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Prevention of electrofilter ash toxic leaching by embedding in composite materials

Disposal of the fly ash may pose a significant risk to the environment due to the leaching of hazardous pollutants. The only sustainable solution for the pollution-prevention is the reapplication of fly ash as one of the components in construction material composites. There is a risk of leaching even when fly ash is built-in the construction composites and the goal of this investigation was to prove that leaching concentrations of toxic elements is in range assigned by actual regulations. Fly ash was applied in several composites: mortar, concrete and asphalt. The leachability of the potentially toxic elements from the fly ash based products was investigated. The leaching behavior and potential environmental impact of the 11 potentially hazardous elements was tracked: Pb, Cd, Zn, Cu, Ni, Cr, Hg, As, Ba, Sb and Se. A detailed study of physico-chemical characteristics of the fly ash is included. The overall results showed that most of the elements are more readily leachable from the fly ash in comparison with the fly ash based composites.

Key words: leaching, toxic elements fly ash, reapplication, construction composites.

1. INTRODUCTION

The request for the adoption of the „reusing” principle in construction materials industry and environmental protection is alerting. This principle refers to waste minimization through planning and design: the final product of the process is incorporated back into the same cycle without additional waste production. The reusing process also refers to the recovery of waste material and transferring it into secondary raw materials [1, 2]. Ash, the by-product of the coal combustion, is one of the hazardous environmental polluters. The annual global fly ash production is over 600 million tons which makes this by-product a serious problem with severe implications for the environment [3-5].

Regulations and contemporary investigations are focused on the recycling of the fly ash and reusing it as a component in construction materials [6]. Namely, the construction industry is the biggest “consumer” of recycled raw materials with reapplication rate of approximately 21 million tons of fly ash per year [5]. The high recycling rate is obtained mainly due to the fly ash pozzolanic behavior. Possibilities for fly ash reapplications are numerous: as bonding agent or aggregate in concrete/mortar [7-10], filler in road base [11], raw material in cement clinker production [12, 13], component in bricks and tiles [14, 15], as injection material, as geopolymers [16-18]. Even though fly ash has been utilized in cementitious

materials for years, there is constantly increasing amount of the unused fly ash. Such occurrence highlights the necessity of developing new recycling methods and products where significant quantity of this waste could be reused.

In addition to the fly ash processing and reapplication in new products, there is also an environmental impact that needs to be evaluated. Namely, there is possibility of toxic metals leaching which could pass through the soil into the ground water, especially when fly ash is used as a construction material. Fly ash may contain some elements of environmental concern, such as arsenic, barium, chromium, cadmium, lead, selenium and mercury, which can limit the potential applications [19-22].

In this study, fly ash based mortars were subjected to mechanical and structural analyses in order to investigate its preliminary characteristics and to evaluate its construction materials utilization potential and implementation safety.

2. MATERIALS AND METHODS

The investigated fly ash samples originated from the filter systems of five coal-fired power plants in Serbia: “Nikola Tesla - A”, “Nikola Tesla - B”, “Kolubara”, “Kostolac - A” and “Kostolac - B”. Fuels applied and power-plant regimes vary, therefore the fly ashes produced have different chemical and mineralogical compositions, as well as varieties in microstructure. Five types of fly ash are investigated (a sample from each power plant) and here subsequently labeled as FA_A, FA_B, FA_C, FA_D and FA_E.

The fly ash grain fraction content was analyzed by diffraction particle size analysis (Cyclo-sizer Warman International LTD, Australia). Grain size distribution of the fly ash samples is given in Figure 1.

