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Processing of high sulphate and free lime calcareous coal fly ash for producing high volume blended cements and complying grade products employed in civil engineering

The present work outlines the results of a research attempt aimed at developing and evaluating the performance of processed fly ash. The raw fly ashes are very coarse, have variable chemical and mineralogical composition and contain higher than allowed amounts of sulphate and free lime. Furthermore these fly ashes contain enhanced levels of radionuclides (²²⁶Ra, ²³²Th, ⁴⁰K), which originate from the parent coal mineral. The above listed properties prevent raw calcareous fly ashes to be employed as they are. Calcareous type lignite fly ash samples that were provided from various power plants were processed in ways which enable the highest possible and safe utilisation ratio of fly ash in cement and some civil engineering applications. The processing consisted mainly on blending and/or co-grinding whereby co-grinding was a combined grinding and hydrolisis in order to reduce the sulphate and free lime content of the blend. Provided an accurate and strict quality management system is available, calcareous fly ashes can easily and safely be employed at up to 50% ratio in blended cements that comply with the European standard requirement for cement. This processing way is an environmentally and economically sound alternative and probably one of the best strategies for the highest possible calcareous fly ash utilisation.

Keywords: calcareous type lignite fly ash, high sulphate, high volume blended cements, complying grade products.

1. INTRODUCTION

High calcium or calcareous (HCFA) fly ash is the combustion residue in electric power plants burning lignite or sub-bituminous coal at temperatures above 1100°C and collected by electrostatic or mechanical precipitation. This fine powder differs from the low calcium or siliceous fly ash (LCFA), since it is rich in reactive lime, CaO (not bound in CaCO₃ and CaSO₄) and sulfates, SO₃, while it also contains reactive silicon dioxide, SiO₂ and aluminum oxide, Al₂O₃. HCFA exhibit hydraulic and pozolanic properties and are referred to as self-cementing materials [1, 2]. The current HCFA utilization comprises high value applications (i.e. Portland cement and concrete), in which the Portland cement clinker is partially replaced by fly ash, and low value ones, in which HCFA is employed as filler in geotechnical or other engineering applications [3, 4].

In Europe, the use of HCFA in blended type cement production is prescribed in EN 197. The specifications of fly ash (both types) for the main engineering applications are prescribed by EN 14227 for hydraulically bound mixtures and EN 13282 for hydraulic road binders. The European

standard for fly ash in concrete, EN 450 is limited to silicious fly ash only (LCFA). High calcium fly ashes are reactive materials that often do not meet the limits of the standards [5, 6, 7]. Efforts to include HCFA into the European standards failed because of some bad experiences with the material in the past and many doubts about the performance in concrete [7].

It is obvious that the economy and environmental protection policy is hampered by the lack of relevant standards for the use of HCFA in concrete products. All of knowledge and experience in using HCFA(s) that have been accumulated for decades has not been transferred into practice and up to now the utilization of HCFA in concrete is not covered by any European standard. Some countries have proceeded by establishing national specifications. In Spain a national standard was issued in 1991, the UNE 83429 "Concrete additions for fly ashes with CaO content in excess of 10%". The example was followed by Greece in 2007 with the Hellenic specification for HCFA, which specifies two categories of fly ash (table 1). The two categories prescribe different limits regarding fineness, given as residue on a 45µm (ROS 45µm), sulphate (SO₃) and free lime contents (CaO_{free}).

