

## LABORATORY TESTING OF FLY ASH

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Preliminary note

Fly ash is one of the most common waste materials created by burning of coal. It is composed of smaller particles, consisting mainly of aluminosilicate-glass, mullite and quartz, which are collected by electrostatic separators (filter) of gaseous products arising from the combustion of coal. This paper presents the pozzolanic properties, mechanical properties, chemical and mineral composition of fly ash obtained in the combustion process in a power plant Nikola Tesla A (PPNT A) with the addition of a binder hydrated lime and cement. The aim of research is to test the possibility of using the fly ash from PPNT A for making elements of road structures (top and bottom layers of the road). Fly ash can be used to create elements for road structures, but it must be taken into account that the embedding is done in segments, isolated from the influence of surface water and groundwater.

**Keywords:** *chemical composition; fly ash; mechanical properties; mineral properties; pozzolanic properties; X-ray diffraction*

### Laboratorijska ispitivanja elektrofilterskog pepela

Prethodno priopćenje

Elektrofilterski pepeo (leteći pepeo) je jedan od najzastupljenijih otpadnih materijala nastao sagorijevanjem uglja. Čine ga sitnije čestice, sastoji se uglavnom od amorfnog-staklastog materijala, mulita i kvarca, koji se sakuplja elektrostatskim separatorima (filterima) iz plinovitih produkata nastalih uslijed sagorijevanja uglja. U radu su prikazana pucolanska svojstva, mehanička svojstva, kemijski i mineralni sastav i svojstva elektrofilterskog pepela dobivenog u procesu sagorijevanja u termoelektrani Nikola Tesla A (TENT A) s dodatkom veziva hidratiziranog vapna i cementa. Cilj istraživanja je mogućnost uporabe elektrofilterskog pepela za izradu elemenata cestovnih konstrukcija (gornji i donji stroj puta). Elektrofilterski pepeo se može primjenjivati za izradu elemenata cestovne konstrukcije, ali treba voditi računa da se ugrađivanje odvija u segmentima, izoliranim od utjecaja površinskih i podzemnih voda.

**Ključne riječi:** *elektrofilterski pepeo (leteći pepeo); kemijski sastav; mehanička svojstva; mineralna svojstva; pucolanska svojstva; rentgenska difrakcija*

## 1 Introduction

Fly ash is one of the most common waste materials created by burning of coal [1]. It is composed of smaller particles, consisting mainly of aluminosilicate glass, mullite and quartz, which are collected by electrostatic separators (filter) of gaseous products arising from the combustion of coal [2]. Serbia produces about 5 million tons of fly ash annually during coal combustion in the power plants [3]. The samples subject to laboratory tests were taken from the thermal power plant Nikola Tesla A (PPNT A) in Obrenovac.

This paper presents the pozzolanic, mechanical, chemical and mineral composition and properties of fly ash obtained in the combustion process in a PPNT A with the addition of a binder hydrated lime and cement. Tests were performed after 7 and 28 days in a humid environment with different amounts of extra binder:

- samples without the addition of binders,
- a sample with the addition of 2 % of binder,
- a sample with the addition of 4 % of binder,
- the sample with the addition of a binder 6 % (hydrated lime and cement).

The mineral composition of fly ash and slag from PPNT A is determined from the diagram of X-ray examination. The chemical composition was obtained by testing samples in accordance with the standards SRPS B.H8.359 369 [4-14] – Methods of test for coal and coke - Determination of the chemical composition of the ash, the basic provisions, and other related standards for the determination of some oxides that are provided standard. The chemical properties of fly ash are the most important indicators to assessing their suitability as construction materials or as raw materials for the production of

building materials. The results were compared with the average results of previous tests in order to determine changes.

Pozzolanic properties are tested according to standard SRPS B.C1.018 2001 [15] due to which the class of pozzolanic material has been determined. Classes of pozzolanic material are obtained by testing the samples to compressive strength and tensile strength in bending after 7 days. Pozzolanic properties of the fly ash determined by mechanical properties are the most important in the application of ash as road-building material. Mechanical properties of fly ash have been determined on the test samples. The experiments were done based on the standards that are in use in the European Union. The following mechanical tests have been made:

- the compressive strength – uniaxial strength,
- the indirect tensile strength – Brazilian experiment,
- the modulus of elasticity.

The aim of research is to test the possibility of using the fly ash from PPNT A for making elements of road structures (top and bottom layers of the road).

## 2 Experimental

### 2.1 Mineral properties of fly ash

Mineral properties of samples of fly ash from thermal plant PPNT A production were determined by X-ray studies at the Laboratory of Crystallography Faculty of Mining - Geology in Belgrade. X-ray diffraction is a non-destructive experimental method based on the usage of the diffraction of X-rays with crystal lattice sized wavelengths. This method is primarily used for quality analysis, due to the fact that it enables the identification of present crystal components, type of crystal lattice, the

presence of certain phases in the system, the deformation of crystal lattice, the size of crystals. It can also be used for quantity analysis, or more precisely, for an estimation of different components share in the content of the sample [16].

All samples were tested on the powder diffractometer PHILIPS PW 1710. The diffraction patterns (diagrams) were obtained with CuK $\alpha$  radiation ( $\lambda = 1,54178 \times 10^{-10}$  m), created in the X-ray tube at 30 mA and a voltage of 40 kV. Record the sample is performed in the range of  $2\theta$  from 5 to 50° with a step of 0,02° and a retention time of 0,5 s at each step.

The data position of the diffraction peaks  $2\theta$  (°), the value of the flange distance  $d$  (m), and the corresponding intensities ( $I$ ) use impulse unite (imp).

Mixtures are made in order to examine the process of activation of fly ash by components present, their mutual reaction and the possible emergence samples of fly ash from the PPNT A with small share of additives (hydrated lime and cement) in a humid environment.

## 2.2 Chemical composition of fly ash

Chemical composition of the fly ash from PPNT A was tested in laboratory for concrete and binders in "Highway Institute A. D.". Chemical analysis was performed on samples with volume of 10 g for "Series 1" (S1) and 10 g for "Series 2" (S2). In period from 1983 to 2004, testing of chemical composition was done for 59 samples and results are presented in Tab. 1.

