

## PROPERTIES OF SELF-COMPACTING CONCRETE PRODUCED WITH WASTE MATERIALS AS MINERAL ADMIXTURE

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*Rapid technological and industrial development in the recent decades caused considerable environmental problems, and one of the most significant is, undoubtedly, disposal and recycling of waste materials and by-products of industrial production. Since concrete is a composite material, waste materials can suitably be used in its composition. In this paper, the research of effects of milled recycled glass from cathode tubes, flotation tailings from a copper mine, red mud and fly ash as mineral admixtures on properties of fresh and hardened self compacting concrete was presented. The test results indicated that the addition of such materials does not cause a decline in physico-mechanical characteristics and properties of durability of self-compacting concretes (SCC), and they even improve some aspects of concrete performance in comparison with SCC made with limestone filler as mineral admixture. Waste materials such as fly ash and recycled glass of cathode tubes (CRT) exhibit a pozzolanic activity, so the performances of the concretes with these admixtures proved to be better after ageing than the concretes with other admixtures.*

**Keywords:** self compacting concrete, recycled CRT, flotation tailings, red mud, fly ash, limestone filler, fresh and hardened properties, durability

### 1. Introduction

The basic principle of sustainable construction is usage of building materials which will not have negative effects on the environment, as well as proper management of waste materials generated during construction or demolishing of structures. Increasing attention is paid to the rapid technological and industrial development in the recent decades which caused big environmental problems, and one of the most significant is, undoubtedly, disposal and recycling of waste materials and by-products of industrial production. On the other hand, concrete, being a composite and frequently used building material is fitting for usage of waste materials as components in its composition. Waste materials in concrete can be used as partial substitution of cement, partial substitution of aggregate or as reinforcement of concrete composite. Exactly integration of those materials into concrete itself can, to a considerable extent, contribute to solving the problem of their disposal. However, in order to achieve this goal, it is important to establish how these materials affect the concrete properties, and what quantities of them can be added without compromising strength and durability of concrete which make it such a suitable building material.

Self-compacting concrete (SCC) is a special kind of concrete which does not require vibration when placed and compacted and which is capable of filling the entire formwork under its own weight,

even when the reinforcement steel is densely installed. Regardless of the high fluidity, such concrete is simultaneously very resistant to segregation and bleeding. SCC has many advantages over conventional concrete, including: eliminating the need for vibration; decreasing the construction time and labor cost; reducing the noise pollution; improving the filling capacity of highly congested structural members; improving the interfacial transitional zone between cement paste and aggregate or reinforcement; decreasing the permeability and improving the durability of concrete; facilitating constructability and ensuring good structural performance [1-3].

The composition of self-compacting concrete can be designed in multiple ways, but one must take care to achieve certain adequate rheological properties of fresh concrete, such as fluidity, viscosity, resistance to segregation [1, 4-6]. Also, designed SCC should meet the requirements for strength, volume stability and durability of the hardened concrete at the same time [7]. In comparison with the conventional concrete, SCC usually has a lower share of coarse aggregate, lower water/cement ratio, increased share of paste and therefore increased share of superplasticizer, and if needs be, an admixture for modification of viscosity. Fluidity and viscosity of concrete mixture are achieved with careful selection of cement and mineral admixtures, and by limiting the ratio of water and fine particles and by adding of superplasticizer and optionally of viscosity

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modifying admixture. It is recommended the use of the largest aggregate grains, 12-20 mm. Very often, inert and pozzolanic, i.e. hydraulic mineral admixture powders, such as limestone filler, fly ash, micro-silica, ground granulated blast furnace slag, rice husk ash, ground dune sand etc [8-12].

Fly ash is very frequently used as admixture when making self-compacting concrete. The research established that addition of fly ash reduces slump flow and slump time T500 [13,14], porosity [15] as well as shrinking and creeping [16]. The spherical shape of ash particles increases mobility and workability of concrete. Experimental investigation of the SCC performance when different types of fibers used, showed that 30% replacement of cement with fly ash improved flowability of micro-reinforced mixes [17]. Due to a slower formation of the pozzolanic reaction products, the early strengths of SCC with fly ash are lower in comparison with the usual values of SCCs without mineral admixtures. However, in time the strength increases and after 90 days, it is similar to the corresponding reference concrete. Because of the reduced porosity, concretes with the admixture of fly ash achieve, in general, higher final strengths [18,19], but this impact considerably depends on the amount of added ash. Excessive quantity of fly ash in concrete can lead to reduction of its strength [20,21]. The research established that fly ash can be used to produce SCC with high performance in terms of durability [16].

In the copper production process, large quantities of waste material are generated whose disposal represents a large environmental issue. Flotation tailings, as one of the by-products of copper production, are rich in iron oxides and silicates, and thus, they are suitable for production of concrete and mortar. Depending on their chemical composition, they can be used either as an admixture to ordinary Portland cement, or as a replacement of fine aggregate particles. This method may solve this large metallurgical environmental issue and bring a great financial benefit, while simultaneously leading to reduction of gases emission and energy consumption related to production of the same quantity of materials whereby natural resources remain preserved [22]. Onuaguluchi and Eren in their researches [23] demonstrated that concretes with admixture of flotation tailings possess improved mechanical characteristics in comparison to vibrated concrete. Compressive, tensile shear and flexural strengths of SCCs with the mineral admixture of flotation tailings are increased. Also, an increased resistance to abrasion and lower chloride penetration depth were observed. Such improved characteristics are more prominent in cases when 5% of flotation tailings are used and when water/cement ratio is high (water absorption is increased, when flotation tailings are used). The same researchers also tested chemical action on.

concretes containing flotation tailings, and on their basis they determined that the increase of presence of flotation tailings in concrete increases acid action resistance, but simultaneously reduces resistance to destructive sulphate expansion [24]. Sharma and Khan [25] researched the impact of fine aggregate replacement with copper slag on durability properties of self-compacting concrete and found that 60% of replacement is an optimum percentage for achieving better concrete performance.

