Optimization of bleaching power by sulfuric acid activation of bentonite

Hale Bayram1, Gokce Ustunisik2,3, Mişref Önal4* and Yüksel Sarkinay4

1Atatürk Faculty on Education, Marmara University Göztepe, Istanbul, Turkey; 2Department of Geology and Geological Engineering, South Dakota School of Mines and Technology, Rapid City, SD 57701-3995, USA; 3Department of Earth and Planetary Science, American Museum of Natural History, New York, NY 10024-5192, USA and 4Department of Chemistry, Faculty of Science, Ankara University, 06100 Tandoğan, Ankara, Turkey

Abstract

Acid activation was carried out by heating muds prepared from Ca-rich bentonite (CaB), sulfuric acid (H2SO4) and distilled water, yielding various bleaching earths (BEs). The roles of each of the process variables, namely mass fraction of H2SO4 (x) in the dried CaB and pure H2SO4 mixture, heating temperature (T) and contact time (t), in the activation were tested. The raw CaB and each BE were examined with X-ray diffraction, chemical analyses, N2 adsorption/desorption, pH-metry and tintometry. The BE contents in both the aqueous and oily suspensions were set to 2% by mass in order to measure the pH value and the bleaching power, respectively. The optimum conditions for the activation to obtain a BE having the same bleaching power (0.60) as Tonsil® Optimum Bleaching Earth for soybean oil were determined as x = 0.35, T = 100°C and t = 2 h. The bleaching power increased only marginally upon increasing the activation time from 2 h up to 10 h at a given x and T. After 10 h, significant changes did not take place in the bleaching power of the BE. The mass fraction of total structural metal oxide (y), pH value, specific pore volume (V), specific surface area (S) and mean internal diameter (D) of the mesopores for the optimized BE are y = 0.10, pH = 3.0, V = 0.31 cm3 g−1, S = 225 m2 g−1 and D = 7 nm. The pH and D values were more important for bleaching the alkaline refined soybean oil compared to the other parameters tested.

Keywords: acid activation, bentonite, bleaching, porosity, soybean oil

(Received 16 March 2021; revised 9 September 2021; Accepted Manuscript online: 22 September 2021; Associate Editor: Stephan Kaufhold)

Crude vegetable edible oils are produced by compression or solvent extraction of soybean, cottonseed, sesame, coconut, olive, safflower, sunflower, corn, rapeseed, mustard seed, peanut and palm oils. They are processed by both chemical and physical refining techniques (Mounts, 1981; Zschau, 1999). The conventional chemical techniques consist of acid degumming, alkali refining, bleaching, deodorization and winterization stages (Rich, 1964; Almeida et al., 2019; Hew et al., 2020).

In addition to organic pigments such as chlorophyll, carotenoids and related compounds, other impurities such as soaps, sulfur, phosphates, trace metal cations and their decomposition products are removed from the alkali-refined oils by adsorption and ion exchange through bleaching (Falaras et al., 1999; Taxiarcho & Douni, 2014; Saneei et al., 2015; Awad et al., 2019). Previous studies on the kinetics of the bleaching of vegetable oils (Brimberg, 1982) showed that the catalytic effect of the bleaching earth (BE) causes lengthy chemisorbed β-carotene molecules to immediately decompose to form shorter molecules. As this decomposition causes a colour change from white to grey-blue, the bleaching power (BP) is more conveniently measured by the adsorption of chlorophyll rather than β-carotene. In addition to the carbonaceous materials (Omar et al., 2003), a variety of raw and modified clays such as attapulgite (Huang et al., 2007), sepiolite (Tian et al., 2014; Laatikainen et al., 2015) and bentonite (Komadel, 2003; Noyan et al., 2007) have also been used for the bleaching of various edible and mineral oils. In general, raw and acid-activated clays are called ‘Fuller’s earths’ and ‘bleaching earths’, respectively (Zschau, 1985; Sohling et al., 2009; Emmerich et al., 2010; Liang et al., 2020). Such adsorbents are also used in other processes such as the clarification of wine, beer and fruit juice, as well as in the removal of contaminants from wastewater (Bomboş et al., 2014; Silva et al., 2014; Zhu et al., 2016; Uddin, 2017).

