The Changes of Material Properties of the Cement Paste with Fly Ash Exposed to High Temperatures

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Abstract: - The paper describes properties of cement paste included addition of the fly ash. Firstly is described the preparation of the cement paste. 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 strength in bending. Finally the relation between material properties and volume weight is discussed.

Key-Words: - Cement paste, fly-ash, concrete, compression strength, Modulus of elasticity, tensile strength in bending.

1 Introduction

The energy intensity of construction is currently one of the important fields of construction production in the economy. Attention is focused not only on the energy performance of buildings operation.

The effective construction output is associated with the use of modern materials that are readily available and cost competitive. Some of the modern building materials are indeed at a high technological level, but the cost of their production is too high. At present, it is possible to observe the trend of replacement of many traditional materials with new, modern and highly technologically advanced materials. This replacement is unfortunately often accompanied by raising prices above the cost-effective level.

Similarly, it is possible to monitor the trend of compensation for the above mentioned materials such as a bricks advanced products. For them, some qualities are favored at the expense of the other properties. The modern brick masonry is an example. The thermal insulation properties are favored at the expense of the mechanical properties such as strength and brittleness.

Construction is the branch where it is possible to process new raw materials, but also recycled building material [1] and in some cases waste. For example, the concrete structure can be constructed completely from new materials, but also by use of recycled materials. The using of concrete granulates for reuse in concrete production is one of the examples of use of the recycled material. The recycled old concrete is used as filler in concrete. To create cement gel is used a new cement. Protecting the natural environment lies in the processing of waste - old concrete that would otherwise be deposited in a landfill.

Recycled concrete aggregate component is replaced, which would have to be extracted and prepared for use.

Another approach consists in finding a substitute for the binder material, while maintaining properties of the original the binder. Production of the binder - cement is highly energy-intensive process. The clinker burning takes place at temperatures above 1300 °C. Typically, the clinker burning temperature reaches 1450 °C [2]. The clinker is milled in the final product after burning and cooling. The burning and grinding process is energy intensive.

The finding substitutes for cement manufacturers try to streamline the energy intensity of production and price.

One approach is a partial replacement of cement by other binder materials. The second approach is a replacement for cement. Suitable materials in both cases appear to fly ash. The fly ash is a waste material that is results from burning coal in coal-fired power plants. The Czech Republic is one of the largest producers of fly ash per person in the world. The reason for this is the high share of electricity production in the coal power plants.

Fly ash is generated by brown coal desulphurization plants. According to the method desulphurization ash divided into two types. The first type, fluidized ash is generated from the combustion of coal at a temperature of 800 °C. The second type of fly ash is produced by burning coal in conventional furnaces at temperatures from 1200 to 1700 °C.

A person in the Czech Republic accounts for almost 800 kg of fly ash per year. The production of Portland cement accounts per person per year approximately 3500 kg [3]. It follows that the amount of fly ash is not negligible in the case of using additives to the cement paste appears to be an optimal quantity.

The fly ash, such a waste during combustion is stored in landfills. Because the fly ash is lightweight material, it is necessary to bind the storage of water, and would not bother to air dust formation.

Fly ash is an inert material. The building materials are suitable for the processing does not endanger the health of people. Fly ash in some way meets this criterion. Standard fly ash has a low content of SO_3 and it is suitable for processing in the concrete. The conversely fluid ash has a higher content of SO_3 and is the basis for the production of gypsum-based materials.

This paper is devoted to processing of the classical fly ash in the cement paste. Cement paste is a binding material in concrete production. It is therefore possible to focus attention especially on the binder between the cement paste and fly ash.

Fly ash is used first as filler in cement paste. The granulometry range is from 1 micron to 1mm. It is therefore able act as fine filler particles and replacing the fine sands.

The comparable content of fly ash in cement mortar with a lot of sand also brings the negative phenomenon. Size of deformations of shrinkage depends on the water content - moisture cement paste. Quartz sand is non-absorbent material. Its proportion in the cement paste has a stabilizing effect in terms of creep.

On the other hand, in the recent past was discovered by a positive quality fly ash – hydration [4]. Fly ash is able, in cooperation with the cement to react only at the beginning of setting and hardening of the cement paste, but also for several months. Thus it is able to support the growth of cement paste such as compressive strength, tensile strength and modulus of elasticity [5].

