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THE EFFECT OF WASTE GLASS SLUDGE USED AS CEMENT REPLACEMENT AND CRYSTALLISING ADMIXTURE ON THE PROPERTIES OF HARDENED CEMENT PASTE

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Two different mix compositions were prepared to compare the effect of waste glass sludge, with and without a crystallising admixture, on the properties of fresh cement paste and hardened cement paste. In Composition 1 cement was replaced by different percentage of waste glass sludge: 0 %, 5 %, 10 %, 15 %, 20 %, 25 %, and 30 %. In Composition 2 the same cement content was used with the addition of the crystallising admixture added at 1 % by weight of the binder. The tests showed that the replacement of 20 % of cement with waste glass sludge improved the properties of hardened cement paste, but the properties of fresh cement paste deteriorated. The results of the tests with composition 2 containing the crystallising admixture showed that the crystallising admixture had a negligible effect on the properties of hardened stone, while the properties of fresh cement paste improved in comparison with the specimens without the crystallising admixture. The overall results showed that 20 % was the optimum amount of cement to be replaced with waste glass sludge and that crystallising admixture could be one of the solutions to improve the workability of the mixtures modified with waste glass sludge.

INTRODUCTION

Sustainable development is gaining importance and increased attention globally with active involvement of researchers as well as business people. In general, sustainable development implies preservation of the environment and nature and reducing the use of energy resources. Rational use of energy resources is one of the areas that has been most extensively studied. Energy-intensive industries, including cement industry, raise great concern. The sustainability issues in cement production are gaining relevance not only from the environmental viewpoint but also concerning the increasing price of cement, which subsequently makes a great majority of construction products more expensive. Cement manufacturing is known to generate high amounts of carbon dioxide and other greenhouse gases [1]. One tonne of Portland cement produced causes the emission of approx. 0.9 tonne of carbon dioxide [2]. Several solutions are available to reduce the harmful effects of cement industry. One of them is the reduction of clinker content in various cement composites by replacing it with pozzolanic materials that are by-products in other industries [3, 4]. Another approach is to produce composite cements incorporating waste materials. Some waste materials, such as blast furnace slag, fly ash, waste glass, cement kiln dust, rice husk ash, metakaolin and silica fume, have been studied by researchers for their application in cement manufacturing. They have demonstrated a positive effect on cement properties and were found to improve the durability of hardened cement paste and its resistance to aggressive waste [5-9]. There is a significant amount of information on ternary blended cements available in literature. It has been found that the use of additional cementitious materials in certain proportions can improve the mechanical strength of cement paste [10].

Fly ash was the most popular pozzolanic waste material used in the production of cementitious composites for a very long time [11]. According to studies, fly ash improves the mechanical strength and durability of cementitious composites [12]. This positive effect of fly ash has made the market dependent on fly ash, but the shift towards renewable energy sources [13] has reduced the generation of fly ash and its availability in the market [14]. The decreasing supply of fly ash has caused its price to rise to the point where it is not viable any more to use fly ash in cementitious composites [15]. Therefore, researchers started looking for other by-products that can be used as alternative pozzolans instead of fly ash [16].

In order to substitute a certain amount of cement in the composites, a pozzolanic material must meet certain requirements. First of all, it must be chemically reactive and able to build a certain structure. Second, it must be easily available worldwide in order to reduce the cost of transportation and the end price of the by-product. One of such by-products comes from the glass industry. Glass waste has been known for a long time, but the scarcity of fly ash has intensified the research into the application of this by-product. Glass waste is attractive because of high silica content and the brittleness, the physical properties similar to those of concrete [17].

In 2015, the amount of glass waste in landfills was estimated to be around 10.4 million tonnes in the United States, where 60 % of glass waste is dumped in landfills, 26 % is recycled and the remaining 14 % is disposed of in incinerators [18]. Glass waste that does not reach the recycling loop is the untapped potential of this raw material. Glass recycling should be increased.

Ground waste glass can be reused in the form of pozzolanic glass powder. It is made from waste glass and has a particle size of less than 45 µm. Ground glass has been investigated because it is readily available in large quantities and due to its good reactivity with Portland cement, since glass is mainly composed of silica [19]. It was also discovered that the use of ground glass in concrete mixes would reduce carbon dioxide emissions. Basically, for every kilogram of glass used, cement producers can cut approx. 0.5 kg of CO_2 emissions [20]. Researchers have found that the negative effect of ground glass on the properties of hardened cement composites is very small. Slightly lower compressive and flexural strengths values were obtained [21, 22]. Other researchers have found that finely ground glass has pozzolanic properties and its behaviour in cementitious composites is similar to that of silica fume [17, 23]. Although the use of ground glass has a positive impact, its use must be limited. One of the drawbacks of using ground glass in cementitious composites is the alkali silica reaction (ASR) [24-31]. Meanwhile, many researchers have been studying ground glass and its applications [32-40].

