

UTILIZAREA PULBERII DE STICLĂ ACTIVATĂ ALCALIN CA ADAOS ÎN CIMENTUL PORTLAND APPLICATION OF ZEOLITIZED GLASS POWDER IN CEMENT-BASED MATERIALS

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There is a lot of industrial and household waste in the world. Rational use of the waste by applying new technological solutions can significantly reduce energy consumption and emissions into the environment. Waste glass is one of such waste that, if non-utilized, fully decomposes in nature within a few thousand years. This paper presents the research of colourless ground waste glass used as a substitute for Portland cement. Glass powders were zeolitized to improve the pozzolanic properties of the material. The research results showed that hydration temperature and compressive strength of the specimens with 5% and 10% of the cement replaced by zeolitized admixture is similar or higher compared with the specimens where non-zeolitized glass powder is used. In summary it can be concluded that zeolitization of ground glass powder is feasible only in the production of specialized concrete products.

Keywords: recycled waste glass, zeolitized glass powders, cement supplementary materials, zeolite

1. Introduction

Cement industry is one of the most energy-intensive industries. Heat requirement is 3100-4400 kJ/kg while making cement clinker by dry means [1]. Partial replacement of clinker in Portland cement by other pozzolanic materials allows not only to reduce the cost of Portland cement, but also to protect the environment by reducing CO₂ emissions. Recently, the accumulation of waste glass causes a lot of environmental problems. Strict EU environmental directives require reducing solid waste, giving priority to recycling. The use of glass waste in construction industry is one of the potential ways of disposal [2].

Glass recycling is the process of turning waste glass into usable products. Sand, an exhaustible natural resource, is the most commonly used fine aggregate in concrete industry. Thus, the use of waste glass enables to solve the ecological problem and to save non-renewable resources. There are many studies where recycled waste glass can be used for partial replacement of fine aggregate [1-3].

Ground waste glass has great part of amorphous silica, therefore it can be used as cement supplementary material and this waste has

pozzolanic properties. The performance of concrete containing glass powder as 0-40% partial substitution of cement was investigated in previous studies [4]. The maximum strength of concrete occurs at around 10% of glass powder. The strength of concrete reduces beyond 10% of glass powder and is lower than that of the control specimen. Nassar et al. [5] found that recycled glass concrete incorporating milled waste glass as partial replacement for cement offers excellent strength and durability properties when compared with normal concrete. The pozzolanic reactions of milled waste glass with cement hydrates improve the microstructure and chemical composition of concrete. Previous studies [6] showed that glass powder in ultra-high performance cement-based materials had exhibited pozzolanic reaction and filling effect, contributing substantially to the increase of concrete strength.

Many researchers have investigated the effects of natural and synthetic zeolites on the structure of concrete and hydration processes in hardened cement paste [7-12]. Zeolites are a large group of natural and synthetic frame-structured hydrated aluminosilicates [13]. Although some natural zeolites occur in large amounts, they offer only a limited range of atomic structures and

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properties. Synthetic zeolites have a wider range of properties and larger cavities than their natural counterparts. Easy chemical modification of synthetic zeolites provides wide opportunities to perform controlled changes in their structure and properties. A number of research works about zeolitization are published [14, 15].

Zeolites have large quantities of reactive SiO_2 and Al_2O_3 ; thus they behave like pozzolanic materials: contributes to concrete strength mainly through the pozzolanic reaction with $\text{Ca}(\text{OH})_2$. Supposedly R. Snellings, E. Küçükıldırım et al. [16–18] have showed that pozzolanic effect of zeolite improved the microstructure of hardened cement paste and reduced the content of the harmful large pores, hence made concrete more impervious. Najimi M. et al. [19] state that zeolite showed more pozzolanic activity than amorphous natural pozzolans. The use of 15% zeolite improved strength and durability of concrete. Zeolite improved chloride penetration and corrosion rate of concrete as well. The shrinkage of zeolite incorporated samples was lower than the control sample [20]. Both natural and synthetic zeolites are increasingly used in construction industry. Natural zeolite can be considered to be an environmental friendly binder with a potential to replace a part of Portland cement in concrete in construction industry [21]. Madandoust et al. [22] determined that despite the observed decrease of proposed composites compressive strength, natural zeolite and metakaolin gained enough strength at the later ages similar to that of normal concrete. Synthetic zeolites can be synthesized from reagent substances and by using production waste. Two types of synthetic zeolite were synthesized from lignite bottom ash. This zeolite improved mortar strength and can partially replace Portland cement [23]. In the previous work [24] synthetic zeolite synthesized from industrial by-product was used like supplementary material in hardened cement paste. The compressive strength of the samples increased by 9 % compared with control samples. This research provides a real opportunity to save cement thus disposing the waste.

Literature review indicates that ground glass powder has weak pozzolanic properties and is mainly used in cement systems as fine aggregate. On the contrary ground glass powder are promising pozzolanic additives. Due to the amorphous Al_2O_3 , SiO_2 and Na_2O are the largest amount of oxides in the glass powder for this reason this material could be suitable for the synthesis of zeolites. Therefore, the aims of this work were to zeolitization of ground

glass powder and to investigate the possibilities of using the obtained material in cement systems.

