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PRE-TREATMENT OF PALM OLEIN-DERIVED USED FRYING OIL AS A FEEDSTOCK FOR NON-FOOD APPLICATIONS

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ABSTRACT

The main focus of this study was to improve the quality of palm olein-derived used frying oil (UFO) as a feedstock for non-food applications using an adsorption process. In addition, the adsorption capability and efficiency of four different adsorbents (activated carbon, activated bleaching earth, silica gel and aluminium oxide) used in treating the oil were assessed. Silica gel, when employed at an optimum treatment level of 20%, was found to be highly effective in the overall improvement of the quality of UFO, leading to significant reductions in free fatty acids (FFA) (percentage improvement, PI = 68.5%), peroxide value (PV) (PI = 85%), anisidine value (An. V) (PI = 33.8%) and total oxidation value (TOTOX) (PI = 45.3%). In terms of adsorption capability in FFA reduction, the Freundlich adsorption isotherm of FFA indicates a similar finding, supporting the pre-treatment of UFO using silica gel with its highest adsorption capacity (K = 1.0017), followed by activated carbon and the other two adsorbents.

Keywords: used frying oil, pre-treatment, adsorbent, adsorption efficiency, free fatty acids.

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INTRODUCTION

In general, all industries generate waste, and waste disposal continues to be hassle-driven due to its associated greenhouse gas emissions. Such problems have become one of the main agenda with which many countries have to deal. Although used frying oil (UFO) has not been alleged as one of the major culprits in environmental degradation, its disposal has been problematic as the deteriorated oil causes significant ecological impact on the environment.

In Malaysia, an estimated 50 000 t UFO are generated as waste from frying activities annually (Loh *et al*., 2006a) compared to several million tonnes produced globally (Zhang *et al*., 2003; Kulkarni and Dalai, 2006; Wang *et al*., 2007). Recycling UFO seems to be an alternative for utilising waste streams in many other more profitable applications, *e.g.* as biodiesel feedstock.

Palm oil as a frying medium, like any other vegetable oils, simply cannot escape from undergoing a series of chemical reactions, *i.e.* thermolytic, oxidative and hydrolytic processes, resulting in a significant reduction in its oil quality (Mittelbach, 1996). The oil quality deteriorates due to the formation of degradation products such as free fatty acids (FFA), glycerol, monoglycerides, diglycerides and other oxygenated products (Kulkarni and Dalai, 2006). Among these, FFA is the most undesirable (Zhang *et al*., 2003; Meher *et al*., 2006; Canakci and Sanli, 2008; Yuan *et al*., 2008) as it affects tremendously the conversion of UFO into biodiesel. This has triggered many attempts to find ways of getting rid of the impurities from UFO so that a rather good quality UFO can be used without much trouble for many applications. Among the many purification methods investigated, such as the membrane processing technique (Subramanian *et al*., 2000), supercritical carbon dioxide extraction (Yoon *et al*., 2000) and the use of filter aid materials (Lin *et al*., 2001; Maskan and Bag˘cı, 2003; Miyagi

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and Nakajima, 2003; Bhattacharya *et al*., 2008), the latter was more reliable, practical and more advantageous as it was simple, relatively less expensive and easy to conduct.

Our earlier studies involved a partial assessment on the effectiveness of activated carbon, silica gel, aluminium oxide and acid-activated spent bleaching earth in removing FFA and reducing the peroxide value (PV) of UFO (Loh *et al*., 2006a). Although results obtained were unable to quantify overall quality improvement of UFO, there was an indication that silica gel was a better adsorbent, similar to the finding by Miyagi and Nakajima (2003). The present study covered a thorough monitoring of the performance of the four adsorbents studied previously in improving the overall quality of UFO by evaluating: (1) a wider spectrum of the oil quality, and (2) the adsorption capacity of these adsorbents in reducing FFA by using the Freundlich adsorption isotherm model (Proctor and Toro-Vazquez, 1996).

