Variation of Material Properties of Cement Pastes with Various types of Fly Ash During Maturation

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Abstract: The article describes properties of cement paste included addition various kinds of the fly ash. Firstly is described the preparation of the cement paste. The variation of material properties are described in time from one to three months. Next part is focused on the testing of the properties. Results of the testing are summarized in properties like a compression strength, Modulus of elasticity and tensile strength in bending. Finally the relation between material properties and volume weight is discussed. The article describes paste made from the same amount of cement and fly ash and mixed with water coefficient of 0.4.

Key-Words: - Cement Paste, Fly Ash, Compression Strength, Modulus of Elasticity, Water-cement Ratio, Cement-Fly Ash Ratio, Maturation of Cement Paste.

1 Introduction

The compressive strength and the tensile strength, elastic modulus and fracture energy are important and interesting material properties. Changes in the properties of cement paste [1] with fly ash to pure cement paste can be documented on those parameters.

The density of fly ash is significantly lower than the density of cement. It moves between 750 and 900 kg/m^3.

Compressive strength is a property determined from uniaxial compressive tests, the specimen is loaded by centric force. The proportion of maximum pressure achieved strength and load-bearing surface element determines the size of the compressive strength [2].

Tensile strength in bending is determined from another type of material test. The most commonly used three-point bending test. The specimen of the section is handled in the middle of the defined range increasing force until to failure. Tensile bending strength is calculated from the dimensions of specimen, distance support and achieved load.

The modulus of elasticity is significant characteristic determined from the compressive stress and the deformation during loading. Most often is determined from pressure tests, as secant value 1/3 strength and the corresponding strain.

Fracture energy is the properties of a given size of the energy required to breach the specimen. It is determined from the three-point bending test. Several methods exists for determination its value. All methods are based on measurement of deformation and vertical forces on the specimen, that has created under an applied force notch defined thickness. These properties are important for the design of structures in terms of capacity [3].

Its outstanding features are the water absorption and porosity. Fly ash is a porous material of high hardness. Article is comparing the above-mentioned material properties of dried and saturated specimens.

The volume density is important properties too. Its size depends on the content of the water. Due these two types is possible divide on the volume density in dry and saturated condition. First one is determined from the sizes of specimens and weight of water dried specimens. Second one is calculated from the water saturated specimens.

2 Material properties and their comparison

The cement paste is the basic part of the concrete. Its properties are crucial for the properties of concrete structures. The resulting material properties are improved by adding aggregates into the cement paste. Into concrete is a used aggregate with better mechanical properties than that of the cement paste in most cases.

The specimens were made by using two types of fly ash. The first is a classic fly ash resulting from combustion of brown and black coal. The combustion temperature is between 1200 and 1350 °C. The second type of fly ash is produced by combustion at lower temperatures. The combustion temperature is between 800 - 850 °C. The technologies of combustion is also different. The milled coal is combusted with the addition of limestone, which is the consist of the sorbet during the combustion of the coal.

Fluidized fly ashes are affected by the higher amount of CaO. The crystalline phase consist the mineralogical neoplasms as: Portlandit, gypsum, calcite.

Although the first type of fly ash is very suitable from viewpoint of use in concrete, the second type of fly ash is useable in concrete too.

It is a stable material for processing in the concrete.

2.1 Cement paste

The cement paste containing the fly ash is a homogeneous material. Homogeneity of the material allows the use of small specimens for testing. Specimens with size 20 x 20 x 100 mm were determined for experiments. The cement paste made from water and Portland cement CEM I 42.5 R was initially chosen for production. Water-cement ratio for the preparation of cement pastes was selected 0.4 [1]. The consistency of cement paste is liquid, well-workable and does not need a plasticizer used for preparation.

The same water-cement ratio was used for the production of cement paste with classic fly ash. The ratio between the amount of cement and fly ash was 1:1 [4]. Specimens were produced in the steel moulds with precise dimensions. This production of the specimens had water-cement ratio 0.4, too. Cement and fly ash are considered under the term "cement" in relation.

Third type of cement paste contained a fluidized fly ash. There were produced three type of specimens. First one was concreted with water-cement ratio 0.6. The other two types were prepared with w/(c + fa) ratio 0.7. The ratio between the amount of the cement and fluidized fly ash (c/fa) was in the first case 70/30, in the second case 60/40 and in the third case of a 50/50.

The specimens containing the fluidized fly ash are very sensitive to water leakage after the concreting. The water content of the specimens was maintained by packaging of fresh specimens into the foil as it displayed in Figure 1.