**Table 1** The results of the chemical composition of ashes from PPNT A for the period from 1983 to 2004

Chemical composition	min	max	mean
	Ash composition, mass / %		
SiO <sub>2</sub>	21,98	71,75	53,30
Al <sub>2</sub> O <sub>3</sub>	7,51	27,64	20,72
Fe <sub>2</sub> O <sub>3</sub>	2,16	10,11	7,23
CaO	2,12	16,10	8,74
MgO	0,64	4,39	2,64
SO <sub>3</sub>	0,66	3,27	1,76
S	0,26	1,31	0,70
Na <sub>2</sub> O	0,08	0,72	0,32
K <sub>2</sub> O	0,25	1,49	0,74
module $R = \frac{(SiO_2 + AlO_3)}{(CaO + MgO + Fe_2O_3)}$	2,18	9,98	4,38
SiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub>	1,92	6,27	2,66

### 2.2.1 The classification of fly ash on the basis of chemical composition

There are several fly ash and slag classifications based on chemical composition, among which the following are the most important:

1. International system of fly ash classification

**Table 2** The international system of classification of fly ash

Group	Ash	SiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub>	CaO / %	SO <sub>3</sub>
I	Aluminosilicate	>2	<15	undefined
II	Silicate - Aluminate	<2	<15	<3
III	Sulfate - Alkaline	undefined	>15	>3
IV	Other (lime)	undefined	>15	<3

2. The classification of fly ash according to the module  $R$  size

$$R = \frac{(SiO_2 + AlO_3)}{(CaO + MgO + Fe_2O_3)} \quad (1)$$

According to module  $R$  size, fly ash can be divided into:

- silicate – calcium  $R < 2$ ,
  - silicate  $R = 2 \div 6$ ,
  - silicate – aluminum  $R > 6$ .
3. Classification of fly ash according to standard ASTM C – 618 from 1994 (ASTM C 618, 1994)

Based on certain oxide content, fly ash can be divided into:

**Table 3** Classification of fly ash to ASTM C-618 [17]

Class	Ash	Chemical composition
F	Sour	Very low in Ca, slather Fe, (SiO <sub>2</sub> +Al <sub>2</sub> O <sub>3</sub> +Fe <sub>2</sub> O <sub>3</sub> ) > 70 (%)
C	Alkaline	High content CaO (SiO <sub>2</sub> +Al <sub>2</sub> O <sub>3</sub> +Fe <sub>2</sub> O <sub>3</sub> ) > 70 (%)

The main flaw of ASTM standard is that it does not take the content of CaO into consideration, which significantly affects the pozzolanic properties of fly ash. Amendment of this, standard was proposed by McCarthy (1994).

**Table 4** Classification of fly ash in accordance with ASTM C-618 amended classification according to McCarthy (1990) [17]

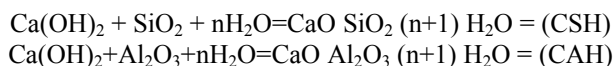
Class	Ash	Chemical composition	McCarthy – CaO / %
F	Sour	Very low in Ca, slather Fe, (SiO <sub>2</sub> +Al <sub>2</sub> O <sub>3</sub> +Fe <sub>2</sub> O <sub>3</sub> )>70(%)	<10
C	Alkaline	High content CaO (SiO <sub>2</sub> +Al <sub>2</sub> O <sub>3</sub> +Fe <sub>2</sub> O <sub>3</sub> )>70(%)	10 ÷ 20
			>20

### 2.3 Pozzolanic properties of fly ash

Pozzolanic properties of the fly ash are tested in the laboratory for concrete and binders in the "Highway Institute". According to standard (SRPS B.C1.018 2001 [15]), pozzolanic materials are natural or artificial, silicate, silicate-aluminate, silicate-carbonate substances or their combination. Pozzolanic materials are getting harder when they are finely milled in the presence of water, they react at environmental temperature with calcium hydroxide (Ca(OH)<sub>2</sub>) and form calcium silicate and calcium-aluminate, which provide them strength. Classification of pozzolanic materials is done in three ways: by the content of reactive silica (SiO<sub>2</sub>), according to the granulometric composition and mechanical properties. Pozzolanic properties of the fly ash determined by the mechanical properties are the most important in the application of ash as road-building material. Depending on the minimum compressive strength and tensile strength in bending after 7 days of making samples, three classes of pozzolanic material can be identified as given in Tab. 5.

The composition of pozzolan mainly includes oxides SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>. These oxides are usually present in the form of an amorphous (glassy phase) that causes pozzolanic activity: during the reaction between Al and Si

oxides from the glass part of the fly ash and  $\text{Ca}(\text{OH})_2$  hydrated aluminates (CAH) and silicates (CSH) of calcium are formed, which are hardly soluble in water.



**Table 5** Classification of pozzolanic materials [15]

Class	Tensile strength / kPa	Compressive strength / kPa	Pozzolanic activity
5	2000	5000	Positive after 15 days
10	3000	10000	
15	4000	15000	

The intensity of these processes depends largely on the ash fineness (particle size and particle size distribution), so that the pozzolanic reactivity increases with the specific surface of ash. International standard ASTM C 311 describes the procedures of chemical and physical methods for characterization of fly ash, and standard ASTM C 618 provides chemical and physical criteria related to classification. Based on the international standard ASTM C 618, chemical conditions that classify any fly ash are given in the Tab. 6.

**Table 6** Classification of fly ash to ASTM C 618 [17]

Characteristics	Class		Natural pozzolan
	F	C	N
$\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ min (%)	70	50	70
$\text{SO}_3$ max (%)	5	5	4
Humidity max (%)	3	3	3
Loss on ignition max (%)	6	6	10

Class F of fly ash is silicate, or silicate and aluminat material whose selfhardening effect is small or absent. In the presence of moisture at room temperature fly ash reacts with calcium hydroxide and forms a product which helps it to be harder.

Class C with pozzolanic properties also has a feature of selfhardening.

Class N is crude or calcified natural pozzolan.

Two series of Series 1 (S1) and Series 2 (S2) samples were taken for testing, weighing about 20g per sample.

## 2.4 Mechanical properties of fly ash

### 2.4.1 Compressive strength – uniaxial strength

For a general overview of the ash characteristics as a building material and design of the stability and capacity, the parameters, obtained by determining the uniaxial compressive strength and the free lateral spreading, are used. For this experiment the cylindrical shape sample is used and prepared by the procedure of Proctor homogenized samples, with diameter of ( $D$ ) 100 mm and height of ( $h$ ) 200 mm, which satisfied the standard height to diameter ratio of 2:1. The samples were specially prepared in a three-part mold and compacted by Proctor procedure in five layers. All prepared samples were stored for a while before the failure. The test was performed with three different series of samples:

- samples cured in a wet chamber for 7 days,
- samples cured in a wet chamber for 28 days,
- samples cured in a wet chamber for 112 days.

