

Influence of Cements Containing Calcareous Fly Ash as a Main Component Properties of Fresh Cement Mixtures

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Abstract. The main goal of presented research was to examine usability of cements containing calcareous fly ash (W) from technological point of view. In the paper the results of tests concerning the influence of CEM II and CEM IV cements containing fly ash (W) on rheological properties, air content, setting times and plastic shrinkage of mortars are presented and discussed. Moreover, compatibility of plasticizers with cements containing fly ash (W) was also studied. Additionally, setting time and hydration heat of cements containing calcareous fly ash (W) were determined. In a broader aspect, the research contributes to promulgation of the possibility of using calcareous fly ash (W) in cement and concrete technology, what greatly benefits the environment protection (utilization of waste fly ash). Calcareous fly ash can be used successfully as the main component of cement. Cements produced by blending with processed fly ash or cements produced by interginding are characterized by acceptable technological properties. In respect to CEM I cements, cements containing calcareous fly ash worsen workability, decrease air content, delay setting time of mixtures. Cements with calcareous fly ash show good compatibility with plasticizers.

1. Introduction

Calcareous fly ash (W) is produced as a result of burning brown coal in conventional furnaces in large amount – in Poland, about 5 million Mg of fly ash W is produced each year [1]. One of the largest manufacturers of fly ash (W) is Bełchatów power plant. Fly ashes (W) are characterized by both pozzolanic and hydraulic activities [1-5]. The main mineral components are: quartz, gahlenite, anorthite, anhydrite and calcium oxide, typical cement clinker phases, i.e. C_2S , $C_{12}A_7$, C_4AF , C_4A_3S were also identified - these phases determine hydraulic properties of fly ashes (W) [1-5]. The pozzolanic activity is determined by the presence of reactive silicon dioxide (SiO_2) and alumina (Al_2O_3) [1-5]. Studies and analyses concerning the use of fly ashes (W) proved that quality requirements of EN 197-1 are met and it is possible to use them as a main cement constituent [2,3,5]. It has been proven that the presence of fly ashes (W) in cement usually does not adversely affect the mechanical properties and durability of hardened concrete and sometimes even improves them [6-11].

Calcareous fly ashes (W) from Bełchatów power plant demonstrate high sieve residue (0.045 mm) what results in significant water demand and problems with concrete workability control [2,3]. Workability loss of fresh concrete is directly proportional to the amount of fly ash (W) added [12-17] and increases with sieve residue (0.045 mm) increase [4]. Negative influence of fly ash (W) on workability of fresh concrete is the problem which considerably reduces the attractiveness of fly ash (W) use in concrete technology [3]. It was shown in [2-4, 16, 18] that negative influence of fly ash (W)



on the workability may be reduced by processing it by grinding, blending or separation. Favoured solution is the production of cement with fly ash (W) as a main constituent - grinding is a routine technological process in cement production [2, 3].

The aim of research was to examine properties of cements containing calcareous fly ash (W) from technological point of view. In the paper, the results of tests concerning the influence of presence and content of fly ash (W) in CEM II and CEM IV cements produced using different methods on rheological properties, air content, setting times and plastic shrinkage of mortars are presented. Moreover, compatibility of plasticizers with CEM II/B-W cements was studied. In a broader aspect, the aim of research is to popularize use of calcareous fly ash (W) in cement and concrete technology, what greatly benefits the environment protection.

2. Experimental

2.1. Research plan and variables

Research plan is shown in table 1. As variable factors in research were adopted:

- Calcareous fly ash (W) content in cement (CEM II/A-W, CEM II/B-W, CEM IV/B-W),
- Method of cement production: by intergrinding of constituents (clinker, fly ash (W), gypsum) in a laboratory ball-mill or by homogenization of earlier prepared materials (CEM I 42,5R, raw or ground fly ash (W)) in a blender,
- Processing of fly ash (W) (in the case of cement produced by homogenization) - raw or ground fly ash (W).