In the Bayer alumina production process, red mud created as a waste is composed mostly of hematite, goethite, quartz, boehmite, calcite, tricalcium aluminate, zinc and magnesium oxide, sodium hydroxide etc. What makes red mud a dangerous polluter of land, ground and surface waters is alkaline liquid phase which filters down from disposal sites into ground waters carrying with it a still high content of sodium [26]. A large number of conducted studies relate to the various aspects of implementation of red mud as a component material of mortars and concretes: as partial cement and fine aggregate replacement in mortars, integral part of geopolymers etc. On the basis of the tests performed on SCC, it was established that red mud admixture increases viscosity, reduces fluidity and considerably reduces segregation and bleeding of concrete, i.e. water separation. On the other hand, porosity of SCC increases, but shrinking reduces [27]. Density of hardened concrete with admixture of red mud also reduces. Compressive strength after 90 days yields higher values in relation to the reference SCC. Flexural and tensile splitting strengths are considerably higher in comparison with the reference self-compacting concrete made without red mud [27,28]. Tang et al. [29] researched the impact of partial replacement of fly ash with red mud on properties of fresh and hardened self-compacting concrete. They established that red mud had a slightly negative impact on the characteristics of fresh concrete, but did not negatively impacted the mechanical strength of hardened concrete. The increase of the red mud share increased the compressive strength and elasticity modulus, while tensile splitting strength slightly declined. Microscopic analysis showed that red mud contributed to improvement of interfacial transition zone.

Limestone filler is most frequently used as a mineral admixture for making SCC. For that reason, there are numerous studies about its effects on SCC properties. The studies [30-31] show that the limestone filler in SCC improves workability with reduced cement content, increases segregation resistance and modifies matrix porosity and pores distribution. In terms of rheology, it causes a decrease of both yield stress and plastic viscosity [32]. Elyamany et al. [33] indicate that chemical and mineralogical properties

of limestone filler to a great extent influence the behavior of SCC in fresh state. Fluidity of SCC made with milled limestone increases with the fineness of the admixture particles [34]. If limestone filler is finer than cement, then it can contribute to increase of strengths. Limestone filler increases the density of the paste, which is particularly important in improving the compressive strength [35]. In comparison to the SCC with fly ash, concrete with milled limestone has a higher water permeability and lower frost resistance [36]. Sideris et al. [37] in their research established that mechanical strength and durability of self compacting concretes produced with limestone filler are lower in comparison to the concrete produced with ladle furnace slag. In the research of the influence of limestone filler on salt frost scaling of SCC, Persson [38] concluded that the amount of filler had no effect on salt frost scaling, while less salt frost scaling was observed in SCC with limestone powder having higher fineness.

There were numerous tests regarding the application of recycled glass as a partial replacement of fine aggregate for making SCC [39-41]. The test results showed that by the increase of recycled glass content in concrete, fluidity and air content increase, but mechanical strengths and static elasticity modulus are reduced. When SCC with limestone filler was tested, it was established that compressive strength and ultrasound pulse velocity (UPV) increase with the increase of recycled cathode ray tube glass content [42]. In the research of the SCC in which a partial replacement of a fine aggregate with recycled glass was performed, UPV value and dynamic elastic modulus increased, while depth of water penetration under pressure decreased with

increase of recycled glass aggregate content [43].

The main target of this research is to investigate the potential for application of waste materials such as milled recycled glass from cathode tubes, flotation tailings from a copper mine, red mud and fly ash as mineral admixtures on properties of fresh and hardened self compacting concrete.

## 2. Experimental research

### 2.1. Materials used in the experiment

The Portland cement CEM I 42,5 R was used for making of concrete mixtures, which complies with all the quality requirements prescribed by SRPS EN 197-1 [44] standard. Physico-mechanical and chemical properties of the cement are given in Tables 1 and 2.

Limestone filler is a by-product in the procedure of crushing stone for concrete aggregate, fly ash is from the Kostolac B coal-fired power plant, flotation tailings are from the Mining and Smelting Combine Bor, red mud is from the Aluminum Plant Podgorica created in the Bayer process of aluminum production. Recycled cathode ray tube glass (RCRTG) was taken from local recycling center and milled in the laboratory mill. All these admixtures are finer than 0,125 mm, because they were passed through an adequate sieve. Chemical composition of used cement and mineral powder admixtures are given in Table 2, while in Figure 1 their SEM and photograph are displayed. Particle size distribution and specific gravity of used mineral powder admixtures are given in Table 3.

Physico-mechanical properties of cement

Standard consistence	28.0%
Setting time	initial 185 min; final 240 min
Soundness – Le Chatelier	1.0 mm
Fineness– sieve residue 0.09 mm	1.3 %
Specific gravity	3.15 g/cm <sup>3</sup>
Loose material bulk density	925 kg/m <sup>3</sup>
Compacted material bulk density	1520 kg/m <sup>3</sup>
Flexural strength	2 days – 6.8 MPa 28 days – 9.1 MPa
Compressive strength	2 days – 32.5 MPa 28 days – 57.3 MPa

Table 1

Chemical composition of used cement and mineral powder admixtures

Parameter		Cement	Limestone filler	RCRTG	Fly ash	Flotation tailings	Red mud
SiO <sub>2</sub>	%	19,30	0.53	60,61	51,68	40.80	32,8
Fe <sub>2</sub> O <sub>3</sub>	%	2,87	0.12	0,58	11,58	41.72	4,63
Al <sub>2</sub> O <sub>3</sub>	%	4,28	0.27	2,88	20,16	5.90	9,92
CaO	%	62,80	53.10	1,31	7,43	7.36	2,37
MgO	%	2,20	0.95	0,53	2,41	0.86	0,22
SO <sub>3</sub>	%	3,05	0.09	0,09	1,02	-	-
P <sub>2</sub> O <sub>5</sub>	%	0,06	0.12	0,03	0,12	-	-
TiO <sub>2</sub>	%	-	0.05	0,30	1,04	-	-
Na <sub>2</sub> O	%	0,21	0.04	7,61	0,88	0.35	0,12
K <sub>2</sub> O	%	0,91	0.02	6,45	1,04	1.20	0,12
LOI	%	2.26	40.25	-	2,57	-	-

