Acta Polytechnica CTU Proceedings **47**:108–112, 2024
https://doi.org/10.14311/APP.2024.47.0108

MECHANICAL PROPERTIES OF CEMENT PASTES CONTAINING PLASMA-TREATED SILICON-BASED MATERIALS

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Abstract. The study delves into the impact of waste silicon-based materials on the mechanical characteristics of the resulting cement pastes. The investigation uses Portland cement CEM I 42.5R in conjunction with silicon-based waste materials sourced from the packaging industry, specifically from Recifa, a.s. Variations between the sample groups arise from differences in the concentration of silicon-based materials and the method of surface treatment. The inquiry seeks to assess the effects of two distinct levels of Si-based material incorporation, namely 10 wt. % and 20 wt. %, along with plasma treatment involving two different gases: oxygen (O_2) and hydrogen (H_2). The mechanical attributes under scrutiny encompass compressive and flexural strengths. The evaluation of mechanical properties is carried out on samples aged 28 days, with dimensions measuring $20 \times 20 \times 100$ mm.

Keywords: Cement pastes, plasma treatment, silicon-based waste materials.

1. Introduction

Silicon-based waste materials refer to waste products or by-products that contain a significant amount of silicon (Si) or silicon dioxide (SiO_2) and are generated from various industrial processes. Most common types of silicon-based waste materials are silica fume, fly ash, rice husk, glass waste and silicon carbide [1]. Silica fume is a by-product of the production of silicon and ferrosilicon alloys in the metallurgical industry [2]. Fly ash is a waste product generated from the combustion of coal in power plants [3]. Rice husk ash is a waste product from the agricultural industry and contains a high percentage of silica [4]. Glass waste materials, such as glass cullet, are rich in silicon dioxide [5]. Silicon carbide (SiC) waste can be generated from the production or use of silicon carbide abrasives and cutting tools [6]. Production of these waste materials depends on several factors, including the type of industry in the area, energy production methods, and the use of raw materials such as silicon and coal. Annually, more than 130 million tonnes of Si-based waste are produced worldwide. These waste materials are commonly used in cement composites [7]. These materials offer several advantages, including improved durability, reduced environmental impact, and cost effectiveness [8]. Silica fume is often used as a supplementary cementitious material in concrete. It improves the mechanical properties and durability of concrete, including increased compressive strength, reduced permeability, and increased resistance to chemical attack [9]. Fly ash is used as a supplementary cementitious material in concrete, and its use in concrete can improve workability, reduce hydration heat, and improve long-term strength and durability [3]. Rice husk can be used in concrete and other construction materials to improve strength and durability [4]. The use of silicon-based materials in concrete is well established and widely accepted in the construction industry. When used correctly and with proper consideration of these factors, they can improve the performance and durability of concrete and reduce the carbon footprint [2].

Alkali-Silica Reaction (ASR) is a significant concern when silicon-based materials are used in concrete. ASR is a chemical reaction that occurs between the alkali (sodium and potassium) present in concrete and certain types of reactive silica materials. This reaction can lead to the formation of a gel-like substance, which can expand and crack concrete over time, compromising its structural integrity [5]. Plasma surface treatment of silicon-based materials is an innovative approach that has the potential to enhance their compatibility with concrete. Plasma treatment can modify the surface properties of these materials to make them more suitable for use in concrete mixes [10]. In the case of potentially reactive silica materials, plasma treatment can help reduce their reactivity and minimize the risk of ASR. Plasma treatment can also increase adhesion between silicon-based material and cement matrix, ensuring better integration and distribution within concrete [11].

This article deals with plasma treatment of glass-based waste material and its incorporation into cement paste.

Set	Percentage replacement [wt. %]	CEM I 42.5R [g]	Waste glass powder [g]	Water ratio w/b	Type of waste materials treatment
REF	0	150	0	0.35	without treatment
S 10 REF	10	135	15	0.35	without treatment
S 10 O	10	135	15	0.35	Oxygen Plasma
S 10 H	10	135	15	0.35	Hydrogen Plasma
S 20 REF	20	120	30	0.35	without treatment
S 20 O	20	120	30	0.35	Oxygen Plasma
S 20 H	20	120	30	0.35	Hydrogen Plasma

Table 1. Composition of tested sets.