Table 1 - Categories of HCFA(s) according to Hellenic specification

	R45 (ROS 45µm)	SO ₃	CaO _{free}
EIT1	45%	7%	-
EIT2	30%	5%	3%

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The testing of EIT1 calcareous fly ash as a constituent for hydraulically bound mixtures was very encouraging for the use of this fly ash in soil stabilization [8]. The EIT2 category, which is an upgraded fly ash, is limited to non reinforced concrete applications. National certifications for the use of HCFA in concrete and road construction have also been issued in Poland. The variability in lime and sulphate of HCFAs is a matter of great concern, because these constituents are often suspected for poor performance of fly ash-cement systems. The problems with calcareous fly ashes refer to setting time, high water demand, low early strength and volume stability. The lime content and particularly the free lime, the sulfate content and fineness seem to be the most influential characteristics of calcareous fly ashes. Different processing of improving the quality of HCFA have been developed and used, such as; sieving for

reducing grain size and unburnt carbon content and milling or milling plus hydrolysis for reducing the free lime content [8]. This research work is about the elaboration of suitable approaches for the production and safe utilization of EIT2 product (based on Hellenic specifications) as well as of high volume fly ash (HVFA) blended cements by employing various local fly ashes at the maximum possible content.

2. EXPERIMENTAL

2.1. Materials

2.1.1. Clinker

The industrial clinker, which was employed to produce blended cements by co-grinding, was provided by a local cement plant. Its chemical and phase compositions are given in table 2.

Table 2 - The chemical and phase composition of the clinker (main oxides and phases).

Property	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	CaO free	MgO	SO ₃	Loss on ignition	C ₃ S	C ₂ S	C ₃ A	C ₄ AF
Content [%]	22.4	5.8	3.3	65.2	1.1	1.8	0.6	0.4	58.2	15.6	10.8	9.1

2.1.2. Fly ash

Representative high calcium fly ash samples HCFA samples from different units of four thermal power plants (TPP) and a LCFA sample from another TPP have been employed for the experiments. The samples were collected according to a sampling plan and during a one month period. A

SEM image obtained on a HCFA sample is shown in figure 1 (left) together with a SEM image of the low calcium fly ash (LCFA) sample (right). Differently from the silicious LCFA sample which has spherical shaped particles, the HCFA sample has particles of irregular shape and variable sizes.

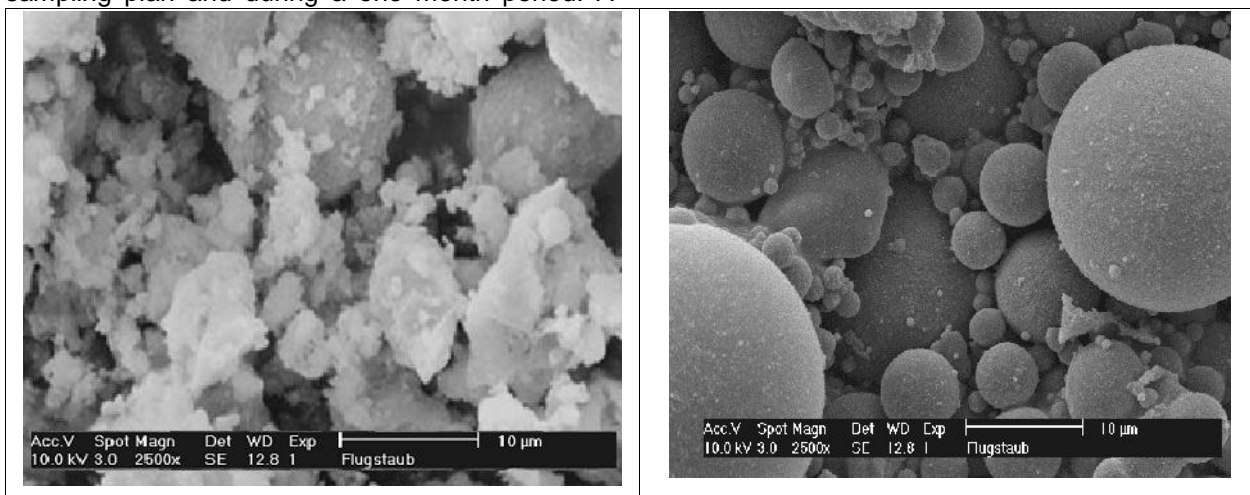


Figure 1 - SEM images of a calcareous (HCFA) and silicious (LCFA) sample.

