste and hot water

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Abstract The main goal of the current investigation was to use sugar cane bagasse ash (SCBA) and to compare its adsorption efficiency with Magnesol XL as synthetic adsorbents to regenerate the quality of used frying sunflower oil. In addition, to evaluate the effect of water washing process on the quality of used frying oil and the treated oil. The metal patterns of sugar cane bagasse ash and Magnesol XL were determined. Some physical and chemical properties of unused, used frying and used–treated sunflower oil were determined. Sunflower oil sample was heated at $180\text{ }^{\circ}\text{C}\pm 5\text{ }^{\circ}\text{C}$, then frozen French fries potato were fried every 30 min. during a continuous period of 20 h. Oil samples were taken every 4 h. The filter aids were added individually to the used frying oil at levels 1, 2 and 3 % (w/v), then mechanically stirred for 60 min at $105\text{ }^{\circ}\text{C}$. The results indicate that all the filter aids under study were characterized by high levels of Si and variable levels of other minerals. The highest level of Si was recorded for sugar cane bagasse ash (SCBA) was 76.79 wt. %. Frying process caused significant ($P\leq 0.05$) increases in physico-chemical properties of sunflower oil. The treatments of used frying sunflower oil with different levels of sugar cane bagasse ash and Magnesol XL caused significant ($P\leq 0.05$) increase in the quality of treated oil, however the soap content of treated oil was increased, therefore, the effect of water washing process on the quality of used frying and used–treated sunflower oil was evaluated. The values of soap and Total polar compounds after water

treatment were about 4.62 and 7.27 times as low as that for sunflower oil treated with 3 % sugar cane bagasse ash (SCBA). The results of the present study indicate that filtration treatment with different levels of sugar cane bagasse ash (SCBA) regenerated the quality of used sunflower oil and possess higher adsorbing effects than the synthetic filter aid (Magnesol XL) in removing oil oxidation products. The current study also recommends using hot water treatment for improving the quality of used frying oil.

Keywords Sugarcane · Industrial waste · Frying · Ash · Adsorbent · Quality and water washing

Introduction

Deep-fat/oil frying is extensively used in food processing both industrially and at home, and fried potato products are one of its largest applications. Deep-fat frying is one of the oldest and most popular food preparation methods. As reported by Garayo and Moreira (2002), the economy of commercial deep fat frying has been estimated to be \$6 billion year⁻¹ in the United States and at least twice that amount for the rest of the world. Fried foods have a desirable flavor, color and crispy texture, which make deep-fat fried foods very popular with consumers (Boskou et al. 2006). Deep-fat frying is a process of immersing food in hot oil with a contact among oil, air and food at a high temperature of 150 to 190 °C (Yamsaengsung and Moreira 2002). The simultaneous heat and mass transfer of oil, food and air during deep-fat frying produces the desirable quality of fried foods. In the presence of oxygen, moisture, trace elements and free radicals, physiochemical reactions such as thermoxidation, hydrolysis, polymerization, isomerization or cyclization take

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place at the high temperatures of the frying process, thus leading to the decomposition of frying oil and formation of monomeric, polymeric, primary and secondary oxidative compounds, thereby affecting the quality of oil and fried product (Andrikopoulos et al. 2002). The oxidized products of fatty acids give off-flavors and odors (hydrolytic rancidity) to the frying medium and fried foods (Lin et al. 2001; Romero et al. 2007; Farag and El-Anany 2007). Several studies reported that using vegetable oils in prolonged frying processes may be a principle or strong reason for inducing or forming mutagenic or carcinogenic diets (Hamilton et al. 1997; Coultate 2002; Gouveia De Souza et al. 2004; Pavia and Gordon 2005; Khalil et al. 2009). The mutagenicity/carcinogenicity of such diets may be due to the fact that the repeatedly used vegetable oils in frying processes undergo degradation by oxidative reactions that lead to form lipid peroxidation (Nagao et al. 1997; Lampe 1999). High intakes of diets containing lipid peroxidation component may be associated with the generation of free radicals (Schut et al. 1997; Lampe 1999; Coultate 2002; Guardiola et al. 2008). These reports indicate the necessity to remove peroxides and these toxic compounds from deteriorated fried oils. In this respect, there are various methods that can be used to remove these products such as filtration through special membranes (Mulflur and Munson 1987; Subramanian et al. 2000) and by using different adsorbent materials including both natural and synthetic adsorbents. Usually, active forms of carbon, calcium, silica, alumina, and magnesia are major constituents of such products (Yates and Caldwell 1992; Lin et al. 1999; Farag and El-Anany 2006; El-Anany and Ali Rehab 2008 and Farag et al. 2009). By these methods, the treated oils can be used again in deep-fat frying process. Many reports have been appeared on the production of low cost adsorbents using cheaper and readily available materials (Babel and Kurniawan 2003; El Nemr et al. 2008). In this regard, Sugar cane is one of the most common raw materials used in sugar and ethanol production. Sugarcane is one of the major crops grown in over 110 countries and its total production is over 1500 million tons (Deepchand 1986). After the extraction of all economical sugar from sugarcane, about 40–45 % fibrous residue (FAO 2006; Partha and Sivasubramanian 2006; Rasul et al. 1999) is obtained, which is reused in the same industry as fuel in boilers for heat generation leaving behind 8–10 % ash as waste (Payá et al. 2002), known as sugarcane bagasse ash (SCBA). The SCBA contains high amounts of un-burnt matter, silicon, aluminum and calcium oxides (Deepchand 1986). The main goal of the current investigation was to use sugar cane bagasse ash (SCBA) and to compare its adsorption

efficiency with Magnesol XL as synthetic adsorbents to regenerate the quality of used frying sunflower oil. In addition, to evaluate the effect of water washing process on the quality of used frying and used-treated sunflower oil.