All specimens were stored in to water bath after the concreting. Testing was performed at the age of one and three months. The article presents the results of the test after 28 days and results of the test after three months, too.

The specimens were removed from the water bath and divided into the two groups before testing before testing. First one is the dried and second one includes specimen saturated with water. All specimens were loaded at high temperatures. For testing were selected at 20, 150, 200, 250 and 300 °C. After removing specimens from the water basin followed by thermal loading, the specimens were heated to a specific temperature at 100 °C / hour. Subsequently, the temperature was defined by specimen is heated for 24 hours and then the temperature was decreased by naturally cooling to 20 °C.

Fig.1 Concreting of cement paste into the steel forms. The foil covering the surface of the specimens against to leakage of moisture.

Saturated specimens were returned back into the water bath and left there two days.

The compression strength was tested by using the six specimens, see Figure 2. The specimens were prepared from the three original specimens so that it was cut into two equal parts the height of 50 mm.

Fig.2 The specimen for material testing and after loading by temperature.

The specimens for the test of tension strength in bending were used in the original size and their quantity was 5.

The Modulus of elasticity was measured together with the execution of the test of compressive strength [5]. Every specimen was loaded 3 times to 1/3 the expected strength and after then the compression test was carried out.

The fracture energy tests were carried out, too. One of the specimens is displayed in Figure 3. Four specimens in each groups were tested with the notch to half the height of section. The fracture energy was calculated from results of measurement in the three point bending test. The loading force and sag in middle of span was measured. The sag was measured in displacement of the crossbeam in accuracy 0.002 mm. Size of the crack opening was measured, too. The extensometer was used for measurement of crack opening.

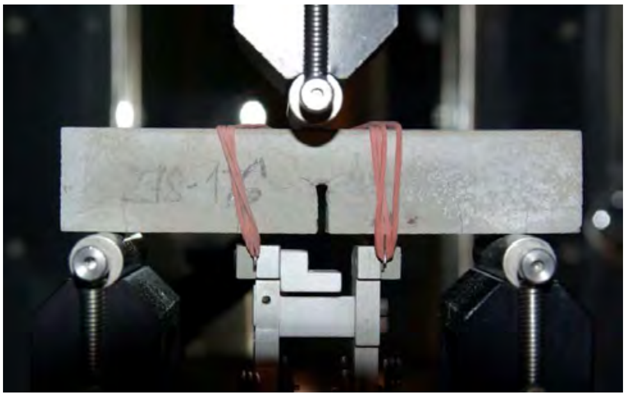

Fig.3 The specimen with notch in fracture energy test.

2.1 Testing equipment

The tests were carried out in the test apparatus 30kN MTS Alliance. This is the electromechanical testing machine with a crosshead displacement measurement in high precision. The compression tests were carried out together with Modulus elasticity measurement.

Display of the result of compression curve was made by using data of load and displacement of crosshead.

The extensometer was used for measurement of the deformation for the modulus of elasticity. The measured length of the extensometer was 25 mm. This corresponds to half the height of the specimen, see Figure 3 and 4 [6]. All compression test were executed with preloading to 1/3 of expected maximal load.

The three bending tests were realized on the specimens 100 mm length. The distance of supports was 80 mm. Specimens without notch were used for measurement of the strength in three point bending test. The specimens with notch were used for fracture energy test. The notch in the specimen is for localization of crack. The post peak curve in the working diagram is possible record with electromechanical testing machine only at specimen with notch.

The notch is situated in middle of the specimen length. The height of notch is between 1/3 an 1/2 of specimen height. There was measured maximal loads between 100 and 500 N for fracture energy test.

