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Application of the Hazardous Waste Vitreous Enamel Generated in the Production Process of Heating Devices as a Partial Replacement for Cement

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Abstract: Solving problems with hazardous waste materials is of crucial importance today. In the presented study, the application of waste vitreous enamel as a cement replacement up to 30% in mortar and concrete production was investigated. The chemical and physical-chemical characterization of the starting material was performed, as well as a leaching test and physical-mechanical characterization of mortar and concrete mixes. Obtained results showed that, due to its chemical composition, the vitreous enamel used must be classified as hazardous waste. At the same time, it possesses pozzolanic properties and satisfies minimal criteria for use as a cement replacement. Testing mortars and concrete mixes indicate that waste vitreous enamel can be applied as a construction material for cement replacement in the maximal amount of 20%. The leaching test was performed in accordance with international standard EN 12457-2 on hardened mortar with a maximal cement replacement of 20%. The results showed that there was no significant release of toxic elements, i.e., that the practical application of hazardous waste vitreous enamel in the construction industry may be fully in line with environmental standards.

Keywords: hazardous waste; vitreous enamel; mortar; concrete; cement replacement; building industry

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1. Introduction

The rapid development of industry as well as a constant need for new and more advanced products has resulted in the generation of large quantities of waste, and its quantities can be measured in millions and billions of tons. Depending on the country, such waste is disposed of at legal (commonly in developed and middle developed countries) and/or insufficiently secured illegal landfills (mainly characteristic of underdeveloped countries where it has a great negative impact on the environment and human health).

Depending on the nature of the waste, its properties, and its origin, different methods of disposal are used, i.e., different types of secured landfills, such as open, controlled, and closed. In order to avoid problems of treatment and disposal, it is necessary to work on the requests for the input of technology on waste minimization [1–4]. In the case of non-hazardous waste, it can be returned to the production process using appropriate technologies with reasonable investments, which is followed with low-cost production of end products, so that, besides environmental gains, economic benefits may be achieved, too. The environmental benefits are visible in the removal of the waste from the environment as well as by reducing consumption of natural resources. This is the practice in almost all highly developed countries, and to a lesser extent in middle-developed countries, with a tendency to apply more in underdeveloped countries.

However, a significant problem is waste, which is hazardous for the environment due to its composition and characteristics. Manipulation of such waste is much more complex and expensive. For such waste it is difficult to obtain useful value and removal from the environment without large investments. For that reason, there are considerably smaller numbers of plants in the world where such waste may be treated, so the transport and management of this kind of waste is very expensive, and it represents a significant individual economic burden for each country. At the same time, hazardous waste at landfills located in the environment, regardless of whether they are legal or illegal, are a constant threat and danger, both, to nature and to human and animal health. For these reasons, it is very important to find a way to treat such waste, remove it from nature, and give it a useful value and a safe applicability in practice. Chen et al. [5] developed supercritical water oxidation technology in order to treat oil-based drill cuttings (OBDC) from a shale gas field and high-nickel-content electroplating sludge (Ni-EPS) obtained as a hazardous waste in an electroplating factory. The authors pointed out that the applied technology is expensive but effective in solving problems with the mentioned hazardous waste. Haji Ali et al. [6] showed that application of nickel foam as an electrode may be used for the electrochemical treatment of liquid hazardous wastes generated during COD (Chemical Oxygen Demand) tests, which contain mercury, silver, and chromium (VI). However, the authors pointed out that the disadvantage of the application of the proposed method is the large-scale production of nickel foam. Gupt et al. [7] used a naturally occurring raw mineral material, bentonite, from a deposit in India in order to ensure that hazardous waste contaminated with heavy metals does not further harm the environment. The authors have shown that bentonite is effective in preserving natural resources from hazardous waste of this type. However, in this way, natural resources are consumed while the amount of waste is not reduced, so that only in the short time can the problem be mitigated or solved. Some authors [8,9] examined various methods by which it is possible to monitor the impact of hazardous waste from landfills on the environment, while other authors [10,11] examined the possibility of legislation to solve the problem of illegal landfills or to find ways for more efficient networking between hazardous waste producers and plants that deal with its processing. Additionally, the problem of hazardous waste, such as bottom ash [12] or fly ash [13–15], can be solved by applying it in the construction industry, as a substitute for cement in the production of mortar and concrete.

Immobilization of hazardous waste by its application in the construction industry (cement or mortar and concrete production industry), where it is used as a substitute for natural raw materials, can be particularly interesting from the aspect of solving environmental and economic problems caused by hazardous waste. The significant benefits of such an approach are the application is very simple and significant amounts of waste can be consumed. Immobilization of hazardous waste materials of different origins in the construction industry is well described in the literature [16–22]. However, comprehensive research must be carried out prior to application as wastes of different origins, deposits, lots, or treatment may have completely opposite properties. For these reasons, each waste must be monitored separately, and complete research must be done. Furthermore, applied materials must meet criteria prescribed by the relevant standards in terms of physical-mechanical and physical-chemical properties ("Rulebook on restrictions and prohibitions on the production, placing on the market and use of chemicals", "Rulebook on the categories, testing and classification of waste", EN 450-1:2012). It is also important that after application, there is no leaching of hazardous components and there is no possibility of their flushing and returning to nature, i.e., meeting the criteria prescribed by the relevant national directives, applicable in the country where the waste material is used ("Rulebook on permitted quantities of hazardous and harmful materials in agricultural land and water for irrigation and methods for testing"—applicable in Serbia).

Production of heating devices on solid, liquid, and gaseous fuels generated a significant amount of waste material, which is mostly vitreous enamels. From the aspect of environmental protection, reducing the deposit of this waste and finding its useful value is a challenge. Namely, vitreous enamel is a transparent or opaque mass, colorless or colored with metal oxides, which is applied by melting to metal plates, ceramic, or glass objects for decoration and protection. The corrosion protection properties of enamel coatings are one of the main reasons for vitreous enamel's broad application in the industry to coat various electrical products and household appliances, dishes, panels, devices, chemical reactors, pipelines for gases, and various liquids and steel products. The vitreous enamel coating matrix possesses a glassy nature and because of that it shows excellent impermeability, washability, and high-temperature (fire) resistance as well. In addition, it is not affected by UV exposure, so its surface aspect remains unchanged in time, which is the main advance in comparison with organic paints and polymers [23,24].