MATERIALS AND METHODS

Materials

Palm olein-derived UFO was obtained from a student hostel at Universiti Teknologi MARA (UiTM). The chemicals used for analyses: isopropanol, phenolphthalein indicator, acetic acid, potassium iodide, sodium hydroxide, sodium methoxide, sodium thiosulphate, starch, 2,2,4-trimethylpentane and bis(trimethylsilyl) trifluoroacetamide (98% GC) were purchased from Merck. Hexane, sodium sulphate (Na_2SO_4) and sulphuric acid (H_2SO_4) were purchased from Fisher Scientific. The adsorbents used, silica gel (silica gel 60; particle size: 0.2-1.0 mm) and activated carbon were purchased from Merck. Activated bleaching earth was purchased from a local manufacturer, Taiko Bleaching Earth Sdn Bhd, while aluminium oxide was supplied by Fisher Scientific.

Methods

Pre-treatment of UFO. UFO was subjected to pre-treatment by mixing it separately with 10, 15 and 20 wt% (based on the oil used) of adsorbent (activated carbon, activated bleaching earth, silica gel and aluminium oxide). The mixture was stirred in a beaker at room temperature for 30 min, and then allowed to settle. The oil was vacuum-filtered and the filtrate was collected for analyses.

Adsorption isotherm study. All adsorbents used were sieved to obtain a particle size in the range of 3.1-7.5 nm. UFO was treated with each of the sieved adsorbent (10, 15 and 20 wt%, based on the oil used) in a beaker. The mixture was stirred at room temperature for 30 min. The adsorbent was allowed to settle and the oil was vacuum-filtered. Five grams of the filtrate was collected for FFA determination which was performed in triplicate, and all the FFA values obtained were incorporated in a graph for the adsorption isotherm study.

Analyses

The surface areas of the adsorbents were analysed using Quantachrome Autosorb Automated Gas Sorption based on the Brunauer-Emmett-Teller (BET) gas adsorption method. The porous structure and surface nature of the adsorbents were analysed on the basis of low-temperature (77°K) nitrogen adsorption-desorption and water vapour adsorption (298°K). An empty tube bath was weighed and reweighed with its cap on. Samples were placed in the weighed tube bath, which was plugged on the degas port of the analyser for degassing.

The physicochemical characteristics of adsorbent-treated UFO (UFO-T) were determined as follows. FFA, PV and anisidine value (An. V) were determined via MPOB Test Methods p2.5: 2004, p2.3: 2004 and p.2.4: 2004 (MPOB, 2005). PV measures the primary oxidation products (peroxide and hydroperoxides) of the unsaturated fatty acids in oil whereas An. V determines the secondary oxidation products (aldehydes, particularly α, β-unsaturated aldehydes). Total oxidation value (TOTOX) is an oxidation index used to determine the total oxidation products in UFO. It was calculated using Eq. (1) based on the values of PV and An. V obtained.

$$\text{TOTOX} = 2\text{PV} + \text{An. V} \quad (1)$$

A GC-MS equipped with a flame ionization detector was used to determine the fatty acid compositions (FAC) of all oil samples. A fused silica capillary column (30 m × 0.32 mm internal diameter) coated with BPX 70 was used with a programmed temperature profile as follows: oven temperature, 100°C (initial temperature) and 350°C (final temperature); injector temperature, 254°C; detector temperature, 360°C; and a carrier gas: helium at 2.0 ml min^{-1} (flow rate) and 10°C min^{-1} (ramping rate). An oil sample (1 ml) was injected into GC-MS as methyl ester derivatives after diluting them with sodium methoxide and hexane. The methyl ester derivatives were prepared via MPOB Test Method p3.4 – Part 1: 2004 (BF_3 method).

A Model 743 Rancimat was used for the measurement of oxidative stability. Oil samples of 3 g, held in the heating blocks at 110°C, were analysed under a constant air flow of 10 litres hr^{-1}. All determinations of induction period were performed in triplicate and the mean values reported.

