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THE EFFECT OF POZZOLANIC WASTE AS CEMENT SUBSTITUTE ON THE PROPERTIES OF CEMENT MORTAR AND RESISTANCE TO ALKALI-SILICA REACTION

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The article analyses the effect of pozzolanic waste on the properties of cement mortar. Seven batches of specimens were made for the tests: control specimens without any pozzolanic waste, three batches of specimens where cement was replaced with 5, 10 and 15 % of wood fly ash, and three batches of specimens where cement was replaced with 5, 10 and 15 % of ground glass. The replacement of 5 % of cement with pozzolanic waste in the mix increased the density, the ultrasonic pulse velocity, compressive and flexural strengths of the mortar. A further increase of pozzolanic waste content degraded the values of physical and mechanical properties, as the lower amount of cement did not ensure a strong bond between the aggregate and the cement matrix. The alkali-silica reaction tests revealed that the expansion of the specimens containing 5 % of pozzolanic waste as cement replacement does not the permissible limit of 0.1 %. With the increase of pozzolanic waste content up to 15 %, the expansion of specimens modified with wood fly ash continues decreasing down to the limit of 0.01 %, whereas the expansion of the specimens modified with ground glass starts increasing to the limit of 0.05 %.

INTRODUCTION

By the end of the 20th century, sustainable development and environment protection became the key goals of modern societies. Civil engineering, especially construction materials industry, play an important role in sustainable development of the built environment, pollution abatement, conservation of natural resources and energy efficiency [1]. Partial replacement of Portland cement by supplementary cementitious materials is seen as the most effective measure for reducing CO_2 emissions related to concrete production [2]. The use of mineral additives as a partial replacement for cement in concrete is a common practice in construction industry [3].

Mineral admixtures are fine silica materials that are added in relatively large amounts during the mixing of concrete. Industrial by-products are the primary source of mineral additives [4]. Mineral additives, when incorporated into concrete mixtures, can have a significant impact on various physical properties of concrete [5]. These additives can modify such properties as strength, workability, durability, shrinkage, and thermal conductivity [6]. The specific effects depend on the type and proportion of the mineral additive used [7]. For the past few decades, mineral additives derived from industry by-products or natural pozzolanic materials, such as fly ash, ground granulated blast furnace slag, silica fume, metakaolin, limestone etc. have been used to partially substitute cement [8].

This paper analyses wood fly ash and ground glass mineral additives. Wood fly ash is a by-product generated from the combustion of wood-based biomass materials in various types of biomass power plants, heating systems, or industrial processes [9]. Wood fly ash can indeed be used as a mineral additive in various construction materials, including concrete. Fly ash derived from wood combustion shares some similarities with traditional coal fly ash but may have different properties due to its source material [10].

Ground glass, also known as glass powder or glass fines, can be used as a mineral additive in construction materials, including concrete and cementitious products [11]. The research into finely ground glass as a pozzolanic material is relatively new and has been stimulated by the continuous accumulation of waste glass and the associated environmental problems [12]. Studies have shown that waste glass has a potential for use in building materials, for example, as an aggregate replacement, a filler in concrete, or a cement replacement [13].

Alkali-Silica Reaction (ASR) is a deleterious chemical reaction that can occur in concrete when certain types of reactive silica-containing aggregates react with

the alkalis (sodium and potassium) present in the cement paste [14]. This reaction can have several adverse effects on concrete, including: expansion and cracking, reduced durability, loss of structural integrity, surface spalling, aesthetic issues, maintenance and repair costs [15]. It is important to note that the severity and progression of ASR can vary depending on different factors, such as the type of aggregates, environmental conditions, and the specific concrete mix [16]. Therefore, a comprehensive understanding of ASR and its potential effects is essential for the design, construction, and maintenance of durable concrete structures [17].

The effect of mineral additives on Alkali-Silica Reaction (ASR) in concrete can vary depending on the specific type of mineral additive, its characteristics, and how it interacts with the concrete mixture [18]. ASR is a chemical reaction that can occur in concrete when certain types of reactive siliceous aggregates react with alkalis in the presence of moisture, leading to the formation of a gel-like substance that can cause expansion and cracking [19].

The effect of wood fly ash on Alkali-Silica Reaction (ASR) in concrete depends on several factors, including the composition and characteristics of wood fly ash, the specific aggregates used in the concrete mix, and the overall concrete mix design [20]. Some types of fly ash, including certain biomass fly ash, have pozzolanic properties [21]. Pozzolanic materials react with calcium hydroxide and other alkalis in concrete to form additional cementitious compounds [22]. This reaction can help mitigate ASR by reducing the availability of alkalis for the deleterious reaction with reactive siliceous aggregates [23]. However, it is important to note that the effectiveness of wood fly ash in mitigating ASR can vary depending on the specific characteristics of the fly ash and the aggregates used in the concrete mix [24].

