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Scientific paper
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An innovative approach for producing high volume fly ash blended cements that meet European standard requirements by employing a silicious coal fly ash exhibiting unusually high water demand and other peculiar properties

The research work is about an innovative approach to produce blended cements with up to 45% silicious coal fly ash. The fly ash which was provided by a local power plant is of silicious type, but it has a pronounced honeycomb particle structure and much lower particle and bulk density, differing significantly from common silicious type fly ashes reported in the literature. This fly ash exhibits an usually high water demand, which makes it a real challenge to produce blended cements with high amounts of this fly ash, even with prolonged time of grinding. The innovation consists on co-grinding of raw fly ash with small amounts of liquid superplasticizer and eventually some small retarder amounts in order to obtain blended cements that conform to type IV cement according to European standard specifications. A series of cements could be developed in laboratory conditions with different rheological behaviour and strength properties. This approach is very promising and can be employed for any kind of fly ash in order to maximize its content in cement, reduce waste and green gases environmental impact, lower costs of cement production and offer an additional posibility to produce cements with tailored properties that can be employed for superior concrete.

Key words: *innovative approach; high volume fly ash blended cements, silicious coal fly ash, high water demand.*

1. INTRODUCTION

The coal combustion process produces solid coal utilization by-products (CUBs) from the non-combustible portion of the coal. The CUBs include fly and bottom ash as well as incinerator slag, whereby fly ash constitutes the majority of CBUs. The chemical and physical properties of fly ash particles are a function of the mineral matter in the coal, the combustion conditions, and post-combustion cooling [1–4]. In a pulverized coal fired boiler, the operating temperatures are typically in excess of 1400°C, at which temperatures minerals in coal become fluid and are then cooled [5].

The heating and cooling have a significant effect on the composition and morphology of each particle. The morphology of a fly ash particle is controlled by combustion temperature and cooling rate. Typically, rapid cooling in the post-combustion zone results in the formation of spherical, amorphous particles of sizes within 1 to 100μm [6]. Specific gravity of fly ashes range from approx. 2.0 to 2.5, the lower values may be due to the formation of hollow spheres [6].

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Hollow fine fly ash particles may have thick shell walls, because the finer the particles are, the higher glass content it has [7]. In hollow fly ashes, the finer the particles are, the more likely the volume of closed pores will decrease. Therefore, the most important factors affecting specific gravity may be the content of closed pores [7]. Specific gravity may become an important technical and economical issue in blended cements that incorporate large volumes of fly ash. The blended cement will have lower than usual specific and bulk density, which might become a problem for handling and production of cement and concrete. One way to increase the specific gravity of fly ash is to grind it, in order to break the hollow particles [8]. During grinding the irregular shaped particles are crushed and the morphology of the fly ashes is changed. Grinding can additionally improve the pozzolanic reactivity of fly ash particles [9-11].

Fly ash can be used in cement production imparting multiple economical benefits [12]. Fly ash can partially replace clinker in blended cements at equal or improved performance and that portion of fly ash can be marketed at the price of cement. Utilization of fly ash in cement minimizes the CO_2 emission problem to the extent of its proportion in cement. Studies show that one tone of Portland cement production discharges in average 0.87 tones of CO_2 into the atmosphere. At least 5% of global carbon dioxide emissions originate from cement production [13]. The current practice of fly ash containing blended cement production involves transport of fly ash from power plant to cement industry since the proportion of fly ash used is less than 25-30% of cement blend [14]. The higher usage in the blended cements is restricted by the low pozzolanic and/or latent hydraulic activity, consequently resulting in slow development of compressive strength as compared to Ordinary Portland Cement (OPC) [14]. This type of cement that uses fly ash as partial replacement of clinker is known as blended cement. Alone at a fly ash replacement ratio of 25% at least 20% of energy savings of cement production can occur [15, 16]. These factors have prompted research projects aiming to boost the fly ash replacement levels in cement. Such research has environmental relevance, in addition to industrial interest.

In this research work we employed a low specific gravity silicious fly ash with honeycomb-like particles morphology and targeted the development and production of high volume fly ash (HVFA) blended cement. High volume fly ash blended cements contain fly ash in excess of 30% (mass percentage) [8, 17]. The employed fly ash, apart from irregular shape and lower than usual specific and bulk density, exhibited a unusually high water demand (approx. 140-145%), which further complicated the employment of high volumes of fly ash. After trying different approaches the most successful one resulted to be the addition of medium and high range water reducers (plasticizers and superplasticizers) along with some setting retarders or accelerators that were added during co-grinding (also named inter-grinding) in a conventional laboratory ball mill of the raw components.

