Utilization of ferroalumina as raw material in the production of Ordinary Portland Cement

I. Vangelatos [a], G.N. Angelopoulos [a,*], D. Boufounos [b]

[a] *Laboratory of Materials and Metallurgy, Department of Chemical Engineering, University of Patras, 26500 Rio, Greece*
[b] *Aluminum Hellas, Saint Nicolas Plant, Paralia Distomou, 32003 Boeotia, Greece*

ABSTRACT

Red mud (RM), the digestion by-product of bauxite processing from "Aluminum Hellas", was dewatered by means of a filter press. The filtrand with water content from 28 to 32 wt% was named ferroalumina (FA). In order to utilize it as a raw material in the production of Ordinary Portland Cement (OPC), mixtures were prepared with limestone, sandstone and 1, 3 and 5 wt% FA addition, respectively. The design of the mixtures was based on the cement's compositional indexes LSF, AM and SM. Burnability tests showed that less than 1 wt% free lime can be obtained in all cases for firing at 1450 °C, except for the mixture with 1 wt% FA addition, which requires 1550 °C. XRD analysis and optical microscopy examination showed that FA addition did not affect the mineralogical phases of the produced clinkers. The characterisation of the produced Portland cements indicated that differences on surface area, water requirement and setting time are negligible. Compressive strength results after 28 days of curing varies from 55 to 63 MPa, which ranks the produced cements in CEM I 42.5N category. More specifically, the cements with FA addition due to their relatively high 2-day strengths (>20 MPa) can be ranked in CEM I 52.5N category. Addition of FA increases the amount of water-soluble chromium proportionally to the amount of total chromium to the mixture; however, conversion of total Cr to hexavalent Cr remains practically constant, in the range of 32–35 wt%. The results indicate that FA can be used as raw material in the production of OPC up to 5 wt% according to the chemical composition of the other raw materials.

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1. Introduction

Red mud (RM) is the by-product of the digestion of bauxite ores with caustic soda in autoclaves according to the Bayer process, in order to produce alumina. Its constituents are mainly Fe_2O_3 and Al, Ca, Si, Ti and Na oxides distributed in various mineral phases. It contains also small quantities of V, Cr, Mn, Cu, Cd, Ni, Zn, Pb, Mg and K oxides as well as rare earth elements [1].

The worldwide alumina industry produces annually 70 Mt of RM [2,3], whilst the amount for Greece is 0.7 Mt. RM is considered as a waste material and is disposed in mud lakes in the form of slurry impoundment or is dry stack in ponds near alumina plants or into a nearby sea. The latter is the case of Greece, where the RM is discharged through a pipeline into Antikyra Bay on the northern shoreline of the central Gulf of Corinth. RM fineness, thixotropic nature, alkalinity and trace metal content are some of the issues for land and sea disposal, which are today's main practices [4]. A significant amount of research has been carried out during the last years in order to develop environmental-friendly procedures for

the disposal [2] as well as for the utilization of RM. In the latter, field applications have been proposed concerning the water and soil treatment [5,6], the use as a catalyst [7] as well as the production of building and structural materials [8–12].

Cement industry, in particular, has already utilized with success a large number of waste materials for clinker production such as metallurgical slags, sludge from waste water treatment, glass, mining and municipal wastes [13–16]. Regarding the utilization of RM in the cement production, published work exists in the area of special and iron-rich cements [1,9] as well as of the Ordinary Portland Cement (OPC) [17,18]. In the case of [17], the addition of RM was 3.5 wt% and the results indicate that the physical and mechanical properties of the produced cement were similar to those obtained from the reference cement. In the case of [18], the addition of RM was 2.5 wt% and the produced cement presented similar physical properties and an improvement in the early day strengths comparing to the reference one. The main problem for the use of the RM slurry as a raw material in the cement industry is the high content in water (∼400 g/l solids), which increases the energy demand and makes difficult its handling and its transportation.