Table 2

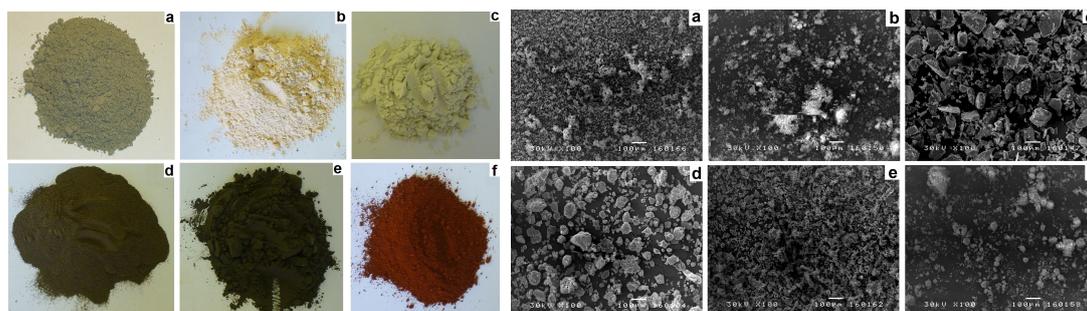


Fig. 1 - Photo (left) and SEM (right) display of used cement and mineral powder admixtures: a) cement, b) limestone filler, c) recycled cathode ray tube glass, d) fly ash, e) flotation tailings, f) red mud

Table 3

Sieve size (mm)	Passing (%)				
	Limestone filler	Recycled glass	Fly ash	Flotation tailings	Red mud
0.125	100.0	100.0	100.0	100.0	100.0
0.09	79.3	63.6	72.8	85.3	97.4
0.063	57.2	38.7	55.3	52.4	88.1
Specific gravity (g/cm <sup>3</sup> )	2.72	2.84	2.24	3.15	2.71

The previous tests according to SRPS B.C1.018 [45] standard established that only fly ash and pulverized recycled ray tube glass exhibit pozzolanic activity. Other powder admixtures can be considered inert.

Three fractions of river aggregate were used (0/4 mm, 4/8 mm and 8/16 mm), and they comply with all the quality requirements prescribed by SRPS EN 206-1 [46] and EN 12620 [47] standard. Particle size distributions of used aggregate are shown in Figure 2. Saturated surface dry specific gravity, water absorption capacity and loose bulk density of the aggregates are shown in Table 4.

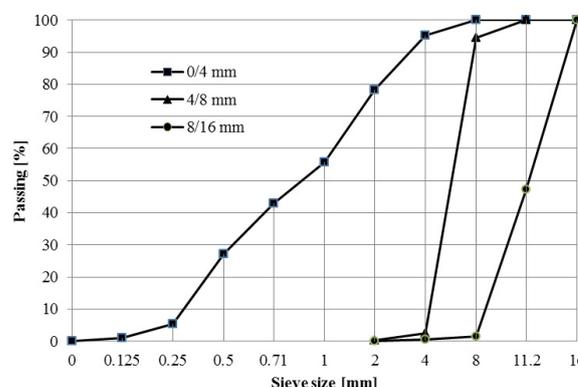


Fig. 2 - Particle size distribution of used aggregate

Table 4

Property	Fraction		
	0/4 mm	4/8 mm	8/16 mm
Saturated surface dry specific gravity (g/cm <sup>3</sup> )	2.62	2.65	2.65
Water absorption capacity (%)	1.26	0.89	0.87
Loose unit weight (kg/m <sup>3</sup> )	1500	1480	1460

The superplasticizer Sika Viscocrete 5380 was used as chemical admixture in the mixtures which specific gravity 1.10 g/cm<sup>3</sup> (based on modified polystyrene esters (modified polycarboxylates).

## 2.2 Concrete mixture composition

A total of five different mixtures of SCCs were made for the requirements of the experimental research, those being: mixture with the mineral admixture of limestone filler (mixture designated LF), mixture with the admixture of pulverized recycled glass of cathode ray tubes (RG), mixture with the admixture of fly ash (FA), mixture with the admixture of flotation tailings (FT)

and mixture with the admixture of red mud (RM). Concrete mixtures differ only in terms of the implemented powder admixtures type. The percentage share of component volume in 1 m<sup>3</sup> of concrete is the same for all the concrete mixtures. All the concrete mixtures were made so as to have a similar flow spread 660±10 mm when concrete flowability is tested. This condition is met by varying the superplasticizer quantity. Compositions of concrete mixtures for 1 m<sup>3</sup> of concrete are given in Table 5.

## 2.3 Types of tests investigated on the fresh and hardened concrete

The following tests were conducted on the fresh concrete: density according to SRPS EN 12350-6 [48] standard, air content in concrete according to SRPS EN 12350-7 [49], slump flow test and T500 spreading test according to SRPS EN 12350-8 [50], passing ability using L-box test according to SRPS EN 12350-10 [51] and the sieve segregation resistance test according to SRPS EN 12350-11 [52].

Table 5

Composition of 1m <sup>3</sup> of concrete mixtures used in the experiment					
	LF	RG	FA	FT	RM
Cement	0.127 m <sup>3</sup> or 400 kg				
Fine aggregate 0/4 mm	0.2962 m <sup>3</sup> or 776 kg				
Coarse aggregate	4/8 mm	0.1158 m <sup>3</sup> or 307 kg			
	8/16 mm	0.2011 m <sup>3</sup> or 533 kg			
Water	0.1815 m <sup>3</sup> or 181.5 kg				
Assumed air content	0.02 m <sup>3</sup>				
Limestone filler	0.055 m <sup>3</sup> or 150 kg	–	–	–	–
Recycled glass	–	0.055 m <sup>3</sup> or 156 kg	–	–	–
Fly ash	–	–	0.055 m <sup>3</sup> or 123 kg	–	–
Flotation tailings	–	–	–	0.055 m <sup>3</sup> or 173 kg	–
Red mud	–	–	–	–	0.055 m <sup>3</sup> or 149 kg
Superplasticizer	0.0045 m <sup>3</sup> or 4.95 kg	0.0040 m <sup>3</sup> or 4.40 kg	0.0050 m <sup>3</sup> or 5.50 kg	0.0043 m <sup>3</sup> or 4.68 kg	0.008 m <sup>3</sup> or 8.80 kg

The tested physical properties of the hardened concrete were the density of water saturated concrete according to SRPS EN 12390-7:2010 [53] standard, determination of total water absorption according to standard SRPS EN 1340:2012 Annex E [54] and ultra sound pulse velocity according to SRPS EN 12504-4:2008 [55] using the specimen cubes having sides of 15cm. The dynamic elasticity modulus of concrete was calculated using Eqs. (1) [56]:

$$E_{dh} = \rho \cdot c^2 \cdot \frac{(1+\nu) \cdot (1-2 \cdot \nu)}{1-\nu} \quad (1)$$

where,  $E_{dk}$  - dynamic elasticity modulus of concrete (MPa),  $\rho$  - hardened concrete density (kg/m<sup>3</sup>),  $c$  - ultra sonic pulse velocity (km/s) and  $\nu$  - Poisson's ratio.; Poisson's ratio was assumed as 0.2 for all concrete mixtures.