In order to improve the properties of vitreous enamel, certain additives are often added to it [24–27], which may contain ingredients that may be harmful for the environment. After the enameling process, a part of such a matrix remains as waste, which as such cannot be reused for coating but must be disposed of in a landfill, where it can affect the environment, and thus human and animal health. For that reason, it is important to find useful value for it. To our knowledge, finding the useful value of waste vitreous enamel and its potential practical application is not well described in the literature and studies on this topic are very rare [28].

In this paper, the application of waste vitreous enamel generated in a plant for the production of heating devices will be tested to our knowledge for the first time in an area, which are realistic bases for the consumption of large quantities of this waste material, i.e., in building and construction industries. For that purpose, testing of vitreous enamel as a potential replacement for cement in the production of mortar and concrete will be performed. The main emphasis in the paper is on practical application, but waste vitreous enamel was also characterized by physical-chemical and physical-mechanical methods and then tested as a replacement for cement in accordance with adequate standards by using prescribed physical-mechanical methods.

2. Materials and Methods

2.1. Sampling

The waste vitreous enamel (Figure 1) was taken from a heating devices production plant in Serbia, one of the largest factories in Southeast Europe, which generates approximately 40 tons of waste vitreous enamel every year.

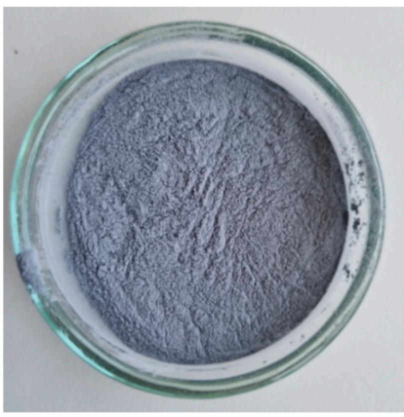

Figure 1. Raw waste vitreous enamel sample.

For this research, the samples were taken from the production plant in a time period of 4 months (October 2019–January 2020). In this time period, a total of 60 samples were taken (15 each month). Then, all samples were mixed and homogenized, and one representative was prepared and taken for analysis. The representative sample was 100% with size <63 µm, and with a specific mass of 2.5 g/cm^3.

Ordinary Portland cement CEM I 42.5R was used from the company Moravacem Popovac, Serbia. The chemical and mineralogical composition of the used cement are given in Table 1 [17].

Table 1. Chemical and mineralogical composition of CEM I 42.5R.

Chemical Composition								
SiO_2	**Fe_2O_3**	**Al_2O_3**	**CaO**	**MgO**	**SO_3**	**Na_2O**	**K_2O**	**LOI**
21.62	2.60	7.00	60.16	2.34	2.55	0.33	0.66	2.68
Mineral properties								
brownmillerite; calcium-silicate-oxide; calcite; larnite; magnesium-silicate; calcium-hydroxide								

2.2. Physical-Chemical Analyses

Chemical analysis and determination of the content of heavy metals in the representative waste vitreous enamel sample and after a leaching test were performed using inductively coupled plasma optical emission spectrometry (ICP-OES) on an ICP spectrometer Spectroflame (Spectro Analytical Instruments, Kleve, Germany). Arsenic concentrations were determined by microwave digestion according to EPA 3051A procedure.

X-ray powder diffraction (XRD) was applied to analyze phase composition of samples. XRD pattern of the waste vitreous enamel sample was collected using a SIEMENS D500 diffractometer, with CuKα radiation at 40 kV and 40 mA, a 0.02° step size over a 2θ range of 20–75°. Using Diffracplus software, the diffractogram was analyzed for identifying phase of samples in terms of the Powder Diffraction File (PDF) cards published by the Joint Committee on Powder Diffraction Standards (JCPDS)—International Centre for Diffraction Data (ICDD).

A Micromeritics ASAP 2020 instrument was used to measure the textural properties of the waste vitreous enamel samples. The nitrogen adsorption–desorption isotherms were obtained by adsorption at −196 °C. Firstly, the sample was degassed at room temperature for 1 h and then, under the same residual pressure, for 24 h at 120 °C. The Brunauer–Emmett–Teller (BET) method was used for determination of the specific surface area (S_{BET}) of the investigated sample [29]. The Dubinin–Radushkevich (DR) equation was applied to the nitrogen adsorption isotherms to obtain the micropore volume (V_{mic}) [30]. Mesopore volume (V_{meso}) was determined by the Dollimore and Heal (DH) method [31].

2.3. Testing of the Pozzolanic Activity of Waste Vitreous Enamel

In order to determine the potential for application of waste vitreous enamel as the type II addition in production of concrete in accordance with standard EN 206, the following determinations were performed: pozzolanic class, activity index, water requirement, and initial and final setting time. The pozzolanic class was determined in accordance with standard SRPS B.C1.018 and the activity index was determined according to standard EN 450-1. The water requirement was determined according to standard EN 450-1 Annex B, while initial setting time and final setting time and soundness were determined according to standard EN 196-3.

2.4. Mortar and Concrete Preparation

For mortar preparation, fine river sand (fraction 0/2 mm) was used with specific gravity and water absorption of 2610 kg/m^3 and 1.52%, respectively. For making concrete, a crushed gabbro with fractions 4/8 mm and 8/16 mm was used as the coarse aggregates with a specific gravity of 2890 kg/m^3 and a water absorption of 0.58%. As the fine aggregates, river sand with a fraction of 0/4 mm, a specific gravity of 2620 kg/m^3, and a water absorption of 1.42% was used.

