



Original Research Paper

Preparation and characterization of carbon/montmorillonite composites and nanocomposites from waste bleaching sodium montmorillonite clay



P. Anadão*, I.L.R. Pajolli, E.A. Hildebrando, H. Wiebeck

Metallurgical and Materials Engineering Department, School of Engineering, University of São Paulo, Av. Prof. Mello Moraes, 2463, CEP 05508-900, Cidade Universitária, São Paulo, SP, Brazil

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ABSTRACT

Waste bleaching sodium montmorillonite clay was used to prepare carbon/clay nanocomposites and composites by calcination in a reducing atmosphere. The main purpose of this study was to determine the influence of the calcination temperature and solvent washing in the material structure and its adsorption properties. X-ray diffraction patterns detected the nanocomposite formation only in the samples calcinated at 350 °C, whose structures were also described by Fourier transform infrared spectra. SEM images showed that all the samples were composed of various agglutinated grains and the non-washed sample calcinated at 350 °C presented the highest carbon recovery as its surface was the smoothest one, as confirmed by thermogravimetry curve. As a result of this higher carbon content, its methylene blue and gasoline adsorption capacities were the highest, albeit a bit lower in comparison to activated carbon due to the hydrocarbon formation onto sample surface. Finally, BET and BJH studies showed that porosity should also be improved in order to achieve higher adsorption values.

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1. Introduction

Carbon/mineral composites are a class of materials that can be used as a support in catalyzers, adsorbents in chromatography, water and wastewater purification technology, hydrogen storage, among others [1–3]. Their versatility is due to their polar (metallic oxides) and non-polar (surface recovered by carbon) portions which allow adsorption of polar and non-polar [4].

Specifically in water treatment, the most used adsorbent is activated carbon which presents high adsorption efficiency due to its high surface area. Nevertheless, besides its expensive cost [5], beds of this material, when pressurized by water, acquire preferential paths which contribute to water percolation in a non-uniform way through the cross section of the activated carbon filter and, consequently, these paths can reduce adsorption potentiality as well as filtration performance and lifetime. Moreover, another disadvantage is that activated carbon also presents low mechanical resistance that reduces the lifetime of the adsorption column since pulverization leads to the loss of granulated material [6].

Considering all the benefits of carbon/mineral composites, research on the preparation of carbon/mineral composites has been performed in the last decades. In 1988, Sonobe, Kyotani and

Tomita proposed a procedure for obtaining a microporous carbon graphite with a high degree of orientation, from a Japanese smectitic clay and polyacrylonitrile [7]. The method consisted in introducing nitrile acid between clay mineral layers by means of monomer vapor contact with the clay for 24 h at room temperature. The monomer was polymerized by the exposition to the radiation of gamma rays and the resulting silicate/polymer nanocomposite was calcinated at 700 °C and grafted by posterior heating at 2300 °C. The resulting carbon graphite showed a high degree of orientation and lamellar distances as compared to materials grafted at temperatures higher than 3000 °C and this highly oriented carbon was a result of the orientation of the two-dimensional carbon precursor produced between the clay mineral layers since when polyacrylonitrile itself was heated, it produced a three-dimensional carbon with a network of intertwined ribbons of stacked graphitic sheets. In addition, this research group also published works related to the preparation of long-range ordered microporous carbons with the structural regularity of zeolite according to the template carbonization method, proving that the template is very important to determine final carbon morphology [8,9].

The successful technique developed by Tomita was then used by several other research groups and new materials, with better properties, were developed. As an example, Bandosz and coworkers prepared, according to Tomitás technique, carbon by the introduction of FA and of polypropylene glycol in taeniolite previously

* Corresponding author. Tel.: +55 11 30912279; fax: +55 11 30912275.

E-mail addresses: priscila.anadao@gmail.com (P. Anadão), ivanpajolli@yahoo.com.br (I.L.R. Pajolli), edemarin@usp.br (E.A. Hildebrando), hwiebeck@usp.br (H. Wiebeck).

pillared with aluminum and zirconium hydroxy cations. The carbon production was dependent on substrate as well as precursor and obtention method. By varying these parameters, materials with a wide range of properties such specific area and microporous volume could be obtained [10].

The research group indeed with an expressive production related to the adsorbent mineral-carbon complexes belongs to Professor Lebeda [11–15], from the University of Marie Curie, in Poland. In most of them, they proposed different matrices from those already used, which were generally aerosols (silica fume) or silica gel, and methylene chloride as a precursor, hence, carbon with different structures could be obtained. Depending on the substrate and time of reaction, a wide spectrum of materials with different topographies, microporosities, pore volumes and specific areas could be obtained.

Moreover, the research on preparing different types of carbon continued and, in the last decade, special attention has also been paid to the preparation of carbon nanostructures, specially, carbon nanotubes [16] and carbon nanofibers [17] by using different substrates. The use of clay minerals, loaded with transition metals, as supports to the catalytic synthesis of carbon nanotubes by catalytic chemical vapor deposition has been described as a successful way to produce these materials [18] and it was found that the quality and the yield of carbon deposits were affected by the clay nature [19]. Important applications have been destined to these carbon nanotubes produced by using clay as support, as, for example, to be applied in hydrogen peroxide biosensor with great final efficiency [20].

