UNIVERSITY OF NIS
FACULTY OF CIVIL ENGINEERING AND ARCHITECTURE

in cooperation with
UNIVERSITY OF NOVI SAD
FACULTY OF TECHNICAL SCIENCES
DEPARTMENT OF CIVIL ENGINEERING AND GEODESY

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GRAĐEVINSKO-ARHITEKTONSKI FAKULTET

u saradnji sa
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PhIDAC 2019 PhIDAC

V INTERNATIONAL SYMPOSIUM
FOR STUDENTS OF DOCTORAL STUDIES IN THE FIELDS OF CIVIL ENGINEERING, ARCHITECTURE AND ENVIRONMENTAL PROTECTION

V MEĐUNARODNI SIMPOZIJUM
STUDENATA DOKTORSKIH STUDIJA IZ OBLASTI GRAĐEVINARSTVA, ARHITEKTURE I ZAŠTITE ŽIVOTNE SREDINE

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Prof. Zoran Grdic
Assoc. Prof. Gordana Toplicic-Curcic
Assis. Prof. Nenad Ristic
Assis. Prof. Vuk Milosevic

TECHNICAL EDITOR:

Assis. Prof. Nenad Ristic

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EDITORAL NOTE:

The Faculty of Civil engineering and Architecture of University of Nis organizes the Fifth International Symposium of Doctoral Studies' Students in the fields of Civil Engineering, Architecture and Environmental Protection "PhIDAC 2019".

The first Symposium of the Doctoral Studies' Students "PhIDAC 2009", held in September 2009 in Nis, confirmed the expectations of Prof. Slavisa Trajkovic and Professor emeritus Radomir Folic, the founders of this symposium, that the two-day meetings of the students of Doctoral studies and their professors would be of invaluable use both for young researchers and their tutors. Namely, a great number of published and presented papers, as well as open discussion on the quality of paper, directions in further researches and relationships between doctoral students and tutors demonstrated that the Symposium fulfilled the expectations of the participants and that the organization of new meetings should be continued.

At the Second Symposium "PhIDAC 2010" held in Novi Sad, the symposium programme was expanded, i.e. the field of environmental protection was also introduced as the third thematic field with the expectation that this multidisciplinary area should be more closely introduced to young researches in the fields of civil engineering and architecture.

The organizers of the Third Symposium "PhIDAC 2011", also held in Novi Sad, decided that the symposium should be international and thus they opened new possibilities for affirmation and development of young researches from Serbia, as well as of their colleagues from the Balkans.

There were 66 papers dealing with topics in the fields of civil engineering, architecture and environment protection that were submitted for the fourth international symposium of students of doctoral studies "PhIDAC 2012". The papers covered a wide range of scientific topics. All the papers were reviewed. On the basis of the reviews, it was concluded that the young researchers provided a significant contribution to the development of scientific thinking.

Members of the international scientific committee actively participated in the preparation of the symposium and reviewing of the papers. For this symposium too, the proceedings including papers in English and Serbian were included, which provides better and more productive communication and exchange of experience with the colleagues from abroad.

We would like to thank all the authors and co-authors of the papers and their mentors, and it is our wish that the young researchers would continue their successful careers and persist in realization of the goals they have set.

Nis, October 2019 Editors

THE CONTENT:

SESSION CIVIL ENGINEERING AND ENVIRONMENTAL PROTECTION / OBLAST GRAĐEVINARSTVO I ZAŠTITA ŽIVOTNE SREDINE

RADOVI NA SRPSKOM JEZIKU / PAPERS IN SERBIAN LANGUAGE

OBLAST ARHITEKTURA / SESSION ARCHITECTURE

OBLAST GRAĐEVINARSTVO I ZAŠTITA ŽIVOTNE SREDINE / SESSION CIVIL ENGINEERING AND ENVIRONMENTAL PROTECTION

PhIDAC 2019

V INTERNATIONAL SYMPOSIUM FOR STUDENTS OF DOCTORAL STUDIES IN THE FIELDS OF CIVIL ENGINEERING, ARCHITECTURE AND ENVIRONMENTAL PROTECTION