Also tested were mechanical properties of concrete, the most important being compressive strength. This characteristic was tested according to SRPS EN 12390-3 [57], on cube shaped specimens having sides of 15 cm at the age of 2, 7, 28 and 90 days. The flexural strength test was performed on the prism shaped specimens, having dimensions 10×10×40 cm at the age of 28 and 90 days according to SRPS EN 12390-5 [58]. The splitting tensile strength test (Brazilian test) was performed on cylindrical specimens having diameter Ø15 cm and length 30 cm at the age of 28 and 90 days according to SRPS EN 12390-6 [59]. „Pull-off“ strength test was performed on the cubes having sides 15 cm at the age of 28 and 90 days according to SRPS EN 1542:2010 [60]. Prior to this test, specimens were prepared by machining – drilling using a Ø50 mm diameter drill to the depth of 15 mm (prescribed drilling depth is 15±5 mm), and then, steel seals (50 mm in diameter and 20 mm in height) were glued on the tested points. Determination of secant modulus of elasticity in compression was performed on

cylindrical specimens having diameter Ø15 cm and length 30 cm at the age of 28 days according to SRPS EN 12390-13 [61].

Also tested were properties of durability of concrete. Depth of penetration of water under pressure in hardened concrete was performed according to SRPS EN 12390-8 [62] on the cubes having sides 15 cm. During the test, the specimens were exposed to water under pressure of 500 kPa for 72h. Determination of freeze/thaw resistance with de-icing salt of concrete was performed according to SRPS EN 1340 [54] Annex D on the cubes having sides 15 cm. A total of 28 freezing and thawing cycles were performed. Measuring of abrasion according to the Böhme test was performed according to SRPS EN 1340 [54] Annex H on 71 mm cube specimens. Drying shrinkage of concrete was performed on the prism shaped specimens, having dimensions 10×10×50 cm according to UNI 11307 [63].

### 3. Results and discussion

The primary goal of this research is testing potential for application of waste materials as mineral admixture for making of SCC. Since the limestone filler is most frequently used for making of SCC, for that reason, the concrete mixture LF can be considered a reference mixture used for comparison with other mixtures which contain admixtures of recycled cathode ray tube glass, fly ash, flotation tailings and red mud.

#### 3.1. Properties of fresh concrete

The test results of fresh concrete are provided in Table 6. The table provides mean values of the obtained test results.

Based on the test results of fresh concrete density given in Table 6, it can be concluded that it primarily depends on the specific gravity of the used mineral admixture, but also on the air content in concrete which is noticeable in the case of concrete mixture with recycled cathode ray tube

Table 6

Characteristic concrete in fresh state						
Properties	Unit	Test results				
		LF	RG	FA	FT	RM
Density	kg/m <sup>3</sup>	2375	2390	2340	2385	2365
Air content	%	2,2	1,2	2,9	2,8	2,6
Test T <sub>500</sub> time	s	3,5	4,5	7,0	6,0	6,5
Slump flow test	mm	660	670	650	670	650
L-box passing ratio (H <sub>2</sub> /H <sub>1</sub> )	(mm/mm)	0,94	0,95	0,91	0,92	0,87
Testing segregation using sieves	%	14,0	12,8	5,6	6,8	6,0

glass admixture (the mixture designated with RG). The highest density is demonstrated exactly by the mixture having the RG designation, which has 15 kg/m<sup>3</sup> more than the reference concrete with limestone filler (LF), and least density is demonstrated by the mixture with fly ash having FA designation, which has 35 kg/m<sup>3</sup> less than the reference concrete.

In terms of air content in fresh concrete, the mixture designated with RG had the lowest value, i.e. 1.2%, and the mixture designated with FA had the highest value (2.9%). Almost all concrete mixtures with the exception of RG have the approximately same air content and they have similar values to the reference LF (Table 6).

As it was already said, all the concrete mixtures were made to have approximately same flow spread (660±10 mm) on the event of testing concrete flowability (border value between slump-flow classes SF1 and SF2 according to The European Guidelines for Self-Compacting Concrete [5]), which was achieved by implementing superplasticizer. In Table 5 can be seen that the lowest quantity of superplasticizer was necessary for making of concrete mixture with recycled glass (RG), slightly more for the mixtures with flotation tailings (FT), limestone filler (LF) and fly ash (FA), and the highest quantity for the red mud (RM) mixture. Red mud is the finest material, and it has a higher water absorption capacity, allowing less water to be available for the slump-flow [27,29]. On the other hand, the pulverized particles of recycled glass have an unfavorable shape (Figure 1), but considerably lower water absorption, because the water (superplasticizer) demand is lower [40]. Fineness and spherical shape of fly ash and limestone filler provide for a better particles packing and in this way they improve the flowability of SSC mixtures.

T<sub>500</sub> test indicates the viscosity of concrete mixture, and represents the time in which the concrete achieves the spread of 500 mm when testing flowability. Based on the test results in the Table 6, it can be concluded that all concrete mixtures with the admixture of waste materials have higher values of T<sub>500</sub> test than the reference concrete. The FA mixture had the highest spread time for 500 mm, the mixtures RM and FT were similar, while the RG mixture had a similar value as the LF reference concrete. A higher viscosity is the

result of the reduced amount of free water in the mixture due to the higher porosity and water absorption property of mineral admixtures.

Filling ability was determined using L-box test, and other methods can be implemented as well: U – box, J – ring and Kajima box. On the basis of the test results in the Table 6, it can be concluded that RG mixture has the best filling ability while the RM mixture has the lowest ability in comparison to the other mixtures. FA and FT mixtures had mutually similar values, but lower than the reference, LF mixture. In general, all SCC mixtures have acceptable filling ability, because all the ratios of H<sub>2</sub>/H<sub>1</sub> are higher than 0.75 (according to The European Guidelines for Self-Compacting Concrete [5]).

Segregation resistance is expressed as the percentage of the amount of concrete which passed through the sieve with 5 mm openings in comparison with the total mass. Based on the results in the Table 6, it can be concluded that all the mixtures with waste materials have a higher resistance in respect to the reference concrete LF, whereby the best resistance was demonstrated by the FA mixture, followed by RM, FT and RG mixtures, respectively. SCC mixtures with fly ash, flotation tailings and red mud, in addition to high viscosity, exhibited a higher segregation resistance. The weaker bond between the glass particles and cement paste caused a lower segregation resistance. In general, all SCC mixtures have a satisfactory segregation resistance, because it is lower than 15% (according to The European Guidelines for Self-Compacting Concrete [5]).