Previous hesitation to utilise glass particles in concrete as an aggregate include concerns regarding durability prompted by the reaction between alkali present pore solutions and amorphous silica present in glass, i.e., alkali-silica reaction (ASR). This issue has resulted in limited use of glass as an aggregate in concrete [25]. Finely ground glass not only increases concrete strength, but could also improve the sustainability and durability of concrete. Most research work has studied pozzolanic reactivity of finely ground glass through the mechanical properties of concrete, i.e., compressive strength and ASR expansion [26]. Finely ground glass powder has a potential to be used as a pozzolanic material; however, glass powder contains more reactive silica than fly ash, which is the most commonly used pozzolan. Besides, finely ground glass powder tends to improve the compressive strength more [27].

EXPERIMENTAL

The tested specimens were made of Portland cement CEM I 42.5 R and wood fly ash (pozzolanic waste) generated in the combustion of biomass. The chemical composition of Portland cement is presented in Table 1 and its physical properties are presented in Table 2. Physical properties of wood fly ash are presented in Table 3 and its chemical composition is presented in Table 4.

Table 1. Chemical composition of Portland cement (%).

Chemical composition of Portland cement (%)							
SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	SO_3	Na_2O	Other
20.76	6.12	3.37	63.50	1.00	0.80	0.30	4.15

Table 2. Physical and mechanical characteristics of Portland cement.

Properties	Portland cement CEM I 42,5 R
Specific surface area ($g \cdot cm^{-2}$)	3700
Particle density ($kg \cdot m^{-3}$)	3200
Bulk density ($kg \cdot m^{-3}$)	1200
Normal consistency cement paste (%)	25.4
Initial set time (min)	140
Final set time (min)	190
Compressive strength at 7 days (MPa)	28.9
Compressive strength at 28 days (MPa)	54.6

Table 3. Properties of wood fly ash and ground glass.

Properties	Wood fly ash	Ground glass
Particle density ($kg \cdot m^{-3}$)	2757	2500
Bulk density ($kg \cdot m^{-3}$)	829	868
Pozzolanic activity ($mg \cdot g^{-1}$)	360	560

Table 4. Chemical composition of pozzolanic waste.

Wood fly ash (%)										
SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	SO_3	Na_2O	P_2O_5	MgO	MnO_2	Cl	Other
61.2	3.71	1.08	12.9	4.71	1.63	0.46	2.27	1.88	0.31	0.39	9.46
Ground glass (%)										
SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	SO_3	Na_2O	P_2O_5	MgO	TiO_2	SrO	Other
70	0.83	0.12	9.84	0.24	0.23	14.20	0.55	3.74	0.05	0.02	0.18

Tables 5 and 6 present petrographic properties and physical properties of sand used for the tests and Table 7 presents the particle size distribution of sand.

Table 5. Mineral composition and petrographic characteristics of sand.

Mineral composition and petrographic characteristics	Sand fraction 0/4
Quartz	60.4 %
Feldspars	12.1 %
Carbonates	15.9 %
Mica	0.1 %
Dark minerals	4 %
Crystal rock	7.5 %

Table 6. Physical characteristics of sand.

Aggregate	Particle density ($kg \cdot m^{-3}$)	Bulk density ($kg \cdot m^{-3}$)
Sand fraction 0/4	2542	1604

Table 7. Particle size distribution of sand.

Particle size distribution of sand								
Sieve (mm)	0.063	0.125	0.25	0.5	1	2	4	8
Fine particles (%)	1	5	24	48	67	83	93	100
Min (%)	0	1	10	25	35	60	90	100
Max (%)	2	6	25	50	75	95	99	100

Table 8 presents the compositions of cement mortar specimens (one batch, six prisms). Seven batches of specimens were prepared for the tests: one batch without any pozzolanic waste, three batches with 5, 10 and 15 % ground glass added to replace cement and three batches with 5, 10 and 15 % of wood fly ash added to replace cement. W/R ratio in all mixes was the same, 0.47.

Dry materials were used to prepare concrete specimens. Cement and pozzolanic waste admixtures were dosed by weight, water and chemical agents were dosed by volume. Cement mortar mixtures were mixed in a forced action mixer, the mixing duration was 2 minutes. Cement mortar compositions were modified by different amounts of wood fly ash added by weight of cement. 40 × 40 × 160 mm prisms were used to determine physical and mechanical properties and ASR resistance of the specimens according to established research methods. The specimens were compacted on laboratory vibrating plate for 20 ± 2 seconds. The specimens were kept in forms for 24 hours at the ambient temperature of 20 ± 2 °C, then removed from the moulds and hardened in water at 20 ± 2 °C for 6 or 27 days. Part of the specimens were placed in a climatic chamber after 24h curing at 20 ± 2 °C and their ASR resistance was tested by expansion.