Materials and methods

Refined sunflower oil (25 Kg) was obtained from Sila edible oil Co. S. A. E., Kom Osheim, El-Fayoum governorate, Egypt. The oil peroxide and acid values were 0.40 (meq/kg oil) and 0.10 (mg KOH/g oil), respectively.

Sugar cane bagasse (SCB)

Sugar cane bagasse (SCB) was obtained from Hawamdiah sugar Refinery Company, El-Hawamdiah, Egypt.

Magnesol XL

Magnesol XL (Hydrous, white, amorphous, and odorless synthetic magnesium silicate) was obtained from Magnesol Product Division, Reagent Chemical and Research, Inc. Houston, Texas, USA.

Chemicals

All chemicals were used in the current study were obtained from Sigma Chemical Company (England, London Ltd., Pools).

Preparation of Sugar cane bagasse ash (SCBA)

Sugar cane bagasse (SCB) was first washed thoroughly with distilled water to remove the dust particles, then dried in an oven at 100 °C for 72 h. The dried product was ground in a grinder (Perten, USA) to a particle size of about 190 nm, placed in a crucible, carbonised in air in a muffle furnace (Furnace 5000; Thermolyne, Iowa, USA) at 650 °C for 12 h and then cooled to room temperature in a desiccator and stored in glass screw-capped bottles.

Determination of metals

The metal contents (Si, Ca, Fe, Al, Mg, K and Na) of various filter aids were determined using a Perkin-Elmer Model 3300 atomic absorption spectrophotometer (The Perkin-Elmer Corporation, Norwalk, USA) after digestion with fluoro-boric acid according to the procedure given by Bernas (1968). As follows: A known weight of the ground sample (ca 50 mg) was placed in a teflon

vessel of the acid digestion bomb. Aqua regia (mixture of HNO_3 (1 part) and concentrated HCl (3 parts), 0.5 ml) followed by HF (3 ml, 48 %) were added to the sample. The contents of digestion bomb were heated at 110 °C for 40 min. After cooling, the contents of the bomb were quantitatively transferred into a 100 ml polyethylene beaker, with deionised water (5 ml). Boric acid (2.8 g) and deionized water (5 ml) were added to dissolve any precipitate. This solution was quantitatively transferred to a 100 ml polyethylene volumetric flask, completed to the mark with deionised water and used for the metal determination. A blank was conducted simultaneously using all reagents except the filter aid. Standard stock solutions (1000 ppm) of the elements: Si, Ca, Fe, Al, Mg, K and Na were prepared from their metals or metal salts using deionised water and few drops of concentrated HCl then stored in polyethylene bottles.

Frying process

Sunflower oil (25 kg) was placed in a stainless steel pan of electric fryer (45 cm depth x 52 cm length x 40 cm width, Frymaster L.L.C., Shreveport, Louisiana, U.S.A) and heated at 180 °C $\pm$ 5 °C. Then 500 g of frozen French fries potato were fried every 20 min. 250 ml of frying oil was withdrawn every 4 h and the continuous frying period was 20 h. The oil samples were left to cool down then stored at -18 °C for physico-chemical analysis and filtration process.

Filtration process

The filter aids were mixed individually with 2500 ml of used frying oil levels 1, 2 and 3 % (w / v), then mechanically stirred for 60 min at 105 °C. The slurry was vacuum filtered through a Whatman No 41 filter paper (Whatman International Ltd, Maidstone, UK). Vacuum filtration facilitated flow of oil through the filter paper. The untreated used frying oil was vacuum filtered through a Whatman No 41 filter paper.

Water washing process

Oil sample (1500 ml) was heated at 70 °C and hot water 80–85 °C was added (1/1 v/v). The mixture was mixed for 15 minutes with the aid of a glass rod, and then left at room temperature for cooling. The upper oil phase was separated by centrifugation at 10,000 rpm for 20 min. The centrifuged oil was left in contact (stirred) with the anhydrous Sodium sulfate for ca. 5 min, then vacuum filtered through a Whatman No 41 filter paper.

Yield and weight loss measurement

The yield is first measured by the following equation

$$\% \text{ Yield} = \text{Weight of treated oil} / \text{Initial weight of untreated oil} \times 100$$

$$\text{Weight losing \%} = 100 - \% \text{ Yield}$$

Quality assurance tests

These tests were conducted on unused, used frying oil and treated oil samples.

Physical properties

Refractive index

Refractive index of the oil was determined according to the A.O.A.C. method (2000) using Abbe - refractometer (NYRL - 3 - Poland).

Colour

Lovibond tintometer (Tintometer Limited Solstice Park, Amesbury, UK) was used to measure the colour of the oil samples under investigation, the yellow glass filter was fixed at 30 and the intensity of red glass colour was measured according to the A.O.A.C. method (2000).

Viscosity

Brookfield LV viscometer Model TC-500 (Brookfield Engineering Laboratories Stoughton, MA, USA) was used to measure the viscosity of the oil samples at 30 °C. according to the method described by Saguy et al. (1996).

Chemical properties

Acid value

Acid value was determined according the A.O.A.C. method (2000) as follows. A Known weight (2 g) of the oil was dissolved in a neutral ethyl alcohol (30 ml). The mixture was boiled on a water bath for 2 min and then titrated with potassium hydroxide solution (0.1 N).

Peroxide value

The peroxide value was determined according to A.O.A.C method (2000) A known weight of the oil sample (2.5 g)

was dissolved in a mixture consisted of glacial acetic acid : chloroform (30 ml, 3 : 2, v/v) then freshly prepared saturated potassium iodide solution (1 ml) was added. Distilled water (30 ml) was added then titrated slowly with sodium thio-sulphate solution (0.1 N).