The average chemical composition of representative samples from the five TPPs under study is shown in table 3. Based on the chemical composition and the European standard for fly ash, the fly ash of TPP A is silicious of LCFA type and those

of TPPs from B to D are calcareous types of HCFAs.

The SO₃ and CaO_{free} contents of the as received fly ashes were tested based on the relevant standards and methods and are given in table 4.

Table 3 - Average chemical composition of as received fly ashes (averaged yearly values)

Content (% weight)	TPP A	TPP B	TPP C	TPP D	TPP E
CaO	7.02	31.3	46.4	38.7	27.69
SiO ₂	48.72	33.3	26.8	32.3	36.89
Al ₂ O ₃	21.19	17.7	10.3	11.3	12.12
Fe ₂ O ₃	12.40	4.97	6.14	7.43	8.26
MgO	2.01	4.68	4.37	2.77	3.20
K ₂ O	2.91	1.18	0.78	0.93	1.87
Na ₂ O	1.20	0.72	0.26	0.49	2.48
TiO ₂	1.42	0.11	0.53	0.70	0.85
P ₂ O ₅	0.22	0.12	-	0.31	0.30
Other oxides	2.91	5.92	4.42	5.07	6.34

Table 4 - SO₃ and CaO_{free} contents of as received fly ashes

TPP	Unit	Sample code	SO ₃ (%)	Ca (OH) ₂ + CaO _{free} (%)	Ca (OH) ₂ (%)	CaO _{free} (%)
A	I	AI	0.57	0.42	-	0.42
B	I	BI	7.10	7.10	0.53	6.57
	II	BII	8.58	7.79	1.21	6.58
	III	BIII	8.85	8.43	2.78	5.65
	IV	BIV	6.22	8.53	2.18	6.35
C	I	CI	5.35	18.85	3	15.85
	II	CII	6.10	18.9	3.7	15.2
	III	CIII	5.10	19.5	3.5	16
	IV	CIV	5.41	19.3	3.5	15.8
D	I	DI	5.62	10.93	2.09	8.84
	II	DII	5.69	10.32	2.28	8.04
	III	DIII	4.89	10.51	2.35	8.16
	V	DIV	4.96	10.4	2.61	7.79
E	I	EI	5.79	7.79	7.78	0.01

Table 5 - Residue on sieve (ROS) results of as received fly ashes

TPP	Unit	Sample code	ROS (%)	
			45 µm	90 µm
A	I	AI	49.5	29.0
B	I	BI	32.5	13.1
	II	BII	26.4	10.3
	III	BIII	24.1	8.1
	IV	BIV	42.0	20.9
C	I	CI	30.2	14.5
	II	CII	33.3	15.9
	III	CIII	33.3	16.0
	IV	CIV	36.0	17.1
D	I	DI	41.8	20.7
	II	DII	42.5	21.0
	III	DIII	46.0	26.8
	V	DV	40.0	17.7
E	I	EI	42.0	25.5

In table 5 are shown the residue on sieve (ROS) values for all as received fly ashes.

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 $20 \pm 1^\circ\text{C}$ 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 $20 \pm 1^\circ\text{C}$ 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 $20 \pm 1^\circ\text{C}$ by an immersion heater and separated refrigerator unit. The prism-shaped specimens were demoulded after 24 hours \pm 15 min and then stored in water at $20 \pm 1^\circ\text{C}$.

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

In order to overcome the problems with fluctuations in composition, high water demand problem and volume stability, it was decided to proceed either with mixing, grinding, grinding and water spraying (hydrolysis), or a combination of those as

was deemed necessary, in order to reduce the size and the CaO_{free} of the as received fly ashes.

Based on the limitations for SO_3 and CaO_{free} put by the Hellenic specifications for HCFA (below table), only seven fly ash samples were employed to produce EIT2 grade products.

