



Long term study of hardened cement pastes containing silica fume and fly ash



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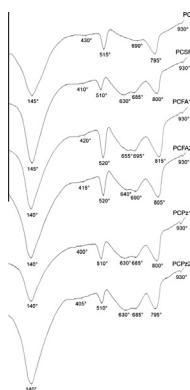
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HIGHLIGHTS

- Fly ash and silica fume participate actively in the hardening of cement paste.
- The used additives stimulate hydrate products with temperature of dehydration >600 °C.
- Polymerization of calcium hydrosilicates is observed after 600 days of hardening.
- The portion of products dehydrating below 340–350 °C is increased.
- Carbonation and polymerization in the hardening cement are enhanced by the additives.

GRAPHICAL ABSTRACT



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ABSTRACT

The hydration products in cements with individual addition of SF and two types of FA from TEPS and mixtures of FA and SF are studied. The phase transformations are followed in a 4-year period. Fly ashes from TEPS "Bobov Dol" and TEPS "Pernik", silica fume from the metallurgical plant "Kremikovtsi", and their mixed product Pozzolit are suitable pozzolanic additions to cement as they participate actively in the processes of formation of hardened cement paste. The above additives stimulate the formation of hydrate products with temperature of dehydration above 600 °C, more clearly expressed after the 48th day of hydration.

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1. Introduction

One of the most widely used active mineral additions are silica fume [1–6] and fly ash from TEPS [7–11]. They exert positive influence on a number of important features of hardened cement paste

and concrete – physical–chemical, non-permeability and corrosion resistance. The addition of a mixture of silica fume and fly ash to cement is one of the ways to obtain concrete with high exploitation characteristics (high performance concrete) [12–20].

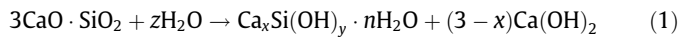
By use of fly ash there can be solved some problems concerned with the workability, which occur in case of addition of greater quantities of silica fume and at the same time silica fume compensates for the relatively low initial strength of the cement composites with added fly ash.

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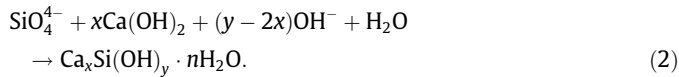
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The hydration processes in cement are most intensive in the initial periods of hardening and then the type and quantity of the hydration products change slower and the degree of hydration is usually evaluated by inspection of the chemically bound water, the quantity of the calcium hydroxide, and the lowering of the quantity of the tri-calcium silicate in the clinker minerals.

Important part in the hydration of cement is the interaction of the tri-calcium silicate (alite, C_3S) with water to form hydrosilicate gel (C–S–H) with ratio $CaO/SiO_2 = 1.6–1.7$ [21,22].



The active mineral additives, known also as pozzolana, react with portlandite according to the following scheme



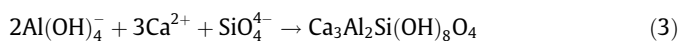
As a result, C–S–H gel is formed with lower CaO/SiO_2 ratio. The above described reaction is known as pozzolanic reaction.

The silica fume (SF) accelerates the early hydration of C_3S ensuring enough quantity of active centres as a basis for the formation of C–S–H. At this stage the intensity of the process is determined by the specific surface of the SF grains [23].

At different stages of hydration of cement with presence of silica fume there are formed three types of hydrosilicates:

- From the hydration of tricalcium silicate – reaction (1).
- From the pozzolanic reaction between SF and portlandite, resulting in formation of Ca hydrosilicates similar to the ones obtained during hydration of C_3S but with lower CaO/SiO_2 ratio. The intensity of portlandite formation in the early stages is higher than the speed of the reaction of its consumption by silica fume.
- When the quantity of portlandite significantly lowers then SF reacts with C–S–H to form hydrosilicates with low CaO/SiO_2 ratio, which at later stages of hardening are characterized with high degree of polymerization [23–26].

The fly ashes, which contain alumina also react with portlandite (reaction (3)) to form Ca alumo-hydrosilicates

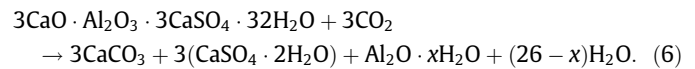


With inclusion of pozzolanic additives in cement the quantity of portlandite cannot be used as a reliable criterion for the degree of hydration due to the continuing pozzolanic reactions in the hydrating cement. In case of high concentrations of the mineral additions it may be very low even missing irrespective of the fact that the hydration is significant [24,27,28]. From another side, the addition of silica fume and fly ash stimulates the polymerization of the hydrosilicates, which are formed during hydration of cement accompanied by evolution of water. Therefore, the quantity of chemically bound water is not a correct indicator for determining the degree of hydration of cements with pozzolanic additions [18,24,29–32].