Phase analysis of pozzolanic waste was done by X-ray diffraction meter SmantLab (Rigaku) equipped with 9kW rotating copper anode X-ray source. X-ray diffraction patterns were recorded at 2θ diffraction angle ranging from 5° to 75° with increments of 0.02°

Table 8. Mixing proportion of mortar specimens.

Pozzolanic	Batches	Cement (g)	Sand (g)	Pozzolanic	Pozzolanic	Water (g)	W/R
	C	975	2193	0	0	450	0.47
Wood fly ash	W5	926.3	2193	5	48.8	450	0.47
	W10	877.5	2193	10	97.5	450	0.47
	W15	828.8	2193	15	146.3	450	0.47
Ground glass	G5	926.3	2193	5	48.8	450	0.47
	G10	877.5	2193	10	97.5	450	0.47
	G15	828.8	2193	15	146.3	450	0.47

and detector step 1° per minute. Database of crystal structures PDF4^{+} (2016) was used for the analysis.

The main physical and mechanical characteristics of hardened specimens were determined in accordance with LST EN 12390. Mortar specimens were dried to a constant mass and their density, compressive strength, flexural strength and ultrasonic pulse velocity values were determined after 28 days of curing. Expansion tests were done after 56 days of conditioning the specimens at 80 °C temperature in 1M NaOH solution.

Ultrasonic pulse velocity was measured with the instrument Pundit 7 (transducer frequency 54 kHz) and ultrasonic pulse velocity (VUIG in m·s^{-1}) was calculated from the equation:

$$UIG = \frac{l}{\tau} \quad (1)$$

where: l - specimen length, m, τ - signal propagation time, s.

Four prisms of each composition were taken for ASR resistance tests. The length of the prisms was measured and the prisms were placed in water of 80 °C. After 24 hours the length of the prisms was measured again (zero reading). Afterwards the specimens were placed in stainless steel containers with 80 °C 1 M NaOH solution. The containers were placed in a climatic chamber and kept at a constant temperature of 80 °C. The length of the specimens was measured regularly at the same hour of the day. The specimens were kept in water at 80 °C for 24 ± 2 hours and their length, i.e. the zero reading (L_0), was measured and used as a reference for the calculation of the specimen expansion. The lengths L_n of the specimens kept in the 1 M NaOH alkali solution at 80 °C were measured after 56 test days ± 2 hours. The expansion was calculated from the equation:

$$\text{Expansion}, \% = 100 \cdot \left(\frac{L_n - L_0}{l}\right) \quad (2)$$

where: L_0 - the length of the specimen prior to its soaking in sodium hydroxide solution, mm; L_n - the length of the specimen soaked in 80 ºC 1 M NaOH solution after 56 test days ± 2 hours, mm; l - the distance between specimen spacers, mm.

Expansion above 0.10 % indicates that cement mortar is not ASR resistant. Particle size distribution was measured by the laser particle size analyser CILAS 1090 Liquid. The microstructure of mortar, wood fly ash, ground glass was tested with the scanning electron microscopy (SEM) device SEM JEOL JSM-7600F. The following electron microscopy parameters were used: power 10 kV and 20 kV, distance to specimen surface 7 – 10 mm. Characteristics of the microstructure was identified by testing the specimen splitting surface. The image was obtained by recording the signal of secondary electrons. Before the testing, the splitting surface was coated with electrically conductive thin layer of gold by evaporating the gold electrode in the vacuum using the instrument QUORUMQ150R ES. The specimens of the tested materials were photographed by the digital camera Sony ILCE 5000.

RESULTS AND DISCUSSION

Figures 1 and 2 illustrate the particle size distribution of pozzolanic waste (wood fly ash and ground glass). Particle sizes were measured in the range of 0.1 – 500 μm. The average size of wood fly ash particles measured was 23 μm and the average size of ground glass particles measured was 15.1 μm.

The distribution of Portland cement particles by size is presented in Figure 3. The particle size was measured in the range of 0.10 – 500 μm and the average cement particle size was 15.05 μm.

The mineral structure of concrete mix ingredients was tested by means of X-ray diffraction analysis. Figure 4 illustrates X-ray Structure Analysis of wood fly ash. The X-ray image shows that silica dioxide (SiO_2) and calcite ($CaCO_3$) are the prevailing minerals in wood fly ash. Calcium hydroxide ($Ca(OH)_2$) and dicalcium silicate (Ca_2SiO_4) are also observed.

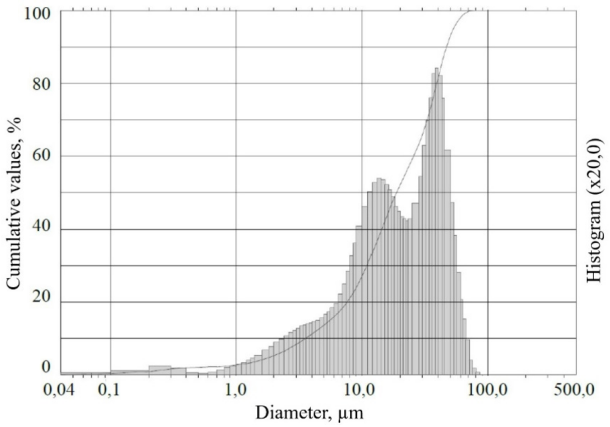


Figure 1. Distribution of wood fly ash particles by size.