2. EXPERIMENTAL

2.1. Materials

2.1.1. Clinker

The industrial clinker which was employed to produce common type cements was provided by a local cement plant. Its chemical and phase compositions are given in table 1.

Table 1 - The chemical and phase composition of the clinker (main oxides and phases)

Property	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**CaO**	**CaO free**	**MgO**	**SO_3**	**Loss on ignition**	**C_3S**	**C_2S**	**C_3A**	**C_4AF**
Content [%]	20.49	4.91	3.36	63.85	0.46	3.74	1.64	0.39	66.38	8.72	7.33	10.22

2.1.2. Gypsum

Gypsum with a $CaSO_4$ x $2H_2O$ content of more than 80% was employed as source of SO_3.

2.1.3. Fly ash

The employed fly ash was of silicious type (CaO < 10%). It has a specific gravity of 1.7-1.8 g/cm^3 and bulk density of 0.65-0.70 g/cm^3. The SEM image in figure 1 (left) shows some particles of the employed fly ash with varying sizes, shapes and morphology. The predominant part of them has a honeycomb-like structure. In the same figure (right) is shown also the SEM image of a "normal" fly ash with almost spherical shape particles. This fly ash exhibited an unusually high water demand (approx. 140-145%). The water demand of fly ashes reported in literature exhibit a water demand lower than 100% compared to the reference paste. The chemical composition of this fly ash is shown in table 2.

Table 2 - The chemical composition of the fly ash (main oxides)

Property	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**CaO**	**CaO free**	**MgO**	**SO_3**	**Cl**	**Loss on ignition**
Content [%]	50.28	24.96	9.28	3.98	0	4.08	0.78	0.0035	1.63

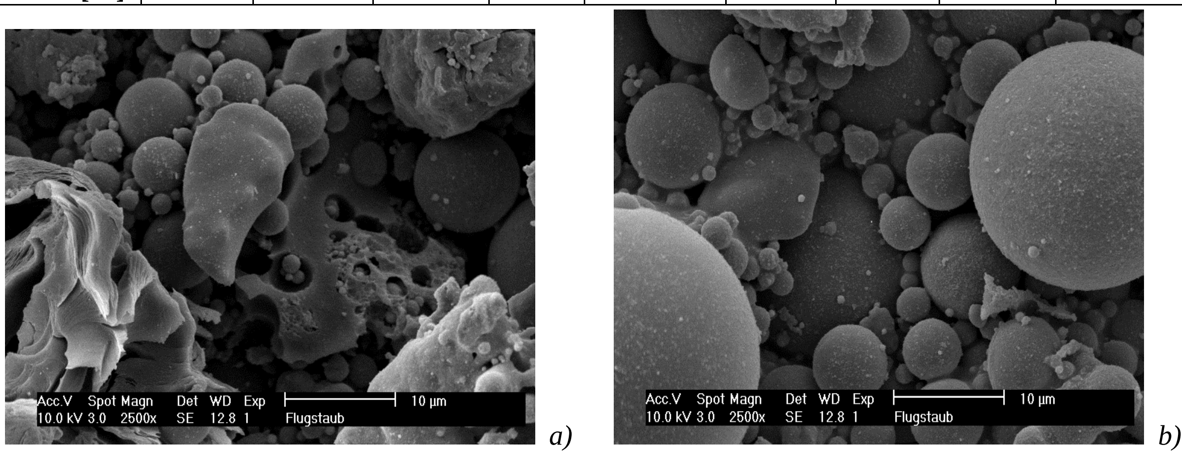

Figure 1 - SEM images of the employed fly ash (a) and a spherical shaped fly ash (b)

2.1.4. Water reducers, setting retarders and accelerators

Some characteristics of the employed water reducers are shown in table 3.

Table 3 - Properties of the employed water reducers

Water reducer code	Type	Water reduction efficiency
FL	Ligno sulphonate	Medium range
SL	Naphthalene sulphonate	Medium range
ML	Melamine sulphonate	High range
SU	Polycarboxylate	High range
MF	Polycarboxylate ether	High range

The setting retarders and accelerators didn't contain chlorides.