Thiobarbituric acid value (TBA)

The method of Sidwell et al. (1954) was conducted to determine the TBA value as follows. A known weight of oil (3 g) was dissolved in a carbon tetrachloride (10 ml) followed by the addition of TBA reagent (10 ml, 0.67 % TBA in 50 % acetic acid). The mixture was transferred to a separatory funnel and the aqueous layer was drawn into a test tube and immersed in a boiling water bath for 30 min. The absorbance of the developed pink colour was then recorded at 532 nm against a blank reagent.

Insoluble polymer content

The insoluble polymers were determined according to the method outlined by Wu and Nawar (1986) as follows. One gram of oil was added to methanol (125 ml) containing 1 % H₂SO₄. The mixture was boiled under a reflux condenser for 2 h and cooled to room temperature. The methanol insoluble were filtered and washed with methanol until no sulphuric acid remained. The washed insoluble polymers were dissolved in petroleum ether (25 ml) and transferred to a pre-weighed flask. The solvent was then evaporated under a stream of nitrogen and the flask was again weighed.

UV spectroscopic indices

An aliquot of the sunflower oil samples (0.1 mL) was dissolved in methanol (3 mL), vortexed and the absorbances at 232 and 288 nm were recorded for conjugated dienes and trienes, respectively as mentioned by Kates (1972)

Total polar compounds (TPC) content

Total polar compounds in sunflower oil samples were measured by column chromatography according to the method described by Walting and Wessels (1981). A slurry of (25 g) silica gel in 80 ml light petroleum-ethyl ether (90: 10, v/v) was poured above the wad of cotton wool which was placed in the bottom of the column (2.1 cm×54 cm) filled with 30 ml light petroleum-ethyl ether (90 : 10 , v/v). An aliquot of oil sample (2.5 g) in 20 ml light petroleum-ethyl ether (90: 10, v/v) was poured at the top of the column. The non-Total polar compounds of oils were eluted with 150 ml light petroleum-ethyl ether (90: 10, v/v) within 60–70 min by adjusting the eluent flow rate. The solvent was evaporated using a rotary evaporator at 60 °C until constant weight

was obtained. Total polar compounds were calculated by difference as follows:

$$\text{Total polar compounds, \%} = ((E - A)/E) \times 100$$

Where: A = weight of non-Total polar compounds fraction—E = weight of sample.

Soap content

Soap content was determined according to method described by Nelson (1973).

Statistical analysis

Data were statistically analyzed in completely randomized design in factorial arrangement according to the procedures outlined by Gomez and Gomez 1986 and the treatments means were compared by least significant differences (L.S.D) and Duncan multiple range using SPSS program package. Data are presented in text and tables as means of three determinations.

Results and discussion

Mineral patterns of synthetic and natural filter aids

Table 1 shows the mineral content of Magnesol XL (synthetic filter aid) and sugar cane bagasse ash (SCBA), sugar cane industry waste). All the filter aids under study were characterized by high levels of Si and variable levels of other minerals. The highest level of Si was recorded for sugar cane bagasse ash (SCBA) was 76.79 wt. % followed by Magnesol XL was 70.92 wt. %. Sugar cane bagasse ash (SCBA) had also other minerals were Al₂O₃, CuO, Fe₂O₃, CaO, K₂O, MgO, and Na₂O (1.87 wt.%, 0.58, wt.% 0.48, 5.81 wt.%, 2.42 wt.% 2.37 and 0.15 wt.%, respectively). Magnesol XL had the highest level of Mg was 17.78 wt. % however it was free from Cu and K. Several authors studied the mineral contents of various natural and synthetic filter aids. For instance, Jacobson (1967) mentioned that the major constituents of filter aids (natural and synthetic adsorbents) were carbon, calcium, silicon, alumina and magnesia. These results are in good agreement with our previous study (Farag and El-Anany 2006), in which we determined the minerals in Magnesol XL to be Si (34.75 %), Mg (27.33 %), Ca (4.36 %), Na (1.92 %) and Fe (1.09 %). Ajay Goyal et al. 2007; Fri'as et al. 2007; Samsudin et al. 2009 determined the minerals of sugar cane bagasse ash (SCBA), their analysis indicate that sugar cane bagasse ash (SCBA) contain Si (from 50.36 to 74.79 %), Al (from

Table 1 Mineral pattern of Magnesol XL and sugar cane bagasse ash (SCBA)

adsorbent	SiO ₂ (%)	CuO (%)	Fe ₂ O ₃ (%)	CaO (%)	K ₂ O (%)	MgO (%)	Na ₂ O (%)
Magnesol XL	70.9 ^b	0	0.25 ^b	6.0 ^b	0	17.7 ^a	4.5 ^a
	±1.5	0	±0.03	±0.50	0	±1.6	±0.35
(SCBA)	76.7 ^a	0.5 ^a	0.3 ^a	8.9 ^a	2.3 ^a	7.4 ^b	3.2 ^b
	±1.9	±0.08	±0.07	±0.40	±0.22	±1.30	±0.26
LSD at 0.05=	4.07	0.12	0.12	1.02	0.35	3.30	0.69

Data are expressed as mean±SD. Values given represent means of three determinations. Values followed by the same letter are not significantly different ($p<0.05$).

1.78 % to 4.38 %), Fe (from 0.45 % to 2.25 %), Na (from 0.15- to 2.05 %), K (from 0.52 % to 3.10 %) an Ti (0.02 %).

