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EXPERIMENTAL TESTING OF FLY-ASH STABILIZED MIXES

V. Mráz[1], J. Suda[2], J. Valentin[3], M. Kopecký[4]

[1,2,3,4]CTU in Prague, Czech Republic

ABSTRACT

The application of fly-ash stabilizers, materials from fluidized combustion fly-ash and other solid coal-burning residues which are called coal combustion by-products (CCB) that have good potential for application in subgrade structures and roadbed materials of roads as well as in the structural pavement layers. One of the many factors limiting the application of some CCB sorts is the relatively low resistance in repetitive contact with water, volumetric changes and the risk of partly unsatisfactory hygienic and environmental parameters.

In regard to the aforementioned negative characteristics of CCB which occurred primarily under the repetitive impact of water and freezing, the experimental examination focused on improving CCB resistance to frost and water, verification of volumetric changes and improvement of pozzolana characteristics of CCB by increasing the percentage of fine particles in the original material.

Currently road construction industry strives to find a suitable substitute of the traditionally applied hydraulic binders as well as expand the existing base of the binders applied. The experience with using CCB as a binder or binder component has not been as extensive so far as to allow any generalisation of conclusions. Therefore, the possibilities of applying alternative additives as a replacement of the binders traditionally applies have been researched.

Keywords: ***Chemical Analysis, Compressive Strength, Fluidized-Bed Fly-Ash, Fly-Ash, Fly-Ash Stabilized Material, High Energy Milling, Mechanical And Chemical Activation, Roadbed Structures***

I. INTRODUCTION

All technically advanced countries have intensified their research and development of utilisation of various types of waste as secondary material sources in recent years. This trend also applies to the brown coal combustion residuals, great quantities of which are a waste in the power industry since they occur as solid residues from coal combustion and residue purification in power plants, heating plants or heat stations. The materials are called coal combustion by-products (CCB) and include e.g. fly-ash, slag, cinder, bottom ash, flue gas desulfurization (FGD) gypsum.

Those countries that support their power policy primarily by generating power in thermal power stations research the options of processing and subsequently effectively utilising CCB. Practical applications have focused primarily on the construction industry so far. One of the paths seems to be applying CCB in roadbed structures of roads, railroads or airfield structures as there is an assumption of processing larger quantities of CCB.

Another possible application of CCB gained after coal combustion is according to [1, 2] use of such material for soil modification or improvements in the roadbed structure. Such modifications of soil usually lead to higher strength properties, improved workability and increased resistance to climatic effects. Other mechanical or physical characteristics of the original soil can be improved as well.

A limiting factor for the use of some CCB types is their relatively low resistance in repetitive contact with water and freezing [3, 4], volumetric changes and in some cases the risk of partly unsatisfactory health and environmental parameters [5, 6]. Particularly for fly-ashes from fluidized combustion, ettringite (high-calcium sulfo-aluminate mineral) might be formed in case of long-term contact with water [7, 8]. Extensive analyses of CCB chemical characterization have been collected and done, e.g. during the planning and preparation of embankment structure for a motorway project in the UK. CCB samples were taken from three different power plants. Leaching analyses have shown increased contents of arsenic, cadmium, chromium, mercury, selenium and sulphates. At the same time the pH value was increased as well as the concentration of polycyclic aromatic hydrocarbons [9].

With respect to the aforementioned negative CCB characteristics which occurred primarily under repetitive influence of water and freezing, the experimental research focused on improvement of CCB resistance to frost and water, verification of volumetric changes and improvement of pozzolana properties of CCB by increasing the percentage of fine particles in the original material (e.g. by means of mechanically and chemically activated fly-ash).

At the same time, the road construction industry strives to find a suitable replacement of the hydraulic binders traditionally used as well as extend the existing base of the binders applied. The experience with application of CCB as a binder or binder component is not as extensive so far as to allow generalisation of its conclusions. Therefore, the possibilities of alternative additive application as a substitute of the binders traditionally used were examined.