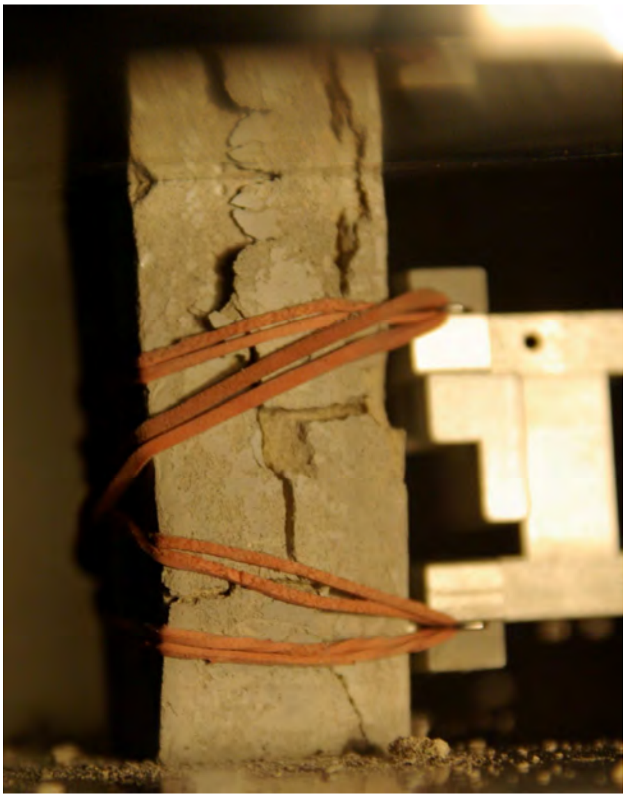

Fig.4 The specimen from cement paste after compression test with the main cracks. On the specimen was clipped extensometer to measurement of strain.

3 Material properties

3.1 Volume density

Firstly, volume densities were compared. Volume density achieves different values for the different temperatures. Table 1 shows the volume density of cement pastes with admixture of fly ash.

The first column contains the temperature at which it was determined the volume density. The average volume weight values are given in the second column of Table 1. Third column shows the change of volume weight of the pastes, which were heated to the specified temperature and then refunded for 2 days in the water. Volume weight of saturated solids mixed with fly ash was near to

1700 kg/m^3, see Table 1. The content of fly ash reduces the volume density of cement paste in both cases of humidity.

Temperature (°C)	Volume density of cement paste with fly ash – dried (kg/m^3)	Volume density of cement paste with fly ash – saturated (kg/m^3)
20	1322	1752
150	1339	1713
200	1303	1736
300	1325	1745
450	1305	1727
600	1301	1729
20*	1297	1774
20**	1215	1738
20***	1153	1689

Table 1: The volume density of cement paste before testing for dried and saturated conditions in age of 1 month. Index * corresponds with fluidized fly ash and relation c/fa 70/30, ** with relation 60/40 and *** with relation 50/50.

Temperature (°C)	Volume density of cement paste with fly ash – dried (kg/m^3)	Volume density of cement paste with fly ash – saturated (kg/m^3)
20	1356	1742
150	1325	1730
200	1348	1715
300	1316	1739
450	1299	1771
600	1268	1774

Table 2: The volume density of cement paste before testing for dried and saturated conditions in age of 3 month.

Tables 1 and 2 described volume densities of cement pastes with classical fly ash. Table 2 includes results after 3 months. Saturated and dried specimens had comparable values of volume densities. The volume density not changes after 1 month of age. The value near to 1750 kg/m^3 are achieved in both cases of age for saturated material. Dried cement paste had volume densities at different temperatures near to 1350 kg/m^3 in age three months. It is about 50 kg/m^3 higher than volume density for one month old cement paste. The slight increasing of values is possible record. The difference between saturated and dried cement paste is 400 kg/m^3.

The cement paste prepared from fluidized fly ash has lower volume density near to 50 kg/m^3 for saturated state and close to 150 kg/m^3 for dried stage and same relation between content of the cement and fly ash.

3.2 Compression strength

The compressive strength of specimen is divided with the content of fly ash into two groups. We can see a comparison between the saturated cement paste with fly ash and dried cement paste with fly ash in Table 3 and Table 4.

Temperature (°C)	Dried conditions (MPa)	Saturated conditions (MPa)
20	34.33	24.68
150	28.39	22.42
200	29.19	22.01
300	29.49	30.69
450	13.88	23.21
600	8.79	20.13
20*	41.84	30.08
20**	29.17	33.00
20***	26.13	26.94

Table 3: Compression strength of the cement paste with fly ash in age 1 month. Index * corresponds with fluidized fly ash and relation c/fa 70/30, ** with relation 60/40 and *** with relation 50/50.

Temperature (°C)	Dried conditions (MPa)	Saturated conditions (MPa)
20	54.07	36.13
150	41.29	34.03
200	38.56	29.64
300	39.70	35.34
450	18.99	26.06
600	16.20	27.10

Table 4: Compression strength of the cement paste with fly ash in age three month.

Third column shows the compressive strength of saturated specimens in both cases. The desiccated specimens in Table 2, had initially at temperature 20 °C lower strength of 10 MPa than saturated cement paste. The strength of dried and saturated specimens mutually corresponds at the temperature of 300 °C in age one month. Difference between dried and saturated cement paste with classical fly ash is significant at temperature 300 °C and age 3 months. The values of compression strength are higher for older material. Dried and saturated cement pastes had same trend of the increasing of values of the compression strength at all temperatures. The trend of the compression strength evolution is similar for comparison of one and three month old paste.

