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ТЕХНОЛОГІЯ І ВЛАСТИВОСТІ МОДИФІКОВАНОГО КОМПОЗИЦІЙНОГО ПОРТЛАНДЦЕМЕНТУ ДЛЯ СТАЛОГО БУДІВНИЦТВА

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Анотація. Більшість пуцоланових добавок, зокрема метакаолін або мікрокремнезем, мають тенденцію підвищувати водопотребу бетонних сумішей. Однією з головних причин збільшення водопотреби тонкодисперсних мінеральних частинок є їх підвищена питома поверхня. Отже, для оптимізації гранулометричного складу портландцементу з добавкою мікрокремнезему та активації процесу тверднення композиційного цементу запропоновано технологію поверхневого модифікування у високовольтному електричному полі. Розроблено установку електричної агломерації для одержання модифікованого композиційного цементу. За результатами сканувальної електронної мікроскопії з енергодисперсійною спектроскопією виконано дослідження частинок модифікованого композиційного цементу. За результати диференційного термокінетичного та рентгенофазового аналізу вивчено процес тверднення композиційного портландцементу з добавкою мікрокремнезему.

Ключові слова: мікрокремнезем, композиційний цемент, поверхневе модифікування, електрична агломерація, домашня частинка, гостьова частинка, цементна паста.

ТЕХНОЛОГИЯ И СВОЙСТВА МОДИФИЦИРОВАННОГО КОМПОЗИЦИОННОГО ПОРТЛАНДЦЕМЕНТА ДЛЯ УСТОЙЧИВОГО СТРОИТЕЛЬСТВА

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Аннотация. Большинство пуццолановых добавок, в частности метакаолин или микрокремнезем, имеют тенденцию повышать водопотребность бетонных смесей. Одной из главных причин увеличения водопотребности тонкодисперсных минеральных частиц является их повышенная удельная поверхность. Таким образом, с целью оптимизации гранулометрического состава портландцемента с добавкой микрокремнезема и активации процесса твердения композиционного цемента предложена технология поверхностного модифицирования в высоковольтном электрическом поле. Разработана установка электрической агломерации для получения модифицированного композиционного цемента. По результатам сканирующей электронной микроскопии с энергодисперсионной спектроскопией выполнено исследование частиц модифицированного композиционного цемента. По результатам дифференциального термокинетичного и рентгенофазового анализа изучен процесс твердения композиционного портландцемента с добавкой микрокремнезема.

Ключевые слова: микрокремнезем, композиционный цемент, поверхностное модифицирование, электрическая агломерация, домашняя частица, гостевая частица, цементная паста.

THE TECHNOLOGY AND PROPERTIES OF MODIFIED PORTLAND-COMPOSITE CEMENTS FOR SUSTAINABLE CONSTRUCTION

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Abstract. Most pozzolanic materials, especially metakaolin or silica fume tend to increase the mixing water requirement for concrete. The most common reason is that fine mineral particles raise the water demand due to increased surface area. So, in order to optimize the grain size composition of Portland cement with the addition of silica fume and to activate the hardening process of composite cement the technology of surface modification in high-voltage electric field is proposed. The electrical agglomeration setup to produce modified composite cement has been designed. An observation of cross-section of modified composite cement particle as well as an analysis of the element distribution by SEM with EDS is shown. The hardening process of composite Portland cement with the addition of silica fume according to the data of the differential thermokinetic and X-ray analysis is considered.

Keywords: silica fume, composite cement, surface modification, electrical agglomeration, host particle, guest particle, cement paste.

1. Introduction

All products used in the construction industry have embodied environmental impacts - whether from raw materials, manufacture or transportation. It is well known, that the cement industry produces approximately 5–7 % of global manmade CO_2 emissions. The CO_2 emissions directly resulting from clinker production fall into two main categories: those derived from de-carbonation of the raw materials and those derived from the fuel burned in the kiln [1].