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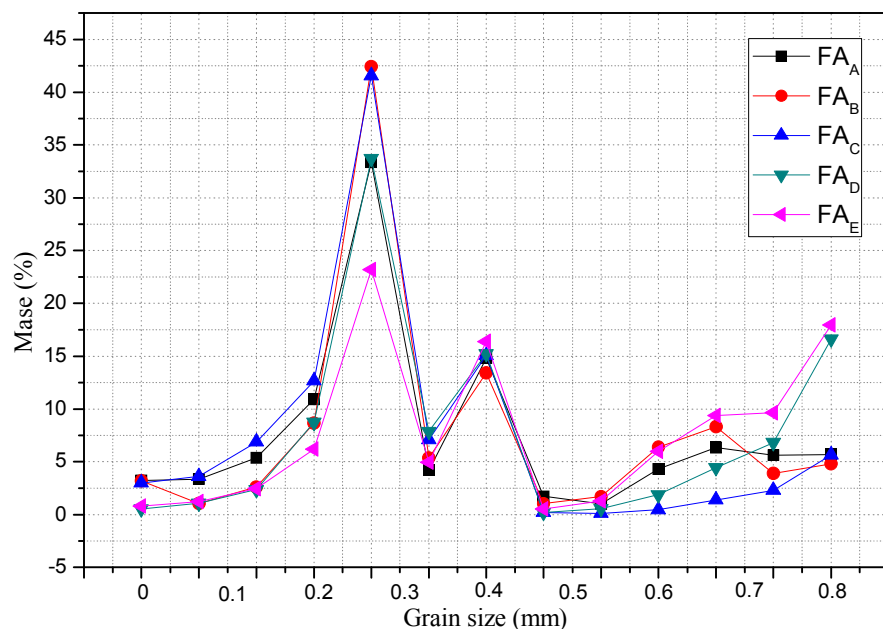


Figure 1 - Grain size distribution of the fly ash samples.

X-ray fluorescence (XRF) technique was used to conduct chemical element analysis. Analysis was performed on the XRF spectrophotometer ED 2000 - Oxford. Thermal stability of crystalline phases within fly ash samples and changes occurring with increasing temperature were investigated by means of the differential thermal analysis. The differential thermal analysis (DTA) of the fly ash was performed with a Shimadzu DTA – 50 apparatus. X-ray powder diffraction analysis was applied in investigation of mineral phases present in fly ash composition. Samples were analyzed by means of X-ray powder diffraction (XRD). The XRD patterns were obtained on a Philips PW-1710 automated diffractometer using a Cu tube operated at 40 kV and 30 mA. The microstructure of the fly ash samples and fly ash based composites was characterized by scanning electron microscopy method (SEM) using a JEOL JSM-6390 Lv microscope

Three different types of fly-ash based composites were prepared for the analyses: fly ash mortar, fly ash concrete and fly ash asphalt. Mix design of the composites was as follows:

- Mortar: binder - PC 42.5R “Lafarge”; aggregate - River aggregate - “Moravac”, grain size fractions 0/4 mm; fly ash replacement coefficient – 35 %
- Concrete: binder - PC 42.5R “Lafarge”; aggregate - River aggregate - “Moravac”, grain size fractions grain size fractions 0/4, 4/8, 8/16, 16/32 mm; fly ash replacement coefficient – 35 %
- Asphalt: binder Bitumen - BIT 60 “Pančevo”; aggregate - stone dust (4 %), fine fractions of

crushed aggregate (grain size 0/4 mm, 68 %), and coarse aggregate (grain size 4/8 mm, 28%); fly ash replacement coefficient – 35 %

Each composite had five variations, i.e. each composite was prepared with different fly ash type (FA_{A-E}), mix-design remaining the same. Concrete, mortar and asphalt composites without addition of fly ash were also prepared. Such composites were “standard” samples, made for comparison with fly ash based composites. The green concrete and mortar mixtures were mixed for 10 minutes in laboratory RILEM-cem mixer and, afterwards, shaped in 10 cm cubic moulds. After 7 days of curing in a climate chamber at 20 °C and humidity 60 %, the samples were demoulded and stored for another 21 days under the same conditions as in the climate chamber. For the asphalt samples, optimal mix design regarding assessment of bitumen as bonding agent was conducted according to the Marshall method: BIT = 6.2 % (m/m).