Bleaching earths are produced through the acid activation of calcium bentonites (Önal & Sarkinay, 2007; Komadel, 2016) after treatment with hydrochloric acid or sulfuric acid (H2SO4). Smeectites such as montmorillonite, saponite, hectorite, nontronite and beidellite are the major clay minerals of bentonites (Bergaya et al., 2013). The mineralogical and physicochemical characteristics of bentonites and other clays vary significantly depending on the acid as well as alkali treatments (Pentrák et al., 2009; Mache et al., 2015).

Although Rich (1964) stated that the oil quality is the most important parameter impacting BP (compared to the clay type and the bleaching method), the physicochemical properties of clay minerals and their effect on BP have been investigated systematically. The role of changing the physicochemical properties...
Table 1. The amounts of H2SO4 and water in the mud samples prepared using 20 g of bentonite. The effect of activation temperature was tested at 2 h and x(H2SO4) = 0.35 based on the optimum BP attained at various total structural metal oxides y(R2O3), as shown in Fig. 3. An activation temperature of 100°C, x(H2SO4) = 0.35 and y(R2O3) = 0.10 were tested to select the various activation times according to the lowest BP value observed in Fig. 4.

| Run # | x(H2SO4) | m(H2SO4) (g) | ν(H2SO4) (mL) | ν(H2O) (mL) |
|-------|-----------|--------------|---------------|-------------|
| 1     | 0.00      | 0            | 0             | 30.00       |
| 2     | 0.10      | 2.222        | 1.12          | 28.88       |
| 3     | 0.20      | 5.000        | 2.53          | 27.47       |
| 4     | 0.30      | 8.571        | 4.33          | 25.67       |
| 5     | 0.35x     | 10.769       | 5.44          | 24.56       |
| 6     | 0.40      | 13.333       | 6.73          | 23.27       |
| 7     | 0.50      | 20.000       | 10.10         | 19.90       |
| 8     | 0.60      | 30.000       | 15.15         | 14.85       |
| 9     | 0.70      | 46.670       | 23.57         | 6.43        |

x(H2SO4), t (h), T (°C)

|       | x(H2SO4) | T (°C) | t (h) |
|-------|----------|--------|-------|
| 10    | 0.35     | 100    | 0     |
| 11    | 0.35     | 100    | 2     |
| 12    | 0.35     | 100    | 5     |
| 13    | 0.35     | 100    | 7.5   |
| 14    | 0.35     | 100    | 15.0  |
| 15    | 0.35     | 100    | 20.0  |
| 16    | 0.35     | 100    | 250   |

x This specific x(H2SO4) concentration of 0.35 is selected for the next set of time and temperature series experiments based on highest BP observed in Fig. 2 (black dashed line).

of acid-activated montmorillonite on its bleaching efficiency was studied by Falaras et al. (1999), who showed that bleaching efficiency increases with increasing acidity and specific surface area, where medium activation of montmorillonite is the most efficient medium activation of montmorillonite. The effect of activation temperature was tested at 2 h and x(H2SO4) = 0.35 based on the optimum BP attained at various total structural metal oxides y(R2O3), as shown in Fig. 3. An activation temperature of 100°C, x(H2SO4) = 0.35 and y(R2O3) = 0.10 were tested to select the various activation times according to the lowest BP value observed in Fig. 4.

Materials and methods

Materials

A white CaB, obtained from the Çamlıdere, Ankara (Turkey) deposit, was used as the starting material after grinding to pass through a 0.074 mm (200 mesh) sieve and drying at 105°C for 4 h. The bulk chemical composition of the dried CaB (mass %) is SiO2: 72.1, Al2O3: 14.8, Fe2O3: 0.8, MgO: 1.6, CaO: 2.2, Na2O: 0.4, K2O: 1.1, TiO2: 0.1 and loss on ignition (LOI): 7.0. The cation-exchange capacity (CEC) estimated based on the methylene blue method was 0.78 eq. kg⁻¹ (Kipling & Wilson, 1960; Hang & Brindley, 1970; Rytwo et al., 1991; Kahr & Madson, 1995). The H2SO4 (98% by mass, 1.98 g cm⁻³) used in this process was of analytical grade. Lithium tetraborate (LiB4O7) for digestion was supplied by Merck. Alkali-refined soybean oil was supplied by a vegetable oil plant located in Marsa, Istanbul. TOBE was procured from Süd Chemie (Germany).