2. Experimental

In this study commercial Portland cement of type CEM I 52.5R was used for the tests. Chemical and mineral composition of Portland cement is shown in Table 1. Colourless waste window glass with oxide composition given in Table 1 was broken by laboratory jaw crusher, then milled for 20 minutes in a ball mill and additionally grounded by vibratory mill until the specific surface of $194\text{m}^2/\text{kg}$ (hereinafter referred to Z0) was reached. The mean diameter of sharp crushed glass particles was $85\ \mu\text{m}$. According to XRD analysis this material is amorphous (Fig 1, 3 cr.). Reagents: NaOH (Chempur, Poland); $\text{Al}(\text{OH})_3$ (Lach-Ner, Czech Republic), CaO (Lach-Ner, Czech Republic).

According to [13], the initial material mixture $\text{Na}_2\text{O} : \text{Al}_2\text{O}_3 : \text{SiO}_2 : \text{H}_2\text{O}$ with molar ratio of 2 : 1 : 2 : 10 was selected for ground waste glass zeolitization reaction. In this case the zeolitization (the syntheses of zeolites) took place on the surface of glass particles from initial materials. Therefore, the additional NaOH, $\text{Al}(\text{OH})_3$ and water were necessary to add after the evaluation of waste glass chemical composition. Breck [13] reported that zeolite could be synthesised from some type primary materials: from solutions of sodium aluminate, sodium silicate and from active SiO_2 and Al_2O_3 source materials. For this reason, two different zeolitization methodologies were used in the research. In the first case (hereinafter, Z1) the dry material (glass powder with reagents NaOH and $\text{Al}(\text{OH})_3$) of the above shown proportion is mixed, and then the required amount of water is added; the obtained paste is thoroughly mixed until homogenous mass. Zeolitization of the mixture is conducted under hydrothermal conditions for 3 hours at $110 \pm 5\ ^\circ\text{C}$. The excess of NaOH was washed with water after the synthesis reaction in the products. The obtained product was dried to constant weight in the oven at $100\ ^\circ\text{C}$. In the second case (hereinafter, Z2), initially and aqueous solution of aluminate was prepared, by dissolving $\text{Al}(\text{OH})_3$ in the aqueous NaOH solution and poured on the ground waste glass. Zeolitization of stirred obtained mass, product purification and drying was carried out under the same conditions as in the first case.

X-ray powder diffraction data was collected by DRON-6 X-ray diffractometer with Bragg-Brentano geometry using Ni-filtered $\text{Cu K}\alpha$

Table 1

Chemical and mineral composition of Portland cement clinker

Chemical composition %								Mineral composition %			
SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3	MgO	K_2O	Na_2O	C_3S	C_2S	C_3A	C_4AF
20.61	5.45	3.36	63.42	0.80	3.84	1.31	0.94	56.7	15.5	8.2	10.7

radiation and graphite monochromator, operating with the voltage of 30 kV and emission current of 20 mA. The step-scan covered angular range of 2°-70° in steps of $\Delta = 0.02^\circ$.

The structures of waste glass and zeolitized glass powders were studied by a scanning electron microscope. A high resolution scanning electron microscope FEI Quanta 200 FEG with a Schottky field emission gun (FEG) was used for the research. Chemical compositions of glass powder and zeolitized glass powder were investigated by an energy-dispersive X-ray spectrometer (EDS) with silicon type drift droplet detector.

Thermographic investigations were carried out using a differential scanning calorimeter STA 409 PC, produced by Netzsch. The temperature ranged from 30 °C up to 600°C under the air atmosphere and the velocity of the increase in temperature was 15 °C/min.

Particle size distribution and specific surface area were determined by "Mastersizer 2000" instrument from Malvern. Red light was produced by helium-neon laser and blue light was obtained from a solid phase source.

The pozzolanic activity was assessed by chemical Chapelle test [7, 25]. By this method, it was proved that calcium hydroxide causes the hydrolysis of the waste glass, followed by calcium silicate hydrate formation. One gram of waste glass is added in 250 ml of distilled water followed by the addition of 2 g of CaO. The mixtures are then boiled for 16 h. After that the solution is filtrated and the solution is titrated with HCl solution (1 M) and phenolphthalein in order to find the amount of Ca(OH)₂ that has not participated in the pozzolanic reaction.

The cement paste hydration temperature was measured by means of 8-channel USB TC-08 Thermocouple Data Logger.

The specimen constant flow in all test was stable and the ratio of water and solid material was constant and equal to W/S = 0.35. The compositions differed by the contents of ground glass Z0, and zeolitized glasses - Z1 or Z2: 0%, 5%, 10%, and 20 % by mass. The compressive

strength of hardened cement paste was determined according to EN 196-1 norm. An automated and computerized Toni Technik 2020 press was used to evaluate the compression strength of hardened cement paste.

3. Results and discussion

3.1. Ground glass powder zeolitization

X-ray diffraction pattern analysis data show that zeolitization reaction in the case of Z1 was slower and the completely unreacted Al(OH)₃ (Fig. 1, curve Z1) was left in the obtained product. Sodium aluminosilicate hydrate and calcium aluminosilicate hydrate (Fig. 1, curve Z2) products prevailed in Z2 case. Ground glass powder (Z0) is in amorphous form (Fig. 1, curve Z0).

