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Preparation, characterization and sludge conditioning performance of modified coal fly ash

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ABSTRACT

Filtration aids have been widely used in improving sludge dewatering performance, while the previous filtration aids have relatively lower efficiency in adsorption and bridging ability. In this study, a modified coal fly ash (MCFA) was prepared by soaking raw coal fly ash (RCFA) with sulfuric acid. Scanning electron microscopy (SEM), X-ray powder diffraction (XRD), Specific Surface Area and Pore Size Analyzer (BET) and X-ray photoelectron spectroscopy (XPS) were utilized to characterize its structure and morphology. The sludge conditioning performance and dewatering performance on secondary sludge was carried out based on the specific resistance to filtration, capillary suction time and LB-EPS protein content analysis. It showed that the optimum conditions for preparing MCFA from RCFA was a soaking time of 2 h, the acid concentration of 5.0 mol/L and the ratio of acid to RCFA 5:1, under which MCFA exhibited a higher efficiency in sludge conditioning. XRD analysis revealed a high proportion of silicon in MCFA at *ca.* 26.80%, whereas XPS implied that more silicon was grafted onto the Si–O structure. SEM and BET measurements indicated that the surface of MCFA had underwent a strong corrosion, exhibiting a larger surface area, high pore volume and small average particle size, which had promoted its adsorption capacity and permeability in aiding sludge dewatering.

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1. Introduction

Sewage sludge is difficult in dewatering, which imposes restrictions on sludge treatment and transportation [1]. This renders sewage sludge pretreatment important and flocculation has been adopted as one of the pretreatment methods for sludge dewatering due to its low operation cost [2,3]. During flocculation, hydration shells around the particles are destroyed and the dispersed colloidal particles agglomerate together to form larger particles and settle down; therefore, solid–liquid separation is pronouncedly promoted [4,5]. High pressure mechanical dewatering was subsequently employed to elevate the solid content of sludge, but the process tends to result in sludge deformation and thus channel closing, which hinders further dehydration [6].

Physical conditioners, known as skeleton builders or filtration aids, *e.g.*, wood chips [7], lignite [4], red mud [8], sawdust [9] and fly ash [10,11], have been paid considerable attention on. These conditioners contribute to the formation of flocs with rigid and permeable internal structure, thereby reducing sludge compressibility [12]. In addition, sludge cake permeability, which reflects the dehydration rate of flocculated suspensions during the cake compression stage, could also be enhanced by the use of physical conditioners [4,12,13].

Composite conditioners, *e.g.*, red mud combined with Fenton's reagent [8], lime combined with Fenton's reagent [14], red gypsum combined with a polymer [15] and Fe^{2+}-activated sodium persulfate combined with thermally pretreated phosphogypsum [16], exhibit the combined merits of each composite conditioner in overcoming the dewatering difficulties. They not only act as chemical conditioners by destroying the extracellular polymeric substance to reduce the viscosity of the sludge system and improve the settling efficiency, but also play an important role in generating a more permeable and rigid lattice structure of the sludge cake as physical conditioners, which help to resist sludge particle deformation and cake void closures. These characteristics of the composite conditioners make them excellent candidates for high pressure mechanical filtering [4,17].

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Coal fly ash is a waste product derived from coal-burning power plants, which may have an adverse impact on environmental quality and human health. In recent years, fly ash has gained widespread application in sludge conditioning [4,10–12,18] and aiding sludge dewatering by acting as a skeleton builder. Recently, acid soaking is reported to promote the exposure of Si, Al and Fe in raw coal fly ash (RCFA), and the modified coal fly ash (MCFA) exhibits larger specific surface area, higher pore volume and adsorption capacity, thus improving sludge dewatering. Previous studies, however, mainly focused on the conditions for sludge dewatering [10], and the internal structure and external characteristics of the physical conditioner have been ignored. Evidently, more systematic studies are required to better understand the internal material and microstructural changes of fly ash and to clarify the mechanism of sludge dewatering based on sludge compressibility and permeability. Furthermore, few studies have evaluated the conditioning performance of fly ash using a combination of capillary suction time (CST) and protein content analysis [10]. A combined aluminum–ferrous–starch flocculant (CAFS) was prepared with starch, aluminum sulfate and ferrous sulfate, with its mesh starch chains grafted polyaluminum and polyferrous [19]. The function of CAFS on flocculation has been previously reported by Lin et al. [19] and CAFS exhibited better sludge dewatering efficiency than $FeCl_3$ [20].