Jelena Bijeljić[1]
Nenad Ristić[2]
Gordana Topličić – Ćurčić[3]
Zoran Grdić[4]
Dušan Grdić[5]
Dejan Krstić[6]

FREEZE – THAW RESISTANCE OF GEOPOLYMER MORTAR BASED ON INDUSTRIAL BY-PRODUCTS

Abstract: *This paper investigated characteristics of geopolymer mortar mixes based on fly ash with admixture of aluminosilicate material – red mud. Four geopolymer mortar mixes were made with fly ash and red mud and one cement mixture. Mixes based on fly ash were made with different share of red mud - 5 % to 20 % of mass.*

The goal of this research was to determine the strengths of geopolymer mortar at the age of 3, 7, 28, 56 and 90 days, and to determine behavior of mortar mixes of such composition after exposure to 25 cycles of the freeze-thaw test. The mixes were cured under the same ambient conditions and had the same water/binder and binder/aggregate ratios.

Key words: *geopolymer mortar, fly ash, red mud, freeze-thaw cycles, mechnical properties*

1. INTRODUCTION

In the recent years, environment-friendly building materials have been in the focus. Geopolymers are composite materials whose characteristics are similar to the traditional cement mixes [1]. Geopolymers are alkali-activated aluminosilicate materials with a much smaller CO_2 footprint than traditional Portland cements, and display good mechanical and durability properties. Geopolymers are mostly based on the waste byproducts, most frequently of industrial production [2].

Until now, a large number of author research potential use of different byproducts for making of geopolymer mortars, whereby various physical, mechanical and other characteristics were tested. The research included potential use of red mud as a partial substitution for fly ash, blast furnace slag, metakaolin etc. and its effect on physical and mechanical strengths of geopolymer mortar. Jian He et al investigated two geopolymer mixtures synthesized from two different raw materials: metakaolin and the mixture of red mud (RM) and fly ash (FA). They researched microstructure, curing duration, and mechanical properties of the resulting geopolymer [3]. M. Zang et al. investigated geopolymer mixtures when synthesized from RM and three FA sources to physical and chemical properties, nominal Si/Al and

[1] Teaching assistant, College of applied technical science Niš, Aleksandra Medvedeva 20, neimar.jelena@gmail.com:

[2] Assistant professor, University of Niš, Faculty of Civil Engineering and Arhitecture Niš, Aleksandra Medvedeva 14, Niš, nenad.ristic@gaf.ni.ac.rs,

[3] Associate professor, University of Niš, Faculty of Civil Engineering and Arhitecture Niš, Aleksandra Medvedeva 14, Niš, Serbia, gordana.toplicic.curcic@gaf.ni.ac.rs

[4] Full professor, University of Niš, Faculty of Civil Engineering and Arhitecture Niš, Aleksandra Medvedeva 14, Niš, : zoran.grdic@gaf.ni.ac.rs

[5] MScCE, Faculty of Civil Engineering and Architecture, University of Niš, Aleksandra Medvedeva 14, Niš, Serbia, dusan.grdic@gaf.ni.ac.rs

[6] Grad. Civ. Eng., Faculty of Civil Enginnering and Architecture, University of Niš, Aleksandra Medvedeva 14, Niš, Serbia, dejan.krstic@gaf.ni.ac.rs

Na/Al molar ratios of raw materials and curing time on the mechanical properties of resulting geopolymers [4]. K. Kaya et al. investigated structural and mechanical properties of RM – metakaolin (MK) based geopolymers with varying RM contents 0 to 40 % RM in MK based geopolymer mixtures. Authors investigated XRD, Fourier Transform Infrared (FTIR) Spectroscopy, Scanning Electron Microscopy (SEM) and Compressive Strength measurements to understand structure-performance relationships in this system [5]. Z. Pan et al. investigated strength development and properties such as resistance against carbonation, simulated seawater, diluted acid, sulfate solution and freeze and thaw cycles of geopolymer mixtures made of 70 % of blast – furnace slag (BFS) and 30 % of RM [6].