2.2. Equipments

2.2.1. Laboratory ball mill

An own designed and constructed, closed circuit "ball mill" was employed for the grinding of clinker, gypsum and fly ash in order to produce the blended cements with the specified fineness. The simple and robust construction of the mill allows for slow horizontal rotational speed. The fineness of the cement was controlled via the optimization of the grinding time.

2.2.2. Setting time apparatus

The setting time of the pastes was determined at 20±1°C according to EN 196-3 using a manual Vicat apparatus.

2.2.3. Paste and mortar mixers

A "ToniMIX" mixer automatically programmable from "Toni Technik" was employed for the preparation of cement pastes and mortar mixes with automatic mixing procedure including water feeding in accordance with EN 196 -1.

2.2.4. Vibrating table

A vibrating table from "Toni Technik" for compacting paste and mortar prisms in either single or triple moulds in accordance with EN 196-1 standard was employed to cast-compact the specimens.

2.2.5. Specimen curing cabinet

Following casting, the specimens (pastes or mortars) were cured in a climate cabinet from "Toni Technik" at 20±1°C and relative humidity r. h.≥ 95% as per EN 196-1 standard. The humidity is maintained from 95% to saturation by water nebulizers. The temperature is maintained to 20±1°C by an immersion heater and separated refrigerator unit. The prism-shaped specimens were demoulded after 24 hours ± 15 min and then stored in water at 20±1°C.

2.2.6. Compression strength testing machine

The measurement of compression strength was carried after the specified time of curing in an automatically controlled hydraulic press type "ToniNorm" from "Toni Technik" according to the EN 196-1 standard. The mortar specimens had prismatic sections of dimensions 40x40x160mm.

2.3. Methods

The aim of the research work was not only to find a solution for the utilization of this fly ash, but also to maximize its utilization. Previous own investigations have shown that the approach for the production of high-volume fly ash (HVFA) blended cement that meet the EN standard requirements depends on the type of the fly ash used. While the production of normal volume fly ash (NVFA) cements that comprise both types of fly ash (either silicious or calcareous) or HVFA cements that comprise calcareous fly ash, which is the most reactive of both types, is not a serious challenge, the production of industrial HVFA cement with silicious fly ash remains a challenge and requires special approaches. The most suitable solution was found the development and production of HVFA blended cements by co-grinding (intergrinding), therefore the second aim was the production of blended cements with maximum possible fly ash content (HVFA) that fully comply with the requirements of the European cement standard EN 197-1. As already mentioned, in order to overcome the issue of low specific gravity and bulk density, the fly ash was ground in the laboratory ball mill.

The most critical component of the blend regarding grindability is fly ash; it is the hardest component to grind. As grinding time, which is a key parameter for the properties of cement, was chosen the time of grinding which is required to produce in laboratory conditions a commercially available cement of same type (ref. cem). The grinding time is deemed satisfactory when the laboratory produced cement (ref. cem) shows as close as possible properties with the commercial cement of type "CEM I 42.5 R" produced under industrial conditions. Based on previous experience with strength development of silicious fly ash mixes, it was deemed satisfactory enough to reach this strength class. This time is referred to as the "optimal grinding time". Also, as indicator of fineness was taken the residue on a specified set of sieves and not the Blaine value. This was purposely chosen due to the high uncertainties of Blaine method when measuring the fineness of blended cements with high amounts of additions. Before the clinker was fed to the grinding mill, it was crushed so that all the particles were less than 2.0 mm. A series of HVFA cements with different compositions and properties were successfully produced in laboratory conditions. In order to overcome the problem of unusually high water demand, various water reducers were added during co-grinding process in the ball mill. The procedures to produce HVFA blended cements that meet the requirements of EN 197-1 consisted of producing blended cement by grinding together (co-grinding) the cement clinker, gypsum, fly ash and small amount of water reducer. Finally in order to optimize the setting and hardening behavior, a series of accelerating or retarding substances were employed that as with water reducers were added during the grinding process. The amount of setting controllers was very low (max. 0.2% of the blend mass).