In Greece, a research project EPAN No. 12252/19-11-02, supported by the General Secretariat of Research and Technology (GSRT), was carried out aiming at the utilization of the RM as raw

material in the field of construction materials. The scope of the project was the dewatering of RM and the utilization of the filtrand in the existing production routes of the heavy clay and cement industries. The project results, led the company "Aluminium Hellas" on the installation of an industrial filter press in its integrated industrial complex. The press of the French company Choquenet has 760 m² active surface with 156 plates covered with double polypropylene filters. Its productivity is 350,000 t FA per year. Its water content varies from 26 to 28%. The cost of the investment was 2,150,000 €. The total cost for the processing of the entire quantity of the produced RM (700,000 t/year) is estimated to 7,000,000 €.

Considering that, 22 Mt of raw materials are used each year in Greece for the production of 15 Mt of OPC [19], additions up to 2–3 wt% in the raw meal will result in the utilization of the majority of the produced RM.

In this work, the results of the research performed in order to evaluate the utilization of the RM in the OPC manufacturing process are presented. RM was dewatered by means of a filter press. The filtrand was named ferroalumina (FA). Two series of experiments were carried out with FA addition up to 5 wt% in the raw meals. In the first series, the physicochemical properties of the produced OPC were evaluated, whereas in the second one, the levels of water-soluble chromium were measured. The design of the meals of both series of experiments was based on the widely applied in industrial practise LSF, AM and SM indexes.

2. Experimental

RM slurry with concentration of approximately 400 g/l was supplied by "Aluminium Hellas". The dewatering was performed by means of a filter press equipped with 20 synthetic fibre cloth filters of active surface 180 × 180 mm² operating at 0.55 MPa. In order to avoid settling of the slurry, the 200 l capacity supply tank was equipped with a two impeller 0.5 kW stirrer operating at 30 rpm, as shown in Fig. 1. After the dewatering procedure, the water content of the produced filtrand (FA), was determined by weighting four samples from different filters along the press, before and after drying at 110 °C. Chemical composition of FA was determined by atomic absorption spectroscopy, whilst its mineralogical composition was determined by X-ray diffraction (XRD) analysis. Other raw materials used for the preparation of the meals were limestone and sandstone, as CaO and SiO₂ carriers, respectively. The above raw materials were delivered by the Greek cement industry "TITAN". They were crushed and milled under 90 μm and their chemical composition was determined by X-ray fluorescence (XRF) analysis.

For the evaluation of the physicochemical properties of the OPC, raw meals with 1, 3 and 5 wt% FA addition were prepared. The design of the FA-containing meals was based on keeping the LSF index similar to the one of the reference mixture and the alumina (AM) and

silica (SM) modulus within the limits that are required for the production of OPC. The reference mixture was also delivered by "TITAN" cement industry. Particle size distribution was measured by a laser scattering analyzer, whereas the thermal behaviour was evaluated by DTA–TG analysis. Burnability tests were performed for the determination of free lime. Approximately 100 g of each mixture was fired at 1450, 1500 and 1550 °C for 40 min and the free lime content in each temperature was determined according to ASTM C114-03 [20]. The aim of these tests was to determine the temperature in which free lime content was below 1 wt%

For the sintering process, the meals with minimum water addition were shaped into 30 mm spherical pellets and preheated at 1000 °C for 4 h in order to remove the physically and chemically bounded water, the volatile organic matter and the CO₂ mainly from the dissociation of limestone. Subsequently, and according to the burnability test results, the pellets were fired in order to produce clinker. In all cases, an electric furnace with MoSi₂ heating elements was used. The chemical analysis of the clinkers was performed by XRF analysis. The microstructure of the clinker was evaluated by optical microscopy in polished sections after etching with 1% nital. Subsequently, the clinker was mixed and co-ground with 5 wt% gypsum. The specific surface of the so-produced cement was measured with a Blaine apparatus according to EN 196-6 [21]. The mineralogical composition of the samples was determined by XRD analysis. The setting time and the water requirement were determined by a Vicat needle apparatus and a Vicat probe, respectively, according to EN 196-3 [22]. Compressive strength of 40 × 40 × 160 mm³ mortar prisms at 2, 7, 28, 90, 180 and 360 days, was determined according to EN 196-1 [23].

