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To cite this article: P Figala and R Drochytka 2018 *IOP Conf. Ser.: Mater. Sci. Eng.* **385** 012012

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# The microstructure of a cement composite based on a secondary raw materials

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**Abstract.** Waste management and its use as a secondary raw material is a matter that is being given increasing attention. Such raw materials also include waste products from the energy industry or municipal waste disposed of from the recycling process. This study explores the use of secondary raw materials in specific cement-based cement composite as a partial binder substitute. Selected secondary raw materials were pre-treated by grinding to a specific surface of 300 and 500  $\text{m}^2 \cdot \text{kg}^{-1}$ . As a reference material, a commercially available mixture was selected to produce cement composites intended for the environment with high physical-mechanical demands. Cement was substituted by secondary raw materials at a rate of 20 and 40% in the reference recipe. Basic physical-mechanical characteristics such as compressive strength and tensile strength at bending were determined on standard 160x40x40 mm prisms. Strengths were determined after 3 and 28 days of ageing. In addition, the influence of binder substitute by the admixtures on the microstructure of cement stone was monitored by sample scanning using an RTG tomography. The positive effect of a part of the binder substitution by selected secondary raw materials on microstructure was proved in all tested samples. From the measured data, it is evident that the higher specific surface of the pre-treated admixtures did not have a significant positive effect either on strengths or the microstructure.

## 1. Introduction

One of the traditional secondary raw materials used in the construction industry is slag. It is an inorganic waste generated on grates or in furnaces by the combustion of coal, flammable slate and other solid fuels. Slag is used in building materials due to the pozzolanic character, but its greater use is limited by the large inhomogeneity and frequent occurrence of more pollutants. The use of slag as aggregate in concrete is influenced by several factors. The quality of slag is influenced by the rate of mechanical degrading pollution of organic or inorganic origin, quality and type of default fuel, combustion method, amount of fuel combustible residues, free CaO and MgO content, sulphur compounds and soluble salts [1][2].

The time and method of its deposition in temporary landfills also influences the characteristics of the slag. Prior to the use of slag, it is recommended to store it for a minimum of six months on free heaps where leaching of soluble salts occurs, which would otherwise cause blistering in concrete. Further, there is oxidation and leaching of sulphur compounds. Standard ČSN EN 450 provides a number of other criteria for slag as an admixture for concrete. For example, there is a requirement for a high SiO<sub>2</sub> content. In addition to the amorphous phase, the slag also contains mullite, anorite and melilite and lime clay rocks such as magnetite or gypsum. [3] Finally, the usability of the slag is limited by the maximum content of combustible substances (10%), the chemical character of the slag extract (pH in the range of 4–8) or by the specific activity ( $<150 \text{ Bq} \cdot \text{kg}^{-1}$ ) [1].

The use of silicate raw materials of amorphous character as building materials has also been the subject of studies for decades. In the alternative raw materials for concrete a whole range of glass



types (solid substances with a high content of amorphous SiO<sub>2</sub>) have a place [4]. The most commonly used is glass recycle, especially container glass from bottles, but also glass from glasses or glass panes, both in clear and coloured form. It is usually made from 50% siliceous sand, 10% Na<sub>2</sub>CO<sub>3</sub> (soda), 12% CaCO<sub>3</sub> (limestone), 18% crushed culled of waste glass and 4% other substances [5]. Not everything can be used for recycling, which is a major environmental problem around the world. For example, only glass sheets made of building demolition in the Czech Republic produce approximately 40 thousand tons per year.[6] Limiting the recyclability of waste glass causes different colouring, impurities, etc. The most widely produced glass type is soda–lime glass, accounting for almost 80% of all glass waste (63% of white coloured glass) [7] [8].

