

Article

Glass Cullet as Additive to New Sustainable Composites Based on Alumina Binder

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Abstract: The article investigated the possibility of reusing heat resistant glass cullet to improve the mechanical properties of high-temperature composites. This is an excellent recycled aggregate that may be used as a substitute for alumina cement, and for fine natural aggregate in the production of concrete based on hydraulic binder. The experimental programme comprised of strength testing conducted on 40 × 40 × 160 mm cuboidal samples. The model mixture was modified by filler that comprised glass recycle, amounting to 5%, 10%, and 15% of the mass of gravel and cement. Given the degree of glass grounding, use was made of two fractions, 0/4 and 0/0.125 mm. Six modified mixtures were produced. Tests were then carried out on their selected physical and mechanical properties as well as the impact of temperature, topography, and chemical composition exerted on the composite. Next, the progress and development of compressive strength and flexural strength after 14 and 28 days of curing were studied. Results showed that concrete with a 5% content of glass dust had a maximum compressive strength at the level of 85.1 MPa. Results also showed that concrete (Zk.I.5) heated at a temperature of 500 °C had a 46% higher compressive strength when compared to basic concrete (Z.I.0). The results show that it is possible to use the described components to obtain a composite that meets requirements imposed on structural materials used in construction engineering.

Keywords: recycling; glass cullet; waste glass powder; recycled aggregates; alumina cement; cement composite; mechanical properties; microstructure



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1. Introduction

Glass has been used for many years and in various fields, including optics, the construction industry, and transport. Glass waste (among others packaging items, i.e., broken or damaged dishes, glasses, or crystal products) form 7–10% of all waste deposited in landfills. Glass recycling helps limit the usage of sand, limestone, soda, and water (by up to 50%) and energy (up to 30%). This in turn reduces emissions released into the atmosphere. In Poland, approximately 30% of glass packaging items are re-melted to obtain cullet. For comparison, elsewhere in the world, in such a way, on average 80–90% jars and bottles are processed. Every tonne of recycled glass cullet conserves 800 kg of sand, 250 kg of soda ash, and 180 kg of limestone dust. Glass recycling contributes to reducing the volume of landfills [1,2].

Glass waste totalled 5% of global solid municipal waste generated in 2016 [3]. In that some year, the glass recycling coefficient ranged from 9% in Turkey to 98% in Slovenia and Belgium. In Europe, in 2017, this index amounted to 71.48% [4].

Glass is produced of glass sand (source of silica SiO₂), borax (source of glass former B₂O₃), sodium and potassium feldspar (source of Al₂O₃), sodium carbonate, limestone, etc. Glass used in the construction industry contains approximately 70–72% SiO₂, 15% Na₂O, 10% CaO, and as MgO, Al₂O₃, along with ingredients that enhance glass properties or the

production process. One of the operations in the glass production process is melting of the set at a temperature of 1400–1500 °C; the molten mass is then clarified and optimised [5]. On the one hand, glass is an extremely brittle material, but on the other hand, it is equally durable and may survive thousands of years intact. It does not lose its properties even if it is repeatedly processed. Given its unique properties, it is a highly ecological and economic raw material that is wholly suitable for processing. Although recycling of cullet is a serious and complex undertaking, it deserves attention. Various researchers are inspired to seek innovative applications, which need to be modern, fulfil performance requirements, and contribute toward environmental protection. The recyclate needs to be free from contamination that could potentially pollute the final product. This makes selective collection of glass waste so important.

Numerous studies [6–18] have proven the suitability of using recycled materials in the production process of ceramics, binders, mortar, concrete, plaster, bituminous materials, etc. In the testing, use is being made of various types of cullet, including car glazing [19], soda-lime glass from containers [20–22], glass from technological lines of the pharmaceutical industry [23–25], cathode ray tubes for TV sets and computer screens [26,27], float glass from window glazing [28], glass from fluorescent lamps [29], glass from heat resistant dishes [30–32] and mirrors, safety glass, crystal glass, and colourless, green, and brown packaging glass [33–37]. Studies are underway in regards to the usage of concrete rubble [38] for the production of “green concrete”.

Waste cullet deposited in landfills does not become decomposed [39,40]; it is a perfect secondary raw material, resistant to mould and humidity. Crushed recyclate may be used in concrete as a substitute for cement, sand, and coarse aggregate. Purified and crushed recyclate are used in several different fields. In the construction industry, it is considered a perfect addition to façade plaster or a semi-product in building mortars (after processing to glass powder). It is also used for the production of foam glass, ceramic mass, or glass fibres. It offers a perfect alternative for natural soils, or natural aggregate used for the production of cement composites. It is also used with success as a binder for the stabilisation of embankments and road subgrades [41,42]. After crushing to powder form (dust fraction), waste glass may be used in the production of decorative tiles and catalyser carriers. Cullet may also be used to produce microbeads for producing reflective paints. The powder phase of crushed glass may be used in concrete as Pozzolan material [43].

Managing glass waste is an action that arises from the currently-binding Directive of the Council of Europe 91/689/EEC, which is directly aimed at protecting the environment. When designing cement composite, cullet may be added to concrete in the form of fibre, powder, or grain. The addition of glass consisting of fibres [17,44–51] enhances the parameters of concrete. Cement composites achieve better strength and become resistant to environmental factors (humidity, solar radiation, thawing, and freeing). If cement is replaced with crushed cullet (to dust fraction) [35–37], the obtained concrete offers better workability, increasing the compressive and tensile strength after an extended curing period. If the cullet is to be used as a substitute for fine aggregate (sand) [33,34,52], it is necessary to take into consideration the type of glass used. If a 10–30% addition of cullet is made, a decrease in strength takes place, equalling to 10–25% as compared to the executed control trial [53]. Scientists also point to the possibility of introducing recyclate to concrete mixture as a substitute for coarse aggregate [54–58]. From an economic viewpoint, this solution seems advantageous. It does not require a large work input, as in the case of fibres or crushing to powder form.

An extensive review of the literature shows that diverse types of waste are used as substitutes of aggregate in the production of concrete [59,60]. To date, no analyses have been carried out in regards to the recycling of used heat resistant dishes in concrete with alumina concrete. The paper presents the possibility of using heat resistant glass cullet, obtained from crushed glass dishes, for the production of concrete. This emphasises the innovative approach to the issue and the interdisciplinary nature of work. An analysis

was performed on selected properties of concrete, particularly the impact of recycle on physical, mechanical, and structural properties.

The study is a continuation of works commenced in articles [30–32], related to the re-use of glass waste. Given the obtained strength parameters and taking into account the necessity of waste management, glass recycling is a feasible solution in the production of mortar and concrete. If results of those studies are deployed in market operations, this may contribute to reduce the amount of waste deposited in landfills.

2. Materials

2.1. Concrete Mixture

Composite was generated from mixing alumina binder with filler consisting of natural and recycled aggregate and water. Heat resistant glass cullet was used as a substitute of traditional aggregate and cement (variable parameter). Control mixture (Z.I.0) was produced along with 6 series modified by glass recycle. The composition of concrete mixture is presented in Tables 1 and 2.

Table 1. Weight and volume composition of the Zk.I mixture, kg/m³.

Component	Z.I.0	Zk.I.5	Zk.I.10	Zk.I.15
Alumina cement	488	488	488	488
Sand 0/2 mm	562	562	562	562
Gravel 2/8 mm	833	791.35	749.7	708.05
Glass aggregate 0/4 mm	-	42	83	125
Glass powder 0/0.125 mm	-	-	-	-
Water	196	196	196	196
w/c ¹	0.40	0.40	0.40	0.40

¹ Water–cement coefficient, ratio of effective water mass to the contents of cement mass in concrete mixture (–).

Table 2. Weight and volume composition of mixture Zc.I, kg/m³.

Component	Z.I.0	Zc.I.5	Zc.I.10	Zc.I.15
Alumina cement	488	463.6	439.2	414.8
Sand 0/2 mm	562	562	562	562
Gravel 2/8 mm	833	833	833	833
Glass aggregate 0/4 mm	-	-	-	-
Glass powder 0/0.125 mm	-	24.4	48.8	73.2
Water	196	196	196	196
w/c ¹	0.40	0.42	0.45	0.47

¹ Water–cement coefficient, ratio of effective water mass to the contents of cement mass in concrete mixture (–).