However, most of the techniques described previously are expensive and complicated. Hence, aiming to decrease final material cost, cheap carbon precursors are also desirable and so, they have been investigated in carbon preparation [21,22]. Besides that, as many naturally-occurring clay minerals in their pristine form can act as adsorbents [23], they can be used to enhance the adsorption properties of the final carbon/mineral adsorbents. As an option of cheap carbon precursor, natural spent clay of bleaching processes can also be considered, which after being used in bleaching processes of several products clarification such as oil [24], juice [4] or still sewage sludge [25], it is composed of a mixture of mineral materials (one or different types of clay) and organic remains. When it is pyrolyzed under low temperatures, carbon can be produced onto clay mineral surfaces.

The use of the spent clay to produce carbon also represents an environmental problem solution. Once soybean oil is a largely used product in the food sector and can be used as a strategic source of renewable energy to produce biodiesel as a replacement to diesel oil and other petroleum-based fuels [26], a large content of waste deriving from soybean oil production has been produced since then and the disposal of the materials used in its production may cause environmental problems. The valorization of these materials, by converting them into functional materials by the deposition of carbon onto clay mineral layers [27], also plays an environmental objective.

Therefore, as clay has been widely used as a template to produce carbonous material, this paper suggests the possibility to use waste bleaching sodium montmorillonite clay (MMT), a resulting by-product of soybean oil clarification, which, after a time of work, lost its ability of oil clarification once a large content of impurities was adsorbed by the MMT. The influence of washing with a solvent and the temperature of pyrolysis was investigated according to their resulting structural and thermal characteristics. Their polar and non-polar affinities were studied by adsorption tests with two substances: methylene blue (polar) and gasoline (non-polar), in order to evaluate their adsorption efficiency.

2. Material and methods

2.1. Materials

The used clay was the waste bleaching soybean oil, which was contaminated with oil and other organic compounds which constituted soybean. The sample was kindly provided by Unilever (Valinhos, SP) and it was an acid activated-montmorillonite clay. N-hexane 99% P.A., ethanol, activated carbon and methylene blue were acquired from Vetec Química LTDA and used as received. Gasoline was supplied by Shell distributor. In all the experiments, distilled water was used. Calcinated montmorillonite clay at 500 °C as well as activated carbon were used as a reference material.

2.2. Preparation of the carbon/clay composites and nanocomposites

50 g of the activated clay was calcinated up to three different temperatures: 350, 500 and 600 °C. The calcination was carried out in a reducing atmosphere up to the final temperature with a heating rate of 10 °C/min, kept at the final temperature for 30 min and cooled to room temperature. For each temperature, one sample was calcinated as received and the other was washed with n-hexane, with a ratio of 1 mL of n-hexane for each gram of sample. After eleven-day air drying, each n-hexane washed sample was heated to eliminate any n-hexane residue.

2.3. Material characterization

The X-ray diffraction (XRD) patterns of the calcinated samples were obtained after passing them through an ABNT 30 mesh (Tyler-28 open of 0.60 mm). A Philips model X'PERT-MPD with Cu K α was used under the following conditions: 40 kV–30 mA; Cu K α radiation ($\lambda = 0.154$ nm); at the rate of 0.001°/min in the range of 2–90° (2 θ).

Fourier transform infrared spectroscopy (FTIR) spectra of the samples were recorded by employing potassium bromide (KBr) pressed disc technique (rate of 1 mg of sample to 200 mg of KBr) from 4000 to 400 cm⁻¹ by using a Fourier Transform Infrared Spectrophotometer (FTIR-8300). For comparison purposes, FTIR spectrum of the calcinated MMT was also recorded.

Moreover, all the samples were analyzed by a Philips XL-30 microscope. They were fixed in a carbon tape and covered by gold with a voltage of 20.0 kV.

Aiming to study the samples thermal behavior, thermogravimetry (TGA) was performed. TGA curves were obtained by using a Shimadzu thermobalance, model TGA-51, at the temperature range of 25–950 °C under N₂ atmosphere with a flow of 50 mL/min, heating rate of 10 °C/min and sample mass of about 5 mg.

2.4. Gasoline adsorption test

Aiming to analyze non-polar adsorption efficiency of this new material, gasoline adsorption was evaluated by triplicate experiments in accordance with the method described in the “Standard Methods of Testing Sorbent Performance of Adsorbents” [28,29]. This test was performed by filling a borosilicate glass vessel up to a 2-cm height (or half of the total volume). 1 g of the sample was placed in a stainless steel basket (mesh 200, opening of 0.075 mm) weighed initially. This set was weighed and placed inside the vessel with the fluid, staying there for 15 min. After this period, the set was allowed to drain for 15 s and was weighed immediately. The content of adsorbed fluid was calculated by Eq. (1):

$$C = [(W_1 - W_2)/W_2] \quad (1)$$

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