

Soybean Oil Bleaching by Adsorption onto Bentonite/Iron Oxide Nanocomposites

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Published online: 25 August 2018

To cite this article: Ghorbanpour, M. (2018). Soybean oil bleaching by adsorption onto bentonite/iron oxide nanocomposites. *J. Phys. Sci.*, 29(2), 113–119, <https://doi.org/10.21315/jps2018.29.2.7>

To link to this article: <https://doi.org/10.21315/jps2018.29.2.7>

ABSTRACT: *The bleaching process of soybean oil using commercial bentonite and bentonite/iron oxide composites has been studied. X-ray diffraction (XRD), Brunauer-Emmett-Teller (BET) surface area measurement and scanning electron microscopy (SEM) were used to characterise the composites generated. SEM results show that the porosity of bentonite after alkaline ion exchange process can be enhanced by the opening of the bentonite's flakes. BET shows that the flakes' structure was more opened, and the porosity was increased from 179.58 m² g⁻¹ for bentonite to 202 m² g⁻¹ for 3 min ion exchanged sample. Changes in basal reflection in XRD peak validated the presence of iron oxide particles. The experimental results indicate that composite prepared for 1 min showed the same efficiency in bleaching crude soybean oil with the bentonite. The greatest reduction in bleaching capacity in soybean oil was achieved using the composite prepared for 3 min. The highest transparency, 1.5-fold in red and 1.25-fold in yellow greater than that of neutralised oil, was obtained with the alkaline ion exchange composite prepared for 3 min. Hence, this process gives a good adsorbent with better bleaching properties than commercial bentonite.*

Keywords: Bentonite composite, iron oxide composite, soybean oil, bleaching, nanocomposite

1. INTRODUCTION

Among the criteria of edible oil quality, colour is the most important factor for its commercial value. The colour is due to the presence of pigments in the crude oil such as chlorophyll-a and β -carotene. The bleaching of edible vegetable oils

involves removal of a variety of impurities, which include phosphatides, fatty acids, gums and trace metals, etc., followed by decolourisation.^{1,2} In refinery processing of vegetable oils, adsorbents are used to remove carotene, chlorophyll and other components formed during the refining process. Common adsorbents are hydrated aluminium silicates, commonly known as bleaching clays. They are purified and activated by a mineral acid treatment, resulting in the de-lamination of the structure, thus increasing clay specific surface and adsorption capacity.^{3,4} Among them, activated bentonites is by far the most common adsorbent for purification and colour improvement of fats and oils.^{1,3,5}

Metal oxides have recently been applied to remove heavy metals and dyes from water and wastewater.^{6,7} Clays/metal oxide composites are one of the most widely studied composites among the new group of developed microporous materials with high surface area.⁸⁻¹⁰ The first and most common method in the preparation of these composites is the ion exchange process with heat treatment.^{9,11} The negative aspect of the ion exchange process is its lengthy processing and multistep prepare procedures. Recently, a number of studies appear in the literature on the application of alkaline ion exchanged clays to prepare antibacterial composites.^{12,13} In alkaline ion exchange method, iron salt is mixed with bentonite and heated at around the melting point of iron salt. In this study, for the first time, bentonite/iron oxide composites were prepared by alkaline ion exchanged method used for decolourisation of soybean oil.

2. EXPERIMENTAL

2.1 Materials

Bentonite clay (Ca^{2+} -montmorillonite), used as a solid support for iron oxide particles, was obtained from Kanisaz Jam Company (Rasht, Iran). Prior to the experiments, the bentonite was sieved to give a particle size of roughly 38 μm . All reagents were of analytical grade and were used as received without further refinement.

2.2 Clays/Iron Oxide Composites

Bentonite was immersed in molten salt, $\text{FeCl}_2 \cdot x\text{H}_2\text{O}$, at 100°C for 1 min, 2 min, 3 min and 5 min. This operation was undertaken using 5 g of bentonite and 5 g of $\text{FeCl}_2 \cdot x\text{H}_2\text{O}$. After ion exchange, the bentonite was adequately washed with distilled water and sonicated. This step was intended to remove any compounds that were not diffused in the bentonite structure. After filtration, the obtained composites were dried in an oven for 24 h at 25°C.

2.3 Characterisation

X-ray diffraction (XRD) patterns of the samples were characterised using an X-ray diffractometer (Philips PW 1050, the Netherlands) with $\text{CuK}\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$, 40 kV and 30 mA, 2θ from 0° to 80° and 0.05° step). The microstructures of the samples were observed through a scanning electron microscope (SEM) (LEO 1430VP, Germany).