Preparation of mortar mixtures and production and curing of specimens was performed according to the standard EN 196-1. A total of 5 different mortar mixes were made: a reference mixture (M-E) and 4 mixtures (M-7.5, M-15, M-22.5, and M-30) in which cement was replaced with 7.5, 15, 22.5, and 30% of waste vitreous enamel. The mass ratio 1:3 and 1:2 was used in all mixtures for quantify binder and sand, i.e., both water and binder, respectively. For improving workability of the mortar mixtures, the superplasticizer was used in the same amount in all mixtures. Table 2 shows quantities of materials for making one series of mortar specimens (3 prisms with dimensions 40 × 40 × 160 mm) in the laboratory mixer "Hobart N-50". Quantities of materials are given in mass and volume ratio.

Table 2. Mix proportion of mortar for one series of samples.

Component	M-E	M-7.5	M-15	M-22.5	M-30
Cement [g (cm^3)]	450.00 (142.86)	416.25 (132.15)	382.5 (121.43)	348.75 (110.71)	331.96 (100.00)
Waste enamel [g (cm^3)]	-	33.75 (11.63)	67.5 (23.28)	101.25 (34.91)	135.00 (46.55)
River sand 0/2 mm [g (cm^3)]	1350.00 (517.24)	1350 (517.24)	1350 (517.24)	1350 (517.24)	1350 (517.24)
Water [g]	225.00 (225.00)	225 (225)	225 (225)	225 (225)	225 (225)
Superplasticizer [g (cm^3)]	1.00 (0.92)	1.00 (0.92)	1.00 (0.92)	1.00 (0.92)	1.00 (0.92)
SUM [g (cm^3)]	2026.00 (886.02)	2026.0 (886.94)	2026.0 (887.87)	2026.0 (888.78)	2026.0 (889.71)

Preparation of concrete mixtures, production, and curing of specimens was performed according to the standard EN 12390-2. A total of 4 different mixtures were used in which cement was replaced with waste vitreous enamel in the 0, 10, 20, and 30% (labels C-E, C-10, C-20, and C-30). A superplasticizer on the base carboxylates was added to all concrete mixtures. Quantities of materials in mass and volume ratio for 1 m^3 of concrete mixtures are given in Table 3.

Table 3. Mix proportion of concrete mixtures for 1 m^3. Assumed air content for all samples was ~2%.

Component	C-E	C-10	C-20	C-30
Cement [kg (m^3)]	380.00 (0.121)	342.00 (0.109)	304.00 (0.097)	266.00 (0.084)
Waste enamel [kg (m^3)]	-	38.00 (0.013)	76.00 (0.026)	114.00 (0.039)
River sand 0/4 mm [kg (m^3)]	808.00 (0.308)	808.00 (0.308)	808.00 (0.308)	808.00 (0.308)
Crushed aggr. (4/8 mm) [kg (m^3)]	376.00 (0.130)	376.00 (0.130)	376.00 (0.130)	376.00 (0.130)
Crushed aggr. (8/16 mm) [kg (m^3)]	696.00 (0.240)	696.00 (0.240)	696.00 (0.240)	696.00 (0.240)
Water [kg (m^3)]	180.00 (0.180)	180.00 (0.180)	180.00 (0.180)	180.00 (0.180)
Superplasticizer [kg (m^3)]	3.04 (0.002)	3.04 (0.002)	3.04 (0.002)	3.04 (0.002)
SUM	2443.04 (0.981)	2443.04 (0.982)	2443.04 (0.983)	2443.04 (0.983)

2.5. Physical-Mechanical Characterization of Mortar and Concrete Samples

For determination of the physical-mechanical properties of the mortar samples, the following tests according to appropriate standards were used: Consistency—by flow table (EN 1015-3); Bulk density of fresh mortar (EN 1015-3); Bulk density of hardened mortar (EN 1015-10); Flexural strength and Compressive strength, (EN 196-1); Water absorption at atmospheric pressure (EN 13755); Water absorption coefficient due to capillary action of hardened mortar (EN 1015-18); Drying shrinkage (SRPS B.C8.029:1979 (ASTM C 596)); Adhesion between mortar and a concrete substrate (EN 1015-12).

For determination of the physical-mechanical properties of the concrete samples, the following tests were done in accordance with adequate standards: Consistency—slump test (EN 12350-2); Density of fresh concrete (EN 12350-6); Air content in fresh concrete (EN 12350-7); Density of hardened concrete (water saturated) (EN 12390-7); Flexural strength (EN 12390-5); Compressive strength (EN 12390-3); Tensile splitting strength (EN 12390-6); Secant modulus of elasticity (EN 12390-13); Depth of penetration of water under pressure (EN 12390-8); Freeze-thaw resistance with de-icing salts—Scaling (CEN-TS_12390-9); Determination of ultrasonic pulse velocity (EN 12504-4).

The leaching test was carried out in accordance with the EN 12457-2 standard. The test was performed on the mortar mixture, where a maximal amount of cement was replaced with waste material. The test was not carried out on concrete because the percentage content of waste material in concrete is lower compared to mortar. The testing procedure was as follows: samples of dry mortar prisms were crushed mechanically and sieved through a 4 mm sieve. Then, 100 g of material finer than 4 mm was poured into a metal grid located inside the glass vessel of the laboratory shaker. 1000 mL of distilled water with a temperature of 200 °C was poured into the glass container, after which the mixer with a vertical rotary propeller was started. The speed of rotation of the propeller was 150 rpm, and the time of water flow around the sample was 24 h. The mortar sample was then filtered through filter paper, and the concentration of heavy metals was determined on the obtained eluate.

3. Results and Discussion

3.1. Physical-Chemical Characterization of the Waste Vitreous Enamel Samples

3.1.1. Determination of Chemical Composition and Heavy Metal Content

Results of the determination of chemical composition and heavy metal content in the waste vitreous enamel samples are presented in Table 4. According to Standard "Službeni Glasnik 56/2010" as well as EN 12457/EN 16192: 2011 applicable in the Republic of Serbia, in the investigated representative waste vitreous enamel sample, the contents of Sb, Se, Zn, Cu, Ni, Cd, Mo, and Pb were higher in comparison with the maximal values for non-hazardous waste prescribed by standards and, because of that, the waste vitreous enamel must be classified as a hazardous waste. For that reason, the disposal of this type of waste in landfills is very dangerous and finding a useful application for it should be a priority.