## 3.2. Properties of hardened concrete

### 3.2.1. Physical properties

The density, total water absorption, ultrasonic velocity and dynamic modulus elasticity test results are shown in Table 7. Each value presented is the average of three measurements.

As for the properties of hardened concrete, densities of hardened concrete are coordinated with the density of fresh concrete. As well as in the case of fresh concrete, differences occur due to the various specific masses of mineral powder admixtures and air content in concrete.

Water absorption of concrete depends on permeable capillary porosity of the concrete

Table 7

Density, total water absorption, ultra sonic velocity and dynamic elastic modulus of concrete mixtures						
Properties	Unit	Test results				
		LF	RG	FA	FT	RM
Density	kg/m <sup>3</sup>	2370	2383	2335	2378	2356
Total water absorption	%	3.23	3.72	3.62	3.95	4.30
Ultra sonic velocity	km/s	4.53	4.69	4.73	4.61	4.56
Dynamic elastic modulus	GPa	43.77	47.18	47.02	45.48	44.09

mixture. Total water absorption of all mixtures ranged between 3.2 and 4.3% which indicates that there are no significant variations in water absorption capacity, i.e. the formed structure of capillary pores is not considerably different. The RM mixture had the highest water absorption which is logical because of the high absorption capacity of red mud, while the LF mixture had the lowest value.

The most important factors affecting ultra sonic velocity values of concrete are the concrete porosity, aggregate type and interfacial transition zone (ITZ) characteristics. It can be seen that the values of ultra sonic velocity were higher than 4.5 km/s which is a boundary for strong concrete. All concrete mixtures produced with waste materials had a higher ultrasonic pulse velocity in comparison to the LF reference. The FA mixture with fly ash had the highest value, which was for 4.3% higher than the reference LF, and the RM mixture had the lowest value among the mixtures with the waste material, and it was 0.7% higher than LF. The mixture with the recycled cathode ray tube glass admixture had a similar ultrasonic pulse velocity value as the mixture with the fly ash admixture, while the mixture with the flotation tailings admixture had 1.7% higher value than LF. SEM analysis showed the presence of cracks in ITZ of LF, FT and RM concrete mixtures which probably contributed to decline of ultrasonic pulse velocity.

The basic factors affecting dynamic modulus of elasticity of concrete are the properties of aggregate, cement paste and interfacial transition zone characteristics. The test results showed that the dynamic modulus of elasticity of concrete changes depending on the type of the used powder admixture. The lowest value of dynamic modulus of elasticity was recorded for the reference mixture LF, and slightly higher values were recorded for RM and FT mixtures, while the highest values were recorded for FA and RG mixtures. The defects in the ITZ structure caused a reduction of ultra sonic velocity value of LF, FT and RM concrete mixtures (which indirectly affected the dynamic modulus of elasticity).

### 3.2.2. Compressive strength

The compressive strength test results of the concrete mixtures are shown in Figure 3. Each value presented is the average of three measurements. Compressive strengths of

concretes, as one of the most important characteristics of concrete, are mutually similar at corresponding age of concrete. At the age of 2 days, the highest strength was exhibited by the FA mixture which is 12.6% higher than LF reference concrete, and the lowest by the FT mixture which is 7.6% lower than LF. The lower quantity of free water and the higher compactness of concrete can be the reasons for higher early compressive strengths of SCC with fly ash and red mud. At the age of concrete of 7 days, the highest strength was demonstrated by the FA mixture which is 3.9% higher than the LF reference, and the lowest was demonstrated by the RM mixture which is 8.4% lower than LF. At the age of 28 days, the highest compressive strength increase was demonstrated by the FT mixture. It simultaneously had the highest compressive strength, as well, which is 6.0% higher than the LF, while the lowest value was exhibited by the RM which was 4.1% lower than the LF. At the age of 90 days, the highest increase and highest value of compressive strength was exhibited by the RG mixture, while the FA mixture exhibited a slightly lower increase, followed by the LF and FT mixtures, while the lowest strength was exhibited by the RM mixture. Compressive strength of the RG mixture was 10.9% higher than LF, while compressive strength of the RM mixture is 12.4% lower than the LF. This was expected, since among all the mineral admixtures, only fly ash and RCRTG demonstrated pozzolanic activity. The obstruction of cement hydration because the grains are enveloped in very fine particles of red mud is a possible reason for the reduced increase of compressive strength of SCC with red mud during aging.

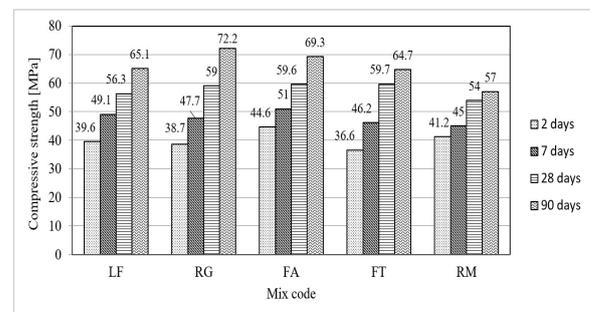


Fig. 3 - Comparison of compressive strength results at the age of 2, 7, 28 and 90 days.

### 3.2.3. Flexural strength

The flexural strength test results of the concrete mixtures are shown in Figure 4. Each value presented is the average of three measurements. The highest value of flexural strength at the age of 28 days was demonstrated by the RM mixture which contained red mud admixture, which was 15.9% higher than the LF reference value, and the lowest value was demonstrated by the FT mixture which contained flotation tailings, which was 15.9% lower than LF. The mixture containing recycled glass from cathode ray tubes had a flexural strength similar to the LF mixture, while the mixture containing fly ash admixture had 11.1% lower value than the LF reference. At the age of 90 days, the highest increase of flexural strength was exhibited by the RM mixture, whose value is 20.0% higher than the LF mixture. The FT mixture had the lowest value of flexural strength, which was 10.0% lower than the LF, while the RG and FA mixtures had 10.0% and 11.4% higher flexural strength, respectively, than the LF mixture. The values of flexural strength of concrete are largely influenced by the quality of bonds between the cement paste and aggregate grains, as well as the method of grain packing. The possible reason for the higher values of the flexural strength of SCC with red mud is the better adhesion between the grains due to the high fineness of red mud. The other reason can be the internal curing provided by the red mud due to its water retention capacity.

