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Regeneration and Reutilization of Oil-Laden Spent Bleaching Clay via in Situ Transesterification and Calcination

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Abstract Landfill bound waste from the oil palm industry, spent bleaching clay (SBC) containing significant amounts of adsorbed crude palm oil (CPO) has the potential to be used for biodiesel production. In this study, SBC was subjected to ultrasound-aided in situ transesterification with a co-solvent to convert the oil into methyl esters (biodiesel). Optimized reaction conditions used were 5.4 wt% KOH, methanol to oil mass ratio of 5.9:1 and 1:1 mass ratio of co-solvent (petroleum ether or ethyl methyl ketone) to SBC. The remaining bleaching clay was calcined at 500 °C for 30 min and reutilized for bleaching. Absence of –CH absorption peaks in the FTIR and TGA-FTIR analysis of regenerated clays shows the regeneration efficiency of the method. In situ transesterification and heat regeneration helped to restore pores without adversely affecting the clay structure. The use of ethyl methyl ketone (EMK) as the co-solvent in the in situ transesterification process produced clay with better bleaching qualities.

Keywords In situ transesterification · Regeneration · Reutilization · Calcination · Spent bleaching clay · Biodiesel

Introduction

Spent bleaching clay (SBC) is one of the major wastes from the edible oil industry. During the refining process, 0.5–2% of bleaching clay is added to crude palm oil (CPO) and stirred. The clay is then separated from the oil and discarded. From Malaysia alone, with an annual production of 17.6 million tonnes of CPO [1], it is estimated that more than 100,000 tonnes of SBC is generated yearly. The most common way of disposing of the oil laden SBC is by dumping into a landfill [2]. This method of disposal has been adopted by oil producing companies worldwide as it is cost effective. However, safety is compromised.

SBC can contain up to about 40% of residual oil and impurities such as coloring pigments, mucilaginous matter, fibers, protein degradation products, ashes and soaps upon disposal [3]. Clearly, disposal of this oil laden waste needs to be treated with care. The current method of disposal raises many concerns regarding possible environmental, fire and chemical hazards. SBC is listed as having pyrophoric properties as the high iodine value oil contained in SBC may spontaneously combust and cause a fire hazard [4]. In order to suppress the possibility of spontaneous combustion, the area for the disposal may be cooled by spraying with water or the distribution of the oil laden SBC must be more widespread. Both the suggested methods result in more wastage of water and land area. Furthermore, oil contained in the disposed SBC may leach into the soil and contaminate the water source, thereby presenting an environmental hazard. As the oil turns rancid, unpleasant odors will be emitted. According to the Environmental, Health and Safety (EHS) Guidelines, Oleochemical Manufacturing [4], the inhalation of dust from bleaching clay may present a risk of exposure to chemical hazards. This could be due to the composition of bleaching clay which contains crystalline silica.

Environmental, Health and Safety (EHS) Guidelines, General EHS Guidelines: introduction [3] defines hazardous wastes as materials that share the same properties of hazardous materials physically, chemically or biologically that are potentially risky to human health or the

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environment if not managed properly. Considering the problems that SBC can pose, it is categorized as hazardous waste. Landfill disposal of SBC is also wasteful as the residual oil adsorbed in the clay is being discarded without any usage. This has prompted urgency in innovating practical and economical ways to utilize SBC. Loh et al. [5] performed solvent extraction and supercritical alcohol extraction of residual oils from SBC and subsequently transesterified the oil into methyl esters. They found that the methyl esters have similar characteristics to diesel fuel. A Soxhlet extractor was used to extract residual oils from the SBC with four types of solvent. Ethyl methyl ketone (EMK) was reported to give the highest extraction, however, the quality of the extracted oil was compromised. Extracted oils can be used in the soap industry or as animal feed if it is of good quality [6]. It has been reported that SBC can be treated with NaOH to convert the residual oil into soap to be discarded. Meanwhile, the remaining bleaching clay has been used as an adsorbent for lead [7]. However, on the downside, the residual oil was discarded without any use and wastewater was generated during soap removal from the treated bleaching clay. Solvent extraction of residual oil seems to be the method of choice for most researchers in this area of study [5]. Lim et al. [8] extracted the oil from the SBC using different types of solvent and subsequently transesterified the extracted oil using CaO as a heterogeneous catalyst.