A typical modern rotary cement kiln with a specific heat consumption of 3,1 GJ/t clinker, burning traditional carbon based fuels such as coal, oil or petroleum coke, emits approximately 0,31 kg fuel derived CO_2/kg clinker. On the other hand, inefficient long rotary kilns burning wet raw materials typically operate at a heat consumption of about 6 GJ/t clinker, and a fuel derived CO_2 emission of about 0,6 kg/kg clinker [2].

Compared to fuel derived CO_2, carbon dioxide derived from the raw materials is relatively high at approximately 0,53 kg/kg clinker. So, the total CO_2 emissions from kilns burning conventional fuels and raw materials therefore range from 0,84 to 1,15 kg/kg clinker depending primarily on the heat consumption of the kiln [1, 2].

The most effective means of achieving significant reduction of CO_2 emissions lies in the replacement of Portland cement clinker by other suitable materials. These replacement materials can be added separately to the concrete allowing a reduction in the content of clinker for the same concrete performance, or used to replace the clinker in composite cements. Replacement materials that react with calcium hydroxide are commonly termed «Supplementary Cementitious Materials», (SCMs). They include fly ash, granulated blast furnace slag, natural pozzolans, and to a lesser extent silica fume, metakaolin, etc. [3–5].

The incorporation of SCMs into cement or concrete systems provides many benefits to properties of both fresh and hardened concrete. However, most pozzolanic materials, especially metakaolin, silica fume or fine milled lime stone tend to increase the mixing water requirement for concrete and lower the rate of strength development. The most common reason is that fine mineral additives raise the water demand due to increasing surface area [5].

On the other hand, fumed silica consists of very fine particles that have a strong tendency to form agglomerates. So, to reduce the water demand of composite cement the particle size distribution should be optimized. This problem can be realized

by surface modification of components of composite cements, in particular by dry particle coating.

Dry particle coating to change the surface properties and/or functionality of powders appears as a very important process for many industries. Typical applications include modification of flowability, wetability (hydrophobic/hydrophilic properties), solubility, dispersibility, flavour, particle properties [6–8]. In such coating processes, powders with relatively large particle sizes (host particles: 1–500 μm) are mechanically coated with the fine particles (guest particles: 0,1–50 μm) in order to create a new functionality or to improve their initial characteristics (Fig. 1) [7].

Dry impact blending to produce co-called «spherical cement» described in a series of papers by a Japanese group [9–12]. The high fluidity of spherical cement comes from its round shape and particle size distribution. In particular, the particle size is distributed in a narrow range and the volume of fine particles under 3 μm is less than that in normal Portland cement.

The packing ratio of spherical cement increases, and the fluidity and workability of concrete using spherical cement also increase as compared to those of normal Portland cement. Furthermore, the strength and durability of concrete on the base of spherical cement are improved because less water is required for mixing. The adiabatic temperature rise of spherical cement is smaller than normal cement due to reduction of the unit weight of cement for the same strength appearance. The authors [9–12] believe that spherical cement is a new type of cement that can be used for several high-quality concrete types including high-fluidity concrete mixtures (SCC), high strength and high-durability concrete (HPC).

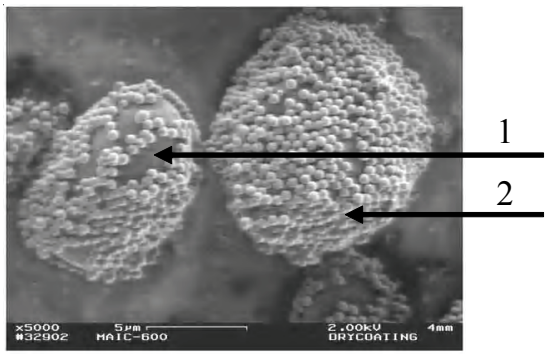

Figure 1. The scheme of particles modified by dry impact blending: 1 – host particle: 1–500 μm; 2 – guest particle: 0,1–50 μm.