Table 4. The content of elements in representative waste vitreous enamel sample.

	Content, mg/kg		
Element	**Determined Amount**	**Limits for Disposal of Non-Hazardous Waste**	**Limits for Disposal of Hazardous Waste**
Mo	15.0	10	30
Hg	<0.15	0.2	2.0
Sb	**9.5**	0.7	5.0
Se	**4.5**	0.5	7.0
Sr	222.0	-	-
Ba	35.5	100	300
Ca	6854.0	-	-
Mg	234.0	-	-
Ti	620.0	-	-
V	18.5	-	-
Mn	7353.0	-	-
Fe	10,153.0	-	-
Co	2872.0	-	-
Cu	**2463.0**	50	100
Zn	**312.0**	50	200
Ni	**68.5**	10	40
Cd	**4.0**	1	5.0
Al	622.0	-	-
Si	53,380.0	-	-
Pb	**14.0**	10	50
As	<0.5	2	25
Be	<0.05	-	-
Cr	59.0	10	70
Tl	48.0	-	-
Sn	<1.0	-	-

Limited values are prescribed by "Službeni Glasnik 56/2010". Dashes mean that the standard does not prescribe limits.

For the Republic of Serbia, the application of waste materials in the building and cement industry is prescribed by the "Rulebook on restrictions and prohibitions on the production, placing on the market and use of chemicals" as well as "Rulebook on the categories, testing and classification of waste", which do not restrict the use of materials on the basis of the content of heavy metals in raw materials. Thus, although the investigated sample is hazardous, it can be used in the building and cement industry. It is only important that there is no release of heavy metals after the leaching test on the mortar mixture is carried out in accordance with the EN 12457-2 standard.

3.1.2. X-ray Powder Diffraction Analysis (XRD)

The results of the X-ray powder diffraction analysis are given in Figure 2.

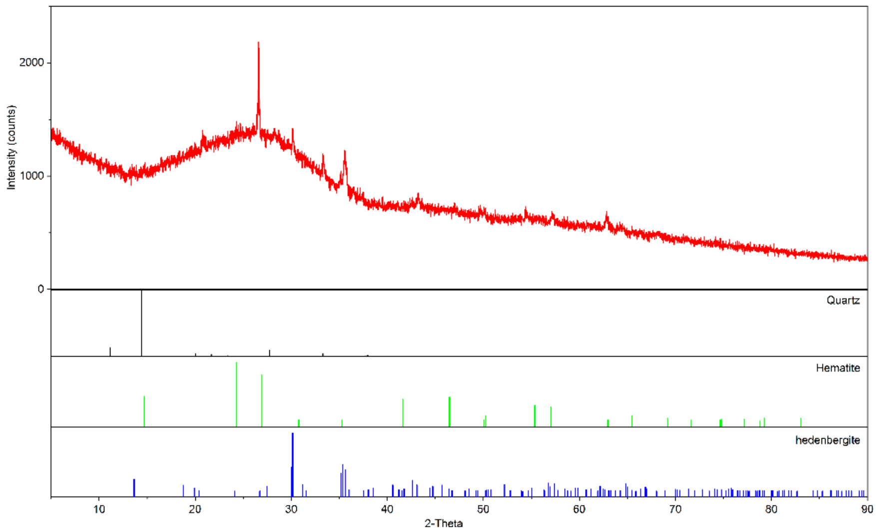

Figure 2. XRD diagram of the waste vitreous enamel.

As can be seen in Figure 2, the waste vitreous enamel is composed from the crystal and amorphous phases. The main composition of the crystal phase includes quartz, hematite, and hedenbergite. The amorphous components are indicated by a big bump background in the 2θ range of 15–35°. The amorphous phase originates from glass matrix of vitreous enamel, which mainly contains Si and Al. A high content of Fe in the vitreous enamel is proved by the results presented in Table 4, and it mainly exists as the form of hematite and hedenbergite.

3.1.3. Textural Properties

The values of textural properties of the waste vitreous enamel are presented in Table 5.

Table 5. Textural properties of the waste vitreous enamel sample.

Sample	S_{BET}, m^2/g	V_{total}, cm^3/g	V_{meso}, cm^3/g	V_{micro}, cm^3/g
Vitreous enamel	15.3	0.0794	0.0654	0.0140

As can be seen from Table 5, the specific surface area of the waste vitreous enamel sample was 15.3 m^2/g. The standard "EN 197-1-Cement—Part 1 Composition, specifications and conformity criteria for common cements", which is applicable in the Republic of Serbia and the EU, prescribes that additions, such as silica fume, should have a S_{BET} of at least 15 m^2/g, which means that the investigated sample satisfied the required criteria and can be used as a cement replacement, from the point of textural properties.

3.2. Testing of the Pozzolanic Activity of Waste Vitreous Enamel

Pozzolanic activity is one of the most important parameters on which largely depends whether the material can be used in concrete production or not. From that point, pozzolanic activity and the activity index of the waste vitreous enamel were firstly determined in accordance with appropriate standards, and the results are presented in Table 6.

Table 6. Results of testing pozzolanic activity and parameters of cement paste with addition of the waste vitreous enamel from an enamel plant.

Property	Standard	Parameters/Results	Requirement	Conclusion
Class of pozzolanic materials	SRPS B.C1.018	Flexural strength: 2.02 MPa Compressive strength: 5.50 MPa	>2.0 MPa >5.0 MPa	class 5 of pozzolanic materials
Activity index	EN 450-1	After 28 days: 79.42% After 90 days—94.75%	>75% >85%	satisfies satisfies
Water requirement	EN 450-1 Annex B	94%	<95%	satisfies
Initial setting time Final setting time	EN 196-3	135 min 165 min	<230 min not prescribed	satisfies -
Soundness	EN 196-3	1.0 mm	<10 mm	satisfies

The obtained results showed that the investigated sample possesses pozzolanic activity and belongs to class 5 of pozzolanic materials due to values of the flexural and compressive strengths. The water requirement, initial setting time, final setting time and soundness also possess values that completely satisfied standard requirements, which means it is possible to use it as a type II admixture for the production of concrete in accordance with EN 206.