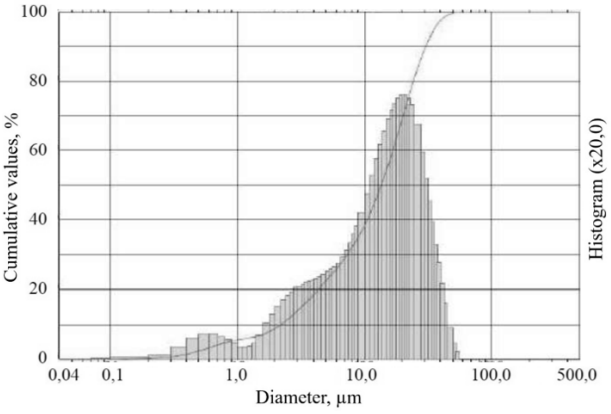


Figure 2. Distribution of ground glass particles by size.

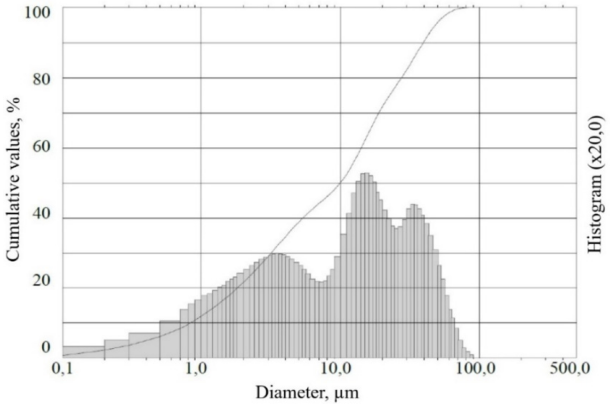


Figure 3. Distribution of Portland cement particles by size.

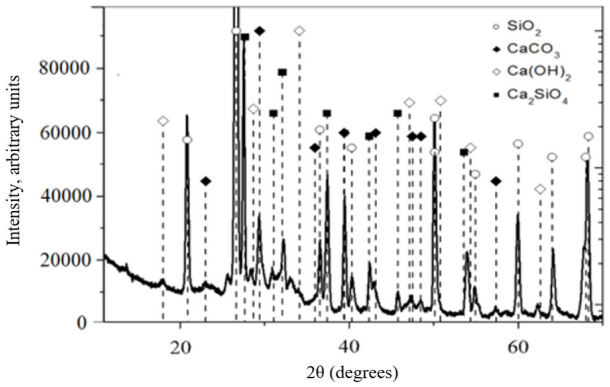


Figure 4. X-ray diagram of wood fly ash.

Figure 5 illustrates the results of ground glass X-ray diffraction analysis. The X-ray image shows the prevalence of quartz.

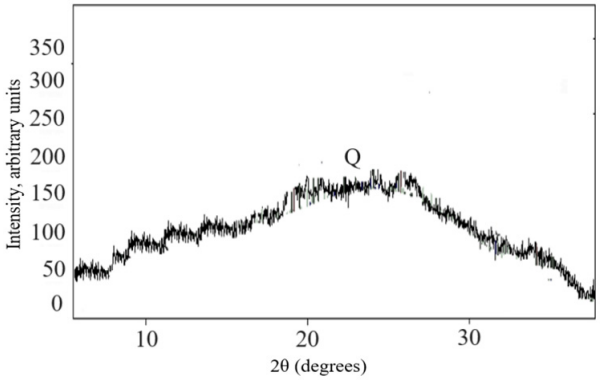
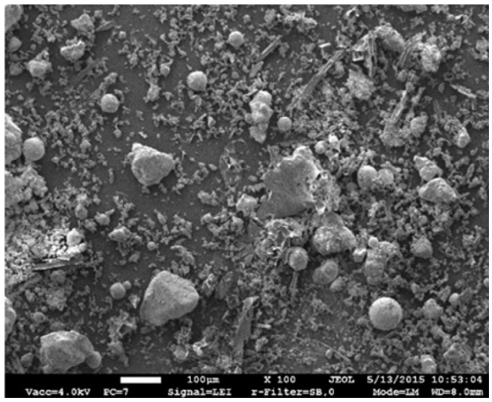


Figure 5. X-ray diagram of ground glass.