Some researchers explore the application of not finely ground but only crushed glass as an aggregate in building materials. Researchers have used waste glass as a micro-filler in the production of asphalt concrete. They found that the addition of up to 18 % of waste glass as a micro-filler increased the stability of the end product, but the values were lower compared to asphalt concrete without the glass additive. A higher amount of waste glass increases the fatigue resistance and flowability of asphalt concrete [41]. The use of waste glass as a fine aggregate in cementitious composites has also been tested. Researchers have found a reduction in mechanical strength along with improved workability, durability, sulphate resistance and constancy of concrete volume [42]. The behaviour of crushed waste glass as a coarse aggregate in concrete was also tested. The replacement of 17.5 % of natural coarse aggregate with waste glass mixed with slag had a negligible influence on the mechanical properties of concrete [43]. Researchers also studied the effect of replacing basalt aggregate with waste glass and found that up to 25 % of the natural aggregate can be replaced. The addition of waste glass up to 25 % was found to significantly improve the mechanical strength of modified concrete, whereas a higher content of waste glass had a negative effect on the mechanical properties of concrete [44].

The application of waste glass sludge (WGS) in concrete has not been widely researched yet. This type of glass waste has received the attention of just a few researchers across the world. Researchers from South Korea studied the effect of waste glass sludge on the durability and frost resistance of concrete. They found that WGS increased frost resistance tested both with and without the deicing salts, chloride resistance and resistance to surface spalling of concrete [45]. Other researchers investigated the effect of WGS on concrete by varying the amount of cement used. Waste glass sludge was found to significantly improve the mechanical properties and microstructure of concrete, reduced its porosity and subsequently frost resistance and durability [46]. The effect of waste glass sludge on cement mortar was also studied by varying the cement content. The results reported by these researchers also showed that waste glass sludge improved the mechanical properties after 28 days of curing. It was found that WGS can reduce ASR caused expansion as effectively as fly ash and that WGS is even more reactive than fly ash [47]. Researchers have also tried to use WGS in the production of ceramic bricks. Their results indicated that up to 25 % of WGS can be added to the clay mix resulting in 37 % increase in the compressive strength of fired ceramic bricks. A decrease in porosity and water absorption was observed in the specimens with a higher WGS content [48].

EXPERIMENTAL

Cement CEN I 42.5 R complying with EN 197-1 requirements was used for the tests [49]. Waste glass sludge was obtained from the enterprise that cuts and polishes glass. Glass particles are extracted by a special equipment that purifies and treats technical water. After the flocculation and sedimentation process the collected material is removed from the technical water. The chemical composition of waste glass sludge (WGS) is given in Table 1 and its physical properties are given in Table 2.

Table 1. Chemical composition of waste glass sludge.

Chemical composition of WGS (%)											
SiO_2	Na_2O	CaO	MgO	Al_2O_3	SO_3	KO_2	CeO_2	Fe_2O_3	LaO_{23}	Cl	Cf
69.0	10.4	8.68	3.55	0.93	0.235	0.15	0.15	0.11	0.07	0.03	6.67

Table 2. Properties of waste glass sludge.

Properties	WGS
Specific surface area ($cm^2 \cdot g^{-1}$)	6670
Particle density ($kg \cdot m^{-3}$)	2500
Bulk density ($kg \cdot m^{-3}$)	826

The particle size distribution of WGS is shown in Figure 1. According to the test results, the average size of WGS particles was 3.98 µm. 10 % of the particles were smaller than 0.98 µm and 10 % were larger than 8.44 µm.

The specific surface area and particle size distribution of WGS were determined in dry dispersion mode using a particle size analyser Cilas 1090 LD in the range of 0.01 µm to 500 µm using air as a carrier. The particles were dispersed in ultrasound background until 12 % distribution of the material in the media was reached. The measuring span was 60 sec. The standard operating system Fraunhöfer was used.