Al-Zahrani and Daous [9], deoiled the SBC with EMK and activated the clay by calcination at 500 °C. Ng et al. [10] used a Soxhlet extractor and supercritical CO_2 at 60 °C and 300 bar to deoil the clay. The clay was then heat treated at 400 °C in a muffle furnace in the air and treated with 20% H_2SO_4. The acidified-heat treated clay was reported to have smaller pores and increased surface area compared to deoiled clay. Supercritical –CO_2 extraction of SBC was reported to be just as efficient as Soxhlet extraction of SBC to yield industrial grade soybean oil and oil-free clay [11].

This work aims reutilize the SBC by an in situ transesterification of the adsorbed oil into methyl esters and subsequent heat treatment to regenerate the SBC. By using the in situ method, the authors are looking into eliminating the much preferred oil extraction step, namely, the Soxhlet extraction step which was deemed to be costly and time consuming as proved by fellow researchers of the same laboratory [8].

Experimental

Materials

SBC and fresh bleaching clay (FBC) was provided by Carotino Sdn Bhd (Johor, Malaysia) and stored below

4 °C. Prior to analysis the clay was desiccated and dried in the oven at 103 ± 2 °C for 2½ h. All the chemicals and solvents used in this study were of analytical reagent grade. Methyl heptadecanoate (>99.5%) was supplied by Fluka (Switzerland).

Characterization of Clays

The oil content of SBC was determined using a simple solvent extraction method which proved to be just as effective as the time consuming and costly Soxhlet extraction method [8]. The acid value of the oil was determined using the standard EN 14104 method. To approximately 2 g of pre dried SBC, 1:1 mass ratio of petroleum ether (60-80 °C) to SBC was added, homogenized using a Vortex mixer (Cenco Instrumenten B. V. 220 V, 20 W) and immersed in a water bath at 55 ± 2 °C for 1–2 min. The homogenized mixture was then centrifuged at 50 rpm for 10 min and the yellowish brown supernatant was collected. The steps were repeated three times and the resulting liquid was dried under a nitrogen stream until a constant weight was achieved. The oil content was taken as the average of the three determinations and reported as weight percent. The process was repeated using EMK as a solvent.

The topography and elemental analysis was conducted using Scanning Electron Micrograph coupled with Energy Dispersive X-ray (SEM-EDX, Hitachi S-3200 N). FTIR analysis was carried out with a Perkin Elmer System 2000 FTIR using the KBr disk technique in the range 4,000–400 cm^{-1} to determine the functional groups of the clay. Thermal properties were analyzed using TGA/SDTA 851^{e} Mettler Toledo coupled with Nicolet 5700 FTIR via a glass cooled transfer line. Peak identification was achieved using Nicolet TGA Vapor Phase library in the OMNIC software. The sample was heated at a rate of 10 °C/min in a temperature range of 50–900 °C, under nitrogen flow (60 ml/min). During TGA analysis, the spectra of the gas released by the sample was acquired at a sampling frequency of one spectrum every 4 s, and with a spectral resolution of 4 cm^{-1}. Surface area and pore structure analysis was done using nitrogen adsorption–desorption isotherms using a NOVA 2000 instrument. The Brauner-Emmett-Teller (BET) surface area was calculated using the adsorption data for relative pressure ranges from 0.00 to 1.00. The pore size distribution curves were obtained based on the differential pore volumes of the Barrett-Joyner-Halenda (BJH) desorption branch.