Spherical cement is prepared by a dry impact blending method (microhybridization technology), where a mixture is formed by covering the surface of large cement «host» particles with ultra fine mineral «guest» particles, in particular microsilica. The adhesion driving forces between particles of spherical cement are due to the van der Waals interaction and electrostatic attraction [11] as well as liquid bridge forces. In turn, the electrostatic interaction between particles is realized by their triboelectric charging. However, it is well known [13] that triboelectric charging is extremely sensitive to environmental conditions. If the electrostatic forces between host and guest particles are relatively weak, insufficient adhesion strength takes place. Thus the fine particles are often peeled off from the surface of core grains during the technological processing such as mixing, pneumatic transport or compression forming where relatively strong mechanical forces are applied to the components of concrete mixtures [14].

To overcome this problem and to increase the attraction of units in a particle system a selective electrostatic charging of single particles can be employed [13]. For this reason the method of electrical agglomeration of Portland cement and microsilica particles as well as the laboratory experimental setup has been developed.

2. Experimental details

2.1. Materials

Ordinary Portland cement CEM I 42.5 N (OPC) and silica fume (SF), which is a by-product of the ferrosilicons production were used as raw materials. The chemical composition and physical properties of materials used are given in Table.

2.2. Experimental electrical agglomeration setup

Agglomeration in an alternating electric field (AC-agglomeration) is a process in which large particles are formed via coagulation of smaller particles. The principle of AC-agglomeration is presented in Fig. 2. Particles are first charged by a corona discharge as in a conventional electrostatic precipitator. Next they enter an alternating electric field where they start to oscillate. The electrical mobility and so also the oscillation velocity depend on the particle size, so that the larger the particle

Table. Chemical composition and properties of the materials used

Composition (%) Properties	OPC	SF
SiO_2	21,4	91,8
Al_2O_3	5,8	1,1
Fe_2O_3	3,4	0,65
CaO	61,5	2,4
MgO	1,7	0,05
K_2O	0,7	0,1
SO_3	2,5	0,35
Loss on ignition	1,2	3,6
Bulk density ($kg \cdot m^{-3}$)	1310	215
Fineness ($m^2 \cdot kg^{-1}$)	365 (Blaine)	18600 (BET)

size the larger are the velocity and the amplitude. The differences of the particles oscillation velocity and amplitude cause collisions between them. So, the particles remain attached to each other [15].

It is necessary to mention the fact that only the charged grains will be moved in an alternating electric field [16]. Better results are expected using bipolar distribution of large and small particles [17].

Particles are charged previously by a corona charger. In this case they are charged bipolarly – Portland cement (PC) in the positive corona discharge and the silica fume (SF) in the negative one (U = +/– 25 kV, I = 30–50 μA). Then the aerosols of charged particles flow into a chamber of alternating electric field: agglomerator. The electric strength of electric field is 3–5 kV/cm. Large charged cement particles oscillate with bigger amplitude and velocity than the small charged microsilica particles. The difference in particle velocities and amplitudes oscillation causes their collisions and agglomeration.

2.3. The host / guest particles mass ratio

The evolution of the percentage by mass of guest particles used in agglomeration experiment is calculated based on the assumption of 100 % surface coverage of the host particles (PC) with a monolayer of guest particles (SF). It is assumed that all guest particles are of same size, both host and guest particles are spherical, and that the shapes of host and guest units do not change during the coating process. Based on these assumptions, the mass percentage (W) of guest particles for 100 % coverage is [6, 8]:

$$W, \% = \frac{(Nd_{guest}^3 \cdot c_{guest})}{(D_{host}^3 \cdot c_{host}) + (Nd_{guest}^3 \cdot c_{guest})} \cdot 100. \quad (1)$$

For $D_{host} >> d_{guest}$ (here, $D_{host} / d_{guest} \approx 10$), the number N of guest particles per host particle is given by the expression:

$$N = \frac{4(D_{host} + d_{guest})^2}{d_{guest}^2}. \quad (2)$$

The average dimensions of particles are: Portland cement (host particles) D_{host} = 20 μm, silica fume (guest particles) d_{guest} = 2 μm. From Eq. (1), the percentage of guest particles needed to coat host particles is 25,5 %.

