



## Research paper

# Influence of magnetic field on the adsorption of organic compound by clays modified with iron



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## ABSTRACT

Insertion of iron into montmorillonite (Mt) resulted in two modified materials, when different treatments were used: i) pillared clay (FePILC) and ii) magnetic clay (FeMAG). The ability of the modified clays to remove the organic dye methylene blue (MB) by adsorption was tested. Additionally, we evaluated the effects of adsorption after exposure to a pulsed magnetic field, the results were monitored by UV–vis spectroscopy and chemical analysis of total organic carbon. All materials were characterized by X-ray powder diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), temperature-programmed reduction (TPR), scanning electron microscopy coupled with energy dispersive spectroscopy (SEM/EDS) and specific surface area measurements. The catalytic activity of the clays modified with iron was evaluated in hydrogen peroxide decomposition reaction. The XRD patterns show the formation of crystalline iron phases on the surface of the clays, besides confirming the pillaring procedure for FePILC with a basal spacing of 1.79 nm, an increase of 0.53 nm over the montmorillonite. The specific surface area of FePILC was 210.9 m<sup>2</sup>/g. FeMAG had part of this original structure broken, with an area of 177.1 m<sup>2</sup>/g and magnetic properties demonstrated by the attraction to a permanent magnet. The TPR profiles and EDS indicated that the different heat treatments exerted great influence on the amount and phases of iron formed. The materials showed high capacity for removal of the MB dye, and the isotherms fit the Langmuir model. Adsorption of the dye significantly increased in the presence of a magnetic field, changing the Langmuir parameters and especially the maximum adsorption capacity for all materials. The best results were observed for FeMAG.

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## 1. Introduction

Pigments and dyes are disposed into wastewaters from various industries, mainly by the textile production (Fatimah et al., 2010; Hai et al., 2011). They are a serious problem when dealing with textile waste because they are water soluble, chemically and photolytically stable (Guimarães et al., 2012). Effluents containing dyes must not be disposed in natural bodies, because they do not allow solar light penetration and decrease the dissolved oxygen amount, damaging aerobic processes (Banković et al., 2012).

Clays have been used by humanity since ancient times for manufacturing, ceramic objects, and more recently in several technological applications. Clay has also become indispensable to modern life; it is the material of many kinds of applications and they are abundant in nature, inexpensive and environmentally friendly (Bergaya et al., 2006; Carretero and Lagaly, 2007). Their structural properties can be modified by simple methods such as pillaring, to produce

materials with higher surface area, porosity, thermal stability and greater capacity for adsorption and/or degradation of contaminants (Bergaya et al., 2006; Kurian et al., 2012; Tong et al., 2009).

The pillared clays, also cited by the abbreviation PILC (Pillared InterLayered Clays), have achieved considerable interest in their use as catalysts and adsorbents in recent years (Gil et al., 2011). The pillarization promotes an increase in basal spacing and surface area, which provides better accessibility of the molecules to the active sites present inside of the clay structure (Gil et al., 2011; Yang et al., 2013). The list pertaining to the engineering and application of these materials is extensive; there are several publications with different aspects of the theme in the recent literature.

Materials containing iron, like iron oxide pillared clay, become very interesting when they display magnetic attraction in combination with high specific surface area. Furthermore, iron is usually employed for adsorbents fabrication, because of the low cost (Mubarak et al., 2013). These represent an innovative and promising class of new materials for removal of contaminants such as organic dyes (Banković et al., 2012; Hou et al., 2010) and contaminants in aqueous media (Mubarak et al., 2013; Rivagli et al., 2014; Zhang et al., 2010). In addition, attention

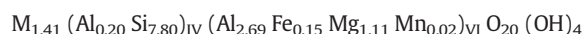
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was focused on utilizing magnetic materials (such as magnetite and magnetite silica composites) for wastewater treatment (Ferroudj et al., 2013; Tuutijärvi et al., 2009; Wu et al., 2012; Yu and Yang, 2010) mainly because of simplicity and speed of separation when these materials are used with aqueous media.

In this work a simple and rapid synthesis was performed for obtaining modified clay with magnetic iron phases exposed. Furthermore, the modified materials were tested for their ability to remove the organic dye methylene blue (MB) from an aqueous medium. The possibility of improving the removal process by implementing a magnetic field was also evaluated.

## 2. Materials and methods

The cation exchange capacity (CEC) of montmorillonite, determined by the ammonium acetate method, is 1.75 mEq/g. Mineralogical composition showed the presence of 5 wt.% of quartz and 10 wt.% of feldspar. Through chemical analysis it was determined that the principal exchangeable cation is sodium and the following is the chemical composition:



### 2.1. Preparation of the modified materials

The materials were prepared by a pillaring procedure of the montmorillonite. This was done by intercalating a dilute aqueous solution (0.4 M) of trinuclear acetato-hydroxo iron(III) nitrate  $[Fe_3(OCOCH_3)_7OH]NO_3$  (Yamanaka et al., 1984) by slow addition at 35 °C to a previously prepared aqueous suspension of the clay (1 wt.%) under constant stirring. The modified material was separated by centrifugation, washed and dried at 60 °C for 24 hours. Two distinct routes were employed for the thermal treatment: i) in an oven with synthetic air flow of 50 mL/min at 500 °C for 1 hour (FePILC) and ii) in an open furnace without gas flow at 500 °C for one hour (FeMAG).