In Situ Transesterification of Adsorbed Oil

The reaction conditions used in this study had been optimized and reported as such elsewhere [12]. To 2 g of SBC

(assumed to contain 27% of oil from the above determination, 6.689×10^{-4} mol), a 1:1 mass ratio of petroleum ether (PE) to SBC was added. Then, 0.108 g of KOH (5.4 wt% of SBC) was dissolved in 3.215 g methanol (methanol to oil mass ratio of 5.9:1). Methanolic KOH was added to the mixture and homogenized using a vortex mixer. A small portion of the aliquot was withdrawn for TLC analysis. The test tube containing the mixture was then immersed in a ultrasound water bath (Bransonic Ultrasound Cleaner, Model B-3200 E2, 47 kHz ± 6%, 340 W) at 55 ± 2 °C for 2 h and centrifuged for 10 min at 50 rpm. At the end of 2 h, three layers were formed, namely, PE, methanol and the SBC layer. The top two layers were transferred into two separate pre-weighed glass bottles. About 3 ml of PE was added to SBC, homogenized and centrifuged. Three layers were formed and separated as mentioned earlier into the respective bottles. The methanol layer was extracted with PE (1×3 ml, 2×2 ml) and added to the bottle containing the PE layer. It was then dried under nitrogen stream, washed with water (1×2 ml and 2×3 ml) and dried with anhydrous sodium sulphate. The resulting SBC was left to dry, calcined at 500 °C for 30 min and kept in desiccators for further analysis. The process was repeated using ethyl methyl ketone (EMK) as the co-solvent. Since EMK is a mutual solvent for methanol and methyl ester, after centrifugation only two layers were formed, the methanol-EMK and SBC layer. The methanol-EMK layer was dried with nitrogen and washed with water until the extract became colorless to phenolphthalein indicator. The SBC was treated the same way as earlier. The dried methyl esters were then subjected to gas chromatography analysis. Analysis was done using the polar capillary column (Supelco Wax, 30 m × 0.25 mm i.d. × 0.25 μm), with nitrogen as the carrier gas and hydrogen and air as the detector gasses. The oven temperature was set at 190 °C, injector temperature at 230 °C and detector temperature at 230 °C. A sample of 0.25 g of methyl esters was mixed with 2 ml of methyl heptadecanoate solution (0.6 mg/ml in heptane). The ester content was determined following the EN 14103 method. Methyl ester peak identification was through a comparison with methyl ester standards.

Regeneration and Reutilization of Clay

The clay remaining after the in situ transesterification was calcined in a preheated furnace at 500 °C for 30 min and desiccated. A 100 g sample of CPO was weighed into a three-necked flask in a paraffin bath and heated to 100 °C while stirring under a nitrogen stream. Then, 1 g of regenerated clay was added to the flask. Heating and stirring was continued for a further 30 min at 100 °C. Figure 1 shows the schematic diagram of the oil bleaching setup.
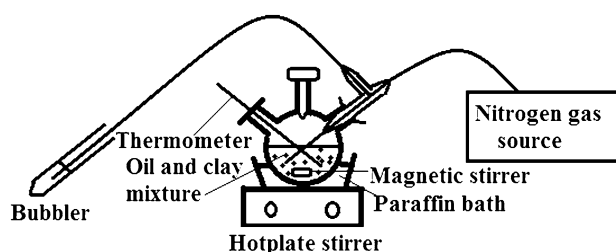

Fig. 1 Schematic diagram of the oil bleaching setup

The hot clay and oil mixture was centrifuged to separate the clay from the oil.