3. Results and discussion

3.1. The shape of cement particles

In this paper both an observation of a cross-section of cement particles by scanning electron microscopy (SEM) and an analysis of the element distribution (EDS) of the surface of modified composite cement are shown. It was observed that Portland cement grains have an angular shape with particle size distribution in the range of 1–50 μm (Fig. 3 a). After the treatment of Portland cement and silica fume in the electrical agglomeration setup the particle size distribution became narrower 5–50 μm with a predominance of large particles. These results are due to the modification from sharp-cornered grains to spherical shape based on the fixing and embedding of fine silica fume particles (Fig. 3 b).

According to the data of energy dispersion spectroscopy the following oxides were found surrounding the surface of modified cement, %: SiO_2 (84,93); SO_3 (11,91); Al_2O_3 (1,85); MgO (0,84); K_2O (0,45) (point 1, Fig. 3 c) – these elements belong predominantly to the chemical composition of silica fume. For comparison, an angular grains on the spheroid surface (point 2, Fig. 3 c) is represented

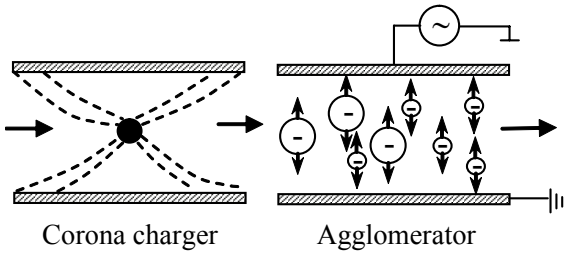

Figure 2. The principle of AC-agglomeration.

by the next set of oxides, %: CaO (51,44); SiO_2 (37,31); Fe_2O_3 (4,21) SO_3 (2,24); Al_2O_3 (1,43); K_2O (3,07), which is very close to the chemical composition of Portland cement.

3.2. Fluidity of cement paste

The flow of cement paste was measured in accordance with [18]. The flow pipe is 50 mm in diameter and 100 mm in height. Flow (F) was measured by averaging two crossing diameters of the spread. The relative flow area ratio (G) as the index of fluidity was calculated by the Eq: $G = F^2 / 50^2 - 1$. The composition of cement paste is: PC = 210 g; SF = 70 g; SNF SP = 2,8 g (dry powder).

It has been found that control cement paste with water-to-cementitious ratio w/c= 0,32 is characterized by the relative flow area ratio G = 1,1. In the case of modified cement the value of relative flow area ratio G = 1,1 is reached when water-to-cementitious ratio is w/c = 0,285 (less than 11 %). So, 28–day compressive strength of samples on the base of modified cement 37 % is higher in comparison to control samples.

3.3. Calorimetric researches of thermal emission kinetics

The next samples were investigated during calorimetric researches of thermal emission kinetics: 1 – Ordinary Portland cement (type I) – 100 %; 2 – Ordinary Portland cement (type I) – 75 % and silica fume – 25 %; 3 – Modified cement: Ordinary Portland cement (type I) – 75 % and silica fume – 25 %.

