



Nano-rod Ca-decorated sludge derived carbon for removal of phosphorus[☆]



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ARTICLE INFO

Article history:

Received 2 August 2017

Received in revised form

8 October 2017

Accepted 22 October 2017

Keywords:

Phosphorus

Ca decorated sludge carbon

Crystallization

Sorption

ABSTRACT

Recovering phosphorus (P) from waste streams takes the unique advantage in simultaneously addressing the crisis of eutrophication and the shortage of P resource. A novel calcium decorated sludge carbon (Ca-SC) was developed from dyeing industry wastewater treatment sludge by decorating calcium (Ca) to effectively adsorb phosphorus from solution. The X-ray diffraction (XRD) and Fourier transform infrared (FTIR) techniques were used to characterize the Ca-SCs, followed by isotherm and kinetic sorption experiments. A preferred design with CaCO₃ to sludge mass ratio of 1:2 was found to have a sorption capacity of 116.82 mg/g for phosphorus. This work reveals the crucial role of well-dispersed nano-rod calcium on the Ca-SC surface for the sorption of phosphorus. Moreover, the decoration of nano-rod calcium was found to further promote the uptake of phosphorus through the formation of hydroxyl-apatite (Ca₅(PO₄)₃(OH)). Thus, the development of decorated Ca-SC for sorption of phosphorus is very important in solving the P pollution and resource loss.

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1. Introduction

Phosphorus (P), as the raw materials in the fertilizer production and the manufacturing of detergents, is active demand. However, it is well known that the P resource is non-renewable and with no known alternatives. Thus, the phosphorus reserves will eventually be depleted. Unfortunately, P, as a major nutrient, is naturally discharged into surface water from agriculture (fertilizer/animal feed run-off), industry (detergents), and sewage, resulting in eutrophication (Lalley et al., 2016). The P pollution of surface waters has been a widely concerned environmental issue for decades (Awual et al., 2011a).

Several techniques including biological (Nancharaiah et al., 2016), physical-chemical (Barbosa et al., 2016; Nguyen et al., 2016) methods have been developed for removing phosphorus.

Biological process such as enhanced biological phosphorus removal (EBPR) (Li et al., 2016a), biofilm (Rai et al., 2016) are effective for removing phosphorus. But these microorganisms are quite sensitive to the water qualities including carbon source (Oehmen et al., 2007) and temperature (Panswad et al., 2003). Besides, precipitation is a common costly physical-chemical process, but producing large amounts of sludge which must be properly disposed (Lee et al., 2016; Okano et al., 2016). Electrocoagulation and electrodi-lysis are effectively, but the high run-cost limited their wide application (Oguz, 2004).

Sorption is an efficient method for removing heavy metal, organic pollutants from aqueous solution based on economic and high efficiency (Awual et al., 2014). In view of the P resource shortage and pollution crisis, it is priority to recycle and recover the P by sorption to compensate for the above crisis (Ramasaahyam et al., 2014; Loganathan et al., 2014).

Consequently, a diversity of natural and synthetic materials such as calcium-rich attapulgite (Yin and Kong, 2014, 2016, 2017) and sepiolite (Yin et al., 2011, 2013), ion exchange fibrous (Awual et al., 2011b), bentonite modified with lanthanum (Kuroki et al., 2014), mesoporous cerium-zirconium binary oxide nanoparticles (Su

[☆] This paper has been recommended for acceptance by Baoshan Xing.

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et al., 2015; Lalley et al., 2016), zeolite/hydrous lanthanum oxide composite (Wang et al., 2016), CeO₂ coated nanofiber (Ko et al., 2016), nanoscale zero valent iron (Eljamal et al., 2016), core-shell bio-ceramic/Zn-layered double hydroxides (LDHs) composites (Zhang et al., 2016), Ca-activated zeolite (Hermassi et al., 2016), magnesium oxide decorated magnetic biochar (Li et al., 2016b) have been adopted for removal and recovery of phosphorus from aqueous solution. Sorbents with advantages in easily available, waste derived, low cost and environment friendly will be prevalent in the view of sustainable development.

Dyeing industry is one of the most prosperous industries in Guangzhou City. A large amount of dyeing sludge was generated during the dyeing waste water treatment process. As a hazardous waste, the dyeing sludge is very urgently to be treated effectively. Carbonizing sludge as adsorbent was the prevalent disposal technique. Henceforth, preparation of sludge derived char for pollution remediation has been widely concerned due to the view of resource and cost effective (Khalili et al., 2000; Bagreev et al., 2001; Bagreev and Bandosz, 2004; Bandosz and Block, 2006; Seredych and Bandosz, 2007; Gómez-Pacheco et al., 2012; Athalathil et al., 2014). Besides, the sludge as a carbon precursor being acted as supporter decorated by metal to enhance the catalytic activity is another novel method for sludge treatment (Tu et al., 2012; 2014). Moreover, more attentions were further paid to the important effect of the initial minerals in sludge on the complexation adsorption (Kong et al., 2014, 2016), catalytic oxidation (Kong et al., 2016; Tu et al., 2012, 2014). Thus, search for the effect of initial minerals in the sludge on the sorption ability is the potential objective for the researchers.