Acid activation

Twenty-three samples, each with a mass of 20 g, were weighed from the dried CaB powder and activated with H2SO4 using the dry method. The homogeneous muds prepared with the specific amounts of CaB, H2SO4 and distilled water were heated at various temperatures. The mass fraction of H2SO4 (x) in the dry CaB and pure H2SO4 mixture of the first eight muds varied between 0 and 0.70. The amounts of water and H2SO4 in the 30 mL solution used for the preparation of the muds were calculated and are given in Table 1. Each mixture was heated at 100°C for 2 h. Based on the first measurements, the highest BP was observed at the 0.35 mass fraction of H2SO4. Therefore, the mass fraction of H2SO4 in all time and temperature series experiments was set to 0.35. Next, for the temperature series experiments, a set of seven muds with x = 0.35 were heated for 2 h at temperatures ranging from 25°C to 250°C. An activation time of 2 h was selected based on the optimum BP attained at various total structural metal oxides. Finally, for the time series experiments, the last seven muds having x = 0.35 were heated at 100°C, with time increasing from 0 to 20 h. After activation, each sample was suspended in water and the solid residue was separated by centrifugation. Each residue was washed repeatedly with distilled water until the wash water was free of SO4²⁻ against BaCl2 solution. The BEs obtained were dried at 105°C for 4 h, ground so that they would pass through a 0.074 mm (200 mesh) sieve and kept...
in tightly closed plastic bottles. A total of 23 different BEs were prepared.

**Analytical procedures**

The pH value for each aqueous suspension having a BE content of 2% by mass was measured using an Orion Star A211 pH-Meter. The X-ray diffraction (XRD) traces for the raw CaB and acid-activated samples were recorded from mounts prepared using the glass slide method and run through a Rikagu D-max 2200 Diffractometer with a Ni filter and Cu-Kα radiation with a wavelength of 0.15418 nm (Moore & Reynolds, 1997). For chemical analyses, ∼0.25 g of dried sample was weighed into a platinum crucible, mixed with 3 g LiB₄O₇, fused at 1000°C for 1 h and cooled to room temperature. Then, the crucible was placed in a beaker containing 1000 mL of 10% HCl and heated to 90°C under stirring to dissolve the fused clays. The mass fractions of undissolved Al₂O₃, Fe₂O₃ and MgO in the CaB after the activation of the first nine samples were determined using atomic absorption spectrophotometry (AAS) with a Hitachi Z-8200 device. The results were given as the mass% of the metal oxides. The N₂ adsorption/desorption data at 77 K for all of the activated samples were obtained at high vacuum using a volumetric adsorption instrument with a Pyrex glass connection (Noyan et al., 2007). Before measurement, each sample was outgassed at 150°C for 4 h under vacuum (10⁻³ mmHg). The S and V values of the raw and acid-activated samples were calculated from the adsorption and desorption data, respectively (Rouquerol et al., 2014). The D parameter of the mesopores assumed to be cylindrical was calculated from the following expression (Onal & Sarıkaya, 2012; Rouquerol et al., 2014):

\[ D = \frac{4V}{S} \]

Bleaching experiments were carried out in an open 400 mL flask containing 2% by mass BE suspension in alkali-refined soybean oil. Each suspension was heated at 105°C for 15 min using the activated samples (Noyan et al., 2007). The oil was then filtered through Whatman No. 41 filter paper. The colour index of the oil in red-yellow units was determined using a Lovibond Automatic Tintometer (Type D) equipped with a 2.54 cm cell according to the American Oil Chemists’ Society (AOCS) Official Method (Taylor et al., 1989). The BP value for each sample was calculated from the expression

\[ BP = \frac{(R_0 - R)}{R_0} \]

where \( R_0 \) and \( R \) are the red colour units on Lovibond scale of the alkali-refined oil prior to and after bleaching, respectively.
Results and discussion