Cullet was added to the concrete mixture, in amounts equalling to 5%, 10%, and 15% of the coarse aggregate mass, limiting the amount of gravel (Zk.I.5–Zk.I.10–Zk.I.15), and in amounts equalling to 5%, 10%, and 15% of the cement mass, limiting the used amount of cement (Zc.I.5–Zc.I.10–Zc.I.15). An assumption was made of a constant ratio of the w/c coefficient at the level of 0.40 for mixtures modified by glass aggregate 0/4 mm. This proportion was used to maximally limit the contents of capillary pores in the structure of the cement matrix [61]. Mixtures modified by glass powder were characterised by different w/c ratio, amounting to 0.42, 0.45, and 0.47. A low water–cement ratio allows obtaining better strength and tightness and, consequently, long-term durability. In her study, Kucharska [62] assumed the w/c coefficient as a basic criterion by which concrete may be broken down into a common type and a fully-valuable one.

2.2. Cement

The concrete mixture was produced with the use of GÓRKAL 70 high-alumina cement (Górka Cement Sp. z o.o., Trzebinia, Poland). The declared performance properties of cement are outlined in Tables 3–5.

Table 3. Chemical composition of alumina cement, %.

Al_2O_3 ¹	CaO ¹	SiO_2 ¹	Fe_2O_3 ¹	$\text{Na}_2\text{O} + \text{K}_2\text{O}$ ¹
69–71	28–30	<0.5	<0.3	<0.5

textsupscrip1 The contents were determined by the XRF or classical method.

Table 4. Mechanical and hydraulic properties of alumina cement ¹.

Bending Strength after 24 h, MPa	Compressive Strength after 24 h, MPa	Commencement of Binding Time, min	End of Binding Time, min
>5	<30	>160	<240

¹ Composition of the mixture: French sand (1350 g), cement (450 g), and water (225 g).

Table 5. Special properties of alumina cement.

Specific Surface Area according to Blaine, cm^2/g	Typical Ordinary Flame Resistance, sP	Density, g/cm^3	Bulk Density, g/cm^3
4000–4500	≥ 158	3.0	1.1

Alumina cement is characterised by high contents of Al_2O_3 and a stable phase composition with a domination of the crystalline phase. The basic phase consists of the calcium monoaluminate CA, which is accompanied by dicalcium aluminate (grossite) CA_2 , with accompanying phases being C_{12}A_7 , αA . Concrete produced on the basis of alumina cement has many applications due to its physicochemical properties. Depending on the composition—its strength (at low and high temperatures) and colour change. White alumina cement is characterised by considerable purity and very good flame resistance; it may be used at a temperature of 1560 °C and gain high compressive resistance after a short time. The phase contents (% by mass of cement) of CA_2 in the cements rose from 30% at 1350 °C to 60% at 1450 °C, whilst the ‘free’ Al_2O_3 content correspondingly declined from 20% to 5% [63]. In white aluminium cement, high temperature resistant CA_6 is a hexagonal phase often occurring with $\alpha\text{-Al}_2\text{O}_3$ at a high temperature. CA_6 is, presently, not hydraulic at ordinary temperatures, and normally only hydrates to any appreciable extent under hydrothermal conditions far above 100 °C.

Due to the significantly higher hydration heat than for Portland cement, it can be used at a temperature of -10 °C.

In addition to temperature extremes, it is sulphate-resistant, because of the lack of CH when hydration arises. The reason for the high resistance to sulphates, as well as sea water and chlorides, is not clear. George [64] reports that this is likely due to the very low permeability of the alumina cement slurries.

Alumina cement with 70–80% Al_2O_3 content is also used for decorative purposes because it contains small, negligible amounts of ferrite C_4AF ; they do not interfere with the whiteness of these cements where they are desired for aesthetic reasons.

2.3. Aggregate

The composite was prepared using a mixture of natural and recycled aggregate. The used gravel had a grading fraction of 2/8 mm (Zakład Produkcji Kruszyw Szumno Sp. J., Szumowo, Poland), ordinary sand from the Vistula with a fraction of 0/2 mm (PolBot Kruszywa S.A., Warsaw, Poland), and glass recycle (Termisil S.A. Glassworks, Wołomin, Poland) having varied grinding levels. The glass was crushed in a crusher and then ground in a ball grinder to obtain fine aggregate with a grading of 0/4 mm, and glass powder

with a grading of 0/0.125 mm. When selecting secondary raw material in the first place, economic and physical reasons were taken into consideration. Properties of cullet obtained from damaged glass dishes are presented in Table 6.

Table 6. Properties of glass obtained from TERMISIL S.A. glassworks.

Component	Requirements
Transformation temperature, °C	535
Temperature of dilatometric softening, °C	635
Lower annealing temperature, °C	520
Upper annealing temperature, °C	550
Permissible working range, °C	−40 ÷ 300
Density at 20 °C, g/cm ³	2.23
Hydrolytic stability of glass grains at the temperature of 98 °C	HGB 1
Average coefficient of linear thermal expansion (30 °C; 300 °C), K ^{−1}	3.55×10^{-6}
SiO ₂ , %	80
Na ₂ O, %	4
K ₂ O, %	1
B ₂ O ₃ , %	13
Al ₂ O ₃ , %	2

Obtained and crushed aggregate from glass waste is presented in Figure 1.

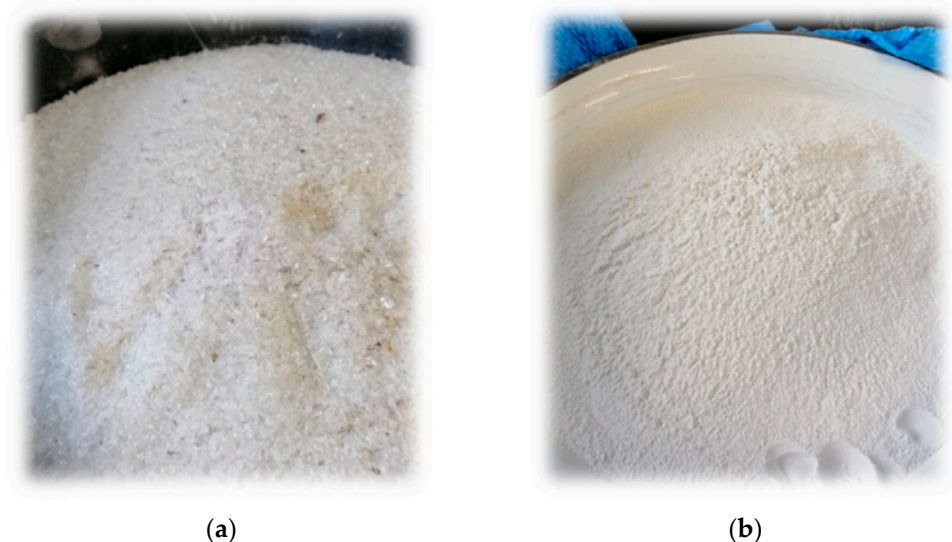


Figure 1. Recycling cullet aggregate: (a) fine aggregate with a fraction of 0/4 mm; (b) glass powder with a fraction of 0/0.125 mm.

2.4. Water

Make-up water for the production of the planned mixture may be obtained directly from the water supply line.

3. Methods

The main objective of the study was to evaluate the feasibility of reusing heat-resistant cullet obtained from broken glass dishes for the production of unconventional concrete types. Pilot testing comprises strength tests, a semi-quantitative analysis of the chemical composition aimed at obtaining a distribution map of elements, and an analysis of topography and phase composition of the planned composite. Moreover, a spot chemical analysis was performed of the material. The impact of high temperature on properties of the planned concrete was evaluated. The cullet was separated into fractions containing

particles of different sizes. The recyclate was sieved through a set of sieves and the grading was then tested.

3.1. Grading Analysis

To determine the size and dimensions of particles of glass recyclate, a grading composition test was carried out using the sieve method, in line with guidelines contained in the standard EN 933-1:2012. The recyclate was sieved and catalogued in a mechanical way to divide it into particular fractions with given diameters of the tested particles. After weighing of subsequent grading classes remaining on the given sieve, percentages of particular fractions in the tested sample were determined in relation to the whole. A grading curve was devised that presented the type of aggregate and its specific properties.

3.2. Strength of Hardened Concrete

Properties of hardened composite were determined on the basis of conducted strength tests. The tensile strength in a three-point bending scheme f_{cf} was determined on cuboidal concrete bars with the dimensions of $40 \times 40 \times 160$ mm. For needs of the tests, the samples were set-up with their concreted surfaces perpendicular to the direction of the applied load. Then, the halves of the samples obtained after the flexural strength test were used to determine the compressive strength f_c [65]. The tests were carried out after the lapse of 14- and 28-day curing periods, according to the standard PN-EN 12390-5:2011 in the Advantest 9 hydraulic press (Controls, Warsaw, Poland).