Trace elements mobility from the fly ash samples, fly ash based composites (concrete, mortar and asphalt samples) and “standard” composites was determined with batch leaching test (procedure conducted according to Standard EN 12457). Following the presumption that atmospheric water is the main resource of penetration through open fly ash landfill and building materials incorporated in exterior elements of structural objects or roads, the water was chosen as leachant. Composite specimens were milled in order to provide greater contact surface between the media and to ease the leaching of

present trace elements. The leaching test was performed at a liquid/solid ratio of 10 L/kg with a stirring time of 24 h and deionized water as the leachant. The content of the trace elements in the leachates were determined by means of inductively coupled plasma atomic emission spectrometer ICP-AES (Perkin-Elmer Optima 5300 DV). Results of the leaching test performed on the fly ash, fly ash based composites and "standard" composites were mutually compared.

3. RESULTS AND DISCUSSION

The fly ash samples chemical composition and physico-chemical characteristics are given in Table 1. and 2., respectively.

Table 1 - Chemical composition of the fly ash samples FA_{A-E}

Oxides (wt %)	FA _A	FA _B	FA _C	FA _D	FA _E
SiO ₂	53.28	56.20	59.39	54.32	59.73
Al ₂ O ₃	21.45	17.38	16.37	18.08	19.97
Fe ₂ O ₃	7.16	9.33	9.48	6.98	7.55
TiO ₂	0.51	0.50	0.52	0.57	0.57
CaO	7.61	7.96	6.96	9.71	4.83
MgO	2.74	2.28	1.98	3.3	2.21
P ₂ O ₅	0.03	0.03	0.02	0.02	0.02
SO ₃	0.78	0.85	1.06	1.29	0.48
Na ₂ O	0.44	0.4	0.31	0.5	0.41
K ₂ O	1.21	0.55	0.64	1.16	1.18
MnO	0.03	0.03	0.03	0.03	0.03
CO ₂	0.25	0.28	0.09	0.11	0.07
LoI	4.51	4.21	3.15	3.82	2.95

Table 2 - Physico-mechanical characteristics of the fly ash samples FA_{A-E}

Sample	FA _A	FA _B	FA _C	FA _D	FA _E
Bulk density (g/cm ³)	2.20	2.32	2.19	2.18	2.18
Compressive strength (MPa)	16.1	10.4	12.5	13.5	15.2
Flexural strength (MPa)	4.6	3.5	4.2	4.1	4.7
Average grain size (mm)	0.118	0.112	0.143	0.089	0.067

Averaged values of main oxides of all five investigated fly ash samples are lying in the range of the values of the fly ashes typical for other origins (Table 3.), which in certain way approves

applicability of Serbian fly ashes, since other countries already showed possibility of using fly ash in building products (SRPS CEN/TR 15677:2009).

Table 3 - Comparison of average chemical composition data for investigated fly ash and typical fly ashes from other origins

Average value (%)	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO
Serbia	56.25	19.98	8.09	7.41
UK	50.09	28.10	11.70	1.62
USA	52.24	19.01	15.71	4.48
Netherlands	50.46	25.74	6.53	4.32
Spain	49.80	17.30	8.70	24.90
Poland	50.80	23.90	8.60	3.60

The heating process of the fly ash generally comprises three separate regions that are monitored by three individual peaks. All DTA curves showed a small peak at approximately 200 °C. The first peak should be attributed to the evaporation of moisture. During temperature interval from 100 to 450 °C hydration water was completely removed. The second region occurring at the temperature range of 400–700 °C incarnates polymorphic transition. Exact peak values are as follows: (FA_A) 530.85 °C; (FA_B) 522.95 °C; (FA_C) 520.74 °C, (FA_D) 503.80 °C; (FA_E) 535.25 °C. Corresponding quantities of heat for each peak are: (Q_{FAA}) 2.77 kJ/g; (Q_{FAB}) 2.05 kJ/g; (Q_{FAC}) 4.13 kJ/g, (Q_{FAD}) 1.21 kJ/g; and (Q_{FAE}) 8.24 kJ/g. The third region represents the beginning of the fusion of fly ash. The peak at approximately 900 °C which is endothermic is induced by presence of aluminosilicates. Exact peak values are as follows: (FA_A) 926.91 °C; (FA_B) 928.41 °C; (FA_C) 871.25-915.17 °C, (FA_D) 928.53 °C. Corresponding quantities of heat for each peak are: (Q_{FAA}) -1,57 kJ/g; (Q_{FAB}) -1,98 kJ/g; (Q_{FAC}) -0,14/-1,51 kJ/g and (Q_{FAD}) -2,78 kJ/g. Melting is not recorded at the temperature 1100 °C for samples FA_{A-D}. This attributes to good thermal and refractory characteristics of fly ash as raw component material.