Table 1. Experimental plan.

Fly ash W content, %	Method of cement production		
	Blended cement		Interground cement
	fly ash W raw	fly ash W ground	
0	CEM I		CEM I (g)
15	CEM II/A-W bu	CEM II/A-W bp	CEM II/A-W g
30	CEM II/B-W bu (PL1, PL2)	CEM II/B-W bp (PL1, PL2)	CEM II/B-W g
50	CEM IV/B-W bu	CEM IV/B-W bp	CEM IV/B-W g

As reference cements CEM I were used. Compatibility of CEM II/B-W cements and plasticizers was also investigated.

2.2. Cement production methods

Blended cements were produced by homogenization of earlier prepared materials: Portland cement CEM I 42,5R, raw or ground fly ash (W) in blender ball - mill within 5 minutes. Cements produced by blending are marked "bu" when raw fly ash W was used, "bp" when processed fly ash W was used.

Interground cements were produced by intergrinding of all the constituents (clinker, fly ash (W), gypsum) in a laboratory ball-mill until specific surface of 4000 - 4400 cm²/g was obtained. Clinker initially was milled in ball mill to the surface of the 2500 cm²/g. Next clinker was ground together with gypsum to the surface of the 3600-3800 cm²/g. Then fly ash (W) were added and were ground to get aimed specific surface area. Cements produced by intergrinding are marked "g".

2.3. Testing methods.

2.3.1. Rheological properties. Influence of cements containing fly ash (W) on rheology was tested using mortars. Rheological behaviour of mortar, as of fresh concrete, may be sufficiently described by the

Bingham model according to equation:

$$\tau = \tau_o + \eta_{pl} \cdot \dot{\gamma} \quad (1)$$

where: τ (Pa) is the shear stress at shear rate $\dot{\gamma}$ (1/s) and τ_o (Pa) and η_{pl} (Pas) are the yield stress and plastic viscosity respectively [19,20]. Yield stress determines the value of shear stress necessary for initiating flow. When the shear stress τ surpasses the yield stress τ_o , flow of the mixture occurs, and the resistance of the flow depends on plastic viscosity η_{pl} ; the higher the plastic viscosity of the mixture is, the slower is its flow. The parameter of particular importance for workability of the mixture is the yield stress τ_o - its value determines the occurrence of flow of the mixture, and, in consequence, the accurate performance of technological processes of concrete production. The technological meaning of the plastic viscosity η_{pl} is marginal in normal concretes with relatively high w/c ratio. However, in the case of mixtures, with characterize by low w/c ratio and by high flow degree (low yield stress τ_o) obtained thanks to addition of the superplasticizer, the plastic viscosity η_{pl} is of significance for their workability and stability (HPC and SCC mixtures). It is necessary to notice that studies on rheology of mortars and concretes indicate that results of rheological measurements obtained for mortars may be suitable for prediction of fresh concrete rheology [20-23]. The rheological parameters of mortar or fresh concrete can be measured by applying no less than two considerably different rotation speed N and the measuring the resulting torque T and are determined by regression analysis according to the relation:

$$T = g + h N \quad (2)$$

where g (Nm) and h (Nm s) are rheological constants corresponding to yield stress τ_o and plastic viscosity η_{pl} respectively. After determining measurement constants of rheometer one may, if necessary, represent the values g and h in physical units. According to [19], in the apparatus like the one used in this work, $\tau_o = 7.9 g$ and $\eta_{pl} = 0.78 h$, but all results are given below in terms of yield stress g and plastic viscosity h .

The mixer and mixing procedure of mortars were compliant with PN-EN 196-1:2006 “Methods of testing cement. Determination of strength”; plasticizers were added 30 sec. after water addition. After mixing mortars samples were transferred to Schleibinger Viskomat NT rheometer and tested. After the end measurement, the mortars were stored in mixer and remixed for 2 min before the next measurement. Additionally, flow test in acc. with PN-EN 1015-3:2000/A1:2005 “Methods of test for mortar for masonry. Determination of consistence of fresh mortar (by flow table) was performed”.