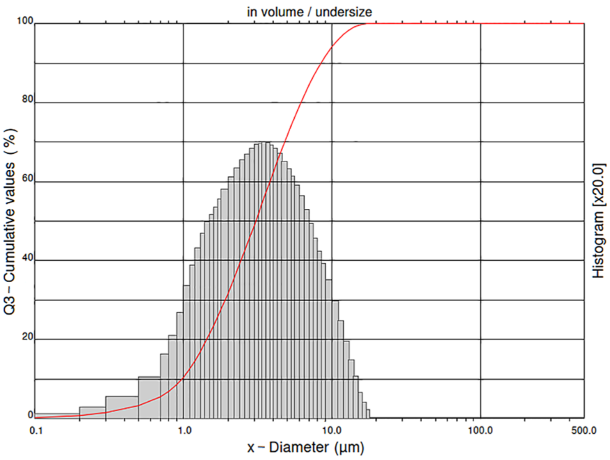


Figure 1. Waste glass sludge particle size distribution.

The XRD analysis was done with X-ray diffractometer BRUKER AXS D8 ADVANCE. The following XRD parameters were used: CuKα radiation, Ni filter, detector step of 0.02°, intensity measuring span of 0.5 s, anode voltage Ua = 40 kV, and the current I = 40 mA. The accuracy of XRD measurements was 2θ = 0.01°.

The X-ray fluorescence spectroscopy was done with the spectrometer Bruker X-ray S8 Tiger WD. Rh target X-ray tube was used, anode voltage up to 60 kV, current I up to 130 mA. The specimens were measured in helium atmosphere. SPECTRA Plus QUANT EXPRESS method was used for the measurements. WGS microstructure was analysed using SEM Helios NanoLab 650.

Vibro Viscometer SV-10 was used to measure the rheological properties of the cement paste. 45-50 ml samples of the cement paste were taken for the tests. Two plates were immersed into the container with the cement paste and vibrated at a uniform frequency and the resultant viscous drag was measured. The parameters of the modified cement pastes were measured at the start and after 5, 10, 15, 20, 25, 30 and 60 minutes.

Other properties of the cement paste were determined according to the respective standards: LST EN 12350-5:2019 [50] for the flow of the paste, LST EN 12390-7:2019 [51] for the density of hardened cement paste, LST EN 12390-3:2019 [52] for the compressive strength, and LST EN 12390-5:2019 [53] for the flexural strength.

The compositions of the cement paste batches tested are given in Table 3. The cement content in the batches was changed at 5 % intervals, up to a total of 30 % of cement being replaced with WGS. In the second stage of testing, the same amounts of cement were replaced and additionally a crystallising admixture was added at 1 % of the binder content. The superplasticiser content in the mixture was reduced in proportion to the crystallising admixture because the admixture has plasticising properties.

Table 3. Mixing proportion of hardened cement paste.

	Mixing proportions with waste glass sludge (%)						
Batches	1	2	3	4	5	6	7
Cement	100	95	90	85	80	75	70
Water	100	100	100	100	100	100	100
Chemical admixture	0.40	0.40	0.40	0.40	0.40	0.40	0.40
Waste glass sludge	0	5	10	15	20	25	30
w/c	0.35	0.35	0.35	0.35	0.35	0.35	0.35
	Mixing proportions with waste glass sludge and crystallising admixture (%)						
Batches	1	2	3	4	5	6	7
Cement	100	95	90	85	80	75	70
Water	100	100	100	100	100	100	100
Chemical admixture	0.30	0.30	0.30	0.30	0.30	0.30	0.30
Crystallizing admixture	1.00	1.00	1.00	1.00	1.00	1.00	1.00
Waste glass sludge	0	5	10	15	20	25	30
w/c	0.35	0.35	0.35	0.35	0.35	0.35	0.35

RESULTS AND DISCUSSION

The microstructure of waste glass sludge is shown in Figure 2. The image magnified 2000 and 10000 times shows that WGS is composed of irregularly shaped crystals.

X-ray diffraction analysis was used to determine the mineral structure of crystallising admixture. The results of the structural analysis of the crystallising admixture by X-ray diffraction are shown in Figure 3. X-ray diffraction image reveals that the prevailing mineral (32.7 %) is sodium carbonate (Na_2CO_3). Authors have noted [54, 55] that Na_2CO_3 can accelerate cement hydration and mostly accelerates C_3S hydration because the layer of hydrates around C_3S particles starts depleting in the carbonate solution due to the surplus of CO_3^{2-} ions. Then the Ca^{2+} and OH ion diffusion rate increa-ses significantly and the hydration of C_3S accelerates. Quartz (SiO_2), Q, is the second mineral in terms of content (31.4 %) in the crystallising admixture. The third mineral (29.4 %) is Tricalcium silicate (Ca_3SiO_4), T, followed by the fourth mineral (6.2 %) portlandite ($Ca(OH)_2$), P.