Major crystalline phases identified in the fly ash samples were: aluminosilicate glass, quartz and mullite. High amount of amorphous matter was present within all investigated fly ash samples. The main peaks on diffractograms relate to quartz. Magnetite, hematite, fluorite and anhydrite are present in relatively negligible amounts.

The water leaching trial gave closer picture of potential mobility of toxic elements existing in the fly ash composition (Cr, Ni, Cu, Zn, As, Se, Cd, Sb, Ba and Pb) in what can be called normal environment influenced only by atmospheric water – rain or snow. Results of the leaching test conducted on the fly ash,

fly ash based composites and “standard” samples are given in Tab 4-7., respectively. The results of the leaching trial performed on standard and fly ash based composites should be compared to the assigned toleration limits which are: 10 mg/l for Pb, 0.5 mg/l for Cd, 100 mg/l for Zn, 10 mg/l for Cu, 50 mg/l for Ni, 30 mg/l for Cr, 0.05 mg/l for Hg, 5 mg/l for As, 50 mg/l for Ba, 5 mg/l for Sb, and 5 mg/l for Se. Upper limit values for toxic metals found in leachate after leach test performed on potentially hazardous wastes used for reapplication/recycling here used are assigned by Serbian Standard EN 12457-(1-4)2002. Even though aluminium, calcium and iron are major constituents in the chemical composition of fly ash their leachability was not investigated due to following reasons: (1) aluminium is element which is poorly leached; (2) calcium is not regarded as an element of concern; (3) iron is mainly present as magnetite mixed in various proportions with hematite, although a minor proportion can be assimilated in the glassy matrix and iron releases are

of little significance in relation to the concentration in the fly ash.

Table 4 - Results of water leaching trial performed on raw fly ash samples

Toxic element (mg/l)	FA _A	FA _B	FA _C	FA _D	FA _E
Pb	0.04	0.05	0.04	0.07	0.03
Cd	-	-	-	-	-
Zn	0.679	0.66	0.674	0.693	0.964
Cu	0.008	0.005	0.009	0.01	0.009
Ni	0.18	0.175	0.185	0.187	0.196
Cr	0.133	0.127	0.129	0.139	0.128
Hg	-	-	-	-	-
As	0.004	0.003	0.009	0.002	0.004
Ba	-	-	-	-	-
Sb	0.038	0.003	0.004	0.001	0.003
Se	0.004	0.004	0.006	0.007	0.005

Table 5 - Results of water leaching trial performed on standard and fly ash based mortars

Toxic element (mg/l)	FA _A mortar	FA _B mortar	FA _C mortar	FA _D mortar	FA _E mortar	Standard mortar
Pb	0.09	0.04	0.12	0.08	0.06	0.04
Cd	-	-	-	-	-	-
Zn	0.647	0.955	0.647	0.656	0.667	0.662
Cu	0.005	0.002	0.007	0.006	-	0.012
Ni	0.178	0.148	0.189	0.179	0.172	0.173
Cr	0.132	0.08	0.127	0.133	0.085	0.151
Hg	-	-	-	-	-	-
As	0.003	0.008	0.008	0.003	0.001	0.001
Ba	-	-	-	-	-	-
Sb	0.01	0.086	0.012	0.005	0.004	0.01
Se	0.004	0.013	0.005	0.008	0.006	0.007