2.3.2. Air content in mortar. Air content in mortars was tested after end of mixing in acc. with PN-EN 1015-7:2000 “Methods of test for mortar for masonry. Determination of air content of fresh mortar”.

2.3.3. Setting times of mortars. Setting times of mortars were tested in acc. with PN-EN 480-2:2008 “Admixtures for concrete, mortar and grout. Test methods. Determination of setting time”.

2.3.4. Plastic shrinkage of mortars. Plastic shrinkage was investigated using Schleibinger Shrinkage Cone [24]. The tests were performed on mortars analogous to rheological tests, at a temperature of 20°C and a relative humidity of 60% (the apparatus was placed in a climatic chamber).

2.3.5. Heat hydration of cement. Heat of hydration of the cement was determined with isothermal microcalorimeters TamAir. With this apparatus, one determines the amount of heat in J/g that is emitted in isothermal conditions during cement hydration from the moment of its contact with water. Measured is the heat stream that forms during the reaction of unhydrated cement sample with water in comparison to inert referential sample of analogous heat capacity. The measurement is conducted on sample

weighting 5 g, mixed with 2.5 g of water. During the measurement, temperature of the cement paste was 20°C. Measurement of the heat of hydration had lasted 72 hours.

2.4. Materials and compositions

Properties of cements are presented in table 2. For the production of cement CEM I 42.5 R, clinker and fly ash (W) was used of properties presented in table 3. Properties of plasticizers are presented in table 4. Proportions of mortars used for testing rheological properties and plastic shrinkage are shown in table 5. In the other tests proportioning of mortars was in acc. with requirements of adequate standards. Standard sand in acc. with [32] was used.

Table 2. Properties of cements.

Cement	Constituents, %				Density, g/cm ³	Blain specific surface, cm ² /g	Water demand, %	Compressive strength 28 days, MPa
	CEM I	Clinkier	W	Gypsum				
CEM I	100	-	-	-	3.09	3630	27.6	50.2
CEM I (g)	-	95	-	5	3.10	3810	25.8	59.2
CEM II/A-W bu	85	-	15	-	2.99	3640	26.5	53.2
CEM II/B-W bu	70	-	30	-	2.95	3570	28	49.6
CEM IV/B-W bu	50	-	50	-	2.85	3420	30.2	38.8
CEM II/A-W bp	85	-	15	-	3.04	4020	30	56.5
CEM II/B-W bp	70	-	30	-	2.99	4070	31	53.4
CEM IV/B-W bp	50	-	50	-	2.92	4150	32.2	49.2
CEM II/A-W g	-	81.1	14.3	4.6	3.04	4190	27.6	58.7
CEM II/B-W g	-	67.7	29	3.3	2.98	4030	30.4	51.1
CEM IV/B-W g	-	49.2	49.2	1.6	2.88	4000	31.4	42.1

Table 3. Composition of cements

Cement	Constituents, %							
	LOI	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	K ₂ O	Na ₂ O	SO ₃
CEM I	2.65	19.18	4.93	2.70	65.08	0.79	0.12	2.74
CEM I (g)	1.92	20.35	4.48	2.06	66.56	0.54	0.24	2.84
CEM II/A-W bu	3.31	21.13	7.02	3.03	59.39	0.72	0.15	3.17
CEM II/B-W bu	3.24	22.85	9.07	3.46	54.80	0.62	0.18	3.45
CEM IV/B-W bu	3.27	25.86	12.23	4.00	47.39	0.47	0.22	3.73
CEM II/A-W bp	3.88	20.33	6.87	3.10	59.96	0.69	0.15	3.1
CEM II/B-W bp	3.58	23.03	9.16	3.48	54.39	0.58	0.18	3.53
CEM IV/B-W bp	3.36	25.34	11.95	4.07	48.15	0.46	0.22	3.84
CEM II/A-W g	2.01	22.38	6.60	2.54	61.29	0.16	0.25	2.84
CEM II/B-W g	2.19	23.89	8.71	3.07	56.56	0.15	0.26	3.05
CEM IV/B-W g	2.30	25.97	11.54	3.76	50.28	0.15	0.28	3.18