A very positive property of the selected structures is the development of lower heat of the hydration when using of fly ash in concrete. The development and the amount of heat of hydration depend on the amount of cement in the massive construction. The content of fly ash causes lower production of the hydration heat in cement substitution.

The mixing water and cement creates CSH gel, from it Portlandit in the cement paste. CSH gel is activator of fly ash, which produces another type of gel [6].

At present, in the known structures is the cement replaced by the fly ash up to a maximum of 60 %. Frequently, the amount of fly ash in concrete is up to 20 %. Known and historically important building is the Orlik dam on the river Vltava [7]. Here it was 50 years ago, used amount of fly ash in the construction of nearly 28 % of the cement.

2 Material properties of cement pastes with addition of fly ash

As has been discussed in the introduction the important material properties are the compressive strength and the tensile strength, elastic modulus and fracture energy. Changes in the properties of cement paste [8] 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 around 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 [9].

Tensile bending strength is determined from another type of test material. 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. Methods for its determination are several. 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 [10].

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. The first one is determined from the sizes of specimens and weight of water dried specimens. The second one is calculated from the water saturated specimens.

3 Material tests and their preparation

The cement paste containing the fly ash is a homogeneous material. Homogeneity of the material allows the use of small specimens for testing.

For the experiments was chosen size of specimens 20 x 20 x 100 mm. For production was initially chosen cement paste made from water and Portland cement CEM I 42.5 R. Water-cement ratio for the preparation of cement pastes was selected 0.4 [11]. 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 fly ash. The ratio between the amount of cement and fly ash was 1:1.

Specimens were produced in steel moulds with precise dimensions.

After the concreting, all specimens were stored in to water bath. Testing was performed at the age of one and three months [12]. The article presents the results of the test after 28 days. By the time the test specimen was stored in a water bath. Before testing, the specimens were removed from the water bath and divided into the two groups. First one is the dried solids. Second one group is the specimen saturated with water. All specimens were loaded at high temperatures. For testing were selected at 20, 150, 200, 250, 300, 450 and 600 °C.

Temperature (°C)	Condition	
	Dried	Saturated
20	15 specimens	15 specimens
150	15 specimens	15 specimens
200	15 specimens	12 specimens
250	15 specimens	15 specimens
300	12 specimens	14 specimens
450	12 specimens	15 specimens
600	14 specimens	13 specimens

Table 1: Conditions of tested specimens.

Number of specimens in Table 1 is determined so that the 6 specimens was used for the compression test, 5 for the test tensile strength in bending and 4 specimens for the determination of fracture energy. Different numbers of specimens are caused by their damage during testing. The same amount of specimens was used for testing at the age of 3 month.

After removing specimens from the water bath followed by thermal loading, the specimens were heated to a specific temperature at 100 °C / hour, as it sees in Figure 1. Subsequently, the temperature was defined by lever of the temperature loading. The specimen was heated for 24 hours and then the temperature was decreased by naturally cooling to 20 °C. During heating, firstly the evaporation of free bound water occurred and the secondly material damage is caused due to the pressure of hot water vapor. Damage of specimens is viewable at Figure 2. High temperatures caused the creation of cracks on the surface of specimens.

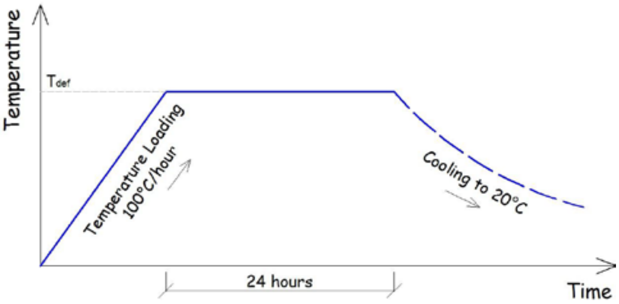

Fig. 1: Temperature loading of cement paste.

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

Fig. 2: Damage of specimens by temperature (600°C).

The compression strength was tested on six specimens. The specimens were prepared from the three solids so that it was cut into two equal parts the height of 50mm.