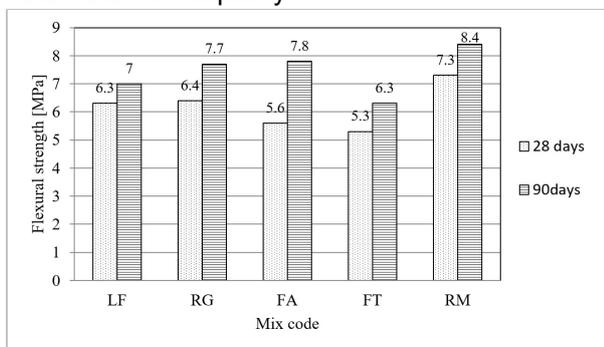


Fig. 4 - Comparison of flexural strength results at the age of 28 and 90 days.

### 3.2.4. Tensile splitting strength

The splitting tensile strength test results of the concrete mixtures are given in Figure 5. Each value presented is the average of three measurements. In terms of tensile splitting strength at the age of 28 days, all the concrete mixtures had the similar strength values. All the concrete mixtures made with waste materials, except the FA mixture with fly ash had the lower tensile split values than the LF reference. The highest value was exhibited by the FA mixture which is 9.1% higher than LF, and the lowest by the FT and RM mixtures, 9.1% lower than the LF. At the age of 90 days, the highest tensile splitting strength increase

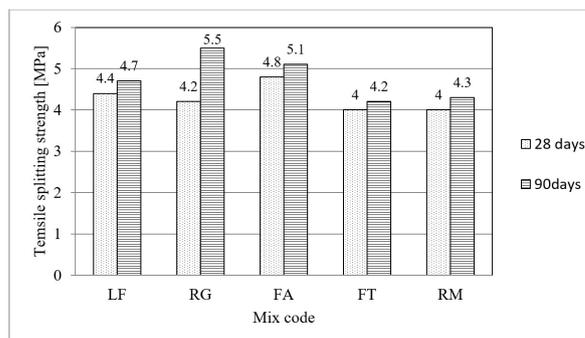


Fig. 5 - Comparison of tensile splitting strength results at the age of 28 and 90 days.

was exhibited by the RG mixture, whose value was 17.0% higher than the LF mixture. The lowest value of tensile splitting strength was exhibited by the FT mixture which was 10.6% lower than LF. The RM mixture had a similar value, while the FA mixture had 8.5% higher tensile splitting strength than the LF mixture. Better particle packing facilitated by the shape of the RCRTG grain and pozzolanic activity are the reasons for the highest value of tensile splitting strength of RG concrete mixture at the age of 90 days. Also, the pozzolanic activity of fly ash contributes to the increase of tensile splitting strength of FA concrete mixture.

### 3.2.5. Bond strength by Pull-off

The bond strength by Pull-off test results of the concrete mixtures is given in Figure 6.

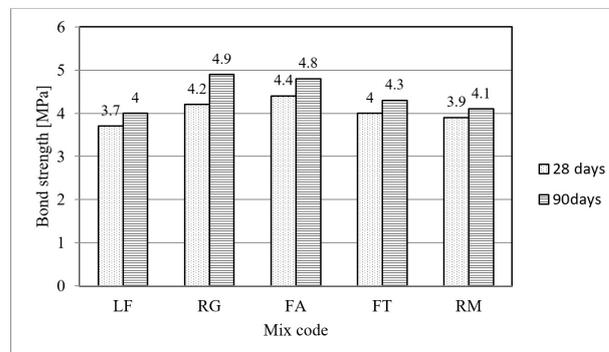


Fig. 6 - Comparison of bond strength by Pull-off test results at the age of 28 and 90 days.

Each value presented is the average of three measurements. In case of the bond strength Pull-off tests, all the concrete mixtures had the similar strength values. At the age of 28 and 90 days, all the concrete mixtures made with waste materials, had the higher bond strengths in comparison with the LF reference. At the age of 28 days, the highest value was exhibited by the FA mixture which was 18.9% higher than the LF reference, and the lowest value of all the waste material mixtures by the RM mixture which was 5.4% higher than the LF. At the age of 90 days, the highest value was exhibited by the RG which was 22.5% higher than the LF reference. The FA mixture had the similar value, while the lowest

Table 8

Depth of penetration of water under pressure in hardened concrete, freeze/thaw resistance with de-icing salt, abrasive resistance of concrete and drying shrinkage

Properties		Unit	Test results				
			LF	RG	FA	FT	RM
Depth of penetration of water under pressure		mm	2	4	3	18	12
Freeze/thaw resistance with de-icing salt (mass loss per unit)		kg/m <sup>2</sup>	0.03	0.06	0.02	0.03	0.54
Abrasive resistance (loss in volume)		mm <sup>3</sup> /5000mm <sup>2</sup>	8280	9190	8960	8830	9150
Drying shrinkage	Age	4 days	0.032	0.045	0.070	0.036	0.168
		7 days	0.146	0.129	0.158	0.125	0.314
		14 days	0.297	0.293	0.353	0.274	0.360
		28 days	0.375	0.385	0.447	0.356	0.393
		56 days	0.419	0.441	0.486	0.393	0.423
		90 days	0.456	0.496	0.512	0.440	0.472
		120 days	0.497	0.536	0.561	0.473	0.512
		180 days	0.542	0.571	0.605	0.517	0.553

value was shown by the RM mixture which was 2.5% higher than the LF. Pozzolanic activity RCRTG and fly ash are the main reasons for the higher values of bond strength of RG and FA concrete mixtures.

### 3.2.6. Secant modulus of elasticity

The most important factors affecting the value of secant modulus of elasticity of concrete are share, density and modulus of elasticity of basic components of concrete, porosity of coarse aggregate and cement paste, water/cement ratio, interfacial transition zone characteristics. The effect of the type of mineral admixtures on the secant modulus of elasticity of the SCC mixtures at 28 days is illustrated in Figure 7. Each value presented is the average of three measurements. The mixture RG had the highest secant modulus of elasticity value, 6.4% higher than the LF reference, while the concrete mixture with red mud RM had the lowest value, 8.3% lower than the LF reference. The mixtures with fly ash (FA) and flotation tailings (FT) had 2.6%, and 3.5% higher value of secant modulus of than the LF reference.

