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## Long-term observation of yeelimite clinker hydration in environment of saturated water vapour

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### Abstract

Due to ecological overloading decrease, industrial by-products are utilized. Fly ash also belongs among these by-products. In Portland-composite cements, the high-temperature fly ash is applied nowadays, but fluidized bed combustion fly ash (FBC fly ash) is not used. The reason is that the FBC fly ash hydrates to form an undesirable ettringite,  $C_3A \cdot 3CaSO_4 \cdot 32H_2O$ . Decomposition temperature of ettringite in ambient conditions is most often mentioned in a range of 50 °C to 110 °C. In order to assess the ettringite long-term stability, ettringite was synthesized by hydration of mineral yeelimite,  $3CaO \cdot 3Al_2O_3 \cdot CaSO_4$ . The aim of this paper is long-term observation of the hydration of yeelimite in environment of saturated water vapour and examination of how stable the products of hydration are under given conditions. Raw materials for preparation of yeelimite were gypsum, limestone and corundum, weighed according to stoichiometric ratio of yeelimite. The mixture was heated at temperature of 1200 °C, soak 3 hours. After quenching and grinding, yeelimite was hydrated with a water-yeelimite ratio of 0.3. The system was cured as pastes and stored in conditions at laboratory temperature and in settings of saturated water vapour (100% R.H.). In order to follow the progress of synthesis regularly, namely up to age of 180 days of hydration, X-ray diffraction (XRD) was chosen using diffractometer Empyrean PANalytical (CuK-alpha). Based on the XRD patterns, only minor part of ettringite was formed, whilst a majority of yeelimite was preserved. It has been found that ettringite was not the only one product of yeelimite hydration. From an age of 20 days of hydration, a small amount of monosulfate was identified, whose quantity rose gradually. Especially at later ages of hydration, transformation of ettringite into monosulfate was observed.

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## 1. Introduction

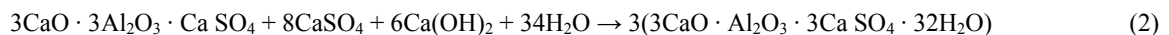
The cement production is, as many other industry sectors, a significant source of CO<sub>2</sub> emissions. Approximately half of that amount is generated due to its energy-intensive burning process when producing cement. Thus utilization of industrial by-products which are not necessary to burn, entails reduction of carbon footprint of cement production.

Fly ash also belongs among these by-products. Nowadays the high-temperature fly ash is applied into Portland-composite cements, but fluidized bed combustion fly ash (FBC fly ash) is not used. The reason is that the FBC fly ash hydrates to form an undesirable ettringite, C<sub>3</sub>A·3CaSO<sub>4</sub>·32H<sub>2</sub>O. Decomposition temperature of ettringite in ambient conditions is most often mentioned in a range of 50 to 110 °C [1,2,3,4]. In order to assess the ettringite long-term stability, ettringite was synthesized by hydration of mineral yeelimite, 3CaO·3Al<sub>2</sub>O<sub>3</sub>·CaSO<sub>4</sub>.

Yeelimite, also known as the Klein's Compound (Klein component/complex, Klein's salt), is a mineral with a composition of Ca<sub>4</sub>Al<sub>6</sub>(SO<sub>4</sub>)O<sub>12</sub>, also 3CaO·3Al<sub>2</sub>O<sub>3</sub>·CaSO<sub>4</sub>, shorthand C<sub>4</sub>A<sub>3</sub>S̄. Its name has the origin from nature deposits Har Ye'elim and Nahal Ye'elim in Israel where yeelimite was first found in nature. This mineral is cubic and creates white or colorless crystals of density of 2.61 g·cm<sup>-3</sup> [5,6].

Yeelimite was originally developed for use in expansive cements. It is the main phase (when the amount of yeelimite is > 50 % wt.) of calcium sulfoaluminate cements (CSA) and the key component (amount of yeelimite > 25 % wt.) of sulfobelite cements. It is used by itself or mixed with Portland clinker cement for its hydration-shrinkage endurance. Raw materials for its manufacture are limestone, bauxite and calcium sulphate dihydrate (gypsum) which are heated up at lower temperature (of about 1200 to 1250 °C) - compared to Portland clinker, which brings benefits such as reduction of production energy demand, decrease of quantity of CO<sub>2</sub> emissions of 35 % and also lower energy required to grind because yeelimite is easy to grind [7,8,9,10,11,12].

When mixed with water, hydration product is monosulfate and aluminum hydroxide, see Equation (1). In the presence of calcium ions and sulphate ions, yeelimite reacts to give ettringite according to Equation (2). When gypsum is depleted, monosulfate formation becomes dominant [11,12,13].



It is known that when molar ratio CaSO<sub>4</sub>·2H<sub>2</sub>O/yeelimite is greater than 2.0, only (2) takes place. However, some authors claim that mix of AFt and AFm phases is formed [8,10,13,14].

Kinetics of yeelimite hydration depends on water/yeelimite ratio, solubility of additional sulphate source and on yeelimite polymorphism [8].

In order to follow the progress of synthesis regularly, namely up to age of 180 days of hydration, X-ray diffraction (XRD) was chosen using diffractometer Empyrean PANalytical (CuK alpha).

## 2. Materials and methods

The experiment is based on the long-term observation of influence of environment of saturated water vapour on yeelimite clinker hydration. Yeelimite hydration gives ettringite, previous studies [13,15,16] verified this procedure as a possible way of ettringite synthesis.

The whole procedure comprised of a few steps: firstly, raw powdery meal for firing of yeelimite was prepared and fired as the next step. Then yeelimite was hydrated. Subsequently, hydrated yeelimite was regularly tested.

Yeelimite was totally prepared in three batches named Y0, Y1 and Y2. It was synthesized from three components: gypsum, CaSO<sub>4</sub>·2H<sub>2</sub>O (by-product from company Precheza, a.s., purity 98.6 %) and from analytical grade chemicals, namely limestone - calcium carbonate, CaCO<sub>3</sub> (purity 99.0 %) and alumina oxide, Al<sub>2</sub>O<sub>3</sub> (purity 100.0 %). All compounds were in powder form.

Raw materials were dried to a constant weight (limestone and corundum at temperature of 105 °C, gypsum at 40 °C) using circulating oven BINDER ED, APT line II with forced circulation. According to stoichiometric ratio of desired yeelimite and considering the three raw materials purities, 38.44 wt. % of limestone, 39.21 wt. % of

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