



Gehlenite and anorthite formation from fluid fly ash

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ABSTRACT

Fluid fly ash could be considered a waste, but, when well treated, it may also become a useful secondary source material. Its rather high content of calcium-containing phases along with thermally treated alumino-silicate residues resulting from coal combustion can lead to the formation of a stable system with newly formatted phases. The high temperature destroys the clay lattice and activates a new configuration of aluminum ions, changing their coordination to oxygen. The effect is accompanied by changes in charge in the surroundings, which are compensated for by calcium ions. The higher the temperature of the fluid ash treatment, the more pronounced the appearance of gehlenite and anorthite in the final mass. Both are natural materials and, together with mullite and anhydrite, they could ensure safety and protection even if exposed to open fire of up to 1150 °C.

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1. Introduction

Previous works on the identification of fluid fly ash (FFA) components and their changes during further temperature treatment showed interesting changes in crystal phases [1–4]. The maximum current temperature of fluid coal burning is 820 °C, which is decisive for the formation of principal components. The addition of powdered calcium carbonate to the crumbled coal leads to the decomposition of calcite during the combustion and to the production of active calcium oxide. This oxide and sulfur trioxide from burned coal form anhydrite (CaSO₄). This reaction captures practically all sulfur from the burned coal and helps prevent the contamination of the environment [5]. Coal residues are mainly formed by clay substances (in the Czech Republic mainly kaolinitic clays), quartz and anhydrite with a small amount of iron oxide [6].

Limited combustion temperature changes the clay lattice; ash studies have revealed the main changes in aluminum ion coordination [3]. The decomposition of the clay lattice involves dehydroxylation at the very beginning of the thermal treatment. The subsequent limited thermal treatment changes the position of aluminum ions to the surrounding oxygen. Apart from silica, contained in clay, XRD analyses have confirmed quartz, which accompanies the amorphous alumina-silicate residue.

The tested products made from fluid ash easily resist the temperature of open fire (1150 °C). A prepared plate placed in the test

kiln has shown that the side exposed to the open fire exhibits changes in the phase appearance [1]. The newly formed gehlenite has been found together with anhydrite and quartz. Phases containing iron could also be detected but do not affect product stability and resistance. The newly formed ash component is gehlenite, generally found in blast furnace slag in a specific nascent form; its formation depends on the furnace temperature and crystallization during the cooling processes. Nascent and also disorganized gehlenite could have very different forms, e.g. (Ca_{1.96}Na_{0.05})(Mg_{0.24}Al_{0.64}Fe_{0.12})(Si_{1.39}Al_{0.61}O₇), when its ideal natural state (Alto Adige, Italy) is Ca₂Al(AlSiO₇) [7].

Fluid burning differs from classic burning in the addition of calcite to coal and in the fact that unburned particles return to the fluid layer several times. This effect could be seen as time dwell at the maximum temperature. This process allows the transformation of aluminum ions into their active five/four-fold coordination to oxygen.

It is known that the transformation of alumina ions to the five/four-fold coordination is hard to achieve at one hundred percent. When clay is treated at similar temperatures (750–800 °C), the transformation level depends on the time dwell at the maximum temperature. When the clay lattice is very well organized, the time dwell can easily reach 8 h. On the basis of the experience on clays confirmed previously [3], the amount of transformed aluminum ions is about 75% when the transformation occurs in a laboratory kiln (750 °C and 4-h time dwell). Also a study of gehlenite and anorthite crystallization based on a mixture of clay and calcite confirmed the reaction at limited temperatures [8]. The kinetics of

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the reaction between kaolinite and calcite were later studied by Ptáček et al. [9] when pure materials and different temperature of treatment were used.

The protection of environment on the one hand and the necessity of energy supply on the other have generated specific coal-combustion processes, which capture sulfur oxides from the coal combustion, transforming them into a solid secondary material – anhydrite. With the exception of anhydrite, the ashes are a mixture of active participants that can be worked with and used. Actually, a large amount of ashes are heaped without any benefits, which could cause environmental problems.

This study focuses on changes in heated fluid fly ashes. The observed transformation which occurs in clayed residues is similar to the thermal treatment of pure kaolinitic clay [10]. Ptáček et al. [9] described the reactions of pure materials (clay materials with calcium oxide) at different temperatures which should be comparable with the heated fly fluid ash.

These specific phase changes are the main aim of the thermal treatment of fluid fly ashes. The main point is to identify the stable final phases after heat exposure for the potential future use of this waste material. When the products are used for heat and fire protection, their stability means safety for the protected constructions and people.