According the EDS analysis it was determined that Al₂O₃, SiO₂, CaO and Na₂O prevails in the ground glass powder and zeolitized ground glass powder (Table 2).

Table 2.

The arithmetic mean of oxide composition of ground glass powder (Z0) and zeolitized ground glass powder (Z1 and Z2)

Oxides	Quantity, %		
	Z0	Z1	Z2
SiO ₂	64.92	61.43	54.24
Al ₂ O ₃	2.81	8.27	14.95
CaO	5.34	8.15	8.11
Fe ₂ O ₃	0.35	-	-
MgO	4.55	4.99	4.02
Na ₂ O	20.52	16.81	18.43
K ₂ O	0.27	0.33	0.24

The morphology of the zeolitized glass powder Z1 and Z2 was examined by scanning electron microscope. SEM analysis of Z1 (Fig. 2b) shows irregular particles in a wide range of size. The aforesaid particles are located on the surface of glass particles (Fig. 2a). Hydrosodalite nano crystals formed on the glass fragments in Z2 case (Fig. 2c, 2d). Other researchers have found similar shape of hydrosodalite crystals in their works [26].

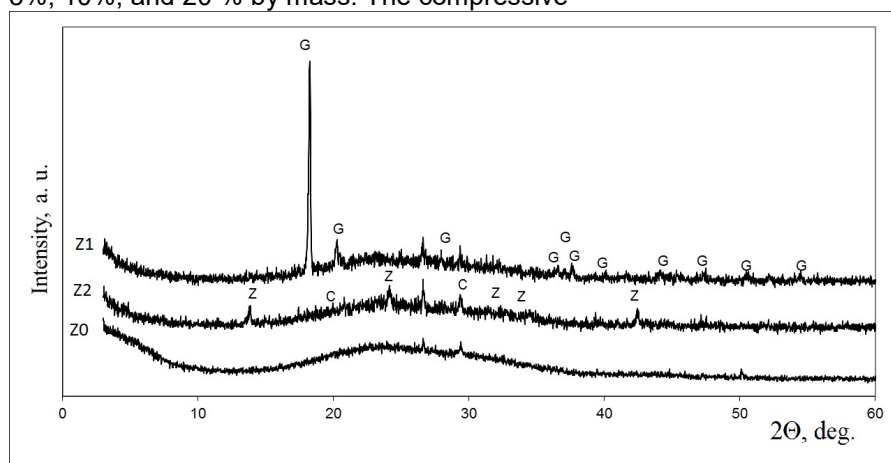


Fig.1 - X-ray diffraction pattern of ground zeolitized glass powder. Notes: G is aluminium hydroxide Al(OH)₃(7-324); Z is sodium aluminium silicate hydrate Na₈(AlSiO₄)₆(OH)₂·2H₂O (41-9); C is calcium aluminium silicate CaAl₂Si₂O₈·4H₂O (20-452).