As FA may contain up to 0.2 wt% Cr, the charge in water-soluble chromium of the produced cements was evaluated in a second series of experiments. In this case, the compositional indexes LSF, AM and SM of the raw meals were maintained similar to the reference one in order to ensure as possible the same mineralogical composition for all clinkers. This was accomplished by adding specific proportions of limestone, sandstone as well as FA up to 3 wt% to the reference meal. The meals were shaped as in the first series of experiments and were fired at 1450 °C. The produced clinkers were co-grounded with 5 wt% gypsum. Subsequently, the so-produced cements were mixed with water in a 10:1 liquid to solid ratio for 15 min followed by filtration. The chromium content of the water solution after filtration was determined by atomic absorption spectroscopy.

3. Results and discussion

3.1. Characterisation of raw materials and meals

FA cake obtained from the filter press had 32 wt% water content and 1.8 g/cm³ apparent density. XRF chemical analyses of FA, lime-
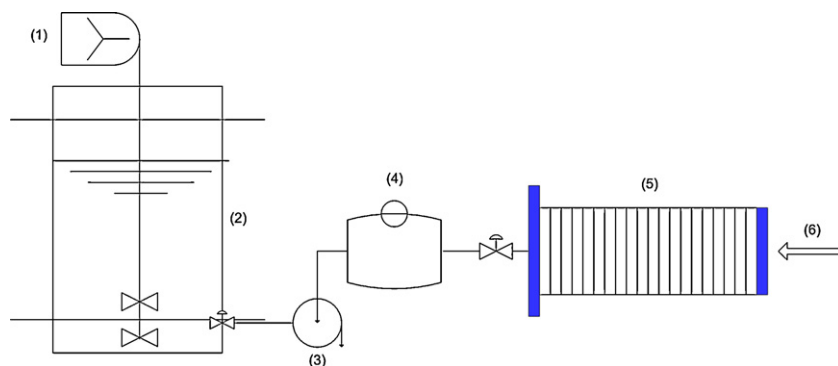
Fig. 1. Layout of laboratory filter press installation, (1) stirrer 0.5 kW operating at 30 rpm, (2) 200 l supply tank, (3) rotary pump, (4) 30 l feed tank operating at 0.55 MPa, (5) HAKU filter press with 20 synthetic fibre cloth filters of active surface 180 × 180 mm², (6) *P* = 30 Mpa.

Table 1
Chemical analysis of raw materials.

Oxides	Content of raw materials (wt%)		
	Limestone	Sandstone	Ferroalumina
SiO_2	7.14	73.53	10.70
Al_2O_3	1.26	15.54	18.70
Fe_2O_3	0.93	3.53	43.00
CaO	50.86	2.24	11.40
MgO	0.79	0.42	0.19
K_2O	0.16	1.44	0.06
Na_2O	0.15	0.31	3.53
TiO_2	ND	ND	4.16
Cr_2O_3	ND	ND	0.20
LOI	39.00	3.00	9.00
Total	100.29	100.01	100.94

ND, not determined.

stone and sandstone, after drying at 110 °C, are given in Table 1. XRD analysis of FA is presented in Fig. 2. The main mineralogical phases detected are hematite [Fe_2O_3], diaspore [AlO(OH)], gibbsite [$Al(OH)_3$], calcite [$CaCO_3$], calcium–aluminium–iron silicate hydroxide [$Ca_3AlFe(SiO_4)(OH)_8$], perovskite [$CaTiO_3$] and cancrinite [$Na_6Ca_2Al_6Si_6O_{24}(CO_3)_2\cdot2H_2O$]. The composition of raw meals as well as their chemical analysis, together with the compositional indexes LSF, AM and SM, are presented in Table 2. Increasing FA addition, the content of Al_2O_3 and Fe_2O_3 increases whilst the content of SiO_2 decreases. The meal with 3 wt% FA addition is comparable to the reference one. The meal with 1 wt% FA addition presents the highest AM and SM indexes whilst the one with 5 wt% FA, the lowest.