The increasing of temperature at the 600 °C in saturated condition means the return of the strength to again close 20 MPa.

The decreasing trend of strength at the temperatures higher of 250 °C is evident. Rate of decline in strength is similar for saturated and dried condition. The strength of dried specimens reaches only 8.79 MPa at 600 °C and 20.13 MPa for saturated cement paste.

Water saturated cement paste with fly ash behaves quite differently. The significant decrease in compressive strength is at higher temperatures, too. But this decline is slower. The strength of saturated specimens is at 450 °C nearly 2 times higher than for the dried specimens.

On the other side, difference between compression strength is not twofold between saturated and dried cement pastes in age 3 months. This value is 7 MPa in favor of the saturated cement paste.

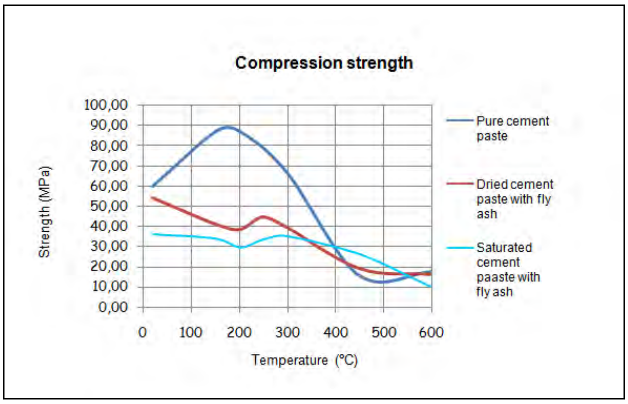

Fig.5 Compression strength of cement paste with content of fly ash compared to pure cement paste.

The saturated cement paste with fluidized fly ash reaches higher values of strength than the cement paste with classic fly ash. The compression strength of dried paste with varying content of fly ash has a variable character. The higher fly ash content causes a decreasing of the compression strength to the value 26.13 MPa. This value is about 8.2 MPa lower than the cement paste with classic fly ash. The results of fluidized cement paste are the same like cement paste included classical fly ash in saturated condition. The results for dried cement paste with fluidized fly ash are better than results of the cement paste with classical fly ash.

The comparison of the development of the compressive strength of cement paste containing fly ash is shown in Figure 5 and Figure 6.

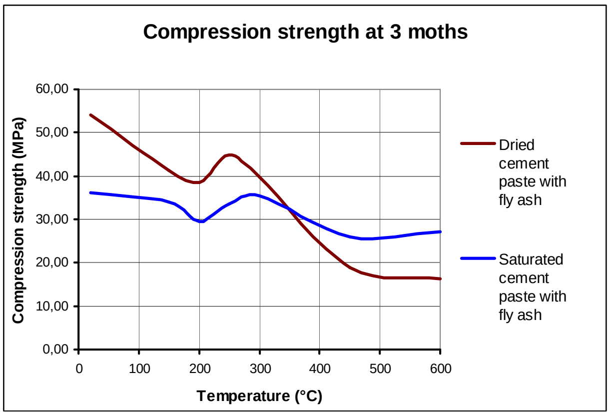

Fig.6 Compression strength of cement paste with content of fly ash in saturated and dried conditions.

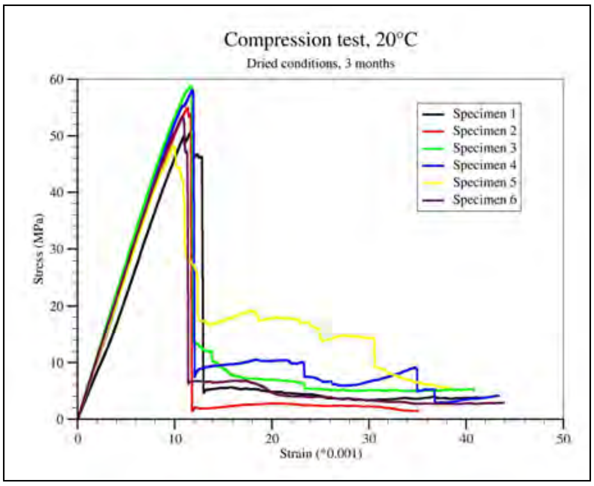

Fig.7 Stress-strain diagrams for saturated cement pastes at 20 °C and age of 3 months.