A micromeritics Brunauer-Emmett-Teller (BET) surface area and porosity analyser (Gemini 2375, Germany) was used to evaluate the products with N_2 adsorption/desorption at constant temperature of 77 K in the relative pressure range of 0.05–1.00.

2.4 Bleaching of Edible Oil

The bleaching process was carried out at a constant temperature of 80°C with a contact time of 30 min. Stirring and heating were carried out by means of a magnet stirrer and an electric heating band. The ratio of the mass of clay to the volume of acid solution was 1:10 (w/v). The hot oil and clay mixture was filtered under vacuum and the colour of the bleached oil was measured spectrophotometrically. The bleaching capacity percentage of the clays was determined from the following equation:

$$\text{Bleaching capacity (\%)} = [(A_0 - A)/A_0] \times 100 \quad (1)$$

where A_0 and A are the absorbance of neutral oil and bleached oil, respectively, at the maximum absorbance wavelength of the neutral oil (410 nm for uptake of chlorophyll-a, and 460 nm for uptake of b-carotene).

3. RESULTS AND DISCUSSION

3.1 Characterisation

Appearance and colour of the parent bentonite was white. Alkaline ion exchange of the bentonite by FeCl_2 changed its colour. By increasing alkaline ion exchange process time at a constant temperature, the colour of bentonite underwent further change. After 1 min, the colour was altered to dark cream, creamy brown after 2 min, and finally after 5 min changed to red. The following equation describes the transition:



Consequently, high temperature resulted in changing the replaced iron ions to iron oxide particles.



It might be concluded that the colour variation is due to the oxidation state of loaded iron to the bentonite.

The morphology of natural bentonite and the prepared composites at different ion exchange times was studied in this paper. The SEM images are shown in Figure 1. In Figure 1(a), the bentonite displays a leafy sheet surface texture with a loose and porous microstructure. This is a typical morphological characteristic of such material. After ion exchange, the structure of the parent bentonite displays some changes. The only difference is that the edges of the leafy sheets of bentonite seem to become thicker and the flakes' structure was more opened.

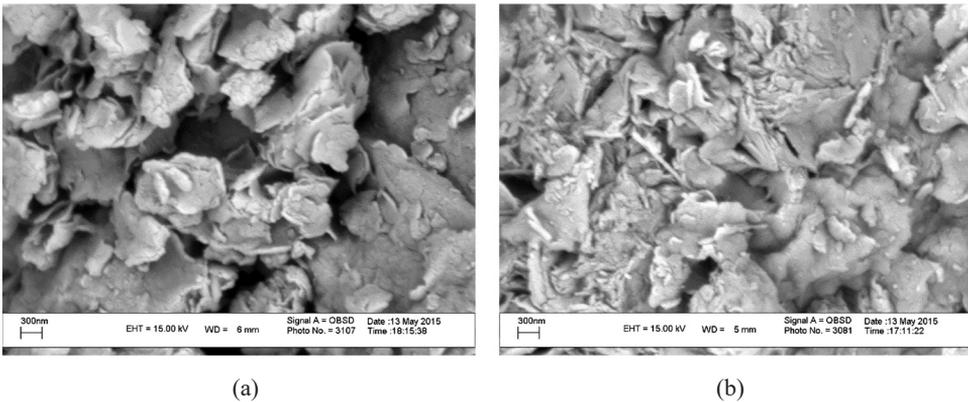


Figure 1: Image of (a) the parent bentonite and (b) ion exchanged bentonite for 3 min.

Figure 2 illustrates the XRD pattern of pure bentonite and composite at different times. A typical pattern was observed for the bentonite, with an intense $2\theta = 6.27$ reflection relative to the basal spacing d_{001} (1.44 nm). The other reflections correspond to montmorillonite's crystalline structure ($2\theta = 19.84^\circ, 20.87^\circ, 26.68^\circ, 27.65^\circ, 32.47^\circ, 34.90^\circ, 50.23^\circ$ and 60.13°).^{12,13} These peaks appear in the patterns of alkaline ion exchange composites. After the ion exchange, the original d spacing in the montmorillonite clay decreased to 1.29 nm for 3 min. This is due to the loss of water initially present in the interlayers. Some reported data showed higher d -spacing after ion exchange, compared with bentonite.^{10,11} The reason may be due to the use of a salt solution for the process of ion exchange, which increased the number of ions between the layers, resulting in swollen bentonite. In this study,

d-spacing of the composite is less than in the parent bentonite. Here, there are two main reasons. First, the reaction is accomplished in a solid phase, and second the high process temperature evaporates the moisture between the layers.

