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# SDS-goethite adsorbent material preparation, structural characterization and the kinetics of the manganese adsorption



## R. Mohammed <sup>a</sup>, Heba H. El-Maghrabi <sup>a</sup>, Ahmed A. Younes <sup>b</sup>, A.B. Farag <sup>b</sup>, S. Mikhail <sup>a</sup>, M. Riad <sup>a,\*</sup>

<sup>a</sup> Catalysis Department, Petroleum Refining Division, Egyptian Petroleum Research Institute, Nasr City, PO Box 11727, Cairo, Egypt
<sup>b</sup> Faculty of Science, Helwan University, Egypt

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#### ABSTRACT

SDS-Goethite and goethite materials were prepared via precipitation technique in an alkaline medium at pH = 12. The structural characterization of the prepared samples established that, the use of either lower concentration (0.05 mol) of SDS and or higher one (0.15 mol) hindered the formation of goethite, and the ferrihydrite phase is the predominant one. On using 0.1 mol of SDS, the X-ray diffraction pattern reveals the formation of a well crystalline goethite material. The surface area and the pore volume decrease from 143.0 and 0.249 for goethite to 113.0 m<sup>2</sup>/g and 0.2010 ml/g for SDS-goethite (0.1 SDS) sample. Langmuir and Freundlich models evaluated the adsorption results. The high adsorption capacity of the prepared SDS-goethite sample towards manganese removal was attained after contact time 60 min with maximum  $Q_m = 76.3$  mg/g as compared with the goethite (19.5 mg/g). The improvement of the adsorption capacity may point to the creative surface hydroxyl groups on using the sodium dodecyl sulphate as a surfactant material.

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#### 1. Introduction

Manganese is the second most abundant metal in nature and is essential for the human system, enzymes activation and micronutrients for plants. In addition, manganese has variety of applications in ceramics, dry battery cells, electrical coils and many alloys.

The excessive use of manganese containing products leads to environmental pollution. According to US Environmental Protection Agency, the maximum manganese concentration level in domestic water supplies is 0.005 mg/L [1], thus, the removal of manganese from water is necessary.

For this reason, the development of reliable accurate, efficient and eco-friendly methods for the removal of manganese researched. Numerous methods investigated to remove toxic metals including chemical precipitation [2], coagulation [3] and adsorption [4]. Among these methods, adsorption is often superior since it is cost effective, easy handling, flexible and selective for heavy metals removal at low concentration level. Xu et al., [5] examined iron oxide nano-materials in the treatment of wastewater, also, Gonga et al., [6] used magnetic multiwall carbon nano-tube -nano-composite for the removal of cationic dyes like methylene blue.

Taffarel and Rubio [7] studied the preparation, and the adsorption properties of manganese oxide coated zeolite towards Mn<sup>2+</sup> removal;

\* Corresponding author. *E-mail address:* maryriad2006@yahoo.com (M. Riad). the  $Mn^{2+}$  uptake attained as high as 1.1 meq. Omri and Benzina [8] prepared activated carbon from Ziziphus spina-christi seeds (ZSAC) to remove Mn (II) from the aqueous solutions. The results emphasized that the maximum adsorption capacity of manganese calculated from Langmuir isotherm was around 172 mg/g.

Rachel et al., [9] investigated the removal of Manganese (II) ions from the aqueous solution by the granular activated carbon (GAC) and the modified activated carbon by iron oxide (MAC). Langmuir adsorption capacity (Qm) was found to be 14.49 mg/g and 6.94 mg/g for MAC and GAC respectively. Qomi et al. [10] clarified that the adsorption capacity for manganese ions reached 58.8 mg/g on using the polyaniline/sawdust (PAn/SD) nano-composite as an adsorbent.

Baker et al., [11] studied the adsorption behavior of the Co/Mo-LDH material for the removal of Mn under experimental conditions: Mn (II) concentration (40–145 mg/L), contact time (15–90 min), pH (2–9), adsorbent mass (0.05–0.35 g/1.0 L), adsorption temperature (25, 35 and 45 °C) and at constant stirring rate 160 rpm. The study presented the high adsorption capacity of the prepared Co/Mo-LDH material towards manganese removal.

As known, goethite is the most stable iron oxy-hydroxide, which crystallizes in the orthorhombic system, and has the ability to accommodate a large number of metal cations into its structure. Several methods used for the synthesis of  $\alpha$ -FeOOH, such as reflux method [12] and directly room temperature hydrolysis method [13].

The incorporation of foreign material into goethite can be functionalized its surface to improve the active hydroxyl groups and consequently enhanced their adsorption properties. In this work, goethite prepared through the simple and inexpensive method assisted with sodium dodecyl sulphate (SDS). The influence of SDS concentrations and the stirring time on the structural characterization and the morphology of the prepared materials investigated. The adsorption efficiency of the prepared materials carried out towards the Mn (II) heavy metal removal.

#### 2. Experimental

#### 2.1. Preparation of goethite

The goethite samples are prepared in an alkaline medium and or assisted by sodium dodecyl sulphate (SDS surfactant). All chemicals reagents are of analytical grade and used without any treatment.

#### a) In an alkaline medium

1.0 mol of Fe (NO3)3·9H2O was dissolved in 40 ml distilled water and then, 6.0 mol KOH solution was added drop wisely (to adjust the pH to 12) under stirring for seven days. The obtained precipitate separated centrifugally at 3000 rpm for 5 min, washed several times with distilled water and dried overnight at 80 °C (labeled G7).