SEM images of wood fly ash microstructure are presented in Figure 6. Figure 6a shows the image of wood fly ash magnified 100 times. Most of the particles have an irregular shape. The particles are of variable size. The image magnified 5000 times (Figure 6b) reveals that the particles have a spherical shape. Figure 7a and b illustrates the microstructure of ground glass. Ground glass particles are of variable size and have irregular shape.


a) 100× magnification

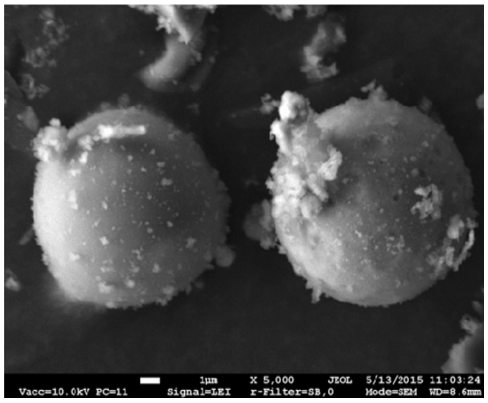
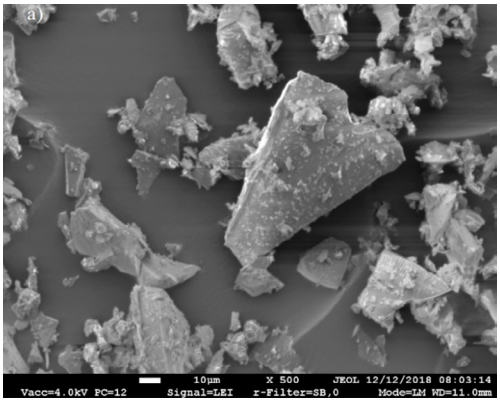

b) 5000× magnification

Figure 6. Microstructure of wood fly ash.


a) 500 × magnification

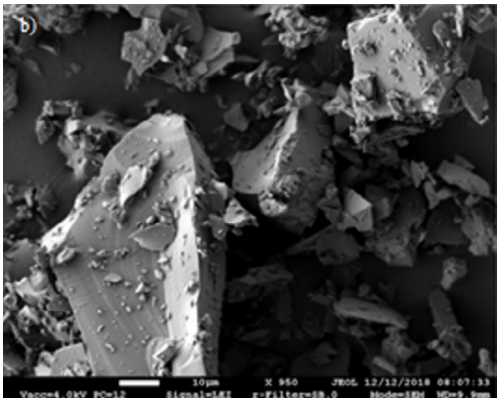
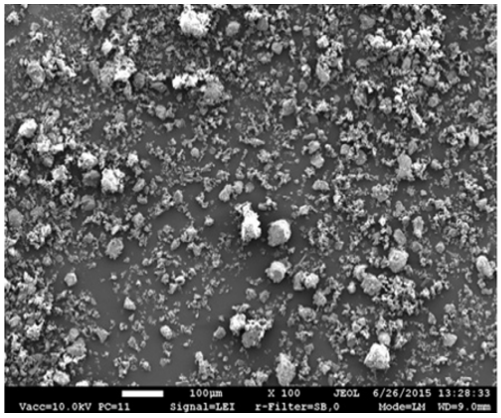
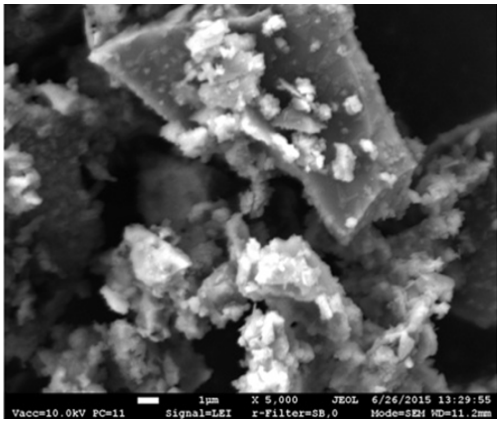

b) 950 × magnification

Figure 7. Microstructure of ground glass.

The images of Portland cement CEM I 42,5 R microstructure are presented in Figure 8. Portland cement particles have irregular shape, as seen in Figure 8a. The image magnified 5000 times (Figure 8b shows that Portland cement particles are made of irregular and angular crystals distributed in different directions.



a) 100× magnification



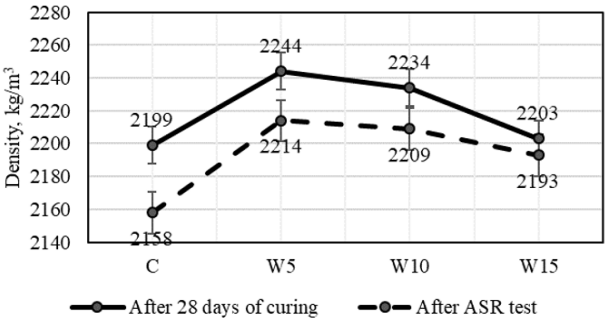
b) 5000× magnification

Figure 8. Microstructure of Portland cement.