The uniaxial strength is tested according to European standard EN 13286-41 [18].

### 2.4.2 Indirect tensile strength – Brazilian experiment

Indirect tensile strength (Brazilian experiment) applies primarily to stabilized bearing layers. The development of this method allows the parameter assessment, such as tensile strength, tensile deformation, modulus of elasticity, Poisson's ratio, at fail and other mechanical properties.

For the experiment the cylindrical shape samples were used  $D = 102$  mm with the length ( $L$ ) 116 mm, and specially prepared in a standard mold by Proctor procedure in three layers. These samples were cured in a wet chamber for 28 days.

Indirect tensile strength – Brazilian experiment, was done according to the standard EN 13286-42 [19].

### 2.4.3 The modulus of elasticity

The modulus of elasticity represents the relationship between stress and deformation. Physical dimension of the modulus is the stress and is expressed in MPa. The experiment was done according to standard EN 13286-43 [20].

## 3 Results and discussion

### 3.1 The chemical composition of fly ash

Results of chemical analysis of fly ash samples taken in Series 1 (S1) and Series 2 (S2) from PPNT A, are shown in Tab. 7.

**Table 7** Results of chemical analyses of fly ash taken in S1 and S2 at location of PPNT A

Chemical composition	S1	S2	Mean values 1983 ÷ 2004
	Ash composition, mass / %		
Loss on ignition 1000 °C	6,43	7,20	-
$\text{SiO}_2$	51,64	51,43	53,30
$\text{Al}_2\text{O}_3$	21,87	22,01	20,72
$\text{Fe}_2\text{O}_3$	5,54	6,03	7,23
CaO	1,05	1,16	8,74
MgO	3,01	2,83	2,64
$\text{SO}_3$	0,85	0,54	1,76
S	0,33	0,21	0,70
$\text{Na}_2\text{O}$	0,57	0,43	0,32
$\text{K}_2\text{O}$	1,05	1,16	0,74
module $R = \frac{(\text{SiO}_2 + \text{AlO}_3)}{(\text{CaO} + \text{MgO} + \text{Fe}_2\text{O}_3)}$	7,65	7,32	3,97
$\text{SiO}_2/\text{Al}_2\text{O}_3$	2,36	2,33	2,57

Comparison of the chemical composition of fly ash taken in S1 and S2, with medium chemical composition of 59 samples of fly ash in corresponding periods from 1983 to 2004, showed the following variations:

- the content of  $\text{SiO}_2$  is lower by about 2 %,
- the content of  $\text{Al}_2\text{O}_3$  is increased by 1 to 1,5 %,
- the content of  $\text{Fe}_2\text{O}_3$  is lower by 1 to 1,5 %,
- the content of CaO in the ash is lower by about 7 %.

### 3.1.1 Classification of fly ash according to chemical composition

According to international system of fly ash classification, the fly ash from PPNT A belongs to group I, that is, aluminosilicate ashes.

Classification of the module R size, fly ash from PPNT A belongs to silicate-aluminum ashes. According to ASTM C-618 standard with McCarthy amendment, fly ash and slag belong to class F – acidic ashes. There are several other ash classifications, by chemical composition, content of specific oxides, means of depositing and other, but previous classifications are most commonly used in practice.

### 3.2 Test results of mineral composition of fly ash

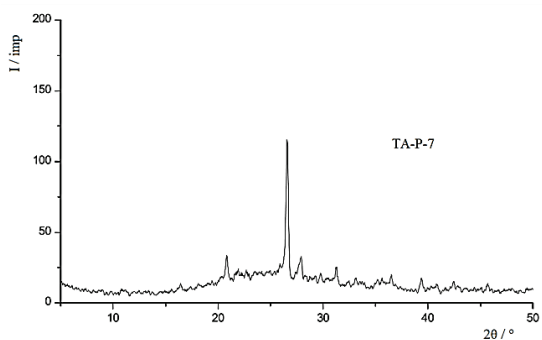
X-ray diffraction is determined by the mineral composition of the ash and slags samples. The characteristic peaks of the mineral composition are show in Tab. 8 and Fig. 2a, 3a, 5a, 6a.

**Table 8** Showing identified crystalline phases in the samples

Name of minerals and label on figure	Formula	Reference peak $\times 10^{-10} / \text{m}$
Quartz (Q)	SiO <sub>2</sub>	3,34
Feldspar (f)	NaAlSi <sub>3</sub> O <sub>8</sub> - CaAl <sub>2</sub> Si <sub>2</sub> O <sub>8</sub>	3,20
Mullit (M)	Al <sub>6</sub> Si <sub>2</sub> O <sub>13</sub>	5,40
Melilite (MI)	Ca <sub>2</sub> MgSi <sub>2</sub> O <sub>7</sub> - Ca <sub>2</sub> Al <sub>2</sub> SiO <sub>7</sub>	2,86
Anhydrite (An)	CaSO <sub>4</sub>	3,50
Cristobalite (Kr)	SiO <sub>2</sub>	4,06
Hematite (He)	Fe <sub>2</sub> O <sub>3</sub>	2,70
Free CaO	CaO	2,40
Portlandite (P)	Ca(OH) <sub>2</sub>	4,90
Gypsum (G)	CaSO <sub>4</sub> 2H <sub>2</sub> O	7,58
Calcium (C)	CaCO <sub>3</sub>	3,04
Alite (C <sub>3</sub> S)	Ca <sub>3</sub> SiO <sub>5</sub>	2,78
Belite (C <sub>2</sub> S)	Ca <sub>2</sub> SiO <sub>4</sub>	2,88
Tricalcium aluminate (C <sub>3</sub> A)	Ca <sub>3</sub> Al <sub>2</sub> O <sub>6</sub>	2,69
Brown millerite (C <sub>4</sub> AF)	Ca <sub>4</sub> Al <sub>2</sub> Fe <sub>2</sub> O <sub>10</sub>	2,64

#### 3.2.1 The sample exposed to 7 days in a humidified environment without addition of binder (TA-P-7)

X-ray examination has shown that the fly ash is mainly amorphous, but there are also present crystal phase feldspar, melilite, mulit, very little anhydrite and quartz (Fig. 1).