The DSC–TG curves are presented in Fig. 3. For the mixtures with FA, the DSC curves presents an endothermic peak at 270–280 °C, which is attributed to the removal of crystalline water from calcium–aluminium–iron silicate hydroxide and most probably sodium–aluminum silicate. Peaks at 340 and 510–540 °C, which were observed elsewhere [24], and correspond to the decomposition of gibbsite and diaspore, respectively, are not present due to the relatively low proportion of FA, up to 5 wt%, in the meals. For temperatures over 670 °C, the dissociation of limestone and other carbonate compounds initiates for all the mixtures and is concluded at about 870 °C.

TG results are in accordance with the DCS analysis. Up to approximately 670 °C a 2–3% weight loss is observed. An abrupt change in weight loss is observed between 670 and 870 °C due to the dissocia-

Table 2
Composition of raw meals.

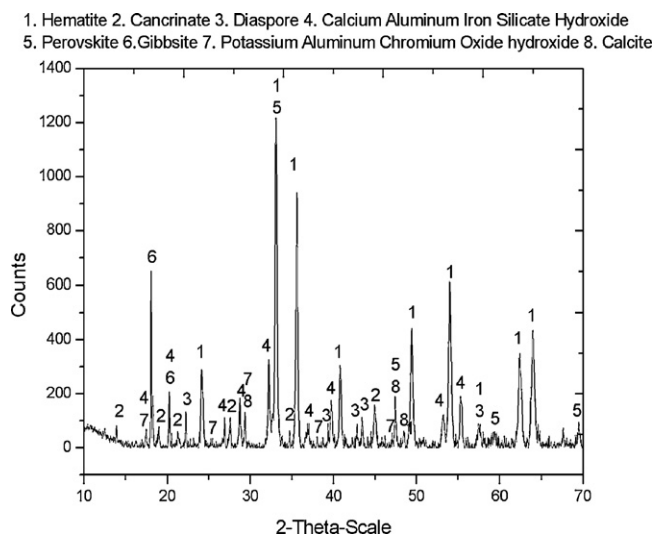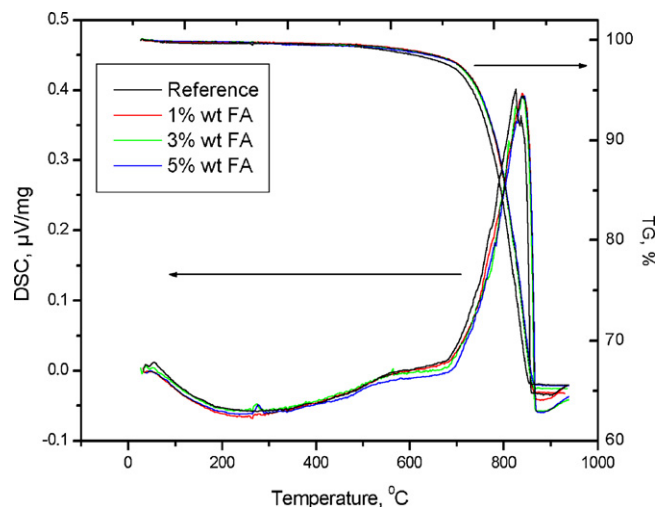|	wt% Reference	1 wt% FA	3 wt% FA	5 wt% FA
Raw materials				
Limestone		87.75	86.50	85.30
Sandstone		11.25	10.50	9.70
Ferroalumina		1.00	3.00	5.00
Oxides				
SiO_2	14.14	14.66	14.22	13.76
Al_2O_3	3.28	3.04	3.28	3.52
Fe_2O_3	2.50	1.64	2.47	3.29
CaO	44.15	44.99	44.57	44.17
MgO	0.69	0.74	0.73	0.72
K_2O	0.56	0.30	0.29	0.28
Na_2O	0.27	0.20	0.27	0.34
TiO_2	0.00	0.04	0.12	0.21
LOI	34.45	35.25	34.80	34.50
Total	100.04	100.87	100.76	100.79
LSF	97.92	98.44	98.41	98.55
AM	1.31	1.85	1.33	1.07
SM	2.45	3.13	2.47	2.02