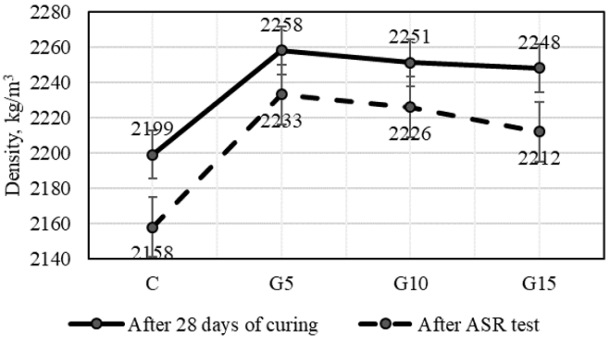
Figure 9 presents the comparison of density values of cement mortar modified with pozzolanic waste (wood fly ash in Figure 9a and ground glass in Figure 9b after 28 days of curing and after ASR test.

The highest density of 2214 kg·m^{-3} was determined in the specimens where 5 % was replaced with wood fly ash. After the ASR test, the density of the specimens where 5 % of cement was replaced with wood fly ash reduced 1.3 % down to 2214 kg·m^{-3}. After the ASR test, the specimens containing 10 % of wood fly ash by weight of cement showed a 1.1 % drop in density value down to 2209 kg·m^{-3}. A 0.5 % drop in the density value down to 2193 kg·m^{-3} was observed in the specimens where 15 % of cement was replaced with wood fly ash.

After the ASR test, the density of control specimen reduced to 2158 kg·m^{-3} and was 1.9 % lower than the density after 28 days of curing. The highest density value was recorded in the specimens where 5 % of cement was replaced with ground glass. After the ASR

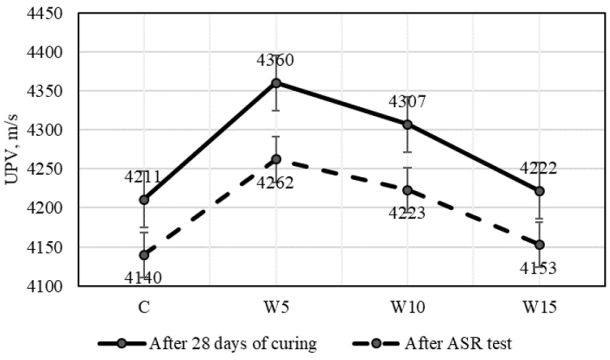


a) Wood biofuel fly ash

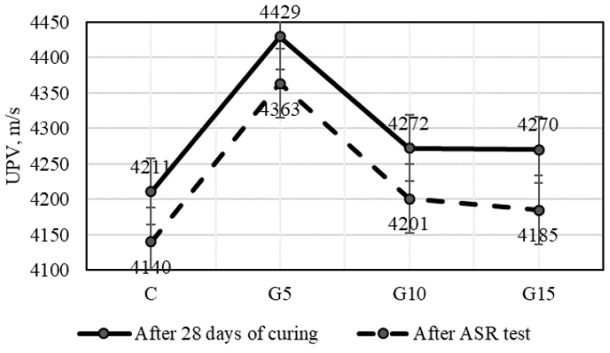


b) Ground glass

Figure 9. Relationship between wood fly ash content (a) and ground glass content (b) and the density of cured mortar specimens after 28 days and after ASR test.



a) Wood biofuel fly ash



b) Ground glass

Figure 10. Relationship between wood fly ash content (a) and ground glass content (b) and UPV values of cured mortar specimens after 28 days and after ASR test, %.

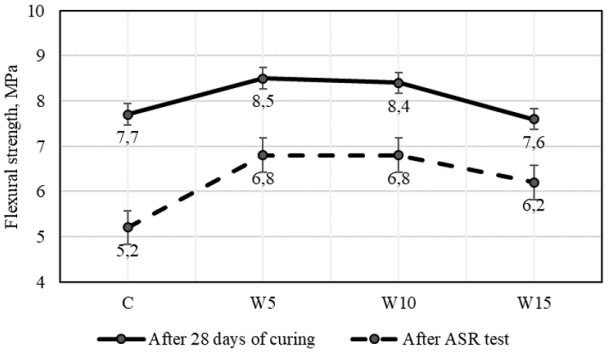
test, the specimens modified with 5 and 10 % of ground glass by weight of cement had a 1.1 % lower density of 2233 and 2226 kg·m^{-3} respectively, and the density of the specimens modified with 15 % of ground glass reduced 1.6 % down to 2212 kg·m^{-3}.

The comparison of ultrasonic pulse velocity (UPV) values of the specimens modified with pozzolanic waste after 28 days of curing and after ASR tests are presented in Figure 10. After 28 days of curing the control specimen had the UPV value of 4211 m·s^{-1} and after the ASR test the UPV value was 4140 m·s^{-1}.