An ASTM oil comparator was used to determine the colour of all oil samples according to ASTM 1500. The sample was initially placed in a test container, and then compared with a 0-8 colour-scaled glass disk in the presence of a specified luminous transmittance and chromaticity.

An automated multi-range viscometer HVM472 was used to measure the kinematic viscosity of the oil samples at 40°C according to ASTM D445.

All determinations were performed in triplicate and the mean values reported.

RESULTS AND DISCUSSION

Characterisation of Adsorbents

The Brunauer-Emmett-Teller (BET) gas adsorption method (Sing *et al*., 1985) was used to determine the physical properties, *i.e.* surface area and pore size, of the adsorbents used. Surface area is one of the important characteristics affecting the adsorptive capacity of an adsorbent (Hung *et al*., 2005). The adsorptive capacity of solid adsorbents is directly proportional to their surface area, *i.e.* a larger surface area contributes to a higher adsorptive capacity of an adsorbent. According to International Union of Pure and Applied Chemistry (IUPAC), adsorbents that have pore diameters <2 nm are classified as microporous solids while those with pore diameters >50 nm are macroporous solids. Adsorbents with pore diameters from 2 to 50 nm are mesoporous solids (Papirer, 2000). As activated carbon and silica gel have considerably larger surface areas compared to activated bleaching earth and aluminium oxide (*Table 1*), they are expected to have a higher capacity in the adsorption process too. As the surface area for activated carbon increases, its pore diameter becomes smaller (mesopores) and *vice versa* for activated bleaching earth and aluminium oxide (macropores).

Physicochemical Characteristics of UFO-T

UFO-T was characterised for its physicochemical properties to evaluate the adsorbents' adsorption capability at different treatment levels in improving the quality of the treated oil. The FAC of UFO and UFO-T (*Table 2*) show the retention of all the major fatty acids present in palm oil: palmitic acid (C16:0), oleic acid (C18:1) and linoleic acid (C18:2). The slight differences in their compositions particularly C18:2 were probably due to the removal of a certain amount of the oxidised intermediates during the adsorption process.

FFA is normally quantified separately from other decomposition products to estimate the degree of oil hydrolysis. It is a useful parameter which predicts the rancidity and the shelf-life of oil. The batch of UFO used in this study was not significantly degraded as its FFA was quite low (1.3%). Nevertheless, FFA in all UFO-T was further reduced at the different adsorbent treatment levels ranging from 10 to 20 wt% based on the oil used (*Figure 1*). The higher dosages of adsorbents had higher capability (*Figure 1c*) in reducing FFA in the oil as compared to the lower dosages of adsorbents employed (*Figure 1a*). Silica gel at the 20% treatment level was able to reduce FFA by a percentage improvement, PI, of 67%

TABLE 1. PHYSICAL PROPERTIES OF THE ADSORBENTS USED IN THE PRE-TREATMENT OF USED FRYING OIL (UFO)

Adsorbent	Surface area (m^2 g^{-1})	Average pore diameter (Å)[a]	Type of porous material
Activated carbon	888.4	30.18	Mesopores
Silica gel	412.0	29.12	Mesopores
Activated bleaching earth	199.2	72.05	Macropores
Aluminium oxide	0.1	75.71	Macropores

Note: [a] 1Å = 0.1 nm.