Specimens for the test in tension in bending were used in the original size and their numbers was 5 The Modulus of elasticity was measured when the test compressive strength was realized [13].

Each specimen was loaded into 3 times to 1/3 the expected strength and then it was carried out compression test [14].

Tests on the fracture energy were carried out in the experiments, too. Four specimens in each group were provided with the notch to half the height of section [15].

4 Material properties – comparison

First of all volume density is possible compare. Table 2 shows the volume density of cement pastes without admixture and with admixture of fly ash.

Temperature (°C)	Volume density of pure cement paste (kg/m^3)	Volume density of cement paste with fly ash (kg/m^3)
20	2058	1322
150	-	1339
200	1700	1303
250	-	1340
300	1625	1324

Table 2: Properties of specimens before measuring of the volume weight.

The table shows a gradual reduction in volume weight of cement paste without fly ash. By heating the specimen at 300 °C, volume weight is reduced of 400 kg/m^3 [16]. Conversely dried specimens containing fly ash still have the same density, although the temperature was raised to 300 °C. The Table 9 shows the change of volume weight of the cement pastes with fly ash, which were heated to the specified temperature and then refunded for 2 days in the water.

Volume weight of saturated specimens mixed with fly ash was near to 1750 kg/m^3 (Table 9).

A comparison of the volume densities can be seen in a significant reduction in the manufacture of mixtures containing fly ash. A very significant difference is obtained by comparing the values for pure cement paste and the paste mixed with fly ash in the dry state. The difference reaches almost 700 kg/m^3.

4.1 Compression strength

The compressive strength of specimen is divided with the content of fly ash into two groups. In Table 3, is possible see a comparison between the cement paste tested after heating and cement paste with fly ash, which was tested in water-saturated state and in the dried state.

Temperature (°C)	Pure cem. paste (MPa)	Cem paste with fly ash – dried (MPa)
20	60.03	34.33
150	86.85	28.39
200	86.92	29.19
250	-	27.82
300	66.49	24.57
450	15.73	13.88
600	-	8.79

Table 3: Compression strength of dried specimens.

Cement paste shows an increase in strength at 200 °C. Change comes at increasing temperatures up to 450 °C, which significantly decreases the compressive strength of 75 % of the strength of 66.49 MPa at 300 °C. The behavior of cement paste with fly ash was expected similar to pure cement paste. Significantly different behavior in compression is evident particularly in the area of 200 °C. For region 200 °C was chosen denser of temperature distribution.

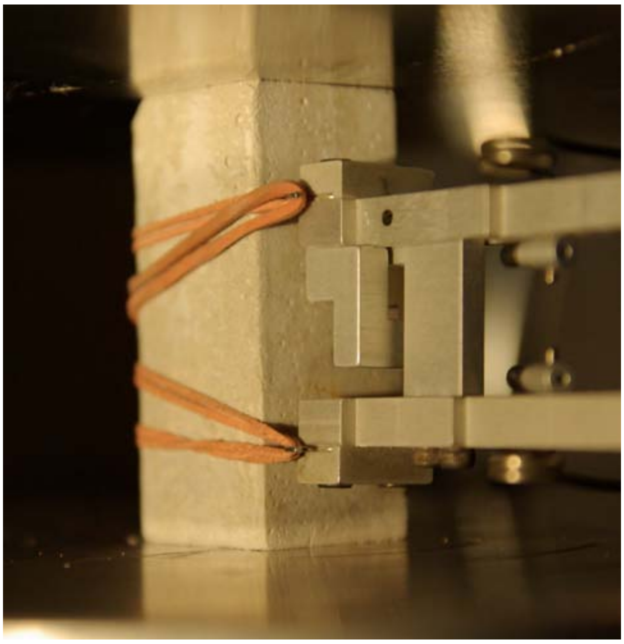

Fig. 3: Cement pastes specimen in compression test. On the surface is fastened the extensometer.

Temperature (°C)	Cem paste with fly ash – saturated (MPa)
20	24.68
150	22.42
200	20.08
250	27.62
300	30.84
450	23.19
600	20.13

Table 4: Compression strength of cement paste with fly ash - saturated specimens.

