



# Effects of sintering temperature on the characteristics of lightweight aggregate made from sewage sludge and river sediment



Mingwei Liu <sup>a,\*</sup>, Chunze Wang <sup>a</sup>, Yang Bai <sup>a</sup>, Guoren Xu <sup>b</sup>

<sup>a</sup> School of Civil Engineering and Architecture, Northeast Electric Power University, Jilin 132000, China

<sup>b</sup> State Key Laboratory of Urban Water Resource and Environment, Harbin Institute of Technology, Harbin 150090, China

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## ABSTRACT

The primary goal of this research is to evaluate the effects of sintering temperature on the characteristics of lightweight aggregate (LWA) made from sewage sludge (SS) and river sediment (RS). The results indicate that LWA made from SS and RS mixed at ratios of 1:1 and sintered at approximately 1100 °C has good physical characteristics with bulk and grain density of approximately 836 kg m<sup>-3</sup> and 1672 kg m<sup>-3</sup> respectively. The attained compressive strength is 13.7 MPa, water absorption is less than 12% and the fine pores (5 μm < pore size < 15 μm) are uniformly distributed on the surface of LWA. The major mineral phases are quartz, albite and hematite at sintering temperatures above 900 °C. High sintering temperature also promoted the binding capacity of Cd, Cr, Cu and Pb in LWA, and the leaching contents of heavy metals did not change at temperatures above 1050 °C even when the LWA bodies were broken. Therefore, solidification effectiveness of heavy metals can be guaranteed in the LWA. This study demonstrates that it is feasible to use SS and RS as raw materials for making LWA, the sintering temperature is one of key factors to determine the characteristics of LWA, and 1100 °C is regarded as the optimal sintering temperature.

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## 1. Introduction

Sewage sludge (SS), the invisible by-product of wastewater treatment, contains abundant organics, pathogenic bacteria and heavy metals, presenting an environmental problem that requires an urgent solution [1,2]. At present, traditional SS treatment methods such as landfilling, incineration and agricultural utilisation face strong public opposition and strict regulatory pressure. Landfilling is becoming increasingly restricted because of groundwater pollution, odour emission and soil contamination [3]. Sludge recycled to agricultural land as fertilizer has been restricted in many countries because of heavy metal pollution [4]. Incineration has been also limited because of its cost and air pollution [5,6]. Therefore, it is particularly urgent to look for a new, environmentally friendly and sustainable approach for sludge disposal.

Pollutants from industrial wastewater and domestic sewage are discharged into rivers, resulting in the deterioration of river water quality. Pollutants such as heavy metals gradually accumulate in the river sediment (RS) and they might be released when the

sediments are disturbed [7]. The bottom sediments might also affect the ecological system for a long time through the food chain. If the RS are not frequently dredged up, sediment accumulation would reduce river depth, and subsequently, would cause trouble in shipping, thereby resulting in muddy waters and the destruction of the marine environment [8,9].

At present, the re-utilisation of solid waste for production of construction materials is one of the energy-efficient and cost-effective measures. For example, utilisation of sludge solids for making bricks [10,11], cement mortars [12], concrete [13]; use of waste materials such as spent glauconite [14], spent zeolite [15], fly ash [16] to manufacture of lightweight aggregate (LWA). SS as raw material to fabricate lightweight aggregate (LWA) is one innovative method for sludge utilisation, which can be widely used in building materials or water-treatment filter material. So far, research has been conducted on LWA made from sludge or sludge ash. Some previous studies have revealed that sludge sintered at 1000–1200 °C and as a raw material could be used to produce porous LWA with high compressive strength and proper density [17–19]. As RS contain abundant mineral phases such as SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, they can be regarded as one of the natural sources for building materials [20]. It was found that river sediment blended

\* Corresponding author.