tion of carbonates. At higher temperatures, a plateau is observed in all cases. Small differences in the overall weight loss were recorded reflecting the differences in calcite content in the samples. The reference sample having the lowest calcium oxide content presents the lowest weight loss value of 34.32 wt%, whilst the mixture with 1 wt% FA, having the highest calcium oxide content, presents the highest weight loss of 35.14 wt%.

The particle size distribution of all samples is depicted in Fig. 4. No significant differences are observed. All samples have practically similar particle size distribution.

The results of the burnability tests are presented in Fig. 5. In order to achieve free lime content less than 1 wt%, the reference meal as well as the ones with 3 wt% and 5 wt% FA required a firing temperature of 1450 °C. The meal with 1 wt% FA addition requires a higher temperature of 1550 °C. This is attributed to the fact that the alumina AM and silica SM modulus for 1 wt% FA are the highest of all meals (Table 2).

3.2. Clinker and cement characterisation

The chemical composition of the produced clinkers was determined by XRF analysis, whilst their mineralogical composition was derived applying Bogue's equations, Table 3. Alite (C_3S) content

1. Hematite 2. Cancrinate 3. Diaspore 4. Calcium Aluminum Iron Silicate Hydroxide 5. Perovskite 6. Gibbsite 7. Potassium Aluminum Chromium Oxide hydroxide 8. Calcite

Fig. 2. XRD spectrum of Ferroalumina.
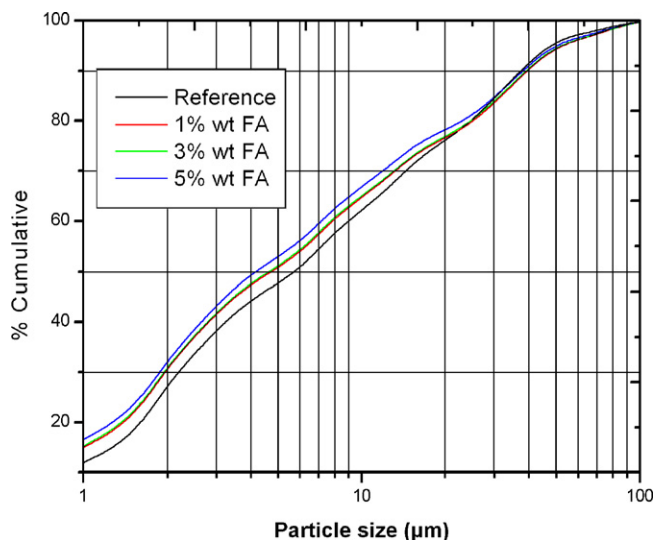
Fig. 3. DSC–TG analysis of raw meals.