Table 4. Properties of plasticizers

Type	Dosage
PL1 lignosulfonate	½ max = 0.25%
PL2 iminodiethanol, bis ethanol, phosphate (V) tri butyl acetate, formaldehyde, methanol, (Z)-octadec-9-enylamine	½ max = 0.25%

3. Results and discussions

Influence of cements CEM II/A-W, CEM II/B-W and CEM IV/B-W on rheological properties, air content, setting times and plastic shrinkage of mortars are presented on figures 1 and 2 and in table 6. Heat of hydration of these cements is presented in table 6.

Table 5. Mortar proportioning

	Content, g/batch
Cement	450
Sand	1350
Water	247.5
w/c	0.55

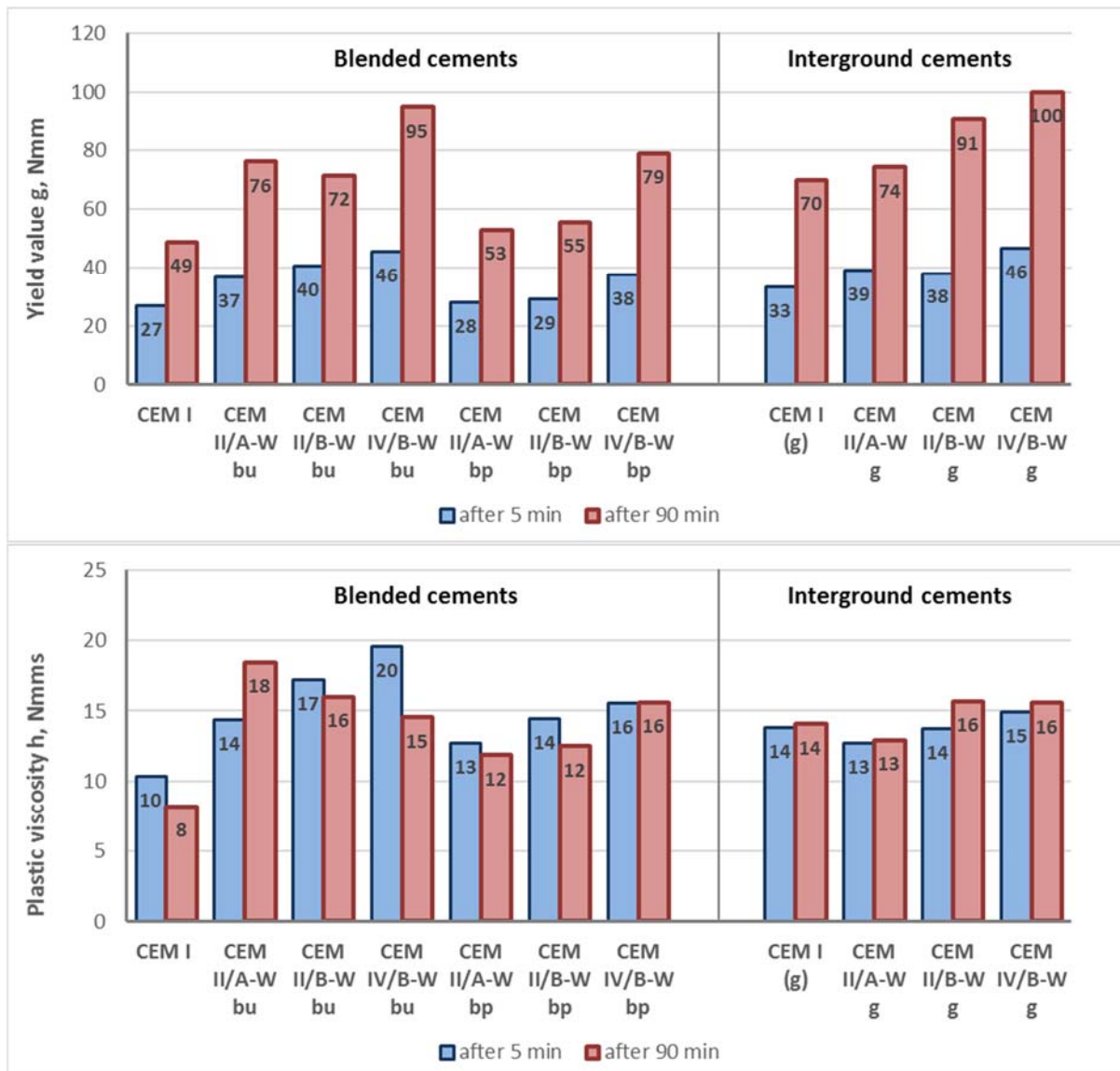


Figure 1. Rheological properties of mortars made of cements CEM II/A-W, CEM II/B-W and CEM IV/B-W, bu - cements produced by blending with raw fly ash W, bp - cements produced by blending with processed fly ash W, g - cements produced by intergrinding

