

THE EFFECT OF FLY ASH ON CEMENT HYDRATION IN AQUEOUS SUSPENSIONS

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Fly ash is waste material formed in coal-using thermal-power plants, and is frequently used as addition to cement and mortars/concrete. Its physical and chemical properties depend exclusively on the quality of coal used and on technological conditions of burning. This study has examined the effect of fly ash from the Plomin 2 Thermal Power Plant, Croatian Power Supply (HEP), Plomin, Croatia, on early stages of cement hydration in aqueous suspensions ($w/s = 4$, $t = 20^\circ\text{C}$). According to the ASTM C618 Standards, fly ash used, belongs to the F class fly ash, i.e. to low CaO content fly ash. Fly ash was added as a replacement for cement in amounts of 0, 5, 11, 15, 20, and 30 wt.%. Conductometrical measurements in suspensions have indicated that the replacement postpones the start of precipitation of $\text{Ca}(\text{OH})_2$, prolongs the duration of that precipitation, and reduces the overall cement hydration rate. XRD and DTA-TG/DTG measurements indicate that the share of $\text{Ca}(\text{OH})_2$ in the solid part of the suspension decreases when the fly ash addition increases, while the results of the DTA-TG/DTG analysis corrected to the cement weight confirm the role of fly ash as nucleation site where hydration products settle, while its pozzolanic activity has not been observed in this period of hydration up to 72 hours.

INTRODUCTION

Various types of additions that improve cement properties have been used in the cement industry for a long time. The most frequently used materials are blast furnace slag, silica fume, fly ash or natural pozzolans such as burned clay, metakaolinite [1]. Fly ash is waste materials from the thermal power plant; it is separated from the flue gas of the power station burning pulverized coal [2]. Factors such as the origin of the coal and the burning condition, strongly affect their chemical and mineralogical composition. According to ASTM C618, two classes of the fly ash are specified Class F and C. Class F fly ash is produced by burning anthracite or bituminous coal and it is known like fly ash with low content CaO. Class C is produced by burning sub-bituminous or lignite coal and Class C is known like fly ash with high content CaO [3,4]. Fly ash is added to Portland cement or directly to mortars or concretes [5]. The use of fly ash as a replacement addition to cement in production is useful for a number of purposes: reduction of CO_2 emission, reduction of power needed for cement production, reduction in the quantity of waste material and protection of natural raw materials. There are numerous studies on using the fly ash as addition to mortars/concrete and it is widely accepted that they affect fluidity, reduce the hydration heat, and block the alkali-silicate reactions [6]. The effect of fly ash on the hydration rate and on final properties of hardened cement composites depends on the chemical composi-

tion, specific surface and chemical reactivity (the content of the amorphous phase) of fly ash in cement systems [7,8].

This study has examined the physical and chemical properties of fly ash and its effect on hydration of cement containing a replacement part of fly ash in aqueous suspension systems.

EXPERIMENTAL

The materials used in the experimental work were fly ash from the thermal power plant Plomin-2 (HEP-Plomin, Croatia) and the industrial Portland cement type CEM IIA-S 42.5N (Dalmacija cement, Croatia). Table 1 shows the chemical composition of fly ash and cement used, and the properties of class F fly ash according to the ASTM C618 Standards.

Aqueous suspensions have been prepared with the replacement part of fly ash in the amounts of 0, 5, 11, 15, 20, 30 wt.% (samples designated PF0, PF5, PF11, PF15, PF20, and PF30, respectively) with the water/solid ratio ($w/s = 4$) at the hydration temperature of 20°C . The cement - fly ash mixture was homogenized in mills with ceramic spheres, the homogenization process lasting for 30 minutes. After adding the homogenized mixture to the thermostated water, the system was left to mix on the magnetic mixer for 5 minutes, after which a conductometrical cell was immersed to trace the changes in conductivity (Schott LF 413-3T).

The Schott CG-853 conductometer was connected to a computer via an RS232 output, and data on conductivity were collected every 5 seconds. The conductivity of the suspensions was monitored continuously up to approx 48 hours, i.e. as long as there were noticeable changes in conductivity.

In order to determine the concentration of Ca²⁺ ions present in the aqueous phase of the suspension and the quantity of Ca(OH)₂ formed in the solid part of the suspension, hydration was interrupted at precisely defined time intervals, at 0.08, 0.25, 0.75, 1, 3, 7, 12, 13,16, 17, 24, 48, and 72 h. Separation solid and liquid phase was made by system vacuum filtration, solid part of the suspension is immediately mixed with acetone in order to stop the hydration and remove free water, after removing free water samples are dried in vacuum at room temperature and placed in desiccator. In liquid phase concentration Ca²⁺ ions is determined by using complexometric methods with EDTA.