Table 6 - Results of water leaching trial performed on standard and fly ash based concretes

Toxic element (mg/l)	FA _A concrete	FA _B concrete	FA _C concrete	FA _D concrete	FA _E concrete	Standard concrete
Pb	0.09	0.17	0.15	0.08	0.19	0.17
Cd	-	-	-	-	-	-
Zn	0.701	0.693	0.667	0.729	0.699	0.677
Cu	0.015	0	0.014	0.017	0.002	0.015
Ni	0.199	0.153	0.182	0.222	0.172	0.186
Cr	0.145	0.122	0.143	0.155	0.145	0.151
Hg	-	-	-	-	-	-
As	0.019	0.007	0.018	0.009	0.005	0.006
Ba	0	0	0	0	0.086	0.09
Sb	0.008	0.009	0.032	0.008	0.005	0.008
Se	0.009	0.011	0.005	0.015	0.004	0.005

Table 7 - Results of water leaching trial performed on standard and fly ash based asphalts

Toxic element (mg/l)	FA _A asphalt	FA _B asphalt	FA _C asphalt	FA _D asphalt	FA _E asphalt	Standard asphalt
Pb	0.08	0.15	0.16	0.1	0.19	0.17
Cd	-	-	-	-	-	-
Zn	0.795	0.701	0.683	0.741	0.695	0.682
Cu	0.013	0	0.001	0.017	0.002	-
Ni	0.208	0.134	0.166	0.202	0.152	0.16
Cr	0.134	0.136	0.141	0.133	0.146	0.142
Hg	-	-	-	-	-	-
As	0.01	0.009	0.005	0.005	0.011	0.004
Ba	-	-	-	-	-	-
Sb	0.01	0.007	0.007	0.023	0.007	0.008
Se	0.018	0.007	0.015	0.018	0.001	0.004

Results of the leaching test conducted on raw fly ash samples (Tab 4.) showed that level of all investigated leached toxic elements was beneath its upper limit value. Content of Zn was higher than content of other toxic elements. Certain toxic elements, precisely Cd, Ba and Hg, were not detected in leachate of tested fly ash samples. Absence of cadmium in fly ash leachate is in accordance with assumption that Cd present in fly ash is consistently immobile while in neutral environment. At the other hand mercury can condensate on surface of fly ash particles and as the water soluble it might pose a risk for groundwater contamination and barium in fly ash forms sparingly soluble compounds with carbonates and sulphates. However, Ba and Hg were not present in investigated fly ash leachates. Concerning other detected elements, lead originates from sulphides found in coal and it is placed within the internal glassy matrix of fly ash and therefore not readily leached. Zinc is in sulphide association in the coal and its mobility in fly ash is high. The level of Zn in fly ash and fly ash based composites leachate was high in comparison to level of other investigated toxic elements, but still under upper level of toleration. Copper is assimilated within the glassy phase and not easily leached. Nickel is distributed between the silicate fraction and the magnetic fraction of fly ash during combustion process and most of the leachable Ni is solubilised from the non-magnetic fraction. Chromium leaching generally poses high threat to the environment since it is recognized as potentially carcinogenic matter. However, Cr quantity in investigated samples was significantly beneath level of tolerance. Selenium shows high solubility in water which makes Se as an element of major concern in

ash due to its mobility. However, detected level of Se was rather low and did not represent any concern for environment. Antimony in coal occurs in sulphides along with pyrite. The leaching behavior has been scarcely covered in the literature, although it is known that Sb is insoluble in all types of leachant. Amount of Sb found in leachate of investigated composites is low. Arsenic is associated with As-bearing pyrite from coal, which is decomposed during the combustion process. Arsenic condenses on the surface of fly ash as sparingly soluble. The leachability varies widely depending on the nature of the fly ash. In investigated ashes and fly ash composites As was not readily leachable.