E-mail address: [mw\\_liu@126.com](mailto:mw_liu@126.com) (M. Liu).

with clay fired at 1050 °C can produce high-insulation brick [21]. Lake sediment also can be used together with SS and cinder to produce porous brick which meet the requirements of government standard [22]. Our previous studies also indicated that RS, as another waste sludge, can be substituted for clay in the production of LWA because it is similarly rich in silicon and aluminium [23,24].

The vitrification temperature of material, plays a very important role on the mechanical strength of LWA [25]. Usually, a viscous glass phase must be produced simultaneously to encapsulate gases released at the vitrification temperature, which contribute to the formation of internal micropore structure in LWA [26]. So the objective of this study is to study the thermal characteristics of SS and RS, the effects of sintering temperature on the physical characteristics of LWA and morphological structures and crystalline phases during the heating process. In addition, the solidification of heavy metals in heat-treated LWA was investigated.

## 2. Materials and methods

### 2.1. Materials

SS was obtained from the Harbin Wenchang Wastewater Treatment Plant in Heilongjiang province, China. The plant has a design capacity of  $1.0 \times 10^6 \text{ m}^3 \text{ day}^{-1}$ . The sludge cake generated from the activated sludge process was approximately  $1.6 \times 10^5 \text{ kg day}^{-1}$  in wet weight with 24% solids, and it went through the direct landfilling process. RS was obtained from the 32.59 km long Hejiagou River in Harbin, China, the longest inner river in Harbin city with a basin area of  $125 \text{ km}^2$  flowing from south to northeast. Since a long time, every year abundant untreated wastewater, municipal sewage and garbage from industries, mining enterprises and communities close to the river have been directly discharged into the Hejiagou River; therefore, water quality has rapidly declined.

The chemical characteristics of SS and RS are shown in Table S1 (Supplementary material). SS and RS were dried at 105 °C until they reached constant mass, and then, were crushed to pass a sieve with a mesh diameter of approximately 0.154 mm. Finally, they were preserved in polyethylene vessels to avoid humidification.

### 2.2. Methods

#### 2.2.1. Preparation of LWA

First, the raw materials were completely mixed at a mass ratio of SS:RS = 1:1, and then, appropriate amounts of water (80 mL water per 100 g raw material) were added. Subsequently, the mixed materials were shaped into pellets with diameters of 6–10 mm and dried at approximately 25 °C for three days. The LWA pellet samples were further dried at 110 °C in a blast roaster for 24 h. The samples were placed in a muffle furnace at 25 °C, and then, heated at a rate of  $8 \text{ }^\circ\text{C min}^{-1}$  and soaked at 200 °C, 600 °C and 800 °C for approximately 10 min and at 900 °C, 950 °C, 1000 °C, 1025 °C, 1050 °C, 1075 °C, 1100 °C, 1125 °C, 1150 °C, 1175 °C and 1200 °C for approximately 30 mins. The samples were left to cool to room temperature, and finally, they were stored in a desiccator.

To investigate the solidification effectiveness of heavy metals (Cr, Cd, Pb and Cu) in the raw materials for making LWA, synthetic metal solutions containing  $0.25 \text{ gL}^{-1} \text{ Cd}^{2+}$ ,  $5 \text{ gL}^{-1} \text{ Cr}^{6+}$ ,  $2.5 \text{ gL}^{-1} \text{ Cu}^{2+}$ ,  $5 \text{ gL}^{-1} \text{ Pb}^{2+}$  were prepared by dissolving  $\text{K}_2\text{CrO}_4$ ,  $\text{Cd}(\text{NO}_3)_2$ ,  $\text{Pb}(\text{NO}_3)_2$  and  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  in deionised water. The solution of heavy metals of  $\text{K}_2\text{CrO}_4$ ,  $\text{Cd}(\text{NO}_3)_2$ ,  $\text{Pb}(\text{NO}_3)_2$  and  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  was added to the raw materials in the process of making the LWA pellets. The contents of Cd, Cr, Cu and Pb were designed on the basis of the basic data obtained from the analyses of the activated sludge at different places in China as shown in Table S2 (Supporting

Information). The simulated concentrations of heavy metals in the SS and RS mixture (1:1) were  $50 \text{ } \mu\text{g mg}^{-1} \text{ Cd}$ ,  $1000 \text{ } \mu\text{g mg}^{-1} \text{ Cr}$ ,  $1000 \text{ } \mu\text{g mg}^{-1} \text{ Pb}$  and  $500 \text{ } \mu\text{g mg}^{-1} \text{ Cu}$ .