Table 4 shows the compressive strength of saturated specimens. In comparison with the desiccated specimens in Table 3, is initially lower strength of 10 MPa. At the temperature of 250 °C, the strength of dried and saturated specimens has already mutually corresponding. The increasing of strength was compared to the values at 200 °C for the saturated material. Conversely dried specimen show sustained slight decrease of strength from the outset.

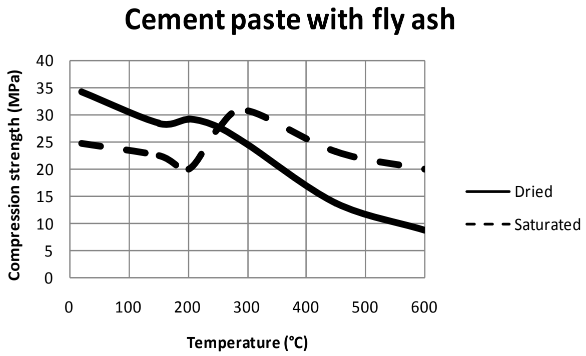

Fig. 4: Compression strength of cement paste with fly ash.

The decreasing trend of strength at the temperatures higher than 200 °C is evident. Rate of decline in strength is formed. The strength of dried specimens reaches only 8.79 MPa at temperature 600 °C. The compressive strength of dried specimens with the addition of fly ash is comparable with the strength of the pure cement paste.

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

Thermal load of 600 °C significantly damage the material structure of the cement paste. Water-saturated cement paste reaches strength 20.13 MPa. Strength of dried cement paste is only 8.79 MPa. This implies a significant effect on the resulting moisture paste compressive strength at high temperatures.

The comparison of the development of the compressive strength of cement paste containing fly ash is shown in Figure 4.

Fig. 5: The specimen after compression test. Typical brittle cracks are viewable on the specimen.

The behavior of the cement paste in compression shows characteristics of quasi brittle failure. Figure 5 shows the main cracks formed in the direction of loading material. Compression test was shear plane in the specimen, which is very well visible in the picture.

The results of tensile strength in bending are shown in Table 5. The table is again compared strength of dry cement paste tested at high temperature and dry cement paste mixed with fly ash.

4.2 Tensile strength in bending

Tensile strength in bending of cement paste culminates at a temperature of 200 °C. Conversely 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 the temperature, as shown in Table 5. The significant decrease occurs after the temperature reaches 300 °C. This behavior corresponds to the compressive strength of dried cement paste. The tensile strength in bending decreases from the beginning when the temperature increases. The trend of uniform decline stops at 250 °C, it is different from the value of the compressive strength of almost 100 °C. The decrease the compressive strength of dried paste is stopped at a temperature of 150 °C. Similar halting the decline of tensile strength in bending is observed above the temperature of 450 °C. The residual strength of dried cement paste with fly ash is observable above the temperature of 450 °C.

The calculation strength in tension in bending is governed by relation for the 3-point bending cited in the first equation.

$$\sigma = 1.5 \cdot \frac{F \cdot l}{b \cdot h^2} \quad (1)$$

Where:

σ is tensile strength in bending;
F is maximum load;
l is span of the supports;
b is width of specimen;
h is height of specimen.

The distance between supports was 79.5 mm. Dimensions b and h match to the cross-sectional dimensions. Force F was considered as the highest load achieved.

Temperature (°C)	Pure cement paste (MPa)	Cement paste with fly ash – dried (MPa)
20	2.69	7.48
150	-	6.36
200	6.06	5.52
250	-	5.08
300	1.89	5.56
450	0.30	0.48
600	-	0.36

Table 5: Tensile strength in bending of the cement paste and the cement paste with fly ash - dried specimens.

The saturated specimens by water show 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. However, this value is considerably higher than in the case of pure cement paste and dry cement paste with fly ash.

Regular decrease of tensile strength is suspended between the temperatures of 200-250 °C. The strength is slightly increased at 250 °C. This phenomenon is very similar to the increase the strength, which carried out around 300 °C for dry cement paste with fly ash as it possible in Figure 6. A further decrease of tensile strength in bending is slower than above the temperature of 300 °C.

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.

It follows that the properties of compressive and flexural strength is 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. The ratio of compressive strength and flexural strength is equal to the value of 25 at 600 °C and dry condition. In contrast, the ratio of strength for the water saturated paste is equal to 8.76 at 600 °C.