II. MATERIALS

2.1 Coal Combustion By-products

The samples chosen for the purposes of experimental research of stabilised fly-ash mixes were the so-called fly-ash from electrostatic separators (filters) of the Melnik power plant (hereinafter “EME”), often called high-temperature fly-ash, and bed ash from fluidized combustion from the Ledvice power plant (hereinafter “ELE”) (see Fig. 1a, 1b). The aforementioned power plants represent the two basic types of desulphurisation; each of them produces CCB of differing technical parameters. EME desulphurisation follows the route of the wet scrubbers. In ELE, CCB from furnace FK4 were used; this is desulphurised by the fluidized combustion method. With respect to the combustion technology applied, the chemical and mineralogical composition of fly-ashes from fluidized combustion fundamentally differs from the composition of classic high-temperature fly-ashes. While the main phases of high-temperature fly-ashes consist of amorphous silicon dioxide, silica, both high-temperature modifications – cristobalite as well as tridymite and mullite – fly-ashes contain aluminosilicate phase, silica, insoluble anhydride II, free calcium oxide, and possibly calcium hydrate and calcium carbonate.

As ensues from the above, high-temperature fly-ashes demonstrate the pozzolana properties exclusively while fly-ashes from fluidized combustion, thanks to the presence of calcium ions, have hydraulic properties even on their own [10].

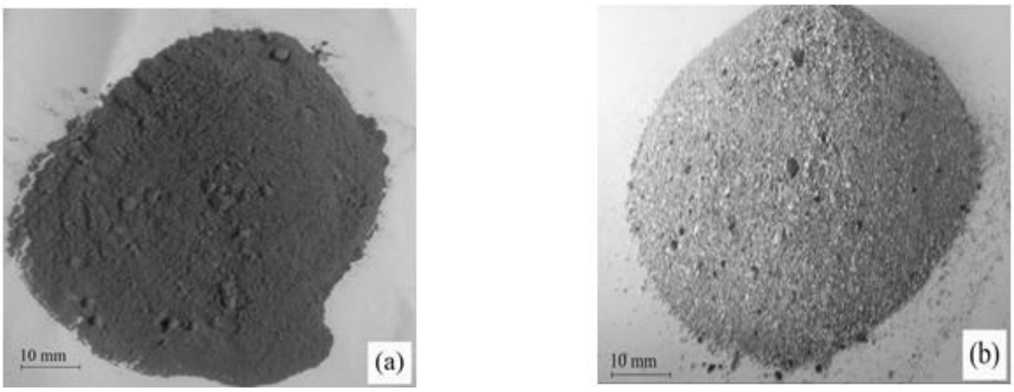

Figure 1: Loose materiál: a) filter fly-ash EME, b) bed ash from fluidized combustion ELE

Within the structural analysis of two selected types of energetic by-products assessment of internal material structure has been carried out by application of electron microscopy. Microscopy and microanalyses have been processed by the environmental scanning electron microscope (ESEM FEI PHILIPS), equipped by the set of electron detectors – scattered electron diffraction (SED) for morphology on micro level and BSED for phase contrast, both in high vacuum mode and environmental mode. The electron diffractions (ED) analysis of secondary X-ray spectra provides the quantitative chemical composition of the selected objects. The quantitative phase mineral composition both on micro and nano level is facilitated by OIM-BSED (Orientational Imaging Microscopy based on Back Scattered Electron Diffraction). This equipment gives the information about the quantitative phase mineral composition and structural orientation maps.

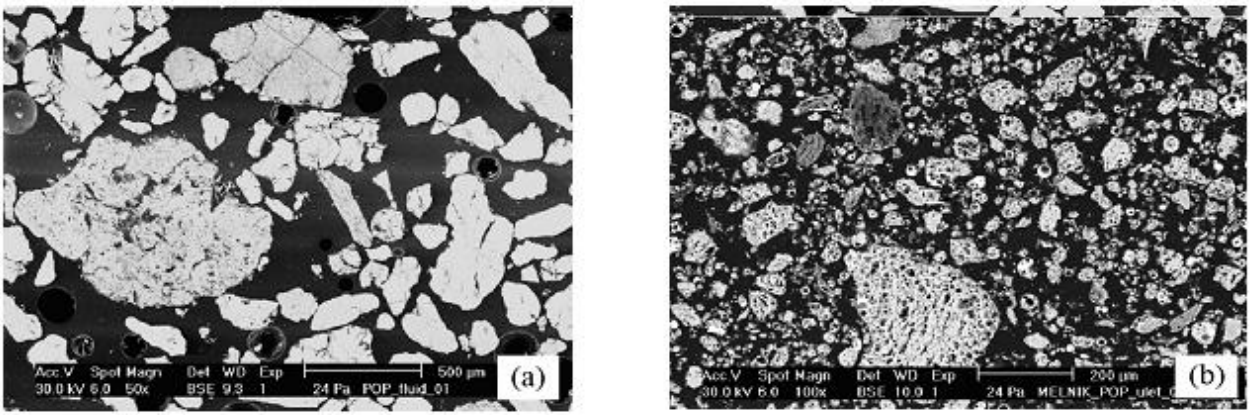

Figure 2: Electron microscopy: a) bed ash from fluidized combustion ELE, b) filter fly-ash EME