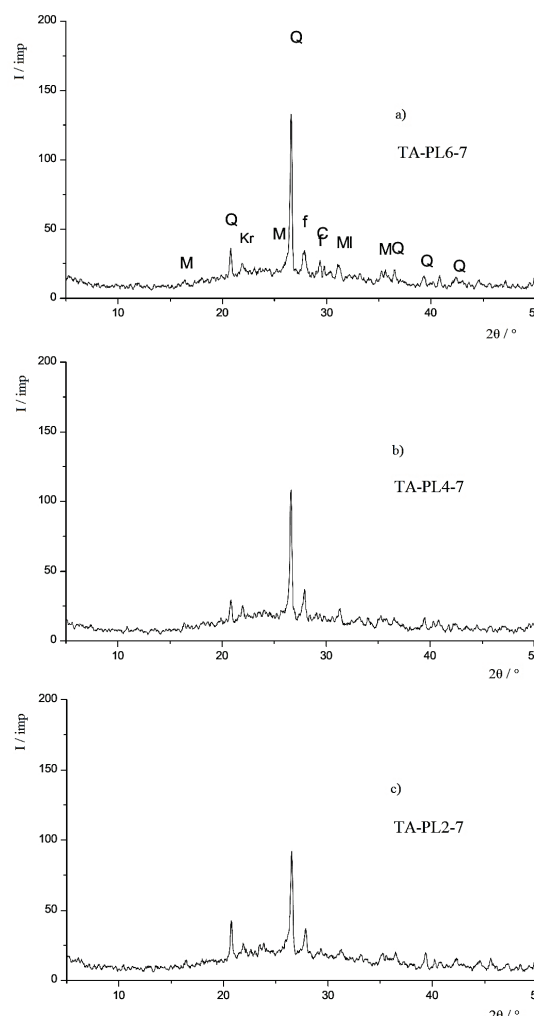


**Figure 1** The X-ray diagram of the sample, which is exposed for 7 days in a humid environment without the addition of binders

#### 3.2.2 The samples were exposed for 7 days in a humidified environment with the addition of hydrated lime, 2, 4 and 6 % (TA-PL2-7, TA-PL4-7 and TA-PL6-7)

Samples with 2, 4 and 6 % limestone are very similar. Their differences and differences in relation to the initial ash are most evident in the diagrams in the field of small-angle  $2\theta$  ( $8 \div 15^\circ$ ) and  $29 \div 30^\circ 2\theta$ .

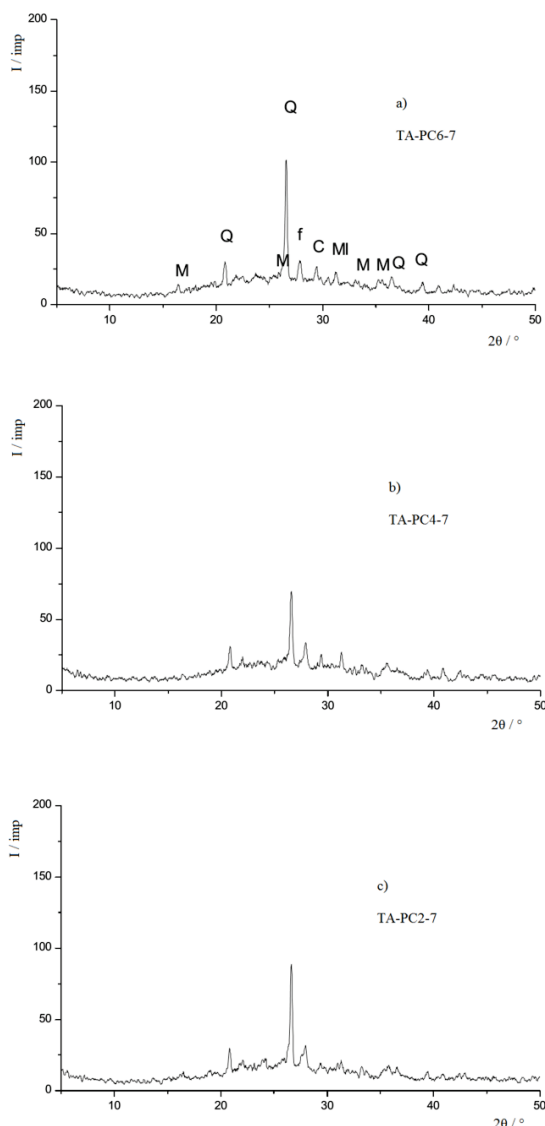
Namely, in the area of small angles, a series of insufficiently defined peaks of very low intensity, which indicate the beginning of crystallization of new phases, according to the data could match the compounds from the group of hydrated Ca - aluminates or sulfoaluminates. At  $29,5^\circ$  a new peak was noted corresponding to the maximum of the strongest mineral calcite. Although the amount of added lime is very small, and the strongest peaks of the dominant portlandite that largely obscured broad diffuse peaks corresponding to a significant share of the amorphous material, it can be confidently said that in the samples this mineral cannot be found and that there is a chemical reaction and transformation in the second phase, and most of the calcite (Fig. 2a, 2b, 2c).



**Figure 2** The X-ray diagram of the sample, which is exposed for 7 days in a humid environment with the addition of lime binders: a) 6 %, b) 4 %, c) 2 %

**3.2.3 The samples were exposed for 7 days in a humidified environment with the addition of cement, 2, 4 and 6 % (TA-PC2-7, TA-PC4-7 and TA-PC6-7)**

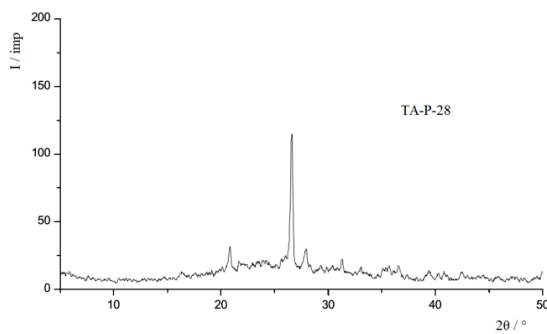
As samples of the hydrated lime, the cement samples also showed a small difference relative to the starting mineral ashes. The biggest differences are in the occurrence of calcite in small quantities and under defined hydration products type Ca - aluminate and sulfoaluminata. In samples with 4 and 6 % of the cement, traces of gypsum occurred (Fig. 3a, 3b, 3c).