TABLE 2. FATTY ACID COMPOSITIONS (FAC) OF USED FRYING OIL (UFO) AND UFO-T BEFORE AND AFTER THE ADSORPTION PROCESS

FAC (wt% as methyl esters)	Before treatment	After treatment (UFO-T)			
	UFO	Silica gel	Activated bleaching earth	Activated carbon	Aluminium oxide
C14:0	1.3	0.81	0.94	1.07	0.84
C16:0	38.5	38.43	38.18	36.71	36.83
C18:0	5.6	4.19	3.77	3.96	4.29
C18:1	45.7	43.92	44.73	44.01	44.82
C18:2	8.8	11.13	11.02	11.81	11.21

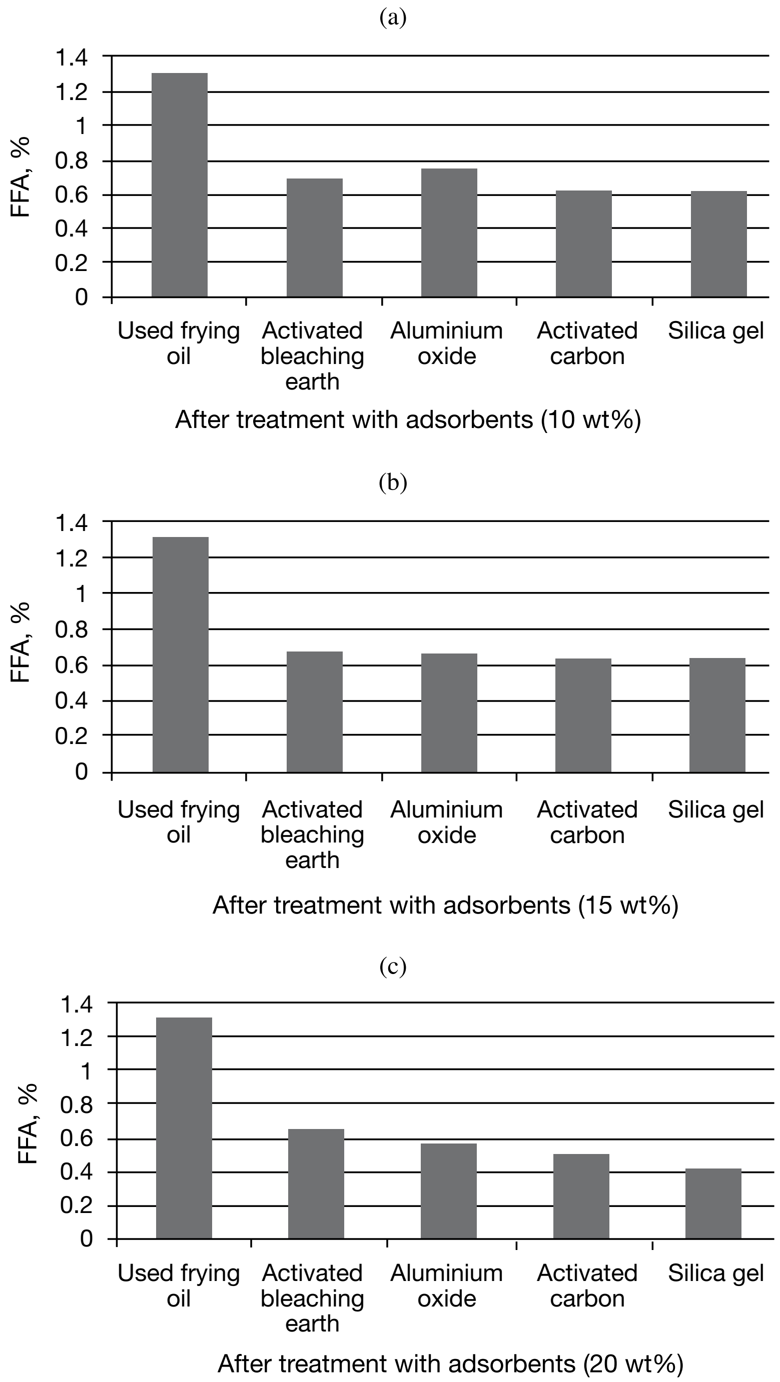

Figure 1. Reduction of free fatty acids (FFA) in UFO-T at adsorbent treatment levels of (a) 10 wt%, (b) 15 wt% and (c) 20 wt%.

