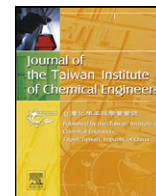




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# Performances of peanut hull ashes in bleaching water-degummed and alkali-refined soy oil

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## ABSTRACT

Peanut hull ashes prepared by calcinating ground peanut hull were evaluated as adsorbent for bleaching the water-degummed and alkali-refined soy oil. Their performances in adsorbing peroxides, phospholipids, pigments,  $\beta$ -carotene and free fatty acids in soy oil were compared with those of peanut hull, rice hull ash, activated clay and regenerated clay. Experimental results indicated that the influence of calcination time on the removal efficiency was not clear. Temperature of 500 or 700 °C showed potential as a best calcination temperature for preparing effective adsorbent. On the basis of adsorbing peroxides, phospholipids, pigments,  $\beta$ -carotene and free fatty acids, activated clay and regenerated clay were found to be superior adsorbents except in adsorbing free fatty acids; peanut hull ashes, effective adsorbents; while peanut hull, non-effective one. Further activation on peanut hull ashes is suggested to obtain a better performance while regenerated clay was found to be a good substituent for activated clay.

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## 1. Introduction

Degumming, neutralization, bleaching and deodorization are the four steps in oil refining (Bockisch, 1993). The function of the bleaching step is to remove or reduce the following components in water-degummed and alkali-refined soy oil: oxidation products, phosphatides, soaps, trace metals and pigments (Erickson, 1995). To achieve this goal, natural clay, acid-activated clay, activated carbon and silicates are normally used as an adsorbent (Erickson, 1995). Among these, activated carbon is expensive while natural clay and silicate is less effective (Erickson, 1995). Therefore, acid-activated clay is the most popular adsorbent employed. However, the clay used in Taiwan must be imported from Indonesia. To save the foreign exchange from buying the imported clay, investigation on searching for a substituent which is abundant and less cost is worthwhile to carry out in Taiwan.

Regenerated clay (Lee and Lin, 2004; Lin and Lin, 2005; Ma and Lin, 2004; Tai and Lin, 2007; Wang and Lin, 2006), soy hull and its derivatives (Gnanasambandam *et al.*, 1998; Proctor and Harris, 1996; Proctor and Gnanasambandam, 1997a, 1997b), rice hull and its derivatives (Chang *et al.*, 2001a, 2001b; Liew *et al.*, 1993; Proctor and Palaniappan, 1989, 1990; Proctor *et al.*, 1992, 1995) as well as

peanut hull and its derivatives are among the potential substituents studied.

Peanut hull is an agriculture waste and is normally treated as a fertilizer in Taiwan. Carbon produced from peanut hull has been found to remove the heavy metal ions in waste water efficiently (Namasivayam and Periasamy, 1993; Periasamy and Namasivayam, 1994, 1995). Unfortunately, no report has been found on examining the possibility of using peanut hull or its derivatives to bleach the soy oil. Therefore, the removal efficiencies of peanut hull and peanut hull ashes (PHA) prepared under different calcination times and different calcination temperatures were examined in this study. To evaluate the removal efficiencies of the different ashes prepared, the concentrations of the following components or the indices of water-degummed and alkali-refined soy oil before and after bleaching were measured: peroxides, phospholipids, Lovibond red index,  $\beta$ -carotene and free fatty acids.

Finally, the removal efficiencies of peanut hull (PH), rice hull ash (RHA), activated clay (AC) and regenerated clay (RC) were also determined for the purpose of comparison.

## 2. Experimental

### 2.1. Materials

Compressed air and nitrogen gas (Yuan-Ron, Taipei, Taiwan) with a minimum purity of 99.99% and 99.55%, respectively, were

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employed. The chemicals, except  $\beta$ -carotene which was of HPLC grade, used for the analysis of the contents of the water-degummed and alkali-refined soy oil were of reagent grade. Many chemicals have been employed in this study. Acetic acid, ethanol, hydrazine sulfate, hydrochloric acid, phosphate, potassium dihydroxide, potassium hydroxide, sodium molybdate, sodium thiosulfate, sulfuric acid, trimethylpentane and zinc oxide were all supplied by Acros Organics (Geel, Belgium). Hexane, pH buffer solution, potassium hydrogen phthalate, potassium hydroxide, potassium iodate and  $\beta$ -carotene were purchased from Mallinckrodt Baker (Louisville, KY, USA), Optimum Instrument & Application (Taipei, Taiwan), Merck (Darmstadt, Germany), Cerac (Milwaukee, WI, USA), Shimadzu's Pure Chemicals (Osaka, Japan) and Sigma (St. Louis, MO, USA), respectively.

Water-degummed and alkali-refined soy oil, the activated clay and the spent clay (Optimum 230FF, P. T. Süd-Chemie, Indonesia) were donated by the TTET Union Corporation (Tainan, Taiwan). The soy oil prior to bleaching was stored in a refrigerator at 4 °C and flown over by a nitrogen stream (3 mL/s) to protect it from being oxidized.

## 2.2. Preparing adsorbents

Peanut hull provided by a farmer of Chang-hua County was washed three times with distilled water, ground by a ball mill and dried in an oven at 100 °C for 24 h. Peanut hull powder between 270 and 325 mesh, obtained by screening, was employed as an adsorbent in the bleaching experiment. Most of the peanut hull was further processed. Eleven to twelve grams of dried peanut hull was then placed in a deep alumina boat of 16 cm  $\times$  4 cm  $\times$  2 cm. A box furnace (Kingswang VT-10, Taipei, Taiwan) was heated up from room temperature under an air stream (20 mL/s). When the furnace was raised to a desired temperature (300–1000 °C) and had been maintained there for 30 min, the boat with peanut hull powder was placed into the furnace. The air was continued to flow during the process of calcination. When a predetermined time (45–225 min) was reached, the boat was removed and air cooled.

