

INFLUENCE OF THE COARSE FLY ASH ON THE MECHANICAL PROPERTIES OF THE CEMENT MORTARS

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This study has examined the effect of fly ash used as replacement addition (in the quantities of 0-40 mass %) to the Ordinary Portland cement (OPC) (type CEM I 42,5R) on the setting time in the system of paste, compressive strength development ($w/s = 0.475-0.5$, $t = 20^\circ\text{C}$) and on drying shrinkage of cement mortars ($t = 20^\circ\text{C}$, $RH \geq 55\%$). Enlarged addition of fly ash affects extension of setting time and also affects reducing of the water, needed to achieve normal consistency. The increased addition significantly improves resistance to drying shrinkage throughout the measurement period. On the other side, increased amount of added fly ash reduces early strengths of cement mortars. In the later hydration stages, when pozzolanic reaction takes place, the strength difference between samples made with and without addition of fly ash decreases. The effect of the pozzolanic reaction, determined by using TG/DTA, can be observed after 60 days of hydration. A detailed chemical (X-ray diffraction), thermogravimetric and morphological (scanning electron microscope (SEM)) analyses of the hydrated samples of mortars are presented.

INTRODUCTION

Great attention has been paid to application of various mineral additions to cement/concrete in the last decades. These additions can be of natural origin (clay, kaoline, metakaoline, carbonates) or artificial, formed as industrial waste materials (blast furnace slag, dust, silica fume, fly ash) [1-3]. Reuse of waste materials plays a very important ecological and economic role in cement production, significantly reducing use of natural raw materials, energy needed for cement production, CO_2 emissions in cement production (1 t cement = 1 t CO_2) and the final cement price at the market. [4].

Fly ash, microsilica (silica fume), metakaoline, and granulated blast furnace slag are pozzolanically active materials that are very often used as replacement additions to cement/concrete [5-9]. Pozzolanically active parts of mineral additions (amorphous and metastable aluminosilicates) chemically react with calcium hydroxide ($\text{Ca}(\text{OH})_2$ – portlandite (CH)) and form a new C–S–H phase. The C–S–H phase is the primary hydration product of OPC and is responsible for setting and hardening of cement and cement composites [10,11]. The start of the pozzolanic reaction, formation of new C–S–H phases, is reflected as an increased quantity of chemically bound

water and as quantity of portlandite formed by hydration decreases, mechanical properties of cement composite materials are improved. Mechanical properties of mortars/concrete (compressive and bending strength, elasticity module, total porosity, shrinking/expanding) result from the developed microstructure, defined by formed hydration products and the pore system [12]. The newly formed C–S–H phase fills the pores in developed microstructure of the cement composite, thereby making the microstructure more even and less porous, which directly increases the durability of the composite [13]. The fineness of used pozzolanic addition is an important factor that, beside the physical effect of occupying the pores, determines the beginning of the pozzolanic reaction. According to studies made so far, the generally accepted rule is: “The greater the specific surface area of the material (microsilica, fly ash...) the pozzolanic reaction starts earlier” [5, 14-16].

This paper concerns the study of coarse fly ash addition up to 40 mass % as the replacement addition for Portland cement (CEM I 42,5 R) on the setting time in the system of cement paste and influence addition on compressive strength, drying shrinkage and microstructure of the cement mortars till 310 days hydration.

EXPERIMENTAL

Materials used in the investigation

Cement

The cement used in the study, is the CEM I 42.5 R, cement, commercially available, produced in Dalmacija cement/CEMEX company, Kaštel Sućurac, Croatia,. Its specific density is 2560 kg/m³. In paste systems, the initial settings after 3.45 hours and final settings after 4.28 hours of hydration are observed. The specific surface area of the used cement is 332.9 m²/kg. Table 1 shows the chemical composition of the cement.

Fly ash

A fly ash, used in this study, (FA) is a waste material formed by stone coal burning in the Plomin 2 thermal power plant (Croatian Power Supply (HEP), Croatia) that has been separated from the waste gases by electrostatic separation. Fly ash generally contains small spherical particles that significantly differ in size and shape. The specific gravity of used fly ash is about 1839 kg/m³ and the specific surface area is about 312.9 m²/kg. The results of the grain size distribution analysis (obtained by sieving) show that the fraction retained on a 0.045 mm sieve is 28.2 % and the fraction passed through a 0.045 mm sieve is 71.8 %, which correspond with the criteria for application in cement/concrete production required by the EN 450:1994 standard. Table 1 shows the chemical composition of fly ash. Fly ash from TE Plomin-2, HEP belongs to class “F” fly ash according to the ASTM C618 standard [17], or to class “V” according to the HRN EN 197-1:2000, both standards classification are based on the low CaO content (content CaO < 10 mass. %). XRD powder pattern of the used fly ash is showed on

the Figure 1. Qualitative analyses of the powder pattern (fly ash sample) were made by comparing experimental diffraction data with the diffraction data for already known compounds stored in the JCPDS database [18]. Results of the analyzed diffraction powder pattern show that the main diffraction maximums arise from the three main crystal phases: mullite (PDF # 15-0776), quartz (PDF # 12-0708) and hematite (PDF # 24-0072). The diffuse bands are present in the region 15° < 2θ < 30° and they indicate a presence of the amorphous (glass) phase. Two diffraction maximums at 2θ = 44.9° and 38.3° arise from the samples holder, not from fly ash sample.