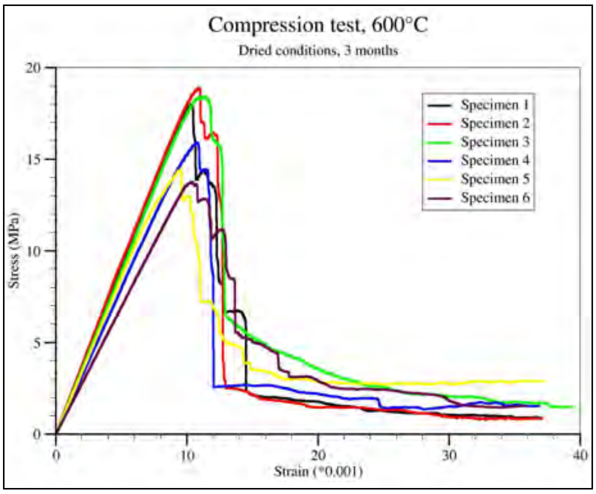

Fig.8 Stress-strain diagrams for saturated cement pastes at 600 °C and age of 3 months.

The comparable results are displayed in Figures 7 and 8. The post peak curves of material at temperature 20 °C are more brittle than curves at temperature 600 °C. The difference between strengths is evident.

3.3 Tensile strength

The results of tensile strength in bending are shown in Table 5 and Table 6. The table compares strength of dry cement paste tested at high temperatures and dry cement paste mixed with fly ash.

Temperature (°C)	Dried conditions (MPa)	Saturated conditions (MPa)
20	7.48	5.70
150	6.36	4.19
200	5.52	3.79
300	5.56	3.14
450	0.84	2.48
600	0.42	2.33
20^{*}	1.40	4.94
20^{**}	2.63	3.50
20^{***}	1.45	5.09

Table 5: Tensile strength of the cement paste with fly ash in age of one month. Index * corresponds with fluidized fly ash and relation c/fa 70/30, ** with relation 60/40 and *** with relation 50/50.

Temperature (°C)	Dried conditions (MPa)	Saturated conditions (MPa)
20	8.34	6.62
150	7.10	4.97
200	7.09	4.27
300	5.39	5.03
450	0.72	1.76
600	1.16	1.60

Table 6: Tensile strength of the cement paste with classical fly ash in age of 3 months.

The dried cement paste with fly ash has consistently decreasing character development of the tensile strength. Initially, the strength value for pastes with fly ash is higher than in case of the pure cement paste.

Tensile strength in bending dried cement paste decreases with increasing of the temperature, as shown in Table 5 and Figure 9. The significant decrease occurs when the temperature reaches 300 °C and increases. This behavior corresponds to the compressive strength of dried cement paste.

The water saturated specimens shows a steady decrease in tensile strength in bending since the temperature of 20 °C. The strength is only 2.48 MPa at temperature of 450 °C, how is displayed in Figure 9. However, this value is considerably higher than in the case of pure cement paste and dry cement paste with fly ash.

Very interesting is the ratio between compressive strength and tensile strength in bending. For example, at 200 °C, the compressive strength of pure cement paste is 14 times greater than the tensile strength in bending. For the dried cement paste with fly ash, this ratio is significantly lower, namely 4x.

The values of tensile strength in bending of cement paste with fluidized fly ash are variable in both cases of water saturation. The values of strength of saturated cement paste is close to value of strength of cement paste with classic fly ash. Dried cement paste with fluidized fly ash achieves much lower values of the tensile strength in bending than the cement paste with classic fly ash.

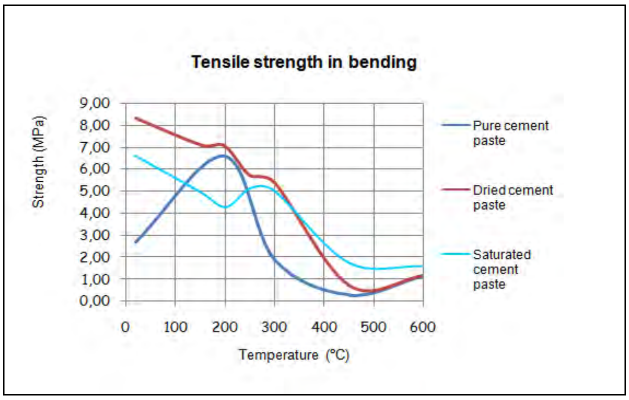

Fig.9 Tensile strength of cement paste with content of fly ash compared to pure cement paste.