Quality parameters of unused and used frying sunflower oil

Refractive index is a good diagnostic parameter for the degree of oil unsaturation and this value increased by increasing number of conjugated double bonds in the oil. The data in Table 2 show that frying process led to gradual increases in the refractive index. This increase in refractive index became significant ($P\leq 0.05$) after 16 and 20 h of frying process. It is well known that frying process caused

conversion some of the non-conjugated double bonds into conjugated ones. This case led to an increase in the refractive index. Colour of oil is one of the most eminent physical properties which attract the consumer appellant. In general this property affects the colour of fried food. The colour of fresh and used frying oil samples was measured at the fixed yellow glass slide (35) and variable red glass slides. The colour of fresh sunflower oil was clear yellow; its colour value was 2.43. Frying caused gradual and significant ($P\leq 0.05$) increase of colour value. The value of colour in used sunflower oil at the end of frying process was about 4.69 times as high as that in unused sunflower oil. The colour of

Table 2 Changes in some physico-chemical properties of sunflower oil during frying (180 °C±5 °C)

Physico-chemical properties	Frying period (h)						LSD at 0.05=
	0	4	8	12	16	20	
Refractive index	1.4724 ^c ±0.00035	1.4734 ^d ±0.0004	1.4748 ^c ±0.0005	1.4770 ^b ±0.0003	1.4820 ^a ±0.0007	1.4825 ^a ±0.0005	0.0008
Viscosity (centipoises)	38.1 ^e ±0.69	43.4 ^d ±1.09	47.6 ^c ±1.31	48.7 ^c ±1.21	52.33 ^b ±0.98	63.52 ^a ±1.45	2.042
Color (Red slide)	2.4 ^e ±0.64	3.5 ^d ±0.50	6.4 ^c ±0.25	8.7 ^b ±0.34	10.6 ^a ±0.30	11.4 ^a ±0.35	0.749
Acid value (mg KOH g ⁻¹ oil)	0.10 ^f ±0.03	0.26 ^e ±0.05	0.42 ^d ±0.07	0.54 ^c ±0.07	0.71 ^b ±0.04	0.88 ^a ±0.04	0.10
Peroxide value (meq.Peroxides/Kg oil)	0.40 ^f ±0.09	2.0 ^e ±0.30	4.9 ^d ±0.06	7.4 ^c ±0.36	10.5 ^b ±0.44	8.4 ^a ±0.42	0.56
TBA value(absorbance at 530 nm)	0 —	0.11 ^d ±0.02	0.25 ^c ±0.05	0.28 ^c ±0.03	0.43 ^b ±0.02	0.49 ^a ±0.05	0.06
Polymer content (%)	0 —	0.29 ^c ±0.04	0.48 ^c ±0.08	0.84 ^b ±0.09	0.93 ^b ±0.14	1.28 ^a ±0.25	0.228
Conjugated dienes (absorbance at 234 nm)	0.09 ^f ±0.005	0.12 ^e ±0.02	0.17 ^d ±0.01	0.22 ^c ±0.01	0.27 ^b ±0.01	0.34 ^a ±0.02	0.026
Conjugated trienes (absorbance at 268 nm)	0.03 ^e ±0.01	0.07 ^d ±0.01	0.09 ^{cd} ±0.01	0.12 ^{bc} ±0.02	0.14 ^b ±0.02	0.17 ^a ±0.01	0.029
Total polar compounds (%)	2.6 ^e ±0.03	2.8 ^e ±0.22	4.6 ^d ±0.41	6.5 ^c ±0.42	9.9 ^b ±0.56	12.5 ^a ±0.80	0.84
Soap (ppm)	0 —	0.11 ^e ±0.02	0.42 ^d ±0.07	0.65 ^c ±0.15	1.4 ^b ±0.2	4.10 ^a ±0.18	0.23

Data are expressed as mean ± SD. Values given represent means of three determinations. Values followed by the same letter are not significantly different ($p<0.05$).

sunflower oil used in frying changed from clear yellow to dark brown especially during the last days of the frying process. Colour darkening is a complicated process involving interaction with fatty acids, dimers, polymers and other minor compounds present in the oil (Xu et al. 1999). The dark colour patterns were significantly different from each heating period and correlated with hours of deep fat frying process (Farag et al. 2009; El-Anany and Ali Rehab 2008). Viscosity is one of the most important parameters required to determine the quality and stability of food system. Table 2 shows the changes in viscosity of unused and used frying sunflower oil. Viscosity of unused sunflower oil at the beginning of the frying experiment was 38.12 centipoises; however the viscosity value of sunflower oil used in frying at the end of frying period was about 1.66 times as high as that in fresh sunflower oil. The increase of viscosity during frying process may due to the formation of high molecular weight polymer compounds leading to the higher the degree of deterioration (Gray 1978; Abdulkarim et al. 2007). These polymer compounds led to increase the viscosity values in frying oil.

Determination of Free fatty acids appears to be the method favored by many operations for quality control evaluation of used frying oil (Stevenson et al. 1984). Free fatty acids in the fryer oil could be considered as a valid oil quality indicator, where the free fatty acids are allowed to go up to >0.5 % in the fryer (Gupta 2005; Abdulkarim et al. 2007). Data presented in Table 2 show that the acid values of sunflower oil used in frying were gradually and significantly ($P \leq 0.05$) increased with increasing the period of frying. The acid value of used frying oil at the end of frying period (20 h) was 0.88, and this value was about 8.8 times as high as that in unused sunflower oil. Free fatty acids (FFA) are formed during oil hydrolysis and during deep-fat frying process (Wu and Nawar 1986). As a result of this, the amount of FFA was increased by almost 9 times (Orthoefer et al. 1996). Hydroperoxides are the primary products of lipid oxidation. Therefore, the determination of peroxides can be used as an oxidation index for the primary stages of oil oxidation. Data presented in Table 2 show that peroxide values of sunflower oil used in frying were gradually and significantly ($P \leq 0.05$) increased during the first hours of frying time and then decreased after 20 h of frying period. The increase in the PV during frying process indicates a decrease of unsaturated fatty acid due to oxidation. However, peroxides are unstable compounds particularly under high temperature conditions; therefore the peroxides decompose to form carbonyl and aldehydic compounds causing the decrease in peroxide value (Perkins 1967; Shahidi and Wanasundara 2002). The amount of hydroperoxides that were formed at the end of the frying process was more than 27.1 times as high as that at the beginning of the frying process. The changes in changes in the TBA values of