Leaching test performed on "standard" cement-mortar and fly-ash based cement-mortar (Tab 5.) showed no presence of Cd, Ba and Hg which was expected due to the lack of these toxic elements in raw fly ash leachate. Mean values of all toxic elements, except Cu and Cr, found in fly ash based mortar leachate are higher than content of adequate element found in standard mortar leachate. Namely, addition of fly ash slightly increased level of most of toxic elements present in mortar composition. However quantities of toxic elements found in fly ash mortar leachate do not exceed upper limit of toleration. The comparison of results of batch leaching test performed on raw fly ash and fly ash based mortar did not show significant differences between level of toxic elements found in leachate, pointing out to the fact that addition of 30 % of fly ash did not cause any unpredicted or unwanted chemical reactions during cement hydration nor additional toxic and/or harmful chemical products were made.

Table 6. gives the results of batch leaching test conducted on samples of fly-ash based concrete and standard concrete. As in case of raw fly ash and fly ash based mortar, test showed no presence of Cd and Hg. However, small quantity of Ba (< 0.1 mg/l) was detected in leachate of FA_E-concrete, probably originating from “standard” concrete whose leachate also contained certain small quantity of Ba. Ba was not found in other investigated samples, probably because its content was beneath detection level. Ba was probably “carried in” as chemical compound found in composition of a component used in concrete design, presumably aggregate which contained some impurity (possibly Ba(SO₄) originating from sedimentary rocks). Quantity of all investigated toxic elements found in concrete leachate was beneath the upper limit values. Same as in case of mortars, the comparison of results of batch leaching test performed on raw fly ash and fly ash based cement did not show significant differences between levels of toxic elements found in leachates. Thus, addition of 30 % of fly ash did not cause formation of any undesired additional toxic chemical product.

Results of the leaching test conducted on samples of fly-ash based asphalt and “standard” asphalt, given in Table 7., pointed out to the fact that there was no Cd, Hg and Ba found in leachate. Cu was in non-detectable quantity in samples FA_B-asphalt and “standards” asphalt. In other samples, Cu originates from fly ash addition. Quantity of all investigated toxic elements was beneath the upper limit values. The results of leaching test did not show any significant change in toxic leaching of the fly ash based asphalt samples in comparison with adequate results of toxic leaching of raw fly ash and “standard” asphalt.

Results obtained from laboratory leaching trial tests performed on three different types of fly ash composites show relatively small environmental risk in these composites are used in building construction. Namely, content of harmful toxic elements is low and not easy leachable. Level of all toxic pollutants found in leachate of both fly ash and investigated composites is significantly lower than assigned level of toleration. This highlights the possibility of fly ash application in construction materials industry. Namely, there is low environmental risk posed by the presence of contaminant trace elements in the fly ash if the fly ash is applied in a construction composite included in either building or road constructive element and exposed to the action of rain and snow. However, it should be stated that wider environmental impact should be investigated only after application of composites in construction element, building or road – namely during a real-time scale researches.

Microstructural analysis of all fly ash samples was performed, however the microstructure of fly ashes FA_{A-E} is rather similar, thus the FA_A was chosen as representative sample and its microstructural elements are presented as original SEM microphotograph in Figure 2.

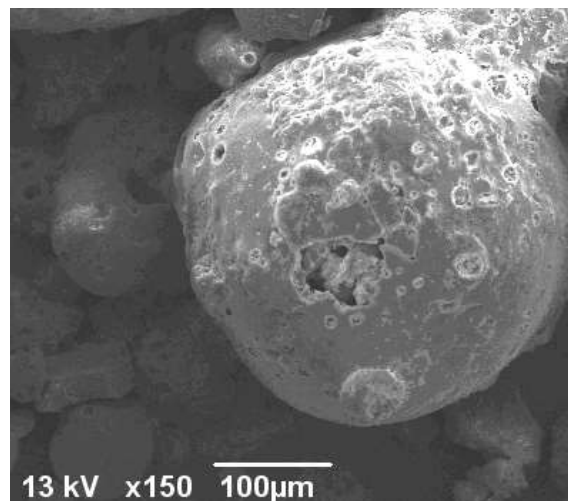


Figure 2 - Microphotograph of FA_A fly ash.