Temperature (°C)	Cement paste with fly ash – saturated (MPa)
20	5.70
150	4.19
200	3.79
250	3.98
300	3.14
450	2.48
600	2.32

Table 6: Tensile strength in bending of the cement paste with fly ash - saturated specimens.

As shown in Table 6 is declining trend in tensile strength in bending observable also in saturated specimens with fly ash. The ratio of the compressive strength and flexural strength is slightly higher than for the dried specimens, specifically 5 times in this case for temperature 150 °C.

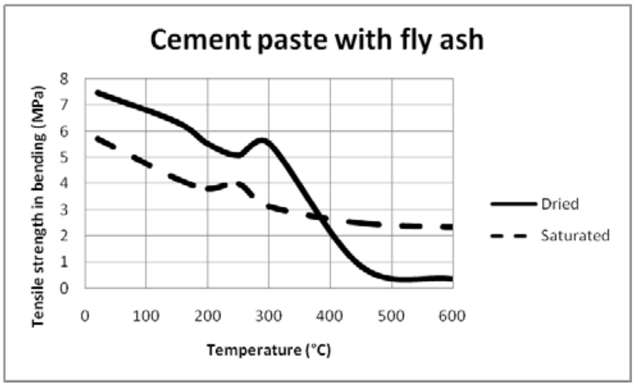

Fig. 6: Tensile strength in bending of cement paste with fly ash.

The ratio between the compression strength and tensile bending strength increases with increasing temperature. It is not possible to say that this strength ratio is constant. The function of this relation is simply linear.

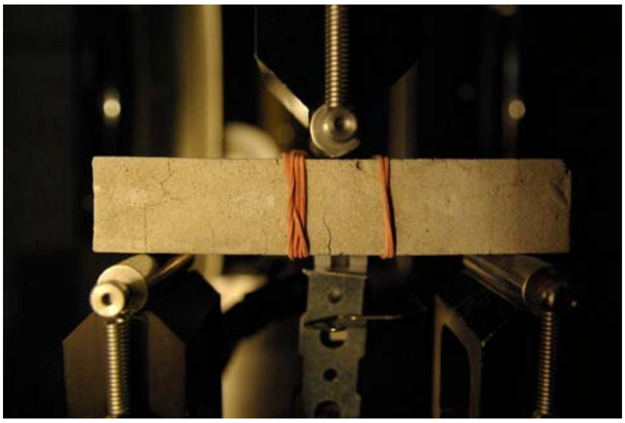

Fig. 7: Instrumentation of the test of tensile strength in bending.

The results of tensile bending are highly depended on the intensity of material failure due to temperature. The Figure 7 shows the marginal crack of the beam in the middle under the load. The regular cracks on the specimen are caused by heating and subsequent cooling of the sample. These cracks greatly affect the size of the maximum force and deflection of specimen. High temperatures cause higher number of cracks. Significant cracks are the cause of large deflections of the material. A material exposed to high temperatures shows higher deflection in three-point bending test. A large deflection in the middle the beam is shown in Figure 7.

4.3 Modulus of elasticity

The modulus of elasticity shows a downward trend as well. The results of values of modulus of elasticity are described in the Figure 8.

The steady downward trend of modulus elasticity is obvious in the case of the dried paste and for temperatures higher than 150 °C. When the temperature reaches 450 °C modulus is only 1.59 GPa. 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 big difference between the saturated and desiccated specimens is shown in Figure 8. At the temperature of 450 °C the saturated specimens reached about 6.67 GPa (Table 6) higher modulus of elasticity than the specimens dried.

The decrease of modulus of elasticity dried cement paste with fly ash is more than five times at 450 °C against the 20 °C. The decrease the value of modulus of elasticity the saturated paste is only doubled. Double decrease is achieved at the temperature of 150 °C.

Temperature (°C)	Modulus of elasticity of cement paste – dry condition (kg/m^3)	Modulus of elasticity of cement paste – saturated condition (kg/m^3)
20	8.3	14.36
150	11.38	7.86
200	8.82	9.65
250	7.36	8.92
300	7.72	9.60
450	1.59	8.26
600	-	9.80

Table 6: Tensile strength in bending of the cement paste with fly ash - saturated specimens.