3 RESULTS AND DISCUSSION

3.1. High volume fly ash (HVFA) cements made by co-grinding

In order to find out the “optimal grinding time” for the laboratory mill, three cement samples that did not contain fly ash (CEM I 42.5 R type) were first produced by employing three different grinding times as in the following, and their main properties were compared (table 4):

S_1:95% clinker and 5% gypsum, (grinding time 15 min)

S_2: 95% clinker and 5% gypsum, (grinding time 20 min)

S_3: 95% clinker and 5% gypsum, (grinding time 25 min)

The sample “S_3” gave the most satisfactory results, the properties of this sample were very close to the commercial cement which does not contain fly ash. Therefore the grinding time of 25 minutes was chosen for the further trials.

Table 5 gives the recipes and the content of fly ash used for the production of HVFA cements.

Table 4 - Trials for the optimal grinding time

Sample	Grinding time [min]	Compressive strength			Fineness	
		2 days [MPa]	7 days [MPa]	28 days MPa]	ROS 90µm [%]	ROS 45µm [%]
S_1	15	15.6	30.4	47.8	7.8	24.0
S_2	20	20.6	35.9	52.4	6.1	21.5
S_3	25	26.5	41.2	56.5	4.8	19.3

Table 5 - Recipes and fly ash content of the HVFA laboratory cements

Constituents [%]	C_0*	HF_1	HF_2	HF_3	HF_4	HF_5
Clinker **Gypsum** **Water reducer + setting retarder or accelerator (WR)**	55.0	55.0	55.0	55.0	55.0	55.0
Fly ash	45.0	45.0	45.0	45.0	45.0	45.0
Constituents [%]	**HF_6**	**HF_7**	**HF_8**	**HF_9**	**HF_{10}**	**HF_{11}**
Clinker **Gypsum** **Water reducer + setting retarder or accelerator (WR)**	55.0	55.0	52.0	55.0	53.5	55.0
Fly ash	45.0	45.0	48.0	45.0	46.5	45.0
Constituents [%]	**HF_{12}**	**HF_{13}**	**HF_{14}**	**HF_{15}**	**HF_{16}**	**HF_{17}**
Clinker **Gypsum** **Water reducer + setting retarder or accelerator (WR)**	55.0	55.0	55.0	60.0	65.0	70.0
Fly ash	45.0	45.0	45.0	40.0	35.0	30.0

*the sample M_0 does not contain water reducer and/or setting accelerator/retarder

For every specimen the amounts of gypsum and activator were varied within the specified range, the amount of the water reducer (WR) in any case was varied between 0.5 - 2% and that of gypsum was within the limits specified by the standard EN 197-1 (calculated as SO_3). The physical properties of the laboratory made HVFA cements (HF_1 - HF_{17}) as well as the properties of one laboratory cement that does not contain fly ash (ref. cem) and a cement that contains 45% fly ash (C_0), but that does not contain water reducer, were determined and are given in table 6.

All cements produced (HF_1 – HF_{17}) meet the requirements of the European standard for cement (table 7).

Table 6 - Physical properties of the HVFA laboratory cements produced by co-grinding

Sample code	Grinding time [min]	Compressive strength			Initial setting time [min]	Fineness	Expansion [mm]	Code of the Water reducer (WR)
		2 days [MPa]	7 days [MPa]	28 days [MPa]		ROS 90µm [%]		
Ref. cem	25	26.5	41.2	56.5	170	4.8	0.1	-
C_0	25	7.6	19.7	29.8	340	5.6	0.1	-
HF_1	25	10.8	26.6	46.6	230	5.9	0.2	SL
HF_2	25	11.2	28.4	48.0	115	4.3	0.1	ML
HF_3	25	15.7	36.8	52.2	185	4.9	0.1	SU
HF_4	25	17.9	40.8	57.3	260	4.2	0.2	SL
HF_5	25	18.7	41.7	54.7	120	3.8	0.1	SU
HF_6	25	20.3	42.1	53.7	100	3.7	0.2	SU
HF_7	25	17.1	39.9	56.0	195	4.4	0.1	SL
HF_8	25	18.3	38.5	57.1	210	4.1	0.1	SL
HF_9	25	16.2	33.7	52.8	170	5.2	0.1	SL
HF_{10}	25	19.9	40.4	54.0	80	5.5	0.2	SU
HF_{11}	25	17.4	37.4	53.8	160	6.5	0.2	SL
HF_{12}	25	16.2	35.2	50.8	185	5.1	0.1	FL
HF_{13}	25	15.0	35.5	54.3	140	7.0	0.1	FL
HF_{14}	25	19.0	38.8	56.8	130	6.0	0.2	FL
HF_{15}	25	21.2	45.7	57.5	170	6.8	0.1	SL
HF_{16}	25	24.7	48.7	58.8	175	5.3	0.1	SL
HF_{17}	25	26.5	47.7	58.5	180	5.0	0.1	SL