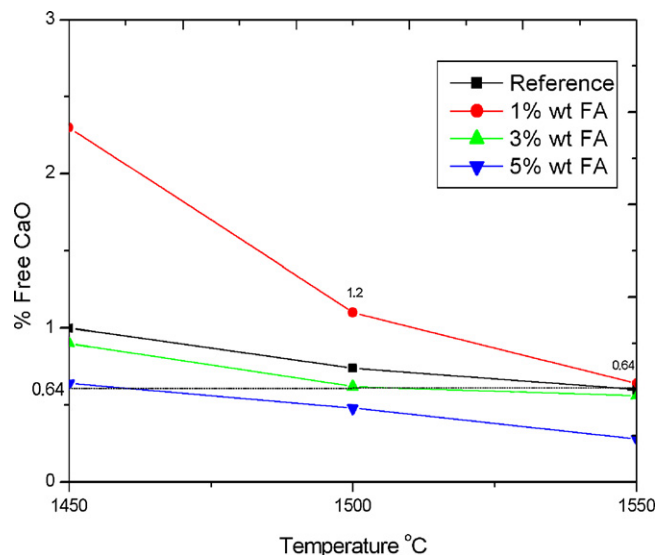**Fig. 4.** Particle size distribution of raw meals.

Fig. 5. Burnability test of raw meals.

Table 3
Chemical and mineralogical composition of the produced clinkers.

	wt% Range [13]	Reference	1 wt% FA	3 wt% FA	5 wt% FA
Oxides					
SiO_2	19–23	22.05	23.61	22.64	21.53
Al_2O_3	3.5–6.5	5.66	4.17	4.52	4.89
Fe_2O_3	2–4	3.38	1.94	3.17	4.40
CaO	62–68	67.12	67.45	66.93	66.07
MgO	–	1.60	0.83	0.82	0.81
K_2O	–	0.54	0.24	0.32	0.38
Na_2O	–	0.23	0.17	0.27	0.38
SO_3	–	0.22	0.08	0.13	0.16
Total	–	100.80	98.49	98.80	98.62
Mineralogical phases [8]					
% C_3S	42–67	62	64	65	66
% C_2S	9–30	16	19	16	12
% C_3A	6–14	9	8	7	6
% C_4AF	1–12	10	6	10	13

varies from 62 to 66 wt% for all meals. Belite (C_2S) content decreases with the increase of FA addition due to the decrease of SiO_2 content of clinkers. Calcium-aluminoferrite (C_4AF) increases with FA addition due to the increase of total Fe_2O_3. The clinker of the reference meal presents similar contents of C_2S and C_4AF to the one with 3 wt% FA addition. Moreover, due to its higher alumina content, it presents higher content of C_3A and lower content of C_3S.