This paper investigated potential use of RM as a binder for making geopolymer mortars. In the examined geopolymer mortar made on the basis of FA a partial replacement of primary binder using RM was performed, where the percentage of RM in the mass was 5 %, 10 %, 15 % and 20 %. Mechanical characteristics of mortar were examined as well as the effects of freeze-thaw cycles on characteristics of these mixes.

2. EXPERIMENTAL DETAILS

2.1. Materials

In this paper, the binders used were fly ash (FA) and red mud (RM). FA generated as a byproduct in the thermal electric power plant Kostolac "B", while RM is an aluminum processing generated byproduct originating from the "Kombinat aluminijuma" Podgorica, Montenegro. Firstly, FA and RM are dried up to the constant mass, and then they are sifted through the laboratory sieve having opening 0.09 mm. The used binders are presented in Figure 1. The aggregate used for making mortar is the river aggregate from the South Morava river, having maximum grain size of 2 mm. The admixture used to improve workability of fresh mortar mix was superplasticizer (SP) SIKA 5380. In those mixes where addition of SP was required, it was added up to a satisfactory flow, as well as extra water which was the same in all mixes.

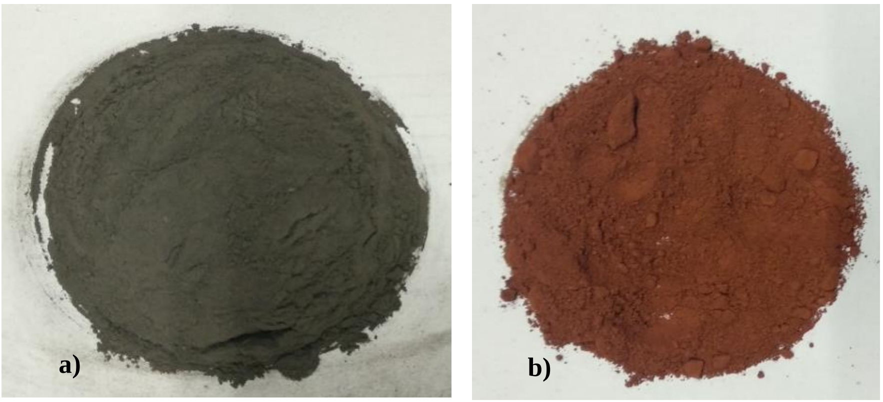


Figure 1 – Used binder materials: a) FA, b) RM

Chemical composition of used FA and RM binders is given in Table 1 and it was obtained using the chemical analysis and ASTM C618 standard. The used alkali activator is Sodium hydroxide – (SH) in the granular form (Oltchim, Romania, p.a. 98,5%), and Sodium silicate – (SS) in the liquid form (Galenika-Magmasil d.o.o. Serbia, with the SiO2:Na2O=1:2 contents ratio. The concentration of SH solution used in this research was 10 molarity (M) and it was obtained by dissolving the granules in water.

Table 1- Chemical composition (wt %) of the raw binder materials

Chemical compounds	SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO	SO_3	P_2O_5	TiO_2	Na_2O	K_2O
FA	51,7	11,6	20,2	7,4	2,4	1,02	0,12	1,04	0,8	1,04
RM	32,8	4,63	9,92	2,37	0,22	-	-	-	0,12	0,12

2.2. Preparation of geopolymer mortar

Four geopolymer mortar mixes and one standard cement mortar mix “E“ were tested for this paper. Geopolymer mortar mixes were made with FA and RM whereby FA was replaced with RM in the amount of 5 %, 10 %, 15 % and 20 % of the mass of the total quantity of binder. Those mixes are labeled as „5 RM“, „10 RM“, „15 RM“ and „20 RM“, respectively. Making of mortar mixes was performed in the laboratory mortar mixer where the mass ratio of binders and sand was 1:3. 24 hours prior to the start of mixing, SH had been dissolved, and it was mixed with SS 30 min prior to the start of mixing. After mixing the alkali activators, binder material was added and the paste was mixed for 5 minutes. After that, dry sand was added and the mixing continued for 5 minutes. The composition of mortar mixes is provided in Table 2.