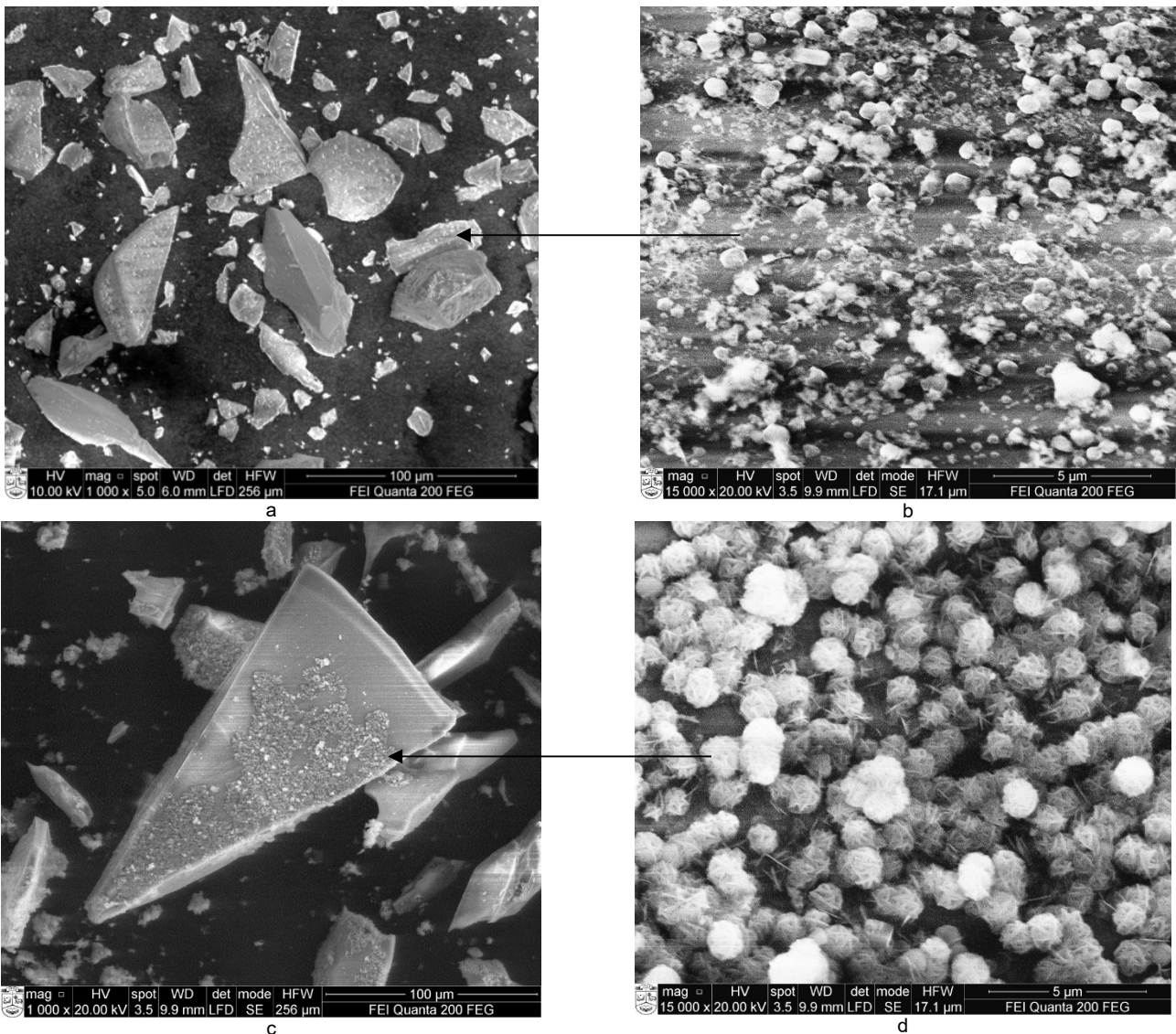


Fig.2 - SEM picture of zeolitized glass powder: Z1 (a and b), Z2(c and d).

This paper presents the results of pozzolanic reactivity under Chapelle test method. The test measures the reduction of Ca(OH)_2 by combination with siliceous or aluminosilicates materials present in pozzolans.

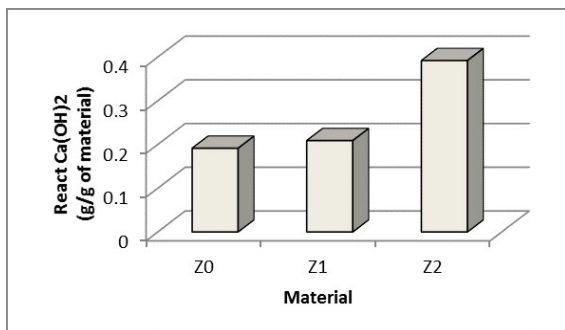


Fig.3 - The pozzolanic activity of ground waste glass (Z0) and zeolitized ground glass (Z1 and Z2).

After the Chapelle test it was determined that pozzolanic activity of the resulting product

obtained by Z1 method was slightly higher (0.209 Ca(OH)_2 g/g of glass) and almost double (0.392 Ca(OH)_2 g/g of glass) for the ground glass (0.191 Ca(OH)_2 g/g of glass) obtained by Z2 method (Fig. 3). A. Khmiri with co-authors [25] received a similar result (from 0.14 to 0.32 Ca(OH)_2 g/g glass) investigating pozzolanic activity of the specific surface of non zeolitized white and green ground glass powder. The increase of pozzolanic activity for the zeolitized glass could be explained by forming zeolitic compounds during zeolitization process. Zeolites are regarded as materials which demonstrate excellent pozzolanic reactivity [9, 11].

According to the results of granulometric analysis it was established that Portland cement particle size varied more evenly from $0.05\mu\text{m}$ to $52.5\mu\text{m}$, forming two peaks at $1.8\mu\text{m}$ (1.75% of all particles) and at $19.42\mu\text{m}$ (2.14% of all particles) in the distribution histogram. The specific surface of Portland cement was equal to $500\text{m}^2/\text{kg}$.

