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# Disintegration of waste activated sludge by a combined treatment of alkaline-modified eggshell and ultrasonic radiation



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#### ABSTRACT

In order to improve disintegration performance of waste activated sludge, alkaline-modified eggshell and ultrasonic radiation were introduced. Eggshell was subjected to modification treatment to produce calcium oxide (CaO). The calcium oxide was characterized by X-ray diffraction (XRD) and thermogravimetric analysis (TGA). XRD patterns showed CaO intense peaks. TGA profile indicated the components of eggshell were constant at above 850 °C. The alone and combined treatment effects of alkaline-modified eggshell and ultrasonic radiation on the disintegration of waste activated sludge were studied. The combination of alkaline-modified eggshell and ultrasonic radiation treatment processes could enhanced the disintegration of waste activated sludge. Optimum conditions were pH of 12, ultrasonic power of 330 W, and disintegration time of 15 min for the combined treatment process. The disintegration efficiency of waste activated sludge reached 45% under the optimized condition. The combined process of alkaline-modified eggshell and ultrasonic radiation was effective and feasible to disintegrate waste activated sludge.

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#### 1. Introduction

Huge amounts of waste activated sludge (WAS) is produced during activated sludge process of wastewater [1]. The components in WAS are very complex, including microorganism zooglea, extracellular polymeric substance (EPS), absorbed inorganic and organic matter and vast amounts water. Myriad hazardous compositions (such as pathogenic microorganisms, parasites, volatile poisonous matter and heavy metals) will produce grievous hazards to public health once the control system loses effectiveness [2,3]. Consequently, the WAS must be effectively disposed to guarantee the public health and prevent environmental pollution. Sludge disintegration technologies, which include thermal, chemical, biological and mechanical processes, are attracting widespread interest today [4]. In order to improve the disintegration performance of the WAS, microwave irradiation [5], acidification [6], ultrasonic pretreatment [7], thermochemical pretreatment [8], Fenton oxidation [9] have been investigated. However, the traditional technologies have some disadvantages such as pollution, high energy consumption, and complicated operation [10].

http://dx.doi.org/10.1016/j.jece.2017.02.034 2213-3437/© 2017 Elsevier Ltd. All rights reserved. Recently, combination processes have aroused wide public concern, such as physico-chemical pretreatment method [11], microwave-hydrogen peroxide [12], microwave-alkaline [13] and so on.

Alkaline method is a commonly examined method whose mechanism is to induce swelling of particulate organics at high pH, making the cellular substances more susceptible to reaction [14]. On the other hand, ultrasonic radiation is an effective technology for the disruption of microbial cells and improvement of dehydration property of the WAS. Therefore, when ultrasonic radiation pretreatment was followed by alkaline method, the efficiency was maximized [15]. However, introducing alkaline reagent causes the total operating costs to increase. Eggshell produced from dining garbage, a problematic basic waste, is one such substance with high level of CaCO<sub>3</sub> (about 87%–90% mass ratio), and the eggshell are dumped into landfill currently [16]. The eggshell use as an auxiliary for disintegration of the WAS would not only improve the ultrasonic disintegration rate but also reduce the production cost and control environment pollution. Improving the disintegration efficiency using an ultrasonic assisting system and the alkaline-modified eggshell as an auxiliary has not been reported.

In order to improve the WAS disintegration performance, this paper investigated the alone and combined influences of ultrasonic

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radiation and alkaline-modified eggshell treatment processes. soluble protein (SP), soluble carbohydrate (SC) and soluble chemical oxygen demand (SCOD), were taken as target parameters. Ultrasonic power, pH and disintegration time were systematically researched in order to provide some insight for the WAS disintegration.