The height of the first peak of the heat evolution rate curve (Fig. 4) of sample 2 is lower than that of OPC (1). In turn, the height of the first peak of sample 3 (modified cement) is lower than that of

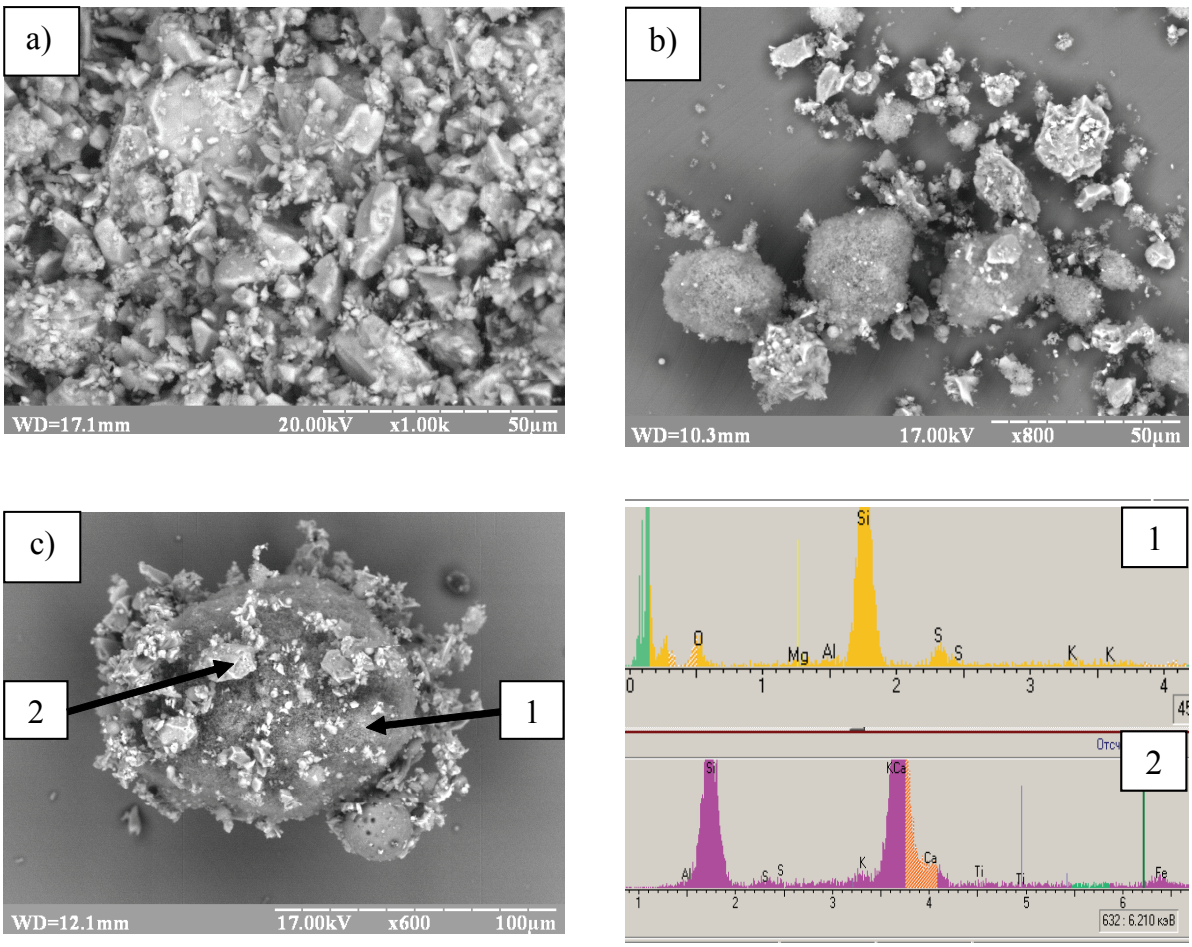

Figure 3. Scanning electron microphotograph of particles of Portland cement (a) and modified composite cement (b, c) with EDS.