Table 2- Mix design of geopolymer mortar mixtures

Mixture no.	CEM III (g)	FA (g)	RM (g)	SS/SH (g)	W/B (g)	B/S (g)	SP (%)	Extra water (g)
5 RM	-	427,5	22,5	0,19	0,45	1:3	0,3	20
10 RM	-	405	45	0,19	0,45	1:3	-	20
15 RM	-	382,5	67,5	0,19	0,45	1:3	-	20
20 RM	-	360	90	0,19	0,45	1:3	-	20
E	450	-	-	-	0,5	1:3	0,5	225

Fly ash (FA), Red mud (RM), Sodium silicate (SS), Sodium hydroxide (SH), Binder (B), Sand (S), Superplasticiser (SP)

After mixing, fresh mortar was placed in metal moulds having dimensions 4 x 4 x 16 cm. The moulds were then wrapped in a plastic foil, and after 2 days the specimens were demoulded. Until the time of testing, the specimens were kept at the laboratory temperature of around 22 ºC. The appearance of fresh mortar mixtures is presented in Figure 2.

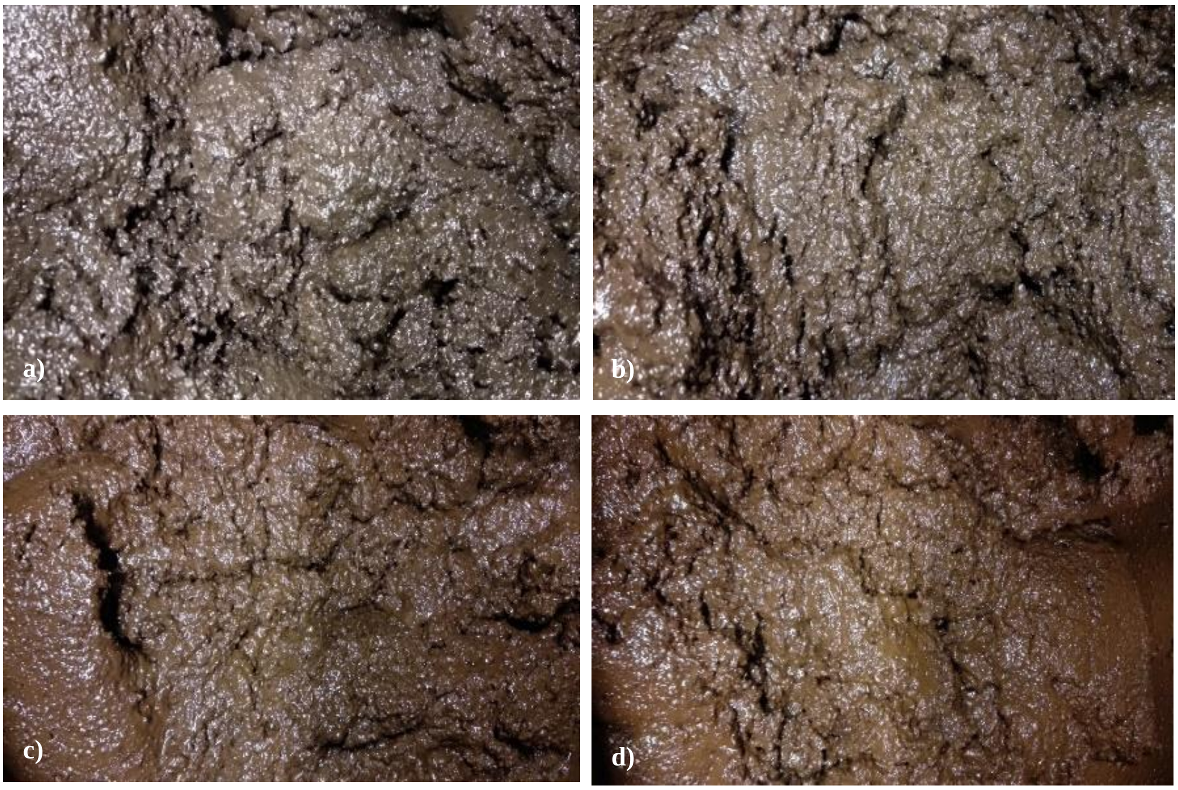


Figure 2 – Fresh geopolymer mortar mixtures: a) “5 RM”, b) “10 RM”, c) “15 RM” and d) “20 RM”

2.3. Strength and freeze – thaw tests

Mechanical characteristics of geopolymer mortar mixes were tested in hardened state. The hardened specimens in the form of prisms, 40 x 40 x 160 mm at the age of 3, 7, 28, 56 and 90 days were tested to compressive and flexural strength according to the SRPS EN 196-1:2018 [7]. standard. A total of fifteen prisms was made for each mortar mix. For each age, three mortar specimen prisms were tested.