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 weight. The fineness of fly ash was expressed as the weight proportion in % of the fly ash retained when sieved on a 0.045 mm mesh sieve, according to Standards EN 450:1994. Differential thermal analysis (DTA) and thermogravimetric analysis (TG) were conducted on a Perkin Elmer simultaneous DTA/TG analyzer, the Pyris Diamond model, in the temperature range from the room temperature until 1000°C with a linear heating rate of 20°C/min in the nitrogen atmosphere. 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 40 mA and voltage of 40 kV, and goniometer

PW 3050/60. Recording was made in the range 4° < 2θ < 64°. The FTIR spectrum was made in KBr pastile on the Perkin Elmer spectrophotometer, the Spectrum One model, in the wave numbers range from 4000-400 cm⁻¹.

RESULTS AND DISCUSSION

Properties of fly ash

Fly ash consists mainly of SiO₂, Al₂O₃, Fe₂O₃ and CaO components, with the (SiO₂ + Al₂O₃ + Fe₂O₃) content of more than 70 wt.%, and with the CaO content of 2.81 wt.%. The data obtained by chemical analysis place the fly ash in the F class, i.e. the low CaO content fly ash. The weight loss (in air atmosphere and in range 30-1000°C) value of 1.60 wt.% reflects the weight of unburned carbon, sulfide, hydrate, carbonate and oxide that is present in the fly ash. Thermal analyses of the samples indicate the continuous weight loss in the range from 30-1000°C. According to the literature data in the temperature range between 500-750°C, in the inert atmosphere, degradation of carbonate can take place, just one process influence on the weight loss. In the same temperature range but in the oxidizing atmosphere unburned carbon can oxidize into carbon dioxide at the same time with the degradation carbonate, both process influences on the weight loss [9]. Figure 1 presents the DTA-TG/DTG measurements of fly ash in the two different atmospheres (air and nitrogen) with the heating rate 5°C/minutes. The quantity of unburned carbon in the fly ash has been determined. Combination heating in the limited temperature range from 438-714°C, both in oxidizing and inert atmospheres, makes it possible to resolve the unburned carbon and carbonate in the fly ash samples. From the difference of weight loss in the oxidizing and nitrogen atmosphere is calculated quantity of carbon which is presence in the fly ash (0.6 wt.%). Table 1 presents the results obtained.

Table 1. Major elements found in fly ash and the composition required by ASTM C618 for class F Fly ash, and composition of the CEM IIA-S 42.5N cement used.

Materials	Cement (wt.%)	Fly ash (wt.%)	Required by Standards ASTM C618
SiO ₂	21.55	59.46	(SiO ₂ +Al ₂ O ₃ +Fe ₂ O ₃)=Min.70%
Al ₂ O ₃	5.96	19.91	
Fe ₂ O ₃	2.61	9.93	
CaO	59.95	2.81	-
MgO	2.84	0.84	5.0 max.
SO ₃	2.83	0.18	5.0 max.
Na ₂ O	0.22	0.40	-
K ₂ O	0.77	1.27	-
C	-	0.60	-
Loss of ignition (1000°C)	2.8	1.6	6.0 max.
Moisture Content	0.2	0.1	3.0 max.
Specific gravity (kg/m ³)	2.501	1.839	-
Specific surface (Blain) (m ² /kg)	409.1	312.9	-
Normal Consistency (%)	28	-	-
Initial settings	2h 26min	-	-
Final settings	3h 15min	-	-

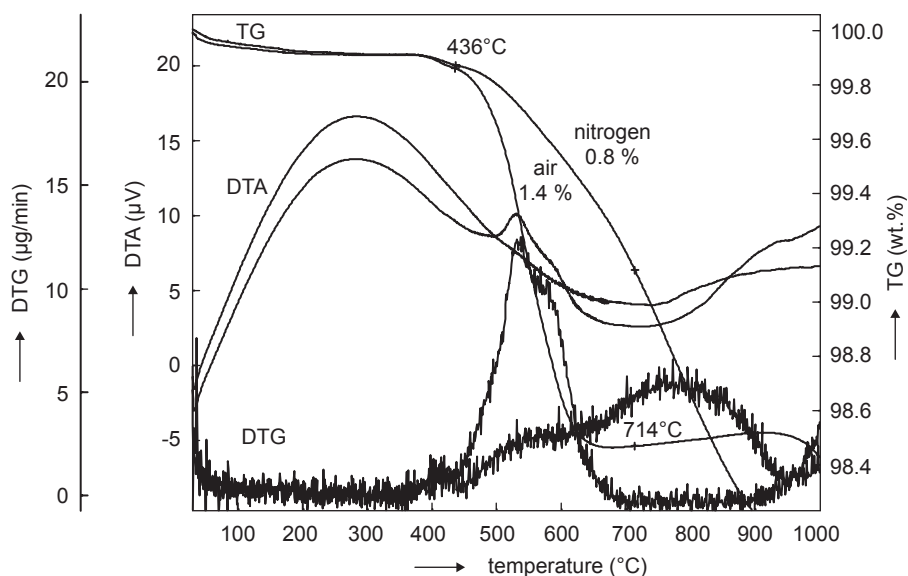


Figure 1. TG/DTA of fly ash sample with 5°C/min heating rate.