Regenerated Clay Efficiency Testing

A 0.1 g sample of bleached oil was weighed into a 25-ml volumetric flask and made up to the mark with *n*-hexane. Spectrophotometric measurements of the bleached oil were taken at 446 and 269 nm. The carotene content of the oil was calculated as *β*-carotene in ppm as follows: $\beta\text{-carotene (ppm)} = \frac{25 \times 383 \times (a_s - a_b)}{100\,W}$, where a_s and a_b is absorption of sample and blank at 446 nm, respectively, W is the weight of sample in grams [13]. The deterioration of bleachability index (DOBI) index was calculated as follows: $\text{DOBI} = \frac{\text{Absorption at 446 nm}}{\text{Absorption at 269 nm}}$ [14]. The carotene content and DOBI index was calculated for the oil bleached with regenerated clays that were prepared using petroleum ether and EMK as co-solvents.

Results and Discussion

Characterization of Clays

SBC was found to contain about 24 and 27% of CPO when extracted with petroleum ether and EMK, respectively. The solvent extraction method for SBC oil determination employed in this study gave comparable results to the Soxhlet extraction method reported by fellow colleagues in the same laboratory [8]. Acidity (expressed as percentage of palmitic acid) of the adsorbed palm oil was determined to be 7.2 wt%. Elemental analysis showed that the elements common to all three types of clay are carbon, calcium, oxygen, iron, magnesium, aluminium, silicon and potassium. However, an extra element, phosphorus, was found in the SBC. This could be due to the addition of phosphoric acid to the oil and clay mixture for degumming during oil refining in the industry. The absence of absorption bands at 2,922 and 2,852 cm^{-1} for $-\text{CH}_2$ deformations and 1,744 cm^{-1} (C = O), characteristic bands for edible oils [15], in FTIR analysis of regenerated clays of petroleum ether and EMK co-solvent, showed that

Table 1 Common signature peaks observed in FTIR analysis of the various types of clay

Signature peaks	Absorption (cm^{-1})
Interlayer water and stretching vibrations of structural hydroxyl	3,460, 3,560
Deformation vibration of adsorbed water	1,650, 1,450
Si–O stretching vibration	1,046, 798
Deformation vibration of Si–O–Al, Si–O–Si (octahedral layer)	800–400

no oil was present in the clay after regeneration. The absence of oil was confirmed when the FTIR spectra (spectra not shown) of the SBC showed large absorption of –CH$_2$ and C = O peaks. Table 1 shows the signature peaks and the corresponding absorption for montmorillonite clay that was present in all clay samples. This shows that the in situ transesterification and heat regeneration method has negligible effect on the structure of the clay.

Thermogravimetric analysis of the clays shows that there are two major mass losses for the fresh bleaching clay and regenerated clays of two different co-solvents, that is, around 30.0–258.5 °C and around 572.4–850.5 °C with a total mass loss of approximately 11%. The first mass loss can be attributed to the loss of adsorbed water molecules and the second due to the loss of hydroxyl groups of clay structure [16]. Only the SBC showed a prominent mass loss between 200 and 480 °C, corresponding to the evolution of volatile products or oil from the SBC. The TG and SDTA curves for regenerated and spent bleaching clay are shown in Fig. 2a–c. Evolved gaseous products were analyzed by coupling the thermobalance online with a FTIR spectrometer and OMNIC software. Analysis showed that only the SBC has an extra band at 2,990 cm^{-1} for C–H stretching in CH$_2$ or CH$_3$ [17] corresponding to the presence of residual oil. Calcination temperature for clay regeneration was fixed at 500 °C for 30 min as the thermogram of SBC (Fig. 2a) shows that oil was fully decomposed at 480 °C (22 min).