The specimens belonging to batches having the same composition were tested by freeze – thaw cycle tests. The tests were conducted on the mortar prisms having dimensions 4 x 4 x 16 cm whereby the variation of compressive and flexural strength was observed. For each batch, three mortar prisms were tested. The samples of geopolymer mixes were cured in ambient conditions to the age of 28 at the temperature of 22 °C, while the cement mix was cured in water saturated condition. Upon reaching the age of 28 days, the samples were subjected to the freeze – thaw test. One cycle consists of alternating freezing and thawing in the water, whereby in each cycle, the samples spent 4 hours frozen at the temperature of -20 °C, after which they spent 4 hours submerged in the standard temperature water. The test was conducted according to the EN 14617-5:2005 standard [8] in 25 cycles. The specimens of mortar mixtures after making are presented in Figure 3. After the conducted freeze-thaw test, the compressive and flexural strength of the specimens was determined. Also was performed the calculation of KMf 25 and KMc 25 (coefficients of freeze/thaw resistance in flexural and compressive strength).

Where it KMf 25 is calculated according to formula:

$$KMf = \frac{RMf}{Rf} \tag{1}$$

$$KMc = \frac{RMc}{Rc} \tag{2}$$

Where:

RMf - is flexural strength average value of specimens after 25 freeze/thaw cycles,

Rf - is flexural strength average value of dried - unfrosted specimens,

RMc - is compressive strength average value of specimens after 25 freeze/thaw cycles,

Rc - is compressive strength average value of dried - unfrosted specimens.