Fly ash is generally found in small spherical particles that differ significantly in size and shape (figure 2). The specific gravity of fly ash used is about 1.839 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 meets the criteria for application in production of cement/concrete required by the EN 450:1994 norm.

Figure 3 shows the XRD powder patterns of the fly ash un-fractionated (a), retained on a 0.045 mm sieve (b), and a fraction passed through a 0.045 mm sieve (c).

Qualitative analyses of the different fractions of fly ash samples were made by comparing experimental diffraction data with the diffraction data for known com-

pounds stored in the JCPDS database [10]. Diffraction powder patterns for all fractions are same, without any difference. Two diffraction maximums at $2\theta = 44.9^\circ$ and 38.3° arise from the samples holder, not from sample fly ash. Other diffraction maximums which arise from the fly ash are analyses, results is the three main crystal phases were found: mullite (PDF # 15-0776), quartz (PDF # 12-0708) and hematite (PDF # 24-0072). The diffuse band are present in the region $15^\circ < 2\theta < 30^\circ$ and it show on presence the amorphous (glass) phase. FT-IR spectrums of fly ash samples are shown in figure 4, detecting the presence of aluminosilicate with a little amount of adsorbed water.

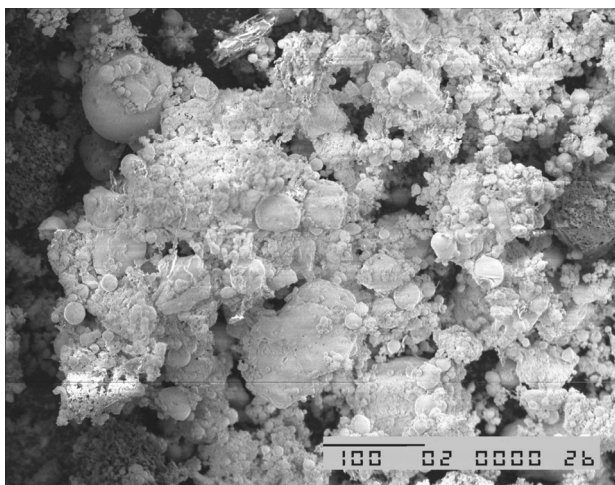


Figure 2. SEM micrograph used fly ash.

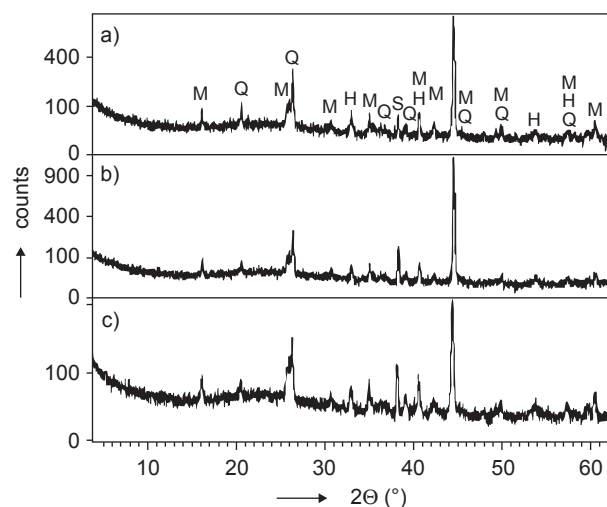


Figure 3. XRD diagram of different fractions of fly ash sample; a) un-fractionated sample, b) retained on a 0.045 mm sieve and c) fraction passed through a 0.045 mm-sieve; M - mullite, Q - quartz, H - hematite, S- sample holder.

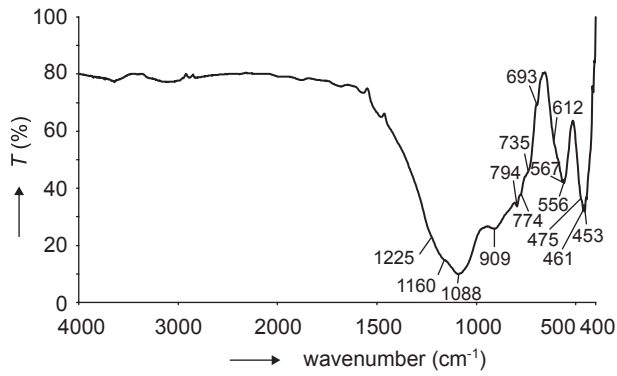


Figure 4. FT-IR spectrum of fly ash sample.