When using glass recycle in cement composites, it is necessary to consider the possible occurrence of an alkali–silica reaction (ASR) between the used glass (source of active SiO<sub>2</sub>) and alkalis. Due to the amorphous structure of glass, significant pozzolanic activity is also expected, which leads to the consumption of portlandite during the formation of neologisms. These are very similar to CSH phases and result from the hydration of Portland cement. These neologisms envelop the reaction edges of the glass grains and reduce the monosulfate level. This phenomenon reduces the risk of ASR occurrence and is closely related to the fineness of glass recycle grinding. Various studies state different limits of glass grading. In general any risk of ASR is eliminated if the average size of particles is below 300 μm [7] [8] [9].

## 2. Material composition

### 2.1. Characteristics of secondary raw materials

Thermal power plant slag (further Slag or EOS) was delivered in a fraction of 0–8 mm. Grounded recycle of container glass was derived from controlled production and was delivered in a fraction 0–32 mm. Pre–treatment of secondary raw materials for research purposes began by adjusting them to a 0–4 mm fraction using a jaw crusher. In the next step, the secondary raw materials were adjusted to predetermined specific surfaces: variant A = 300 m<sup>2</sup>·kg<sup>-1</sup> (± 50 m<sup>2</sup>·kg<sup>-1</sup>) and B = 500 m<sup>2</sup>·kg<sup>-1</sup> (± 50 m<sup>2</sup>·kg<sup>-1</sup>). The grinding drum for discontinuous grinding was selected for grinding. For minimization of the variables the mill rotation speed and emptying times were identical in all cases. For all samples of secondary raw materials, the following physical characteristics were verified density with a helium pycnometer, specific surface with a permeable method according to Blain's device and granulometry using laser diffraction analysis (table 2).

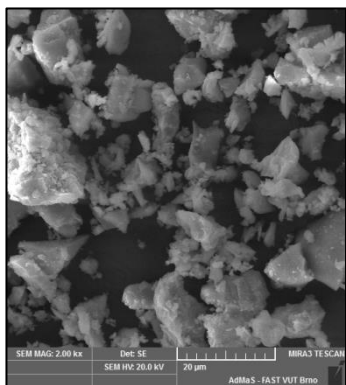
**Table 1.** Physical characteristics of pre–treated secondary raw materials.

Raw material	Designation	Density (kg·m <sup>-3</sup> )	Variant	Specific surface (m <sup>2</sup> ·kg <sup>-1</sup> )
<b>Slag</b>	EOS	2610	A	298
			B	530
<b>Waste glass</b>	Glass	2560	A	300
			B	541

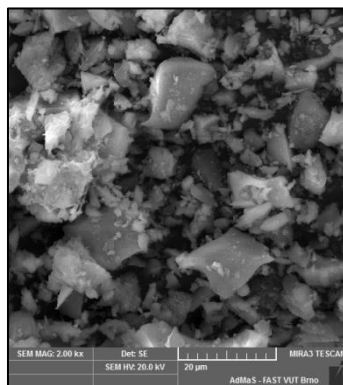
**Table 2.** Granulometry of pre–treated secondary raw materials.

Raw material	Variant	%>0,063 mm	0,01>%> 0,006 mm	%< 0,01 mm
<b>Slag</b>	A	42.83	55.63	1.48
	B	2.44	91.49	6.07
<b>Waste glass</b>	A	44.55	54.04	1.41
	B	12.47	77.89	9.64

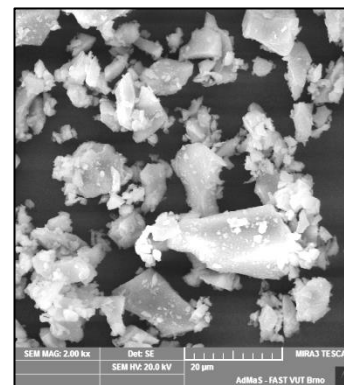
At the same time, pre-treated grain microscopy was monitored by SEM analysis (Figure 1–3).



