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Properties of Cement Mortars with the Addition of Waste Copper Slag Mineralized Using CO_2 – Preliminary Study

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Abstract. In response to the growing amount of copper slag, generated as a by-product of copper smelting and stored in landfills, an attempt was made to use this waste as a valuable partial replacement for natural aggregate in cement mortars. Comparative tests were carried out on four series of mortars designated as: REF – reference, GR – with the addition of copper slag, GR22 and GR50 – with the addition of slag mineralized using CO_2 at temperatures of 22 °C and 50 °C, respectively. The article presents the results of tests of flow diameter and bulk density of fresh cement mixes, flexural tensile strength and compressive strength of hardened mortars, brittleness index, and the microstructure of 7-day-old hardened mortars examined with a scanning electron microscope. Based on the conducted tests, it was demonstrated that replacing part of the natural aggregate with copper slag aggregate caused: a reduction in consistency and an increase in bulk density of fresh cement mixes, an increase in flexural tensile strength in later curing periods and a reduction in compressive strength of hardened mortars, as well as very favourable values of the brittleness index compared to the reference mortar. Based on the obtained results, mineralization of copper slag with CO_2 at 22 °C was considered advantageous.

Key words: cement mortars; copper slag; CO_2 utilization; CO_2 slag mineralization; mortar properties

1. INTRODUCTION

According to [1], Poland is among the leading countries in the world in terms of copper ore extraction. A by-product of copper smelting is copper slag. According to [2], approximately 2.2 tons of slag are generated per ton of copper produced, which is legally classified as waste [3]. The amount of copper slag produced worldwide each year is currently measured in millions of tons, of which about one million tons are generated in Poland [4]. This slag is typically stored in landfills. Considering the growing global demand for copper, further increases in the amount of copper slag deposited in landfills should be expected [5], since its current utilization in industry does not solve the problem. Hence, it is justified to seek new possibilities for its application. One of them may be the use of copper slag as aggregate, serving as a valuable partial replacement of natural aggregate in cement composites. A similar approach was adopted in studies on blast furnace slag, which demonstrated that the fineness and chemical composition of slag determine the properties of fresh and hardened mortars [6]. In the literature, many cases have been reported where mineral additives such as fly ash improve the microstructure and durability of concrete [7]. This is, in many respects, a pro-ecological approach, as it reduces the consumption of non-renewable natural resources, increases the use of waste material while reducing the amount stored in landfills, contributes to lowering the carbon footprint of concrete [8], and limits greenhouse gas emissions [9], among others.

To increase the usability of copper slag in cement composites, its valorisation may be beneficial. Several methods of valorising waste materials have been described, including mechanical, chemical, thermal, biological processes, or combinations thereof [10] [11]. Currently, there is growing interest in mineralization with carbon dioxide for this purpose — a greenhouse gas captured from emission sources and stored in liquid form. Examples of CO_2 utilization in the construction industry include sequestration of CO_2 for curing precast concrete elements through carbonation [12], improving the properties of “old” cement mortar permanently bound to recycled aggregate used in concrete production [13] [14], and the use of CO_2 as an admixture during concrete mixing [15]. The process of chemical activation of copper slag with CO_2 can positively affect the microstructure and some parameters of cement composites made with its use [16] [17]. Similar observations regarding the importance of activation conditions for obtaining favourable mechanical properties have been reported in [18]. Likewise, [19] showed that the combination of fly ash and nanosilica leads to improved microstructure and increased strength of mortars and concretes with these additives. As explained in [17], CO_2 reacts with calcium-rich cementitious materials and forms nanoscale calcium carbonate

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that favourably influences cement hydration. This contributes to the formation of a denser microstructure, which may result in improved mechanical properties and durability of these composites [17] [20].

The selection of appropriate parameters for copper slag mineralization — such as CO_2 solution temperature, CO_2 concentration, and mineralization time — requires further research [21] [9]. Studies are also needed to determine, among other factors, the influence of the amount of copper slag in the mix, its particle size distribution, and maximum grain size on the rheological, mechanical, and microstructural properties of cement mortars and concretes. At present, these remain research gaps. The aim of this study is to conduct preliminary tests and, on their basis, evaluate selected mechanical, and structural properties of cement mortars containing waste copper slag, both untreated and mineralized with CO_2 at two different positive temperatures, in comparison with a reference mortar.