2.1.1 Sample preparation used for electron microscopy analysis

Before the electron microscopy analysis representative quantity of fly-ash sample (10 ccm) was mixed with low viscose epoxy resin. After hardening (at 25°C), the sample was brushed (emery papers) and polished (diamond paste) in dry mode. Obtained surface was cleaned by pure methanol. This method of sample preparation prevents eventual reaction of water-sensitive fly-ash particles.

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Methods of electron microscope assessment were based on phase contrast taken by back-scattered electrons detection (BSE) and energy dispersed X-ray analysis (EDS).

2.1.2 Analyses of Fly-ash from Melnik Coal Power Plant

Fly-ash particles (FAP) are represented by the very porous slaggy debris some of larger particles are almost foamy. The character of FAP is very monotonous, nearly all particles have the same character: porous and foamy. The size of FAP varies from first microns up to several tenths of mm. The compact like glassy balls of FAP or iron oxides particles do not occur in this type of fly-ash from EME power station.

2.1.3 Analyses of Fly-ash from Ledvice Coal Power Station

Fly-ash particles (FAP) are mostly represented by calcium sulphates (gypsum, hemihydrate, anhydrite etc.). Some grains are alumosilicates (mullite, quartz). Only a small portion of FAP is porous (in comparison with FAP from Melnik).

2.1.4 Analyses of Fly-ash from Plzen Generation Plant

Fly-ash from fluidized combustion originated from Plzen generation (heating) plant which is equipped with the fluidized bed furnace technology that is installed in a number of power-generating operations in the Czech Republic. The types of fly-ash obtained are formed primarily during fluidized combustion of soft coal and limestone powder which, from the perspective of further application in the construction industry, are rather significant. The ash was mechanically activated concretely the material was driven between the rotors of a twin-rotor contra-rotating high speed mill – disintegrator – under mutual peripheral speed of the rotors of approx. 204 m.s-1 and power consumption at the level of approx. 20 W per kg of pulverized ash.

The fly-ash from the Plzen heating plant contain unusually small quantities of free lime; such ash loses its self-binding ability and an addition of a certain quantity of lime or cement would be recommended in a certain stage of compacted mix production. The contents of the amorphous phase are rather high in such type of fly-ash. The results of XRD analysis for mechanically activated fly-ash is indicated in the following Table 1 and Fig. 3.

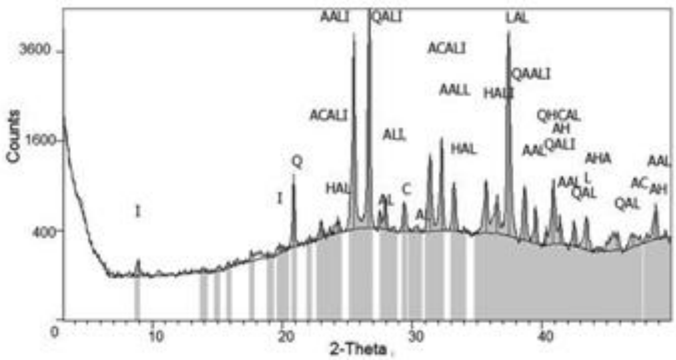

Figure 3: XRD data record of mechanically activated fly-ash from fluidized combustion Plzen

Table 1: Evaluation of phase composition of mechanical chemically activated fly-ash from fluidized combustion based on XRD data record

Ref. Code	Compound Name	Score	Total Lines	Scale Factor	SemiQuant [%]
Fly-ash from fluidized combustion – generation plant Plzen					
01-085-0794	Quartz	69	7	0.994	24
01-074-2421	Anhydrite	63	16	0.722	31
01-079-0007	Hematite	46	7	0.136	3
01-078-0649	Lime	49	2	0.800	13
01-083-0578	Calcite	43	9	0.071	2
01-089-6423	Albite	26	83	0.076	9
00-026-0911	Illite-2\ITM\RG#1 [NR]	36	17	0.427	18

2.2 Mechanical Activation

The mechanical process or interference with the structure of a substance that increases its chemical reactivity can be called mechanical activation. Such an intervention in the substance structure can consist of grinding/pulverization. According to the classic interpretation, grinding is defined as mechanical dispersion of solid substances which results in reduced particle size and a simultaneous increase of specific surface and surface energy within the system; nevertheless, mechanical effects occurring in the course of dry grinding of solid particles might cause significant structural changes and chemical reactions in the material ground. The character of the surface, or morphology thereof, distribution of charges, chemical nature of the thin grain surface film have a very distinctive impact on reactivity as well [11, 12, 13, 14].