	R45 (ROS 45 μm)	SO_3	CaO_{free}
EIT1	45%	7%	-
EIT2	30%	5%	3%

The employed samples were as follows:

- CU II
- CU II + 3% H_2O^*
- CU II + 4% H_2O
- CU II + 5% H_2O
- DU V
- DU V + 2% H_2O
- DU V + 3% H_2O

*- (% weight based on fly ash amount)

Two other samples were employed to produce EIT2 grade fly ash product with blending and co-grinding of fly ashes, namely:

- EU I 75% + AU I 25%
- DU V 25% + AU I 75%

Seven fly ash samples were employed to produce blended cements, either by co-grinding or by simultaneous co-grinding and hydrolysis, namely:

- AU I 50% + clinker 50%
- EU I 50% + clinker 50%
- DU V 50% + clinker 50%
- DU V 50% + clinker 50% + 3% H_2O
- DU V 50% + clinker 50% + 4% H_2O
- BU IV 50% + clinker 50%
- CU II 50% + clinker 50%

3. RESULTS AND DISCUSSION

3.1. EIT2 products

3.1.1. EIT2 production by blending and co-grinding of fly ashes

After a careful study of the properties of the as received fly ashes, we have chosen two ways to produce EIT2 grade fly ash that comply with the Greek national specifications;

- blending and co-grinding of as received fly ashes and the other processing way was
- simultaneous co-grinding and hydrolysis of fly ash from the same source.

The results for ROS45, CaO_{free} and SO_3 contents of the two blends shown in tables 6-8 confirm our theoretical calculations for the production of EIT2 grade fly ash. Both blends produce complying grade EIT2 products (table 9).

Table 6 - SO_3 and CaO_{free} results for EIT2 produced by blending and co-grinding of fly ashes

Sample code	SO_3 (%)	Theoretical SO_3 (%)	$Ca(OH)_2 + CaO_{free}$ (%)	$Ca(OH)_2$ (%)	CaO_{free} (%)	Theoretical CaO_{free} (%)
EU I 75% + AU I 25%	4.37	4.48	3.81	2.97	0.84	0.11
DU V 25% + AU I 75%	1.68	1.67	1.79	-	1.79	2.26

Table 7 - ROS (45 μm) results for EIT2 produced by blending and co-grinding of fly ashes

Sample code	ROS 45 μm (%)
EU I 75% + AU I 25%	7.5
DU V 25% + AU I 75%	6.0

Table 8 - Compliance for EIT2 grade products produced by blending and co-grinding of fly ashes

	SO_3 (%)	EIT2 standard req. (max. %)	CaO_{free} (%)	EIT2 standard req. (max. %)	ROS 45 μm (%)	EIT2 standard req. (max. %)	Complying
EU I 75% + AU I 25%	4.37	5	0.84	3	7.5	30	Yes
DU V 25% + AU I 75%	1.68	5	1.79	3	6.0	30	Yes

3.1.2. EIT2 production by CaO_{free} reduction through hydrolysis and co-grinding of fly ashes

In order to produce EIT2 grade fly ash through hydrolysis and co-grinding process, two high free lime fly ashes was chosen to study the extent of free lime reduction via the hydrolysis process. The results of tables 9-10 indicate that up to 3% water is required to reduce the free lime content of the chosen fly ashes and produce complying grade EIT2 product (table 10). The percentage of CaO_{free} that can be converted into $Ca(OH)_2$ is in good agreement with the stoichiometrically calculated amount. These results proved also that the theoretical calculations and assumptions were accurate to a satisfactory degree and that combined grinding

and hydrolysis is an efficient approach for reducing the free lime content to acceptable levels.