The main and very broad absorption zone appears centered at 1088 cm^{-1} attributed to asymmetric stretching of O–Si–O and O–Al–O bonds, the bands at 567 and 556 cm^{-1} are attributed to the bending Al–O–Si and Si–O–Si bonds, the bands at 461 cm^{-1} attribute on bending O–Si–O bonds. Assigned strong and very strong absorption bands at 1160, 1088 and 794 cm^{-1} attribute on the presence quartz, shoulder 612 cm^{-1} and strong absorption bands at 567 cm^{-1} attribute on the mullite phase but the one absorption bands characteristic for mullite phase at 1138 cm^{-1} is hid with very broad band

centered at 1088 cm^{-1} . Absorption bands at 1088, 612, 556, 475 cm^{-1} attribute on the presence hematite phase in the sample. Broad absorption band in the area 500–650 cm^{-1} indicate on the silicate and aluminosilicate glasses phase [11–14].

Conductivity in suspensions

Measurements of conductivity in cement suspensions, pastes, and mortars are very suitable for monitoring the progress of hydration. A change in conductivity in cement suspensions relative to hydration duration has been divided into three hydration phases according to Maximilen: a) mixing, b) dormant and c) acceleration period [15].

When non-hydrated cement particles get in contact with water, various simultaneous hydration processes take place on clinker materials contained in cement, and various alkali oxides and sulfates contained in cement dissolve completely, which results in the increase of cations and anions present in the aqueous suspension, such as Ca^{2+} , K^+ , Na^+ , SO_4^{2-} , OH^- . The first conductometric maximum is determined by the number and mobility of ions present [16]. C–S–H and the ettringite phase, as hydration products on the surface of non-

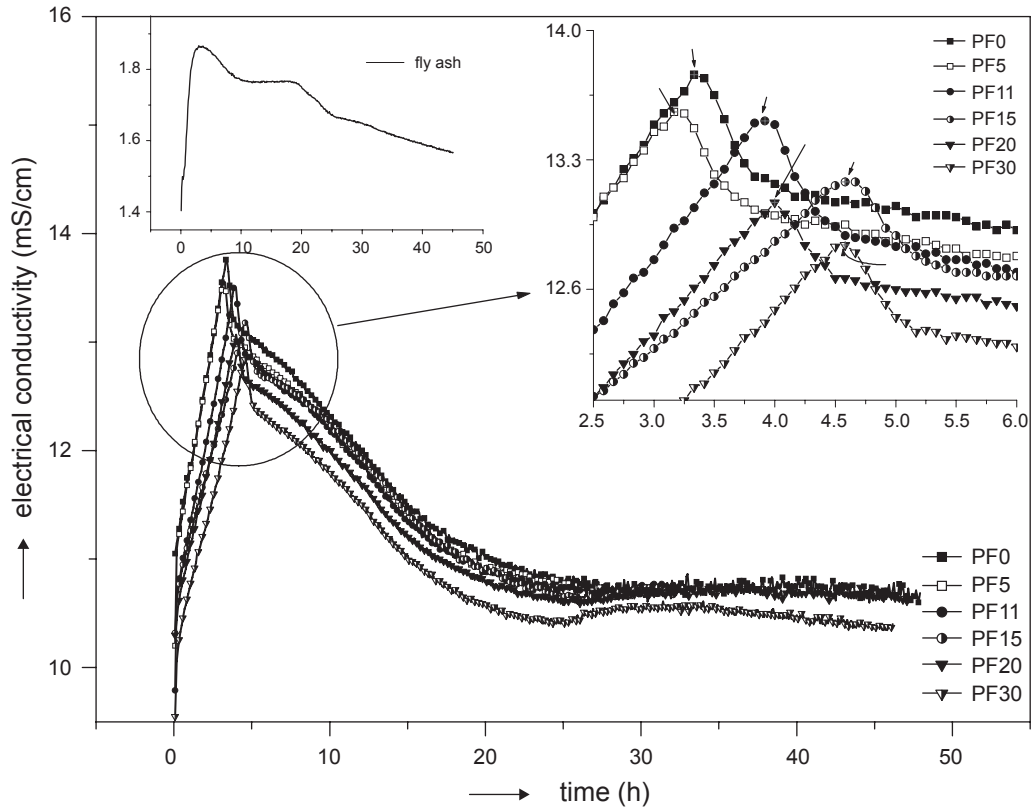


Figure 5. Change in electrical conductivity in aqueous suspension ($w/s = 4$) for sample PFO, PF5, PF11, PF15, PF20 and PF30 relative to hydration duration.