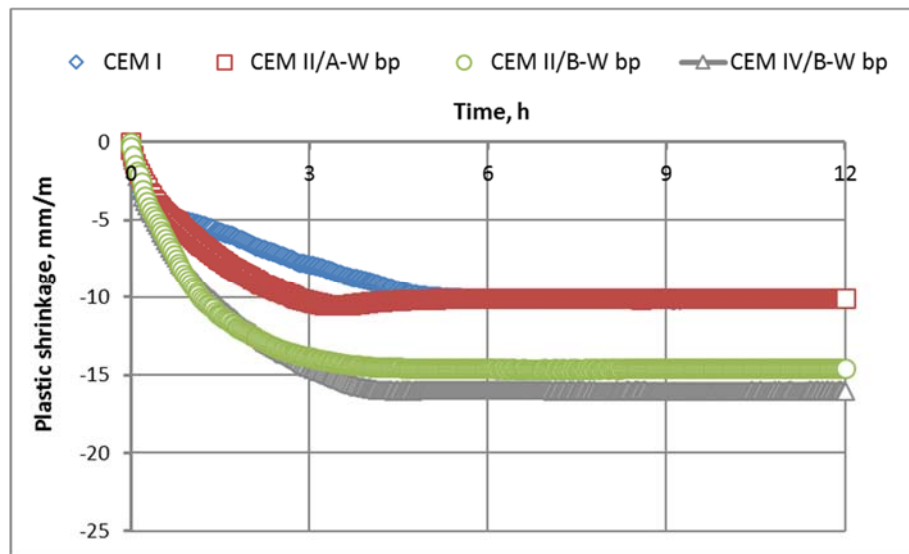


Figure 2. Plastic shrinkage of mortars made of cements by blending, fly ash W processed

Table 6. Flow, air content, setting times of mortars, heat of hydration of cements

Cement	Flow, mm		Air content, %	Setting time, min		Hydration heat, J/g		
	5 min	90 min		initial	end	12 h	24 h	72 h
CEM I	21.5	19.7	5.4	121	215	59.76	146.1	250.0
CEM I (g)	20.8	17.8	2.5	135	180	66.96	161.1	266.6
CEM II/A-W bu	21.0	17.0	4.2	135	180	56.41	143.0	243.9
CEM II/B-W bu	20.4	17.3	3.4	188	298	45.21	117.7	226.5
CEM IV/B-W bu	19.6	16.5	4.0	193	348	39.04	95.62	208.2
CEM II/A-W bp	21.0	19.0	1.6	136	211	57.53	152.8	244.5
CEM II/B-W bp	21.2	19.3	2.5	186	296	45.45	124.0	221.7
CEM IV/B-W bp	20.5	16.7	3.4	276	346	45.09	121.9	210.7
CEM II/A-W g	20.4	16.9	2.4	153	223	63.12	156.7	261.4
CEM II/B-W g	20.7	17.0	2.6	180	250	50.47	149.7	241.2
CEM IV/B-W g	19.3	16.8	2.9	188	258	40.45	109.5	205.9