Table 7 - Extract of the European standard (EN 197-1) requirements for clinker-based cements depending on the strength class

Strength class	Compressive strength [N/mm^2]				Setting time [minutes]	Soundness (expansion) [mm]
	Early strength		Standard strength			
	2 days	7 days	28 days			
32.5 N	-	≥16	≥32.5	≤52.5	≥75	≤10
32.5 R	≥10	-				
42.5 N	≥10	-	≥42.5	≤62.5	≥60	
42.5 R	≥20	-				
52.5 N	≥20	-	≥52.5	-	≥45	
52.5 R	≥30	-				

The HF_1 – HF_{15} cements belong to the Pozzolanic type (Cem IV/B), whereas the cements HF_{16} - HF_{17} belong to the Portland – fly ash type (Cem II/B-V). Any targeted strength that is specified in the European standard can be obtained by varying the amount of fly ash and the water reducer. Eventhough any type of the employed water reducers could produce the targeted strength properties, the naphthalene sulphonate (SL) type, which is a medium range water reducer, seems to work best with this fly ash. It seems also that there is no need to employ a high range water reducer to reach the best cement propeties, a medium range water reducer is sufficient for this purpose. The desired cement properties can be reached by optimising the water reducer + accelerator/retarder and fly ash content. The soundness of all cements is within the limits specified by this standard. Also all cements have loss on ignition values, insoluble residues, sulphate and chloride contents within the limits specified by the standard. For the pozzolanic cement the standard requires additionally to assess whether the cement is pozzolanic or not (the test should be positive). Using the Rio-Fratini method, the pozzolanicity is assessed by comparing the quantity of calcium hydroxide in the aqueous solution in contact with the hydrated cement, after a fixed period of time, with the quantity of calcium hydroxide capable of saturating a solution of the same alkalinity. The test is considered positive if the concentration of calcium hydroxide in the solution is lower than the saturation concentration. The results of the Rio-Fratini test for a HVFA cement paste with

45% fly ash show that $Ca(OH)_2$ produced during cement hydration has been consumed by pozzolanic reactions the point is below the solubility curve of $Ca(OH)_2$.

In table 8 are compared some technical properties of two selected HVFA cements with few comercially available cements. One of the worries with this fly ash has been its lower than usual specific gravity and bulk density and the aim has been to produce HVFA cements with bulk densities comparable to the commerical cements. The data of table 8 show that this property and other ones were similar to the properties of commercial cements.

Table 8 - Comparison of properties of two laboratory produced HVFA cements with some commercially available cements

Type of Cement & Producer	*In. Setting time [min]*	*Consistence [%]*	*Bulk density [kg/m³]*	*Compressive Strength*	
				2 Days [MPa]	*28 Days [MPa]*
HF_{11}	160	29.6	950	17.4	53.8
HF_{12}	185	30.4	980	16.2	50.8
CEM IV/B-V 32.5 R	297	30.0	1100	16.2	52.3
CEM IV/B-(W-P) 32.5 N	270	29.6	1000	25.6	56.0
CEM IV/B 32.5 N	154	32.2	1060	18.7	54.5

Finally in figure 2 are shown the particle size distribution curves (cumulative percentages) of some HVFA cements. The fact that the size distribution curves lie relatively close to each-other indicates that the milling has a relatively good reproducibility on grinding performance.