#### 2.2.2. Characterisation of LWA

The composition of SS and RS was determined using a Philips PW 4400 XR spectrometer (XRF, PANalytical, Netherlands). The thermal behaviour of the samples was examined by differential scanning calorimetry and thermogravimetric analyses (DSC–TGA) (TA-Q600, USA). The samples were heated from room temperature to 1200 °C in dry air atmosphere at a rate of  $8 \text{ }^\circ\text{C min}^{-1}$ . Powder XRD patterns of LWA were recorded on a D/max-g b X-ray diffractometer with 50 mA and 40 kV, Cu Ka radiation (D/max-2500, Rigaku, Japan). Data were collected at  $2\theta$  in the range 10–90° at a scan rate of  $0.02 \text{ }^\circ \text{ s}^{-1}$ . Water absorption, bulk and grain density tests were employed to characterise the quality of the sintered pellets. Water absorption and bulk density were determined according to GB/T 17431.2–2010 [27]. Grain density was obtained by the dry mass and volume of the sintered pellets. Individual grain density was calculated according to the Archimedes' principle. A sintered LWA with a diameter of 5–8 mm was placed vertically on the platform of an automatic material testing machine Instron 5569, (Instron, USA) and was pressed at a crosshead speed of  $0.5 \text{ mm min}^{-1}$  until it was crushed. Comprehensive strength is the average value of three tests per sample, and it is given by  $S = 2.8P_c / \pi X^2$ , where  $X$  is the diameter of the LWA specimens and  $P_c$  is the rupture load. SEM analyses were conducted with a Quanta 200 FEG scanning electron microscope (FEI, Hillsboro, USA) under an accelerating voltage of 20 kV.

The heavy metals extraction procedure employed in this study using a revised method derived from the toxicity characteristic leaching procedure (TCLP; US EPA Method 1311) and solid waste-extraction procedure for leaching toxicity horizontal vibration method (China, HJ 557–2010) [23,24]. Heavy metal leaching content was analyzed using an Optima 5300 DV Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES, Perkin Elmer, USA).

## 3. Results and discussion

### 3.1. Thermal behaviour of SS and RS

The thermal behaviour of SS and RS was examined separately by DSC–TGA. The TGA diagram in Fig. 1 (A) shows that weight loss of SS is more than 70% when the temperature increases from room temperature to 1200 °C. Approximately 9.8% weight loss is seen in the TGA curve when the temperature increases from 25 °C to approximately 135 °C, which is attributed to the evaporation of the absorbed water in SS, and this phenomenon corresponds to the endothermic peak in the DSC curve. Approximately 53% weight loss is seen in the TGA curve from 210 °C to 585 °C, which could be attributed to the elimination of the structural water and the combustion of organic matter and certain inorganic salts. Approximately 4% weight loss is seen in the TGA curve from 585 °C to 1200 °C that corresponds to the endothermic reaction in the DSC curve, and suggests that the carbon dioxide formed from calcium carbonate is eliminated and the sulphur dioxide formed by thermal decomposition is lost [28]. The TGA curves are shown in Fig. 1 (B) and the weight loss of RS is not more than 10% from room temperature to 1200 °C because RS mainly consists of inorganic components. A small weight loss is seen in the TGA curve when the temperature increases from room temperature to approximately 200 °C; however, an endothermic change is observed in the DSC curve at approximately 133 °C, which is attributed to the evaporation of water absorbed in RS. The major weight loss occurs from

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