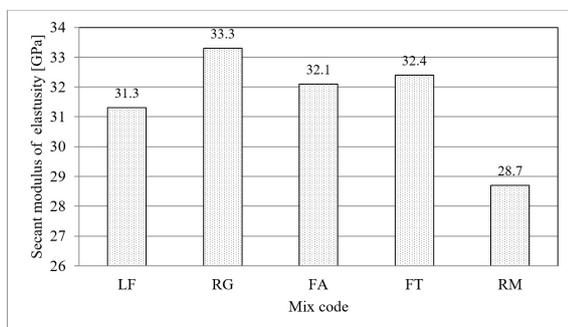


Fig. 7 - Comparison of secant modulus of elasticity at the age of 28 days.

### 3.2.7. Durability properties of concrete

The depth of penetration of water under pressure in hardened concrete, freeze/thaw

resistance with de-icing salt, abrasive resistance of concrete and drying shrinkage test results are shown in Table 8. Each value presented is the average of three measurements.

The test results of depth penetration of water under pressure showed that the highest penetration was recorded for the concrete made with flotation tailings admixture - FT (18 mm), followed by the red mud mixture RM (12 mm), while the concrete mixes LF, RG and FA had very low penetration values (2 – 4 mm). These results are in agreement with the other tests of SCC with fly ash [16], flotation tailings [25], limestone filler [32] and recycled glass [41]. In addition, and according to Neville [54], these mixes can be considered as waterproof as none of them has a penetration greater than 30 mm.

After 28 freezing-thaw cycles with de-icing salt, there was a considerable surface damage of the concrete made with the red mud admixture. Cement paste was entirely damaged, and coarse aggregate grains were visible (Figure 8). Other concrete types had a very small concrete surface damage in the form of scaling of fine mortar and they meet the quality requirements for the XF4 exposure class concrete according to SRPS EN 206-1 [40] and prefabricates such as concrete kerbs, concrete paving blocks, concrete paving flags (maximum permissible mass loss is 1 kg/m<sup>2</sup>).

The basic factors affecting the abrasive resistance of concrete are hardness and toughness of aggregate, strength and porosity of cement stone and interfacial transition zone characteristics. The loss of volume after 16 cycles of abrasion of all mixtures was in the range 8280 to 9190 mm<sup>3</sup>/5000 mm<sup>2</sup> which indicates a very good abrasive resistance. All SCC meet the quality requirements for the XM3 exposure class concretes according to SRPS EN 206-1 [44] and prefabricates such as concrete kerbs, concrete paving blocks, concrete paving flags (maximum permissible abrasion value is 18000 mm<sup>3</sup>/5000mm<sup>2</sup>).

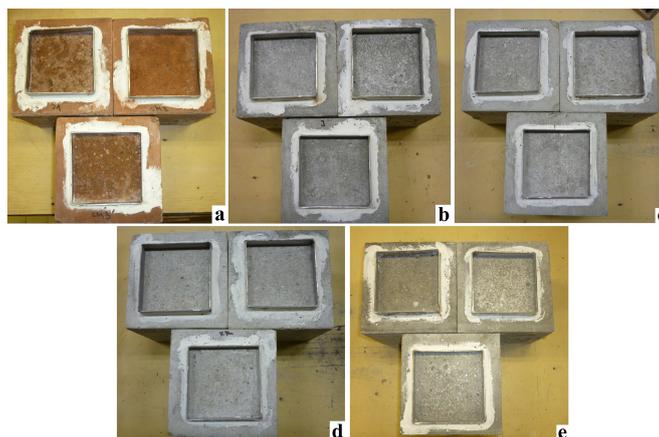


Fig. 8 - Appearance of the concrete surface after 28 freeze-thaw cycles with de-icing salt: a) RM, b) FT c) RG, d) LF and e) FA.

The SSC mixture with red mud (RM) had the highest drying shrinkage during the first seven days of exposure to air. The rapid shrinkage could be due to the loss of water in the coarse capillary pores which were saturated during water curing. However, with ageing, the increase of drying shrinkage decreases and the final shrinkage becomes the same as in the reference LF concrete. The possible reason for this might be attributed to the internal curing ability of the red mud. Red mud being a porous material absorbed a large amount of free water in the fresh state but the water gradually migrated from the red mud and was used for concrete curing particularly after the concrete had been dried for some time. The FA concrete mixture slightly higher initial drying shrinkage than the reference concrete. The other

SSC mixtures had a similar drying shrinkage at all ages of concrete. The FA concrete mixture had the highest drying shrinkage in the period from 28<sup>th</sup> to 180<sup>th</sup> day, and the RG concrete mixture had a slightly lower shrinkage. One of the possible explanations is that the pozzolanic reaction products, fly ash and RCRTG contributed to a more intensive shrinkage of concrete samples in the mentioned period. The shrinkage of the FT concrete mixture does not considerably differ from the reference concrete (FA).

### 3.2.8. SEM observations

SEM images were taken on the SCC mixtures to examine the microstructure characteristics in interfacial transition zone (ITZ) and hardened cement paste. A visual inspection of

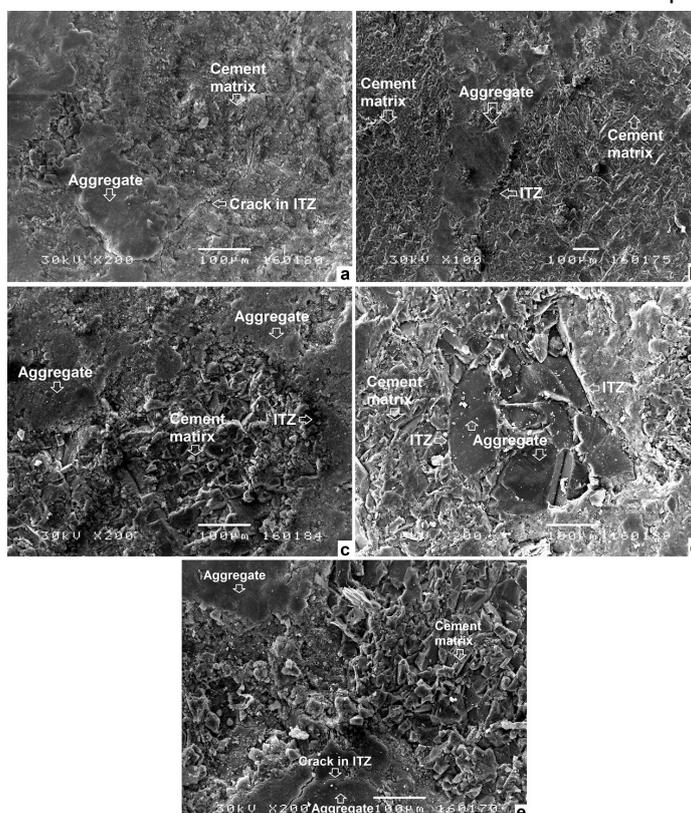


Fig. 9 - SEM images of SCCs: a) LF, b) RG c) FA, d) FT and e) RM.