3.3. Effects of Waste Vitreous Enamel on Mortar Properties

Test results of physical and mechanical properties of mortar mixtures based on Portland cement, natural sand, and addition of waste vitreous enamel are given in Tables 7 and 8 and Figures 3–6.

Table 7. Test results of physical properties of mortar mixtures.

Property	Unit	M-E	M-7.5	M-15	M-22.5	M-30
Consistency—by flow table	mm	135 ± 2.0	144 ± 2.5	147 ± 3.0	153 ± 2.5	164 ± 3.0
Bulk density of fresh mortar	kg/m^3	2299 ± 8	2294 ± 6	2289 ± 7	2281 ± 9	2275 ± 8
Bulk density of hardened mortar	kg/m^3	2294 ± 7	2290 ± 8	2285 ± 6	2278 ± 8	2270 ± 9
Water abs. at atm. pressure	%	7.54 ± 0.12	7.45 ± 0.10	7.36 ± 0.09	7.25 ± 0.11	7.16 ± 0.08
Water abs. due to capillary action of hardened mortar: for mortars other than renovation mortars	$m^2 \times min^{-0.5}$	0.24 ± 0.02	0.23 ± 0.01	0.23 ± 0.02	0.22 ± 0.01	0.21 ± 02
Water absorption due to capillary action of hardened mortar: for renovation mortars	kg/m^2	5.50 ± 0.03	5.53 ± 0.04	5.57 ± 0.05	5.62 ± 0.04	5.65 ± 0.03

Table 8. Drying shrinkage of the mortar mixtures.

Designation of Mortar	M-E	M-7.5	M-15	M-22.5	M-30
Age [days]	$\varepsilon_{sm,sr}$ [mm/m]				
3	0.00	0.00	0.00	0.00	0.00
4	0.25 ± 0.02	0.23 ± 0.03	0.20 ± 0.03	0.18 ± 0.02	0.16 ± 0.03
7	0.38 ± 0.03	0.36 ± 0.02	0.33 ± 0.01	0.30 ± 0.03	0.28 ± 0.02
14	0.59 ± 0.01	0.59 ± 0.02	0.58 ± 0.02	0.57 ± 0.02	0.56 ± 0.02
21	0.69 ± 0.03	0.70 ± 0.03	0.70 ± 0.03	0.71 ± 0.03	0.72 ± 0.03
28	0.91 ± 0.02	0.90 ± 0.03	0.88 ± 0.02	0.85 ± 0.02	0.84 ± 0.02
56	0.93 ± 0.02	0.92 ± 0.02	0.90 ± 0.02	0.87 ± 0.01	0.86 ± 0.01
90	0.94 ± 0.03	0.93 ± 0.01	0.92 ± 0.01	0.89 ± 0.02	0.88 ± 0.02

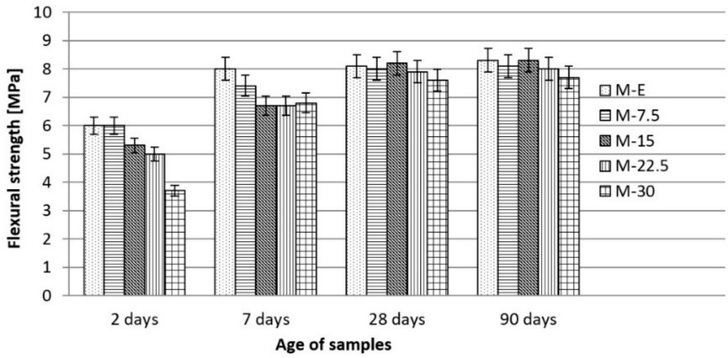

Figure 3. Flexural strength of mortar samples.

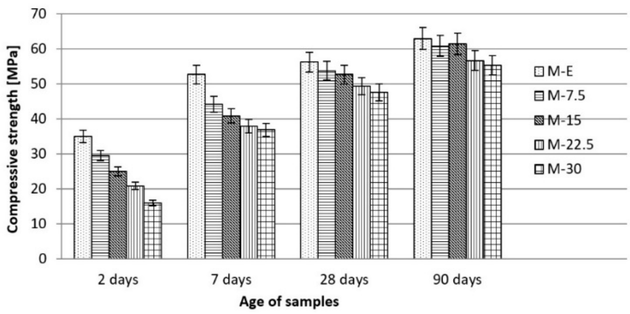

Figure 4. Compressive strength of mortar samples.

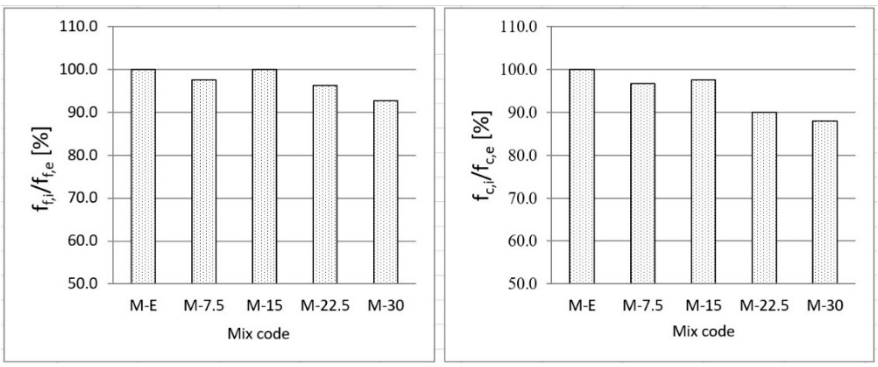

Figure 5. Percentage ratio of flexural (**left**) and compressive strength (**right**) of mortar with waste vitreous enamel and reference mortar at 90 days.