3.3. Tests of Concrete at Elevated Temperature

The $40 \times 40 \times 160$ mm beams were dried in a laboratory drier KC-100/200 (Zalmed, Warsaw, Polska) until constant mass was achieved, at a temperature of 105 ± 5 °C. Four sets of samples were taken for the research. The samples were heated at temperatures of 200 °C, 400 °C, 600 °C, and 800 °C in a special furnace—PK 1100/5 (Thermolab S.C., Warsaw, Poland) furnished with powered heating compartments. The thermal process was programmed with the use of dedicated ThermoPro software. The temperature in the furnace remained for 30 min until completion of the heating process, which was monitored using a NiCr-Ni thermocouple attached to the control sample. The temperature distribution was illustrated by the standard curve “temperature-time”. Each set was baked at a different temperature, then cooled and tested separately. The results were compared with reference samples. All heated elements were subjected to measurements of tensile strength in a trial of bending and compressive strength.

3.4. Studying Morphology and Elemental Composition in SEM and EDS

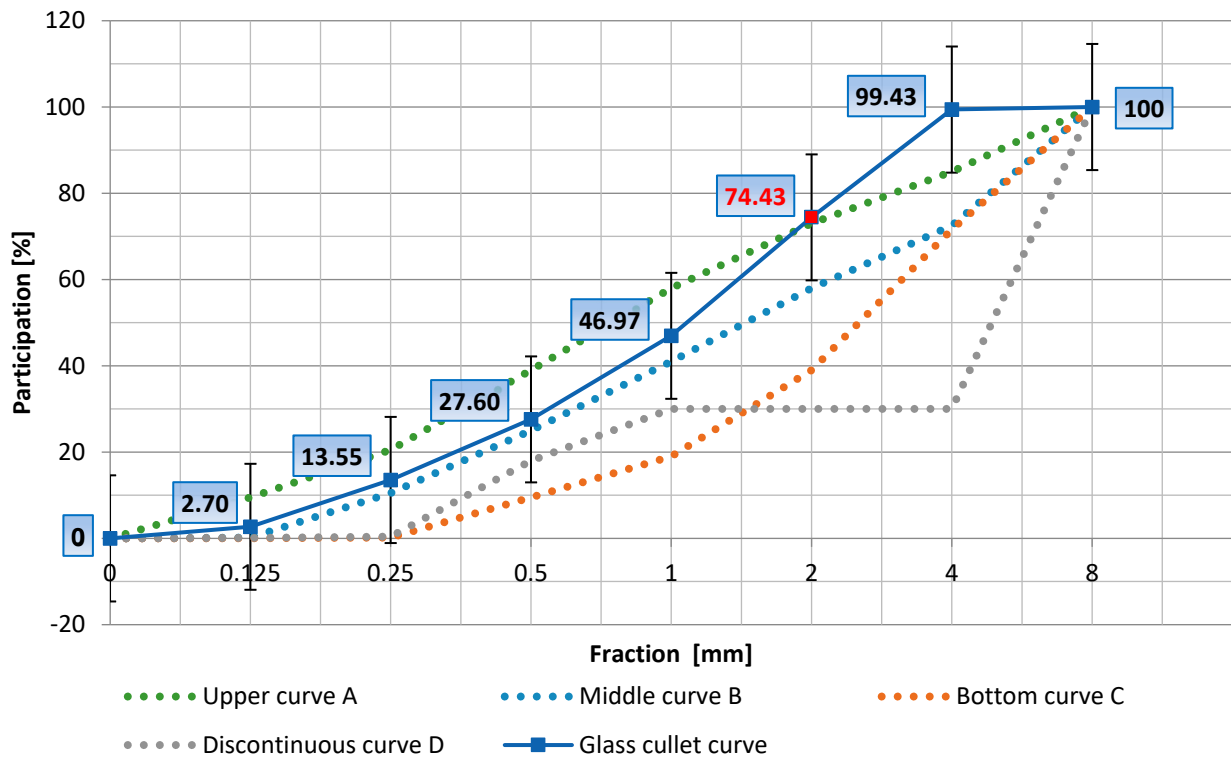
Material tests of the execution and of the top layer of the composite, including primarily morphology and the elemental composition, the scanning electron microscope (SEM) type FEG Quanta 250 was used (FEI, Hillsboro, OR, USA) with an attachment for X-ray microanalysis in the EDS mode (EDAX, Mahwah, NJ, USA). The research material was obtained from fragments of concrete bars remaining from strength tests. Samples for SEM tests were powder painted and fixed to the grip with the use of a carbon binder. The samples were coated with a thin carbon layer (approximately 50 µm) as a result of cathode spraying.

4. Results and Discussion

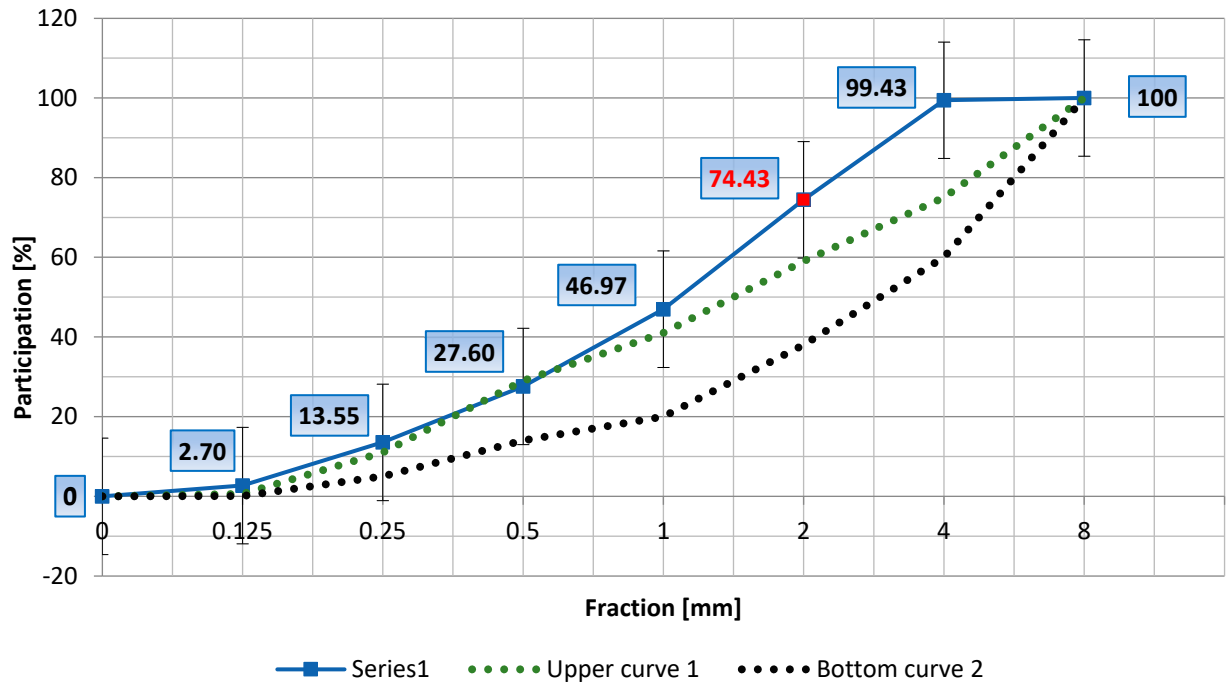
4.1. Sieve Analysis of the Waste Cullet

For comparison, results of the granulometric analysis were applied onto a semi-logarithmic grid, obtaining a sieving curve (Figure 2). When determining the composition of mixtures, aggregate and aggregate composition, attempts were made to select them, ensuring that they were contained between grading curves and that they had the smallest voids between particles. Recommendations contained in relevant applicable standards

were taken into consideration in this respect. Pursuant to the curve obtained from testing, a calculation was made of diversity and curvature indices.



(a)



(b)

Figure 2. Grading curve of used recyclate as compared to the standard good grading field: (a) DIN 1045-2; (b) PN-B 06250.