**Figure 1.** Microscopic images of cement particles (CEM I).



**Figure 2.** Microscopic images of slag particles (EOS).



**Figure 3.** Microscopic images of waste glass particles (Glass).

*2.2. Test recipes*

As a reference substance (hereafter referred to as PH3), a high-strength floor screed from the renowned building materials manufacturer was chosen for research purposes (table 3). Substance PH3 was delivered as a dry homogeneous mixture and its material composition was based on a commercially available product. For the purpose of research, the manufacturer reduced the amount of CEM I 42.5 R Portland cement in dry mixture to 50%, so the other 50% could be substituted by secondary raw material. The reference dry mixture (hereinafter referred to as REF) was composed of the PH3 substance and the missing binder and was homogenized with a vertical screw homogenizer. The resulting composition of substance REF was based on the manufacturers prescribed ratio: 5:1:1 (PH3:CEMI42.5R:water). The water coefficient for the reference substance was determined by the manufacturer for 0.17 kg of water/1 kg of dry mixture with 100% dose of cement. The methodology of the dry mixture homogenization as well as the methodology of production and storage of prisms, which is not excluded by the technical sheets of the PH3 screed, was determined for research purposes. The standardized beams of dimensions 160×40×40 mm were selected as ideal test bodies for research purposes.

**Table 3.** Approximate material composition of the reference substance.

Material	The proportion in the mixture (%)
Portland cement CEM I	37–46
Coarse aggregate 4–8 mm	14–20
Fine aggregate 0–4 mm	24–38
Pozzolan admixture	3.5–4.4
Ground limestone	3–5
Mixture of additives	1.15–1.95

The other test recipes were based on the modified composition of the reference recipe. The binder was partially substituted by grounded slag (recipe marked with EOS) and glass recycle (marking with Glass) in a 20 and 40% rate of the total Portland cement CEM I 42.5 R dose. Overall, eight recipes were designed and manufactured containing two different admixtures, which were modified to two finenesses of grinding. Individually modified mixtures weighing 20 kg were mixed with a screw homogenizer and the homogenization time was set to 30 minutes to ensure perfect mixing.

In the next step, the optimal water coefficient was determined for each test recipe by the consistency test on a jogging table (CSN EN 1015–3). The water coefficient was chosen so that the consistency of the fresh substance was always the equivalent of REF. Subsequently, standardized

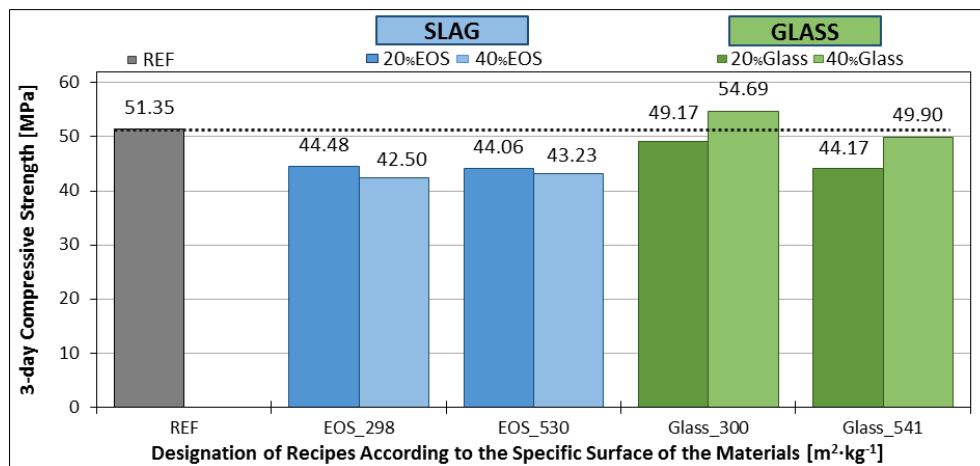
prisms of 160×40×40 mm were made and stored in a laboratory environment under PE foil and treated regularly with water. Test recipes has been marked as a follow-up to the secondary raw material used with a definite specific surface.