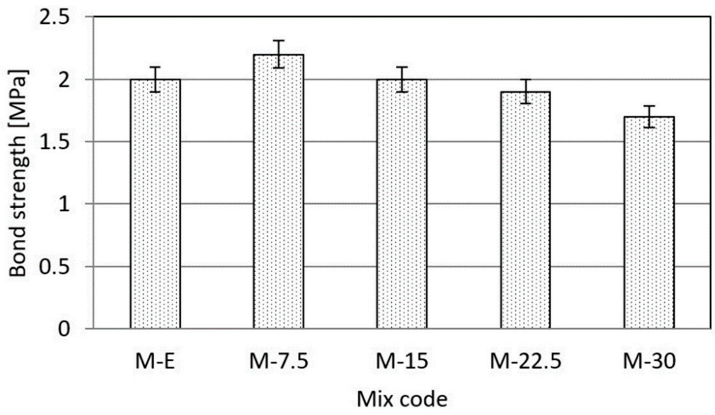

Figure 6. Adhesion between mortar and a concrete substrate.

Based on the consistency test results presented in Table 7, it can be concluded that with the increase in the percentage of cement replacement by waste vitreous enamel, the flow of mortar on the flow table also increased. The highest flow was achieved on the mortar made with 30% replacement of cement by waste vitreous enamel, which is 21% higher than the reference mortar. The main reason is the lower water requirement of vitreous enamel compared to cement, as a result of which the amount of free water in the mortar mixture increases. In general, the addition of waste vitreous enamel contributed to the improvement of mortar workability.

The replacement of cement with waste vitreous enamel contributed to a slight reduction in the bulk density of mortar in the fresh and hardened state. This reduction is up to 24 kg/m^3 for the fresh and up to 25 kg/m^3 for the hardened state.

The replacement of cement with waste vitreous enamel did not significantly affect the water absorption values, regardless of the test method. Water absorption of reference mortar at atmospheric pressure was the lowest, while with the increase in the percentage of cement replacement with waste vitreous enamel, the absorption value slightly decreased. A similar trend was observed for determination of the absorption coefficient due to capillary absorption of water. In the case of a 90-min test (test for non-repair mortars), the lowest absorption coefficient was recorded for mortars marked "M-30", while in the case of a 24-h test (test for repair mortars), the lowest absorption coefficient was recorded at reference mortar. Results of shrinkage measurements are given in Table 8.

Dry shrinkage was performed on mortar prisms with dimensions of 160 × 40 × 40 mm. Shrinkage is expressed as the ratio of the change of the length and the length of the prism ($\varepsilon = \Delta l/l$). Based on the shrinkage measurement results, due to the drying of cement mortars it can be observed that mortar mixtures with the addition of waste vitreous enamel had lower shrinkage values in the initial curing phase (first 7 days) than the reference mortar. At 14 days, all mortar mixtures had almost the same shrinkage values, while in the later curing phase (days 21 to 90), the reference sample had less shrinkage compared to the mortars in which partial replacement of cement by waste vitreous enamel was performed. This is logical, given the fact that the pozzolanic reaction occurs later after the formation of portlandite (calcium hydroxide) in the hydration process of the cement, which is necessary for its initiation. The pozzolanic reaction results in the contraction of hydration products, which caused increased shrinkage of the mortar with the addition of waste materials [32,33].

Mechanical strength, and especially compressive strength, are very important parameters for cement composites. The flexural and compressive strength tests were performed at the mortar specimen age of 2, 7, 28, and 90 days. A graphical representation of the results is shown in Figures 3 and 4.

It can be observed that with increasing cement replacement by waste vitreous enamel, the strength of mortar specimens decreased at early ages (2 and 7 days). On the other hand, with increasing age (28 and 90 days) the strength of mortar with waste vitreous enamel approached the values of the reference mortar. This is attributed to the pozzolanic reaction of waste vitreous enamel, which manifests itself at a later stage of hardening. For that reason, the percentage of possible replacement of cement with waste vitreous enamel was estimated on the basis of strength at 90 days.

In Figure 5, the percentages of flexural and compressive strength of the mortar prepared with partial replacement of cement by waste vitreous enamel and the reference mortar at 90 days of age are presented.

Figure 5 shows that the decrease in flexural strength that occurs with the increase in the percentage of cement replacement by waste vitreous enamel was very low, whereas in the case of 15% replacement there was no decrease. The compressive strength decreased slightly with an increase in the percentage of replacement, with a maximum decrease of 13%. If 15 % is the acceptable level of strength decrease, then it can be concluded that in the production of mortar, up to 30% of cement can be replaced by waste vitreous enamel.

The testing results of adhesion between mortar and a concrete substrate are shown in Figure 6.

The replacement of cement with waste vitreous enamel resulted in a reduction in the adhesion of the mortar in comparison with concrete substrate, with an exception for cement replacement of 7.5%, when an adhesion strength increased 10% and 15%, where the adhesion is equal to that of M-E. With an increase in the percentage of cement replacement in an amount higher than 15%, the adhesive strength decreased with a maximum reduction of 15% for the M-30 sample. The lower limit of adhesive strength for repair mortars is 1.5 MPa, thus, all tested mortar mixtures meet this condition. It should be noted that in all samples the fracture was at the junction between the mortar and the substrate.

3.4. Effect of Waste Vitreous Enamel on Concrete Properties

Test results of physical and mechanical properties of concrete mixtures based on Portland cement, natural sand, coarse crushed aggregate, and addition of waste vitreous enamel are given in Tables 9–11 and Figures 7 and 8.

Table 9. Test results of physical properties of concrete mixtures.

Property	**Unit**	**C-E**	**C-10**	**C-20**	**C-30**
Consistency—slump test	mm	200 ± 10	210 ± 12	220 ± 9	240 ± 11
Density of fresh concrete	kg/m^3	2466 ± 12	2495 ± 9	2488 ± 10	2481 ± 12
Air content in fresh concrete	%	2.6 ± 0.19	1.9 ± 0.16	1.5 ± 0.18	1.3 ± 0.15
Density of hardened concrete (water saturated)	kg/m^3	2455 ± 10	2490 ± 12	2485 ± 8	2477 ± 11
Determination of ultrasonic pulse velocity	km/s	5.21 ± 0.022	5.23 ± 0.020	5.20 ± 0.018	5.18 ± 0.015

Table 10. Test results of mechanical properties of concrete mixtures.