### 2.2. Instrumental characterization

FTIR spectra were recorded on a Digilab Excalibur spectrometer with spectral range from 400 to 4000  $cm^{-1}$ . Samples were ground to powder and pressed in KBr pellets. FTIR spectra with a resolution of 4  $cm^{-1}$  were collected over an average of 32 scans. XRD analyses were performed on a Rigaku (Japan) D/Max 2500 VB2 +/PC X-ray powder diffractometer operating with  $K\alpha$  radiation of Cu ( $\lambda = 1.54056 \text{ \AA}$ ), and a generator voltage of 45 kV and a current of 30 mA in  $2\theta$  ranging from 2° to 60°. Nitrogen adsorption isotherms were obtained with an Autosorb1 MP, Quantachrome. The specific surface area was calculated using the BET model. The total pore volume was estimated from the amount of nitrogen adsorbed at  $P/P_0 = 0.99$ , the pore size distribution (PSD) was calculated based on the DFT equation. All samples were degassed overnight at 200 °C before each adsorption measurement. TPR profiles were obtained with Chembet 3000 Quantachrome equipment. In these TPR experiments, the samples are heated in the presence of  $H_2$  and the reduction reactions monitored by the hydrogen consumption. Scanning electron microscopy (JEOL Ltd.) was coupled with an energy dispersive X-ray analyzer (INCA 350, Oxford Instruments). The hydrogen peroxide decomposition study was carried out with 2 mL of  $H_2O_2$  (0.3 mol/L) and 30 mg of each material (FePILC, FeMAG and Mt), at pH 6. The mixture was stirred and the decomposition reaction of hydrogen peroxide (Eq. (1)) was monitored by  $O_2$  gaseous formed in a volumetric glass system. The decomposition with organic molecule was carried out in the same conditions, with added of 5 mL of methylene blue (50 mg/L).



### 2.3. Adsorption experiments

The adsorption isotherms were obtained in a batch equilibrium experiment using 20.0 mg of the materials dissolved in 20.0 mL of 25, 50, 100, 250, 500, and 1000 mg/L standard solutions of the organic dye methylene blue (MB); these solutions were kept stirring for 24 hours at room temperature (25 °C) and at a pH of 6.0, adjusted with dilute HCl. The solid was separated by magnetic attraction (Fig. 8) and MB concentration in the supernatant was monitored by UV–vis spectroscopy at 665 nm (Biosystems SP-2000).

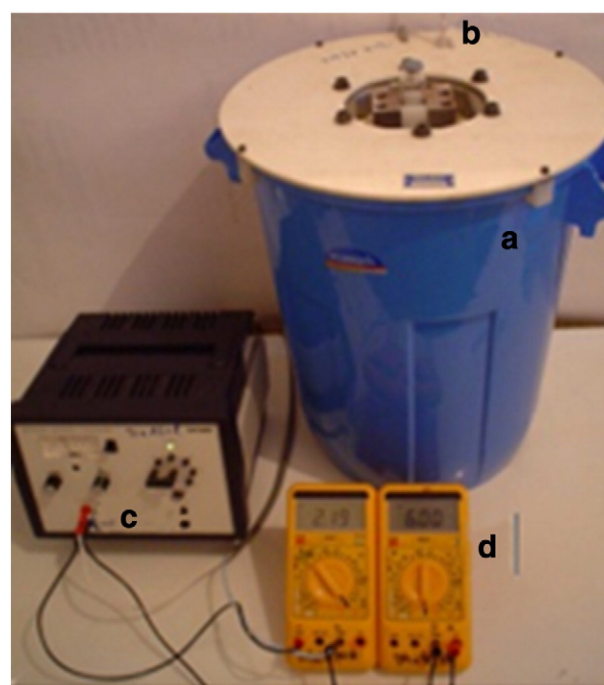
### 2.4. Influence of magnetic field on the dye adsorption

The adsorption test in the presence of the magnetic field was performed with a device developed by the Federal University of Lavras (Batista et al., 2007), designated: Device Inductor of Adsorption (Fig. 1) which generates magnetic field lines uniformly from an electromagnet. The generated field was continuously measured by a Teslameter PHYWE apparatus, with an observed value of 50.0  $\mu T$ , the voltage was kept at 6.0 V and the electric current at 2.20 A. The tests were conducted under the same conditions and concentrations cited for the adsorption tests without a magnetic field. After adsorption experiments in the presence of the magnetic field were completed, analysis of the total organic carbon content (TOC) was performed with a Shimadzu 5000 A, in order to obtain data on the amount of remaining carbon.

## 3. Results and discussion

### 3.1. Structural characterization

The FTIR spectra of the clays are presented in Fig. 2. A strong absorption in the region around 3600  $cm^{-1}$  for the montmorillonite corresponds to the stretching of the hydroxyl groups and cations in the octahedral sheet. In these modified materials, this absorption appears overlapped with another in the region of 3400  $cm^{-1}$  that is related to the presence of adsorbed water on the surface of the clays. Also in



**Fig. 1.** Adsorption inductor device. (a) polypropylene container with cooling water system, (b) opening for sample introduction, (c) power supply, and (d) multimeters for controlling current and voltage.

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