Sand

In cement mortar preparation, quartz sand produced in Germany, marked as CEM Normsand DIN EN 196-1 was used, which meets the quality requirements for standard sand as a fine aggregate for examination of mechanical properties of cements according to the HRN EN 196-1 standard [19].

Table 1. Physical properties and chemical composition of used cement and fly ash.

Compounds	CEM I	Fly ash
SiO ₂	20.38	62.13
Al ₂ O ₃	5.41	20.83
Fe ₂ O ₃	2.81	9.50
CaO	61.77	2.65
MgO	1.76	0.83
SO ₃	3.01	0.20
Na ₂ O	0.17	0.40
K ₂ O	1.00	1.27
L.o.i. (1000°C)	2.80	1.60
Moisture Content	0.20	0.10

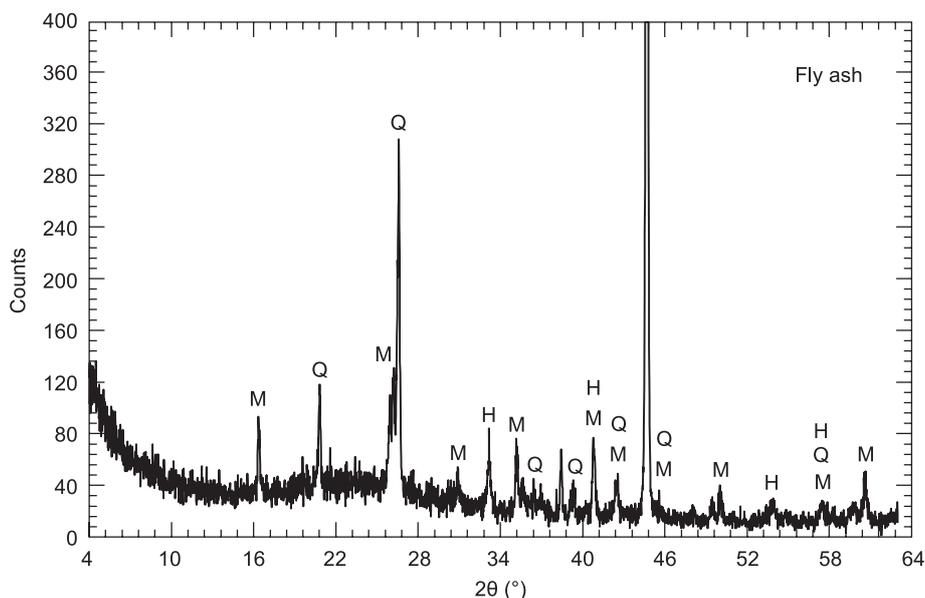


Figure 1. XRD powder pattern of the fly ash (Q - Quartz, M - Mullite, H - Hematite).

Mixture proportions and sample preparation

Preparation cement pastes

Samples of CEM I 42,5R cement and fly ash were dried for 1 hour at a temperature of 105°C and kept in a dessicator until the preparation. In order to determinate influence of the fly ash on the setting time, cement pastes were prepared without and with the replacement addition of fly ash in the amounts of 5, 10, 15, 20, 25, 30, and 40 mass % of the cement mass. Samples were marked as it follows: PB0, PB5, PB10, PB15, PB20, PB25, PB30, and PB40. Sample made without any replacement addition, a reference sample, was marked as PB0. Distilled water was used in preparation of paste samples. Normal consistence, start and the end of binding were determined on prepared samples by means of the Vicat method.

Preparation of cement mortars and testing procedures

The cement mortar was prepared according to the standard HRN EN 196-1. The standard quartz sand was used as aggregate. The aggregate/binder (cement + fly ash) ratio was constant in all mixtures and was 3:1. The quantity of water needed in mortar preparation differed, so the mortar samples were prepared in such a way that they had the same consistency, measured by mortar spreading on a flow bed within the limits of 156 - 165 mm. Mortars containing FA were prepared by replacing cement with 5, 15, 20, 25, 30, and 40 mass % FA. Mortars were marked as it follows: PB5, PB15, PB20, PB25, PB30, and PB40 (Table 2). A sample without FA was marked as PB0. Prepared mortars, with the dimensions of 4×4×16 cm, were cured for 24 hours in a conditioned chamber at a temperature of 20°C and relative humidity (RH) higher than 95 %, after which samples were taken from the mould and cured in conditions appropriate for the intended use for the rest of the time until examination. Mortars intended for examination of mechanical properties (compressive strength) were cured in thermostatted pools filled with water (RH = 100 %, T = 20 ± 1°C).