**Figure 3** The X-ray diagram of the sample exposed to 7 days in a humid environment with the addition of cement binder: a) 6%, b) 4%, c) 2%

**3.2.4 The sample exposed for 28 days in a humid environment without the addition of binders (TA-P-28)**

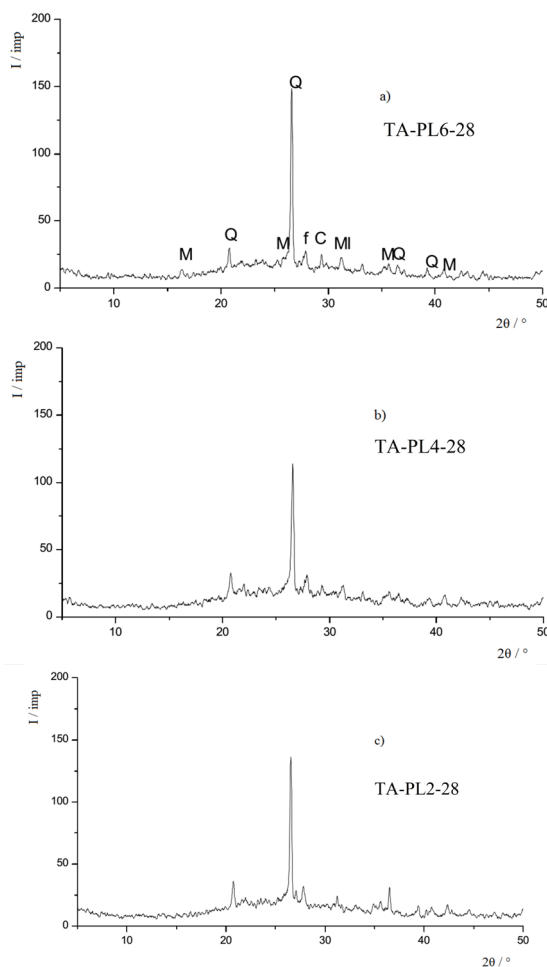
A sample of the mixture of fly ash and water after 28 days of exposure was very similar to the sample after 7 days exposure. Apart from the starting mineral ash in the sample occurs newly created trace of calcite (Fig. 4).



**Figure 4** The X-ray diagram of the sample, which is exposed for 28 days in a humid environment without the addition of binders

**3.2.5 Samples were exposed for 28 days in a humid environment with the addition of lime, 2, 4 and 6 % (TA-PL2-28, TA-PL4-28, and TA-PL6-28)**

Diffraction patterns with hydrated lime after 28 days of exposure do not show significant differences compared to the previous one. As a product, calcite is ascertained in small quantities. The intensity of the reference peak of this mineral clearly increases with increasing participation lime in the mixture. Hydrated Ca phases are still not sufficiently defined (Fig. 5a, 5b, 5c).



**Figure 5** The X-ray diagram of the sample, which is exposed for 28 days in a humid environment with the addition of lime binders: a) 6 %, b) 4 %, c) 2 %

### 3.2.6 Samples were exposed for 28 days in the humid environment of cement with the addition of 2, 4 and 6 % (TA-PC2-28, TA-PC4-28 and TA-PC6-28)

In addition to clearly defined calcite whose intensity slightly increases with the share of cement in the mixture, a second phase was not observed. It can be said that there are the beginnings of the formation of new products due to the presence of weak and poorly defined peaks at low angles (Fig. 6a, 6b, 6c).

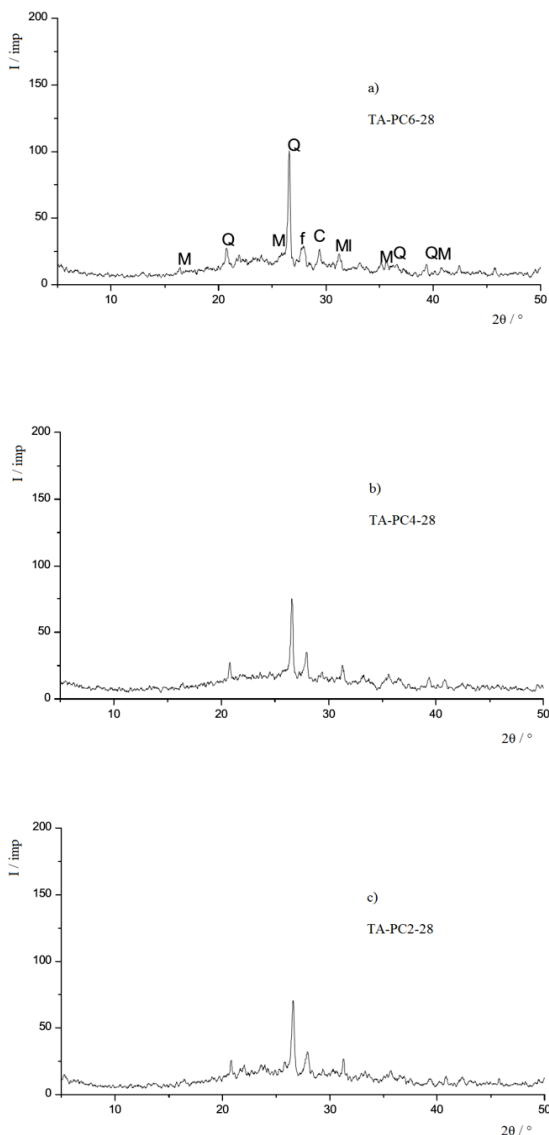


Figure 6 The X-ray diagram of the sample, exposed for 28 days in a humid environment with the addition of cement binder: a) 6 %, b) 4 %, c) 2 %

### 3.3 Mineral composition of fly ash

In all samples tested calcite was only mineral in crystalline form, which has been identified as the product of the reaction system in addition to the ash-water. Its amount is relatively small (below 10 %) and varies depending on several factors: the nature of the fly ash, the type of additive (hydrated lime or cement), the percentage of the additive and partly of the time of exposure. Fig. 7 presents the trend of calcite formed in the samples of mixtures based on intensity (in the registered pulses in the

diffractogram) of the reference peak (at 29,5° 2θ) for this mineral. From Fig. 7 it can be noted that the participation of mainly calcite increases with increasing participation of extras.