Surface area analysis showed that the regenerated clays using two different co-solvents have similar specific surface area and average pore diameter. However, the specific surface areas (Fig. 3) of the regenerated clays are much lower than the FBC. This could be due to the carbonized residual carbon that closed in the smaller pores, thereby, resulting in a loss of specific surface area and pore volume [15]. The BET specific surface area of the regenerated clays corresponds to that of unactivated montmorillonite clay, which is between 31 and 61 m^2/g [18]. The acid wash treatment after regeneration was proved to increase the surface area of the regenerated clays [10, 13, 15]. It has been claimed that the acid wash helps to remove the carbon
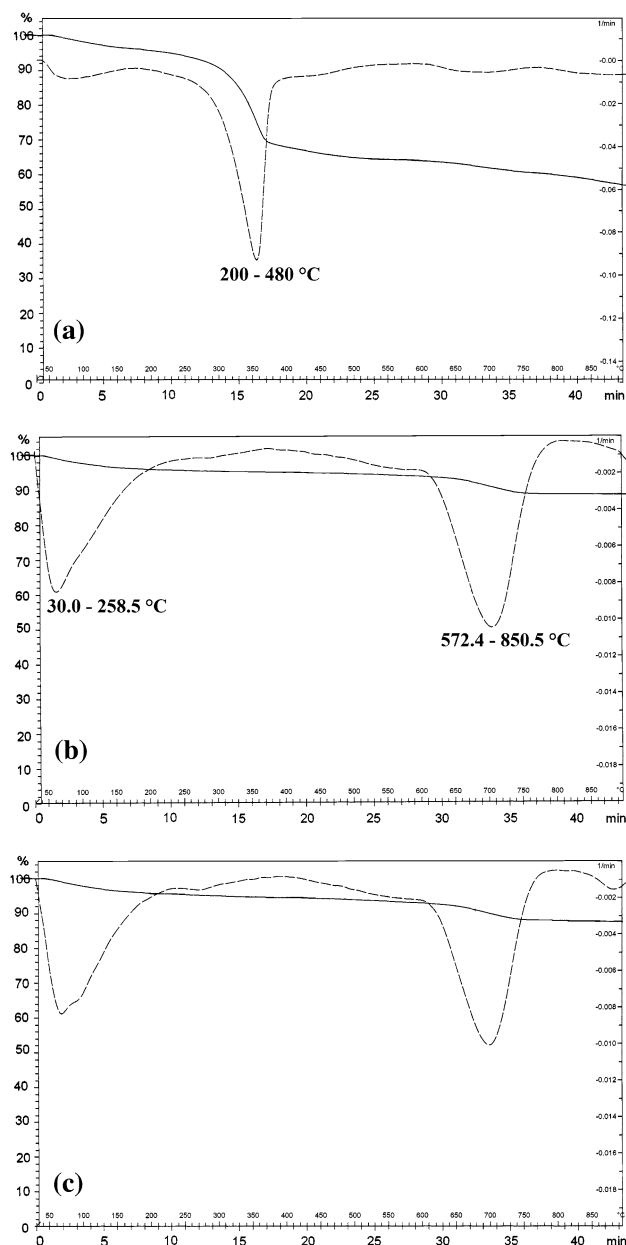
Fig. 2 Thermogravimetric analysis of (**a**) spent bleaching clay, (**b**) regenerated clay with EMK co-solvent and (**c**) regenerated clay with PE co-solvent