For examination of shrinking by drying in the air, cement prism samples were prepared (4×4×16 cm) with

marks inbuilt according to the HRN B.C8.029 standard [20] that prescribes curing conditions and frequency of measurement. Samples were cured for the first 24 hours in a conditioned chamber (20°C, RH=100 %), while measurement were done in conditions of 20°C and RH ≥ 55 %. Dimension changes of mortar prisms were measured after 3, 7, 14, 28, 60, 90, and 310 days of hydration. The dilatometer Mitutoyo Absolute, Mitutoyo Corp. Japan was used to observe the changes in dimensions of prepared mortar samples, with the precision of measurement of 0.001 mm. Changes in mortar sample dimension due to drying in the air were calculated as the ratio $\Delta l = (l_1 - l_2)/160 \times 100$, where l_1 is the initial dimension of the mortar prism (after 24 hours of hydration) (mm), l_2 is the dimension of the mortar prism (mm) after a specific period of time spent drying.

The beginning and the end of binding of cement paste samples containing different additions of fly ash was determined by the Vicat method according to the HRN B.C8.023:1982 standard [21]. Examinations of mechanical properties (compressive strengths) of mortars were carried out on the Toni Technik modular system of hydraulic presses. The rate of increase of compressive load was 1.50 Nmm⁻²s⁻¹. The content of Portlandite (Ca(OH)₂) and chemically bound water was determined by the Thermal method. The differential thermal analysis (DTA) and thermo gravimetric analysis (TG) were conducted on a Perkin Elmer simultaneous DTA/TG analyser, the Pyris Diamond model, in the temperature range from room temperature until 1000°C with a linear heating rate of 20°C/min in nitrogen atmosphere. The content of the chemically bound water in the system was determined by reducing the overall loss from 105 to 1000°C by the loss of bound water and CO₂ due to decomposition of Ca(OH)₂ and CaCO₃. The content of the chemically bound water and Ca(OH)₂ was corrected to 100% cement mass. XRD powder pattern was recorded on a Philips X'Pert Pro, the PW 3040/60 model with an X-Ray tube PW 3373/00 Cu LFF DK119707 at a current of 40mA and voltage of 40kV, and goniometer PW 3050/60. Recording was made in the range 4° < 2Theta < 64°. The observed phases were indexed by comparison with the distances indicated in the ICDD (International Centre for Diffraction Data) guidelines. The mortar microstructure was examined by Scanning Electron Micro-

Table 2. Composition of the mixture and denotations of samples of prepared cement mortars.

Materials	Samples						
	PB0	PB5	PB15	PB20	PB25	PB30	PB40
Sand /g	1350	1350	1350	1350	1350	1350	1350
Cement /g (c)	450	427.5	382.5	360	337.5	315	270
Fly ash /g (FA)	0	22.5	67.5	90	112.5	135	180
Water /cm ³ (w)	225	223	219	216	215	214	214
w/c	0.5	0.521	0.57	0.6	0.637	0.679	0.79
Broadcast /mm	165	164	164	159	163	157	156
w/(FA+c)	0.5	0.495	0.486	0.48	0.477	0.475	0.475

scopy/Energy Dispersive Spectroscopy (SEM/EDS) on the TESCAN instrument, using the software support VEGA TC. Mortar samples, on which carbon was deposited, were used to examine the microstructure of mortars in conditions of low vacuum. The process of depositing carbon on mortar samples was carried out on the Sputter Coater SCD 050, Bal-Tec instrument, at low vacuum (0.05 bars; 25°C).

RESULTS AND DISCUSSION

Results of examination effect of fly ash on the start and ends of settings of cement type CEM I 42.5 R by using the Vicat instrument are showed in the Table 3 and Figure 2. Results indicate that the increase of the FA addition shifts the start and end of settings to later hydration times and prolongs the setting time in comparison with the reference sample. The FA addition of 20 mass % prolongs the setting time for 26 minutes, while a higher addition of FA (more than 20 mass %) shortens the setting time but it is still longer than setting time measured in the reference sample. In paste preparation it has been observed that the quantity of water needed to obtain normal paste consistency decreases with the increase of the fly ash addition; the water/solid (w/s) ratio ranges from 0.2960 to 0.1896; the water/cement (w/c) ratio increased and ranges from 0.2960 (for the reference sample) to 0.3160 (for the

sample containing 40 mass % FA). This can be attributed to the effect of increased quantity of spherical particles due to the fly ash addition to the mixture [22].