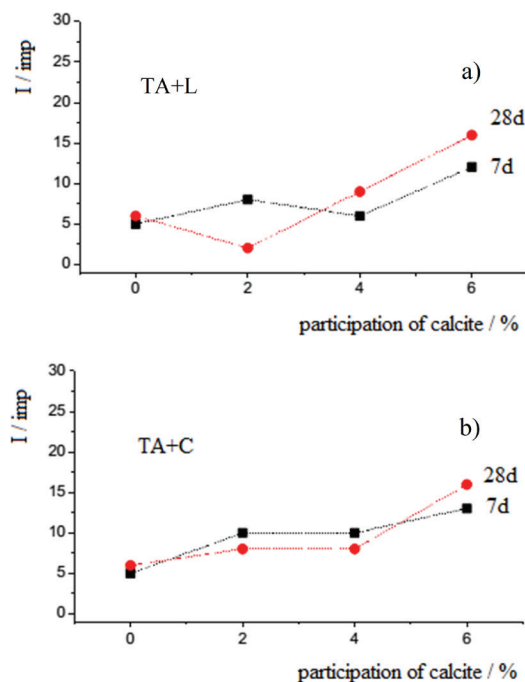


Figure 7 Shares of calcite formed in the examined samples of mixtures based on intensity: a) lime (L), b) Cement (C)

### 3.4 Pozzolanic properties of fly ash

Based on the sum of oxides SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, and according to the ASTM classification, tested ashes belong to class F artificial-industrial pozzolanic. Having in mind that the tests are not carried out on samples of the ash that was previously fragmented, but the patterns of the original particle size distribution, the results are favorable. Compressive strength and tensile strength were tested for samples in S1 and S2, taken from the thermal PPNT A and the results are shown in Tab. 9.

Table 9 The test results of compressive strength and tensile strength

Tested characteristics	Month of sampling	Test results		
		Single	Average	Average for S1 and S2
Tensile strength / kPa	S1	2090	1950	1670
		1730		
		2020		
	S2	1120	1390	
		1610		
		1450		
Compressive strength / kPa	S1	7350	6990	5570
		6740		
		7350		
		7050		
		6130		
		7350		
	S2	3980	4140	
		3980		
		4290		
		4290		
		3980		
		4290		

According to this standard fly ash has pozzolanic properties and is in Class 5 of pozzolanic materials. The tests were performed on samples of the original particle size distribution, which were not pre-shredded.

### 3.5 Mechanical properties of fly ash

#### 3.5.1 Compressive strength – uniaxial strength

After 7 days in a wet environment the samples were taken from PPNT A, and tested on uniaxial compressive strength, and were in a strength range from 398 to 1333 kPa. Test results are shown in Tab. 10, and it can be seen that the cement (C) is better binder than the lime (L). The maximum value of the uniaxial compressive strength is obtained for the sample with the cement addition of 4 %, and while the binder amount increases the compressive strength decreases. The maximum value of compressive strength is obtained by adding the lime binder of 6 %.

**Table 10** The uniaxial compressive strength after 7 days in the wet chamber

Sample No.	Compressive strength $R_c$ / kPa	Sample No.	Compressive strength $R_c$ / kPa
0 %	398,3	0 %	398,3
0 %	423,5	0 %	423,5
0 %	402,0	0 %	402,0
L 2 %	790,7	C 2 %	1139,6
L 2 %	783,1	C 2 %	1159,0
L 2 %	787,5	C 2 %	1147,2
L 4 %	753,7	C 4 %	1308,5
L 4 %	864,9	C 4 %	1333,4
L 4 %	817,2	C 4 %	1324,4
L 6 %	735,8	C 6 %	1250,6
L 6 %	950,4	C 6 %	1202,8
L 6 %	832,6	C 6 %	1227,1

The value ranges of the uniaxial compressive strength after 28 days in wet environment are from 518 to 2390 kPa. The maximum strength is obtained by adding 6 % of the lime as the binder. The test results are shown in Tab. 11.

**Table 11** The uniaxial compressive strength after 28 days in the wet chamber

Sample No.	Compressive strength $R_c$ / kPa	Sample No.	Compressive strength $R_c$ / kPa
0 %	518,8	0 %	518,8
0 %	538,4	0 %	538,4
0 %	527,2	0 %	527,2
L 2 %	1147,8	C 2 %	1139,6
L 2 %	1039,3	C 2 %	1159,0
L 2 %	1087,4	C 2 %	1147,2
L 4 %	1890,1	C 4 %	1308,5
L 4 %	1726,8	C 4 %	1333,4
L 4 %	1772,8	C 4 %	1324,4
L 6 %	2312,0	C 6 %	1250,6
L 6 %	2390,8	C 6 %	1202,8
L 6 %	2387,4	C 6 %	1227,1

The value ranges of the uniaxial compressive strength after 112 days in wet environment are from 1352 to 4196 kPa. The maximum strength is obtained by adding 6 % of the lime as the binder. The test results are shown in Tab. 12.

**Table 12** The uniaxial compressive strength after 112 days in the wet chamber

Sample No.	Compressive strength $R_c$ / kPa	Sample No.	Compressive strength $R_c$ / kPa
L 2 %	1489,5	C 2 %	1663,8
L 2 %	1352,7	C 2 %	1542,2
L 2 %	1406,0	C 2 %	1523,4
L 4 %	2668,9	C 4 %	1901,0
L 4 %	2723,7	C 4 %	1610,6
L 4 %	2689,2	C 4 %	1748,7
L 6 %	4196,1	C 6 %	1831,2
L 6 %	4094,7	C 6 %	1883,6
L 6 %	4008,0	C 6 %	1872,5

#### 3.5.2 Indirect tensile strength – Brazilian experiment

The value ranges of the indirect tensile strength after 28 days in wet environment are from 33 to 422 kPa. The maximum tensile strength is obtained by adding 6 % of the lime as the binder. The presented results that contain the lime as binder have significant deviations in the contents of the same quantity of binder added in the percentage in regard to the cement. The test results are shown in Tab. 13.

**Table 13** The indirect tensile strength after 28 days in wet chamber

Sample No.	Indirect tensile strength $R_{it}$ / kPa	Sample index	Indirect tensile strength $R_{it}$ / kPa
0	36,83	0	36,83
0	45,08	0	45,08
L 2 %	33,60	C 2 %	74,10
L 2 %	127,4	C 2 %	77,51
L 4 %	317,22	C 4 %	114,03
L 4 %	255,53	C 4 %	106,95
L 6 %	422,47	C 6 %	209,52
L 6 %	286,90	C 6 %	161,05

#### 3.5.3 The modulus of elasticity

After 7 days of the sample storage in the wet chamber, the modulus of elasticity was tested and was in range from 58 000 to 289 000 kPa. The maximum modulus of elasticity is obtained by adding 4 % of the cement as the binder. With the greater amount of cement content the modulus of elasticity decreases and with the lime added as the binder the test results are obtained with significant variations in the content of the same binder quantity. The test results are shown in Tab. 14.