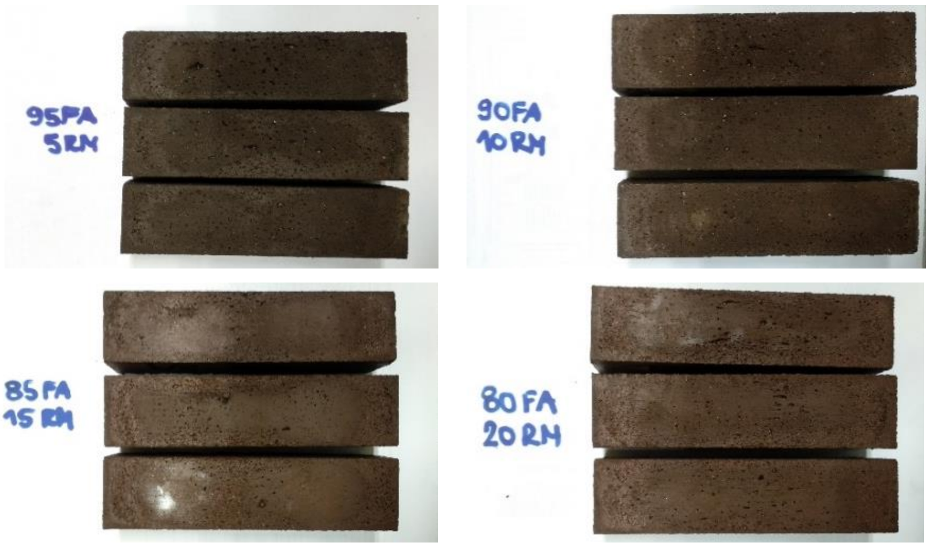
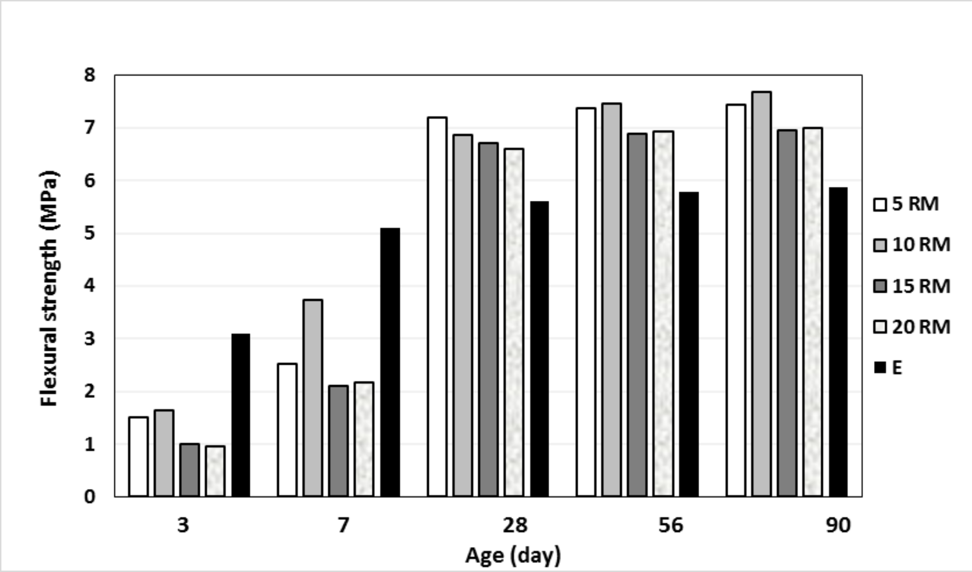


Figure 3 – Specimens of hardened geopolymer mortar mixtures after demoilding

3. EXPERIMENTAL RESULTS AND DISCUSSION

Hardened mortar prisms were tested according to the procedure prescribed by the standard [7]. The flexural strength value was obtained by measuring three specimens at each age, while the value of compressive strength was measured on six specimens. The results of flexural and compressive strength of the tested specimens are presented in Figures 4 and 5.