2. Material

2.1. Fluid fly ash Kladno

Fluid fly ash from the circulating fluidized-bed boilers from Kladno (Central Bohemia) power station was selected. This ash contains a rather high amount of “free” calcium oxides and a residue of aluminum silicate, confirmed by XRD (see below). This fluid technology is specific for the reappearance of coal particles in the fluid layer, which decrease the content of unburned carbon, defined by a low L.O.I. (loss on ignition) value [5].

The XRD pattern has identified a major amount of lime (CaO), anhydrite (CaSO₄) and quartz (SiO₂), complemented by a small amount of hematite (Fe₂O₃) and unreacted calcite (CaCO₃) and mullite (3Al₂O₃·2SiO₂) in traces. The XRD pattern indicates roentgen-amorphous phases, which should be identified as transformed alumino-silicate (theoretically more than 56 wt % of the kaolin lattice residue).

The results of differential thermal analysis (DTA) and thermogravimetric analysis (TGA) (Fig. 2) have shown that the moisture and residual water contained in the samples are released during the decomposition. The graph indicates that the carbonates identified by X-ray analysis are decomposed [11]. The decomposition began at 589.7 °C and was terminated at about 760 °C. The loss corresponds to 4.2 wt %.

3. Sample preparation

The average samples of fluid fly ash (in the amount of 200 g for each test) were thermally treated in a laboratory muffle kiln using an un-glazed porcelain bowl. They were heated to the final temperature at the rate of 100 °C/hour and subsequently cooled down to laboratory temperature for 12 h. From the temperature of 1000 °C, the fired material was slightly sintered and the sample was carefully milled for further analyses. The samples studied included the original fluid fly ash and samples treated at 900 °C, 1050 °C, 1100 °C and 1150 °C.

4. Methods

The obtained samples of ashes were treated in a laboratory dryer up to the constant weight and then pulverized by a Retsch friction mill (a particle size of 0–2 mm). Homogenized ash was reduced by quartering to 50.0 g and this amount was ground by a vibration mill down to an average diameter at 50%: 15–20 μm. The ash treated in this manner was used in subsequent analyses.

Differential thermal analysis (DTA) and thermogravimetric analysis (TGA) were carried out under air atmosphere (0.005 m³ h⁻¹) by a MOM Budapest analyzer (MOM Budapest, Hungary). The sample was heated at 10 °C per minute up to the final temperature of 1000 °C. Data collection and evaluation were performed with the Terry software.

Non-destructive X-ray fluorescence (XRF) spectrometry (Spectro IQ, Kleve, Germany) was used, with the target material being palladium and the target angle from the central ray being 90°. The focal point was a 1 mm × 1 mm square, with the maximum anode dissipation of 50 W and 10 cfm forced-air cooling. The instrument was equipped with a HOPG Barkla crystal. The tested samples were prepared by the pressed-pellet method: 4.0 g of the material (a particle size of 15–20 μm) were mixed for 10 min with 0.9 g of a

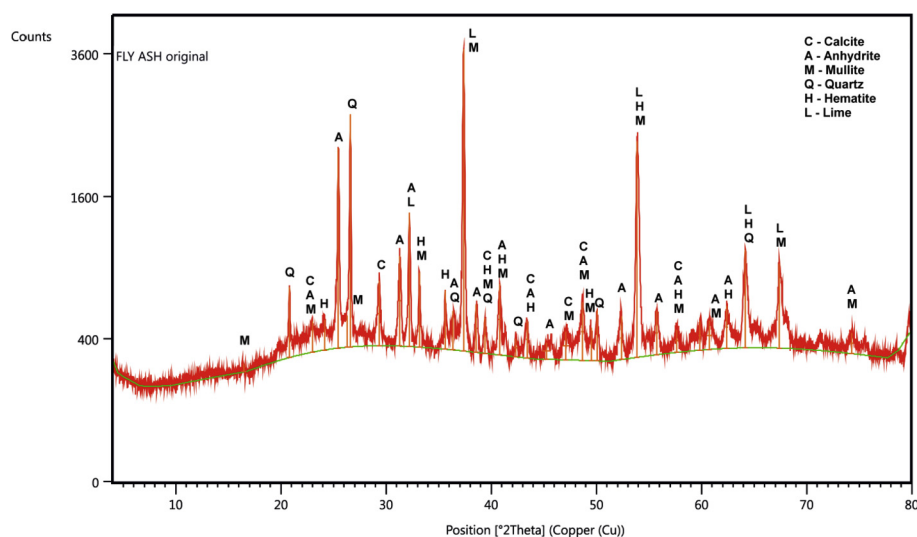


Fig. 1. The XDR pattern of the fly ash.

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