The compressive strength of prepared cement mortars was examined after 3, 7, 14, 28, 60, 90, and 310 days of hydration. The results (Figure 3) show that the developed compressive strengths increase with time for all the samples. The comparison of developed compressive strength in mortar samples containing 5-40 mass % of a fly ash with the reference mortar (without a fly ash addition (PB0)) shows that developed compressive strengths after 60 days of hydration are lower for all mortar samples containing addition of fly ash. It is only after 90 days of hydration when the developed compressive strength for the mortar sample containing 5 mass % fly ash (PB5) becomes higher than strength of the reference sample, which is probably due to the pozzolanic activity of fly ash. Specifically, the pozzolanic reaction between FA and hydration-formed portlandite, $\text{Ca}(\text{OH})_2$ (CH) forms new C-S-H phases, which dominantly determinate the mechanical properties of cement composite materials. Other examined mortar samples (above 5 mass % FA) show the developed compressive strength constantly increase until 90 days of hydration, but the values are still lower than those observed in reference sample. This can be attributed to the effect of dilution, i.e. increased fly ash addition in the systems examined. However, the measurements of developed compressive strengths after 310 days of

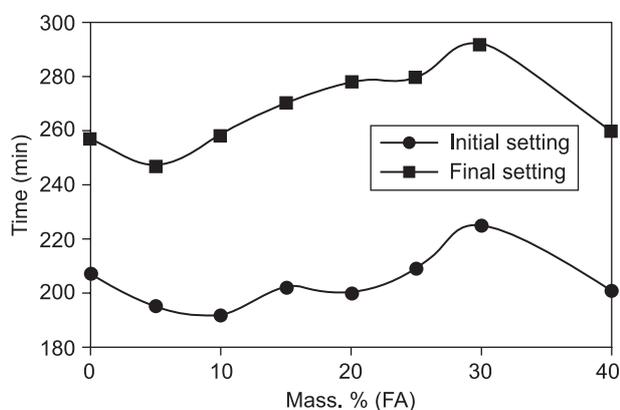


Figure 2. Effect of mass % of fly ash on the initial and final setting.

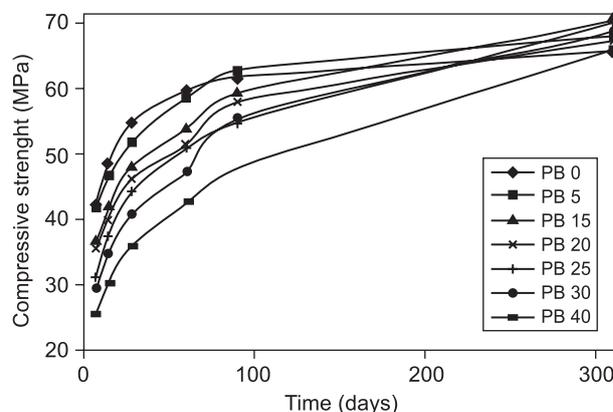


Figure 3. Development of compressive strength in mortars with fly ash replacement addition (0-40 mass %) relative to hydration duration (7-310 days of hydration).

Table 3. Initial and final settings and duration of settings time relative to mass % of fly ash.

Samples	w(FA) (%)	w/s	w/c	Initial settings (min)	Final settings (min)	Duration settings (min)
PB0C	0	0.2960	0.2960	207	257	50
PB5C	5	0.2840	0.2990	195	247	52
PB15C	15	0.2527	0.2973	202	274	72
PB20C	20	0.2403	0.3004	202	278	76
PB25C	25	0.2295	0.3060	211	281	70
PB30C	30	0.2156	0.3080	225	292	67
PB40C	40	0.1896	0.3160	201	270	69

hydration show higher values of developed compressive strength in all mortar samples containing fly ash, added in the quantities from 5-40 mass %, than strength developed in the PB0 reference sample. This observed increase of developed compressive strengths is also due to the pozzolanic reaction between pozzolanically active components of fly ash (amorphous aluminosilicates) and hydration-formed CH, where longer hydration period is obviously needed to eliminate the dilution effect.

The functional correlation between activation index (AI) and compressive strength has been established according to the HRN EN 450:1993 standard, where AI is directly proportional to the developed compressive strength of mortar containing fly ash ($f_c(PBX)$), and inversely proportional to developed compressive strength of the reference mortar ($f_c(PB0)$) at a specific time, $AI = f_c(PBX)/f_c(PB0)$, where $X = 5, 15, 20, 25, 30, 40$. The values obtained for AI indicate the activity of fly ash and its contribution to compressive strengths of cement mortar samples (Figure 4).