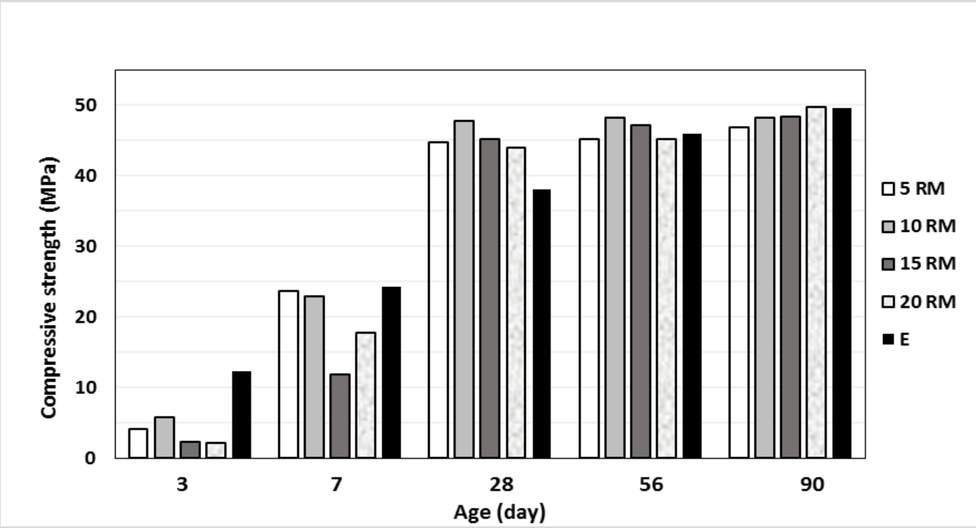


Figure 4 and 5 – Flexural and compressive strength of geopolymer ("5 RM", "10 RM", "15 RM" and "20 RM") and cement ("E") mortar mixtures

The results of testing of compressive and flexural strength of the specimens at the ages of 3, 7, 28, 56 and 90 days are shown in Figures 4 and 5. It can be concluded that the more mature the samples the stronger the mortar. At the age of 3 days, the sample "10 RM" has the highest flexural and compressive strength, and the form of the diagram on both diagram segments presented in Figure 4 and 5 is the same.

The measured flexural strength of the "10 RM" mix specimens, at the age of 7 and 28 days was 3,73 MPa and 6,87 MPa, respectively. At the age of specimens of 28 days, the highest value was measured for the "5 RM" mix samples, which was 7,2 MPa, which is for around 4,5% more than the "10 RM" mixture. At the older ages of specimens, these two mixes have the approximately identical values. When testing the flexural strength of the "15 RM" and "20 RM" mixes, at the age of samples of 28 days, the values 6,7 MPa and 6,6 MPa were measured (respectively) and until the end of testing, the increase of flexural strength of around 5 % was measured.

According to the compressive strength testing results, presented in Figure 5, the highest value of compressive strength at the tested age of specimens of 3 days is demonstrated by the reference mix "E" prisms, which is about 2,1 more than the "10 RM" mixture where the highest compressive strength was measured. At the age of specimens of 28 days, the "10 RM" mix specimens had the highest value of compressive strength and it was 47,82 MPa which is for around 25 % more than the reference mix „E" made with CEM III cement. The shape of the compressive strengths diagram at the age of specimens of 28 and 56 is the same, with a slight increase of strength up to 2 MPa, which was measured for the “15 RM“ mix. At the age of samples of 28, 56 and 90 days, the increase of compressive strength is evident, but they, like the flexural strengths, are slight, with a maximum increase up to 12 %.

It can be concluded that all geopolymer mortar mixes meet the basic requirements in terms of compressive and flexural strengths.

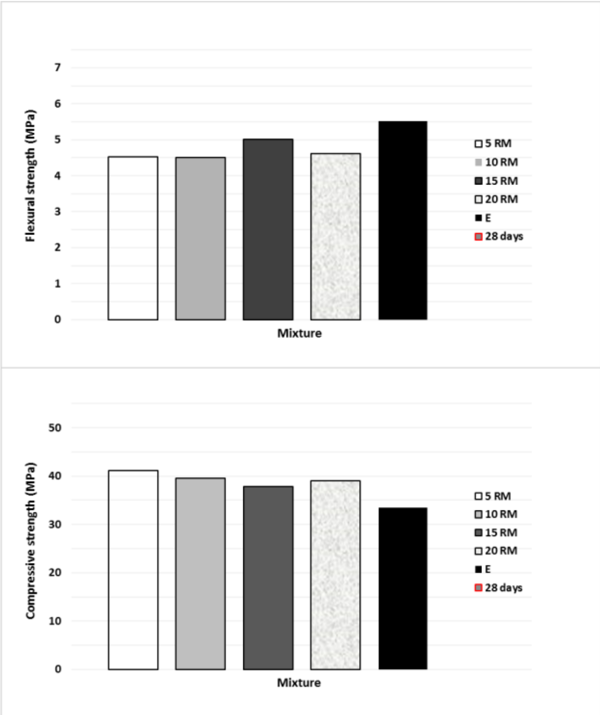


Figure 6 and 7 – Flexural and compressive *strength of geopolymer („5 RM“, „10 RM“, „15 RM“ and „20 RM“) and cement („E“) mortar mixtures after 25 freeze-thaw cycles*

The results of testing flexural and compressive strengths after 25 freeze/thaw cycles are presented in Figures 6 and 7.