Presumably, C_3S, present in the crystallising admixture, was used to accelerate cement hydration and increase the early strength of concrete, as indicated in the reference [56]. According to the new findings about the hydration kinetics of tricalcium silicate, Na_2CO_3 accelerates early strength development [57].

The microstructure of the crystallising admixture is presented in SEM images in Figure 4. The image magnified 1000 times (a) shows that particles of irregular shape prevail. The image magnified 6500 times (b) shows that the particles of the crystallising admixture are made of irregular shape crystals and angular crystals distributed in different directions.

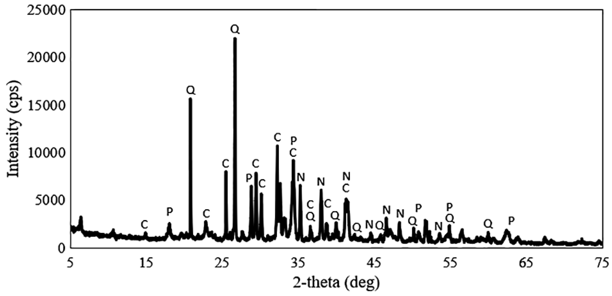
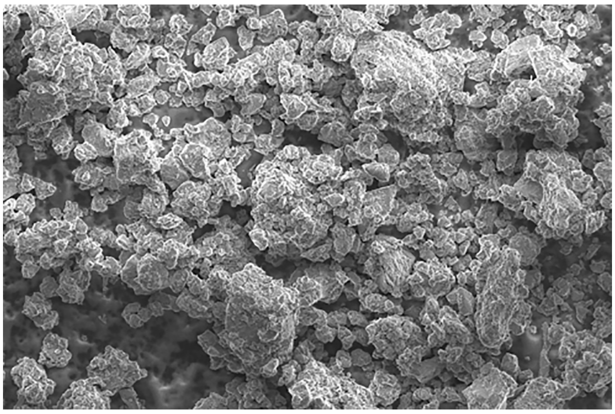
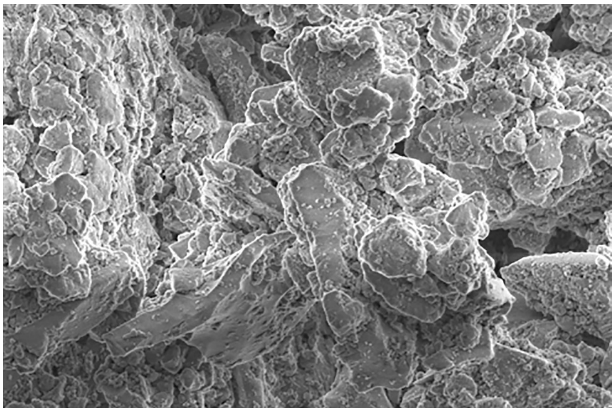


Figure 3. X-ray image of crystallising admixture: N – sodium carbonate (Na_2CO_3), C – tricalcium silicate (Ca_3SiO_4O), Q – quartz (SiO_2), P – portlandite ($Ca(OH)_2$).