The cullet curve (Figure 2b) was not contained within the area of the limiting sifting

curves (upper and lower ones). The recycle failed to meet standard requirements of particularly good grading. The recycle curve was over the upper limiting curve. The ground cullet was too coarse. However, that does not discriminate recycle as an additive to concrete. Glass aggregate appeared much better after its application onto the grid of standard curves (Figure 2a). The area between the curves was determined as a good grading zone. Limiting curves contained in the standard DIN 1045-2:2014 made it possible to allow for the majority of concrete types, while limiting curves determined by the standard PN-B 06250 were characterised by a more extensive range of application.

To allow the delimiting of properties of aggregate composition, the sand content (PP) of glass cullet was determined, which amounted to 74.43%, and this conformed to suggestions that may be found in literature sources [66] for a mixture with particles not larger than 8 mm. The sand content was of considerable importance, assuring the required workability to the mixture. Aggregate having a sand content equal to 45–60% is suitable for application in vibro-pressable mixtures with a moist or dense plastic consistency, such as paving setts, in elements with no reinforcing, or with sparse reinforcing, which have a simple and uncomplicated shape of the section [8].

The obtained summation curves for particular types of aggregate were used to determine substitute diameters of grain (reliable diameter d_{10} , diameter d_{30} , and average diameter d_{60}), which constituted 10%, 30%, and 60%, respectively of the aggregate mass. Two parameters were calculated to characterise grading: uniformity coefficient (degree of uniformity) C_U and the coefficient of grading curvature C_C . Definitions of the parameters are described by Formulas (1) and (2). The above mentioned values are presented in Table 7.

$$C_U = \frac{d_{60}}{d_{10}}, \quad (1)$$

$$C_C = \frac{d_{30}^2}{d_{60} \cdot d_{10}}. \quad (2)$$

Table 7. Grading indices of glass cullet.

d_{10} , mm	d_{30} , mm	d_{60} , mm	C_U	C_C
0.21	0.56	1.37	6.53	1.11

Indicator C_U is a measure of inclination of the sieving curve. The uniformity coefficient for glass aggregate is 6.53, while the curvature coefficient is 1.11. As per the classifications available in the literature sources, the studied recycles are with several fractions [67], well-graded [68–71] and fine fractions that fill out the pores between larger particles.

4.2. Analysis of Results Obtained from Strength Tests

Laboratory strength testing was performed on cuboidal concrete bars with dimensions of $40 \times 40 \times 160$ mm. Changes of sample strength were executed by variations of the recipe of concrete mixtures. The samples were produced in accordance with guidelines contained in the standard PN-EN 206-1:2003. Laboratory tests were conducted at the temperature of 20 °C after 14 and 28 days from demoulding of the samples. The composite was also tested after heating to temperatures of 200 °C, 500 °C, and 800 °C after 28 days of curing.

The results of the conducted tests, including the statistical processing, which comprised the determination of average sizes, standard deviations, and variability indices, are shown in Tables 8–12.

Table 8. Basic statistical parameters of concrete flexural strength.

Concrete	Flexural Strength after 14 Days, MPa	Standard Deviation, MPa	Variability Index, %	Flexural Strength after 28 Days, MPa	Standard Deviation, MPa	Variability Index, %
Z.I.0	11.9	0.2	1.65	11.9	0.6	5.31
Zk.I.5	10.5	0.9	8.48	11.2	0.3	2.85
Zk.I.10	11.2	1.3	11.07	11.0	1.0	9.28
Zk.I.15	11.8	0.8	6.72	10.7	0.5	4.58
Zc.I.5	12.0	0.7	5.81	11.7	0.3	2.73
Zc.I.10	11.5	0.2	1.48	11.6	0.7	6.23
Zc.I.15	11.1	0.5	4.42	10.7	1.3	12.06

Table 9. Basic statistical data of compressive strength of concrete.

Concrete	Compressive Strength after 14 Days, MPa	Standard Deviation, MPa	Variability Index, %	Compressive Strength after 28 Days, MPa	Standard Deviation, MPa	Variability Index, %
Z.I.0	68.1	2.6	3.75	76.9	4.1	5.33
Zk.I.5	64.4	2.9	4.46	74.4	5.8	7.82
Zk.I.10	58.6	2.6	4.47	77.2	3.5	4.59
Zk.I.15	62.8	2.2	3.50	78.4	4.3	5.43
Zc.I.5	63.6	3.0	4.75	85.1	3.3	3.82
Zc.I.10	57.1	8.4	14.67	79.8	6.3	7.92
Zc.I.15	59.2	3.5	5.84	75.6	4.5	5.97

Table 10. Basic statistical characteristics of the composite after thermal shock—200 °C.

Concrete	Bending Strength, MPa	Standard Deviation, MPa	Variability Index, %	Compressive Strength, MPa	Standard Deviation, MPa	Variability Index, %
Z.I.0	7.1	0.8	11.13	60.5	1.8	2.88
Zk.I.5	7.3	0.2	2.70	61.7	2.8	4.56
Zk.I.10	7.7	1.3	17.33	60.0	2.6	4.30
Zk.I.15	6.4	0.9	14.26	48.8	3.0	6.13
Zc.I.5	7.4	0.4	5.32	51.8	2.3	4.34
Zc.I.10	7.3	0.8	10.51	54.8	3.1	5.61
Zc.I.15	6.7	0.3	4.67	50.2	1.4	2.69

Table 11. Basic statistical characteristics of the composite after thermal shock—500 °C.

Concrete	Bending Strength, MPa	Standard Deviation, MPa	Variability Index, %	Compressive strength, MPa	Standard Deviation, MPa	Variability Index, %
Z.I.0	1.4	0.2	10.04	39.8	6.5	16.25
Zk.I.5	3.3	1.0	30.29	58.1	6.3	10.76
Zk.I.10	2.6	0.8	30.30	46.6	4.0	8.60
Zk.I.15	1.7	0.2	8.94	47.2	6.6	14.03
Zc.I.5	2.3	0.7	29.45	38.7	8.0	16.66
Zc.I.10	2.2	0.8	35.25	38.2	5.6	14.72
Zc.I.15	2.7	0.4	15.84	48.4	3.7	7.55

Table 12. Basic statistical characteristics of the composite after thermal shock—800 °C.

Concrete	Bending Strength, MPa	Standard Deviation, MPa	Variability Index, %	Compressive Strength, MPa	Standard Deviation, MPa	Variability Index, %
Z.I.0	1.0	0.2	21.23	35.3	5.2	14.80
Zk.I.5	0.8	0.1	14.83	38.4	3.7	9.66
Zk.I.10	1.1	0.4	41.27	35.2	2.3	6.39
Zk.I.15	1.4	0.2	14.53	32.0	3.6	11.32
Zc.I.5	1.6	0.2	14.13	37.7	2.9	7.59
Zc.I.10	0.8	0.2	29.43	23.8	3.6	15.29
Zc.I.15	0.8	0.2	22.57	26.4	3.2	12.09

4.2.1. Flexural Strength

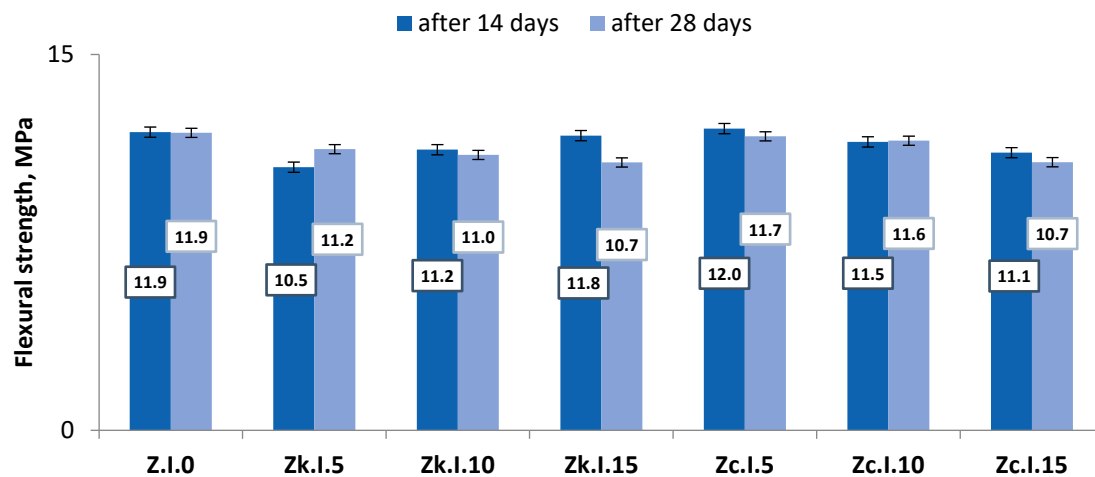
A cuboidal beam with a cross section of 40 × 40 was subjected to bending, growing loads, until the moment of its destruction. Consequently, the flexural strength was calculated according to Formula (3).

$$f_{cf} = \frac{3 \cdot F \cdot l}{2 \cdot d_1 \cdot d_2^2} \cdot 10, \quad (3)$$

where:

- f_{cf} —flexural strength (MPa);
- F—maximum load in bending test (kN);
- l—distribution of sample supports (cm);
- d_1, d_2 —dimensions of sample cross-section (cm).