#### b) Assisted by SDS

1.0 mol of Fe (NO<sub>3</sub>)<sub>3.</sub>9H<sub>2</sub>O was dissolved in 40 ml distilled water and mixed with 100 ml of 0.05 mol of sodium dodecyl sulphate, 6.0 mol KOH was stepwise added to the mixture (until pH reached 12) under stirring for three hours. The precipitate separated centrifugally, washed with distilled water and absolute ethanol several times, and then dried at 60 °C. Different concentration of SDS were used (0.05, 0.1, 0.15 mol), the obtained samples labeled, GSDS-0.05, GSDS-0.1 and GSDS-0.15.

#### 2.2. Characterization of the prepared goethite materials

The obtained powders structurally characterized by different techniques.

- X-ray diffraction pattern was recorded in 20 range from 4 to 80° using a Shimadzu XRD 6000 (Japan) equipped with CuKa radiation ( $\lambda = 1.5406 \text{ A}^\circ$ ), with a 0.028/min scan increment.
- Fourier-transform infrared (FTIR) spectroscopy experiments performed on spectrometer Perkin-Elmer-Spectrum-1 in the range of 4000–400 cm<sup>-1</sup> at a resolution of 4 cm<sup>-1</sup>. The samples grounded to fine powder and dispersed in KBr to compress into pellets before measurements.
- Dynamic light scattering performed to determine the particle size distribution of samples using Zeta sizer nano series HT (Nano-ZS) (Malvern Instruments).
- Thermal and thermo-gravimetric analyses performed to study the structural changes of the materials with thermal treatment. 10 mg of sample heated up to 1000 °C, with a heating rate of 10 °C min<sup>-1</sup> in an airflow rate 100 ml/min on SDTQ-600 (TA-USA) thermo balance instrument.
- High resolution transmission electron microscope (HRTEM): The samples prepared by dispersing in ethanol and sonicated for 20 min. The suspension dropped on a carbon coated copper grid for TEM observations.
- The images of the materials obtained in JEOL JEM-2100F microscope operating at voltage of 200 kV.
- The textural properties determined from the N<sub>2</sub> adsorption-desorption isotherms at liquid nitrogen temperature (-196 °C) using NOVA 3200 S Unite, automates gas sorption analyzer (Quantachrome Corporation). All samples degassed at 300 °C for 4 h in nitrogen atmosphere prior to adsorption to ensure a dry clean surface. The pore size

distributions were determined from Barrett–Joyner–Halenda (BJH) method applied to the desorption branch of the isotherm.

#### 2.3. Adsorption method

The batch adsorption experiments carried out by using 0.15–0.6 g adsorbent (goethite) added to Mn(II) solution at pH (3 - 10) in the reagent bottles and agitated in a temperature controlled shaker at a constant speed of 200 rpm for 15–120 min. at room temperature. At the end of agitation, the suspended particles separated and the filtrate analyzed by complex titration to determine the un-adsorbed Mn content. The amount of equilibrium adsorption of metals ( $q_e$ ) calculated according to Eq. (1):

$$q_{\rm e}({\rm mg/g}) = (C_{\rm i} - C_{\rm e})V \tag{1}$$

where,  $C_i$  and  $C_e$  are Mn(II) concentration measured before and after adsorption, W is the dry weight of the adsorbent and V is the volume of aqueous solution.

#### 2.3.1. Kinetic studies

The kinetics of heavy metal adsorption are evaluated by using both pseudo-first-order and pseudo-second-order models. Between these two models, the criterion for their applicability based on judgment on the respective correlation coefficient ( $R^2$ ) and agreement between experimental and calculated values of  $q_e$ .

#### a) Pseudo first order kinetic model

This model assumes that the rate of change of solute up take with time is directly proportional to the difference in equilibrium concentration and the amount of solute adsorbed with time. The rate law for a pseudo-first-order reaction expressed in Eq. (2):

$$\ln(q_e - q_t) = \ln q_e - k_1 t \tag{2}$$

The plot of  $\ln (q_e - q_t)$  versus t gives a straight line with slope of  $-k_1$  and intercept  $\ln q_e$ .

#### b) Pseudo second order kinetic model

The sorption kinetics represented by pseudo-second-order model given as in Eq. (3):

$$\frac{1}{q_{\rm t}} = \frac{1}{k_2 q_{\rm e}^2} + \frac{t}{q_{\rm e}} \tag{3}$$

The plot of  $t/q_t$  versus t gives a straight line with a slope of  $1/q_e$  and intercepts  $1/k_2q_e^2$ . Using the value of  $q_e$  calculated from the slope, the value of  $k_2$  is determined from the intercept.

#### 2.3.2. Adsorption isotherm study

The Langmuir model essentially describes the monolayer type of adsorption. It is expressed as follows (Eq. 4):

$$q_{\rm e} = \frac{q_{\rm m} b C_{\rm e}}{1 + b C_{\rm e}} \tag{4}$$

where b (L/g), is the Langmuir constant and  $C_0$  (g/L) is the initial concentration in the liquid phase. The linear form of Langmuir isotherm given as Eq. (5):

$$\frac{C_{\rm e}}{q_{\rm e}} = \frac{1}{K_{\rm L}q_{\rm m}} + \frac{C_{\rm e}}{q_{\rm m}} \tag{5}$$

where  $q_{\rm m}$  (mg/g) and  $K_{\rm L}$  (L mg<sup>-1</sup>) are the maximum adsorption capacity and empirical constant, respectively.

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