hydrated cement grain, are known to be poorly conductive phases that upon formation lead to a drop in conductivity (phase b), but as the CH nucleation process is rather slow, the concentration of Ca^{2+} and OH^- ions increases in the solution which in turn increases conductivity until supersaturation which reaches its maximum there. The curves of changes in conductivity in the fly ash - cement aqueous suspension systems, shown in figure 5, indicate a conductivity maximum in the interval between 3.3 and 4.6 hours of hydration. Addition of fly ash to the system postpones the maximum of conductivity, which decreases with the increase of the addition (table 2). The change of the slope of the conductivity change line relative to hydration duration in phase b reflects the effect of the addition on the cement hydration rate: the lower the line slope, the lower the hydration rate [17]. The results indicate that the addition of fly ash slows down cement hydration. However, when the solubility product constants are reached, K_{sp} [$\text{Ca}(\text{OH})_2$], portlandite suddenly precipitates which is manifested by a sudden drop in the conductivity of the system [15,16]. The precipitation of $\text{Ca}(\text{OH})_2$ takes 26 to 54 minutes, followed by a slower drop in conductivity, which suggests further progress of hydration in the system. This is probably due to breaking of the membrane of hydration products formed around the non-

hydrated cement grain (due to the action of the osmotic pressure) and to transformation of ettringite into monosulfate with constant separation of portlandite and continuous drop in conductivity until approx. 24 hours of hydration, after which conductivity becomes balanced and hydration processes are strictly limited to diffusion processes of hydration [18,19]. In systems with addition of 20 or 30 wt.% of fly ash, a mild increase of conductivity in the system is encountered again after 24 hours of mixing.

Figure 6 shows that the change in concentration of Ca^{2+} ions in the aqueous suspension phase, expressed as the change in CaO concentration in the systems, follows the change in conductivity in the suspensions (figure 5).

The maximum concentration of Ca^{2+} ions present in the liquid phase is achieved after 3 hours of hydration, followed by a continuous drop in concentration which becomes slower after 24 hours of hydration, and concentration continues to decrease until 72 hours of hydration. In systems with increased addition of fly ash (15, 20, and 30 wt.%), there occurs an increase in the concentration of Ca^{2+} ions in the period of hydration after 24 hours, which confirms the results obtained by conductometric measurements, indicating that hydration continues and more active in the systems with addition fly ash.

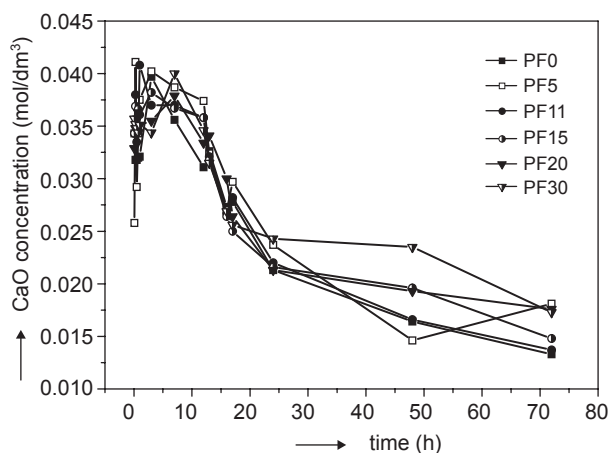


Figure 6. Change in Ca^{2+} ion concentration (expressed as CaO) in the cement suspension solution relative to hydration duration.

XRD analysis of the solid residue

The mineralogical composition of the solid hydration residue has been analyzed by means of the XRD method, as shown in figure 7. Diffraction patterns of a sample without fly ash (PF0) and of a sample containing fly ash (PF30) confirm the presence of the same phases after 24 hours of hydration. The major crystalline phases present in the system are: tri-calcium silicate (C_3S) (PDF # 49-0442), di-calcium silicate (C_2S) (PDF # 33-0302), tri-calcium aluminate (C_3A) (PDF # 33-0251), calcium hydroxide ($\text{Ca}(\text{OH})_2$) (PDF # 04-0733), calcium carbonate (CaCO_3) (PDF # 05-0586), ettringite (PDF # 72-0646), and brownmillerite (C_4AF) (PDF # 30-0226). If the share of fly ash is higher than 11 wt.%, the diffraction patterns show diffraction maximums that can be attributed to quartz (PDF # 70-2517) and mulite (PDF # 15-0776) and which are due to the fly ash addi-

Table 2. Results obtained with the conductometric measures.

Sample	Initial conductivity (mS/cm)	Maximum conductivity (mS/cm)	Duration of max. conductivity (h)	Duration of precipitation $\text{Ca}(\text{OH})_2$ (min)
PF0	11.15	13,76	3.33	29
PF5	11.09	13.56	3.17	26
PF11	11.66	13.51	3.92	40
PF15	10.62	13.18	4.00	33
PF20	10.44	13.07	4.58	54
PF30	10.06	12.84	4.58	54

tion: this is best shown in the PF30 sample as in figure 7. The presence of the amorphous C-S-H gel has been observed in all the system, indicated by the presence of a wide diffusion diffraction maximum in the angular range of 20-42° 2θ. The crystal form of gypsum has not been found in the system as it has probably been consumed in formation of ettringite in the early hydration phase.