The XRD traces (Fig. 1a) revealed that the raw bentonite contained Ca-Sme, illite (Ilt), a kaolin group mineral (Kn), orthoclase (Or), quartz (Qz), opal-CT (Opl-CT) and probably some opal-A (Opl-A). The abbreviation symbols are after Warr (2020). As the major clay mineral was Ca-Sme, the raw material is CaB. Ilt and Kn are the minor clay minerals, whereas Or, Qz, Opl-CT and Opl-A are non-clay impurities. The basal spacing ($d_{001}$) values for the Ca-Sme, Ilt and Kn are 1.52, 1.00 and 0.72 nm, respectively. The XRD traces (Fig. 1) showed that the Ca-Sme and Ilt particles decomposed completely through acid activation at $x = 0.35$, but Kn, Or, Qz, Opl-CT and Opl-A were still observed. The structural cations Al$^{3+}$, Fe$^{3+}$ and Mg$^{2+}$ of Ca-Sme and Ilt dissolved during the H$_2$SO$_4$ activation. The mass fraction ($y$) of the corresponding metal oxides Al$_2$O$_3$, Fe$_2$O$_3$ and MgO and their sum, $R_xO_y$, decreased curvilinearly with increasing $x$ in the prepared muds (Fig. 2). Dissolution ceased at $x = 0.70$. The remaining Al$_2$O$_3$ was probably present in undissolved Kn and Or impurities. These results are in agreement with the present study, suggesting a correlation between specific surface area, acidity and pH. In a similar work, Mache et al. (2015) showed that Al, Mg and Fe were leached from the octahedral and tetrahedral sheets of Ca-Sme, while the SiO$_4$ groups of the tetrahedral sheets remained intact. Partial leaching of octahedral cations was correlated with the decrease in the MgO, Fe$_2$O$_3$ and Al$_2$O$_3$ contents during the acid activation of smectitic clays. Finally, Sanei et al. (2015) focused on the optimization of acid activation parameters during the bleaching of sepiolite and observed a similar decrease in structural cations and reduction in pH.

Changes in the $V$, $S$, pH and BP parameters were dependent on the $x$ and $y$ variables defined above (Fig. 3). The broad maxima of $V$ and $S$ are in the ranges $0.20 < x < 0.60$ and $0.04 < y < 0.16$. By contrast, the pH and BP values show a sharp minimum and a sharp maximum, respectively, for the same intervals. Their extreme values are pH = 3.0 and BP = 0.60 at $x = 0.35$ and $y = 0.10$. In addition, TOBE has an identical BP with the optimum BE that is used in the present study. Therefore, the acid-activation conditions for preparing the most suitable BEs are $x = 0.35$, $T = 100^\circ$C and $t = 2$ h (Fig. 3). Other investigated physicochemical properties of this sample were $y = 0.10$, $V = 0.31$ cm$^3$ g$^{-1}$ and $S = 225$ m$^2$ g$^{-1}$. Consequently, the BP was observed to be more dependent on the pH (acidity) than the $V$ and $S$ values. Our results are in partial disagreement with the work of Falaras et al. (2000) in terms of the BP of TOBE because Falaras et al. demonstrated that

\[ y(\text{Al}_2\text{O}_3, \text{Fe}_2\text{O}_3, \text{MgO}) = 0.10 \]

\[ y(\text{Fe}_2\text{O}_3) = 0.08 \]

\[ y(\text{MgO}) = 0.02 \]

\[ x = 0.35 \]

\[ y(\text{Fe}_2\text{O}_3) = 0.06 \]

\[ y(\text{MgO}) = 0.04 \]

\[ y(\text{Fe}_2\text{O}_3) = 0.02 \]
pillared, acid-activated montmorillonites have greater bleaching efficiency compared to the pillared products of the untreated clay. Therefore, the Al-pillared, acid-activated montmorillonite reached the same BP as TOBE. In the present study, we only reached a similar BP as TOBE for acid-activated CaB without Al pillaring. The conclusions of Falaras et al. (2000) on the increases in surface area, pore volume and acidity and decrease in octahedral sheet oxides during acid activation are in accord with our results when a similar treatment was applied to CaB.