Figure 3 presents results of strength tests in the triaxial bending scheme. The results of testing obtained for each series are the mean arithmetic values received from three measurements.

**Figure 3.** Flexural strength after 14 and 28 days of curing.

Destructive tests were performed at room temperature. Results showed that both samples, modified by aggregate and by glass powder, had a similar strength after 14 and 28 days of curing. The differences in values are insignificant and do not lower significantly strength parameters as compared to reference concrete. The obtained results may be considered satisfactory. The target strength recorded on control samples after 28 days of curing remained within the range of 10.7–11.7 MPa. The highest obtained strength was 11.7 MPa for Zc.I.5, and the lower one was 10.7 MPa for Zk.I.15 and Zc.I.15. The obtained strength values were lower by 0.2 and 1.2 MPa, respectively, compared to the reference concrete.

A slightly lower flexural strength was recorded for concrete with the addition of powder and for glass aggregate. The obtained strength-related results correlated with the Pozzolanic activity of the cullet. A slight impact was observed from the presence of cullet on the decrease of strength after 14 and 28 days of curing compared to the control sample.

The requirements for the tensile strength when bending curbs made of paving stones and concrete curbs were described in [72,73]. The results obtained in the test exceeded 10.5 MPa; therefore, they should be considered satisfactory. Similar results concerning the strength of glass–sand concrete were previously described by Du and Tan [74] and Góra and Franus [7].

Table 8 presents the average strength f_{cf} along with elementary statistical characteristics.

The variability index for the designed concrete after 14 and 28 days of curing was lower than 10% (the only exception were Zc.I.15—12.06%; Zk.I.10—11.07%). The quality of composite, determined after 14 and 28 days from demoulding, was considered good given concrete Zc.I.15 and Zk.I.10, for which the variability index was lower than 15%. The obtained parameters allowed the presumption that the designed concrete types were of a homogenous nature.

4.2.2. Compressive Strength

Results of compressive strength testing of concrete (Figure 4) along with their statistical processing, which comprised the determination of average values, standard deviations, and variability indicators, are presented in Table 9. Tests were carried out for six samples for particular periods of their curing. The testing was conducted at room temperature.

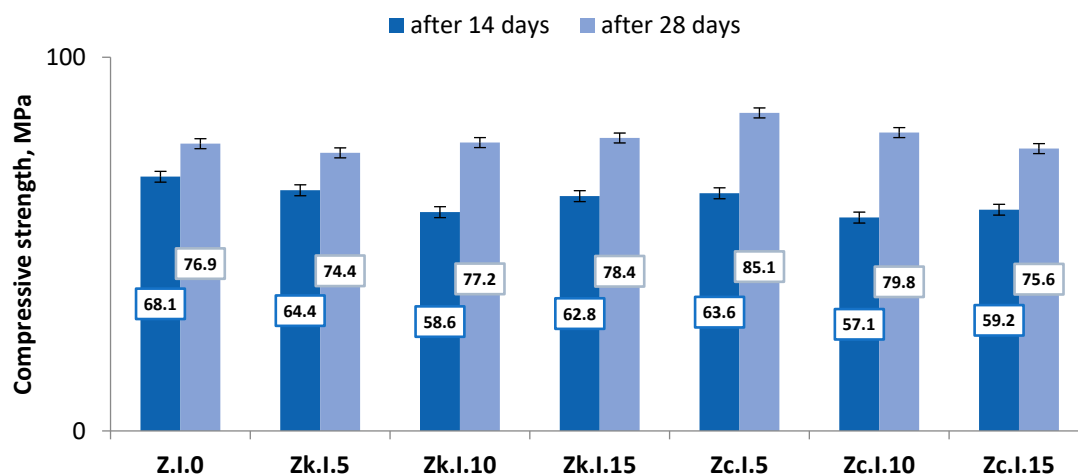


Figure 4. Average compressive strength after 14 and 28 days.

The compressive strength of concrete after 28 days of curing significantly improved thanks to the usage of recycle, especially when 5% of cement was replaced by glass powder (Zc.I.5) and 15% of gravel was replaced by glass aggregate (Zk.I.15). The maximum compressive strength was 85.1 MPa, 78.4 MPa, respectively, which showed an increase by 10.66%, 1.95%, as compared to the control sample. The lowest compressive strength was recorded for Zk.I.5 and Zc.I.15. When compared to the control sample, the concrete samples had a slight decrease in strength, by 3.25% and 1.69%. The usage of waste glass powder amounting to 5% of the substance increased strength by 8.2 MPa.

Results of the compressive tests of concrete after 28 days were found to have a low variability index, below 8%. This proves that this concrete offers very good quality. A downtrend was recorded of the value of the variability index with the increase in strength of concrete over time for Zc.I.5–Zc.I.10–Zc.I.15. On the other hand, in regards to concrete produced with glass aggregate Zk.I.5–Zk.I.10–Zk.I.15, the variability index tended to grow with an increase in strength over time. Figure 5 presents dependencies between the strength of concrete at flexural strength and compressive strength.

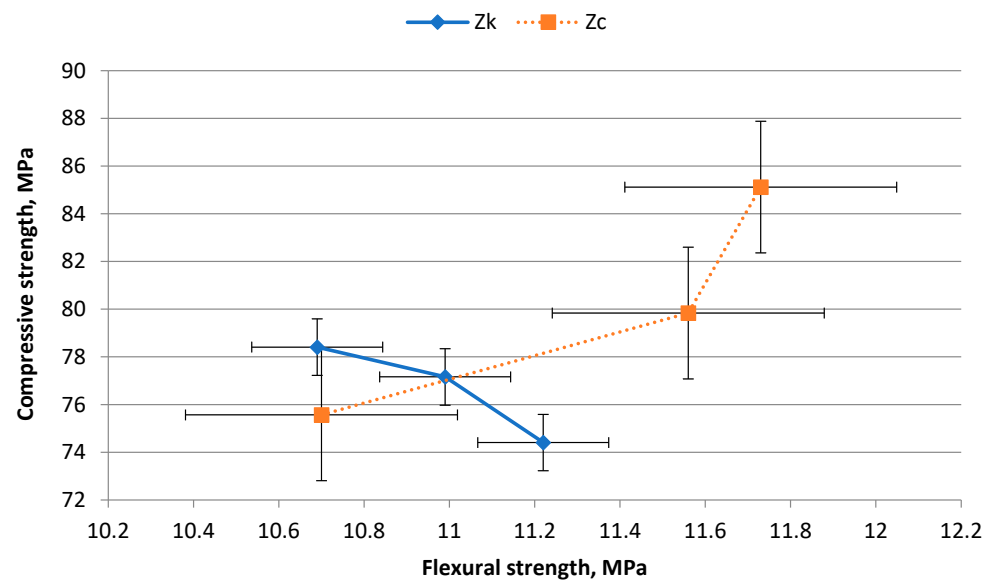


Figure 5. Dependence of flexural strength of concrete on compressive strength.

The continuous line depicts the dependence for the composite produced using recycle with a grading fraction of 0/4 mm. As the content of the recycle grows, flexural strength tends to grow, while compressive strength tends to decrease. On the other hand, the intermittent line defines the dependence of strength of composite modified by glass powder. Compressive strength and flexural strength grow with an increase of the content of the powder fraction in concrete.

4.2.3. Strength of Composite under High Temperature Load

Cuboidal samples were heated up at the temperatures of 200 °C, 500 °C, and 800 °C for 30 min and tested after their naturally cooling down to ambient temperature. Results of testing, of compressive and tensile strength for samples under the temperature load, are presented in Figures 6 and 7. Results obtained for each of the series are an arithmetic average, from three measurements for flexural strength and from six measurements for compressive strength; moreover, a comparison was made of the standard deviations of results from each series of samples. The basic statistical characteristics are presented in Tables 10–12 for particular periods of their curing.