Presented results indicate a favourable contribution of FA to the development of compressive strength already after 90 days of hydration ($AI > 100$) in the sample with 5 mass % FA (PB5), while this effect can be observed in other samples only after 310 days of hydration.

The changes of the portlandite content and chemically bound water content in the mortars are presented on the Figures 5 and 6. The changes of hydration-formed portlandite content clearly indicate that its content increases in all mortar samples containing fly ash as cement replacement until 28 days of hydration, and decreases afterwards. These changes in the portlandite content are accompanied with the increase and decrease of chemically bound water content, and indicate that pozzolanic reaction does not take place in this hydration period, yet. The effect of the FA pozzolanic reaction can be observed only after 60 days of hydration in the sample containing 5 mass % of FA (PB5). This sample shows a decrease in the content of hydration-formed portlandite with the simultaneous increase of the content of chemically bounded water, which confirms that the pozzolanic reaction takes place in the sample containing 5 mass % of fly ash (PB5) after 60 days of hydration. In any other sample containing replacement additions of 15-40 mass % FA, this effect can be observed only after 90 days of hydration. These changes, the decrease in the content of hydration-formed portlandite with simultaneous increase of chemically bound water content (due to formation of new quantities of the C-S-H phase), confirms that the pozzolanic reaction takes place in examined mortar samples between pozzolanically active constituents of FA and hydration-formed CH.

In its original form, fly ash contains spherical particles, cenophenes i.e. spherical empty particles (Greek kenos - empty) and plerospheres, i.e. cenophenes filled with several small spherical particles (Greek. pliris - full) [23]. Microphotographs (Figure 7) of mortar samples

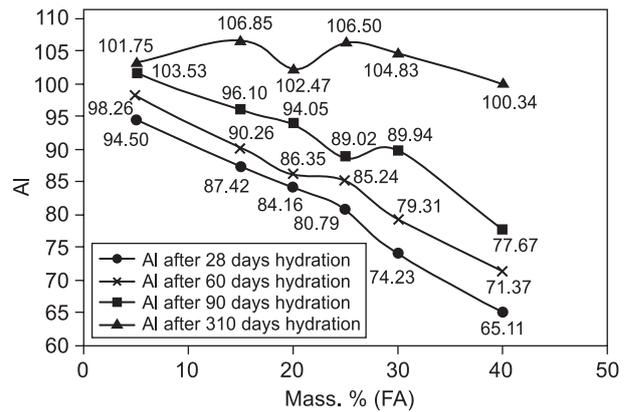


Figure 4. The activation index relative to fly ash replacement addition and hydration time (after 28, 90, and 310 days of hydration).

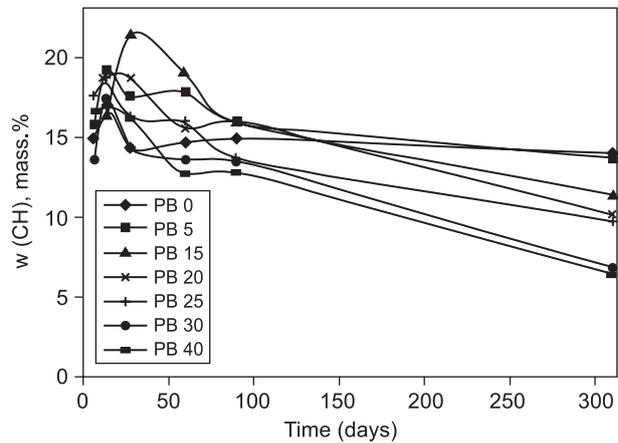


Figure 5. Change in the portlandite content in mortar samples without and with FA replacement addition (5-40 mass %) relative to hydration time (7-310 days of hydration).