Portlandite and the Ca-hydrosilicates, which comprise about 85% of the weight of the hardened cement paste, are capable of forming carbonate in a longer time period. When the cement composites are subjected to the action of carbon dioxide, the latter is dissolved in the pore-liquid of the cement pastes ensuring CO_3^{2-} ions, which react with Ca^{2+} ions of portlandite and the Ca-hydrosilicate gel to form calcite – $CaCO_3$. In case of prolonged contact of the cement pastes with air and water it is possible to reach complete carbonation with small residue of ettringite, carbo-aluminates and hydrosilicates. The depth of the formed carbonate layer depends on the time of contact with CO_2 and its concentration in the surrounding environment as well as on the coefficient

of diffusion of the hardened cement paste [33–35]. The volume changes, which accompany the processes of carbonation lead to filling of the empty pore volumes with Ca-carbonates and densify the structure of the hardened cement paste.

The carbonation of portlandite, Ca-hydrosilicates, and ettringite proceeds according the following scheme [33,36]



The carbonation in the presence of SF and fly ash is a more complicated process due to the pozzolanic reactions. The products, subjected to carbonation (CH and C–S–H) participate in pozzolanic reactions or they themselves are obtained as a result of these reactions [33,36]. The carbonation of CH and C–S–H proceeds simultaneously [37–39]. With the development of the process and the accompanying lowering of the portlandite quantity the mean length of the polymer chains of C–S–H progressively increases, the excess of water is released [38] and with those processes one can explain the carbonation shrinkage [40]. The process is markedly expressed in cements with pozzolanic additives in which there are formed Ca-hydrosilicates with low CaO/SiO_2 – ratio as well as in aggressive carbonating media [41]. Thus the pozzolanic additions modify the cement matrix, alterations occur in it, which develop with time. The changes depend on many factors – chemical composition and quantity of the additions, specificity of the interactions with the hydration products of cement, the quantity of water expressed by the water to cement ratio. Therefore, to fully clarify the above dependencies there is need to conduct a long-term study of the type and quantity of the hydration products and their transformations in respect to any additive alone or in combination. The studies on cements with separate addition of silica fume or fly ash are numerous but the studies on the structure of hydration products in cements for cases of combined addition SF and FA are restricted [19,42].

The recent paper reports results on studies on the type and quantity of the hydration products in cements with individual addition of SF and two types of FA from TEPS and mixtures of FA and SF, the latter being a product according the trade mark “Pozzolit” and their transformations are followed in a 4-year period.

2. Materials and methods

2.1. Materials

The cement (PC) is produced by the cement plant “Titan” (Zlatna Panega, Bulgaria) – CEM I 42.5 N with mineral composition: C_3S – 57.52%; C_2S – 23.48%; C_3A – 5.54%; C_4AF – 11.7%, with addition of 1.76% gypsum and density of 3 g/cm^3 . Two types of FA are used (Table 1), both with the particle size 100% below $63 \mu\text{m}$, respectively, from “TEPS “Pernik” (Pernik, Bulgaria) (sample FA1) and TEPS “Bobov Dol” (Bulgaria) (FA2) as well as silica fume (SF) with particle size 90% below $10 \mu\text{m}$ and 60% below $1 \mu\text{m}$. The specific surface of the particles of the mineral additives (determined by BET method) is: SF – $18.6 \text{ m}^2/\text{g}$; FA1 – $1.76 \text{ m}^2/\text{g}$; FA2 – $2.67 \text{ m}^2/\text{g}$. Applying mechanical mixing of FA and SF in weight ratio 50%:50% there have been prepared two mineral additives Pozzolit 1 (Pz1) and Pozzolit 2 (Pz2), respectively from FA1 and SF and from FA2 and SF.

2.2. Samples

The samples were prepared with water to binder ratio $W/C = 0.5$ (Table 2). The cement and the mineral addition were homogenized in a dry state. The dry materials were added into water and the resulting mixture was stirred for 5 min. The hydrating pastes were hermetically closed in plastic containers up to the 24-th hour and then the material was released from the containers and kept in water at 20°C up to the age of testing.

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