Grinding might be a possible solution for rough fluid combustion separation process residuals. The grinding process causes large plerospheres (porous particles) to disintegrate and reduces particle roughness. Such reduction, together with the increased reactivity of the fly-ash, improves strength. The grinding of cenospheres increases density and fineness which results in higher pozzolana reactivity of the fly-ash. The grinding time affects the particle size, shape and, consequently, also the need for water [15].

2.3 Stabilized Fly-ash Mix

The so-called fly-ash stabilizate was chosen as the first type of compacted mix for experimental verification. Fly-ash stabilizate is a solid mass which usually arises by wetting a mix of fly-ashes or ashes with a binder (e.g. lime or cement) or by wetting a mix of fly-ashes from fluidized combustion that demonstrate self-setting properties. Ettringite might form during this process which causes volumetric changes. Ettringite forms in fly-ash stabilizate from soluble compounds of calcium, aluminium and sulphur in wet, alkali environments. In some cases, its expansion ability might damage the solidified stabilizate. According to the composition of the mix and mutual component proportions, quantity of added water and the processing method, a mass whose strength and other physical properties (permeability, weight, and thermal conductivity) are similar to those of lightweight concrete.

A deponate is understood as non-solidifying mass (with no additional additives) which is only strengthened by dehydration, drying or thixotropy.

The following additives were applied to prepare the fly-ash stabilizates:

- cement CEM II/B-M 32,5R;
- lime CL90S;
- mechanically activated or combined mechanically and chemically activated fluid ashes (from the Plzen heating plant);
- mechanically activated dolomitic limestone (from the Krty u Strakonic quarry);
- mechanically activated or combined mechanically and chemically activated recycled mix from concrete (recycled aggregate generated by crushing and sorting of reclaimed concrete from the airport Ruzyne);
- chemical additives Iterstab, Zycosoil.

III. EVALUATION METHODOLOGY FOR COMPACTED FLY-ASH MIXES

The Department of Road Structures, CTU, Faculty of Civil Engineering, determined the workability, strength characteristics and, with respect to the negative results in case of repetitive exposure to water, a test of resistance to frost and water immersion including the verification of volumetric changes of the mixes examined.

Mixes from both desulphurisation technologies and mixes with various binder proportions were tested. For CCB, the binders used as a standard were replaced by inorganic loose binders obtained by means of mechanical activation of fly-ashes from fluidized combustion, dolomitic limestone and reclaimed concrete materials. Besides such binders, the Iterstab (additive used primarily in soil improvement and stabilization) and Zycosoil (additive on a nanotechnology basis using silane groups) chemical additives were verified; the main benefit is preventing water from entering the mix. The objective was observing the same principles in CCB property modification as well.

One of the objectives of applying mechanically activated materials in CCB was eliminating ettringite formation which is generally one of the limiting factors of CCB application in embedding in roadbed structures of roads.

3.1 Compaction Assessment

Workability of fly-ash mixes was tested by the Proctor standard tests which simulate the compaction achieved by construction rollers very well. The laboratory test of CCB compaction quality is an important test for the assessment of applicability in road construction. CCB compaction effort is related to particle shapes and sizes. The mix compaction quality was examined by the standard Proctor test under CSN EN 13286-2 [16]. Compaction was started after a certain time elapsed from the wetting of the mix – this models the delays caused by transportation, spreading and other handling during real-life paving of the mix. Cement, lime or other additives were added to the dry CCB in samples with additives. The mix was dry-homogenised and wetted only afterwards.

3.2 Compressive Strength Testing

The laboratory test of compressive strength was performed according to the CSN EN 13286-41 [17] standard where a test specimen shaped like a right circular cylinder was loaded by the growing axial stress σ until its failure. The test principle consists in loading the test specimen of hardened energetic by-products with uniaxial compression with a simultaneous measurement of deformation. Strength characteristics after different curing times were studied in detail.

EME fly-ash and ELE bed ash were used to make test specimens by compaction in the laboratory with dimensions of R=100 mm and the height of 120 mm. The test specimens were cured for 7, 14, 21, 28, 60 and 90 days (for some mixes even for 1 year) in a laboratory environment in an airtight cover. The test specimen was also tested for immediate strength after compaction where the prepared specimen was cured at a laboratory temperature of 20-23°C for approx. 2-3 hours.