**Table 14** The modulus of elasticity after 7 days in the wet chamber

Sample No.	Modulus of elasticity in compression $E_c$ / kPa	Sample No.	Modulus of elasticity in compression $E_c$ / kPa
0 %	78 000	0 %	78 000
0 %	82 000	0 %	82 000
0 %	58 000	0 %	58 000
L 2 %	187 000	C 2 %	214 000
L 2 %	127 000	C 2 %	225 000
L 2 %	165 000	C 2 %	231 000
L 4 %	192 000	C 4 %	282 000
L 4 %	164 000	C 4 %	289 000
L 4 %	173 000	C 4 %	285 000
L 6 %	134 000	C 6 %	279 000
L 6 %	201 000	C 6 %	253 000
L 6 %	143 000	C 6 %	264 000



The values of the modulus of elasticity after 28 days in the wet environment are from 147 000 to 458 000 kPa. The maximum strength is obtained by adding 6 % of the lime as binder. The test results are shown in Tab. 15. The presented sample results that contain the lime as the binder have the significant deviation in the content of the same binder amount in percentages, and in the addition of cement as the binder the deviation results are very small, even negligible.

**Table 15** The modulus of elasticity after 28 days in the wet chamber

Sample No.	Modulus of elasticity in compression $E_c$ / kPa	Sample No.	Modulus of elasticity in compression $E_c$ / kPa
0 %	158 000	0 %	158 000
0 %	147 000	0 %	147 000
0 %	162 000	0 %	162 000
L 2 %	257 300	C 2 %	214 000
L 2 %	235 700	C 2 %	225 000
L 2 %	196 800	C 2 %	231 000
L 4 %	365 000	C 4 %	282 000
L 4 %	304 000	C 4 %	289 000
L 4 %	266 000	C 4 %	285 000
L 6 %	389 000	C 6 %	279 000
L 6 %	433 000	C 6 %	253 000
L 6 %	458 000	C 6 %	264 000

The values of the modulus of elasticity after 112 days in the wet environment are from 87 000 to 411 000 kPa. The maximum strength is obtained by adding 6 % of the lime as the binder. The test results are shown in Tab. 16. The presented sample results that contain the lime as the binder have the deviation in the content of the same binder amount in percentages, and in the addition of cement as the binder the deviation results are greater than those of the lime.

**Table 16** The modulus of elasticity after 112 days in the wet chamber

Sample No.	Modulus of elasticity in compression $E_c$ / kPa	Sample No.	Modulus of elasticity in compression $E_c$ / kPa
L 2 %	297 000	C 2 %	332 000
L 2 %	217 000	C 2 %	325 000
L 2 %	238 000	C 2 %	760 000
L 4 %	402 000	C 4 %	347 000
L 4 %	371 000	C 4 %	329 000
L 4 %	377 000	C 4 %	481 000
L 6 %	440 000	C 6 %	374 000
L 6 %	469 000	C 6 %	312 000
L 6 %	455 000	C 6 %	338 000

### 3.5.4 Dependencies between the mechanical properties

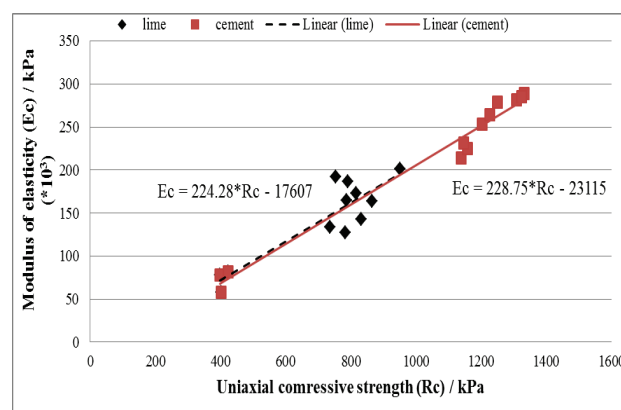
After the mechanical properties test completion according to EN standards, the dependencies were made between:

- the uniaxial compressive strength and the modulus of elasticity after 7, 28 and 112 days,
- the uniaxial compressive strength and the indirect tensile strength after 28 days,
- the modulus of elasticity and the indirect tensile strength after 28 days.

The testing was made with the samples that contain the cement and the lime as the binder (of 2, 4 and 6 %

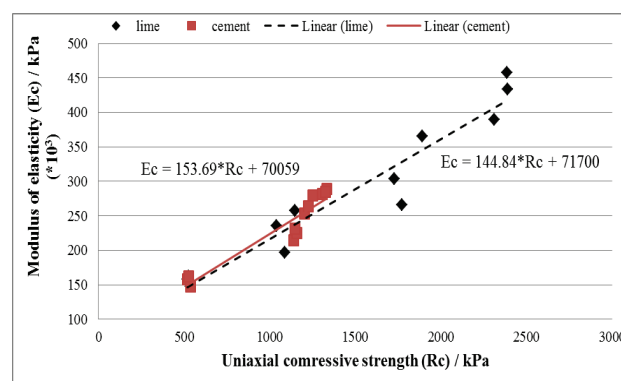
and with the control sample (100 % of the ash). The linear dependence was made for all variants, and after that the correlation coefficient was calculated. The correlation coefficient is characterized and classified in the specific correlation group.

After 7 days in the wet chamber, the maximum uniaxial strength and the modulus of elasticity are obtained in the samples that contain the cement as the binder, while the samples with the lime have lower values (Fig. 8). The correlation coefficient with the cement content and the control sample is calculated and is 0,990, and with the lime content and the control sample is 0,899. After the calculated coefficient values it can be seen that the samples with the cement belong to the very strong correlation group, and the samples that contain the lime belong to the strong correlation group.



**Figure 8** The dependence of the modulus of elasticity and the uniaxial compressive strength after 7 days in the wet chamber

After 28 days in the wet chamber, the maximum uniaxial strength and the modulus of elasticity are obtained in the samples that contain the lime as the binder (Fig. 9). The correlation coefficient with the lime content and the control sample is calculated and is 0,966, and with the cement content and the control sample is 0,954. After the calculated coefficient values it is found that the samples with the lime and the cement belong to the very strong correlation group and differ by 1 %.



**Figure 9** The dependence of the modulus of elasticity and the uniaxial compressive strength after 28 days in the wet chamber

After 112 days in the wet chamber, the maximum uniaxial strength and the modulus of elasticity are obtained in the samples that contain the lime as the binder (Fig. 10). The correlation coefficient with the lime content is calculated and is 0,951, and with the cement content is -



0,461. After the calculated coefficient values it is obtained that the samples with the lime belong to the very strong correlation group, and with the cement belong to the very low correlation group, and the coefficients differ by 50 %.