#### 2. Materials and methods

#### 2.1. Materials

The WAS samples were collected from the secondary sedimentation tank of the Yuyang wastewater treatment plant in Yulin, China. The pH of the WAS was in the range of 7.6–9.5. Moisture content of the initial WAS sample was about  $98 \pm 1.5\%$ . Before the experiments, moisture content of the WAS sample was adjusted to 99.0% (wt/wt) by moving supernatant or diluting with ultrapure water. Total chemical oxygen demand (TCOD) of the samples at 99.0% was about 7998 mg/L, while soluble chemical oxygen demand (SCOD) was about 68 mg/L. Eggshells were collected from a restaurant in Yulin, China. Eggshells were washed and dried, then grinded (Porcelain crucible,  $\Phi$  30 mm, Tangshan Porcelain Factory, China) and sieved (Stainless steel sieve, 100 mesh, Comio Chemical Reagent Co., Ltd. Tianjin, China) to remove impurities. The eggshells powder was heated in the muffle furnace at 800 °C for 3h before use. Hydrogen chloride (HCl, analytical reagent) and sodium hydroxide (NaOH, analytical reagent).

#### 2.2. Experimental procedure

The pH values of the WAS samples were adjusted to 10, 11, 12 and 13 using the alkaline-modified eggshell, and corresponding amount of the alkaline-modified eggshell was about 0.15 g/mL, 0.25 g/mL, 0.35 g/mL, 0.45 g/mL, respectively. The ultrasonic radiation treatment was carried out with an ultrasonic generator (88-1, New league electrical and mechanical equipment factory, Beijing, China) and an ultrasonic horn. The working frequency of ultrasound was fixed at 20.024 kHz. According to previous works [17], the experiments were carried out with a 100 mL of the WAS at 300, 330 and 360 W ultrasonic powers. The ultrasonic horn was introduced to the middle of the WAS, approximately 2 cm up from the bottom of the WAS. For the combination of ultrasonic radiation and alkaline-modified eggshell treatment process, firstly, the WAS pH was adjusted with mentioned methods above, then the ultrasonic radiation treatment of the WAS was performed with

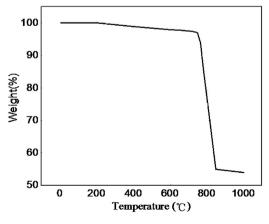


Fig. 1. TGA profile of the eggshell.

the same mentioned methods above. The all experimental times were varied in the range of 5–35 min at 5 min increments.

#### 2.3. Analyses

The X-ray diffraction (XRD) characterization of the eggshell was determined on a X-ray diffractometer (D/MAX-2400) using Cu k $\alpha$  radiation source carried out at 25 mA and 30 kV over a 2 $\theta$  range from 10° to 70° with a step size of 0.02° (2 $\theta$ ) and a scan step time 0.5 s. In order to ascertain calcination temperature of the eggshell, thermogravimetric analysis (TGA) is performed with SDT2960 instrument. The produced CO<sub>2</sub> during calcination was removed using nitrogen gas (flow rate: 60 mL/min). 30 mg of the eggshell was put into the sample holder, then heated from 25°C to 1000°C (heating rate:10°C/min).

TCOD and SCOD were measured using the Standard Methods [18]. pH was measured by a pH meter (PHS-3C, China). The soluble carbohydrate (SC) and protein (SP) measurements were carried out by the phenolsulphuric acid method and Lowry method [19], respectively. In order to compare the WAS disintegration efficiency of the different processes, the disintegration efficiency (DE) was calculated by Eq. (1) [20]:

$$DE = \frac{SCOD_{after} - SCOD_0}{TCOD_0 - SCOD_0} \tag{1}$$

where  $SCOD_0$  is the SCOD of the initial WAS, (mg/L),  $SCOD_{after}$  is the SCOD of the WAS after treatment, (mg/L) and  $TCOD_0$  is the TCOD of the initial WAS, (mg/L).

Colony-Forming units (CFU) were analyzed with beef extractpeptone agar plates for 48 h incubation at 36 °C.

#### 3. Results and discussion

#### 3.1. Eggshell characterization

The TGA profile of eggshells is showed in Fig. 1. Weight loss was small when calcination temperature was less than 500 °C. This phenomenon was mainly caused by the elimination of water and some volatile organic matters in the eggshell. Eggshell presented a larger weight loss of 42.93% when the calcination temperature

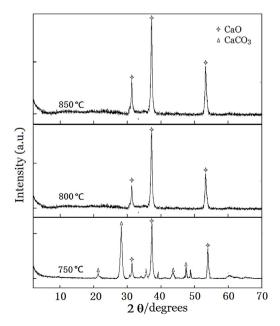


Fig. 2. XRD patterns of eggshell at different calcination temperatures.

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