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Characterization and pozzolanic properties of calcined alum sludge

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ABSTRACT

Microstructure and pozzolanic properties of water treatment sludge calcined for 3 h at 600–900 °C were investigated by XRD, FTIR, SEM, and TGA techniques and Chapelle test. Calcination of alum sludge was accompanied with sequence of chemical reactions including; dehydration of colloidal $Al(OH)_3$, formation of Al_2O_3, and crystallization of amorphous silica. Calcination conditions greatly affect the microstructure and pozzolanic activity of calcined alum sludge. Under the hydration at high temperature of calcined alum sludge with lime, boehmite (AlOOH), and amorphous silica react with lime forming amorphous calcium aluminate hydrates and calcium silicate hydrates. The amount and density of hydrates increased with calcination temperature up to 800 °C then decreased after that. Calcination of alum sludge at 800 °C is enough to improve its pozzolanic properties.

1. Introduction

Alum sludge continuously accumulates in drinking water treatment plants during the flocculation-clarification process using alum coagulant and further removed from the liquid phase by sedimentation/filtration processes [1]. Sludge amount and composition mainly depend on water quality, removal efficiency as well as type and dose of coagulant. The amount of sludge ranges from 1 to 5% of the total untreated water quantity [2]. Sludge contains suspension of inorganic and organic substances typically, silica, hydrated aluminum oxide, and iron oxide [3]. The moisture content of the wet sludge is generally above 80 wt%. The organic matter content of the dried sludge is about 25% and particle size distribution is under 100 μm [4,5]. Sludge management process in European countries comprises the following stages: sludge gathering and storage, pumping to thickening area, thickening, storage of thickened sludge, pumping to dehydration area, dehydration, atomization, and final storage [6]. On contrast, sludge discharges directly into nearby stream. The traditional practice of discharging the sludge directly into a nearby stream is becoming less acceptable because this discharges can violate the allowable stream standards [7]. The discharging of sludge into water body leads to accumulative rise of aluminum concentrations in water, aquatic organisms, and, consequently, in human bodies. Some researchers have linked aluminum's contributory influence to occurrence of Alzheimer's disease, children mental retardation, and the common effects of heavy metals accumulation [8].

The most feasible alternatives to solve the problem of sludge discharge including several reuse options were identified globally such as; for coagulant recovery and reuse [9], as coagulant in wastewater treatment [10], as adsorbent for phosphorus [11], manganese [12] and fluoride [13] from aqueous solutions, as co-conditioning and dewatering with sewage sludge [14], as constructed wetlands substrate [15], as soil buffers [16], nutrients reduction in laden soils and runoffs [17], in brick making [18–20], in ceramic making [21,22], in pavement and geotechnical works [23], for structural soil improvement [24] and in manufacture of cement [25–29], cementitious materials [30–36], and light weight aggregate [37,38]. Replacement part of clay by sludge improves the compressive strength of produced cement paste. Moreover, the microstructure of the sludge based clinker was similar in terms of alite and belite crystal size, and composition to OPC clinker [29]. Sludge used as supplementary cementitious material and sand substitute in preparation of cement mortar and concrete [32,33]. Sludge inhibits the setting and hardening of cement paste because aluminium sulphate delays setting of Portland cement [34] as well as the presence of sodium ions [35]. Addition of nano-SiO_2 improves the engineering properties of cement paste containing sludge calcined at 800 °C [36]. Bearing in mind that the calcination conditions i.e., temperature and duration affect the microstructure and the pozzolanic properties of industrial solid wastes [39] and as a result control the mechanical properties of pozzolanic cements [40,41]. Hence, the aim of this study is to study the microstructure and pozzolanic properties of water treatment sludge calcined at

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600–900 °C for 3 h. The investigation was accomplished by XRF, XRD, FTIR, SEM, and TGA instrumental techniques as well as Chapelle test for pozzolanic activity.

2. Materials and experimental techniques

Sample of wet sludge cake was collected from nearby drinking water treatment plant during the cleaning process of one of the decanters. The conventional water treatment plant uses aluminum sulfate coagulant in water treatment. Raw sludge dried and milled in steel ball mill to fine powder. Raw sludge was calcined in electrical muffle furnace with a heating rate 10 °C min^{-1} at 400–900 °C for 2 h. Calcined sludge was recharged from the muffle furnace and cooled to room temperature in desiccator. Calcined sludge was milled in steel ball mill and sieved using 90 μm sieve. Freshly prepared lime was prepared by calcination of limestone powder (purity > 99%) in electrical muffle furnace at 1000 °C. Lime was cooled to room temperature in desiccator, milled, and stored in tightly closed plastic bag to avoid carbonation. The pozzolanic activity of calcined sludge was evaluated according to Chapelle test. Equal amounts (0.375 g) both of calcined sludge and lime were added to 75 ml of distilled water in double cape plastic bottles of 100 ml maximum capacity. A blank sample contains the same amount of lime without addition of calcined sludge. Bottles were heated at 80 °C for 8 days. At the end of the run, lime treated calcined sludge was filtered, washed with distilled water, and dried in microwave oven. Filtrate solution was completed to 100 ml with distilled water. Amount of residual lime (in mmol l^{-1}) was estimated by titrating of 10 ml of the filtrate solution against 0.05 N HCl solution in presence of phenolphthalein indicator. Fixed lime content was estimated from the difference between the amount of lime measured for blank and sample solutions [42]. X-ray fluorescence (XRF) and X-ray diffraction (XRD) analyses were carried out by Philips X-ray diffractometer PW 1370, Co., with Ni filtered CuKα radiation (1.5406 Å). The Fourier transform infrared (FTIR) analysis was measured by spectrometer PerkinElmer FTIR System Spectrum X in the range 400–4000 cm^{-1} with spectral resolution of 1 cm^{-1}. Scanning electron microscopy (SEM) was investigated by Jeol-Dsm 5400 LG apparatus. The thermogravimetric (TGA) and differential thermogravimetric analyses (DTG) were carried out with the aid of Shimadzu Corporation thermo analyzer with DTG-60H detector with 10 °C min^{-1} heating rate from room temperature up to 1000 °C, under nitrogen atmosphere at 40 ml min^{-1} flow rate, the hold time at the appropriate temperature is zero.