The comparison of integrated intensities belonging to the Ca(OH)₂ phase in the angular range where there is no overlapping with other phases, i.e. in the angular ranges from 17-19° 2θ and 46-48° 2θ, (figure 8) indicates that the content of Ca(OH)₂ formed by hydration decreases with the increase of the fly ash addition in the mineral mixture.

Diffraction patterns of polycrystalline samples indicate that the largest share of the crystal Ca(OH)₂ phase is observed in samples without fly ash (PF0) for all hydration durations observed (until 72 hours).

DTA-TG/DTG analysis was used to quantify the Ca(OH)₂ formed in the solid part of the suspension blocked by hydration. Hydration in aqueous suspensions was interrupted at precisely defined hydration times (5 minutes - 72h).

The Ca(OH)₂ decomposition takes place in the temperature interval from 400-550°C (figure 9). The values obtained for the Ca(OH)₂ content in samples with the

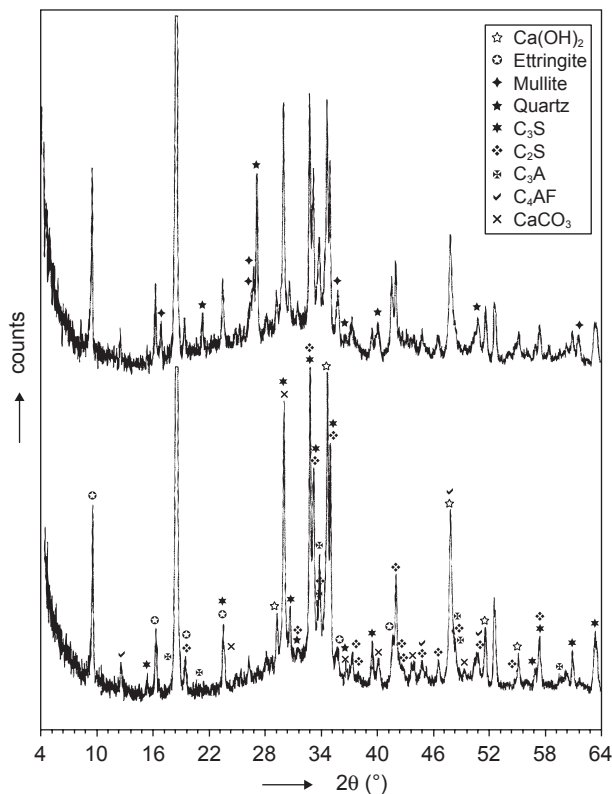


Figure 7. XRD patterns of PF0 and PF30 samples after 24 hours of hydration.

addition of fly ash have been corrected to 100 % cement weight. Figure 10 presents the results for the change in Ca(OH)₂ content in samples examined during hydration from 5 minutes to 72 hours, corrected to 100 % cement weight.

The results obtained indicate that in the cement suspension systems Ca(OH)₂ precipitates much sooner than assumed based on conductometric measurements. DTA-TG/DTG measurements have indicated the presence of Ca(OH)₂ even in systems 5 minutes old. The DTA signal and the weight loss in the temperature interval from 330-460°C indicate an endothermic change that may be attributed to the Ca(OH)₂ decomposition, but which cannot be confirmed by XRD because of a very high share of amorphous and crystal phase in the system and a very small share and low degree of order in the crystal Ca(OH)₂ phase in that hydration period. A sudden increase in the Ca(OH)₂ content occurs in the period between 3 and 7 hours of hydration. In the period between 7 and 13 hours of hydration, the Ca(OH)₂ content is observed to be almost constant [20], followed

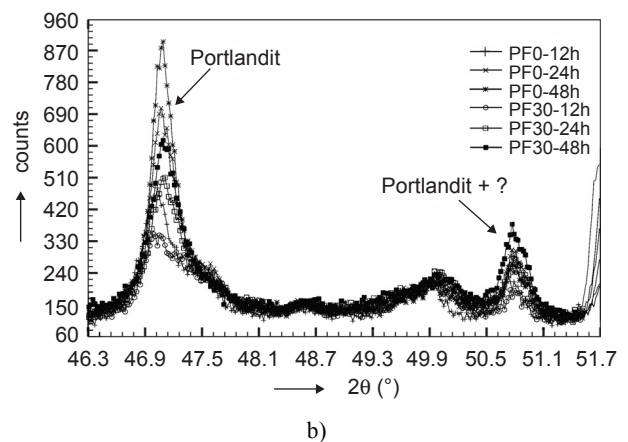
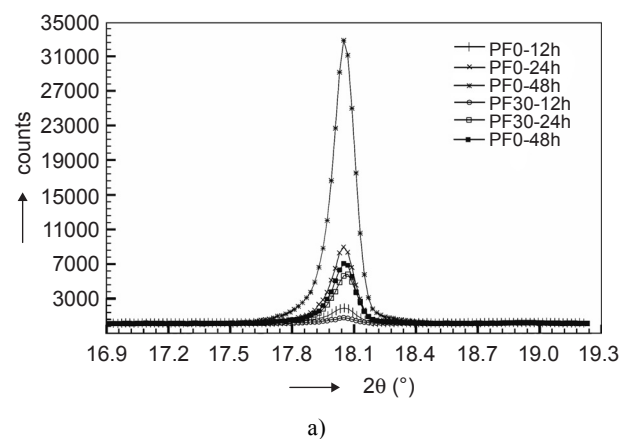