residue and consequently increases the surface area [15]. The isotherms in Fig. 3 shows that the adsorption capacity of regenerated clays decreases compared to the fresh bleaching clay. This is as such because regenerated clays in this study were not subjected to acid activation. According to the Brauner, Deming, Deming and Teller (BDDT) classification, all the nitrogen adsorption isotherms shown in Fig. 3 are of type IV, which are characteristic of a mesoporous solid. According to de Boer, specific pore structures can be deduced from certain shapes of the hysteresis loops. The type H3 hysteresis loops (Fig. 3) are
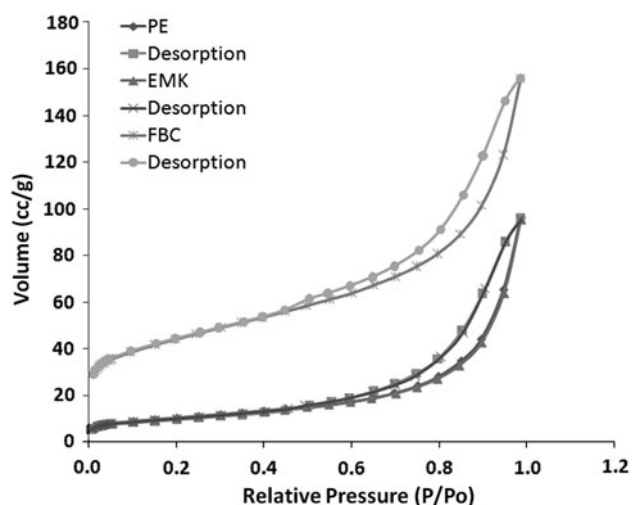
Fig. 3 Adsorption isotherm of nitrogen of FBC and regenerated clays with EMK and PE co-solvents
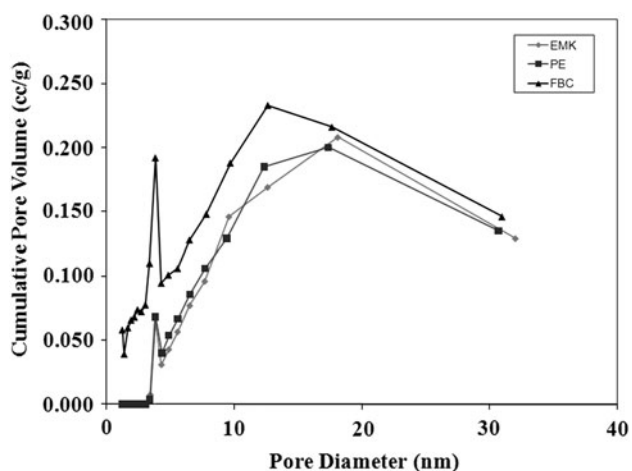

Fig. 4 BJH pore size distributions of FBC and regenerated clays with EMK and PE co-solvents

observed for non-rigid aggregates of plate like particles giving rise to slit-shaped pores. The plate like particles or layer structure is typical of clay structure. This shows that regeneration by calcination does not alter the layer structure of the clay. Pore size distribution curves based on the differential pore volumes of Barrett-Joyner-Halenda (BJH) desorption branch (Fig. 4) shows that the major peak is at 12 nm and the majority of pores of FBC and the regenerated clays are distributed between 3.0 and 30.0 nm. According to IUPAC classification of pore sizes, this range falls in the mesoporous pore structure which is between 2 and 50 nm. Calcination helps in the removal of substances that were adsorbed in the pores during bleaching and could not be removed by deoiling [10]. As conducted in this study, ultrasound-aided in situ transesterification helps to deoil the used clay to a certain extent before calcination to

remove the remaining stubborn substances. Therefore, it is evident that in situ transesterification and heat regeneration helps in the restoration of the pores without disrupting the clay structure.

In Situ Transesterification

Ultrasound aided in situ transesterification of the adsorbed oil gave approximately 60 wt% of methyl esters for the EMK co-solvent and 75 wt% of methyl esters for the petroleum ether co-solvent. Compared to the oil extraction efficiency, as reported in "Results and Discussion" of "Characterization of Clays", EMK gave higher oil extraction. This could be due to EMK being a more polar solvent (dielectric constant = 18.5) than petroleum ether (dielectric constant = 2.0). EMK, a polar aprotic solvent is prone to solvate both the polar molecules (FFA) and non polar components of CPO adsorbed on SBC, producing a homogeneous mixture with methanolic KOH (as stated in "Experimental" of "In Situ Transesterification of Adsorbed Oil"). The polar molecules consumes the KOH in methanol to form soap, thus lowering the methyl ester yield. However, petroleum ether, a non polar solvent, solvates the non polar components (oil) which is then transesterified to methyl esters. Therefore, this study shows that although EMK is a good solvent for overall removal of oil components from SBC, petroleum ether is a better co-solvent for biodiesel production. Co-solvent and ultrasound enhances the production of methyl esters and the subsequent regeneration of the clay. The co-solvent helps to overcome the mixing problem, promotes oil/methanol miscibility and enhances the contact of the reactant molecules [19]. The use of ultrasound for in situ transesterification of the adsorbed oil managed to eliminate the oil extraction step. Cavitational methanolic/co-solvent bubbles were produced near the methanolic KOH/co-solvent/SBC/oil boundaries; resultant ultrasonic jets of methanolic KOH/co-solvent impinge into the oil adsorbed on SBC [20]. The cavitational bubbles help to dislodge the oil from SBC for the transesterification process to take place. The high amount of KOH used in this study is to convert the free fatty acids into soap to be removed during water washing and to compensate for the loss of KOH that could be stuck to the walls of SBC rendering it useless.