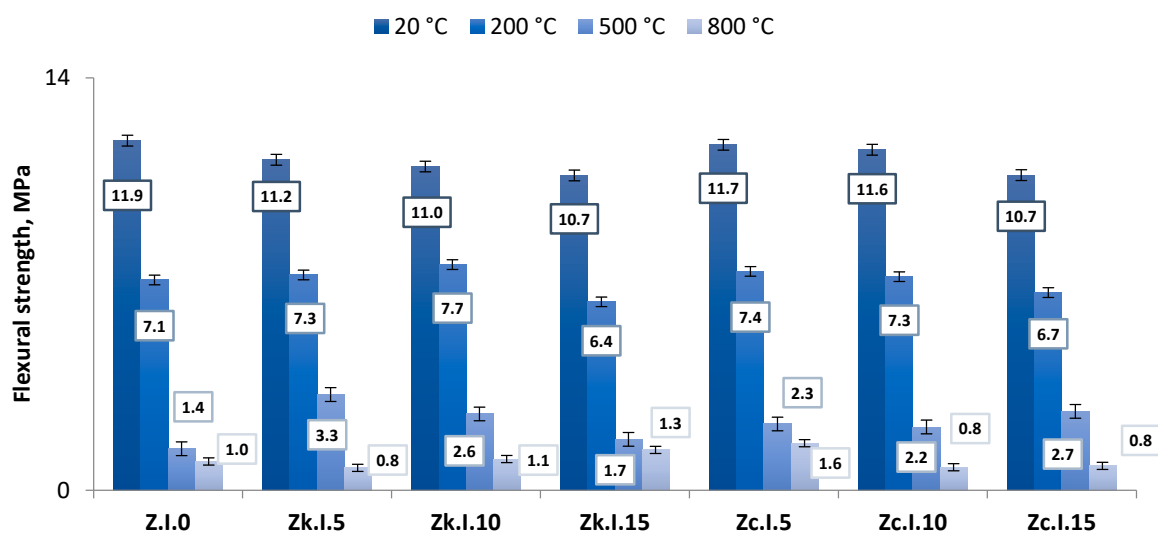


Figure 6. Flexural strength of samples under temperature load.

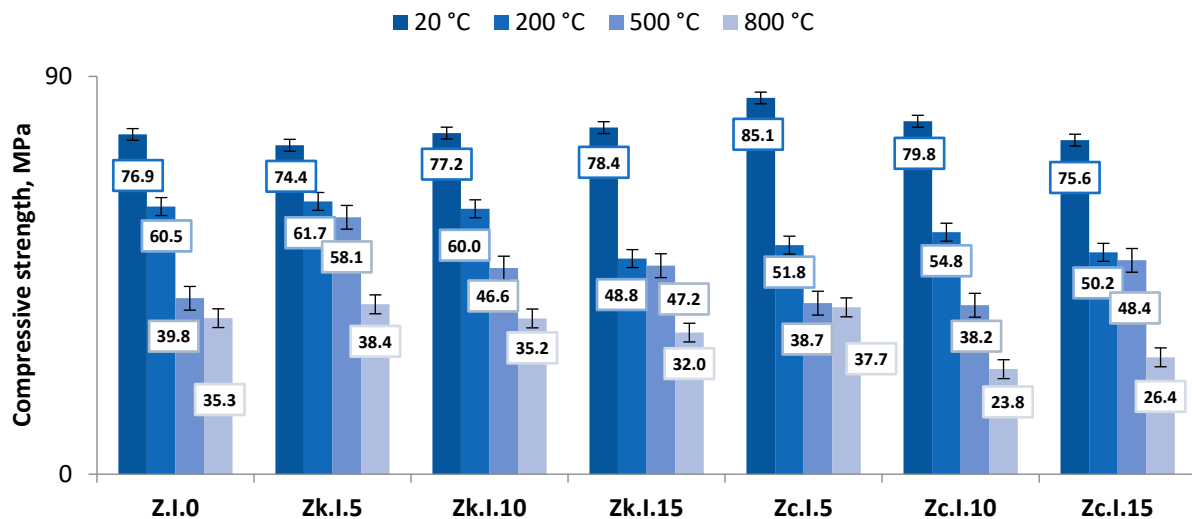


Figure 7. Compressive strength of samples under temperature load.

The above presented diagram shows that, after adding glass recycle, an improvement was recorded in the results of bending tensile strength, especially for concrete heated at a temperature of 500 °C. The peak value of strength for this temperature was recorded for concrete mix Zk.I.5, for which an increase was ascertained by 43.38% as compared to the reference concrete. The lowest strength value of 1.7 MPa was recorded for Zk.I.15—slightly higher than the strength of Z.I.0 concrete, by 22.70%. Concrete modified by a 15% addition of glass powder was characterised by a strength increase by 47.39%, as compared to reference concrete. All types of concrete heated up to 200 °C achieved a similar mean value of bending tensile strength.

Based on the obtained results, a positive impact was ascertained in regards to high temperatures on the compressive strength of concrete. Samples heated at 500 °C, after 28 days of the hardening period, reached the highest strength limit, equal to 58.1 MPa, for the Zk.I.5 mixture, showing an increase of 45.99%, as compared to the control sample. Strength tends to decrease with further replacement of glass aggregate. The lowest strength value was slightly higher than the control sample, by 17.16%, as shown in the above diagram. In regards to mixtures with the addition of glass powder, the highest strength level was recorded for Zc.I.15, which amounted to 48.4 MPa, and suggested an increase of 21.55%, as compared to the reference concrete.

The variability index for the designed concrete to be exposed to thermal shock after 28 days of curing was below 10% for 200 °C. The composite offered very good quality and had a homogenous nature. Concrete heated to the temperature of 500 °C and 800 °C still maintained good quality. The obtained results allowed the presumption that adequate component proportions in the mixture were retained. In addition, aggregates play an important role (due to chemical and physical stability) in concrete exposed to high temperatures, as aggregates usually occupy 60–80% of the volume of concrete.

Concretes made on the basis of alumina cement are used in structures where resistance to temperature is important (refractory concretes). Thanks to the use of ground glass recycle, it is possible to reduce the total cost of production of concretes resistant to high temperatures. The cost of producing alumina cement is about 4–5 times that of Portland cement. Due to the high cost, the consumption of alumina cement reaches approximately 1/1000 of the consumption of Portland cement [75].

4.3. Temperature Distribution in the Material

The tests attempted to assure that temperature distribution was similar to conditions prevailing during a real fire. Samples were heated according to the standard “temperature-time” curve until the furnace chamber temperature reached 200 °C, 500 °C, or 800 °C.

The concrete bars were heated at the pre-set temperature for a period of 30 min, and then cooled down for 24 h until ambient temperature was reached. Before heating, a measuring thermoelements (CH 8–CH 10) was fixed to the pilot samples (Figure 8), which enabled the determination of temperature distribution. Regulating thermoelement (RT) was introduced through the posterior wall and was situated in the upper part of the special furnace, PK 1100/5. The testing was conducted in accordance with the standard EN 1991-1-2:2005. The temperature distribution curves are presented in Figure 9. Afterwards, concrete bars were subjected to testing of flexural strength. The remaining sample halves were used to test compressive strength.



Figure 8. Mounting of thermal elements in the heating chamber before commencement of testing.

Temperature distribution allows ascertaining concrete behaviour in the case of a fire. Heating was applied to concrete bars from particular groups of designed mixtures designated for tests of flexural strength.