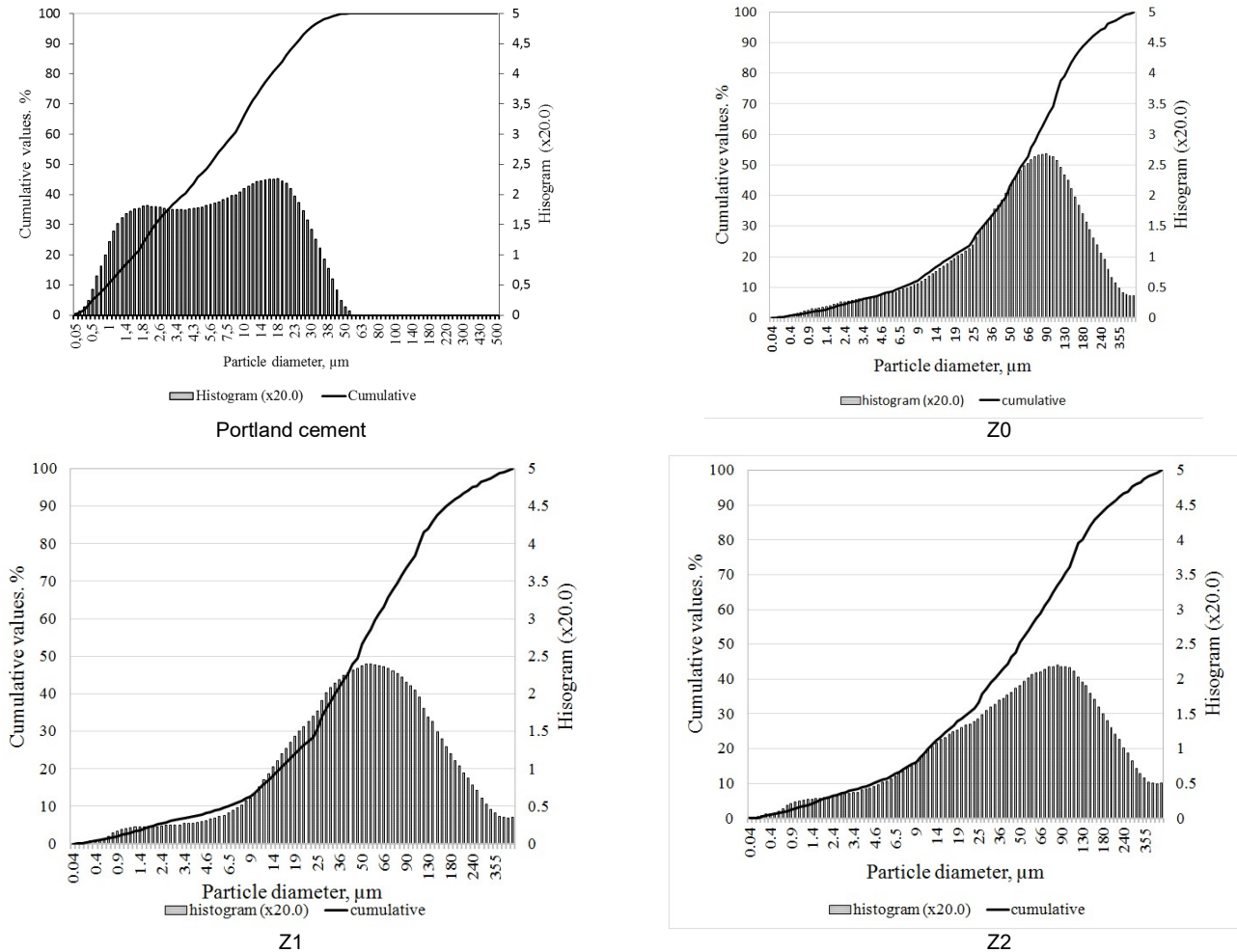


Fig. 4 - Particle size distribution for Portland cement, Z0, Z1 and Z2.

While the Z0, Z1 and Z2 particle distribution is similar, the specific surface of Z0, Z1 and Z2 is 194 m²/kg; 219 m²/kg; 258 m²/kg respectively. This is due to the fact that zeolitization of ground glass powder increases the specific surface. So, Portland cement is more than two times finer than the particles of ground glass powders and zeolitized ground glass powders (Fig. 4). This result suggested that the more fine material could cause a greater pozzolanic reaction. This aspect is a good agreement with the correlation between pozzolanic activity and particle size distribution proposed by Cordeiro et al. [27]. It was concluded that a good correlation exists between the 'external' specific surface areas, that is, the surface is associated with the measured particle size distribution, and the pozzolanic activity.

3.2. Properties of hardened cement paste specimens

Hardened cement paste specimens were formed by replacing 0, 5; 10 and 20% of Portland cement by ground glass powder (Z0) or zeolitized glass Z1 or Z2.

X-ray diffraction analysis of hydrated specimens (Fig. 5, a) show that calcium silicate

hydrate, alite, belite, portlandite and calcite, as well as small amount of ettringite dominated in the hardened cement paste with and without supplementary cementitious material (Z0, Z1, Z2). Portlandite main peak (0.493 nm) intensity was close to the peak intensity of the control specimen in the specimen with 20% of Portland cement replaced by Z0. It is likely that portlandite amount should therefore be similar to that of the control specimen (Fig. 5, b). The main Portlandite peak ($d = 0.493\text{nm}$) reduces almost twice when Z1 or Z2 is used as a supplementary cement material. It can be concluded that their placement of Portland cement binds portlandite to secondary calciumsilicate hydrates during the hydration. Uzal et al. and Mertens et al. determined that zeolite has a high pozzolanic activity [28, 29]. The high pozzolanic reactivity of zeolite is attributable to its specific surface area and reactive SiO₂ content.

Thermal analysis was carried out with hardened cement paste specimens containing 0 and 20 % of supplementary cementitious material. The thermograms (Figure 6) showed that endothermic peaks of DSC curves in the temperature range between 103 °C and 268 °C indicated the elimination of water at C-S-H gel phase and calcium aluminosilicate hydrates. The