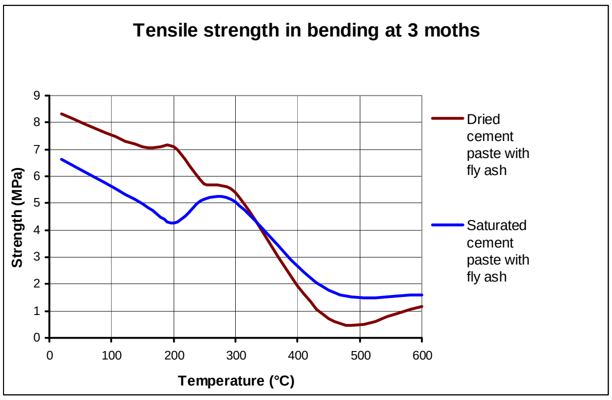

Fig.10 Tensile strength of cement paste with content of classical fly ash in time three months.

It follows that the properties of compressive and flexural strength are balanced for the dried specimens. On the other hand, the decrease in strength is greater for the dried cement paste than for the saturated cement paste above 300 °C. As shown in Table 5 is declining trend in tensile strength in bending observable also in saturated specimens with fly ash. In this case, the ratio of the compressive strength and flexural strength slightly higher than for the dried specimens, specifically 5 times.

Slight increasing of the tensile strength in bending is observable in comparison of the values in one and three months. This improvement is stronger at temperatures to 300 °C. The strengths at higher temperatures than 300°C are comparable for the different old specimens, like is possible see in Figure 9 and 10.

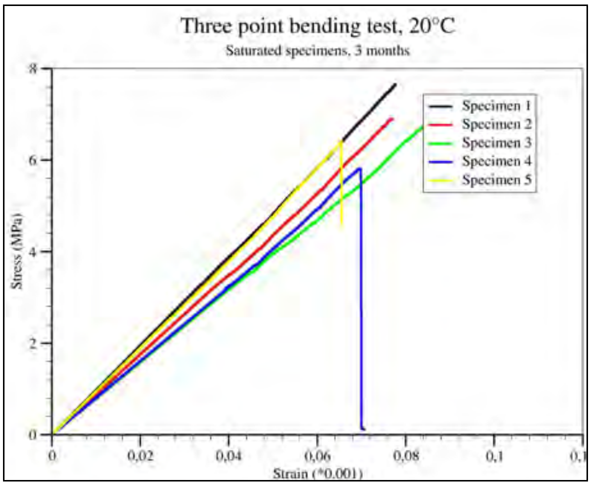

Fig.11 Tensile stress in bending-strain diagrams for saturated cement pastes at 20 °C and age of 3 months.

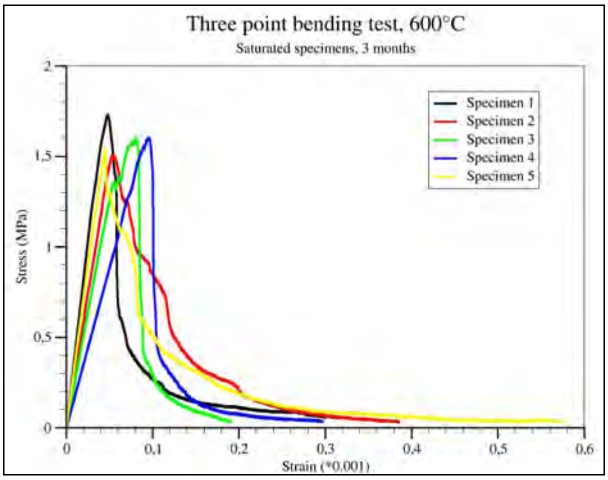

Fig.11 Tensile stress in bending-strain diagrams for saturated cement pastes at 600 °C and age of 3 months.

The Figure 11 and 12 show difference between post peak behaviour of material. The specimens without notches have quasibrittle behaviour after achieving of the maximal load. More ductile behaviour is observable for material at higher temperatures, where the maximal load is lower.

3.4 Modulus of elasticity

The modulus of elasticity shows a downward trend as well. Downward trend of the temperatures higher than 150 °C is steady in the case of the dried paste. The modulus is only 1.59 GPa when the temperature reaches 450 °C. The steady value of the modulus of elasticity is evident for the saturated specimens. From temperature 150 °C the strength value is between 8-10 GPa.

The saturated specimens reached about 6.67 GPa higher modulus of elasticity than the specimens dried at the temperature of 450 °C how is possible see in Table 7. The results are displayed in the Figure 12.