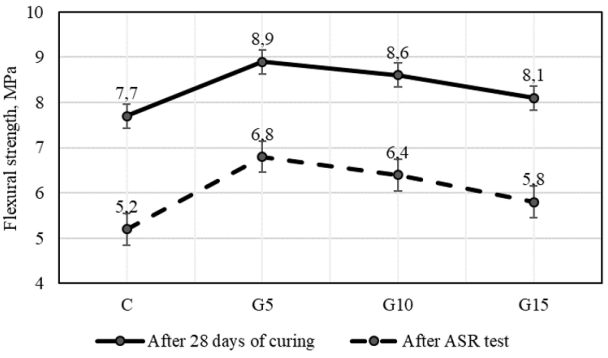
The comparison of UPV values of the specimens modified with wood fly ash after 28 days of curing and after ASR tests are presented in Figure 10a. The highest UPV value of 4262 m·s^{-1} was recorded in the specimens modified with 5 % of wood fly ash. It was 2.2 % lower than the UPV value after 28 days of curing. After the ASR test, the specimens containing 10 % of wood fly ash by weight of cement showed a 2.0 % drop in UPV value down to 4223 m·s^{-1}. A 1.6 % drop in the UPV value down to 4153 m·s^{-1} was observed in the specimens where 15 % of cement was replaced with wood fly ash. Figure 10b illustrates the UPV values after ASR test in the specimens modified with ground glass. The highest UPV value of 4363 m·s^{-1} was recorded in the specimens modified with 5% of ground glass. It was 1.5 % lower than the UPV value after 28 days of curing. After the ASR test, the specimens containing 10 % of wood fly ash by weight of cement showed a 1.7 % drop in UPV value down to 4201 m·s^{-1}. A 2.0 % drop in the UPV value down to 4185 m·s^{-1} was observed in the specimens where 15 % of cement was replaced with ground glass.

Figure 11 illustrates the relationship between the flexural strength of the specimens and pozzolanic waste content (Figure 11a) for wood fly ash and (Figure 11b) for ground glass. After the ASR test, the flexural strength of control specimen reduced 32 % down to 5.2 MPa.

After the ASR test, specimens modified with 5 and 10 % of wood fly ash by weight of cement had the highest flexural strength value of 6.8 MPa. The flexural strength changed the least (18.4 %) during ASR test and was 6.2 MPa in the specimens where cement was replaced with 5 % of ground glass. After 28 days of curing these specimens had the highest flexural strength of 6.8 MPa, but the ASR test caused the flexural strength to drop 22.5 %. After the ASR test, the specimens containing 10 % of ground glass by weight of cement showed a drop in flexural strength value down to 6.4 MPa, and the specimens where 15 % of cement was replaced with ground glass had the flexural strength of 5.8 MPa. The flexural strength changed the least during ASR test in the specimens where cement was replaced with 5 % of ground glass.

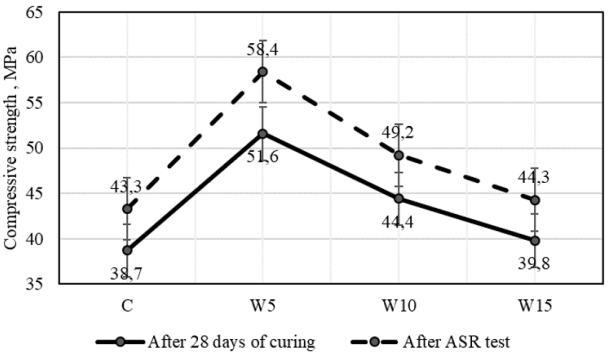


a) Wood biofuel fly ash

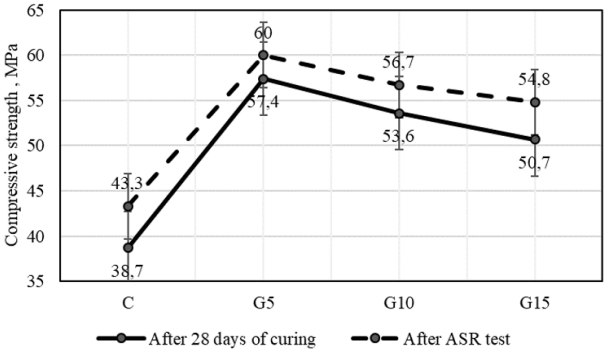


b) Ground glass

Figure 11. Relationship between wood fly ash content (a) and ground glass content (b) and flexural strength values of cured mortar specimens after 28 days and after ASR test, %.



a) Wood biofuel fly ash



b) Ground glass

Figure 12. Relationship between wood fly ash content (a) and ground glass content (b) and compressive strength values of cured mortar specimens after 28 days and after ASR test, %.

The effect of pozzolanic waste (wood fly ash) and ground glass on the compressive strength of mortar after 28 days of curing and after ASR test is presented in Figure 12. After the ASR test, the compressive strength of control specimen reduced 11.9 % down to 43.3 MPa.