Figure 8. XRD of PF0 and PF30 samples after 12, 24, and 48 hours of hydration: a) 2θ from 17.4° to 18.8°; b) 2θ from 46.8° to 48.1°.

by a further increase in the $\text{Ca}(\text{OH})_2$ content in the solid suspension residue. If the content of fly ash in the systems is compared to $\text{Ca}(\text{OH})_2$ formed, it can be seen that after 48 hours of duration the $\text{Ca}(\text{OH})_2$ content is lowest in systems without fly ash addition. The $\text{Ca}(\text{OH})_2$ content in systems with addition of more than 5 wt.% of fly ash is higher than that in the cement without fly ash addition (the PF0 sample) already after 24 hours of hydration.

The values obtained for the content of chemically bounded water in the solid suspension residue have been calculated to 100 % cement weight and shown in figure 11. There is a continuous increase of chemically bounded water in all systems in the whole measuring range. The increase of bound water is observed in the

period after 24 hours in the systems containing the fly ash addition in comparison with the system without the fly ash addition. This indicates the increase in hydration products. As the share of chemically bound water increases and there is a simultaneous increase of the $\text{Ca}(\text{OH})_2$ content in systems containing the fly ash addition, it may be assumed that fly ash increases the cement hydration degree. This indicates that the addition of fly ash plays a very important role as nucleation site where hydration products precipitate, which accelerates the hydration reaction. As no drop in the $\text{Ca}(\text{OH})_2$ content has been observed in samples containing the fly ash addition, the pozzolanic reaction may be assumed to take place later in comparison with the cement hydration reaction.

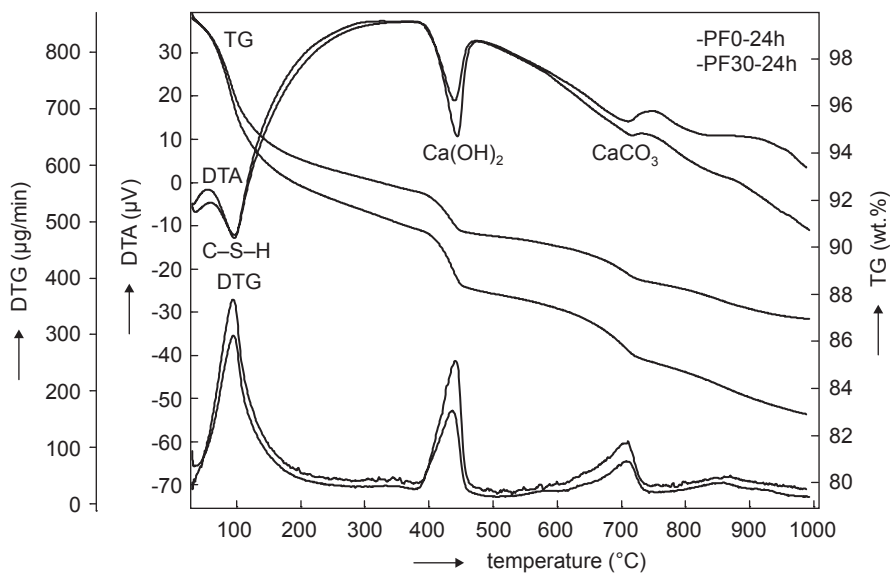


Figure 9. DTA-TG/DTG thermal decomposition of hydration products in the PF0-24h and PF30-24 h samples.