2. RESEARCH PROGRAM

2.1. Materials and mix composition.

Four series of cement mortar specimens were tested, designated as REF, GR, GR22, and GR50. These specimens were prepared from cement mixes also designated as REF, GR, GR22, and GR50, with compositions given in Table 1. Portland cement CEM I 42.5R ensuring higher repeatability and clearer interpretation of the observed effect was used as the only binder. Quartz sand with grain sizes of 0.5–1.4 mm, granulated, amorphous copper slag (Table 2) with grain sizes of 0.063–1.0 mm, and a plasticizer were used for the mixes.

For the REF mix, quartz sand was used as the only aggregate, while for the other mixes, aggregates consisted of both quartz sand and granulated copper slag. In the case of mix GR, non-mineralized copper slag was used, whereas the slag used for mixes GR22 and GR50 underwent mineralization.

TABLE 1. Components and designation of cement mixes

Mix components	Mix designation			
	REF	GR	GR22	GR50
Cement CEM I 42,5R [kg/m^3]	300	300	300	300
Quartz sand [kg/m^3]	880	750	750	750
Granulated copper slag [kg/m^3]	0	130	130	130
Plasticizer 3 % [kg/m^3]	9	9	9	9
Water [kg/m^3]	120	120	120	120
w/c [-]	0.4	0.4	0.4	0.4

TABLE 2. Chemical composition of copper slag [%]

SiO_2	CaO	Fe_2O_3	Al_2O_3	MgO	K_2O	Na_2O	ZnO
34.85	21.58	17.37	12.56	5.92	3.22	1.61	1.18

2.2. Mineralization of Copper Slag with CO_2

Granulated copper slag, dried to constant mass at 105 °C, was subjected to mineralization. Portions of 250 g of slag were poured into a 2-liter glass vessel and mixed with 1 litre of 0.3 M sodium hydroxide solution. Two gases were introduced into the vessel via hoses: carbon dioxide at a flow rate of 8 L/min and nitrogen at a flow rate of about 4 L/min. The mixture was continuously stirred using a laboratory stirrer. The mineralization process was carried out for one hour, with the pH decrease rate controlled by diluting the CO_2 stream with nitrogen. After mineralization, the vessel contents were filtered to separate the liquid and then dried in a laboratory oven to constant mass at 105 °C. The slag used for mix GR22 was mineralized at a temperature of 22 °C ± 1 °C, while that used for mix GR50 was mineralized at 50 °C ± 1 °C. The applied mineralization approach was based on previously reported aqueous carbonation methods for alkaline industrial residues [22] [23], in which NaOH provides a strongly alkaline environment that enhances partial dissolution of the amorphous (glassy) phase of copper slag, enhancing the release of calcium ions, facilitating subsequent carbonation reactions leading to $CaCO_3$ precipitation. The mineralization procedure was carried out in an aqueous system, which is commonly applied in laboratory studies on CO_2 mineralization of alkaline industrial residues, as the alkaline solution enhances CO_2 dissolution and facilitates the formation of carbonate ions that react with calcium released from the slag to form $CaCO_3$.

2.3. Test methods

The density and water absorption of slag were determined in accordance with PN-EN 1097-6:2013 [24]. After mixing the components, the flow diameter of all four mixes (REF, GR, GR22, and GR50) was determined using a flow table. The bulk density of these mixes was also measured. Next, prismatic specimens with dimensions of 40 × 40 × 160 mm were cast in moulds. After 24 hours, the specimens were demoulded and placed in water at 22 °C ± 1 °C, where they were stored until testing.

After 3, 7, and 28 days of curing (counted from the time of casting), flexural tensile strength and compressive strength tests were carried out in accordance with PN-EN 1015-11 [25]. For each testing age, three prisms from each series were tested for flexural strength, and six prism halves were tested for compressive strength (series REF, GR, GR22, GR50). Based on the strength test results, a coefficient characterizing the brittleness of mortars was also calculated as the ratio of flexural strength to compressive strength. For each series, average strength values and standard deviations were determined. In addition, after 7 days of curing, microstructural tests of mortars from series GR, GR22, and GR50 were performed. The prepared samples were imaged using a JEOL JSM-6610LV scanning electron microscope (SEM) in high-vacuum mode, with an accelerating voltage of 20 kV, a working distance of 20 mm, and a backscattered electron (BSE) detector in compositional mode to reveal so-called Z-contrast features. The carbonation degree of slag and the amount of hydration products after 7 days was analysed using thermogravimetric

analysis (PerkinElmer TGA 8000) under air conditions, in the temperature range from 30 °C to 1000 °C, at a heating rate of 10 °C/min.