The highest compressive strength value of 58.4 MPa was recorded in the specimens modified with 5 % of wood fly ash. It was 13.2 % higher than the strength after 28 days of curing. After the ASR test, the specimens containing 10 % of wood fly ash by weight of cement showed a 10.8 % increase in compressive strength value up to 49.2 MPa. A 11.3 % increase in compressive strength value up to 44.3 MPa was observed in the specimens where 15 % of cement was replaced with wood fly ash.

The highest compressive strength value of 60.0 MPa was recorded in the specimens modified with 5 % of ground glass. It was 4.5 % higher than the strength after 28 days of curing. After the ASR test, the specimens containing 10 % of ground glass by weight of cement showed a 5.8 % increase in compressive strength value up to 56.7 MPa, and the specimens where 15 % of cement was replaced with ground glass showed an 8.1 % increase in compressive strength value up to 54.8 MPa.

Figure 13 illustrates the expansion values of hardened mortars modified with pozzolanic waste (ground glass and wood fly ash) conditioned in 1M NaOH solution at 80 °C for 56 days.

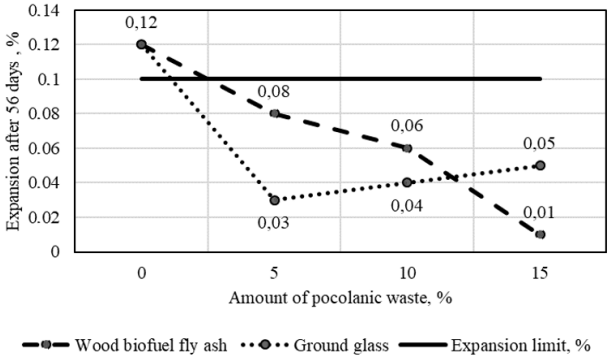


Figure 13. Expansion values of the specimens modified with wood fly ash and ground glass after conditioning in 1 M NaOH solution at 80 °C for 56 days, %.

The lowest expansion after 56 days was observed in the mortar where 5 % of cement was replaced with ground glass (0.03 %) and 15 % of cement was replaced with wood fly ash (0.01 %). When cement was replaced with 5 – 10 % of ground glass and 5 – 10 % of wood fly ash, the expansion values did not exceed the limit value of 0.10 %, beyond which the alkali-silica reaction processes start occurring in cement mortars.

The tests showed that with the ground glass content above 5 % the expansion of cement mortars started increasing, whereas the increase of wood fly ash content above 5 % reduced the mortar expansion.

The test results lead to the conclusion that pozzolanic waste, i.e. wood fly ash and ground glass, can substitute cement in cement-based mortars. In this way the amount of cement in the mortars can be reduced and wood fly ash and waste ground glass can be utilised. Modified cement mortars resistant to alkali-silica reaction can be produced by replacing 5 % of cement with waste ground glass or up to 15 % of cement with wood fly ash.

CONCLUSIONS

Pozzolanic waste was found to increase the density, ultrasonic pulse velocity, compressive and flexural strengths of hardened cement mortar. Physical and mechanical properties start deteriorating when more than 5 % of cement is replaced with pozzolanic waste. This happens because the reduced cement content does not ensure a strong blond between the filler and the cement matrix.

The highest density (2244 kg·m^{-3}), UPV (4360 m·s^{-1}), compressive strength (58.4 MPa), and flexural strength (8.5 MPa) values were recorded in the mortars modified with 5 % of wood fly ash.

When 5 % of cement was replaced with ground glass, the following highest values were recorded after 28 days of curing: density 2258 kg·m^{-3}, UPV 4429 m·s^{-1}, flexural strength 8.9 MPa, and compressive strength 60 MPa.

The tests revealed that after 56 days the highest expansion of 0.12 % was recorded in control specimens and the lowest expansion of 0.03 % was observed in the specimens modified with 5 % of ground glass. With an increase of ground glass content, the expansion increases to 0.05 %. Thus, the mortar modified with 5 % of ground glass experienced the least detrimental effect of alkaline environment.

The expansion was found to decrease when a higher content of wood fly ash was used. Hardened mortars modified with 15 % of wood fly ash had the least expansion of 0.01 % after 56 days of soaking in 1M NaOH solution at 80 °C. The test results lead to the conclusion that wood fly ash increases the resistance of cement mortars to alkali-silica reaction.

The comparative tests showed that during the ASR test the least changes in density, UPV, compressive and flexural strengths occurred in the specimens where cement was replaced with 5 % of ground glass and 15 % of wood fly ash. The structure of these mortars experienced the least impact during the ASR test.

The test results lead to the conclusion that pozzolanic waste, i.e. wood fly ash and ground glass, can substitute cement in cement mortars. In this way the amount

of cement in the mortars can be reduced and wood fly ash and waste ground glass can be utilised. Modified cement mortars resistant to alkali-silica reaction can be produced by replacing 5 % of cement with waste ground glass or up to 15 % of cement with wood fly ash.

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