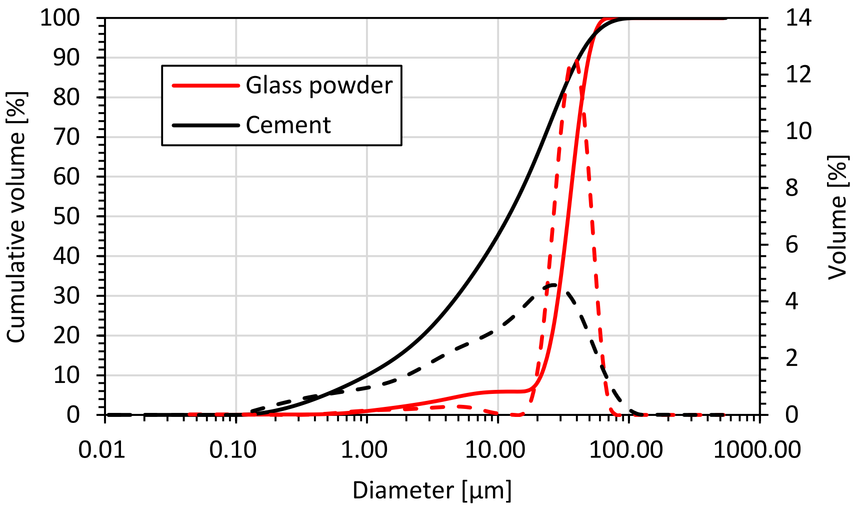

Figure 1. Particle size distribution of tested materials.

2. Materials and samples

Experiments were conducted on cement pastes in which a fraction of the cement was substituted with waste glass powder. The Portland cement used was CEM I 42.5R Radotín, characterized by a notable C_3S content of 74.6 %, a lower C_3A content of 8.1 %, C_2S content of 7.2 %, C_4AF content of 8.5 %, and a MgO content of 1.6 %.

Glass powder was created through the recycling of waste generated from glass packaging. As part of the recycling process, the glass shards were crushed and ground. The whole process of recycling was carried out by Recifa a.s. The grain size curve of the glass waste powder compared to Portland cement is shown in Figure 1, with an average particle size (D[4,3]) of 35.9 microns.

Based on Energy-Dispersive X-ray Spectroscopy (EDS) analysis, the glass powder exhibited a substantial composition with 40.0 % oxygen (O), 28.5 % carbon (C), 18.5% silicon (Si), and 7.1 % sodium (Na). This composition profile suggests a high likelihood of ASR.

The composition of all the mixtures subjected to testing is detailed in Table 1. The reference mixture contained exclusively Portland cement. In the remaining mixtures, a portion of the cement was substituted with glass powder at two weight percentages: 10 % and 20 %. The water-to-binder ratio (w/b) was consistently set at 0.35. Additionally, the glass powder was subjected to two distinct plasma treatment processes generally used for surface wetting properties modification [12]. The radiofrequency (RF) plasma treatment was carried out at low temperature using a large-area, low-pressure system (AK 400, Roth & Rau). The treatment lasted for 5 minutes and was performed in either oxygen or hydrogen environments at a pressure of 15 Pa, RF power 600 W, substrate stage bias 10–15 V, with temperatures not exceeding 50 °C. This treatment is referred to as “room temperature” treatment and was repeated four times with glass powder mixing for better homogeneity.

All tested samples featured nominal dimensions of 20 × 20 × 100 mm and were manufactured using steel triple molds. Each mixture resulted in three samples. On the second day after production, the test specimens were demolded and subsequently immersed in a water bath at a controlled temperature of 22 ± 1 °C. After 28 days of curing, the samples were subjected to various tests.

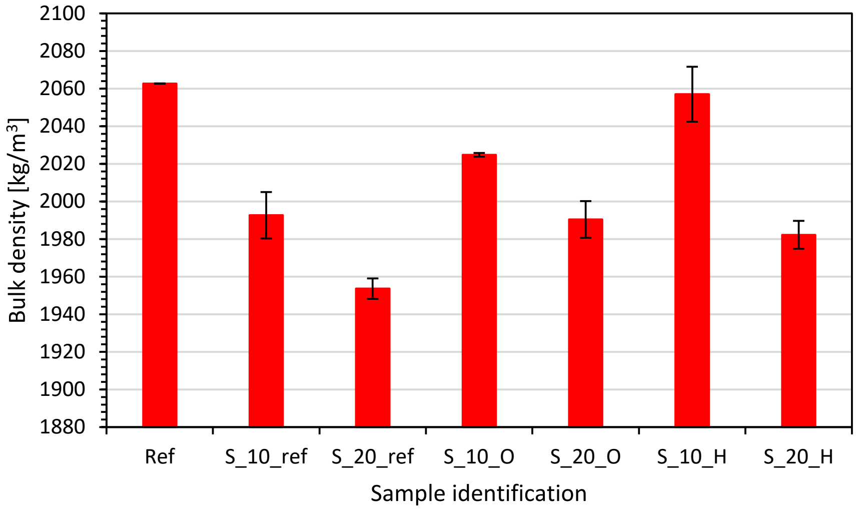

Figure 2. Bulk density of test materials with standard deviations.