fresh and used in frying sunflower oil are shown in Table 2. Frying process led to a gradual and significant ($P \leq 0.05$) increases in the TBA values (absorbance at 535 nm). TBA value of sunflower oil used in frying at the end of frying period was more than 49 times as high as that in unused sunflower oil. This finding clearly indicates that the strong effect of the frying period on the formation of secondary oxidation products that were formed during frying process and could be explained by the fact that the primary oxidation compounds (i.e. hydroperoxides) are very unstable and decompose into a series of aldehydes, ketones, hydrocarbons, alcohols, and many more reaction products as the oil oxidation process continues. These carbonyl compounds react with TBA reagent to produce TBA derivatives that have a high absorbance at 535 nm. (Przybylski and Eskin 1995). Data presented in Table 2 show the changes in the content of each conjugated dienes and trienes in unused and used in frying sunflower oil. These data show that unused sunflower oil had low levels of conjugated diene and trienes 0.09 and 0.03, respectively. Frying process led to increase the values of conjugated diene and trienes, this increase was gradually and significantly with increasing frying period. Used sunflower oil had significantly ($P \leq 0.05$) the highest levels of conjugated diene and trienes at the end of frying period 0.34 and 0.17, respectively. Frying process caused positional rearrangement in sunflower oil double bonds and part of the non-conjugated system was converted to conjugated diene and triene double bonds (Farag et al. 2009). Polymer content is one of quality indicator of the validation of fried oil. The oil quality control regulations (IUPAC 1987) indicate that the polymer levels must not exceed 1.5 %. Data presented in Table 2 show that unused sunflower oil did not contain polymers. Frying process caused significant ($P \leq 0.05$) increase in the level of polymer materials, this increase was gradually and significantly with increasing frying period. Sunflower oil used in frying had significantly ($P \leq 0.05$) the highest levels of polymer materials 1.28 % at the end of frying process (20 h). It is well known that the polymer content increases as the oxidative rancidity of oil exposed to frying temperatures proceeds (Yoon et al. 1987). The increase in polymer content is due to the formation of higher molecular weight substances by polymerization occurring at elevated frying temperature (Peled et al. 1975). The concentration level of total polar compounds is a good indicator of the overall quality of frying oils. Many European countries have established regulatory limits for TPC in frying oils (Blumenthal 1996). Most of these countries have considered a limit of 25 % TPC. The changes in the TPC content of fresh and used in frying sunflower oil are shown in Table 2. Unused sunflower oil had low level of total polar compounds 2.60 %, this low level indicate the high quality of fresh sunflower oil used in this investigation. In general, most of the unused oils

have polar compound levels lower than 5 % while levels can be highly variable in used frying oils (Ruiz Méndez et al. 2008). Frying process caused significant ($P \leq 0.05$) increase in the level of total polar compounds. Total polar compounds of sunflower oil used in frying was gradually increased during intermittent frying, there were significant differences in the contents of the total polar compounds at any of the measured frying times (Table 2). The total polar compounds (TPC) content of sunflower oil used in frying at the end of frying period was about 4.83 times as high as that of unused sunflower oil. Polar compounds are sum of non triglycerides of oil including fatty acids, alkaline pollutions, sterols, tocoferols, mon and di triglycerides, alcohols, aldehydes, ketons and other soluble compounds in oil that are more polar than triglycerides (Melten et al. 1994). Table 2 shows the soap content of unused and used in frying sunflower oil. Fresh sunflower oil did not contain soap materials. Frying process caused gradual and significant ($P \leq 0.05$) increases in the formation of soap content. Sunflower oil used in frying had 4.10 ppm of soap materials at the end of frying period. The formation of these soap materials during frying process was probably due to the interaction of some fried food components with oil during frying process, the metals, naturally present in the food, can form soap by reacting with the free fatty acids in the oil. Soap, thus formed in the oil, can produce an oil/water solution in the fryer and promote hydrolysis in the oil. (Blumenthal and Stockler 1986; Gupta 2005).

Effect of sugar cane bagasse ash (SCBA) and Magnesol XL filter aids on the quality of used frying sunflower oil

Although some adsorbent suppliers and researches recommended between 0.1 to 2.0 % for single adsorbent (Brooks 1991). Therefore, we decided to evaluate the efficiency 1–3 % concentration of each adsorbent.

Applying different filtration treatments resulted in weight losing of treated oil samples. Filtration with sugar cane bagasse ash (SCBA) and Magnesol XL caused significant ($P \leq 0.05$) weight losing ranged from 12.2 to 14.6 %. Non-significant differences in weight losing were observed between sugar cane bagasse ash (SCBA) and Magnesol XL filter aids at various levels (Table 3). The changes in some physico-chemical properties of treated sunflower oil are shown in Table 3. Used frying sunflower oil had significantly ($P \leq 0.05$) the highest value of refractive index 1.4825 at 25 °C. The treatment with different levels of Magnesol XL and sugar cane bagasse ash (SCBA) caused a significant ($P \leq 0.05$) decreases in the values of the refractive index. This decrease was gradually and significantly increased with increasing the adsorbent concentration. The reduction in refractive index due to treatment with various filter aids indicate the removal of some unsaturated compounds

resulted from frying at 180 ± 5 °C for 20 hr. The treatment with 2 and 3 % of SCBA had significantly ($P \leq 0.05$) the highest efficiency in removing some of some unsaturated compounds resulted from frying process. No significant differences were observed between the efficiency of 1 % SCBA as well as 2 and 3 % of Magnesol XL on lowering the refractive index of treated sunflower oil. The viscosity of sunflower oil used in frying at 180 °C for 20 h was 63.52 centipoises. The treatment of used sunflower oil with filter aids at different levels induced a significant lowering effect on the viscosity. It is worth noting that the value of viscosity was dependent upon the level of adsorbent, the higher level caused the higher lowering effect on the value of viscosity. The efficiency of SCBA at level 3 % was significantly ($P \leq 0.05$) the highest in reduction the viscosity of used sunflower oil. The efficiency of SCBA at level 2 % in lowering the viscosity of used sunflower oil was significantly ($P \leq 0.05$) the same as that of Magnesol XL at levels 2 and 3 %. The reduction in viscosity of treated oil indicates that polymers, which are the high molecular weight fraction of the degradation products, are reduced in the oil (Subramanian et al. 2000).