Property	**Unit**	**C-E**	**C-10**	**C-20**	**C-30**
Flexural strength	MPa	28 days: 7.0 ± 0.2	28 days: 6.4 ± 0.3	28 days: 5.9 ± 0.1	28 days: 5.6 ± 0.3
Compressive strength	MPa	90 days: 7.4 ± 0.3	90 days: 6.8 ± 0.2	90 days: 6.4 ± 0.2	90 days: 6.1 ± 0.2
			See Figure 6		
Tensile splitting strength	MPa	28 days: 3.9 ± 0.2	28 days: 3.5 ± 0.2	28 days: 3.0 ± 0.3	28 days: 2.8 ± 0.2
Secant modulus of elasticity	GPa	28 days: 33.0 ± 0.3	28 days: 33.5 ± 0.2	28 days: 33.2 ± 0.2	28 days: 33.0 ± 0.3

Table 11. Depth of penetration of water under pressure and freeze/thaw resistance with de-icing salt of concrete mixtures.

Property	Unit	C-E	C-10	C-20	C-30
Depth of penetration of water under pressure	mm	12	10	14	16
Freeze-thaw resistance with de-icing salts—Scaling	kg/m^2	0.14	0.11	0.15	0.19

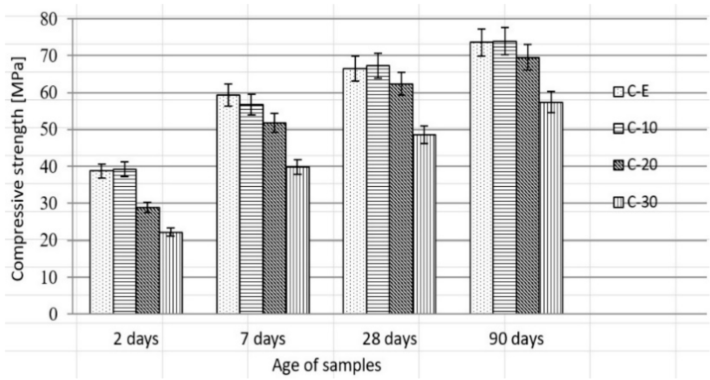

Figure 7. Compressive strength of concrete samples.

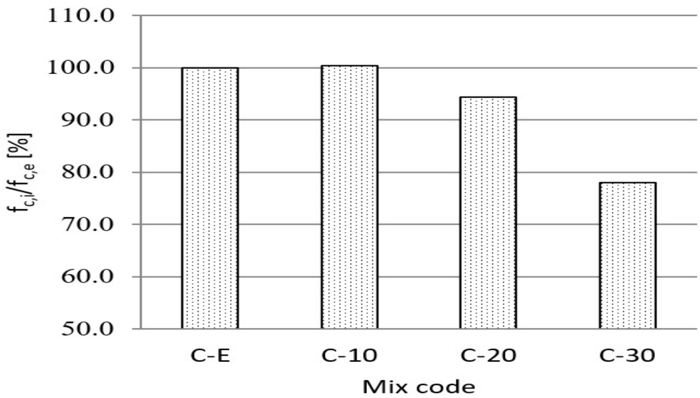

Figure 8. The percentage ratio of compressive strength of concrete with waste vitreous enamel and reference concrete at 90 days.

Based on the results of concrete consistency testing, it can be concluded that by increasing the content of waste vitreous enamel, the size of the slump increased. A maximum slump of 240 mm was recorded for the concrete mixture, C-30, which was higher than the reference mixture by 20%. As in the case of mortar mixtures, the main reason for this phenomenon is the lower water requirement of vitreous enamel compared to cement.

The content of air entrained into fresh concrete decreased with the increasing percentage of cement replacement. The reference mixture had the highest percentage of entrained air (2.6%), while the C-30 mixture had the lowest (1.3%). This can be attributed to the better placing of concrete mixed with waste material.

The replacement of cement with waste vitreous enamel contributed to the slight increase in the concrete bulk densities in the fresh and hardened state, which was primarily a consequence of the lower content of air entrained.

The concrete porosity, aggregate type and interfacial transition zone (ITZ) are the most important factors that affect the ultrasonic velocity values of concrete. Based on the results presented in Table 9, it can be seen that all concrete mixtures had values of ultrasonic velocity that was higher than 4.5 km/s, which is a limit for strong concrete [32]. The differences in the values of the ultrasonic velocities were insignificant, which indicates that all concrete mixtures had a uniform quality in terms of physical and mechanical characteristics.

The flexural strength test for the samples was performed at 28 and 90 days. Based on results presented in Table 10, it can be observed that the flexural strength decreased with the increase of cement replacement, and ranges between 9 and 20% at 28 days' age and between 8 and 18% at 90 days' age, in comparison to the C-E.

Compressive strength is the most significant characteristic of concrete, and it was determined at the age of 2, 7, 28, and 90 days. The results are shown at Figure 7. From presented results, it can be seen that the C-10 concrete mixture had a higher value of compressive strength relative to the reference concrete for all ages. On the other hand, with the increase of cement replacement by waste vitreous enamel by more than 10%, the strengths of concrete specimens decreased for all ages. In the first 7 days, the concrete strengths of mixtures C-E and C-10 had the highest increase, while after increasing ages, the compressive strength of concrete made with waste vitreous enamel was higher than the reference one due to the delayed pozzolanic reaction. A similar conclusion was reached by authors [32,34,35] in the study of durability of concrete supplemented with recycled CRT glass as cementitious material.

Figure 8 shows the percentages of compressive strengths of concrete samples prepared with partial replacement of cement by waste vitreous enamel and reference concrete for 90 days.

The diagram shows a slight decrease in compressive strength with an increase in the percentage of cement replacement for more than 10%, while in the concrete mixture, C-10, there was a slight increase in strength in some cases. The acceptable level of compressive strength decrease is 15%, so that it can be concluded that up to 20% of cement can be replaced by waste vitreous enamel in concrete production.