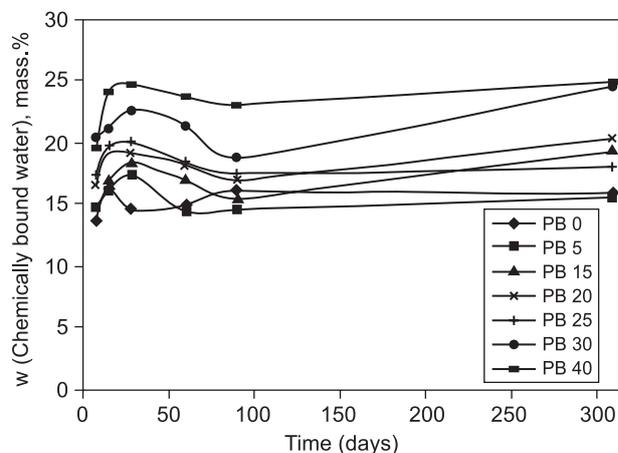
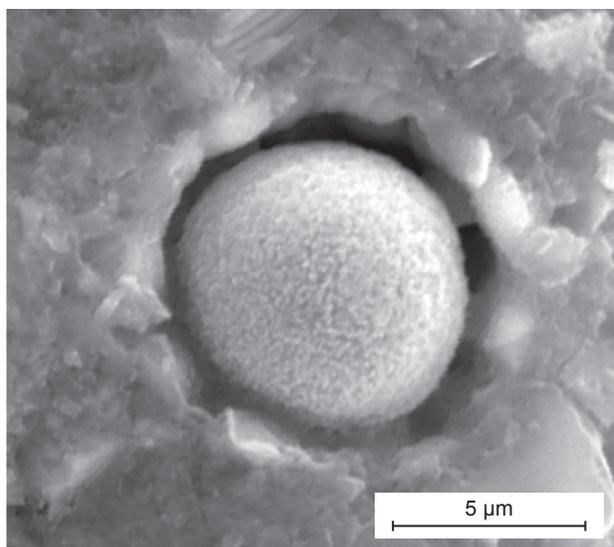


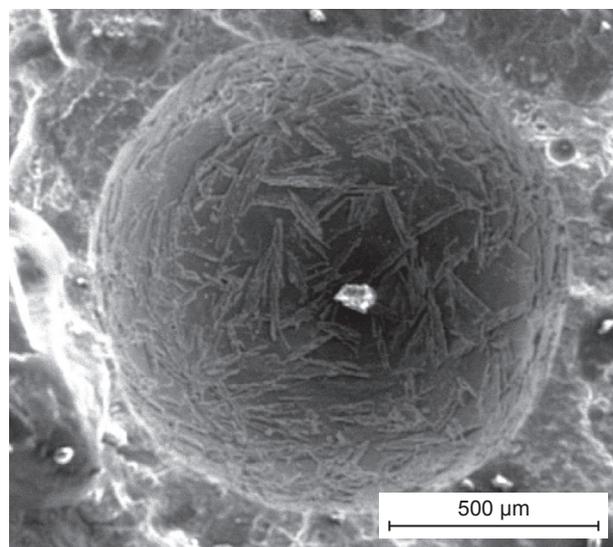
Figure 6. Change in the content of chemically bound water in mortar samples without and with FA replacement addition (5-40 mass %) relative to hydration time (7-310 days of hydration).

containing fly ash replacement addition, obtained by means of scanning electron microscopy (SEM), clearly show the remained spherical FA particles in all mortar samples after 310 days of hydration. The remained fly ash particles seen in microphotographs indicate

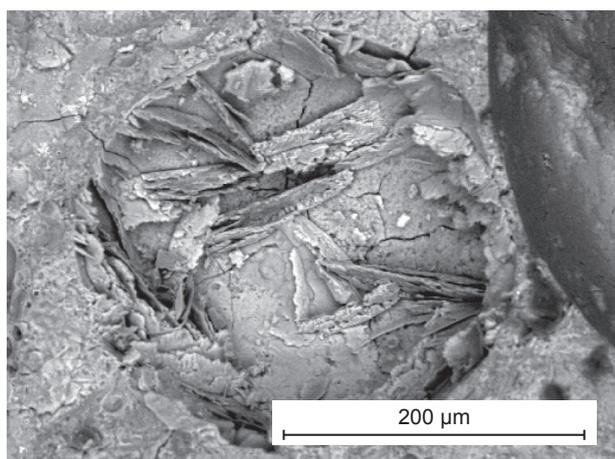
that pozzolanic reaction between fly ash particles and hydration-formed portlandite is incomplete (Figure 7), due to it, additional increase in mechanical properties in later hydration times (after 310 days) can be expected.