3.3 Resistance to Frost and Water Immersion

The preparation and curing of test specimens followed the same process as in the case of compressive strength test. Once the 28-day curing was completed, the test samples were placed on a felt pad partly sunk in water and left to saturate through capillaries until the set weight so that the weight increment for at least 1 hour would not exceed 1 %. All test specimens were saturated in the course of 20 minutes from putting on the felt pad.

Subsequently, the test specimens were placed in a freezer box for 6 hours under -20°C to -22°C. After freezing, the test specimens were taken out of the freezer box and stored on a felt pad partly sunk in water for 18 hours to allow further capillary saturation. Simultaneously, de-frosting under +20°C to +25°C occurred. The test continued by another round of freezing and repeated 10 cycles according to the method stipulated in the National Annex NB CSN EN 14227-5 [18]. Once the last cycle was completed, a strength test was carried out according to the standard CSN EN 13286-41 [17].

3.4 Swelling Susceptibility of Fly-ash Stabilizates

Monitoring of CCB volumetric changes is of crucial importance from the perspective of pavement structure durability. Volumetric changes might be demonstrated by shrinking or expansion and, subsequently, result in deterioration of the technical and environmental parameters or, often, complete destruction of the pavement structure.

Further factors affecting volumetric changes of CCB include:

- chemical and physical properties of the input materials;
- risk component content and variability;
- mix design;
- production technology;
- environment in which the CCB is placed (e.g. humidity and thermal parameters, pressure and combination of these factors);
- other specific factors [19].

In relation to the risk of undesired volumetric changes, this test must be viewed with more emphasis. The experimental research examined the impact of the mix on volumetric changes.

The subject matter of swelling measurements for fly-ash stabilizate was determining the linear and volumetric coefficient of swelling. Volumetric changes are understood as increasing of the fly-ash stabilizate volume caused by physical and mechanical processes ongoing in the material, or by additional water absorption.

A CBR bin and other equipment used for the preparation and facilitation of CBR testing under CSN EN 13286-47 [20] were used for the purposes of this test.

The mix saturated to w_{opt} according to the Proctor Standard test was compacted in the CBR cylinder by means of Proctor Standard (PS) energy.

Fly-ash stabilisers were cured for 7 and 28 in moulds under (20±2)°C in impermeable wraps and, subsequently, saturated by water until all deformations ceased to occur. Within the time intervals as mentioned, the changes of surface level of the compacted, saturated samples loaded by a weight were measured.

IV. RESULTS AND DISCUSSION

4.1 Fly-ash Stabilized Mixes

4.1.1 Compaction

From the results of optimum compaction assessment done on fly-ash form EME implies, that optimal moisture content of fly-ash mixes with 6 % of CaO addition are only slightly dependent on the content of hydraulic binder in the mix and reach for guidance around 20 %.

Filter fly-ash without any additives showed optimum moisture content for compaction at 21 %, whereas filter fly-ash from EME with 6 % CaO reached optimum moisture content at 20 %.

Compaction quality of fluidized-bed ash from ELE was reached with optimum moisture content about 35 %, if 6 % of pulverized dolomitic lime was added the optimum moisture content increased slightly to 36 %. In case of 10 % activated fly-ash from fluidized combustion the value went up to 38 %. Overall results of mix compactability are given in the Table 2.

Table 2: Compactability parameters of fly-ash stabilizates

Mix	Fluidized-bed fly-ash from ELE without additive	Fluidized-bed fly-ash from ELE with 3 wt % of cement CEM II/B-M 23,5R	Fluidized-bed fly-ash from ELE with 6 wt % of pulverized dolomitic limestone	Fluidized-bed fly-ash from ELE with 10 wt % of pulverized fluidized-bed ash	Fly-ash from EME without additive	Fly-ash from EME with 6 wt % CaO
Maximum density [kg/m3]	1122	961	996	1084	1080	1060
Optimal moisture [%]	36,7	34,7	36,2	35,3	21,0	20,0

4.1.2 Compressive Strength Results

From the Fig. 4 and 5 it is apparent, that best values of compressive strength are reached for mixes where mechanically activated fly-ash, dolomitic lime and cement are represented. These mixes fulfilled required threshold limits for compressive strength according to the technical specifications TP 93 [21]. By applying mechanically activated fly-ash form fluidized combustion, as well as dolomitic lime or pulverized recycled

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concrete the possibility of substituting traditional hydraulic binders by these alternative materials has been proven.