Table 9 - CaO_{free} reduction and dependence on the sprayed water quantity

Sample code	$Ca(OH)_2 + CaO_{free}$ (%)	$Ca(OH)_2$ (%)	CaO_{free} (%)
CU II	18.90	3.7	15.2
CU II + 3% H_2O	14.02	2.18	11.84
CU II + 4% H_2O	13.22	2.71	10.51
CU II + 5% H_2O	13.04	3.01	10.03
DU V	10.4	2.61	7.79
DU V + 2% H_2O	7.04	2.33	4.71
DU V + 3% H_2O	6.25	3.7	2.55

Table 10 - Compliance for EIT2 products produced by water spraying and co-grinding of fly ashes

Sample code	SO_3 (%)	EIT2 standard req. (max. %)	CaO_{free} (%)	EIT2 standard req. (max. %)	ROS45 μm (%)	EIT2 standard req. (max. %)	Complying
CU II + 3% H_2O	6.10	5	11.84	3	n.m	30	No
CU II + 4% H_2O	6.10	5	10.51	3	n.m	30	No
CU II + 5% H_2O	6.10	5	10.03	3	n.m	30	No
DU V + 2% H_2O	4.96	5	4.71	3	8.3	30	Yes
DU V + 3% H_2O	4.96	5	2.55	3	9.8	30	Yes

n.m – not measured

3.2. Blended cement products produced by co-grinding process

The results for the cements produced either by co-grinding or co-grinding and hydrolysis are represented in tables 11-13. The values of ROS45,

SO_3 , CaO_{free} , expansion and strength qualify the produced cements to fulfill the requirements of the European standard for cements. The fly ash content of these cements can be as high as 50% without any risk for expansion or other failure.

Further it can be said that no hydrolysis is needed in order to produce the blended HVFA cement, even when the initial CaO_{free} content of raw fly ash is up to 16%, since soundness of all cements is within the limits specified by the standard. Some of

the produced cements belong to the Pozzolanic type (Cem IV/B-W) based on the European standard for cements. Also all cements have setting time values within the limits specified by this standard.

Table 11 - SO_3 and CaO_{free} results of cements produced by co-grinding

Sample code	SO_3 (%)	Theoretical SO_3 (%)	$\text{Ca}(\text{OH})_2 + \text{CaO}_{\text{free}}$ (%)	$\text{Ca}(\text{OH})_2$ (%)	CaO_{free} (%)	Theoretical CaO_{free} (%)
AU I 50% + clinker 50%	1.26	1.74	7.46	5.38	2.08	0.96
EU I 50% + clinker 50%	3.07	4.34	8.02	6.94	1.08	0.76
DU V 50% + clinker 50%	2.99	3.93	5.61	2.8	2.81	4.64
DU V 50% + clinker 50% + +3% H_2O	3.93	3.93	6.4	5.26	1.14	1.3
DU V 50% + clinker 50% + +4% H_2O	3.93	3.93	5.21	4.35	0.86	0.24
BU IV 50% + clinker 50%	3.28	4.56	6.35	2.23	4.12	3.92
CU II 50% + clinker 50%	2.41	4.5	12.25	2.52	9.73	8.35

Table 12 - Main parameters of cements produced by co-grinding of clinker with as received fly ashes

Sample code	ROS (%)		Setting time (min)		Expansion (mm)	Compressive strength (MPa)		
	45 μm	90 μm	Initial	Final		2 days	7 days	28 days
AU I 50% + clinker 50%	6.5	0.2	265	340	0.5	11.31	23.84	45.95
EU I 50% + clinker 50%	7.0	0.3	110	170	1.5	13.85	30.3	48.52
DU V 50% + clinker 50%	12.6	0.6	210	265	2	7.88	19.10	38.22
DU V 50% + clinker 50% + 3% H_2O	12.5	0.6	215	270	1	9.95	19.17	37.16
DU V 50% + clinker 50% + 4% H_2O	8.5	0.1	165	200	1	21.03	32.44	47.32
BU IV 50% + clinker 50%	10.0	0.1	215	305	0.5	22.47	44.05	60.44
CU II 50% + clinker 50%	11.7	0.1	110	160	5	24.26	38.72	58.04

Table 13 - Compliance of cement products produced by co-grinding of clinker with as received fly ashes