The nominal, standard curve time-temperature defines the course of temperature inside a premise in which combustion takes place, of primarily lignin and cellulose materials, such as furniture and paper, described by Formula (4):

$$\theta = 345 \cdot \log(7t + 1) + 20. \quad (4)$$

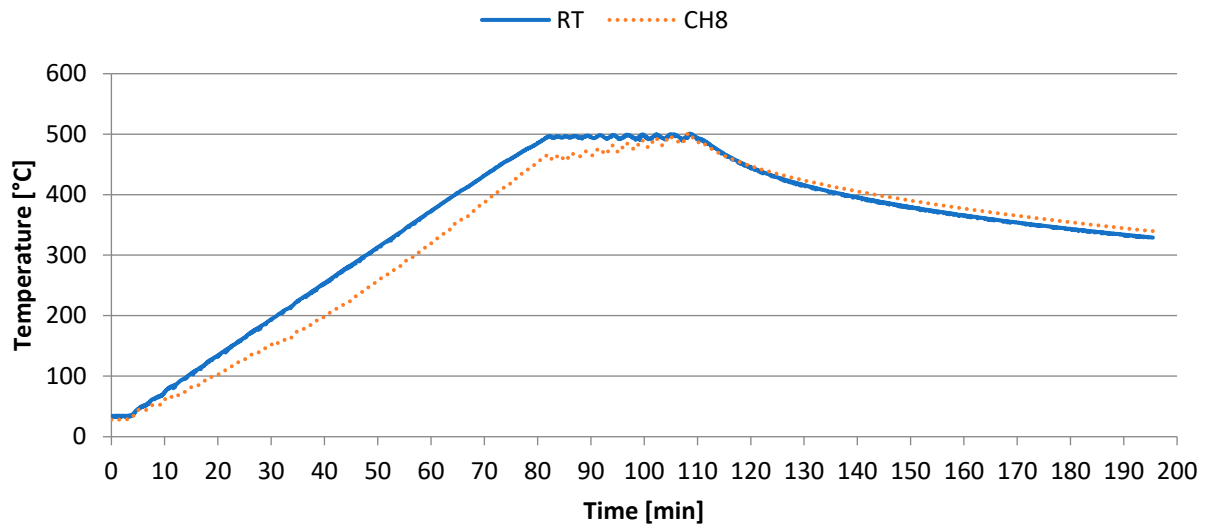


Figure 9. Distribution of temperature in the pilot sample Zc.I.10 heated up to 500 °C.

The accumulation of temperature during a standard fire depicts processes that take place in concrete during the heating process. Once the level of 100 °C is achieved, the dehydration process commences, i.e., dewatering of components of the cement slurry C-S-H. At a temperature of 350 °C, transformations in gravel commence, while it is 570 °C in silicate aggregate. The decay of calcium hydroxide $\text{Ca}(\text{OH})_2$ takes place between 400 °C and 600 °C. Silicate concrete is less resistant to the impact of fire, because at 573 °C, quartz transforms from type “a” to type “b”. The decay of calcium carbonate CaCO_3 takes place at 700 °C–800 °C. In regards to load bearing, concrete heated up to a temperature of 600 °C should be considered destroyed. Therefore, aggregate, due to its chemical and physical stability, plays an important role in concrete exposed to high temperatures, because 60–80% of its volume is made of aggregate concrete. In concrete elements, nonlinear temperature distributions occur, as well as stress from thermal deformations [76]. The samples were evenly heated over their entire volume—this may be proven by the point at which the temperatures become balanced. In the cooling down process, a temperature increase takes place in internal layers [31].

4.4. Analysis of Morphology and Topography of Composite Surface

The microstructure of concrete was tested with the use of a scanning microscope. The tests were carried out on the surface of cracking of samples, which were destroyed. The results are shown in Figure 10, which reflect the place of study. The samples were tested using the same procedure and the same magnification. The results pertained to analyses of the surface, from the given area, along the pre-set line and a spot analysis.

SEM images did not disclose the occurrence of any significant differences in the morphology and microstructure before and after addition of recycle to the cement matrix. Mechanical properties of the samples were modified as compared to the control sample. Scratches were ascertained on the surface of the analysed glass material. Data published in [77–79] allow the presumption that composite containing glass addition is less susceptible to deformation due to thermal expansion.

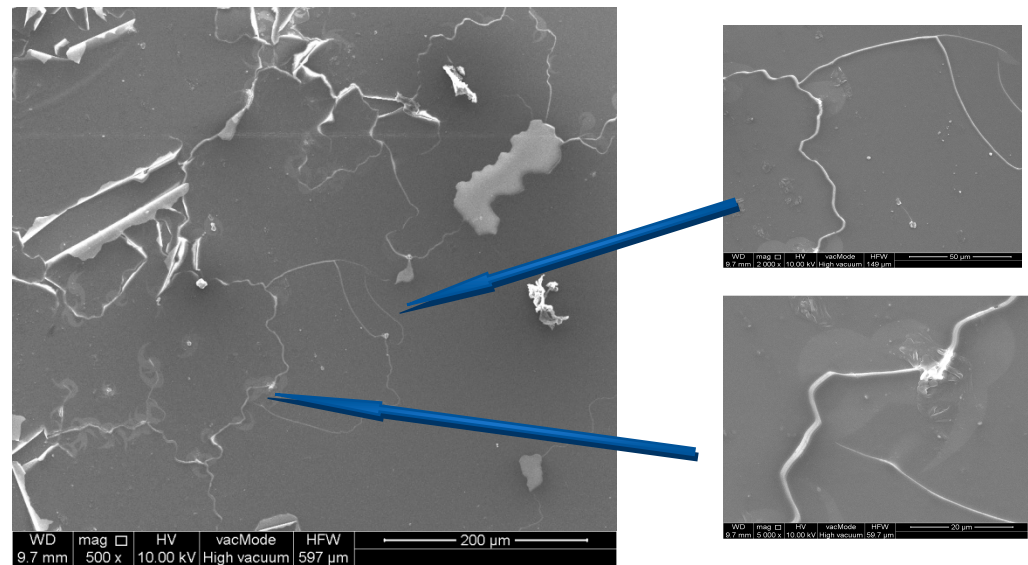


Figure 10. Topography of scratches in the glass material, 20 °C.

4.5. Elemental Analysis

Results of the elemental analysis are presented as digitally recorded spectra. This allows making analyses of particular spectra fragments beyond the research device. Identification of elements follows an analysis of all the recorded spectrum peaks. Next, calculations were carried out of the material being tested. This allowed obtaining information concerning the concentration of identified elements as percentage (%) by weight and % by atom. Next, the computational error was determined. The obtained results are specified in Table 13. It is also possible to calculate stoichiometry of the oxides (Table 14).

Table 13. Phase composition of a glass sample defined from point A, 20 °C.

Oxide Composition	Wt, %	Mol, %	K-Ratio	Excitation Efficiency Z	Likelihood of Absorption A	Secondary Fluorescence F
C ₂ O	30.11	39.95	0.0301	1.0285	0.1617	1.0005
Na ₂ O	2.00	1.71	0.0058	0.9466	0.4092	1.0036
Al ₂ O ₅	3.09	1.23	0.0084	0.9420	0.7053	1.0139
SiO ₂	64.34	56.85	0.2356	0.9696	0.8080	1.0001
K ₂ O	0.46	0.26	0.0032	0.9181	0.9164	1.0000
Total	100.00	100.00				

Table 14. Analysis of chemical composition of glass point A, 20 °C.

Chemical Compound	Net Intensities	Background Intensity	Intensity Error	Peak to Background Ratio P/B
CK	67.40	1.21	1.87	55.91
NaK	36.51	8.76	3.04	4.17
AlK	51.98	11.33	2.51	4.59
SiK	1318.39	11.26	0.42	117.09
KK	12.49	8.33	6.52	1.50

The analysis of the chemical composition was conducted using the EDS attachment. The percentage of particular oxides were determined from point A and B (Figure 11). The oxide composition of glass is characterised by high contents of SiO₂ and C₂O at the level of 64.34% and 30.11%, respectively. Spectra of the chemical composition are presented in Figure 12.