Regeneration and Reutilization of Clay

The regenerated clays were used to bleach CPO and the oil was tested for carotene content and DOBI index. Spectrophotometric measurements were taken at 446 nm (β-carotene) and 269 nm (conjugated trienes) to determine the reusability of the clay. A reduced carotene content of bleached oil compared to CPO is a good indicator of the

Table 2 Deterioration of Bleachability Index (DOBI) of CPO and CPO bleached with various types of clay

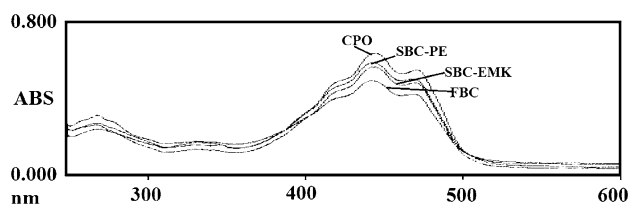| Oil sample	DOBI
SBC-EMK	2.90
SBC-PE	2.50
FBC	2.45
CPO	2.99

Fig. 5 Spectrophotometric spectrum of CPO, oil bleached with FBC and regenerated clays

efficiency of the regenerated clays. About 500–700 ppm of α and β-carotene are known to be present in good quality CPO. However, the color of palm oil becomes fixed as a result of the iron-catalyzed oxidation of carotene and becomes more difficult to bleach [21]. Figure 4 shows the spectrum of CPO and CPO bleached with various types of clay. The regenerated clay of EMK co-solvent showed the highest efficiency for carotene content reduction and had a higher multi point BET surface area as can be seen in Fig. 3. However, the reduction in carotene content is not the ultimate indicator of the bleaching capacity of the clays, as in the refining industry; most of the carotene content is removed during the deodorization step [22]. Therefore, it is more important to make sure that the clay is not contributing to the deterioration of the quality of the oil after bleaching. DOBI is widely used as an indication of the oxidative status of CPO [23]. DOBI indicates the ratio of the amount of carotene to the amount of secondary oxidation products. As a result, any increase in the secondary oxidation products in the oil after bleaching with the regenerated clay will cause an increase in absorption at 269 nm and subsequent decrease in DOBI. This indicates the deterioration of the oil quality. Table 2 shows that the DOBI of the oil bleached with regenerated clay using EMK co-solvent is similar to CPO. According to the DOBI values and respective oil quality set by Palm Oil Research Institute of Malaysia (2005), oils with a DOBI index between 2.4 and 2.9 are of fair quality. Thus, it is clear that none of the regenerated clays are detrimental to the quality of bleached oil (Fig. 5).

Conclusion

Ultrasound-aided in situ transesterification of adsorbed CPO using EMK as a co-solvent, followed by calcination at 500 °C for 30 min was found to be the best set of conditions to regenerate the hazardous oil laden SBC. The quality of oil bleached with regenerated clay using the EMK co-solvent showed more resemblance to the CPO. This study succeeded in providing an alternative way to produce a green energy source while simultaneously lessening the pollution from hazardous materials.

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