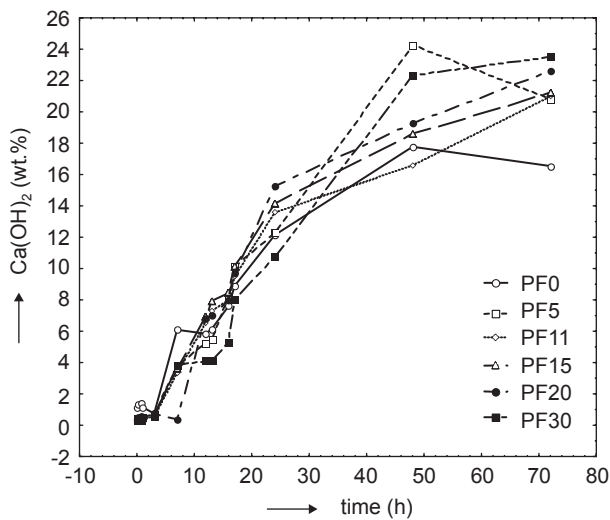


Figure 10. Change in the content during 72-hours' hydration (wt.%) $\text{Ca}(\text{OH})_2$ in solid hydrated residue after filtering and stopping of further hydration.

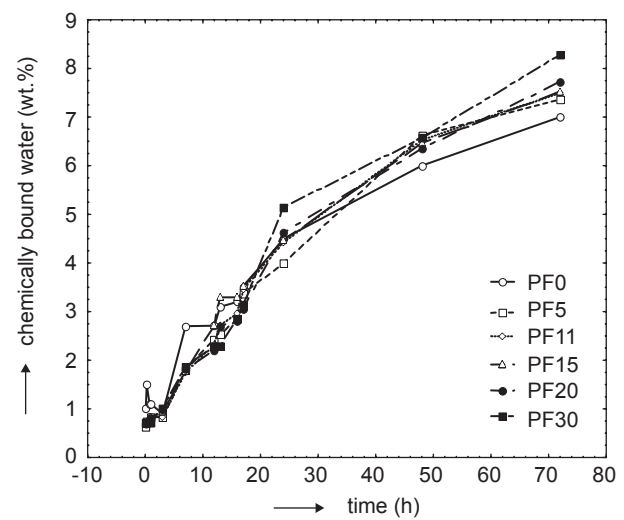


Figure 11. The content the chemically bound water in the PF0, PF5, PF11, PF15, PF20 and PF30 samples.

CONCLUSION

The fly ash from TE Plomin-2 belongs to the low CaO content fly ash group by its physical and chemical properties and according to the ASTM C618 Standards, which places it in the F class and makes it applicable as addition to cement/concrete according to the EN 450:1994 Standards. The results of examination of aqueous suspensions ($w/s = 4$) of the cement-fly ash system indicate that the replacement part of fly ash from 5 to 30 wt.% slows down the hydration rate, increases the cement hydration degree, postpones the beginning of Ca(OH)_2 precipitation by as much as 78 minutes, and increases the time of sudden precipitation by 28 minutes in comparison with the cement without the fly ash addition. For all hydration times, the Ca(OH)_2 content (without correction to 100 % cement weight) is highest in samples without the addition of fly ash, which leads to the conclusion that the addition of fly ash reduces the content of free chemically reactive Ca(OH)_2 . The results of the DTA-TG/DTG analysis confirm XRD measurements. However, after correction to 100 % cement weight, the results obtained indicate that the hydration degree is higher in the systems containing the fly ash addition than in the pure cement, which can be seen from the content of Ca(OH)_2 and the chemically bound water. Continuous increase in the content of chemically bound water and Ca(OH)_2 indicates that the pozzolanic reaction is postponed in the cement-fly ash aqueous suspension systems until 72 hours of hydration in comparison with cement hydration reactions.

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VLIV POPÍLKU NA HYDRATACI CEMENTU VE VODNÝCH SUSPENZÍCH

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Popílek je odpadní materiál vznikající při spalování uhlí v tepelných elektrárnách a je hojně používán jako přísada do cementu, malty či betonu. Jeho fyzikální a chemické vlastnosti závisejí výhradně na kvalitě použitého uhlí a na technologických podmínkách spalování. Tato studie zkoumala popílek z tepelné elektrárny Plomin 2, Chorvatské energetické závody (HEP), Plomin, Chorvatsko v počátečních fázích hydratace cementu ve vodných suspenzích ($w/s = 4$, $t = 20^\circ\text{C}$). Podle normy ASTM C618 odpovídá použitý popílek třídě F, tj. popílkům s nízkým obsahem CaO. Popílek byl přidáván jako náhrada cementu v množstvích 0, 5, 11, 15, 20 a 30 hmotn. %. Konduktometrická měření suspenzí ukázala, že náhrada odsunuje počátek srážení Ca(OH)_2 , prodlužuje trvání tohoto srážení a snižuje celkovou rychlost hydratace cementu. XRD a DTA-TG/DTG měření ukazují, že podíl Ca(OH)_2 v pevné části suspenze se zvyšujícím se přidavkem popílků klesá, zatímco výsledky DTA-TG/DTG analýz korigované na hmotnost cementu potvrzují funkci popílků jako nukleačních zárodků v místě usazování produktů hydratace, zatímco jeho pucolánová aktivita v této fázi hydratace (do 72 hodin) nebyla pozorována.