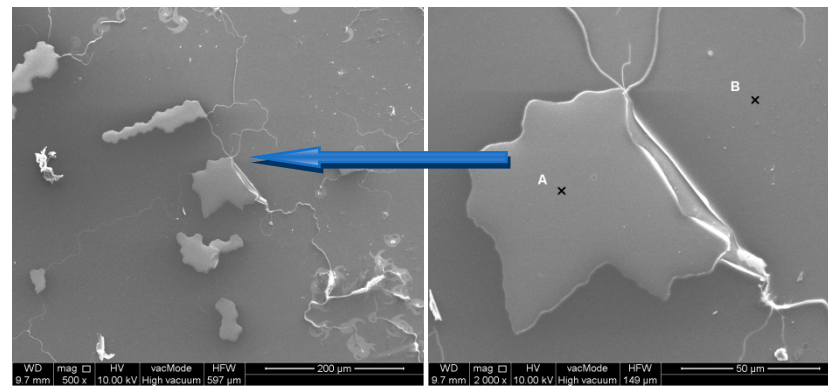
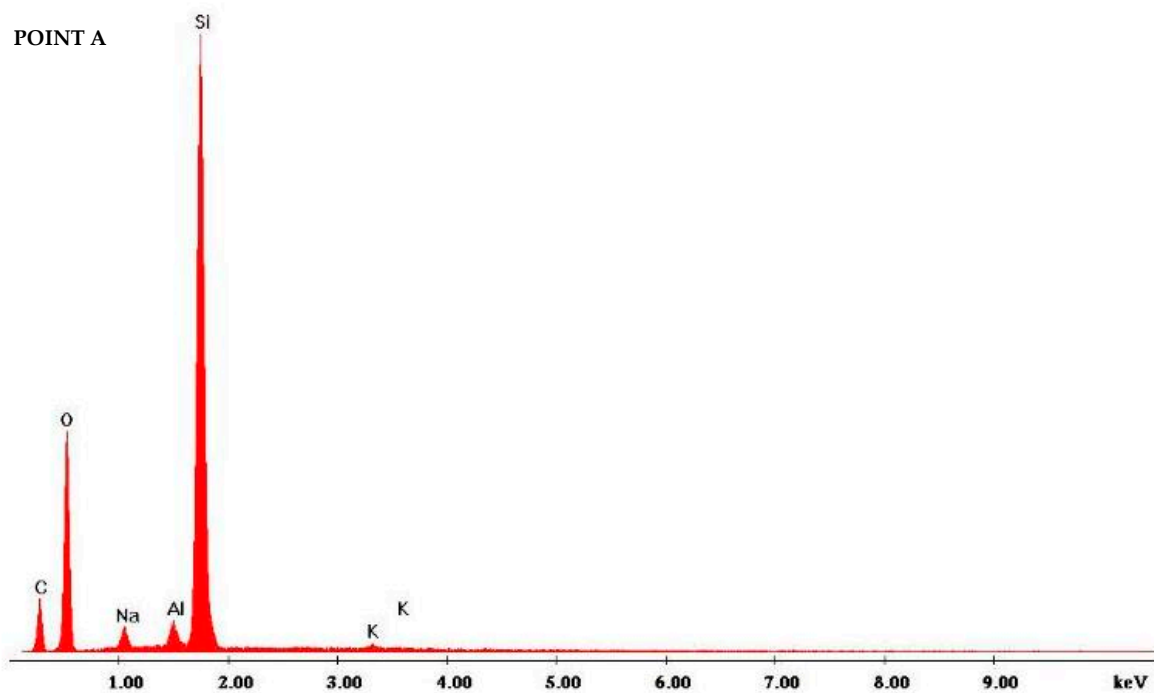


Figure 11. Topography of glass from points A and B, 20 °C.

An analysis of the chemical composition and the obtained spectra confirm the presence of elements comprised by glass recycle and point to minimum differences in the contents of main components. Peaks were recorded coming from quartz (SiO_2), potassium oxide (K_2O), sodium oxide (Na_2O), and aluminium oxide Al_2O_3 . According to studies described in the literature [77–79], the above mentioned oxides contribute to better mechanical, chemical, and thermal properties of silicate glass. The designed concrete is less susceptible to cracking that may occur after thermal shock.



(a)

Figure 12. Cont.

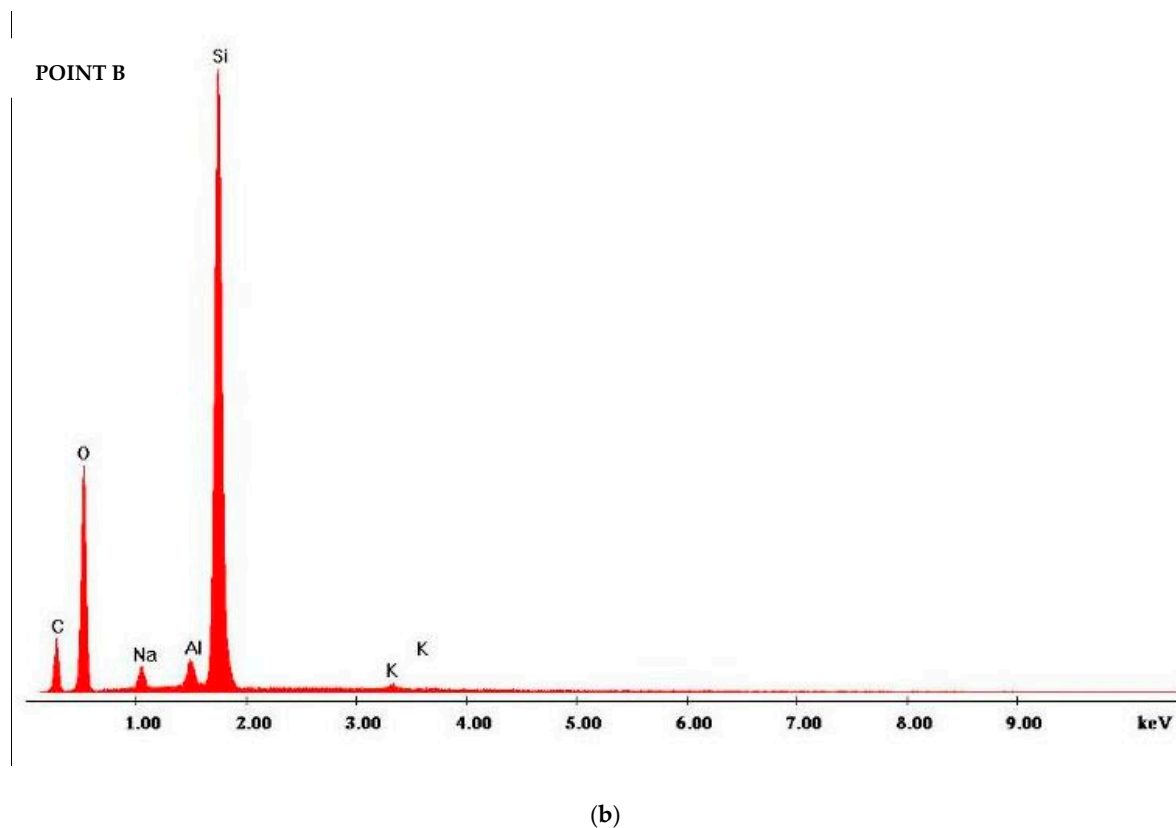


Figure 12. SEM analysis of glass surface determined from point A and B (a,b), 20 °C.

5. Conclusions

An analysis of the obtained results of the experiments allowed the formulation of the following conclusions:

1. If glass powder is used as a substitute for cement, after 28 days of curing, concrete Zc.I.5 was found to have the biggest increase of compressive strength by approximately 10% with the addition of cullet by approximately 5%, as compared to the control sample.
2. Comparable parameters were obtained from bending tensile strength, after 14 and 28 days of curing with the control trial. This proves the Pozzolanic reactivity of cullet.
3. The correlation between the average compressive strength and the average flexural strength is significant. In regards to concrete Zk, a decrease in compressive strength was recorded, as well as an increase in flexural strength, with an increase of the amount of cullet used in concrete. Meanwhile, for concrete Zc, the compressive strength and the flexural strength grew as the amount of recycle in concrete increased.
4. The obtained results provide proof that cullet positively affects the concrete strength in situations of a fire. Flexural strength, for all concrete samples heated up to temperature of 500 °C, considerably grew; concrete produced with the use of glass powder Zc increased to at least 52%. If 5% of fine glass cullet was added, concrete Zk.I.5 had a maximum strength equalling to 3.25 MPa, proving a significant increase (130%) in strength as compared to the basic sample. Tests of compressive strength of concrete types subjected to temperature loads of 500 °C, in which cullet was used as a substitute for aggregate, showed that an approximate increase was achieved by 46%–17%–18% for Zk.I.5–Zk.I.10–Zk.I.15, respectively.
5. The level of compressive strength determined for all samples modified by glass aggregate, heated up to 800 °C, was slightly higher (or similar) in strength as compared to the control trial.

6. Concentrations of elements in the tested material remained within admissible limits.
7. Waste consisting of cullet is fully valuable material that can be reused in the production of concrete. Recyclate does not undergo biodegradation, and may be used both as substitute of fine aggregate and of cement.

To date, research shows that an insignificant replacement of cement or fine aggregate with glass waste containing finer fractions may prove advantageous; however, the achieved results continue to be rather contradictory, which shows that the optimum mixture formula has not been established yet. The topic at hand requires further long-term studies to allow an in-depth evaluation of the feasibility of using cullet as a recycling aggregate for the production of unconventional concrete.

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