ITZ and hardened cement paste was performed in order to detect potential non-uniformity and micro-cracks in the structure which could affect mechanical strengths, stiffness and permeability of concrete. In Figure 9 are displayed SEM images of the tested concrete mixtures. It may be observed that the cement matrix of all the mixture is fairly uniform, without any clearly visible cracks or large pores. The crystallization has been almost complete, with no visible particles that failed to react which indicates that mineral powder admixtures did not have any adverse effects on hydration. Interfacial transition zone of SCCs produced with fly ash and recycled glass is well structured, providing good mechanical strength and properties of durability of these concretes. The presence of cracks in ITZ of the concrete with limestone filler did not cause decline of performances, because they are sporadic and narrow. However, in the case of red mud mixtures, the cracks in ITZ directly caused increased water permeability of concrete and a reduced freeze/thaw resistance with de-icing salt. The SEM analysis of the concrete mixture with flotation tailings indicated that there are discontinuities in ITZ which contributed to the increase of water permeability of concrete, but did not have adverse effects on other performances.

#### 4. Conclusion

In the present work, a study of the effects of milled recycled glass from cathode tubes, flotation tailings from a copper mine, fly ash, red mud and limestone filler as mineral admixtures on properties of fresh and hardened self compacting concrete was carried out. On the basis of the experimental research results, the following conclusions can be drawn:

- It is possible to make SCCs with waste materials such as milled recycled glass from cathode tubes, flotation tailings from a copper mine, red mud and fly ash as mineral admixtures that comply with the criteria of self-compacting recommended in The European Guidelines for Self-Compacting Concrete [5].
- Density in fresh state of the examined SCCs primarily depended on the specific gravity of the used mineral admixtures, and on the content of entrained air in concrete. Porosity, absorption capacity, fineness and shape of the particles of used mineral powder admixtures are the basic parameters which affected flowability, filling ability and segregation resistance of SCC.
- There are no considerable differences in the water absorption ability, i.e. in the formed capillary pore structure, because the total water absorption of all concrete mixtures ranged between 3.2 and 4.3%.
- In all concrete mixtures, the values of the ultra sonic velocity were higher than 4.5 km/s which is a boundary for strong concrete. Defects in the ITZ structure caused reduction of the value of ultra sonic velocity in the case of the KF, FT and RM concrete mixtures. It had an indirect effect on the dynamic elastic modulus of SCCs.
- All concrete mixtures had a high performance in terms of mechanical strengths. Due to the pozzolanic activity of fly ash and pulverized cathode ray tube glass, FA and RG concrete mixtures had the highest values of almost all mechanical strengths except the flexural strength which was the highest for the RM mixture.
- Secant modulus of elasticity ranged between 28.7 and 33.3 GPa whereby the RG concrete mixture had the highest value, and the RM mixture the lowest value.
- According to Neville [50], all used concrete mixtures can be considered as waterproof as none of them has a penetration greater than 30 mm.
- All concrete mixtures exhibited high freeze/thaw resistance with de-icing salt apart from the RM mixture, and they meet the quality requirements for the XF4 exposure class concretes according to SRPS EN 206-1 [40].
- All SCCs had a high degree of abrasive resistance and meet the quality requirements for the XM3 exposure class concretes according to SRPS EN 206-1 [40].
- The SSC mixture with red mud (RM) had the highest drying shrinkage during the first seven days. The FA concrete mixture had the highest drying shrinkage in the period from 28<sup>th</sup> to 180<sup>th</sup> day, and the RG concrete mixture had a slightly lower shrinkage. The other SCC mixtures had a similar drying shrinkage at all ages of concrete.
- SEM analysis showed that the cement matrix of all the mixtures is fairly uniform without clearly visible cracks and large pores. The presence of cracks and discontinuities in ITZ of LF, FT and RM concrete mixtures contributed to the decline of mechanical characteristics and properties of durability of these concretes.
- In general, it can be concluded that SCCs made with waste materials such as milled recycled glass from cathode tubes, flotation tailings from a copper mine, red mud and fly ash do not cause a decline in physico-mechanical characteristics and durability properties of self-compacting concretes (SCC), and they even improve some aspects of concrete performance in comparison with SCCs made with limestone filler as mineral admixture.
- Making of SCCs with such materials also contributes to preservation of the environment and to solving the issue of waste materials disposal.
- The future research should be focused on SCCs produced by combining mineral powder admixtures from the waste materials treated in this study.

## ACKNOWLEDGEMENTS

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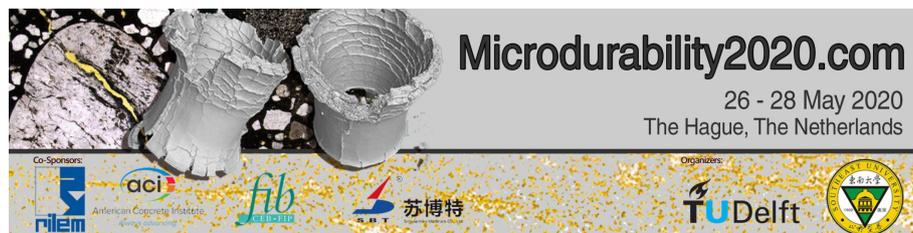
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## MANIFESTĂRI ȘTIINȚIFICE / SCIENTIFIC EVENTS



### 4<sup>th</sup> International RILEM Conference on Microstructure Related Durability of Cementitious Composites, 26-28.05.2020, Madurodam, The Hague, The Netherlands

#### Main Topics

- Alternative binders, supplementary cementitious materials and industrial or regional wastes used in concrete
- Hydration and microstructure formation
- Transport properties in cracked and uncracked concrete
- Chemical and physical degradation under coupled loading conditions.
- Effect of time dependent phenomena and aging on microstructure and durability
- New techniques for evaluation of hydration, microstructure and service life
- Development and application of smart cementitious materials for enhanced durability
- Modeling of microstructure, transport, degradation processes and design for durability

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