3. RESULTS AND DISCUSSION

Figure 1 presents the results of densities and water absorption of copper slag. Comparing the dry material density and water absorption with the literature review [26], it can be concluded that the slag meets the requirements for high-quality aggregate (oven-dried material density ≥ 2.5 g/cm^3, water absorption ≤ 3.0%). It should be noted that these parameters refer to the untreated copper slag.

Figure 2a presents the results of flow diameter tests for cement mixes REF, GR, GR22, and GR50. For mix REF, the diameter was 150 mm, while for the other mixes it was smaller and equalled 120 mm. The results indicate that mineralization of copper slag with CO_2 did not affect the consistency of the mixes. Figure 2b shows the results of bulk density tests for mixes REF, GR, GR22, and GR50. The lowest bulk density was recorded for mix REF, amounting to 2224 kg/m³. Partial replacement of quartz sand with copper slag in mixes GR, GR22, and GR50 resulted in increased bulk density. This increase compared to the reference mix may be related to the replacement of aggregate in the 0.5–1.4 mm fraction with finer copper slag particles in the 0.063–1.0 mm fraction, which can improve packing density.

However, mineralization of copper slag with CO_2, compared to mix GR, caused a slight decrease in bulk density. This effect may be associated with the formation of carbonate phases (mainly $CaCO_3$) during mineralization, which can modify the surface texture of the slag particles and potentially increase their micro-porosity.For mixes GR22 and GR50, the bulk density values were 2324 kg/m³ and 2356 kg/m³, respectively, while for mix GR it was 2388 kg/m³.

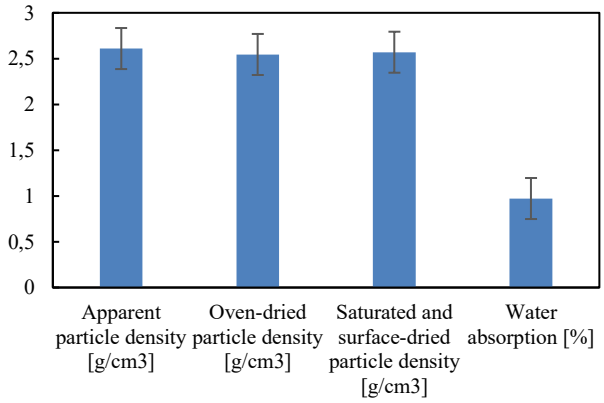

Fig.1. Results of densities and water absorption of copper slag

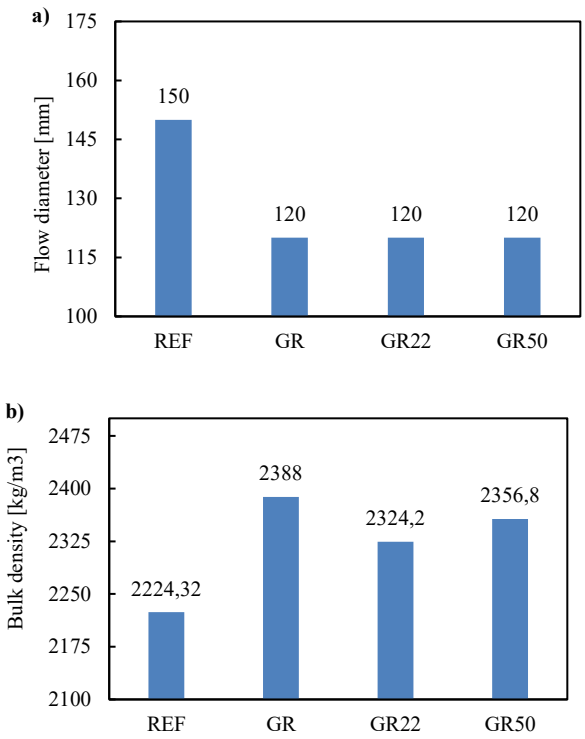

Fig.2. Results of cement mix tests for REF, GR, GR22, and GR50: a) flow diameter b) bulk density

Table 3 presents the results of flexural tensile strength and compressive strength tests obtained after 3, 7, and 28 days of curing for specimens from series REF, GR, GR22, and GR50. The average values are also illustrated in Fig. 3.

As shown in Fig. 3a, the incorporation of untreated copper slag (GR) resulted in a reduction in flexural tensile strength at early ages compared to the reference mortar. However, the mineralization treatment improved this parameter. In particular, the GR22 series exhibited higher flexural strength than GR at all curing times and approached the values obtained for the reference mixture. After 28 days, the highest flexural tensile strength among the modified mixtures was observed for GR22, indicating that the carbonation process carried out at 22 °C positively influenced the flexural performance of the composite. The GR50 series showed intermediate results, remaining close to the reference values but generally lower than GR22.

