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Synergistic effect of water content and composite conditioner of Fenton's reagent combined with red mud on the enhanced hydrogen production from sludge pyrolysis

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A B S T R A C T

This study investigated the synergistic effect of water content and a composite conditioner of Fenton's reagent combined with red mud (Fenton-RM) on the pyrolytic products (fuel gas, tar, and solid char) of deep-dewatered sludge. The catalytic effect of metal oxides in Fenton-RM could be promoted by the presence of water during sludge pyrolysis, showing higher gas yield with increased water content. Maximum gas outputs of the deep-dewatered sludge conditioned with Fenton-RM (S-Fenton-RM) and the conventional dewatered sludge conditioned with polyacrylamide (S-PAM), both appeared at 900 °C with a water content of 65 wt%, and were 0.257 and 0.189 L/g dry solid (DS), respectively. At the same temperature and with the same water content, the hydrogen (H_2) yields of the S-Fenton-RM samples were always higher than those of the S-PAM samples. At 900 °C, the maximum H_2 yield of the S-Fenton-RM samples was 0.102 L/g DS, which was 85.5% higher than that of the S-PAM samples. The results indicated that water in the wet sludge provided the steam atmosphere for pyrolysis, and the water vapor then involved in secondary cracking reformation of tar and char gasification reactions, which would be catalyzed by the presence of metal oxides in the Fenton-RM conditioner, thus increasing the yield of fuel gas, especially hydrogen. The H_2 production cost from the S-Fenton-RM system is less than that from the S-PAM system. The results suggest that pyrolysis of the wet deep-dewatered sludge conditioned with Fenton-RM is an economical and promising alternative for sewage sludge dewatering and disposal/reuse.
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1. Introduction

Sewage sludge is a by-product from municipal wastewater treatment. It has high contents of organic matters and water, and is difficult to be effectively dewatered. Increasing production of sewage sludge has been a burden and a source of secondary pollution to the environment (Feng et al., 2015). Reducing sludge volume, stabilizing organic matters and heavy metals and meanwhile achieving sludge resource utilization are desirable to eliminate the adverse impact on human health and the environment, as well as facilitating waste recycling (Liang et al., 2015; Xie et al., 2016; Yuan and Dai, 2014, 2016). Pyrolysis has been demonstrated as one of the most promising sewage sludge management alternatives due to its effective transformation of organic matters to fuel gases (e.g. hydrogen and methane) and other useful products (Manara and Zabaniotou, 2012; Fonts et al., 2012; Smoliński and Howaniec, 2016).

The operating parameters, kinetics, products, and catalysis of pyrolyzing sludge cakes have been extensively studied (Shao et al., 2008; Inguanzo et al., 2002; Liu et al., 2015; He et al., 2015). Nevertheless, those experiments were conducted using dry sludge feedstock, wherein energy-intensive pretreatment and dewatering

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Abbreviation list

DS	Dry Solid
Fenton-RM	Composite conditioner of Fenton's reagent combined with red mud
FTIR	Fourier Transform Infrared Spectroscopy
GC	Gas Chromatography
GC-MS	Gas Chromatography-Mass Spectrometer
MS	Mass Spectrometer
NIST	National Institute of Standards and Technology
PAHs	Polyaromatic Hydrocarbons
PAM	Polyacrylamide
RM	Red mud
S-Fenton-RM	Deep-dewatered sludge conditioned with Fenton's reagent and red mud
S-PAM	Dewatered sludge conditioned with PAM
RS	Raw Sludge
TCD	Thermal Conductivity Detector
TIC	Total Ion Chromatogram

process were needed. For pyrolysis of sludge, the water content is critical to the distribution of the final products (i.e., fuel gas, tar as well as solid char), total energy consumption, and capital investment. It has been reported that water in the sludge could participate in the pyrolytic reactions, and subsequently elevate the yield of fuel gas, especially H_2 (Domínguez et al., 2006a). More specifically, Zhang et al. (2011) have revealed the formation of polyaromatic hydrocarbons (PAHs) in tar and production of hydrogen-rich fuel gas via the Diels-Alder reaction model during pyrolysis of sewage sludge with a moisture content of 84.2 wt%. Gai et al. (2016) found that water played an important role in the fuel gas production in steaming gasification of hydrochar derived from hydrothermal carbonization of sewage sludge. Liu et al. (2014) also elucidated the effects of sludge moisture content on the production of tar, char and syngas using both dry sludge and wet sludge. Xiong et al. (2013) suggested that water in wet sewage sludge could generate a steam-rich atmosphere on the surface of sludge, and the steam would then participated in the degradation of organic matters and secondary cracking reformation of tar, which led to the production of hydrogen-rich fuel gas.