### 3. Evaluation of experimental testing

When designing the quantity of mixing water for individual recipes, it was found that for all recipes with the addition of secondary raw materials, the consumption of mixed water was reduced while maintaining the reference consistency. The major influence was the different water absorption of cement and alternative raw materials; the higher portion of the glass phase in the slag and in the glass was also important. Despite the lower water coefficient and the similar consistency set on the jogging table, the workability of substances with grounded glass was significantly better than the reference substance or the substances with slag. The effect was not only water absorption of glass but also a suitable form of the grains leading to a simpler compaction of the substance. The used screed was characterized by a considerable development of the hydrating temperatures after the beginning of the hydrating processes. This phenomenon was probably caused by the hydrating of the contained aluminous cement. Due to the significant development of hydrating heat and a very rapid increase of the initial strengths, it was suggested that the microstructure of the cement stone and the influence of the secondary raw materials on the development of the hydrating cracks will be studied on samples of selected recipes.

#### 3.1. Physical–mechanical characteristics

Compressive strength is the most significant material characteristic of cement composites. The production of test bodies and their testing complied with the standard CSN EN 13892–2, resp. standard for the testing of screed systems. The effect of the cement substitution rate by individual secondary raw materials on short–term strength after three days maturation and on long–term strength after 28 days maturation was monitored.



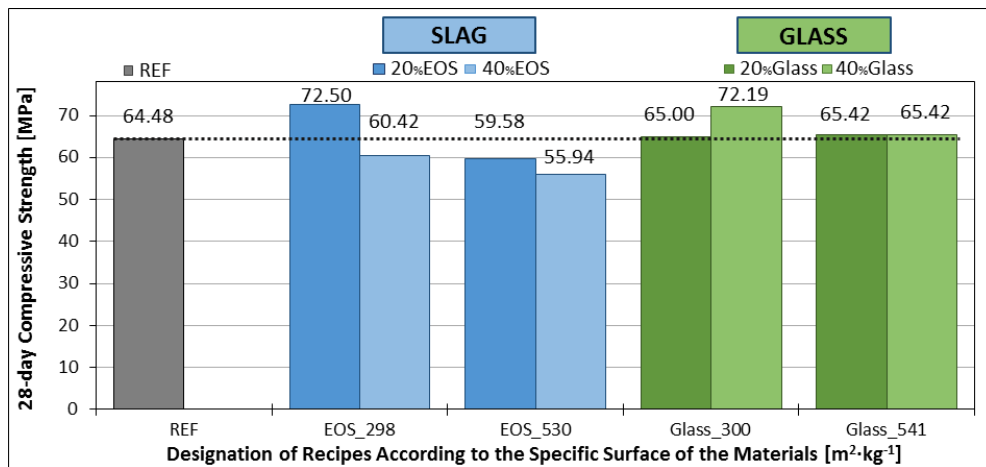
**Figure 4.** Compressive strength after three days of maturation.

Determination of early compressive strengths has shown that it is possible to replace larger amounts of cement in the screed while achieving similar or even higher strengths. These strengths (figure 4) were achieved with the use of grounded glass at a recipe of 40% Glass300.

For long–term compressive strengths (figure 5), the dependence between the lower specific surface of secondary raw materials ( $300 \text{ m}^2 \cdot \text{kg}^{-1}$ ) and higher compressive strengths was proven. The highest strengths after 28 days of maturation were achieved with 20%EOS298 recipes (113% of reference strength) and 40%Glass300 (112% of reference strength).

Long-term compressive strengths were determined also after 90 days of maturation and the typical pozzolanic trend of strength growth was confirmed. But the indicative properties of the specific

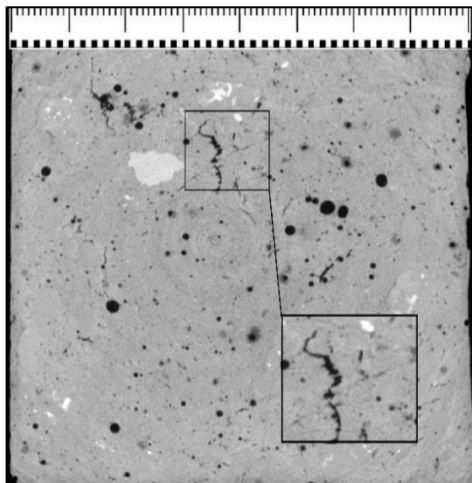
material are after 28 days of maturation, so the results after 90 days are not essential and are not part of this article.