Saturated cement paste with fly ash is in terms of deformation at higher temperatures better then the dried paste. Saturated material retains deformation properties almost the same as at 20 °C.

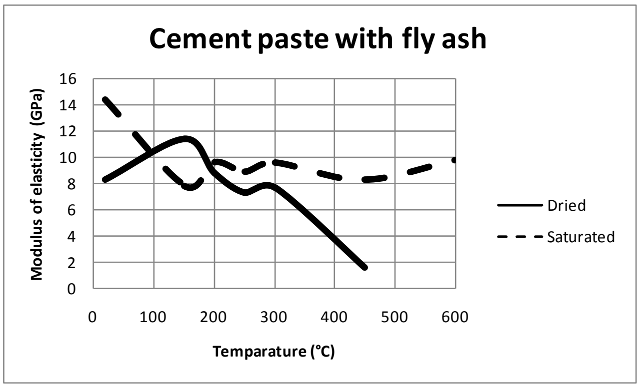

Fig. 8: Compression strength of cement paste with fly ash.

4.3 Correlation of the data

It can be assumed that some properties of cement paste will affect the quality of the other. Very interesting is the correlation between the parameters of cement paste and results. The basic parameters of cement paste as size, weight, volume weight. On the other hand, the results are compression and tensile bending strength, elastic modulus and fracture energy. The resulting properties can be estimated according to the parameters already.

The correlation is defined by:

$$r_{xy} = \frac{s_{xy}}{\sqrt{s_x^2 s_y^2}} \quad (2)$$

Where:
r_{xy} is coefficient of correlation;
s_{xy} is covariance;
s_x is variance of the first variable;
s_y is variance of the second variable.

The compression strength of the cement paste with fly ash was correlated with the volume weight. The volume weight was measured at several stages of preparation of the specimens. The specimens were subjected to the first measuring the weight and volume immediately after production and before their dipping into the water.

The second specimens' weight measurement was carried out before testing the material properties. First, the weight of the dried specimens was measured (Table 7) and the second water-saturated mass specimens was measured (Table 8). Water-saturated specimens were weighed after saturation of the material with water. It was carried out after heating the specimens to the specified temperature. The volume weight was determined form the data of 6 specimens intended for the compression test.

Temperature (°C)	Volume weight of cement paste – dry condition (kg/m^3)	Volume weight at start of the maturation (kg/m^3)
20	1326	1542
150	1350	1555
200	1309	1651
250	1339	1605
300	1324	1648
450	1298	1658
600	1298	1675

Table 7: Volume density for temperature. It is state for testing of the dried specimens.

The volume weight was same at the beginning after removing the formwork as it shown in Table 7 and Table 8. The dried specimens had density 1300 kg/m^3 close, but from the increasing temperature this value declined. Volume weight of water-saturated cement paste was close to the value of 1730 kg/m^3.

Temperature (°C)	Volume weight of cement paste – saturated specimens (kg/m^3)	Volume weight at start of the maturation (kg/m^3)
20	1743	1611
150	1698	1618
200	1736	1532
250	1704	1695
300	1737	1609
450	1737	1502
600	1725	1648

Table 9: Volume density for temperature. It is state for testing of the saturated specimens.

Variances of the achieved compressive strength and volume weights before the test and after the concreting of the specimens were used in the Equation 2.

The compressive strength and the density of dry cement paste achieve the correlation coefficient 0.7048. Conversely, the compressive strength and the density of cement paste with fly ash after concreting achieved the correlation coefficient -0.7540. It is possible to conclude that the dry strength of the cement paste depends strongly on the density in both cases.

On the contrary, a strong association between water saturated specimens and the compressive strength was not demonstrated. The correlation coefficient between the compressive strength and density of saturated specimens was 0.006. The correlation coefficient between the compressive strength and density of the vault was 0.3496.

5 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. The material properties of paste depends on the temperature on which was loaded. The behavior of saturated cement paste with fly ash achieved better performance at high temperatures than that of dried paste. The compared data showed a significant relationship between the input parameters and the resulting properties.

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, and therefore the need of only half the amount of cement. 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.

Acknowledgements

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