The $V$, $S$ and BP values did not change with further increases in $T$ after reaching the optimum activation conditions (Fig. 4). Based on these results, the optimum acid-activation temperature was specified as 100°C. In contrast, the $V$, $S$ and BP values increased with increasing $t$ until they reached a plateau after 10 h of treatment (Fig. 5). Even though increasing the acid-activation time from 2 to 10 h caused an increase of $V = 0.46 \text{ cm}^3 \text{ g}^{-1}$, $S = 260 \text{ m}^2 \text{ g}^{-1}$ and BP = 0.65, 10 h of heating for the activation of mud is probably not economic given the modest improvement in performance.

The mean mesopore size for each acid-activated sample was calculated from Equation 1. The variation of the BP as a function of $D$ indicates that the effective bleaching mesopore sizes range from 4 to 9 nm. The optimum $D$ value against the maximum BP is 7 nm (Fig. 6). Consequently, in addition to pH, $D$ is more impactful on the bleaching of vegetable oils compared to the $V$ and $S$ values of the BE. Huang et al. (2007) investigated the impacts of pore-size distributions and specific surface areas of three attapulgite samples on the bleaching of soybean oil and suggested that the most effective pore size for the best BP is 0.8–3.2 nm. The similarity of the pore size of attapulgite to the diameter of the pigments enhanced the BP by enabling the rapid removal of these pigments. Even though various BEs were used in this study and that of Huang et al. (2007), the conclusion of Huang et al. regarding the importance of compatibility between the BE and pigments on the BP is in accordance with the present work.

The adsorption of coloured pigments onto the BE depends not only on the physicochemical interactions between large organic molecules and the BE surface, but also on the acidity and mesopore size. The compatibility in size between the pigments and internal widths of the mesopores in the BE is the most important parameter that controls the BP.

**Conclusions**

Various BEs were produced using $H_2SO_4$ activation of a CaB under varying mass fractions of $H_2SO_4$, heating temperature

**Fig. 3.** Variation in $V$, $S$, BP and pH depending on the mass fractions of $x(H_2SO_4)$ and $y(R_xO_y)$. 

https://doi.org/10.1180/clm.2021.28 Published online by Cambridge University Press
Fig. 4. Variation in $V$, $S$ and BP depending on the process temperature in experiments conducted for 2 h at the constant value of $x(H_2SO_4) = 0.35$ and corresponding $y(R_xO_y) = 0.10$.

Fig. 5. Variation in $V$, $S$ and BP depending on the process time in experiments conducted at 100°C at the constant value of $x(H_2SO_4) = 0.35$ and corresponding $y(R_xO_y) = 0.10$. 

$V$ (cm$^3$ g$^{-1}$) 
$S$ (m$^2$ g$^{-1}$) 
BP
and contact time in an attempt to bleach an alkali-refined oil. The main conclusions are as follows:

(1) The 2:1 clay minerals such as montmorillonite and Ilt decomposed completely after treatment with an adequate amount of H2SO4. In contrast, the 1:1 kaolin group clay mineral Or as well as Qz, Opl-CT, and Opl-A were less affected by the activation.

(2) Activation results in a non-linear decrease in the Al2O3, Fe2O3 and MgO of the clays with increasing mass fraction of H2SO4. To fully constrain this relationship, the direction and surface area of bentonite, illite, and kaolinite by methylene blue adsorption capacity of bentonites from the island of Milos and Chios, Aegean, Greece. Applied Clay Science, 12, 329–347.

(3) The micropore and mesopore volumes, specific surface area and BP of the BEs reach their maximum values when the temperature of the activation, but also depend on the type and ratio of the raw bentonite and H2SO4, as well as the temperature and duration of activation.

(4) The most effective internal width of the mesopores is 7 nm.

(5) The optimum conditions for the production of the most effective BE for an alkali-refined vegetable oil depends on the type and ratio of the raw bentonite and H2SO4, as well as the temperature and duration of activation.

(6) To obtain more comprehensive results, the optimum parameters need to be determined individually for each raw bentonite and edible oil pair.

Acknowledgements. The authors thank the reviewers and S. Kaufhold for their detailed and constructive comments that improved the quality and clarity of the paper. The authors are also grateful to G. Christidis for the editorial handling of this manuscript.

Financial support. This research was supported by Ankara University Scientific Research Projects Coordination Unit (19L0430007).

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