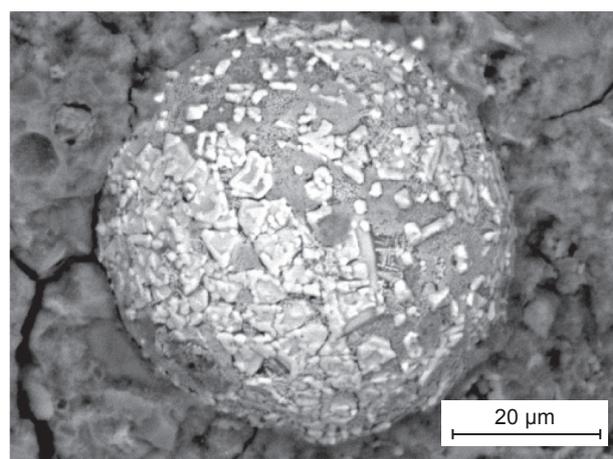
a) FA 5 mass %



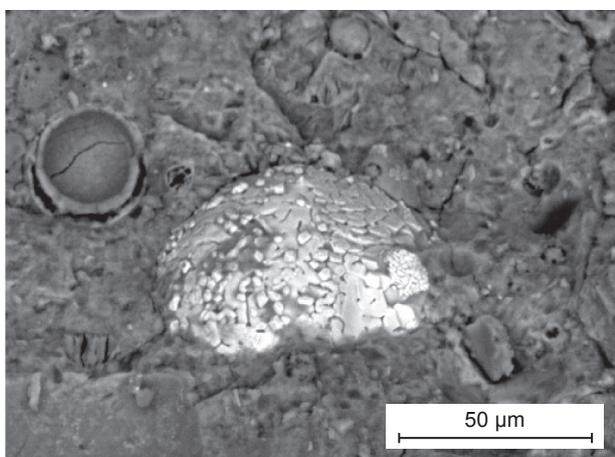
b) FA 15 mass %



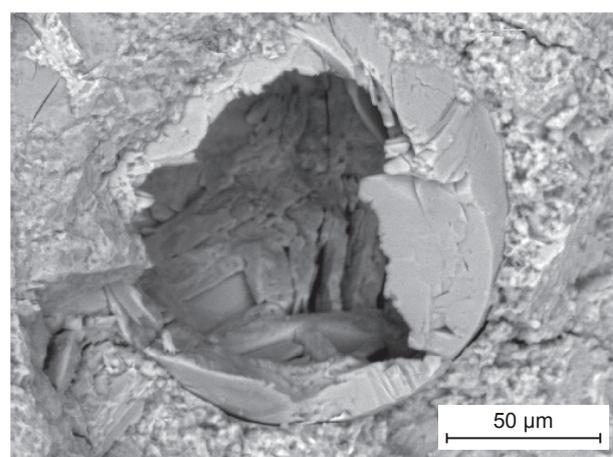
c) FA 20 mass %



d) FA 25 mass %



e) FA 30 mass %



f) FA 40 mass %

Figure 7. SEM images of mortar samples with replacement addition of FA (mass %) after 310 days of hydration.

The mineralogical composition of the mortars after 310 days of hydration has been analyzed by means of the XRD method. Diffraction patterns of the sample without fly ash (PB0) and of samples containing fly ash confirm the presence of the same phases after 310 days of hydration as those present into reference mortar PB0, list of found compounds is show in the Table 4.

Only difference between diffraction pattern of the reference mortar and mortars with higher addition of fly ash is the increase of the diffraction peak intensity which arise from compounds that are already present in the fly ash, such as Quartz, Hematite and Mullite, (Figure 8). Presence of these compounds after 310 days hydration indicates that they are very low reactive compounds and that only amorphous aluminosilicate phase from a fly ash participate in pozzolanic reaction.

The results of dimension changes examination of mortars with and without FA replacement addition due to dry shrinking in the period of 310 days are shows on the Figure 9. The results show that increasing the FA addition reduces shrinking throughout the whole examination period up to 310 days of hydration in comparison with the reference sample (PB0). Shrinking is the lowest in the mortar sample containing 40 mass % of FA replacement addition, dry shrinking of this

sample (PB40) is 15.67 % lower than shrinking observed in the reference mortar (PB0) after 90 days of hydration and 21.38% lower after 310 days of hydration. This is due to smaller amount of water used in cement mortars preparation because of increased fly ash addition, which consequently improves certain engineering properties of such mortars. However, it must be noted that all types

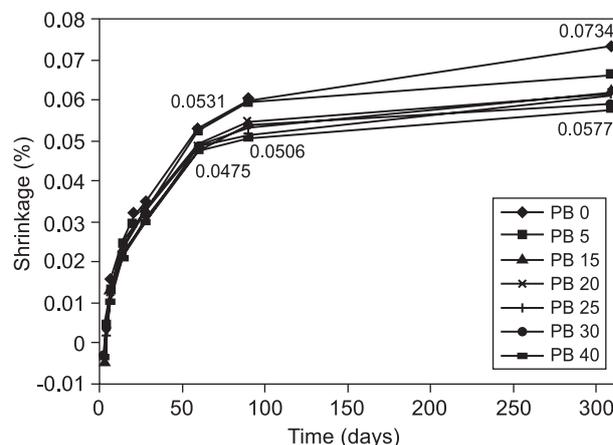


Figure 9. Drying shrinking of mortars containing fly ash replacement addition in the quantity from 0-40 mass %.

Table 4. Crystal phase which are presence in the reference mortars after 310 days, assignation phase by PDF code (Ref. Code), compounds name and chemical formula.