The value of intensity of red glasses of used sunflower oil was 11.40. Treatment with various levels of adsorbents caused significant ($P \leq 0.05$) decreases of the red-coloured glasses. The values of red-coloured glasses were gradually and significantly ($P \leq 0.05$) decreased with the increasing the level of the adsorbent. No significant differences ($P \geq 0.05$) in the efficiencies of 2 and 3 % SCBA as well as 2 and 3 % of Magnesol XL in reducing the values of red-coloured glasses were observed. The effectiveness of magnesium silicate in colour improvement and removing the colour producing materials was reported by (Lin et al. 2001; Farag and El-Anany 2006; Farag et al. 2009; Suseno et al. 2011). Table 3 shows that used oil had significantly ($P \leq 0.05$) the highest value of acid 0.88 mg KOH g⁻¹ oil. The treatment with various levels of adsorbents caused significant ($P \leq 0.05$) decreases of the values of free fatty acids. The values of acidity were gradually and significantly ($P \leq 0.05$) decreased with the increasing the level of the adsorbent. The treatment with SCBA and Magnesol XL at levels 2 and 3 % as had significantly ($P \leq 0.05$) the highest efficiencies in reducing the levels of FFA content of used sunflower oil. The differences between the FFA content of oil treated with 1 % of SCBA and those treated with 1 % of Magnesol XL were insignificant ($P \geq 0.05$). The treatment of used frying sunflower oil with various levels of adsorbent caused gradual and significant ($P \leq 0.05$) decrease in peroxide values. Peroxide values were gradually and significantly decreased with the increasing of the adsorbent level. The highest reduction was observed in peroxide value in those samples treated with 2 and 3 % of Magnesol XL or SCBA. In the same time, no significant differences were observed

Table 3 Changes of some physico-chemical properties of used frying sunflower oil treated with different levels of sugar cane bagasse ash (SCBA) and Magnesol XL filter aids

Physico-chemical parameters	Used frying oil	Used oil—treated with						LSD at 0.05=
		Sugar cane bagasse ash (SCBA).			Magnesol XL			
		1 %	2 %	3 %	1 %	2 %	3 %	
Refractive index	1.4825 ^a ±0.0005	1.4740 ^c ±0.0003	1.4741 ^c ±0.0007	1.4770 ^b ±0.0003	1.4727 ^d ±0.0006	1.4729 ^d ±0.0006	1.4742 ^c ±0.0004	0.0009
Viscosity (centipoises)	63.5 ^a ±1.45	44.3 ^c ±0.60	44.4 ^c ±1.70	47.5 ^b ±0.99	40.0 ^d ±1.60	45.2 ^c ±0.87	48.6 ^b ±1.38	2.250
Color (Red slide)	11.4a ±0.35	3.2c ±0.24	3.2c ±0.52	5.6b ±0.85	2.7c ±0.61	3.2c ±0.42	5.5b ±0.65	1.27
Acid value (mg KOH g ⁻¹ oil)	0.88 ^a ±0.04	0.15 ^c ±0.07	0.20 ^c ±0.1	0.40 ^b ±0.06	0.16 ^c ±0.02	0.17 ^c ±0.01	0.42 ^b ±0.09	0.110
Peroxid value (meq.Peroxides/Kg oil)	8.4 ^a ±0.42	0.90 ^c ±0.01	0.94 ^c ±0.05	1.9 ^b ±0.26	0.91 ^c ±0.09	0.96 ^c ±0.10	1.9 ^b ±0.14	0.60
TBA value (absorbance at 530 nm)	0.49 ^a ±0.05	0.10 ^c ±0.00	0.16 ^{bc} ±0.02	0.23 ^b ±0.05	0.11 ^c ±0.01	0.15 ^{bc} ±0.04	0.21 ^b ±0.03	0.062
Polymer content (%)	1.28 ^a ±0.25	0.13 ^c ±0.01	0.33 ^{bc} ±0.08	0.48 ^b ±0.12	0.14 ^c ±0.02	0.34 ^{bc} ±0.10	0.45 ^{bc} ±0.12	0.21
Conjugated dienes (absorbance at 234 nm)	0.34 ^a ±0.02	0.10 ^c ±0.00	0.16 ^{bc} ±0.05	0.20 ^b ±0.06	0.10 ^c ±0.00	0.15 ^{bc} ±0.04	0.22 ^b ±0.02	0.061
Conjugated trienes (absorbance at 268 nm)	0.17 ^a ±0.01	0.07 ^d ±0.01	0.09 ^{cd} ±0.00	0.13 ^b ±0.02	0.07 ^d ±0.00	0.08 ^{cd} ±0.00	0.11 ^{bc} ±0.01	0.026
Polar content (%)	12.5 ^a ±0.80	4.2 ^d ±0.36	6.1 ^c ±0.46	7.5 ^b ±0.47	4.6 ^d ±0.32	5.1 ^d ±0.41	7.2 ^b ±0.48	0.864
Soap (ppm)	4.1 ^c ±0.18	15.2 ^a ±1.87	11.8 ^{ab} ±2.05	8.5 ^b ±1.44	14.5 ^a ±2.13	11.5 ^{ab} ±1.62	8.3 ^b ±1.03	2.80
Weight losing %	—	14.4 ^a ±1.01	13.5 ^a ±0.49	12.3 ^a ±0.65	14.6 ^a ±1.3	12.9 ^a ±0.75	12.2 ^a ±1.05	1.62