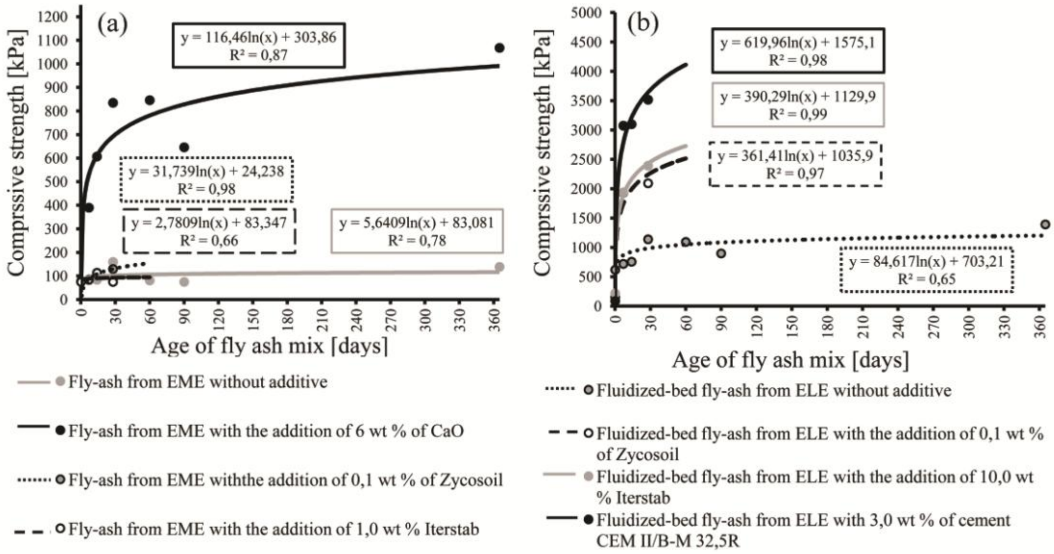

Figure 4: Compressive strength results of compacted energetic by-products with different additives

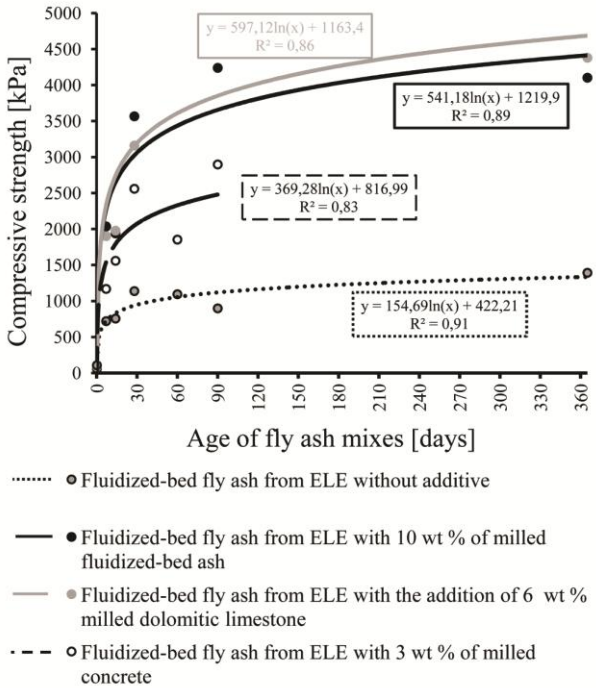

Figure 5: Compressive strength results of compacted energetic by-products with added mechanically and chemically activated materials

4.1.3 Resistance to Frost and Water Immersion

Resistance to freezing and water depends to a great degree on the composition of the original mix. Stabilizates (deponates) prepared with material from wet scrubber process mixes disintegrated after the first freezing cycle already, or even during the saturation stage. Stabilizates prepared with material from fluidized combustion technology disintegrated after 2 to 3 freezing cycles. Stabilizates made of compacted hydrated mix with an addition of mechanically activated fly-ashes from fluidized combustion, dolomitic lime, reclaimed concrete or chemical additives improve the resistance feature of fly-ash mixes against the effects of water and freezing better than stabilizates with no additives. The drop in strength after freezing ranged from 20 to 30 %. For the stabilizate from fluidized-bed ash with the addition of 6 %-wt. mechanically activated dolomitic lime, the original compression strength even increased by 11 % after defrosting. The values must be confirmed again with more test specimen sets.