The microstructure of the clinkers is presented in Fig. 6. Addition of FA did not affect qualitatively the formed mineralogical phases. The texture of all clinkers is similar. The matrix contains prismatic C_3S crystals, round C_2S crystals and interstitial phase. The bluish or tan subhedral and euhedral alite crystals exist in a variety of sizes. Differences in coloration occur most probably due to differences in crystallographic orientation, structural state and chemical composition. In several cases, alite crystals are strongly zoned. The latter is more noticeable in the case of reference sample. Moreover, alite contains inclusions of belite, which are more conspicuous in the case of the reference and 1 wt% FA samples. Belite crystals in most
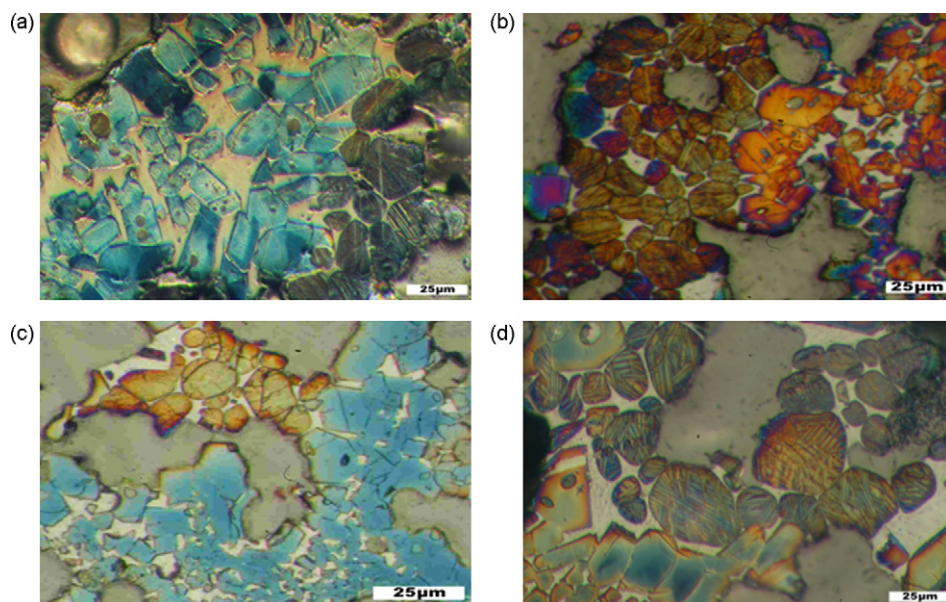
Fig. 6. Microstructure of Portland cement clinker; (a) and (b) reference and 1 wt% FA, respectively, strongly zoned prismatic alite crystals with inclusions of round belite, (c) 3 wt% FA, clustering of belite in interstitial melt, (d) 5 wt% FA round belite crystals with typical multidirectional lamellae.
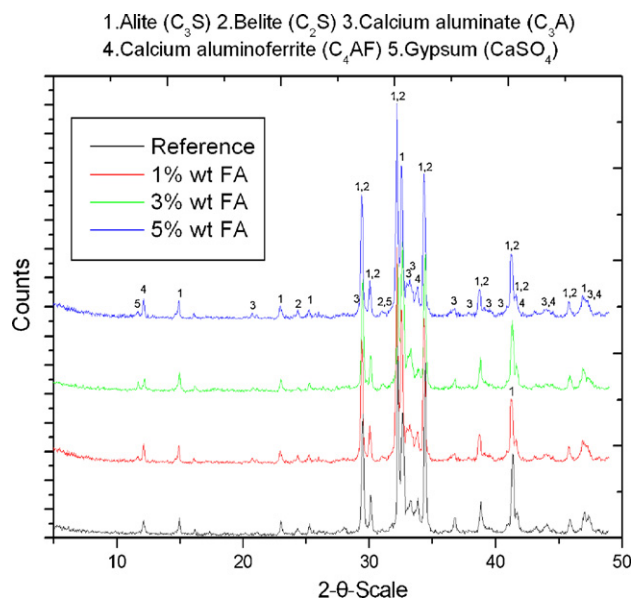
1.Alite (C_3S) 2.Belite (C_2S) 3.Calcium aluminate (C_3A)
4.Calcium aluminoferrite (C_4AF) 5.Gypsum ($CaSO_4$)

Fig. 7. X-ray diffraction of the produced Portland cements.

Table 4
Physical properties of the produced cements.

Physical properties	Reference	1 wt% FA	3 wt% FA	5 wt% FA
Specific Surface (cm^2/g)	3730	3810	4010	3870
Initial Setting Time (min)	100	100	80	90
Final Setting Time (min)	160	140	160	150
Water Demand (%)	21.6	22.4	22.6	22.2

cases occur in lumps. In the case of the reference, are brown-blue coloured, whereas in samples with FA, are brown-tan coloured. In all cases, they present typical multidirectional lamellae except for sample with 3 wt% FA addition, which shows minimal development of lamellae. The interstitial melt of relatively high proportion is better differentiated in the case of the sample with 3 wt% addition. At higher magnification, not presented herein, it can be noted that the interstitial melt is crystallized to a mixture of ferrite (C_4AF) and calcium aluminate (C_3A), that is, not of a dendritic texture.

All clinkers were co-ground with 5 wt% gypsum for 10 min, in order to produce OPC. The XRD analyses of the produced cements, Fig. 7, indicate that addition of FA did not affect the formation of the mineralogical phases. The FA addition cements present similar diffraction peaks to the reference one, with the phases C_3S, C_2S, C_3A, C_4AF and anhydrite to dominate.