In this study, a physical conditioner (MCFA) was prepared with coal fly ash and sulfuric acid. Meanwhile, scanning electron microscopy (SEM), X-ray powder diffraction (XRD), a specific surface area and pore size analyzer (BET) and X-ray photoelectron spectroscopy (XPS) were utilized to characterize its structure and morphology. Furthermore, key parameters, including the specific resistance to filtration (SRF), capillary suction time (CST), LB-EPS protein content, compressibility and permeability, were employed to evaluate the sludge conditioning ability and dewatering performance. The objectives of this study were: (1) to demonstrate the feasibility of prepared material using MCFA as a skeleton builder in combination with CAFS; (2) to clarify the mechanism of the composite conditioner related to the adsorption ability of MCFA and sludge cake compressibility; and (3) to investigate the effects of micropores on sludge cake permeability in the physical conditioner.

2. Materials and methods

2.1. Material

MCFA: The raw coal fly ash (RCFA) obtained from Huangpu Power Plant (Guangzhou, China) was soaked with sulfuric acid (5.0 mol/L) at a ratio of 3:1 (mL/g) for 2 h. The mixture was then filtered and dried at 105 °C for 24 h. Subsequently, the dried MCFA was milled and sieved under 100 mesh to produce MCFA.

Combined aluminum–ferrous–starch flocculant (CAFS): CAFS was prepared according to our previous papers [19,20].

Raw sludge: The raw sludge was collected from the secondary sedimentation tank of the Lijiao municipal waste water treatment plant in Guangzhou City, China. The samples were transported to the laboratory using polypropylene containers and stored in a refrigerator at 4 °C. Subsequently, secondary samples were taken to measure the moisture content, SRF, CST, LB-protein content and pH value, which were determined to be 95.0%, 98.3×10^{11} m/kg, 120 s, 8.85 mg/L and 6.75, respectively.

2.2. Flocculation experiments

An optimized dosage of MCFA was added under rapid stirring at 250 rpm for 1 min, followed by slow agitation at 50 rpm for 5 min. The experiment with sludge co-conditioned by MCFA and CAFS was conducted by adding MCFA under rapid stirring at 250 rpm for 1 min, followed by slow agitation at 50 rpm for 5 min. Subsequently, CAFS was added under the same operating condition. All experiments were performed in triplicate, and the results were averaged for further analysis.

The CST of the sludge system was measured using a TYPE304M Capillary Suction Timer (Triton Electronics, UK) by adding approximately 2 mL of sample into tubes [21]. SRF was determined by the Buchner funnel method [21]. Sludge conditioned by different types of flocculants was centrifuged (4000 rpm, 10 min) using a TD5A centrifuge (China) to separate the solids and supernatants. The organic matter in the supernatant was assumed to be equivalent to loosely bound EPS (LB-EPS) in the sludge mass [2,3,22] and the protein content in the LB-protein content was measured with a UV spectrophotometer (722E Model) following the modified Lowry method [23], adopting the standards for bovine serum albumin content analysis.

2.3. Evaluation of using MCFA as the skeleton builder

The function of filtration aids on sludge dewatering was closely linked to its rigid and porous structure [12,24]. The use of a skeleton builder has showed an effective method for achieving deep dewatering [13]. Therefore, permeability and compressibility were used to determine the role of MCFA in sludge dewatering combined with CAFS.

Permeability tests were conducted on the compressed cake by passing water through the sample [4]. The equilibrium permeability of a compressed cake (K) can be expressed by the following equation:

$$K = \frac{Q\mu H}{A\Delta P_c} \tag{1}$$

where Q is the volumetric flow rate of the fluid through the compressed cake (m^3/s), μ is the fluid viscosity of water, $\triangle P_c$ is the fluid pressure drop across the cake, H is the height of the sludge cake and A is the frontal sectional area of the sludge cake.

The measurement of sludge compressibility was conducted under five pressure gradients over the range from 0.15 to 0.65 MPa by method described by Zhao and Bache [16].

2.4. Characterization of RCFA and MCFA

The surface morphology was investigated by scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX, Hitachi SU8010, Japan). The XRD patterns of the samples were obtained by X-ray diffraction (D/MAX-Ultima IV, Japan). Therefore, the degree of crystallinity (X_{cr}) can be expressed by the following equation:

$$X_{cr}(\%) = \frac{I_c \times 100}{I_c + I_a} \tag{2}$$

where I_c and I_a are the area under the crystalline peaks and the halo, respectively [25].

The surface elemental composition of the samples was determined by X-ray photoelectron spectroscopy (Axios Pw4400, Holland). The specific surface area, pore volume and aperture of samples were tested by a specific surface area and pore size analyzer (JW-BK112, USA). Briefly, RCFA and MCFA were degased and air dried at 200 °C prior to the subsequent N_2 adsorption measurement conducted at a temperature of -196 °C. The specific surface area was obtained using multipoint BET method, while pore volume and aperture size were evaluated using the Barrett–Joyner–Halenda (BJH) method by computation of nitrogen isotherms.

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