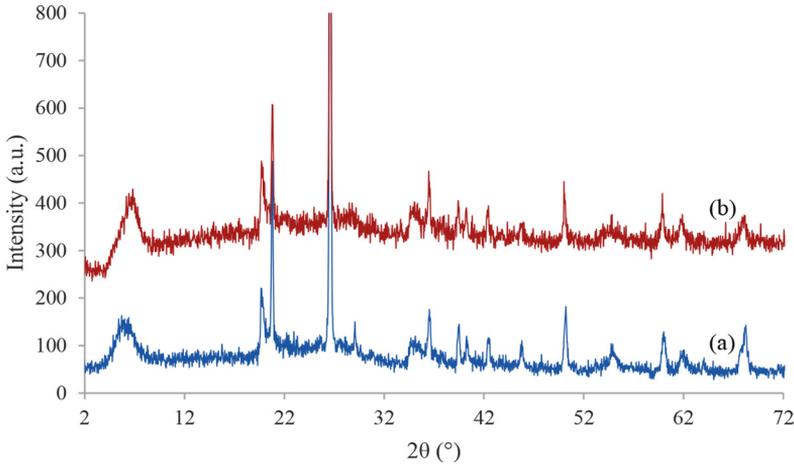


Figure 2: The XRD pattern of (a) pure bentonite and (b) ion exchanged bentonite for 3 min.

A comparison of porosity for parent bentonite and alkaline ion exchange composites by BET analysis was carried out. At first, the bentonite sample had $179.58 \text{ m}^2 \text{ g}^{-1}$ porosity. After 3 min, porosity was increased to $202 \text{ m}^2 \text{ g}^{-1}$. The reason is due to diffusion of iron ions to the bentonite. Given the SEM results, the porosity of composite can be enhanced by the opening of the bentonite's flakes.

3.2 Bleaching Efficiency

When bleaching refined soybean oil, chlorophyll reduction is the most important quality parameter. Results from the bleaching runs for soybean oil using alkaline ion exchanged bentonite and commercial bentonite used as a reference are presented in Figure 3. The greatest reduction in neutralised oil using the commercial bentonite was achieved in red by a factor of 26.2% and in yellow units by a factor of 27.1%. The experimental results indicate that composite prepared for 1 min has the same efficiency in bleaching crude soybean oil with the bentonite. The highest transparency, 1.5-fold in red and 1.25-fold in yellow greater than that of neutralised oil, was obtained with the alkaline ion exchange composite prepared for 3 min. Substantial removal of colour substances makes the oil highly transparent. It is obvious that composites displayed more bleaching efficiency than the commercial bentonite currently in use by the oil industry.

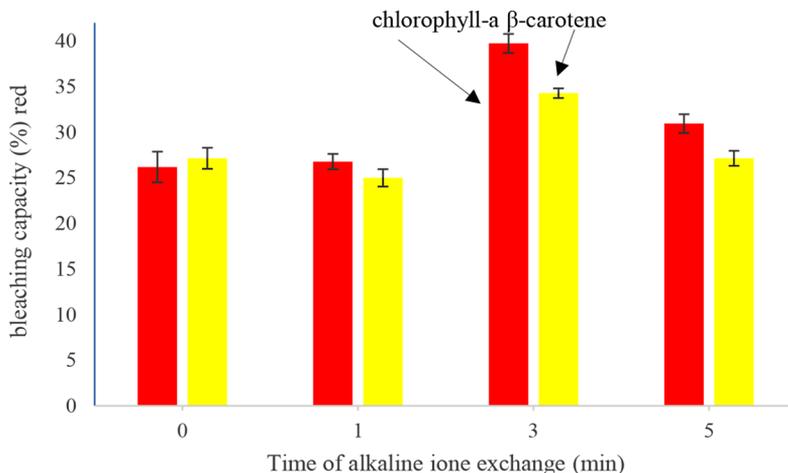


Figure 3: Bleaching capacity by using commercial bentonite and alkaline ion exchanged bentonite prepared at different times.

Briefly, iron oxide particles enhanced removal capabilities towards various pigments in the crude oil. On the other hand, alkaline ion exchange process was found to increase the porosity, which implied an increase in surface area, and hence the adsorption capacity.

4. CONCLUSION

The alkaline ion exchange process increased the surface area of the bentonites. Based on the test data, the highest transparency, 1.5-fold in red and 1.25-fold in yellow greater than that of neutralised oil, was obtained with the alkaline ion exchange composite prepared for 3 min. Thus, bentonite/iron oxide can efficiently decolourise crude soybean oil through removal of colouring agents like b-carotene. This process gives a good adsorbent with better bleaching properties than commercial bentonite.

5. REFERENCES

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