Our previous study demonstrated that using a composite conditioner of Fenton's reagent combined with red mud, referred as Fenton-RM hereafter, in sewage sludge dewatering could enhance hydrogen production in pyrolysis of the dry dewatered sludge through facilitated decomposition of organic matters and accelerated secondary cracking reformation of tar (Song et al., 2016). However, the synergistic effect of water content and the Fenton-RM conditioner remains unclear. The objective of this study was to investigate the effect of water content and composite conditioner of Fenton-RM on the pyrolytic products of deep-dewatered sludge at different temperatures, and the results were compared with that of dewatered sludge conditioned with conventional polyacrylamide (PAM). The schematic of this study is shown in Fig. 1. The gas composition was analyzed with Gas Chromatography (GC); the tar was determined with Gas Chromatography-Mass Spectroscopy (GC-MS); and the solid char was measured with Fourier Transform Infrared Spectroscopy (FTIR). The results of this study are expected to provide a reference for the practical application of the Fenton-RM conditioner for deep dewatering and further pyrolysis of sewage sludge.

2. Materials and methods

2.1. Materials and sample preparation

A mixture of primary sludge and secondary sludge from Tangxunhu wastewater treatment plant in Wuhan, China was used as the raw sludge (RS) in this study. The water content of the RS after gravitational thickening was around 97 wt%. The deep-dewatered sludge sample produced from sludge conditioned with Fenton's reagent and RM is denoted as S-Fenton-RM hereinafter. The RM, acting as a skeleton builder in the dewatering process and a catalyst in the pyrolysis process, was collected from an alumina plant using the Bayer process in China. It is mainly composed of Fe_2O_3 (40.85 wt %), Al_2O_3 (13.20 wt%), SiO_2 (11.07 wt%), TiO_2 (7.12 wt%), and other oxides like Na_2O, MgO and CaO (Song et al., 2016). The dewatering process followed the procedure in our previous study (Zhang et al., 2014). The RS was first conditioned with Fenton's reagent (32 mg Fe^{2+}/g dry solid (DS) combined with 34 mg H_2O_2/g DS) and RM (275 mg/g DS), and mechanically dewatered using a diagram press filter under a pressure of 0.8 MPa. For comparison, the conventional dewatered sludge conditioned with PAM (0.005 t/t DS) (obtained from the same wastewater treatment plant) was labelled as S-PAM hereinafter.

Results of the proximate and ultimate analyses on the RS, the S-Fenton-RM and the S-PAM samples are summarized in Table 1. These analyses were conducted following the Chinese National Standards of Proximate Analysis of Coal (GB/T 212-2008). The ash content was measured by placing the sample in a muffle furnace as the temperature rised from room temperature (25 ± 2 °C) to 500 °C for 30 min, and then rised to 815 °C, and kept for 1 h. The ultimate analyses were measured using an elemental analyzer (Vario Micro cube, Elementar, Germany). As shown in Table 1, the volatiles and fixed carbon of the S-Fenton-RM sample are smaller than those of the RS and the S-PAM samples due to the addition of inorganic RM.

To investigate the effect of water content on the pyrolysis, the S-Fenton-RM samples with an initial water content of 65 wt% were dried at 55 °C for different time intervals to get sludge samples with different water contents (14, 40, 52 and 65 wt%). For comparison, the S-PAM samples with an initial water content of 85 wt% were dried at 55 °C to get sludge samples with different water contents (14, 40, 52, 65 and 85 wt%).

2.2. Experimental apparatus and procedure

Pyrolysis experiments were carried out in a horizontal quartz reactor (1200 mm length × 70 mm I.D.), heated by an electrical tube furnace. The schematic diagram of the tubular furnace pyrolysis system can be found in our previous study (Song et al., 2016). The whole pyrolysis system consists of a quartz tube, a cooling system for condensation of water and organic vapors, a gas cleaning/drying system, a wet gas meter, and a gas sampling bag. In each experiment, a certain amount of wet sludge sample with 4.5 g of dry solids was used. Triplicates were conducted for each experiment.

Detailed description on the pyrolysis process can also be found in our previous study (Song et al., 2016). Briefly, temperature of the furnace was first elevated to a pre-set temperature (700, 800 or 900 °C). Nitrogen of 99.99% purity was passed through the reactor at a flow rate of 100 mL/min to keep an inert atmosphere throughout the whole pyrolysis process. Sludge sample was then injected into the reaction zone for 30 min. The gas collected in the sample bag was analyzed by Gas Chromatography (GC) (Agilent 7820A, Agilent, USA) equipped with a Thermal Conductivity Detector (TCD). The National Institute of Standards and Technology (NIST) mass spectral data library was used to identify chromatographic peaks, and the relative percentage content was calculated

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