Based on the test results from Table 10, it can be seen that the tensile splitting strength slightly decreased with the increase in the percentage of cement replacement by waste vitreous enamel. The reference concrete had the highest strength value (3.9 MPa), while the concrete mixture with 30% cement replacement had 28% lower tensile splitting strength.

The secant modulus of elasticity of concrete was tested at a sample age of 28 days. As can be seen from Table 10, the modulus of elasticity of concrete increased with the increase in the percentage of cement replacement by waste vitreous enamel up to 10%, when it reaches the maximum value (33.5 GPa). With the further increase of cement replacement, the modulus of elasticity slowly decreased and reached a minimum for concrete, with 30% cement replacement (33.0 GPa).

The depth of penetration of water under pressure in hardened concrete and freeze/thaw resistance with de-icing salt of concrete are shown in Table 11. The results of depth penetration of water under pressure showed that the C-10 mixture had the lowest value of penetration (10 mm), which is slightly less than for reference mixture (12 mm). When the content of waste vitreous enamel in concrete was greater than 10%, the depth of water penetration through concrete increased moderately. According to Neville and Brooks [36], all concrete mixtures can be considered as waterproof as none of them has a penetration greater than 30 mm. It can be concluded that waste vitreous enamel does not have a negative effect on the resistance of concrete mixtures to the pressurized water action.

After 56 freezing-thaw cycles with de-icing salt, the least scaling of concrete surface was achieved for concrete mixture with 10% replacement of cement (C-10). In Table 11 it can be seen that with a further increase of waste vitreous enamel content, the freeze/thaw resistance of concrete reduces. The C-10 mixture had decreased scaling in comparison to the reference mixture (E), while the C-20 and the C-30 mixtures had slightly higher damage compared to sample E. A possible reason for such a result is the consequence of the formation of hydration phases during the pozzolanic reaction of waste vitreous enamel, which caused a reduction in permeability to fluids. A similar conclusion was reached by Jihwan et al. [37], who tested the durability of concrete with recycled glass as a partial replacement for cement.

3.5. Leaching Test

The leaching test was performed on the mortar mixture with maximal allowed cement replacement, where 20% of cement was replaced with waste vitreous enamel. The leaching test was carried out in accordance with the standard EN 12457-2, and the results are shown in Table 12.

Table 12. Concentration of the released elements after the leaching test.

Released Element	Concentration, μg/dm^3	* Allowed Values, μg/dm^3
Cu	4	<100
Zn	17	<1000
Ni	20	<100
Cd	2	<10
Pb	30	<100
Cr	3	<500
Hg	<0.1	<1
As	<5	<50
Mg	5	-
Fe	27	-
Co	8	-
Al	47,500	-
Sn	<10	-
Si	665	-
Mo	<1	-
Sr	<1	-
Ca	140	-
Mn	<1	-
V	<10	-

* According to the Rulebook on permitted quantities of hazardous and harmful substances in soil and irrigation water and methods of their testing, "Službeni glasnik RS 23/1994"—is applicable in Serbia. Dashes mean that the Rulebook does not prescribe limits.

From the results presented in Table 12, it can be seen that according to the rulebook, which is applicable in Serbia, after a leaching test during 24 h, the concentrations of the released elements from a mortar sample with 20% cement replacement with waste vitreous enamel were much lower than allowed.

In order to fully confirm that the application of waste vitreous enamel is safe for the environment, the leaching test was continued and performed for the next 60 days. For the whole examined time interval, the concentration of released elements did not deviate significantly in relation to those shown in Table 12. Thus, it may be concluded that application of the waste vitreous enamel as a replacement for cement in mortar and concrete productions is completely acceptable from ecological as well as human health safety aspects.

4. Conclusions

Based on the results of testing the pozzolanic activity of waste vitreous enamel, as well as its effect on the properties of cement paste, mortar, and concrete, the following can be concluded:

1. The waste vitreous enamel possesses pozzolanic activity and belongs to class 5 of pozzolanic materials. Additionally, the activity index, water requirement, setting time, and soundness possess values that completely satisfied standard requirements, which means it is possible to use a type II admixture for the production of concrete in accordance with EN 206;
2. Replacing cement with waste material contributed to a reduction in compressive strength of up to 12% and flexural strength of up to 7%, and also contributed to a reduction in shrinkage due to drying and water absorption. Due to its glassy structure, waste enamel has a positive effect on the consistency of the mortar by increasing its workability by approximately 20%. Generally, replacement of cement with waste

vitreous enamel in the amount of up to 20% in mortar does not greatly reduce its physical and mechanical characteristics compared to the characteristics of the reference mortar made with 100% cement;

3. The use of waste vitreous enamel in concrete, as a partial replacement of cement, contributes to a slight decrease in mechanical properties, while on the other hand it does not compromise the durability of the concrete. Replacement of cement with waste vitreous enamel contributes to the improvement of concrete consistency (increases settlement by 10–30 mm) and reduction of entrained air content by 20–50% compared to reference concrete. Compressive strength decreases by 6% at 20% replacement of cement, i.e., 22% at 30% replacement, while flexural strength decreases by a maximum of 13%. The depth of penetration of water under pressure in hardened concrete and freeze/thaw resistance with de-icing salt of concrete with waste material are in range of reference concrete for replacement of cement to 20%. Generally, the physical and mechanical properties of concrete mixed with up to 20% of cement replacement with waste vitreous enamel do not significantly differ from the reference concrete;
4. Bearing in mind that the use of waste vitreous enamel solves the problem of its disposal, and it can be used as a replacement for cement, it can be used in the production of mortar and concrete whose quality is slightly lower than cement composites made only with cement as a binder;
5. The application of the hazardous waste vitreous enamel as a replacement for cement in mortar and concrete productions is completely acceptable from ecological as well as human health safety aspects;
6. Further research should be focused on the study of the effect of pozzolanic reaction of waste vitreous enamel on the characteristics of the interfacial transition zone and the porous system of concrete composites in general.

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