Regardless of the production method of cement, CEM II/ A-W mortars are characterized by higher yield stress σ (and smaller flow diameter) than CEM I mortars, and faster increase of the yield stress σ in time (reduction of flow diameter in time). With the increase of the amount of fly ash (W) in the cement (CEM II/B-W, CEM IV/B-W), the yield stress σ of mortars and its increase in time increase. The negative influence of the presence of fly ash (W) in cement on the rheological properties of mortars is the strongest in case of cements blended with raw fly ash (W), and the weakest in case of interground cements - this is due to the beneficial effect of using processed fly ash. In case of mortars with blended cements with processed fly ash and with interground cements with the amount of fly ash (W) up to 30% (CEM II/A-W and CEM II/B-W) the worsening of fluidity is moderate, and becomes significantly higher only when the amount of fly ash (W) is increased to 50% (CEM IV/B-W). In case of using cements with raw fly ash (W) the significant worsening of fluidity can be observed already when the amount of fly ash reaches 15% (CEM II/A-W). The mortars with calcareous fly ash W are usually characterised by the higher plastic viscosity η than the mortars with CEM I cement, and with the increase of fly ash (W) content, the plastic viscosity η also increases.

The nature of the influence of fly ash (W) on the rheological properties of mortars when it is used as a cement constituent or as a type II additive is analogous (figure 3). However, if fly ash (W) is used as the cement constituent, its negative influence on the rheological properties is clearly lower. It is probably caused by the additional grinding of the fly ash (W) during the process of cement production. Using the fly ash (W) as a cement constituent enables to obtain the mortars (and concrete mixes) with better workability when analogous amount of fly ash (W) is added directly to the mix.

The presence of fly ash (W) in cement does not affect or decreases ~~of~~ the amount of air in the mortar in comparison to mortars with CEM I cement (table 6). Cements with fly ash (W) are characterized by the delayed initial and final setting times in relation to CEM I cement (table 6). The delay is the greater, the higher is the fly ash (W) content. The delay of the initial setting time depends to a small degree on the method of cement production, whereas the delay of final setting time is the highest in case of the blended cements with processed fly ash (W), and the lowest for interground cements. Plastic shrinkage of the mortars with fly ash (W) cements is higher than of CEM I mortars and it raises proportionally to the final setting time of cement.

In the time range of first 12 to 72 hours, the amount of heat generated by cements with fly ash is smaller than by CEM I cements – in case of CEM II/A-W by about 3%, CEM II/B-W by approx. 10%, CEM IV/B-W by approx. 20% (table 6). The method of cement production does not affect the amount of heat generated during the hydration.