A similar trend can be observed for compressive strength (Fig. 3b). The addition of untreated copper slag caused a noticeable reduction in compressive strength compared to the REF series, particularly at early curing stages. Nevertheless, the decrease became less pronounced with increasing curing time. The carbonation treatment improved the compressive strength compared to the untreated slag mixture. Among the modified materials, the GR22 series consistently achieved higher strength values than GR and GR50 throughout the investigated curing period.

Based on the analysis of the obtained test results, it can be observed that the incorporation of copper slag in mortars GR, GR22, and GR50 initially reduced their flexural tensile strength

compared to the reference mortar. However, after 7 and subsequently after 28 days, this strength was higher than that of the REF mortar. It can also be concluded that mineralization of copper slag with CO_2 at 22 °C had a positive effect on flexural tensile strength. For mortar GR22, this strength after both 7 and 28 days of curing was the highest compared to mortars REF, GR, and GR50. This improvement may be attributed to modifications of the slag particle surface induced by the carbonation process. The formation of carbonate crystals, such as calcite or aragonite, increases the surface roughness of the particles and enhances the interaction between the aggregate and the cement matrix within the interfacial transition zone. In combination with the progressive densification of the cement matrix during curing, these microstructural changes may improve stress transfer in the composite. From a mechanistic perspective, this effect may act similarly to dispersed nano-scale reinforcement, which contributes to the observed increase in flexural performance [27] [28].

The results indicate that although the use of copper slag reduces compressive strength, the mineralization process partially mitigates this effect. The improvement observed for the GR22 mixture suggests that the carbonation conditions applied at 22 °C promote favorable modifications of the material, which positively affect strength development. It is also worth noting that the reduction in compressive strength relative to the reference mixture decreased with curing time, which raises the question of how these materials would perform over longer curing periods (e.g., 90 or 180 days). As indicated in [29], curing conditions and duration play a crucial role in the full development of the mechanical properties of cement-based composites. Although the present study concerns Portland cement-based systems, previous research on alkali-activated slag concretes has also demonstrated that curing temperature significantly affects strength development, albeit through different chemical mechanisms [30].

TABLE 3. Results of flexural tensile strength and compressive strength tests for specimens from series REF, GR, GR22, and GR50

Flexural tensile strength [MPa]				Compressive strength [MPa]		
Series	Mean value $f_{ct,t}$	Standard deviation S_d	Coeffi. of variation v [%]	Mean value $f_{ct,t}$	Standard deviation S_d	Coeffi. of variation v [%]
REF (3d)	5.00	0.27	5.4	32.14	2.71	8.4
GR (3d)	4.06	0.27	6.7	20.21	0.95	4.7
GR22 (3d)	4.69	0.00	0.0	25.84	0.96	3.7
GR50 (3d)	4.85	0.27	5.6	24.38	0.63	2.6
REF (7d)	5.31	0.27	5.1	37.92	2.61	6.9
GR (7d)	5.63	0.00	0.0	29.38	2.26	7.7
GR22 (7d)	5.94	0.27	4.6	34.58	0.72	2.1
GR50 (7d)	5.31	0.27	5.1	30.21	0.95	3.2
REF (28d)	5.94	0.72	12.1	42.29	0.72	1.7
GR (28d)	6.56	0.47	7.1	35.63	1.65	4.6
GR22 (28d)	6.88	0.27	3.9	35.83	1.57	4.4
GR50 (28d)	6.09	0.47	7.7	30.42	4.02	13.2