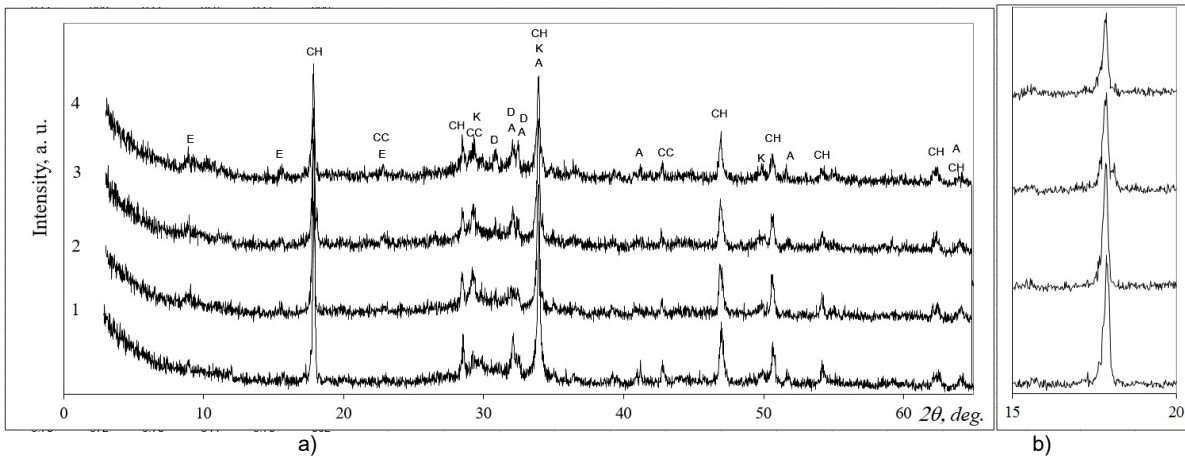


Fig.5 - X-ray diffraction patterns of hardened cement paste after 28 days. Notes: Ref is reference specimen.CH is portlandite (44-1481); A is alite (13-272); K is calcium silicate hydrate (33-306); D is larnite (83-460); E is ettringite (41-1451); CC is calcite (72-1651).

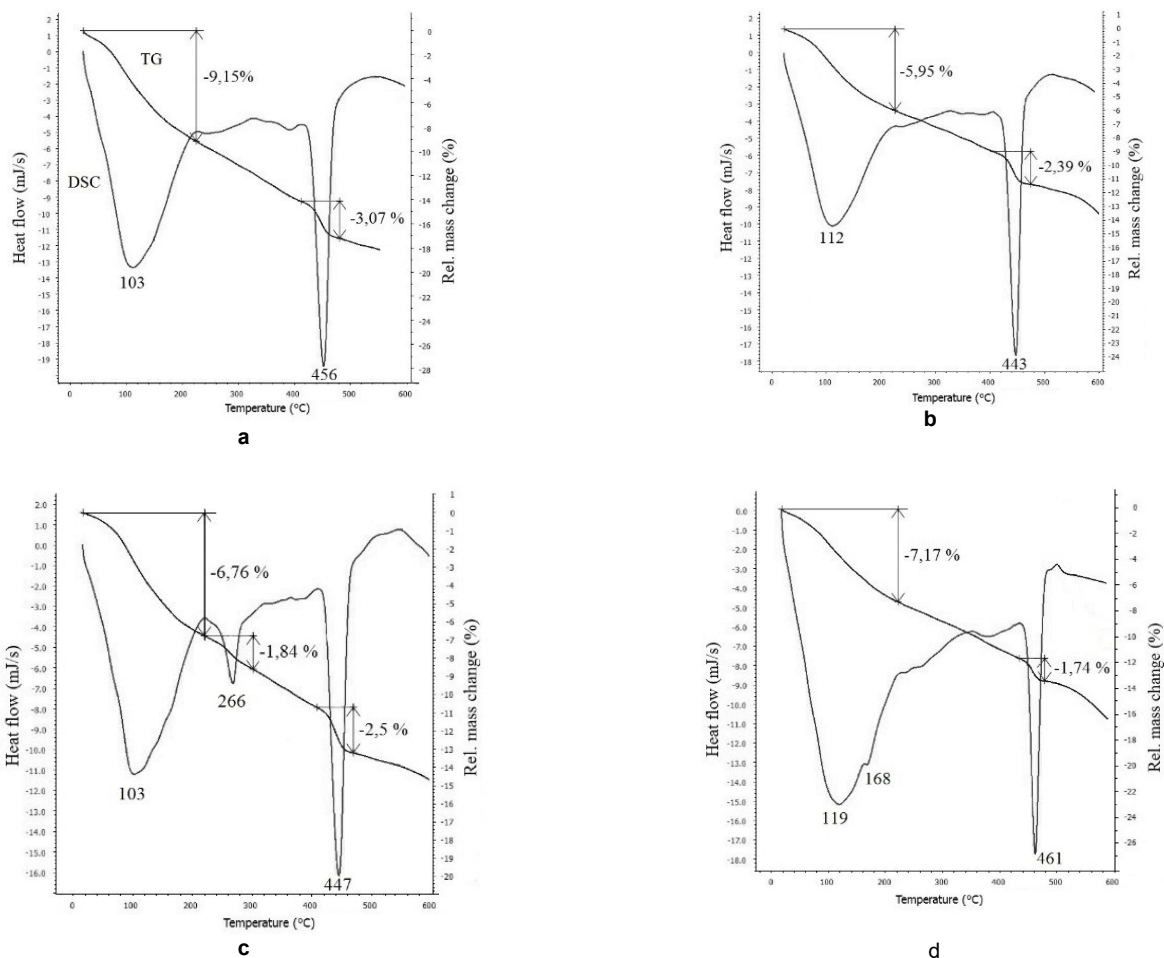


Fig.6 - DTA curves of hardened cement paste after 28 days. Reference specimen (a), 20 % Z0 (b), Z1 (c), Z2 (d) was used as supplementary cementitious material.

second intensive endothermic peak with the temperature maximum about 443 °C - 456 °C indicated the dissociation of portlandite $\text{Ca}(\text{OH})_2$.