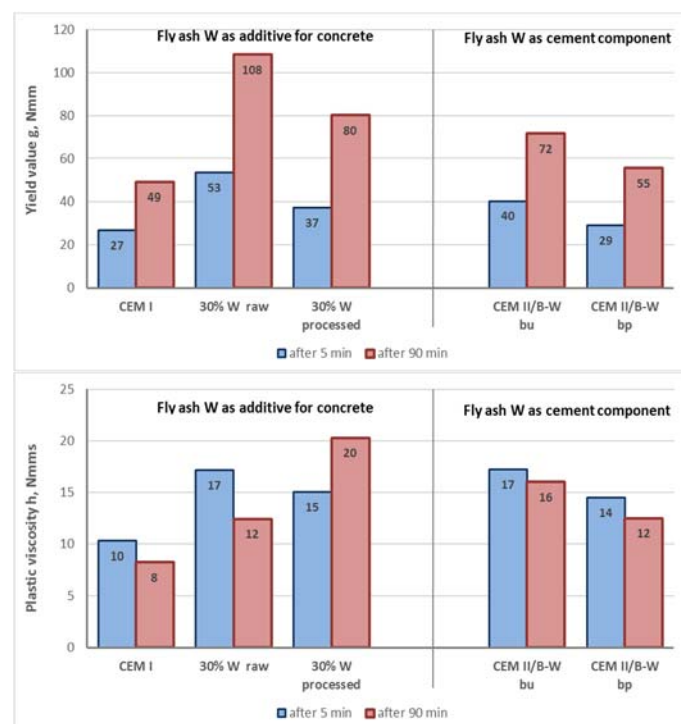
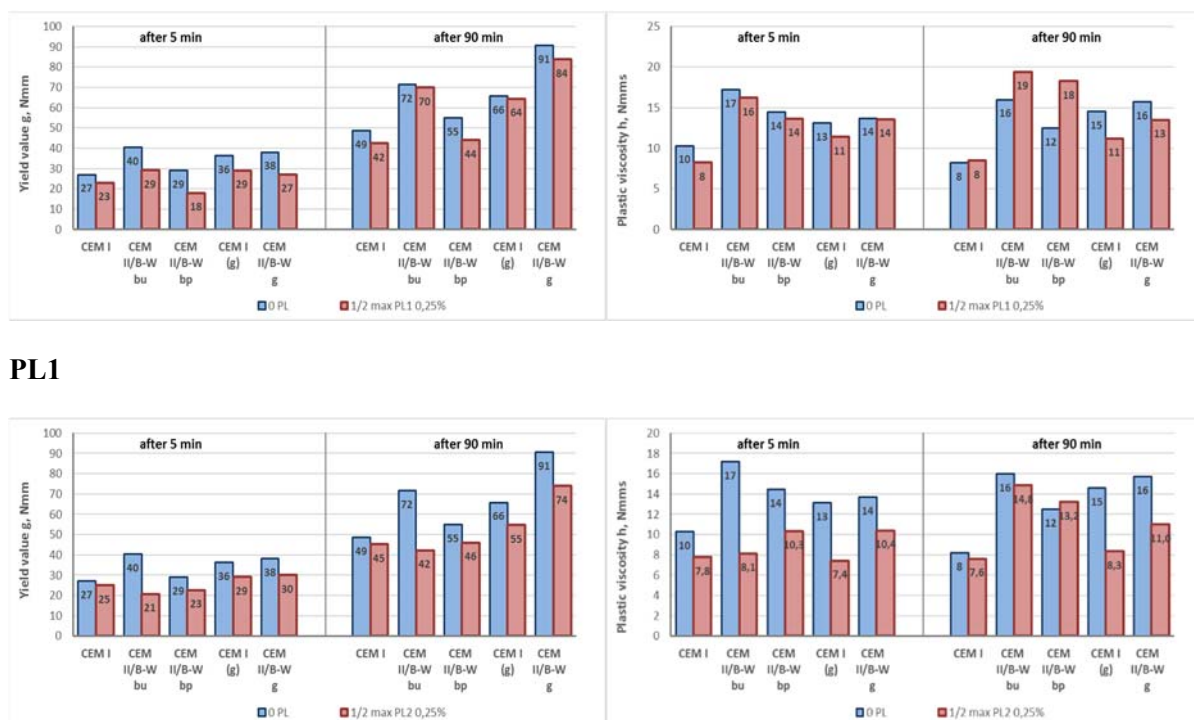


Figure 3. Rheological properties of CEM II/B-W mortars and mortars with fly ash W as mineral additive for mortar (as part of cement replacement), bu - cements produced by blending with raw fly ash W, bp - cements produced by blending with processed fly ash W