The cements with FA present greater specific surface for the same grinding time and greater water demand than the reference one in order to acquire the desirable consistency. The setting time for all the produced cements varies between 40 and 80 min (Table 4). The compressive strength results for 2, 7, 28, 90, 180 and 360 days of curing, are displayed in Table 5. The results indicate that all the produced mortars may be ranked as Portland cements of 42.5N category according to EN196. More specifically, the pastes with FA addition can be ranked into a higher category, the 52.5N one, as their 2-day compressive strength are over 20 MPa.

3.3. Evaluation of FA addition on chromium charge

The design of the second raw meals series is presented in Table 6 whilst in Table 7, the total chromium content of the cement (Cr_{tot}), the water-soluble hexavalent chromium content (Cr^{+6}) as well as the chromium conversion defined as $Cr^{+6}/Cr_{tot} \times 100$ are presented, respectively. Total chromium, from 0.159 mg/g in the reference

sample, increases to 0.271 mg/g in the sample with 3 wt% FA. Water-soluble chromium increases too for the same samples, from 0.053 to 0.086 mg/g. It is observed that chromium conversion remains at similar levels from 32 to 35%. This value is higher than the 10–20% conversion that is reported in previous study [25]. For European member states, the limit of the water-soluble chromium in cement is 0.0002 wt% according to the directive 2003/53/EC of the EU Parliament. An excess of the above-mentioned limit can be affronted with measures already existing in the industrial practice such as use of a reducing agent such as ferrous sulphate [26].

4. Conclusions

RM was dewatered by means of a filter press. The filtrand named FA presents water content from 26 to 28%.

The addition of FA as a raw material in the raw meal for the production of cement is feasible up to the investigated 5 wt% according to the chemical composition of the other raw materials. FA addition does not affect the thermal behaviour of the meals. Burnability tests showed that free lime less than 1 wt% can be obtained for firing temperature of 1450 °C in all cases except for mixture with 1 wt% FA addition, which requires 1550 °C. XRD analysis and optical microscopy examination showed that FA addition did not affect qualitatively the mineralogical phases formed in the produced clinkers. Characterisation of the cements showed that differences on surface area, water requirement and setting time are negligible. Compressive strength after 2 days curing is over 20 MPa, whereas after 28 days, it varies from 55 to 63 MPa. These values rank the obtained FA cements in CEM I 52.5N category. Finally, the addition of FA increases the amount of water-soluble chromium proportionally to the amount of total chromium of the mixture.

Table 5
Compressive strength of the produced cements.

	CEM I [13]		Compressive strength (MPa)			
	42.5N	52.5N	Reference	1 wt% FA	3 wt% FA	5 wt% FA
2 days	≥10.0	≥20.0	19.2	26.5	27.4	30.8
7 days	NR	NR	39.2	48.2	42.9	44.3
28 days	≥42.5	≥52.5	55.6	62.9	57.5	58.4
90 days	NR	NR	57.5	69.6	65.2	63.0
180 days			60.2	71.2	67.8	68.3
360 days			64.3	73.1	69.8	70.2

NR, no requirement.

Table 6
Composition of raw meals, second series.

Raw material	% Raw meals constituents			
	Reference	1 wt% FA	2 wt% FA	3 wt% FA
Reference meal	100.00	97.80	55.00	55.00
Limestone	–	1.20	35.00	34.50
Ferroalumina	–	1.00	2.00	3.00
Sandstone	–	–	8.00	7.50
Total	–	100.00	100.00	100.00
LSF	96.21	96.12	96.30	96.32
AM	1.31	1.34	1.22	1.20
SM	2.45	2.42	2.25	2.19

Table 7
Conversion of initial Cr into water soluble in the produced cements.

Cr	Reference	1 wt% FA	2 wt% FA	3 wt% FA
Initial in raw meal (mg/g)	0.153	0.187	0.229	0.271
Water soluble in cement (mg/g)	0.053	0.066	0.072	0.086
% Conversion	35	35	32	32

Acknowledgements

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