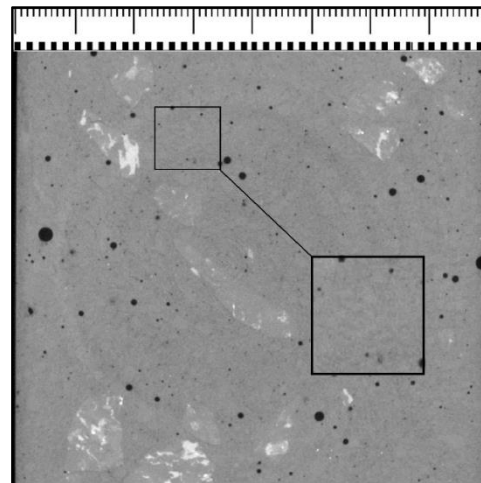
**Figure 5.** Compressive strength after 28 days of maturation.

### 3.2. Microstructure

The internal microstructure of prisms was studied using an X-ray (RTG) tomography. It is a method based on the radiography of the sample from a variety of angles in one plane, resulting in several hundred projections. These are subsequently reconstructed, and the output is a surface section of the examined object. Based on the evaluation of the strengths determined for the individual test recipes, two representative recipes for the study of the cement stone microstructure were selected.



**Figure 6.** A tomographic image of the test body structure REF.



**Figure 7.** A tomographic image of the test body structure 40%Glass300.

There were a large number of shrinkage cracks in the REF reference screed (Figure ), as demonstrated by scanning the prisms with a tomography. This confirmed a research concern, namely that due to the high hydrating temperatures developed at the early stage of the PH3 screed maturation, a disruption of the cement stone integrity occurs. By studying the microstructure of recipe bodies 40%Glass300 (Figure 7), a significant contribution of grounded glass as a raw material for the floor screed has been proven. The premise that the glass calms down the hydrating processes was confirmed. According to images of the screed internal structure, almost no shrinkage cracks occurred. Simultaneously, the number of larger cracks occurring by the processing of the fresh screed was significantly reduced in the prisms.

#### 4. Conclusion

Each figure should have a brief caption describing it and, if necessary, a key to interpret the various lines and symbols on the figure.

Based on the results of experimental research it can be stated that:

- Substitution of the binder with finely ground slag and glass has a positive effect on the workability of all modified substances regardless of the substitution rate or the admixtures fineness of grinding.
- The dependence between the lower specific surface of used secondary materials and the higher compressive strengths has been proven. The effect of cement substitution by secondary raw materials on compressive strength increased with maturation time and was more pronounced in milled glass recipes due to the higher pozzolanic activity of the admixture. Higher compressive strengths of up to 12% compared to the reference recipe were achieved.
- Due to the time and economic demands of concrete admixtures grinding, the optimal specific surface was chosen as  $300 \text{ m}^2 \cdot \text{kg}^{-1}$ . With this fineness of grinding, there is also no risk of an alkali–silica reaction occurring in the ground glass.
- The lower the specific surface of the secondary raw materials leads to the higher strengths from the insights of the characteristic properties of the reference screed. These secondary raw materials were less reactive and contributed to slowing down hydration processes and the development of smaller hydration cracks.
- By studying of the screed microstructure with a partial substitution rate of cement by secondary raw materials, the positive influence of all alternative raw materials was proven. The best results were achieved with a higher rate of cement substitution with ground sodium–calcium glass. The use of ground glass recycle of a specific surface of  $300 \text{ m}^2 \cdot \text{kg}^{-1}$  influenced the microstructure of the screed very positively.

#### Acknowledgments

This paper has been worked out with financial support from the budget under the project FAST–S–18–5406, “Study of advanced silicate composites structure with emphasis on the characterization of secondary raw materials influence” and under the project No. LO1408 "AdMaS UP – Advanced Materials, Structures and Technologies", supported by Ministry of Education, Youth and Sports under the “National Sustainability Programme I”.

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