(*Table 3*). Similar results have been found previously (Miyagi and Nakajima, 2003; Loh *et al*., 2006a) in FFA reduction by adsorption with silica gel, thus indicating that silica gel at a 20 wt% treatment level was the most effective adsorbent to effectively reduce FFA in UFO. For commercial exploitation, a treatment level beyond 20 wt% is not economical anymore, and also poses difficulty in handling due to the formation of a thick slurry in the oil.

PV and An. V of the oil after frying for a week increased from 2.0 meq kg^{-1} and 4.0 meq kg^{-1} to 40.04 meq kg^{-1} and 274.80 meq kg^{-1}, respectively. The tendency of the four adsorbents in removing the primary and secondary oxidation products in UFO was realised using the optimised adsorbent treatment level*s* of 20 wt%. *Table 3* shows PI of the four adsorbents in reducing PV and An. V of UFO. Aluminium oxide, silica gel and activated bleaching earth successfully reduced PV by >80% while activated carbon caused PV reduction by only 25%. On the other hand, silica gel adequately removed 34% of the secondary oxidation products

TABLE 3. PERCENTAGE IMPROVEMENTS (PI) OF FREE FATTY ACID (FFA), PEROXIDE VALUE (PV), ANISIDINE VALUE (AN.V), TOTAL OXIDATION VALUE (TOTOX) AND OXIDATIVE STABILITY OF UFO-T AT 20 WT% ADSORBENT TREATMENT LEVELS

Characteristic	Before treatment UFO	After treatment (UFO-T)							
		Aluminium oxide		Silica gel		Activated bleaching earth		Activated carbon	
			(%)[a]		(%)[a]		(%)[a]		(%)[a]
FFA[b] (%) ± 0.51	1.30	0.58	55.38	0.41	68.46	0.62	52.31	0.5	61.54
PVb (meq kg^{-1}) ± 0.21	40.04	5.80	85.50	6.01	84.99	7.51	81.25	30.03	25.00
An.Vb (meq kg^{-1}) ± 9.85	274.80	190.00	30.86	182.00	33.77	203.00	26.13	196.50	28.49
TOTOX (meq kg^{-1})	354.88	201.61	43.19	194.02	45.33	218.02	38.57	256.56	27.71
Oxidative stability[b] (hr) ±1.24	20.79	20.18	-	14.08	-	10.61	-	3.99	-

Note: [a] PI.
[b] Values are means ± standard deviation of triplicate determinations.
UFO – used frying oil.

TABLE 4. FLUIDITY AND OIL LIGHTNESS OF UFO-T AT 20 WT% ADSORBENT TREATMENT LEVELS

Physical property	Before treatment UFO	After treatment (UFO-T)			
		Aluminium oxide	Silica gel	Activated bleaching earth	Activated carbon
Kinematic viscosity @ 40°C ($mm^2 s^{-1}$)	45.658	45.327	45.402	45.625	45.637
Oil lightness scale	5 (reddish)	2 (yellowish)	3 (Lemon Yellow)	4.5 (reddish)	4.5 (reddish)

Note: UFO – used frying oil.

while activated bleaching earth showed the least reduction in An. V.

The values for PV and An. V were computed into Eq. 1 to produce the corresponding TOTOX values which determine oxygen-directed oil degradation in UFO (Miyagi and Nakajima, 2003). The TOTOX values (*Table 3*) of UFO-T after treatment with silica gel, aluminium oxide, activated bleaching earth and activated carbon were 194.02, 201.61, 218.02 and 256.56 meq kg^{-1}, respectively. Silica gel showed the highest PI for the TOTOX value whereas the other three adsorbents had lower capability. This overall quality improvement profile agreed well with the findings of Miyagi and Nakajima (2003).