When measuring flexural strength, and calculating the flexural strength resistance coefficient, in all the specimens of geopolymer mixes with the RM admixture, a decline of flexural strength was observed. The value of this coefficient was determined for the following mixes: „5 RM“, „10 RM“, „15 RM“, „20 RM“ and „E“ and it is 63 %, 66 %, 75 %, 70 % and 98 %, respectively. The frost resistance parameter for mixtures „15 RM“ and „E“ is higher than 75 % so it can be concluded, that those specimens are frost-resistant for 25 cycles.

The compressive strength resistance coefficient was also calculated for the following mixes: „5 RM“, „10 RM“, „15 RM“, „20 RM“ and „E“ and it is 92 %,83 %, 84 %, 89 % and 88 %, respectively. The compressive strength resistance parameter is higher than 75 % for all tested specimens. It can be concluded that the tested specimens of geopolymer mortar mad on the FA basis, with admixture of RM are frost resistant for 25 cycles. Specimens after freeze – thaw test are presented in Figure 8.

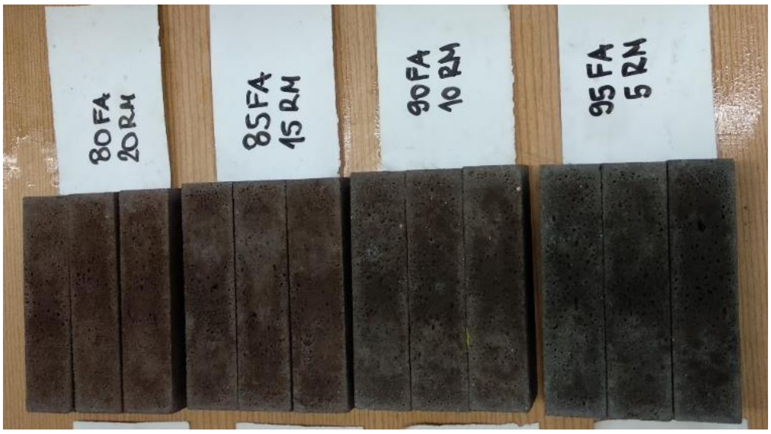


Figure 8 – Specimens of geopolymer mortar after 25 freeze-thaw cycles

4. CONCLUSIONS

According to the obtained characteristics, it can be concluded that the geopolymer mortars based on FA originating from TEP „Kostolac B“ with the admixture of RM can be an adequate substitutions for traditional cement mixes. Also, the following conclusions can be drawn:

- At the age of samples of 28 days, all the tested samples had the compressive strength exceeding 44 MPa, i.e. exceeding 46,5 MPa at the age of 90 days.
- All geopolymer mortar mixes meet the basic requirements regarding compressive and flexural strengths.
- Testing the frost resistance for 25 cycles leads to conclusion that the flexural strength resistance parameter is higher than 75 % only in the „15 RM“ and „E“ mixes.
- By calculating the compressive strength resistance parameter it was concluded that all the specimens of geopolymer mortar made on the basis of fly ash with the red mud admixture are frost resistant for 25 cycles.
- Environmental and economic advantages of using geopolymer materials are reflected in use of waste material, whose CO2 emission, when transforming it from a byproduct into a binder, is low.
- It is necessary to conduct more detailed tests of physical and mechanical characteristics on mortars, as well as on concretes.

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