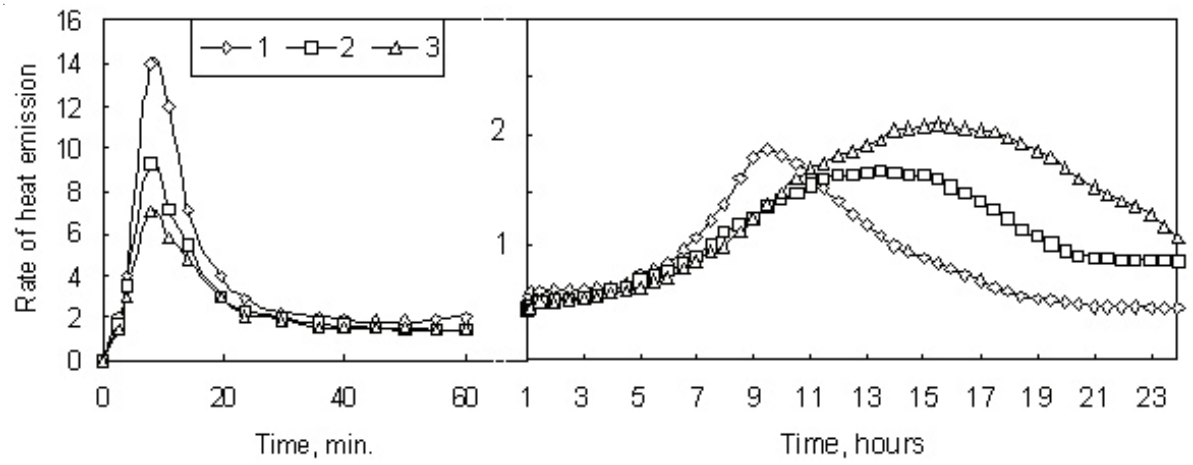

Figure 4. Heat evolution rate curve of cement paste.

sample 2 (control cement). Perhaps this is due the fact that the layer of ultra fine silica fume particles blocks the penetration of water to the surface of Portland cement.

The height of the second peak of the heat evolution rate curve is recorded at 9,5 hours of hydration for Ordinary Portland cement paste (1) and at 13–14 hours for the control sample (2) and 15–16 hours for the modified sample (3). On the other hand, the maximum quantity of heat evolution is observed for the modified composite cement, which indicates increasing degree of hydration. These data are consistent with the X-ray diffraction analysis of 28 days cement paste (Fig. 5). The intensity of diffraction reflections lines of tricalcium silicate (d = 0,386; 0,277; 0,176 nm) and portlandite (d = 0,491; 0,311; 0,263; 0,193; 0,170 nm) of modified composite cement is less in comparison with control samples. At the same time the higher intensity of the calcium silicate hydrates lines (C–S–H) (d = 0,304; 0,280; 0,182 nm) is observed.

Conclusion

The method for surface modification of Portland cement with addition of ultra fine silica fume in high-voltage electric field is proposed. The electrical agglomeration setup to produce modified composite cement has been designed. It was found that Portland-composite cement modified by electrical agglomeration exhibits high fluidity, thereby improving the workability of concrete as well as the higher strength and durability.

The results of this investigation can be considered as the comprehensive set of the basic principles of the sustainable construction.

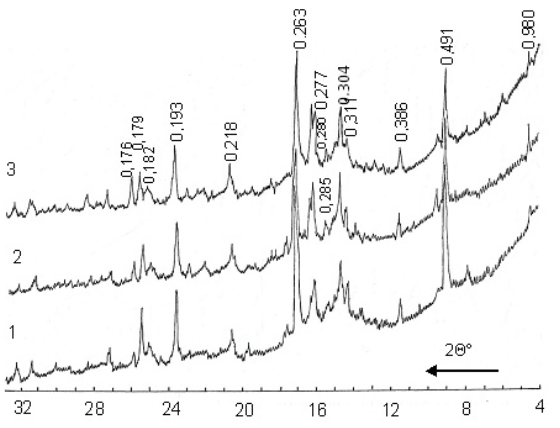

Figure 5. XRD-patterns of cement paste.

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