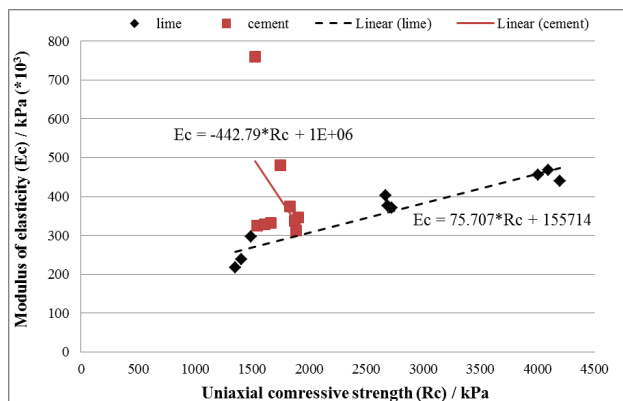


Figure 10 The dependence of the modulus of elasticity and the uniaxial compressive strength after 112 days in the wet chamber

After 28 days in the wet chamber, the maximum uniaxial strength and the indirect tensile strength are obtained in the samples that contain the lime as the binder (Fig. 11). The correlation coefficient with the lime content and the control sample is calculated and is 0,917, and with the cement content and the control sample is 0,686. After the calculated coefficient values it is obtained that the samples with the lime belong to the very strong correlation group, and with the cement belong to the lower correlation group, and the coefficients differ by 24 %.

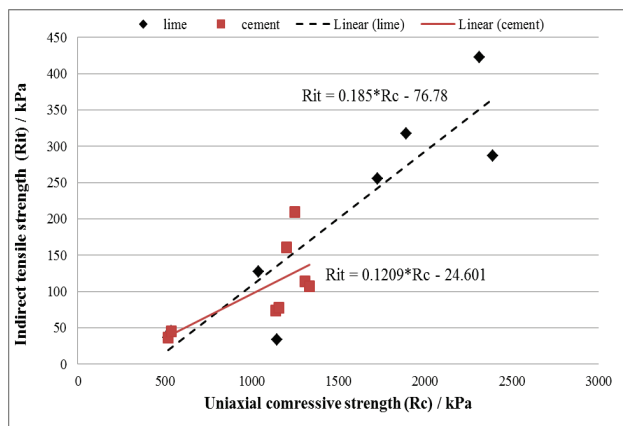


Figure 11 The dependence of the indirect tensile strength and the uniaxial compressive strength after 28 days

After 28 days in the wet chamber, the maximum of the modulus of elasticity and the indirect tensile strength is obtained in the samples that contain the lime as the binder (Fig. 12). The correlation coefficient with the lime content and the control sample is calculated and is 0,876, and with the cement content and the control sample is 0,771. After the calculated coefficient values it is obtained that the samples with the lime and the cement belong to the strong correlation group and differ by 11 %.

Based on the above presented results of the dependencies between mechanical properties, it was found that the cement binder is suitable for the first seven days because the large uniaxial compressive strengths and the modulus of elasticity were obtained, and after 28 days

it is obtained that the lime binder is suitable refer to the cement. The correlation coefficient shows that the relationship between the ash and the lime as the binder is much better than with the cement as the binder. The difference of the correlation coefficient is in the range from 1 to 50 %, whereby the minimum difference is achieved in the dependence between the modulus of elasticity and the uniaxial compressive strength after 28 days, and the maximum difference between the modulus of elasticity and the uniaxial compressive strength is achieved after 112 days.

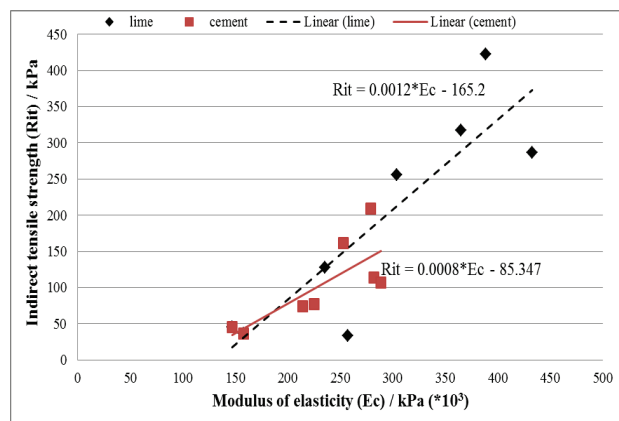


Figure 12 The dependence of the indirect tensile strength and the modulus of elasticity after 28 days

#### 4 Conclusions

Based on the study results, we have come to the following conclusions:

- all samples of ash that were for 7 days exposed in a humid environment contain a substantial amount of amorphous glassy-matter, but also crystal phase of feldspat, melilit, mullite, very little anhydrite and quartz is present. In the specimens that were subjected for 28 days besides the already mentioned minerals a trace of calcite occurs in the sample;
- in all tested samples calcite mineral is the only mineral in crystal form which has been identified as the product of the reaction system in the ash-binder-water, and the specimens without binders do not contain calcite or it is present in trace,
- a change in the chemical composition of the tailings is produced by the presence of inorganic composition that occurs in coal. Samples S1 and S2 contain a smaller amount of silicon dioxide ( $\text{SiO}_2$ ), a greater amount of aluminum dioxide ( $\text{Al}_2\text{O}_3$ ), calcium oxide ( $\text{CaO}$ ) and magnesium oxide ( $\text{MgO}$ ), while the content of iron oxide ( $\text{Fe}_2\text{O}_3$ ) is similar to samples that were examined in the period of 1983-2004,
- according to the international classification system, fly ash belongs to the first group of aluminosilicate ash. According to the size of the module R, it belongs to silicate ash,
- fly ash and slag have pozzolanic properties and belong by the test standard SRPS B.C1.018 2001 in Class 5 of pozzolanic materials, but according to standard ASTM classification the investigated ashes belong to artificial-industrial pozzolan of class F.

- the mechanical properties of the ash are much better when the lime is used as the binder refer to the cement.

All of the tested mechanical properties are significantly higher when the lime is used as the binder refer to the cement. The cement is suitable as the binder at the beginning of the first 7 days, while the lime as the binder is better after 28 days and 112 days.

Based on the results above, the general conclusion is that the fly ash from PPNT A can be used to create elements for road structures. It must be taken into account that the embedding is done in segments, isolated from the influence of surface water and groundwater, because with this, in terms of road operation, it prevents the leaching of harmful chemicals from layers of ash and slag, and their input in the bearing environment [21].

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