Sample code	SO_3 (%)	Cement standard req. (max. %)	CaO_{free} (%)	Expansion (mm)	ROS45 μm (%)	Complying
AU I 50% + clinker 50%	1.26	4	2.08	0.5	6.5	Yes
EU I 50% + clinker 50%	3.07	4	1.08	1.5	7.0	Yes
DU V 50% + clinker 50%	2.99	4	2.81	1	8.5	Yes
DU V 50% + clinker 50% + +3% H_2O	3.93	4	1.14	2	12.6	Yes
DU V 50% + clinker 50% + +4% H_2O	3.93	4	0.86	1	12.5	Yes
BU IV 50% + clinker 50%	3.28	4	4.12	0.5	10.0	Yes
CU II 50% + clinker 50%	2.21	4	9.73	5	9.7	Yes

In table 14 are compared the bulk densities and specific surface areas (Blaine) of a selected HVFA cement with two commercially available cements. The data of table 14 show that bulk density and specific surface area of the laboratory produced cement are well comparable to these of commercial cements.

Finally in figures 2 and 3 are shown the particle size distribution curves (cumulative percentages) of two selected HVFA cements. The fact that the size distribution curves follow same trend indicate that the milling has a relatively good reproducibility on grinding performance.

Table 14 - Bulk density and specific surface area (Blaine) of a HVFA and two commercial cements

Sample code	Density (g/cm ³)	Specific surface (cm ² /g)
Cem II/B-M	3.06	5132
Cem II/A-L	3.09	4564
50% DU V+ 50% clinker	2.94	5128

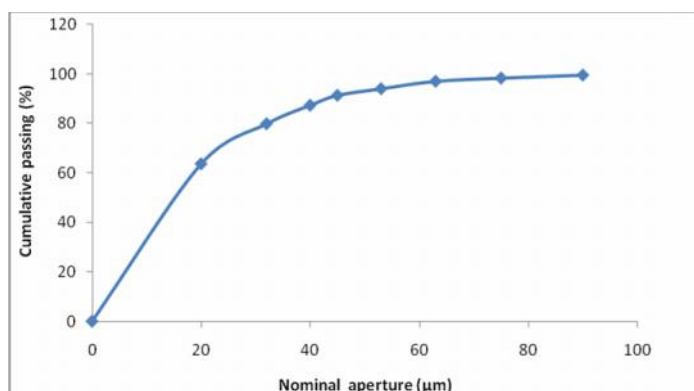


Figure 2 - Particle size distribution of the HVFA cement produced with 50% DU V +50% clinker.

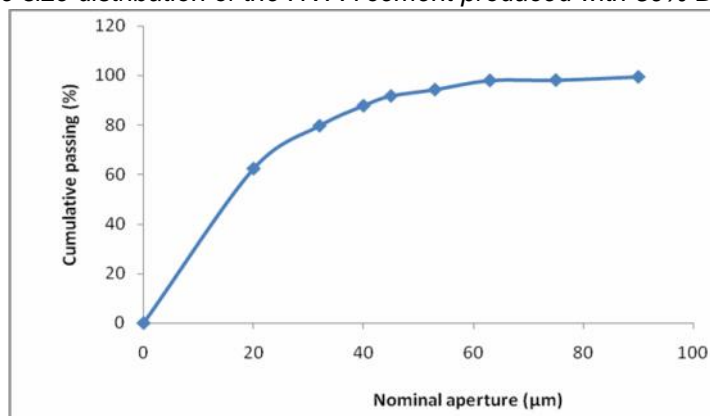


Figure 3 - Particle size distribution of the HVFA cement produced with 50% CU V +50% clinker.

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.

3.3. Gamma spectroscopic analysis for radioactivity of raw fly ashes

The homogenized samples were measured by gamma spectroscopy with an overclean germanium scout of 70% corresponding performance.