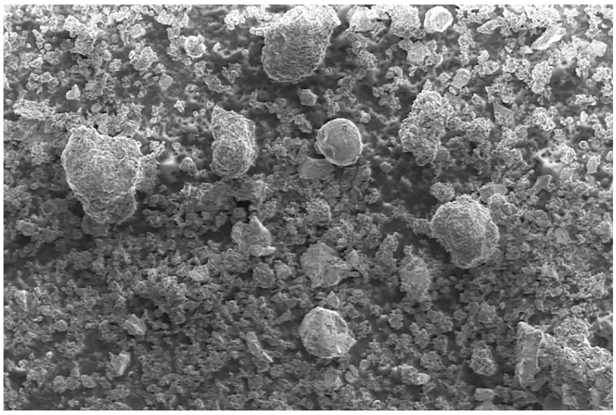


a) ×2 000

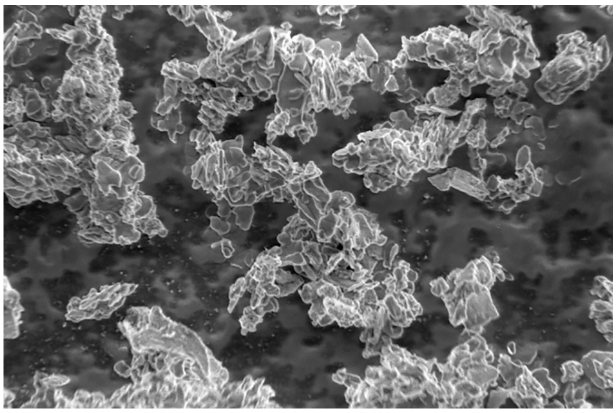


b) ×10 000

Figure 2. Microstructure of dry waste glass sludge: a) ×2 000 magnification; b) ×10 000 magnification.



a) ×1 000



b) ×6 500

Figure 4. The microstructure of the crystallising admixture: a) ×1 000 magnification; b) ×6 500 magnification.

The cement paste flow tests showed that the flow values decrease when cement is replaced with WGS. The results of the tests are given in Figure 5. The flow reduces in proportion to the higher WGS content in the mix. Cement paste specimens containing 30 % of WGS had a 36 % lower flow value compared to the control specimen. The decrease in flow values can be explained by the particle size. WGS particles are finer than cement particles; therefore, more superplasticiser or water is required to achieve the same flow results. When crystallising admixtures are added to the mixtures, the same trend is observed, i.e., the flow values decrease with the higher content of waste glass sludge. The flow of the cement paste where 30 % of cement was replaced with WGS decreased 37 %. After the addition of the crystallising admixture, less adverse dilution effect of WGS was observed and higher flow values were obtained because the admixture has good plasticising properties.

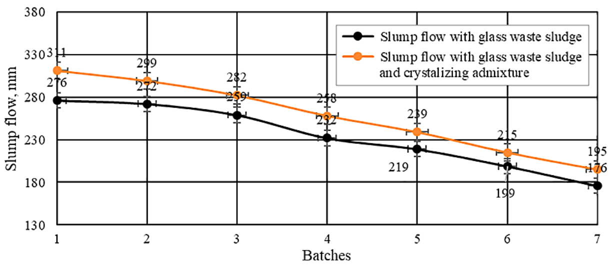


Figure 5. Slump flow of cement paste with waste glass sludge and with waste glass sludge and crystallising admixture.

The viscosity of the cement pastes was determined for the specimens, in which cement was replaced with WGS from 5 % to 30 %. The results are given in Figure 6. The results show that the viscosity of the specimens increased in proportion to the amount of cement replaced by WGS. The specimens had the same viscosity immediately after the mixing, but after 90 seconds the specimens containing WGS started to become more viscous. The viscosity of the control specimen remained practically unchanged throughout the test, while the viscosity of the specimens where 30 % of cement was replaced with WGS increased significantly. The results show that an intensive reaction of WGS with water starts after 90 seconds. The reaction changes the amount of free water in the mix and thus the viscosity of the mix increases [58].

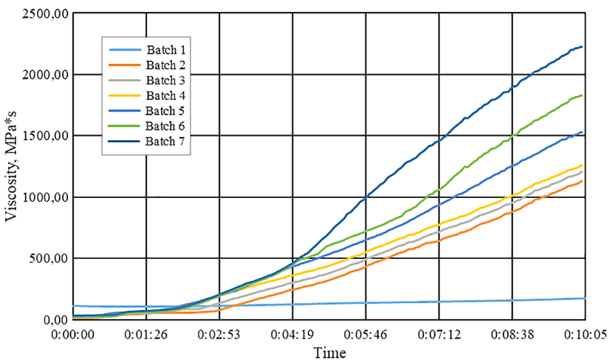


Figure 6. Viscosity of fresh cement mortar with waste glass sludge and crystallizing admixture.

Density values of different mix designs are given in Figure 7. The results show that the density increases with the amount of WGS present in the mix, i.e. the higher is the WGS content, the denser are the specimens. The density of the specimen containing 30 % of WGS is 52 kg·m^{-3} higher compared to the control specimen. The density of the specimens modified with the crystallising admixture was higher compared to the specimens with WGS only. The same trend of density increase with higher WGS content was also observed.