Temperature (°C)	Dried conditions (GPa)	Saturated conditions (GPa)
20	8.3	12.99
150	11.38	8.50
200	8.82	9.65
300	7.36	8.77
450	1.59	8.90
600	1.03	5.65

Table 7: Modulus of elasticity for the cement paste with fly ash in age one month.

Temperature (°C)	Dried conditions (GPa)	Saturated conditions (GPa)
20	10.22	22.12
150	19.97	16.90
200	9.25	10.74
300	7.34	7.14
450	1.73	4.50
600	2.26	4.48

Table 8: Modulus of elasticity for the cement paste with fly ash in age 3 months.

The saturated cement paste with fly ash had higher values of the Modulus of elasticity at higher temperatures then dried pastes. This behavior is same for one and three months old specimens. Saturated material retains deformation properties almost the same as at 20 °C. Three months old cement paste with classical fly ash reaches two times higher value of the modulus of elasticity in saturated condition than dried paste. The modulus of elasticity have similar values like a pure cement paste or concrete to the temperatures 200 °C.

Fig.12 Specimens prepared for test of modulus of elasticity after temperature loading at 300 °C.

The trends of the decreasing of modulus of elasticity with increasing temperatures are similar like in the case of compression strength and tensile strength in bending as is possible see in Figure 13 and 14. The exception is visible for cement paste saturated of the water in age of one month. There are values of Modulus of elasticity close to 9 GPa for the higher temperatures.

The modulus of elasticity of the cement paste with fluidized fly ash has not be determined.

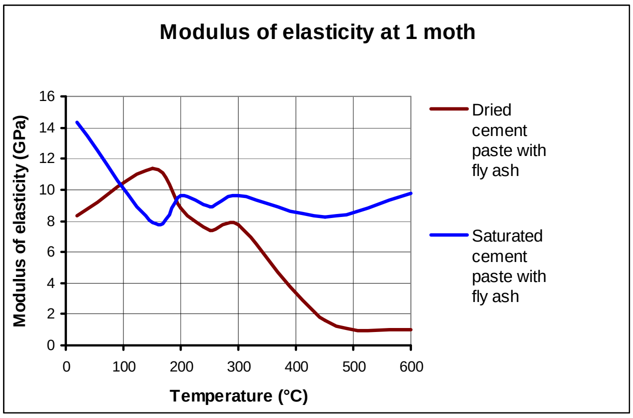

Fig.13 Modulus of elasticity for cement paste with content of fly ash in age 1 month.

The decreasing of values is clear for temperatures higher of 300 °C. The significant influence have cracks on the specimens after temperature loading, like is possible see on Figure 12. The cracks causes creation of the higher deformation in the measurement by extensometer.

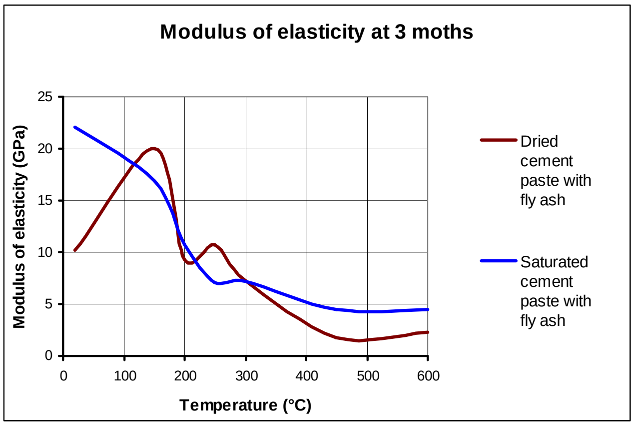

Fig.14 Modulus of elasticity for cement paste with content of fly ash in age three months.

3.4 Fracture energy

Figure 15 shows the evolution of fracture energy in dependence on increasing temperature. The cement paste with fly ash has a fracture energy range between 18 to 46 N / mm. The steady development is evident for dry cement paste and increasing temperature. Conversely fracture energy of saturated cement paste with fly ash has a growth trend with increasing temperature, see Table 9 and Table 10.

Temperature (°C)	Dried conditions (N/mm)	Saturated conditions (N/mm)
20	33.73	18.58
150	28.38	21.69
200	36.84	23.13
300	41.87	28.95
450	-	47.58
600	-	-

Table 9: Fracture energy of the cement paste with fly ash in age one month.

Temperature (°C)	Dried conditions (N/mm)	Saturated conditions (N/mm)
20	52.57	20.53
150	43.61	22.58
200	37.37	33.71
300	41.03	36.58
450	32.83	31.53
600	33.14	31.41

Table 10: Fracture energy of the cement paste with fly ash in age three month.