Data are expressed as mean ± SD. Values given represent means of three determinations. Values followed by the same letter are not significantly different ($p < 0.05$).

between the efficiency of synthetic adsorbent (Magnesol XL) and natural adsorbent in removing the hydroperoxides resulted from frying at $180 \pm 5^\circ\text{C}$ for 20 hr. Used frying oil had significantly the highest TBA value at the end of frying period 0.49 as determined by the absorption at 530 nm. Generally, the treatment of used sunflower oil with various levels of adsorbents caused significant ($P \leq 0.05$) and gradual decrease in TBA values of sunflower oil. The reduction in TBA values was increased with the increasing the adsorbent level. Used oil treated with SCBA and Magnesol XL at levels 3 % had significantly ($P \leq 0.05$) the lowest amount of secondary oxidation products 0.11 and 0.10 as determined by the absorption at 530 nm, respectively. Table 3 demonstrates the polymer content of used-treated sunflower oil with different concentrations of Magnesol XL and SCBA. Used sunflower oil at $180 \pm 5^\circ\text{C}$ for 20 hr had 1.28 % polymers. The treatment with different levels of Magnesol XL and SCBA induced significant ($P \leq 0.05$) decrease in the polymer content. In this respect the used oil had nearly 5.80 and 5.50 times of polymer content as great

as that in used sunflower oil treated with 3 % of sugar cane bagasse ash and Magnesol XL respectively. These findings indicate that these filter aids had significantly ($P \leq 0.05$) the same efficiency in removing the polymer compounds from sunflower oil used for frying. The levels of conjugated dienes and trienes of used sunflower at the end of frying period were 0.34 and 0.17 as determined by the absorption at 234 and 268 nm, respectively. The treatment of fused sunflower oil with various levels of Magnesol XL and SCBA caused significant ($P \leq 0.05$) decrease of the contents of conjugated dienes and trienes. The levels of conjugated dienes and trienes were gradually and significantly ($P \leq 0.05$) decreased with the increasing the level of the adsorbent. The values of conjugated dienes and trienes of used oil treated with 3 % of SCBA were about 3.4 and 2.4 times as low as in used oil without treatment, respectively. Used sunflower oil at the end of frying period (20 h) had significantly the highest level of total polar compounds 12.56 %. Deterioration of frying oils is generally followed by changes in free fatty acid (FFA) level, colour of the used oil, or an

increase in polarity of the oil (Paradis and Nawar 1981). Treatment with different levels of Magnesol XL and SCBA induced significant ($P \leq 0.05$) decrease in the total polar compounds content. The treatment with SCBA at levels 2 and 3 % as well as Magnesol XL at level 3 % had significantly ($P \leq 0.05$) the lowest values of the total polar compounds content 5.18, 4.62 and 4.24 %, respectively. These values agreed well with the oil quality regulations (IUPAC 1987). Used in frying sunflower oil at the end of frying period (20 h) had a soap content of 4.10 ppm. Treatment with different levels of Magnesol XL and SCBA induced significant ($P \leq 0.05$) increase in the soap content. This increase was gradually and significantly increased with increasing the adsorbent concentration. The soap content of sunflower oil treated with 1, 2 and 3 % of SCBA were about 2.03, 2.57 and 3.06 times as high as that in used oil without filtration. Soap formed during filtration process due to the reaction of FFA with sodium silicate, the majority of it was released into the oil. Further, stirring the oil also would have induced release of the soap into oil (Kalapathy and Proctor 2000). The results indicate also that the treatment with Magnesol XL had nearly the same effects in increasing the levels of soap content in treated sunflower oil.

These results are in good agreement with our previous study (Farak and El-Anany 2006; El-Anany & Ali Rehab 2008), in which we observed that the treatment of used frying oils with Magnesol XL at level 2 % resulted in a significant improvement of the quality of treated oils. In this respect, Lin et al. 1999 found that the acidity of used oils was remarkably reduced by treatment with adsorbents, the FFA values after treatment dropped by 25–50 %. The treatment of Lemuru (*Sardinella lemuru*) oil with Magnesol XL at 1, 3 and 5 % levels reduced the acid value, the treatment with Magnesol XL at level 1 % resulted in a reduction of the acid value by 46 %. The use of 3 % Magnesol XL resulted in no further reduction of the acid values (Suseno et al. 2011). The treatment of fish oil with 3 % Magnesol for 5 minutes reduced PV by 59.5 %, giving the treated fish oil much lower peroxide values (Suseno et al. 2011). The treatment with hull ashes and Magnesol XL at level 2 % resulted in a significant decrease of the peroxide value of used sunflower oil from 34.50 to 2.0 (Farak et al. 2009). The treatment of used sunflower oil with normal and modified silica gel adsorbents caused a significant decrease of secondary oxidation products by about 70–77 % (Farak and Basuny 2009).

Table 4 Effect of hot water (80–85 °C) washing process on physico-chemical properties of used frying (F1) and used-treated (F2 & F3) sunflower oil