While UFO had poor characteristics for PV, An. V and TOTOX, its oxidative stability associated with the degree of unsaturation in the fatty acids present was considerably better (20.79 hr). With the adsorbent treatment, UFO-T exhibited poorer oxidative stability (14.08, 10.61 and 3.99 hr, respectively) with silica gel, activated bleaching earth and activated carbon than UFO-T with aluminium oxide (20.18 hr). The decrease in oxidative stability in the three former treatments was associated with the adsorbents' selective removal of antioxidants, particularly carotenes (Loh *et al.*, 2006b) previously present in UFO.

In assessing the fluidity of oil after frying, it was found that the viscosity of UFO changed only slightly after frying for a week (45.66 $mm^2 s^{-1}$ *vs.* 43.08 $mm^2 s^{-1}$ for fresh oil), and no differences in viscosity was observed after the adsorption process (*Table 4*). This finding together with the low FFA value in the oil could support the fact that palm oil as a frying medium is able to highly withstand oil deterioration as it has equal compositions of saturated and unsaturated fatty acids compared to most of the other oils which have compositions of more than half of which are susceptible to oxidation. However, this cannot be strongly concluded as palm oil tends to exhibit high PV and An. V after a period of frying. Further research should concentrate on evaluating other rancid-affecting quality parameters such as total polar component (TPC) and other products, *e.g.* polymeric triglycerides.

The colour of frying oil provides an indication as to whether the oil should be discarded. Two of the adsorbents used in this study, *i.e.* activated carbon and activated bleaching earth were able to discolor UFO from an oil lightness scale of 5 (reddish) to 2 (yellowish) and 3 (lemon yellow), respectively, but the other two adsorbents were ineffective in doing the job (*Table 4*). A similar finding showing activated bleaching clay to be the most effective adsorbent in discoloring UFO was also reported by Miyagi and Nakajima (2003).

Freundlich Adsorption Isotherm

The FFA adsorption capacity of the four adsorbents used in this study was investigated using the Freundlich adsorption isotherm empirical model (Proctor and Toro-Vazquez, 1996; Özgül-Yücel and Türkay, 2003). This model was chosen as it accounts for various adsorption factors such as surface roughness, inhomogeneity and adsorbate-adsorbate interactions without which the study on FFA adsorption by the four adsorbents will bring about significant deviations. FFA adsorption capacity is expressed as Q_e, the amount of FFA adsorbed per unit weight of adsorbent (adsorption efficiency of the adsorbent), as a function of C_e, the residual FFA concentration at equilibrium at constant temperature. The adsorption efficiency of a particular adsorbent is directly related to specific adsorption x/m, where x is the quantity of FFA adsorbed and m the quantity of the adsorbent used. The linear form (logarithmic form) by plotting Q_e as a function of C_e based on Eq. (2) is shown in *Figure 2*.

$$\log \frac{x}{m} = \log K + n \log C_e \qquad (2)$$

The Freundlich adsorption isotherm for FFA in log function shows a slope equals to n and an intercept K. The intercept K represents the adsorption capacity while slope n is the energy of adsorption (Proctor and Toro-Vazquez, 1996). The K and n values for silica gel, aluminium oxide, activated carbon and activated bleaching earth determined this way are shown in *Table 5*. The adsorption isotherm for FFA showed that the higher the K value, *i.e.* the adsorbent's ability to adsorb FFA, the lower the energy of adsorption (n) required. Silica gel had the highest ability to adsorb FFA (K = 1.0017) compared to the others. Its excellent adsorption capability relates to a proposed ionic adsorption mechanism (Özgül-Yücel and Türkay, 2003). The mechanism involves the ionic interaction between the polar carbonyl group and the hydrophilic silanol (≡Si-OH) and siloxane (≡Si-O-Si≡) groups (Siwinska-Stefanska *et al*., 2008). This causes decreased surface tension at the interface of FFA and silica, hence resulting in a higher chemical affinity and an increased adsorptive potential of silica to FFA. The isotherm provides some evidence to support the earlier finding that a higher adsorption capacity and efficiency of adsorbents in reducing FFA is related to having larger surface areas and are mesoporous such as silica and activated carbon (*Table 1*). On the other hand, macroporous materials such as aluminium oxide and activated bleaching earth having smaller surface areas in their electrostatic situations are found to be the least effective adsorbents. The order of decreasing adsorption capability in FFA reduction of the absorbents in this study is: silica