3. Results and discussion

3.1. Characterization of alum sludge ash

Table 1 illustrates the chemical composition of alum sludge calcined at 900 °C as determined by XRF. Calcined sludge ash composes of SiO_2, Al_2O_3, and small amounts of Fe_2O_3, CaO, MgO, Na_2O, and K_2O. The sum of SiO_2, $A1_2O_3$, and Fe_2O_3 satisfies the requirements stated for pozzolana [43]. Fig. 1 illustrates the XRD patterns of raw sludge and that calcined at 600–900 °C. The major crystalline inorganic matter that has been present in the raw alum sludge are quartz (SiO_2), albite ($NaAlSi_3O_8$), and calcite ($CaCO_3$).

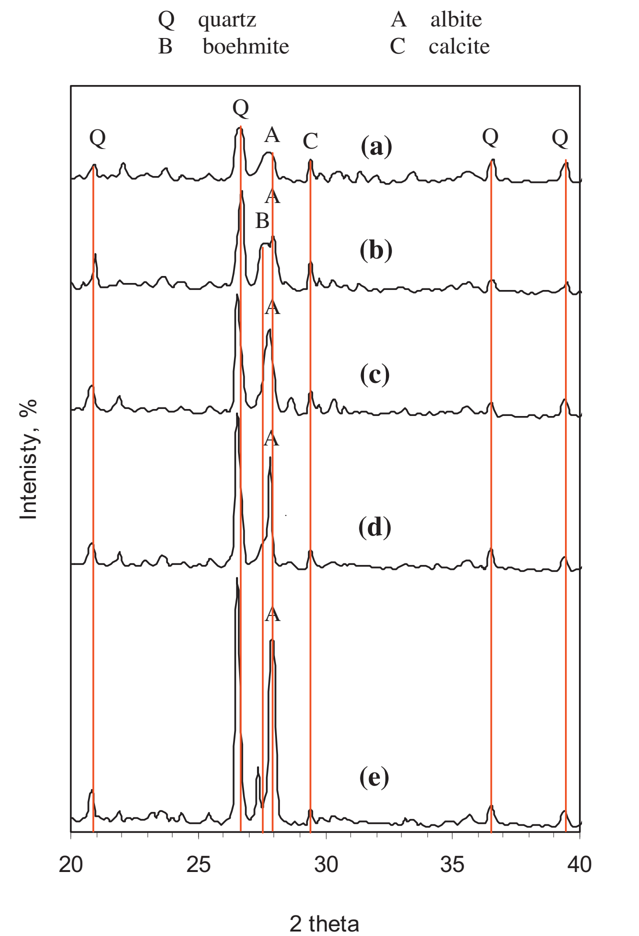

Fig. 1. XRD patterns of (a) raw alum sludge and that calcined at (b) 600 °C, (c) 700 °C, (d) 800 °C, and (e) 900 °C.

Absence of $Al(OH)_3$ peaks confirms its amorphous nature. Boehmite (AlOOH) appears in alum sludge calcined at 600 °C due to partial dehydration of amorphous $Al(OH)_3$. Boehmite disappears in alum sludge calcined at 700 °C transforming to amorphous alumina polymorphs, i.e., γ-, θ- and δ-alumina [44]. These amorphous humps were not observed due to existence of sharp intense peaks of crystalline solids. The formation of Al_2O_3 was proved in TGA/DTA analysis (Fig. 4). The amount of quartz increases markedly with calcination temperature. This may be due to the crystallization of amorphous silica into quartz that starts around 245 °C [45].

Fig. 2 illustrates the FTIR spectra of raw sludge and that calcined at 600–900 °C. The absorption bands of silica appear at 1105, 800, and 474 cm^{-1} which are attributed to asymmetric stretching vibration of Si—O—Si, symmetric stretching vibration of Si—O—Si and bending vibration of O—Si—O respectively [46]. The absorption bands of $Al(OH)_3$ at 530, 1628, and 3458 cm^{-1} are corresponding with the Al—O stretching vibration, bending vibration of water molecules chemically associated with $Al(OH)_3$ as well as OH

Table 1
Chemical composition of alum sludge calcined at 900 °C inferred by XRF.

Wt%	44.21	16.47	4.62	4.12	0.74	2.02	0.61	0.31	0.018	26.68	99.49
Oxide	SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	SO_3	Na_2O	K_2O	Cl^-	LOI	Total