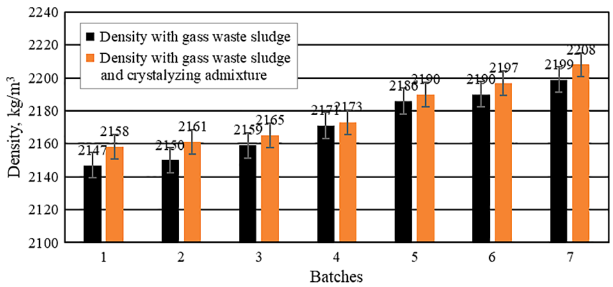


Figure 7. Density of hardened cement pastes with waste glass sludge and with waste glass sludge and crystallising admixture.

The results of the compressive strength tests show that WGS increases the mechanical strength of the specimens. The obtained compressive strength results are given in Figure 8. The chart shows that the compressive strength increases with the higher WGS content in the mix. The comparison of the control specimens with the specimens in which 30 % of the cement was replaced with WGS revealed an almost 17 % increase in strength. The specimens with the crystallising admixture had a slightly lower strength compared to the specimens without the admixture. Presumably, the crystallising admixture slows down the strength gain [59].

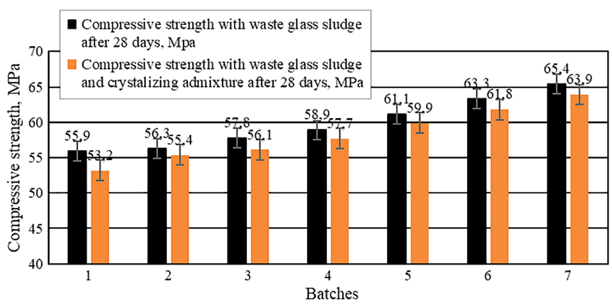


Figure 8. Compressive strength of hardened cement pastes with waste glass sludge and with waste glass sludge and crystallising admixture after 28 days.

The same trend was also observed in the flexural strength results given in Figure 9. The flexural strength increased in proportion to the higher WGS content in the mix. The specimens in which 30 % of the cement was replaced with WGS showed a 12 % increase in flexural strength compared to the control specimen. Similar to compressive strength results, the flexural strength results in the specimens with crystallising admixture were slightly lower.

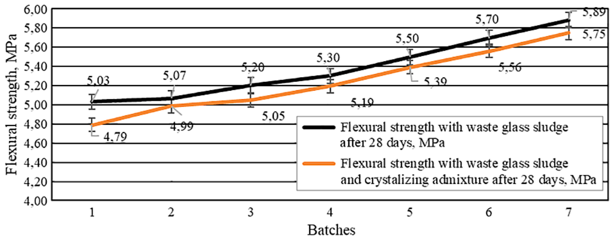


Figure 9. Flexural strength of hardened cement pastes with waste glass sludge and with waste glass sludge and crystallising admixture after 28 days.

CONCLUSIONS

The flow of the fresh mortar decreases with a higher WGS content in the mix. The more cement is replaced, the lower cement paste flow values are obtained. The specimens containing 30 % of WGS showed a 36 % decrease in flow compared to the control specimen. The addition of the crystallising admixture to the mix causes the flow of the cement paste to decrease at higher WGS content.

It was found that the mixes become more viscous when more WGS is added. The viscosity of the control specimen remained practically unchanged throughout the test, while the viscosity of the specimens where 30 % of cement was replaced with WGS increased significantly after 10 minutes.

The density of the mixes, both with and without the crystallising admixture, tended to increase with higher WGS content. The density of the specimens where 30 % of cement was replaced with waste glass sludge was 52 $kg \cdot m^{-3}$ higher compared to the control specimens, while the specimens with the crystallising admixture in the mix design had higher density compared to the specimens modified with WGS alone.

The compressive and flexural strengths were found to increase significantly with a higher waste glass sludge content. In comparison to the control specimens, the compressive strength increased 17 % and the flexural strength increased 12 % in the specimens, in which 30 % of cement was replaced with WGS. The specimens modified with a crystallising admixture showed slightly lower flexural and compressive strengths.

The obtained results lead to the conclusion that 20 % of cement can be replaced with waste glass sludge. Higher replacement rates deteriorate the workability of the cement paste, but the crystallising admixture compensates for the negative effect of WGS and improves the technological properties of the mixes. For this reason, it is possible to use a higher amount of waste glass sludge in the mixtures with the crystallising admixture.

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