Rice hull obtained from a rice miller in Tainan county was washed with distilled water three times, dried at 100 °C for 24 h and then calcinated at 500 °C under 20 mL air/s for 50 min.

The activated clay and the regenerated clay employed in previous study (Lin and Lin, 2005) were used in this work, again. The regeneration conditions for the regenerated clay are briefly reported here: 500 °C, 20 mL air/s and 50 min.

The contents of silicon and metals of the PH, PHA500\* (peanut hull ash prepared at 500 °C for 180 min) and RHA were determined by an inductively coupled plasma-mass spectrometer, ICP-MS (Sciex Elan 5000, PerkinElmer, Shelton, MA, USA) while the contents of carbon in PH, PHA300\*, PHA500\*, PHA700\*, PHA1000\* and RHA were determined by a PerkinElmer 2400-CHN elemental analyzer (PerkinElmer, Norwalk, CT, USA). PHA300\*, PHA500\*, PHA700\* and PHA1000\* mentioned above represent peanut hull ashes prepared at 300, 500, 700 and 1000 °C, for 180 min, respectively as well as PHA300, PHA500, PHA700, PHA1000, prepared at 300, 500, 700 and 1000 °C, for all calcination time, respectively. The morphologies of these adsorbents were observed with a scanning electron microscope (S360, Cambridge, Cambridge, United Kingdom). Mean particle diameters and true densities of the adsorbents were measured by a laser diffraction particle size analyzer (LS-230, Beckman Coulter, CA, USA) and a density meter (Accupy 1330, Micromeritics, Norcross, GA, USA), respectively. Specific surface areas, specific pore volumes and average pore diameters were obtained from a surface area analyzer (AS1MP-LP-VP2, Quantachrome, Boynton Beach, FL, USA).

## 2.3. Procedure for bleaching experiments

One hundred grams of water-degummed and alkali-refined soy oil was poured into a 300 mL three-neck round bottom flask and 0.6 g of adsorbent was stored in the neck above the valve prior to bleaching. The oil in the flask was stirred by a magnetic stirrer and heated up by an oil bath. A vacuum was maintained in the system by a mechanical pump during the heating stage and the consequent bleaching process. When the oil reached 110 °C and had been maintained there for 15 min, the valve was opened and the regenerated clay was dropped into the oil with continuous stirring. The bleaching was then started isothermally. Twenty minutes later, the bleaching was stopped by replacing the oil bath with an ice/water bath. After cooling to room temperature, the slurry in the flask was filtered by a Toyo 5C filter paper using vacuum suction and the filtered oil was collected to determine the concentrations of the following components or the indices: peroxide value (PV) (Tai and Lin, 2007), phospholipids (PL) (Lee and Lin, 2004), Lovibond red index (LRI) (Chang et al., 2001b),  $\beta$ -carotene ( $\beta$ C) (Ma and Lin, 2004) and free fatty acids (FFA) (Wang and Lin, 2006).

The operating conditions for an industrial bleaching step are normally as follows: 50 mmHg vacuum, 100–110 °C, 20–30 min and 0.03–0.006 g adsorbent/g oil (Erickson, 1995). To be close to these, following conditions are chosen in this work: 50 mmHg vacuum, 110 °C, 20 min, 0.006 g adsorbent/g oil, and 800 rpm.

Each determination was made in triplicate and the average value was employed.

## 2.4. Calculation of removal efficiency

Five types of removal efficiencies based on different components concentrations or indices can be calculated according to following equations:

$$RE(PV) = \frac{C_{PV_0} - C_{PV}}{C_{PV_0}} \times 100\% \quad (1)$$

$$RE(PL) = \frac{C_{PL_0} - C_{PL}}{C_{PL_0}} \times 100\% \quad (2)$$

$$RE(LRI) = \frac{C_{LRI_0} - C_{LRI}}{C_{LRI_0}} \times 100\% \quad (3)$$

$$RE(\beta C) = \frac{C_{\beta C_0} - C_{\beta C}}{C_{\beta C_0}} \times 100\% \quad (4)$$

$$RE(FFA) = \frac{C_{FFA_0} - C_{FFA}}{C_{FFA_0}} \times 100\% \quad (5)$$

where  $C_{FFA}$  and  $C_{FFA_0}$  are the concentrations of free fatty acids (%) at  $t = t$  and  $t = 0$ , respectively;  $C_{LRI}$  and  $C_{LRI_0}$  the dimensionless Lovibond indices at  $t = t$  and  $t = 0$ , respectively;  $C_{PL}$  and  $C_{PL_0}$  the concentrations of phospholipids (ppm) at  $t = t$  and  $t = 0$ , respectively;  $C_{PV}$  and  $C_{PV_0}$  the peroxide values (mequiv./kg) at  $t = t$  and  $t = 0$ , respectively; as well as  $C_{\beta C}$  and  $C_{\beta C_0}$  the concentrations of  $\beta$ -carotene ( $10^{-7}$  mol/L), respectively.

## 3. Results and discussion

### 3.1. Contents and physical properties of adsorbents

The contents and physical properties of PH, PHA and RHA were determined in this work while those of AC and RC were reported in a previous study (Tai and Lin, 2007). The results are shown in

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