The weight loss of the reference specimen without supplementary cementitious material, associated with $\text{Ca}(\text{OH})_2$ decomposition, was 3.07%. So, it would be 12.62% of portlandite according to mass losses. In the specimens with

20% supplementary cementitious materials (Z0, Z1 or Z2) the quantities of portlandite decrease till 9.82%, 10.28%, and 7.15% respectively (Table 3). It can be observed that the consumption of portlandite was due to the pozzolanic reaction occurring, when Z0, Z1 or Z2 was incorporated and the formation of smaller amount of hydration products because of lower concentration of Portland cement in the system.

Table 3

The quantity of portlandite in hardened cement paste after 28 days.

The type of admixture	Amount of admixture in samples of pure OPC, %	Data according to the TG method	
		Mass losses at 400–480 °C, %	W (Ca(OH) ₂), %
Ref		3.07	12.62
Z0	5	2.89	11.88
Z0	10	2.75	11.31
Z0	20	2.39	9.82
Z1	5	2.62	10.77
Z1	10	2.51	10.32
Z1	20	2.50	10.28
Z2	5	2.38	9.78
Z2	10	2.02	8.30
Z2	20	1.74	7.15

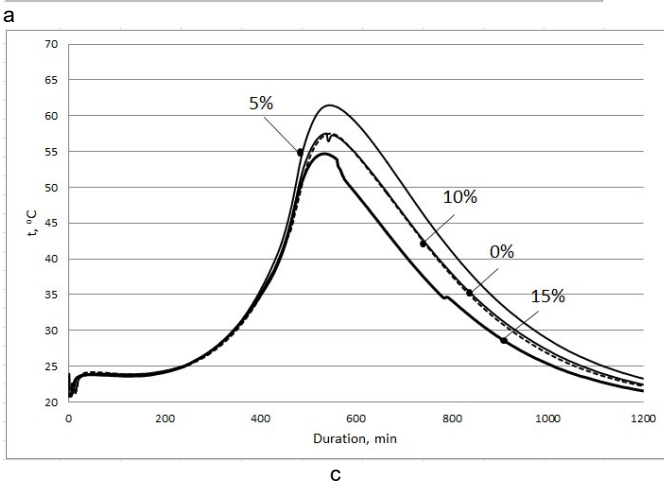
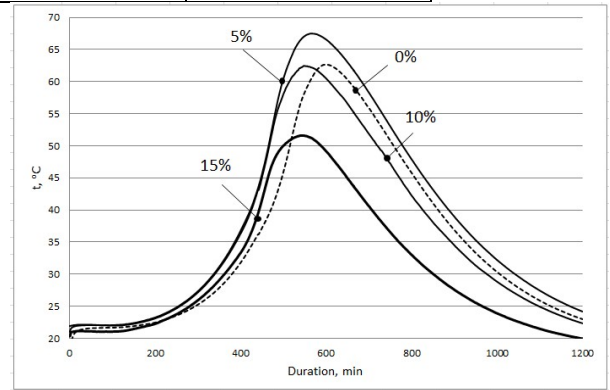
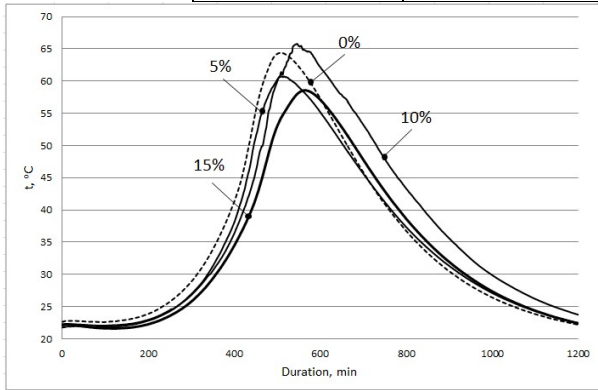


Fig.7 - Hydration temperature kinetic curves of cement paste with 5; 10 and 20% Z0 (a), Z1 (b) Z2 (c) admixtures.

After the X-ray diffraction and thermal analysis, the hydration temperature of cement paste was measured (Fig. 7). It was determined that the main peak is higher in the specimen with 10% of Z0 admixture (Fig. 7, a.). This indicates that admixture Z0 slightly delays the hydration of hardened cement paste. It could be explained by the lowest surface area, as the surface of particles

should act as nucleation sites for hydrates.

By using admixture Z1, the main reaction of hydration in all cases was shortened. Acceleration period decreases from 578 min till 546 min comparing to the control specimen (Fig. 7, b). Investigating specimens with Z2 supplementary material, hydration duration was very similar to the control specimen (Fig. 7, c)