For the deponate prepared with fly-ashes from EME with no additives, the specimen collapsed in 50 minutes from placement on the felt pad partly soaked in water (see Fig. 6a). Specimens collapsed after the first cycle also in the case of application of chemical additives with 0.1 %-wt. Zycosoil and with 1 %- wt. Iterstab in fly-ashes from EME.

The stabilizate prepared from the EME fly-ashes with 6 %-wt. calcium oxide resisted 10 cycles; however, it demonstrated minor longitudinal and alligator cracks (see Fig. 6b). Compression strength after the last cycle fell to 0.41 MPa, i.e. approx. half of the strength detected in the design stage.

The stabilizate from fluidized-bed ash from ELE showed a transverse crack; the specimen broke after approx. 4 hours' freezing in the second cycle (see Fig. 6c).

In the case of the test specimen from fluidized-bed ash with the addition of 3.0 %-wt. cement CEM II/B-M 32.5R, compression strength fell by 26.5 % after freezing.

Stabilizates from fluidized-bed ash from ELE with the addition of 1 %-wt. Iterstab and 0.1 %-wt. Zycosoil survived 10 cycles and demonstrated the greatest resistance; nevertheless, the strength indicators fell by 19 % for Zycosoil and by 38 % for Iterstab. The stabilisers failed to meet the requirements of CSN EN 14227-14 [22] and, therefore, frost susceptibility according to CSN 72 1191 [23] has always to be determined.

For the stabilizates from fluidized-bed ash with the addition of 10 %-wt. mechanically activated fly-ashes from fluidized combustion, a transverse crack formed after the 10^{th} cycle (see Fig. 6d).

The stabilizate from fluidized-bed ash with the addition of 6 %-wt. mechanically activated dolomitic lime (see Fig. 6e) which was stressed by a graded number of freezing and defrosting cycles demonstrated a value of strength even higher than the initial compression strength. These values must be reconfirmed with more specimen sets.

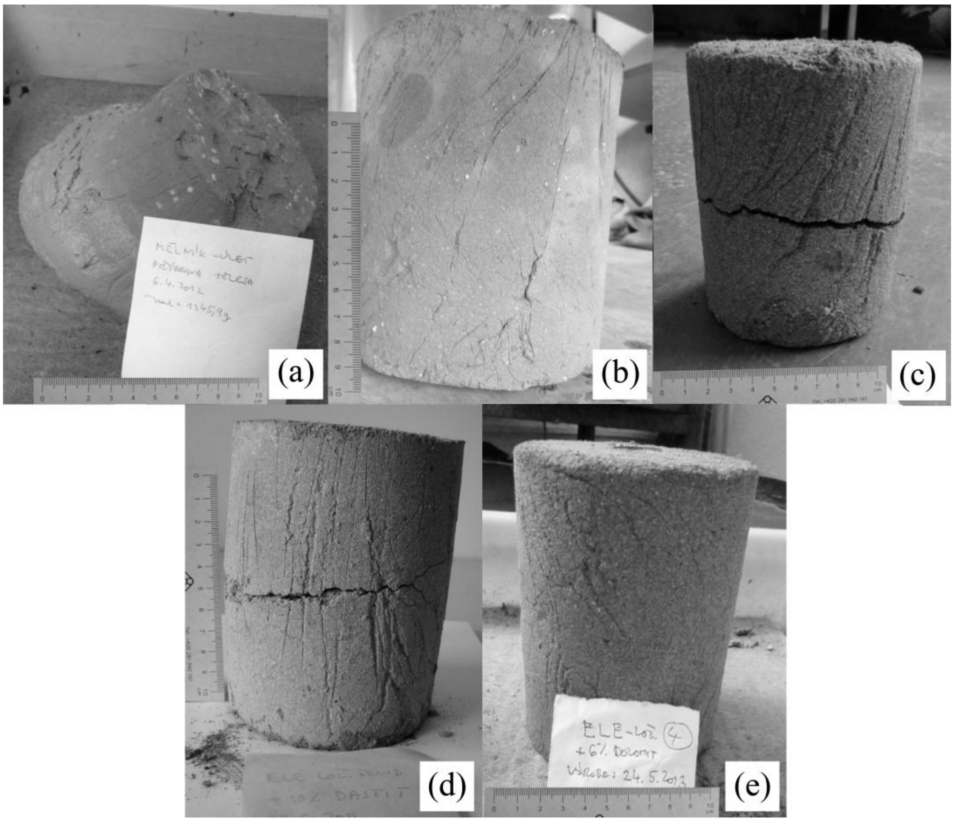

Figure 6: a) Filter fly-ash mix, EME, b) Stabilizate EME + 6 wt. %. CaO, c) Stabilizate from bed ash ELE without additives, d) Stabilizate from bed ash ELE + 10 wt. % of pulverized fly-ash from fluidized combustion – generation plant Plzen, e) Stabilizate from bed ash ELE + 6 wt. % pulverized dolomitic limestone