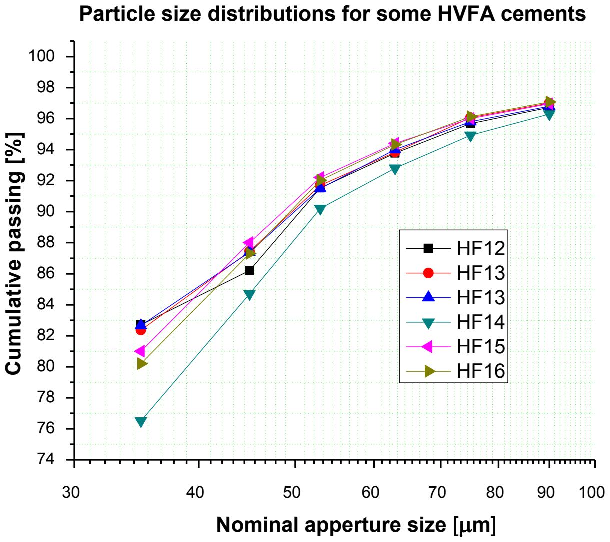

Figure 2 - Particle size distribution curves (cumulative percentages) of some HVFA cements

It should be recognized that all the above data are valid only to a closed circuit grinding, and probably more specifically, to grinding in similar mills and with similar grinding balls. The production of cement via co-grinding fly ash and clinker in commercial ball mills that practically contain more than one compartment and equipped with air separator may yield different results. In view of this, these laboratory data should be considered as a starting point, and trial grindings should be run in commercial ball mills before adopting this technology.

4 CONCLUSIONS

A silicious type fly ash exhibiting unusually high water demand and low specific gravity has been successfully employed for the production of high volume (HVFA) blended cements. The fly ash content of the cements, whereby the fly ash is replacing clinker, can be as high as 50% (optimum for this fly ash about 45%). The properties of the produced cements are well comparable with the properties of cements that do not contain fly ash and most of them meet the EN specifications and requirements for blended cements. This innovative approach consists on co-grinding of raw fly ash with small amounts of liquid water reducer and eventually some small retarder amounts in order to obtain blended cements that conform to type IV cement according to European standard specifications. A series of cements could be developed in laboratory conditions with different rheological behavior and strength properties. Any targeted strength that is specified in the European standard can be obtained by varying the amount of fly ash and the water reducer. Eventhough any type of the employed water reducers could produce the targeted strength properties, the naphthalene sulphonate (SL) type, which is a medium range water reducer, seem to work best with this fly ash. It seem also that there is no need to employ a high range water reducer to reach the best cement propeties. The desired cement properties can be reached by optimising the water reducer + accelerator/retarder and fly ash content. This approach is very promising and can be employed for any kind of fly ash in order to maximize its content in cement, reduce waste and green gases environmental impact, lower costs of cement production and offer an additional possibility to produce cements with tailored properties that can be employed for superior concrete.

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IZVOD

INOVATIVNI PRISTUP ZA PROIZVODNJU VELIKOG OBIMA PEPELA U SMEŠI CEMENTA KOJI ZADOVOLJAVAJU EVROPSKE STANDARDE ZA KORIŠĆENJE PEPELA IZ SILICIJUMSKOG UGLJA, KOJI ZAHTEVAJU VISOKU POTRAŽNJU VODE I IMAJU DRUGA SVOJSTVA

Istraživački rad je posvećen inovativnom pristupu za proizvodnju cementa pomešanog sa 45 % silicijum letećeg pepela uglj a. Pepeo, koji je obezbeđen od strane lokalne elektrane, je silicijumskog tipa, ali ima izraženu saćastu strukturu čestica i mnogo manje granula u nasutom stanju , znatno se razlikuje od uobičajenih silicijumskih pepela prijavljenih u literaturi. Ovaj pepeo obično ispoljava visoku potražnju za vodom, što ga čini pravim izazovom za proizvodnju cementa pomešanim sa visokim količinama ovog pepela, čak i sa produženim vremenom brušenja. Inovacija se sastoji u ko - mlevenju sirovog pepela sa malim količinama tečnosti superplastifikatora i eventualno nekim malim dodatkom u cilju dobijanja smeša cementa koje su u skladu sa tip IV cementa prema evropskim standardnim specifikacijama. Niz cementa se mogu razviti u laboratorijskim uslovima sa različitim ponašanjem i reološkim svojstvima. Ovaj pristup je veoma obećavajući i može se koristiti za sve vrste letećeg pepela u cilju maksimalnog iskorišćenja sadržaja u cementu , smanjenja otpada i uticaja gasova na životnu sredinu , niže troškove proizvodnje cementa a nude i dodatnu mogućnost da se proizvede cement prilagođenih osobina, koji se mogu koristiti za vrhunski betona.

Ključne reči: *inovativni pristup; visok obim pepela u mešavini cementa, pepeo silicijumskog ugalja, visoka potražnja vode.*

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