The influence of plasticizers PL1 and PL2 on the rheological properties of mortars is shown on figure 4, and on the setting time, air content, and heat of hydration in table 7. In the presence of CEM II containing fly ash (W) PL1 and PL2 plasticizers act effectively, lowering the yield stress g and plastic viscosity h , and inhibiting the changes of rheological parameters in time. Importantly, the yield stress of

CEM II/B-W mortars, as a result of adding plasticizer in the amount of $\frac{1}{2}$ of max. dosage is usually after 5 min lower than in case of CEM I mortars with analogous dose, and for PL2 this effect is sustained even up to 90 min. This means that the process of choosing the right plasticizer requires experimental optimization. Plasticizer PL2 greatly lowers the plastic viscosity of mortars with all of the tested cements. This can probably be attributed to the air-entraining effect of this admixture (plasticizer PL1 does not exhibit similar characteristics).

Plasticizer PL 1 practically does not influence air content of mortars, while plasticizer PL2 shows air entraining action. In case of CEM II/B-W mortars, air content amounts from 10 to 13% and is significantly smaller than in case of CEM I mortars. The PL1 and PL2 plasticizers delay the initial setting time. Plasticizer PL2 reduces the amount of heat generated during first 12 and 24 h, however after 72 h this effect disappears.



PL2

Figure 4. Influence of plasticizers PL1 and PL2 addition ($\frac{1}{2}$ maximum dosage (0.25% C)) on rheological properties of mortars made of CEM II/B-W cements produced by blending (with fly ash W raw (bu) or processed (bp)) and by intergrounding (g)

Table 7. Influence of plasticizer on properties of mortars

	Air content, %	Initial setting time, min	Hydration heat, J/g		
			12 h	24 h	72 h
CEM I	5.4	182	59.76	146.1	250.0
CEM I + PL1	4.1	301			
CEM I + PL2	20.5	349	30.853	107.0	249.5
CEM II/B-W	2.5	219	45.45	124.0	221.7
CEM II/B-W + PL1	2.1	385			
CEM II/B-W + PL2	10.5	444	33.774	56.946	225.6

Due to the fact that cements with fly ash (W) are characterized by higher water demand than in case of CEM I cements, to obtain a set workability of mixture for a set w/c ratio, it may be necessary to add a higher dose of plasticizer. Using a plasticizer one can neutralize the effect of higher workability loss in case of cements with calcareous fly ash (W).

4. Conclusions

Calcareous fly ash can be successfully used as a main component of a wide assortment of cements. Cements produced by blending with processed fly ash (W) or by interginding are characterized by acceptable technological properties and do not differ significantly from other currently used cements. It is not recommended to use cements obtained by blending with raw fly ash (W). Calcareous fly ash used for cement production should be selected on the basis of its properties.

In respect to CEM I mortars:

- mortars with CEM II/A-W, CEM II/B-W, CEM IV/B-W are characterized by worse workability and faster workability loss. Those effects are the higher, the higher is the fly ash (W) content in cement. It must be noted, however, that the negative influence of fly ash (W) used as cement component on workability loss is significantly lower than if the fly ash (W) is used as an additive to mortars.
- Presence of fly ash (W) in cement does not affect or contribute to the loss of air content in the concrete mix.
- Initial and final setting times of cements with fly ash (W) are delayed. During first 12-72 h the amount of heat generated by cements with fly ash (W) is lower by 10 to 20%.
- Plastic shrinkage is proportional to the final setting time of cement and is higher for mortars with cements containing fly ash (W). This must be taken under consideration while choosing the proper curing method for concretes with those cements - intensive moisturising should be applied from the moment of finishing the process of concrete laying.

The effectiveness of plasticizers in presence of CEM I cements and CEM II/B-W do not differ significantly or even increases. The method of production of cement does not significantly affect the effectiveness of plasticizers.

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