This effect is probably related to saturation of the material with water and a number of cracks from thermal loading. The older saturated cement paste have growing trends of the fracture energy to the temperature 300 °C, but growing trend stops near to value 31 N/mm at higher temperatures. This steady state is visible for dried specimens, too.

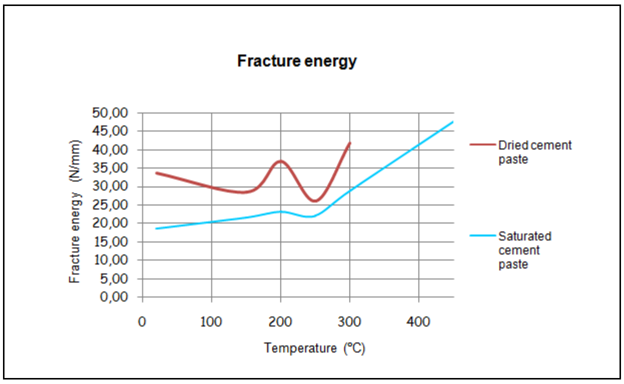

Fig.15 Fracture energy of the cement paste with content of fly ash compared to pure cement paste.

4 Conclusion

As it was shown in the article the material made from fly ash reaches comparable parameters such as cement paste made from Portland cement.

The cement paste mixed with fly ash reaching interesting properties at higher temperatures [7]. The material properties of paste depends on the temperature of which was loaded. The behavior of saturated cement paste with fly ash at high temperatures achieved better performance than that of dried paste [8].

The tests proved the dependence of the dried material and reached the compressive strength and the independence of the parameters for the case of water-saturated material.

A significant effect of energy saving is in the use of fly ash, and therefore the need of only half the amount of cement [9]. It is processed material, which was stored in landfills. The positive trend is reflected in the long term, when the properties cement paste with addition of fly ash is improving.

Very interesting is the perspective of the use of fly ash in the structures of fire protection when at elevated temperature reaches the same and better properties than pure cement paste.

Values of compressive strength and tensile strength in bending of the cement paste with fluidized fly ash is comparable with cement paste with classic fly ash. The material properties and volume density are dependent on the content of water and the proportional representation of fly ash in the cement paste [10].

Acknowledgements

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References:

[1] Neville, A.M., *Properties of Concrete*, John Wiley & Sons, (1997), ISBN 0-470-23527-6 .

[2] *Czech building industry in numbers 2011*, Czech Statistical Office, ISBN 978-80-250-2201-6.

[3] Morsy, M.S., Alsayed, S.H., Aqel, M., Effect of Elevated Temperature on Mechanical Properties and Microstructure of Silica Flour Concrete, *International Journal of Civil & Environmental Engineering*, IJCEE-IJENS Vol: 10 No: 01, ISSN 2077/1185.

[4] Bentz, D. P., Hansen, A. S., Guynn, J. M., Optimization of cement and fly ash particle sizes to produce sustainable concretes, *Cement & Concrete Composites 33 (2011)*, pp. 824–831, www.elsevier.com/locate/cemconcomp.

[5] Padevět P., Mechanical Properties of Cement Pastes, *International* Confernce *70 Yeasr SvF STU.* Bratislava, 2008.

[6] Van Mier, J.G.M., *Fracture Processes of Concrete* CRC Press (1997), ISBN 0-8493-9123-7.

[7] Bahar Demirel, B., ,Kelestemur, O., Effect of elevated temperature on the mechanical properties of concrete produced with finely ground pumice and silica fume, *Fire Safety Journal*, 45 (2010) 385–391, www.elsevier.com/locate/firesaf.

[8] Padevět, P., Bittnar, P., Zobal, O., Changing the Properties of Cement Paste with Addition of Fly Ash in Time, *Experimental Stress Analysis 2012*, June 4-7, 2012, Tabor, Czech Republic, ISBN 978-80-01-05060-6.

[9] Vytlačilová V., "Effect of Individual Components on Characteristic of Cement Composites with Recyclate", in *Proceedings of the 4th WSEAS International Conference on Engineering Mechanics, Structures, Engineering Geology (EMESEG'11),*Corfu, July 2011, Greece, pp. 324-328.

[10] Padevět P., "Measuring of Creep of Cement Paste Specimen", *Proceedings of the 2nd WSEAS International Conference on Applied Mathematics, Simulation, Modeling (ASM'09),* Athens, Greece, 2009, pp. 33-39.