Physico-chemical parameters	Used frying oil (F1)	Used- treated with 3 % (SCBA). (F2)	Used- treated with 2 % Magnesol XL (F3)	F1-treated with hot water	F2-treated with hot water	F3-treated with hot water	LSD at 0.05=
Refractive index	1.4825 ^a ±0.0005	1.4727 ^d ±0.0006	1.4741 ^c ±0.0007	1.4800 ^b ±0.0006	1.4720 ^d ±0.0008	1.4730 ^d ±0.0004	0.0010
Viscosity (centipoises)	63.5 ^a ±1.45	40.0 ^d ±1.60	44.4 ^c ±1.70	50.2 ^b ±1.83	39.3 ^d ±0.62	41.0 ^d ±1.00	2.54
Color (Red slide)	11.40 ^a ±0.35	2.70 ^c ±0.61	3.26 ^c ±0.52	5.00 ^b ±0.54	2.50 ^c ±0.30	3.00 ^c ±0.50	0.87
Acid value (mg KOH g ⁻¹ oil)	0.88 ^a ±0.04	0.16 ^{cd} ±0.02	0.20 ^c ±0.1	0.42 ^b ±0.07	0.10 ^d ±0.02	0.12 ^d ±0.01	0.06
Peroxide value (meq.Peroxides/Kg oil)	8.4 ^a ±0.42	0.91 ^c ±0.09	0.94 ^c ±0.05	4.2 ^b ±0.53	0.72 ^c ±0.12	0.68 ^c ±0.09	0.50
TBA value (absorbance at 530 nm)	0.49 ^a ±0.05	0.11 ^c ±0.01	0.16 ^c ±0.02	0.30 ^b ±0.08	0.10 ^c ±0.02	0.10 ^c ±0.00	0.074
Polymer content (%)	1.2 ^a ±0.25	0.14 ^c ±0.02	0.33 ^c ±0.08	0.75 ^b ±0.11	0.14 ^c ±0.05	0.16 ^c ±0.08	0.22
Conjugated dienes (absorbance at 234 nm)	0.34 ^a ±0.02	0.10 ^b ±0.00	0.16 ^b ±0.05	0.20 ^b ±0.09	0.10 ^b ±0.03	0.10 ^b ±0.04	0.076
Conjugated trienes (absorbance at 268 nm)	0.17 ^a ±0.01	0.07 ^b ±0.002	0.09 ^b ±0.00	0.15 ^a 0.03	0 0	0 0	0.022
Total polar compounds (%)	12.5 ^a ±0.80	4.6 ^c ±0.32	6.1 ^b ±0.46	3.5 ^d ±0.64	1.0 ^e ±0.17	1.50 ^e ±0.26	0.877
Soap (ppm)	4.1 ^c ±0.18	14.5 ^a ±2.13	11.8 ^b ±2.05	1.2 ^c ±0.13	2.0 ^c ±0.24	1.5 ^c ±0.20	2.16
Weight losing %	—	—	—	11.12 ^a ±1.44	4.22 ^b ±0.85	5.16 ^b ±0.91	

Data are expressed as mean ± SD. Values given represent means of three determinations. Values followed by the same letter are not significantly different ($p < 0.05$).

The data of the present study demonstrate that filtration treatment with different levels of sugar cane bagasse ash (SCBA) regenerated the quality of used frying sunflower oil and possess higher adsorbing effects than the synthetic filter aid (Magnesol XL) in removing oil oxidation products. In addition, sugar cane bagasse ash (SCBA) is cheap and useful for regenerating the quality of frying oils. Hence, one would recommend implementing SCBA as natural filter aid to improve and regenerate the quality of used frying oils.

Effect of water washing process on the quality of used frying and used-treated sunflower oil

The abovementioned results showed that treated sunflower oil with different levels of SCBA and Magnesol XL caused significant increase in the quality of oil, however soap content of treated oil was increased also, therefore, the current experiment was performed to evaluate the effect of water washing process on the quality of used (F1) and treated sunflower oil (Table 4). According to the abovementioned results the treatment of used in frying sunflower oil with 3 % of SCBA and 2 % of Magnesol XL (F2 and F3, respectively) performed significantly the highest values in removing the oxidation products. Therefore, these treatments were selected to evaluate the efficiency of hot water washing process in improving the quality of used (F1) and treated sunflower oil. The applying of hot water for curing the used frying sunflower oil mimics degumming process during refining of crude oils. Hot water treatment caused significant ($P \leq 0.05$) weight losing, in all treated oils. Used in frying sunflower oil had significantly ($P \leq 0.05$) the highest weight losing 11.12 %, which indicate the important role of hot water treatment in improving the quality of sunflower oil used for frying, no significant differences in weight losing were observed between F2 and F3 oil samples treated with hot water. Hot water treatment improved the viscosity, colour, acid, peroxide, TBA, total polar compounds and soap content values of used frying sunflower oil by 21.28, 56.14, 52.27, 50.00, 38.77, 28.50 and 70.70, respectively. Hot water treatment induced significant reduction in soap and total polar compounds in all treated samples. The significant ($P \leq 0.05$) decreases in these values after water treatments could be explained by the fact that *Polar molecules* dissolve best in Polar solvents. Polar molecules can hydrogen bond with polar solvents, such as water, hence increasing their solubility. The decrease in soap and total polar compounds of used frying sunflower oil which was recovered with 3 % sugar cane bagasse ash (F2) and treated with hot water were about 4.62 and 7.27 times as low as that for (F2) oil. These findings were recorded for the hot water treatment of sunflower oil which was recovered with 2 % Magnesol XL filter aids (F3). The soap content of oils treated with hot water ranged from 1.2 to 2.0 ppm. The level

of soap should be less than 30 ppm after neutralization and less than 1 ppm after the bleaching (Erickson 1995). Therefore, water washing process leads to remove most of soap and total polar compounds products due to its polar nature. This means that water washing process improved the quality of used frying sunflower oil.

Conclusion

The data of the present study demonstrate that filtration treatment with different levels of sugar cane bagasse ash (SCBA) regenerated the quality of used sunflower oil and possess higher adsorbing effects than the synthetic filter aid (Magnesol XL) in removing oil oxidation products. In addition, SCBA is cheap and useful for regenerating the quality of used oils. Hence, one would recommend implementing sugar cane bagasse ash (SCBA) as natural filter aid to improve and regenerate the quality of used frying oils. The results also indicate that hot water treatment improved the viscosity, colour, acid, peroxide, TBA, total polar compounds and soap content values of used frying sunflower oil by 21.28, 56.14, 52.27, 50.00, 38.77, 28.50 and 70.70, respectively. Therefore, the current study recommends using hot water for improving the quality of used frying oil.

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