3. Experimental methods

The mechanical properties of the resulting cementitious compounds were assessed by experimental techniques. The test was carried out on samples aged 28 days. The key mechanical properties investigated included flexural strength and compressive strength.

Before testing, the samples were subjected to precise measurements and weight determination. These measurements were integral in determining the bulk density of the tested materials, a critical parameter for the comprehensive evaluation of the mechanical characteristics.

Flexural strength assessment was carried out using a Heckert machine (hydraulic press), specifically model FP100. Using a three-point test configuration, the methodology combines bending and shear loading. The test was carried out under displacement control at a constant rate of $0.5\,\mathrm{mm\,min^{-1}}$. The distance between the supports during the three-point bending test was fixed at 90 mm.

The formula used for the calculation of flexural strength (f_t) is expressed as follows:

$$f_t = \frac{3F_{b,\max} \cdot L_s}{2ab^2}\,. \tag{1}$$

Compressive strength testing was performed using the Heckert hydraulic press, model FP100, with a uniaxial compression test setup. Testing was carried out under displacement control at a constant rate of $3\,mm\,min^{-1}$. The test was carried out on a suitable section of a fractured beam from a previous three-point bending test, with effective dimensions of $20 \times 20 \times 20$ mm.

Compressive strength (f_c) of the material, representing the magnitude of stress at the point of failure where the material loses its integrity, was determined based on the maximum force achieved during the test ($F_{c,\max}$) and is calculated as follows:

$$f_c = \frac{F_{c,\max}}{ab}\,. \tag{2}$$

4. Results and discussion

The findings concerning bulk density, as illustrated in Figure 2, reveal a discernible trend of diminishing values in relation to the quantity of waste glass powder. This observation aligns with the observations made by other researchers in the field [13–15]. In particular, the reference sample exhibits the highest average bulk density, quantified at $2\,062 \pm 1.1\,\mathrm{kg\,m^{-3}}$. Furthermore, in all instances of plasma treatment applied to waste glass powder, a marginal increment in bulk density, approximately 3 %, was consistently observed.

The outcomes pertaining to flexural strength (Figure 3) exhibit a consistent alignment with the bulk density results. An evident reduction in the average bending strength value is observed as the quantity of waste glass powder increases. The reference sample stands out with the highest bending strength, measuring 11.6 ± 0.1 MPa. Upon a meticulous comparison of samples featuring surface-treated and untreated waste glass powder, noteworthy distinctions emerge. In most cases, the differences in values are within the range of standard deviation, highlighting the similarity in flexural strength. However, an intriguing exception emerges in the case of 20 wt. % glass powder treated with oxygen plasma, demonstrating a remarkable 13 % increase in flexural strength compared to the reference mixture with 20 wt. % glass powder.

The results of compressive strength (Figure 4) align with the trends observed in bulk density and flexural strength. As the amount of waste glass powder increases, a corresponding decrease in compressive strength is evident. Other authors have also made similar observations in their respective studies [13–15]. In particular, the reference specimen achieves the highest compressive strength value, measuring

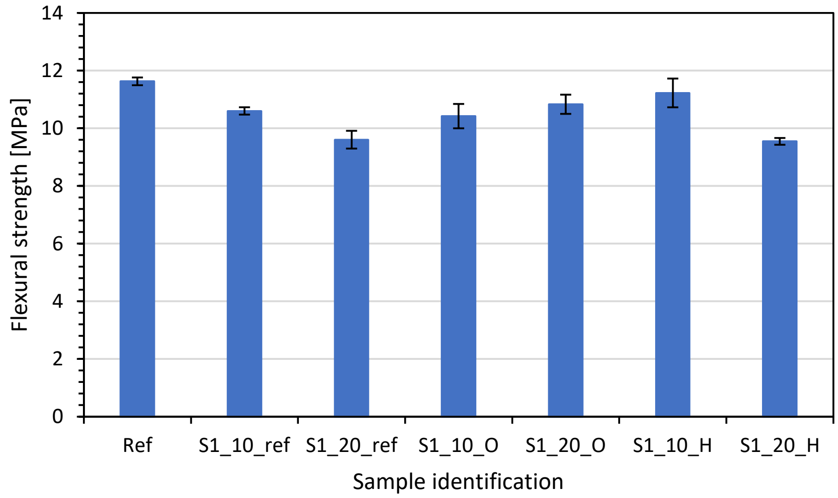

Figure 3. Flexural strength of test materials with standard deviations.