Ref. Code	Compound Name	Chemical Formula	Notation
00-046-1045	Quartz, syn	SiO ₂	Q
00-033-0306	Calcium Silicate Hydrate	Ca _{1.5} SiO _{3.5} ·xH ₂ O	A
00-004-0733	Portlandite, syn	Ca(OH) ₂	CH
00-029-0374	C-S-H (II)	Ca ₂ SiO ₄ ·3H ₂ O	B
00-012-0008	Calcium Aluminum Oxide Hydrate	Ca ₂ Al ₂ O ₅ ·6 H ₂ O	C
00-007-0348	Potassium Calcium Silicate	Ca _{1.917} K _{0.166} SiO ₄	D
00-018-0275	Calcium Aluminum Oxide Sulfate Hydrate	Ca ₄ Al ₂ SO ₁₀ ·12 H ₂ O	E

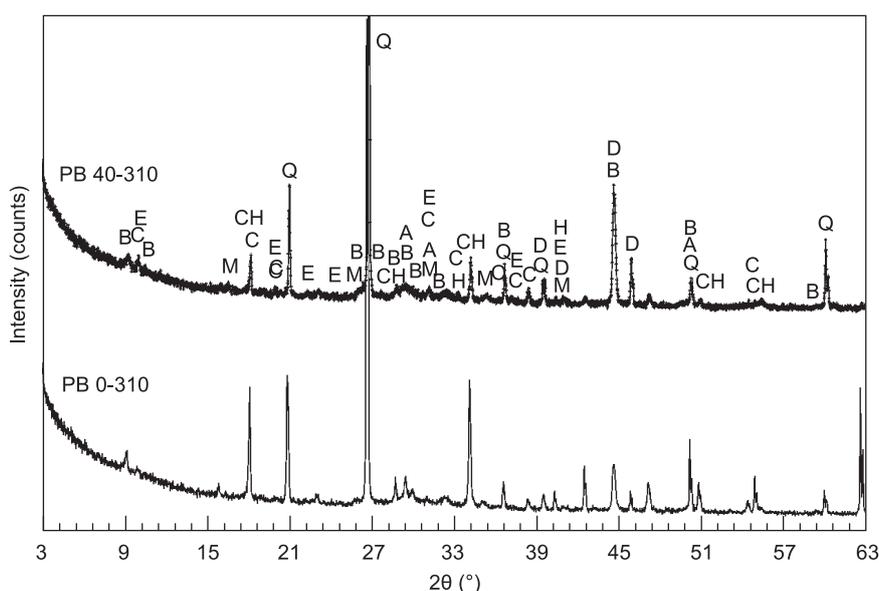


Figure 8. Comparison diffraction pattern for reference mortar (PB0) and mortar PB40 with higher part of fly ash (replacement addition fly ash is 40 mass%) after 310 days of hydration (Q - Quartz, M - Mullite, H - Hematite, CH - portlandite).

of fly ash doesn't cause water reduction, but only those fly ashes that contain spherical, round particles formed as "products" of burning at high temperatures, around 1300°C or higher [24]. Microphotographs of mortars after 310 days of hydration confirm the presence of spherical particles of fly ash that are present in the pure original form of fly ash as raw material for preparation of cement mortars [25].

CONCLUSIONS

Fly ash addition (5-40 mass %) as replacement addition to OPC in the system of cement paste has the following effect: reduction of needed water, shift of the start and the end of setting time towards later hydration times and prolongation of the setting time related to the reference sample (without FA addition). This indicates that FA as replacement addition to cement plays the role as water reducer and cement setting retardant.

In the system of the cement mortars the chemical role of the coarse fly ash as pozzolanically active addition is evident in later hydration phases (after 60 days of hydration for the sample with 5 mass % FA, determined by TG/DTA), when new C-S-H phases are formed by the pozzolanic reaction which then contribute the increase of the final compressive strength. Due to the pozzolanic reaction, mortars with 40 mass % of FA replacement addition after 310 days have higher compressive strength than the reference mortar sample. According to mineralogical mortars composition after 310 days of hydrations indicate that the fly ash addition does not influence on hydration product, namely hydrations products are same in all samples with or without added fly ash. Comparison of XRD powder pattern of the samples with and without addition fly ash (PB40, PB0) after 310 days of hydration show that the crystal phases are still rest liberated in the mortars with addition fly ash (PB40), as they were in original fly ash. This fact indicates that the crystals phase which arises from the fly ash (quartz, mullite and hematite) does not contribute pozzolanic reaction; just the amorphous (or metastable) aluminosilicates phase contributes the pozzolanic reaction. The increased fly ash addition significantly affects improvement in drying shrinkage and it directly indicates a reduced possibility of cracks formation in the cement composite microstructure, which should greatly affect resistance and durability of cement composite materials.

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