4.1.4 Swelling of Fly-ash Stabilizate

Based on the results of measurements of swelling during hardening and setting of the fly-ash stabilizates indicated in Fig. 7, it can be noted that the volumetric changes of the stabilizate amount to rather low values. The greatest volume increase (by approx. 1.5 %) was demonstrated by stabilizate from ELE with no additives after 7 days of curing. It shows no signs of setting even after 40 days. The remaining mixes show a slower volume increase, roughly linear with the hardening time. It can be assumed that the limit value < 3 % of swelling in CBR cylinder under TP 93 [21] will not be reached by all fly-ash stabilizates tested even after a longer examination period. It is likely that a hydration reaction occurs once the stabilizate components are mixed with water and compressed; ettringite forms during the reaction. In contrast to other materials with hydraulic bonds (e.g. concrete, cement-stabilized soil), ettringite's slight tendency to swell is a prevention against shrinking during hydration.

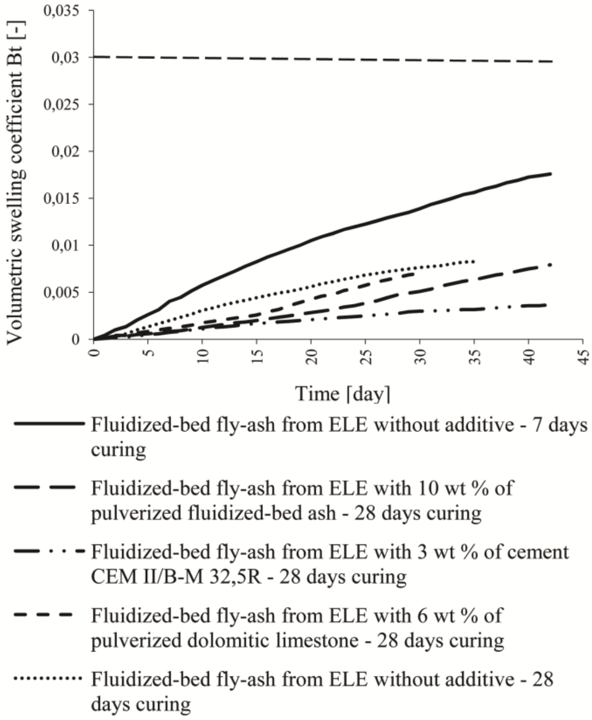

Figure 7: Time dependent progression of fly-ash stabilizate swelling

V. CONCLUSION

The present article focused on the possibility of using CCB by-products directly or applying them after certain modifications (through improvement of the properties required) in roadbed structures of roads, or even as structural layers of the pavements.

Laboratory research of CCB has emphasised the dominant influence of the technology applied to desulphurisation on the resulting CCB characteristics. The results of long-term CCB testing have proven that besides the deposit from EME, mechanical properties of fly ash stabilisers improve significantly in time. The results obtained have confirmed that highest values of simple compressive strength are achieved by mixes with a representation of ground (mechanically and chemically activated) fluidized fly-ash, pulverized dolomite limestone or recycled concrete and, therefore, such mixes meet the minimum values required for simple compressive strength according to technical specifications TP 93, [21].

Based on the results yielded by the measurement of fly-ash swelling capacity, it can be concluded that the volumetric changes of the stabiliser amount to relatively small levels.

Frost and water susceptibility testing has indicated that the deposits and stabilisers tested are not resistant to frost and water. The majority of the samples tested disintegrated in the course of ten freezing and thawing cycles or, subsequently, compressive strength fell considerably after the last cycle.

A stabiliser made of compacted, slightly wet mix with the addition of mechanically and/or chemically activated mineral materials or chemical additives partly improves the characteristics of fly-ash mix resistance to water and frost when compared to a stabiliser alone (no additives). The application of mechanically and chemically activated materials or used additives facilitates elimination of some CCB problems, while indicating a possibility of substitution for lime, or cement.

VI. ACKNOWLEDGMENT

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