The fly ash samples were mainly composed of unbounded glassy spherical hollow particles, even though certain amount of grains of different size and irregular shape was present. Differently sized and shaped particles belong to different mineral phases which were previously detected by means of XRD. One of the characteristic spherical fly ash particles in Fig. 2. It can be noted that superficial porosity of fly ash grain is high. High porosity of grains leads to elevated water demands when composite mixture is prepared. Also, grain porosity might induce higher apparent porosity of the final product (mortar, concrete or asphalt), which might in return enable easier leaching in natural surroundings. However, the leaching test performed on fly ash and fly ash based composites showed no possible toxic leaching threat, since all toxic elements in leachates proved to be assigned limits. Larger particles which may be either spherical or slightly irregular usually contain variable amounts of the “bubbles” and other differently shaped crystalline minerals.

Microstructure of fly ash based composite FA_A-concrete is given as SEM microphotograph in Fig.3. Microphotographs of other sub-types of composites are not provided here because due to application of same raw materials – same bonding agents and aggregates and only varying the type of fly ash, there are no significant visible differences between them.

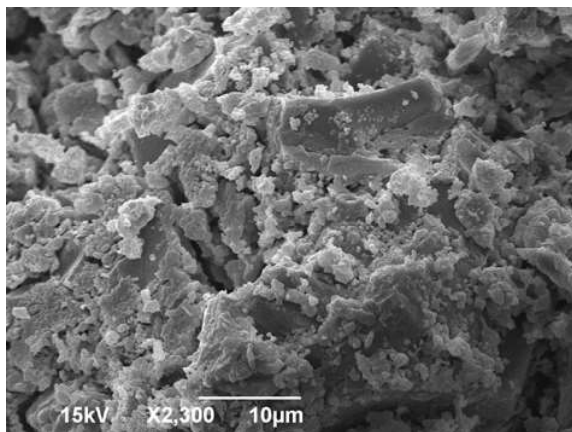


Figure 3 - Microphotograph of FA_B -concrete.

In Figure 3. fly ash particles are incorporated in “cementing zone” between aggregate grains. The fly ash which is merged with cement during process of setting and afterwards hardening of the concrete behaves like standard pozzolanic material. Namely, cement and fly ash together form hardening bonding-agent paste locking the aggregate grains together in stone-alike composite structure. In such structure, fly ash porous grains are being interlocked in the space between particles of bonding agent and/or filler making internal barriers within pore channels, “cutting” the path of the water and preventing the larger scale toxic leaching. Also, fly ash particles, due to its spherical shape, improve the “packing” of the concrete microstructure and fill in potentially left structural voids. Namely, fly ash acts like super-plasticizer making the concrete structure more compact and decreasing the level of porosity. The role of the fly ash is same in all composites: concrete, mortar and asphalt.

4. CONCLUSIONS

The investigation performed on the fly ash originating from electro-filters of five different power plants and fly ash based composites - asphalt, concrete and mortar led to the conclusion that investigated fly ash can be considered as non-hazardous in terms of its reapplication as raw material in building materials. The conventional powder processing of lignite coal fly ash, which did not include additional thermal or chemical activation, allowed the formation of relatively high density/low porosity building materials (mortars, concretes and asphalts) which proved to have optimal composition regarding prevention of toxic leaching. The toxic element leaching obtained for experimentally designed ash based composites was comparable to that of commercially produced engineering composites - standard mortars, concretes and asphalts.

Potential toxic and/or leachable elements in leachates of investigated fly ashes and fly ash based building composites were under upper limit of toleration assigned by official regulative. The following potential pollutants – Pb, Cd, Zn, Cu, Ni, Cr, Hg, As, Ba, Sb and Se proved to be of low concern when fly ash is exposed to standard environmental conditions approximated by laboratory batch water leaching test. As the leaching tests carried out on the fly ashes and fly ash based composites dismissed the possibility of potential larger scale transition of toxic elements from the building material in contact with water, fly ash reapplication might be regarded as safe process.

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