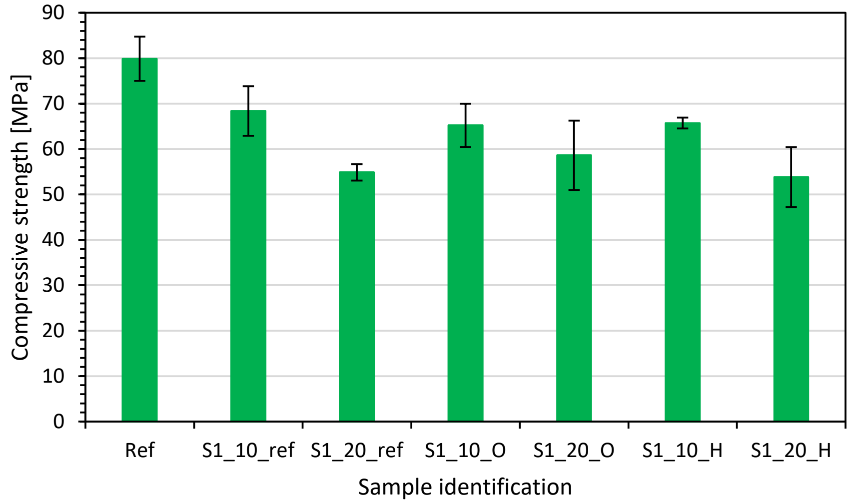

Figure 4. Compressive strength of test materials with standard deviations.

79.9 ± 4.8 MPa. In the context of plasma treatment applied to waste glass powders, no discernible impact on compressive strengths is observed. Variations in compressive strengths among mixtures containing the same amount of waste glass powder fall within the range of standard deviation.

5. Conclusions

This study is focused on investigating the impact of plasma treatment on waste silicon-based materials on the mechanical properties of cement pastes. Surface plasma treatment was carried out using two gases, namely hydrogen and oxygen. The cement pastes consist of Portland cement CEM I 42.5R and glass powder at two different concentrations: 10 wt. % and 20 wt. %.

Based on the results obtained, the following conclusions can be drawn:

- With an increase in the amount of glass powder in the cement composite mixture, there is a corresponding decrease in bulk density, flexural strength, and compressive strength.
- Both types of plasma treatments applied to waste glass powders had a positive influence on the bulk density of the resulting cement composite, resulting in a 3 % increase.
- Oxygen plasma treatment exhibited the most substantial positive effect on flexural strength, leading to a 13 % increase in flexural strength for samples containing 20 wt. % of surface-treated glass powder.
- Both types of plasma treatment did not significantly impact the resulting compressive strength of the cement composite with surface-treated glass powder content.

Future research endeavors will build upon the findings presented in this article. Subsequent investigations will utilize electron microscopy to elucidate phase changes within the structure of the cement composite and describe alterations in the cement matrix surrounding both treated and untreated glass powder grains.

Acknowledgements

This research has been supported from the Global Postdoc Fellowship Program of the Czech Technical University in Prague and by the Czech Science Foundation project 23-05500S Effect of surface treatments on the performance of silicon-based secondary materials in cementitious composites. The authors would like to extend their gratitude to Recifa a.s., and in particular, to doc. Ing. Martin Lidmila, Ph.D., for providing the valuable samples and materials for this study and M. Mozerov a J. Lorinc for technical assistance in plasma treatment.

List of symbols

f_t the flexural strength [Pa]

f_c the compressive strength [Pa]

$F_{b,\max}$ the maximum force during bending test [N]

$F_{c,\max}$ the maximum force during compressive test [N]

L_s distance between the supports during bending test [m]

a the width of the specimen [m]

b the height of the specimen [m]

C_3S tricalcium silicate

C_2S dicalcium silicate

C_3A tricalcium aluminate

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