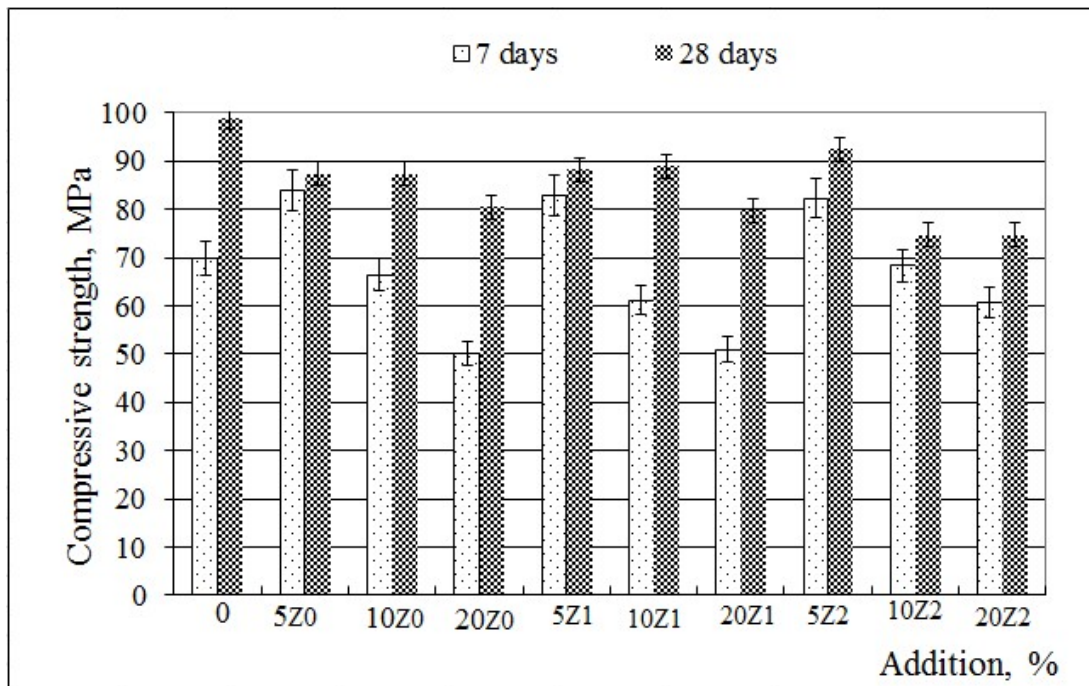


Fig.8 - The dependence of hardened cement pastes compressive strength on the type and quantity of admixture after 7 and 28 days of hydration.

In all investigated cases, by using 5 % Z1 and Z2 or by using 10 % of Z0, cement paste hydration temperature increased compared with the reference specimen temperature. These temperature increases can be explained by filler effect. The physical presence of particles enhanced the hydration of the clinker phases. This effect is called the filler effect. This effect has been attributed to the increase of the number of nucleation sites provided by the extra surface from the particles [30, 31]. After 7 days when 5% of Portland cement was replaced by Z0, Z1 or Z2, the compressive strength of specimens was higher. Higher than 5% of admixture content reduced the compressive strength compared to the control specimen (Fig. 8). The decrease could be explained by the binder dilution and, thus, by reducing the amount of Ca^{2+} resulting from Portland cement hydration, necessary for hydrosilicates and hydroaluminates formation. These results are similar to those obtained from blended cement pastes prepared with natural or synthetic zeolite admixtures [7]. When zeolite admixture is present in the cement and water mixture, high pH of the zeolite causes disintegration and depolymerization of the aluminosilicate framework. In depolymerization phase $[\text{SiO}(\text{OH})_3]^-$ and $[\text{Al}(\text{OH})_4]^-$ ions enter the solution and react with Ca^{2+} ions thus forming hydrated calcium silicates and calcium aluminates, very similar to those compounds, which develop during the hydration of ordinary cement.

The compressive strength (after 28 days) test showed that, although the best result is obtained when 5% of the cement is replaced by the supplementary cementitious material Z2, its

strength was 6.6% lower than in the control specimen; the compressive strength was obtained 10.9% and 11.7% lower in the other cases, than the strength of the control specimen (Fig. 8). Consequently, this insignificant decrease in strength could be due to the dilution effect.

Based on the experimental results, it can be concluded that zeolitized glass powder is a pozzolanic material. Glass powder zeolitization is useful to be used in specialized cement products for specific applications[32, 33]. In the other cases, zeolitization of waste glass is not appropriate, because the specimens with ground glass admixture after 28 days of hydration gained similar or higher compressive strength compared to specimens containing zeolitized ground waste glass admixture.

4. Conclusions

1. It was determined that it is appropriate to zeolitize ground glass powder according to Z2 methodology (by using aqueous solution of sodium aluminate instead of $\text{Al}(\text{OH})_3$ and NaOH aqueous solution) and it was the best method among the three investigated methods. In this case, sodium aluminosilicate hydrates and calcium aluminosilicate hydrates were formed in zeolitization product. Their specific gravity decreased by about 3.5%, the specific surface area increased by about 32% and the pozzolanic activity increased almost twice ($0.392 \text{ Ca}(\text{OH})_2 \text{ g/g}$ of glass) comparing to ground glass powder(Z0).

2. The compressive strength test of hardened cement paste specimens after 28 days showed that using the admixtures in all cases the

specimen strength was lower than of the control specimen (without waste glass addition). The best results were obtained in specimens where 5% of cement was replaced with Z2 admixture; in this case, the compressive strength decreased by 7% compared to the control specimen. This result suggested that a higher specific surface of glass admixture could cause a greater pozzolanic reaction and also the fine grains of glass fills the hardened cement paste voids, thus producing a compressive strength higher than that obtained using coarser glass admixture.

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