TABLE 5. *K* AND *n* VALUES FOR SILICA GEL, ALUMINIUM OXIDE, ACTIVATED CARBON AND ACTIVATED BLEACHING EARTH

	K	*n*
Silica gel	1.0017	0.5293
Activated carbon	0.8878	0.5292
Activated bleaching earth	0.8191	0.3220
Aluminium oxide	0.3058	0.8264

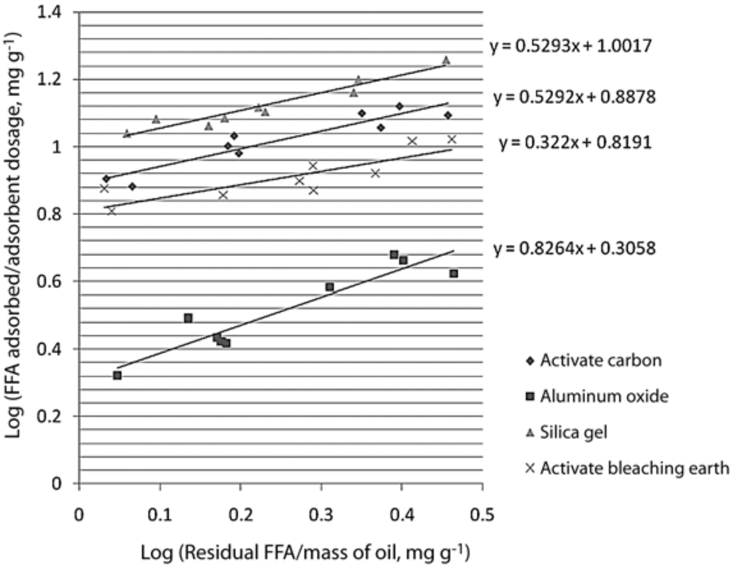

Figure 2. The adsorption isotherms for free fatty acids (FFA) of silica gel, aluminium oxide, activated carbon and activated bleaching clay in logarithmic form.

gel > activated carbon > activated bleaching earth > aluminium oxide.

The influence and the effectiveness of the four adsorbents used are clearly illustrated by the percentage improvement of each quality parameter assessed as given in *Table 3*. Based on these findings, the order of decreasing effectiveness in FFA reduction is: silica gel > activated carbon > aluminium oxide > activated bleaching earth; in PV reduction is: aluminium oxide > silica gel > activated bleaching earth > activated carbon; in An. V reduction is: silica gel > aluminium oxide > activated carbon > activated bleaching earth; and in TOTOX reduction is: silica gel > aluminium oxide > activated bleaching earth > activated carbon. The findings from the Freundlich adsorption isotherms indicate a similar order of decreasing adsorption capability in FFA reduction by the four adsorbents used.

CONCLUSION

The data from this study demonstrate that UFO can be essentially regenerated for various different non-food applications by adsorption processing. Among all the adsorbents used, removal of FFA in UFO using silica gel is highly recommended due to its highest adsorption capability shown in the Freundlich adsorption isotherm for FFA followed by activated carbon, activated bleaching earth and aluminium oxide. Silica gel, being mesoporous, is not merely used to reduce FFA in UFO significantly, but also effectively improves PV, An. V and TOTOX. Although these properties are somewhat improved, their levels in UFO-T are not acceptable for use as food. As a result, the oil cannot be conveniently recycled back as cooking oil or as an ingredient for making other food products unless strong evidence on its edible characteristics is found. Thus, UFO-T can only be considered for non-food applications, *e.g.* as biodiesel feedstock.

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