The duration of the measurement was > 6h. Artificial radionuclides were not detected. Radionuclides which were detected belong to the natural radioactivity. The minimum tracing limits were determined by the Curie method in a level of confidence 95%. The activity concentration indexes (I) were calculated using the following formula: $I = (C_{Ra-226}/300Bq \cdot kg^{-1}) + (C_{Th-232}/200Bq \cdot kg^{-1}) + (C_{K-40}/3000Bq \cdot kg^{-1})$. The results for I-indexes are presented in table 15.

Taking into account the stringiest dose criterion (0,3 mSv a⁻¹) the maximal I-index must be limited to 0.5 for concrete. Under these circumstances all HVFA cements that contain up to 50% fly ash comply with the limits for radionuclides content, therefore all cements are safe to be employed for production of concrete as final building material.

Table 15 - Activity concentration indexes for raw fly ashes and concrete that contains up to 15% HVFA cement

	TPPA	TPPB	TPPC	TPPD	TPPE
The activity concentration index (I)					
- raw fly ash	3.78	1.99	2.47	4.37	1.29
- concrete*	0.49	0.26	0.32	0.57	0.17
*- values calculated based on concrete with approx. 15% HVFA cement containing up to 50% fly ash.					

4. CONCLUSIONS

The research work was about producing complying grades fly ash as well as composite cements using as much as possible calcareous fly ash (HCFA). The EIT2 grade fly ash products complying with the requirements of the Greek national specifications can be produced either by blending and co-grinding of raw fly ashes or combined co-grinding and hydrolysis. The approach to follow depends on the CaO_{free} content of raw fly ashes. The amount of water to be sprayed and the reduction of CaO_{free} follow the figures calculated stoichiometrically. The employment of higher amounts of water do not result in higher percentage of CaO_{free} converted into $\text{Ca}(\text{OH})_2$. Blended cements of type Cem IV/B-W with as high as 50% of fly ash without any risk for expansion or other failure could be produced. The results indicate that no hydrolysis is needed in order to produce the blended HVFA cement, even when the initial CaO_{free} content of raw fly ash is up to 16%, since soundness of all cements is within the limits specified by the standard. Provided an accurate and strict quality management system is available, calcareous fly ashes can easily and safely be employed at up to

50% ratio in blended cements that comply with the European standard requirement for cement. This processing way is an environmentally and economically sound alternative and probably one of the best strategies for the highest possible calcareous fly ash utilization.

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IZVOD

OBRADA PEPELA KRE NJA KOG UGLJA ZA PROIZVODNJU CEMENTA I POŠTOVANJE KVALITETA PROIZVODA KOJI SE KORISTE U GRA EVINARSTVU

Ovaj rad prikazuje rezultate istraživanja u cilju razvijanja i vrednovanja rada prera enog pepela. Sirovine „leteci pepeo“ su veoma grube, imaju promenljiv hemijski i mineraloški sastav i sadrže ve e koli ine od dozvoljenih sulfata i slobodnog kre a. Osim toga, lete i pepeo sadrži aktivne radionuklide (^{226}Ra , ^{232}Th , ^{40}K), koji poti u iz uglja. Kre nja ki tip uzorka lignita pepela, koji su obezbe eni iz raznih elektrana, su obra eni na na in koji omogu avaju najviši mogu i bezbedonosni odnos iskoriš enosti pepela u cementu i pojedinim gra evinskim aplikacijama. Obrada se sastojala uglavnom u mešanju i / ili ko-brušenju, gde je ko-brušenje kombinovano brušenje i hidroliza kako bi se smanjili sulfati i sadržaj kre a u mešavinama. Obezbe ivanje ta nog i strogog sistema upravljanja kvalitetom je dostupno i kre nja ki lete i pepeo može da se lako i bezbedno koristi do 50% u pomešanom cementu, koji se u skladu sa evropskim standardom zahteva za cement. Ovakav na in obrade je ekološki i ekonomski opravdan i verovatno jedan od najboljih strategija za mogu e koriš enje kre nja ki lete eg pepela.

Ključne reči: kre nja ki tip lignita pepela, visok obim umešanosti cementa, sulfati, kvalitet proizvoda

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