In the past, concrete sludge was treated by acid soaking to release the Ca²⁺. It was regarded as an environment-friendly adsorbent for extracting the P from aqueous solution. The main action is due to the released Ca²⁺ (Okano et al., 2016). Fortunately, the Ca is widely approached and environmental friendly, herein, an extraordinary approach was designed to decorate the cost-effective sludge derived carbon (Ca-SC) by impregnating CaCO₃ powder, further being conducted to enhance the ability for removal of phosphorus in aqueous solution. Structural property of Ca-SC was obtained by SEM analysis. Sorption kinetics and isotherms were conducted to describe the sorption behavior of Ca-SC to P. The effect of the varied mass ratio of CaCO₃ to sludge on the sorption characteristics of phosphorus on the Ca-SC was investigated. The crystal change on the Ca-SC before and after sorption was investigated by XRD analysis to explore the sorption mechanism.

2. Materials and method

2.1. Materials

The precursor was dewatered sewage sludge collected from a Xintang dyeing wastewater treatment plant, Guangzhou, China, and subsequently being dried in air at 105 °C. As shown in Table S1, the incombustible mass content of the dried sludge is 72.76%. The main mineral elements were Ca, Fe and Mn. The calcium carbonate (CaCO₃), potassium phosphate (K₃PO₄), Dipotassium phosphate (K₂HPO₄), monopotassium phosphate (KH₂PO₄) were chemical grade purchased from Aladdin Industrial Corporation, Shanghai, China.

2.2. Preparation of Ca-SC

Firstly, 13 g and 26 g micro-powder CaCO₃ were weighted, further being dosed into two beakers each containing 100 g dewatered sludge slurry (water content 74%), assuring that the dried mass ratio of CaCO₃ to sludge is 1:2 and 1: 1. And determined

deionized water was added to adjust the slurry mixtures. The mixtures were stirred by Magnetic Stirrers adequately one night to ascertain the slurry being mixed uniformly, being dried at a water bath at 80 °C by stirring to form gels. After that, the gels were dried at 105 °C in an oven to a constant weight. The dried sludge mixtures in varied mass ratios were directly put into ceramic ark, and being heated in a programmable tube electric furnace (SKF-210, Hangzhou Lantian Instrument Co., China) at a rate of 20 °C/min to 800 °C in the presence of N₂ holding for 2 h. After cooling to room temperature, the product was ground to 80-100 meshes, further being washed with deionized water to remove the hetero ions until the conductivity of washing solution was constant, further being dried at 105 °C. The obtained product was dried at 105 °C in an oven overnight. The obtained products were named as Ca-SC-0, Ca-SC-1 and Ca-SC-2 according to the dried mass ratio of CaCO₃ to sludge in 0:1, 1:2 and 1:1, respectively.

In order to investigate the effect of the decorated Ca on the sorption of phosphorus, the carbonized products were washed by excessive HCl solution to remove the decorated Ca, being named as Ca-SC-0-A, Ca-SC-1-A and Ca-SC-2-A according to the mass ratio of CaCO₃ to dried sludge.

2.3. Characterization and analytical method

The crystals of the Ca-SCs were characterized by X-ray Diffraction (XRD) using D/max 2200 vpc Diffractometer (Rigaku Corporation, Japan) with a Cu K α radiation at 40 kV and 30 mA. The morphologies of the obtained char were observed using a field emission scanning electron microscope (SEM) (JEOLJSM-6330F) with energy dispersive X-ray spectrometer (EDS) (Inca300, Oxford). The relative P amount in the char was analyzed with SEM-EDS-X-ray elemental analysis. Fourier Transform Infrared Spectroscopy (FTIR) was recorded by a Thermo Scientific Nicolet IM IS10 (America). The S_{BET} of the tested samples were analyzed using an auto-adsorption system (Auto-sorb-6, Quantachrome) at 77 K and calculated using the Brunauer–Emmet–Teller equation (Kong et al., 2013).

2.4. Sorption experiments

The phosphorus stock solution in 1000 mg/L was prepared by dissolving 6.8399 g K₃PO₄ in 1 L deionized water and was serially diluted to obtain desired concentrations. The KH₂PO₄ and K₂HPO₄ stock solutions were also prepared by the above method to ascertain the concentration of phosphorus was 1000 mg/L. The sorption experiments were conducted in a series of conical flasks containing desired phosphorous concentrations. The adsorbent dosage is 3 g/L. Sorption kinetics were investigated in an initial phosphorus concentration of 200 mg/L. Samples were withdrawn at each pre-determined interval and filtered through a 0.45 μ m cellulose acetate filter membrane, measured using an ultraviolet spectrophotometer at the wavelength of 700 nm (HITACHI U-2910, Japan). Isotherm sorption characteristics were investigated by varying the initial phosphorus concentrations (200, 300, 400, 500, 600, 800 and 1000 mg/L). The residual concentration of P was measured as described above. The sorption experiments of KH₂PO₄ and K₂HPO₄ were also conducted as described previously to compare the sorption characteristics. Coexisting anions NO₃⁻, Cl⁻, SO₄²⁻ and HCO₃⁻ in 10 mM are conducted in the K₃PO₄ solution to investigate the effect of competing anions on the sorption behaviors.

The sorption amount P (mg) per unit mass of adsorbent q_t (mg/g) was calculated using Eq. (1):

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