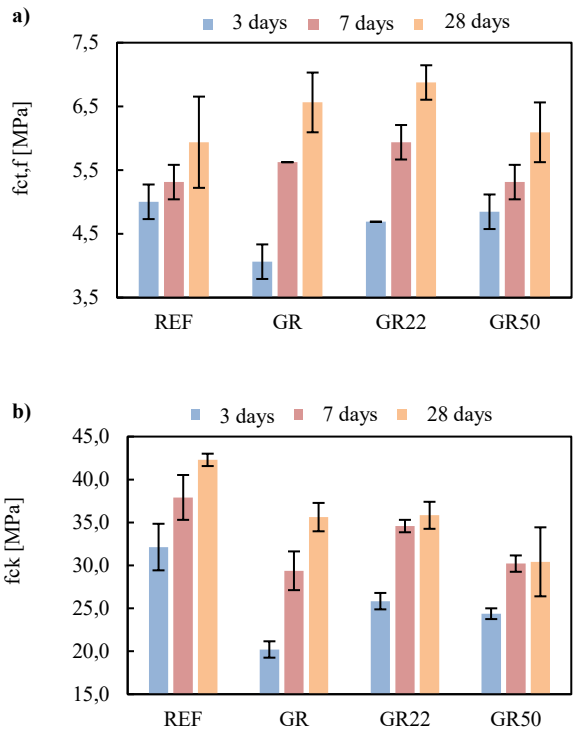

Fig.3. Average strengths of specimens from series REF, GR, GR22, and GR50: a) flexural tensile strength b) compressive strength

Based on the experimentally obtained results, namely the average flexural tensile strength f_{ct} and the average compressive strength f_{ck}, the brittleness index k was calculated for the tested mortars using the following relationship (1):

$$k = \left[\frac{f_{ct}}{f_{ck}}\right] * 100\%. \quad (1)$$

The results of these calculations are illustrated in Fig. 4. Their analysis shows that in the case of mortars GR, GR22, and GR50, made with copper slag, the brittleness index k is very favourable. The values of this coefficient are significantly higher than those for the reference mortar REF, both after 3, 7, and 28 days of curing. Specifically: after 3 days, they were higher by 25%, 12.5%, and 25% for mortars GR, GR22, and GR50, respectively; after 7 days, they were higher by 36%, 21%, and 29% for mortars GR, GR22, and GR50, respectively; and after 28 days, they were higher by 29%, 43%, and 43% for mortars GR, GR22, and GR50, respectively.

It should be noted that the increase in the brittleness index is mainly related to the change in the ratio between flexural and compressive strength, particularly due to the reduction in compressive strength observed in mortars containing copper slag.

It is also noteworthy that in the case of mortars GR22 and GR50, made with copper slag mineralized using CO_2, the brittleness index does not decrease with curing time but rather increases.

However, this effect should be interpreted primarily because of the relative relationship between compressive and flexural strength rather than a direct improvement in overall mechanical

performance. Therefore, the obtained results indicate a change in the mechanical response of the composites containing copper slag, reflected in the modified ratio of flexural to compressive strength.

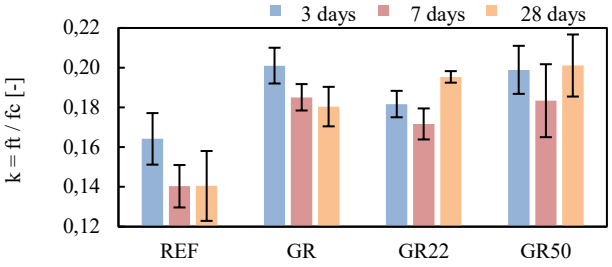

Fig.4. Brittleness index values for tested mortars REF, GR, GR22, and GR50 after 3, 7, and 28 days of curing.

Figures 5, 6, and 7 present preliminary microscopic images of the microstructure of 7-day-old mortars GR, GR22, and GR50, obtained using a scanning electron microscope (SEM). In the images shown in Figures 5a, 6a, and 7a, copper slag is easily identifiable by its bright color in the BSE detector, as it is characterized by a higher average Z value (heavier elements). The dark areas in these images correspond to pores. The figures show that hydration products tightly fill the mortar microstructure. The interfacial transition zone between slag and cement paste is most visible in mortar GR (Figure 5a) and is the densest in mortar GR22 (Figure 6a). In mortars GR22 and GR50, a denser structure at the slag–cement paste interface was observed compared to mortar GR. This effect may be associated with the mineralization process carried out prior to mortar preparation. During CO_2 mineralization, carbonate phases form on the surface of the slag particles, modifying their surface characteristics. The formation of these products can increase surface roughness and promote improved mechanical interlocking between the slag particles and the cement paste, resulting in a locally denser interfacial transition zone. The chemical composition analysis of mortars GR, GR22, and GR50 (Figures 5b, 6b, and 7b) shows copper slag as a material rich in many chemical phases such as Al, C, Ca, Cu, Fe, K, Mg, Na, O, and Si. The purple-colored areas in these images correspond to regions where unreacted cement grains are still present. Chemically, the interfacial transition zone between slag and cement paste consists of the same phases in mortars GR, GR22, and GR50, except that in mortar GR50, Cu is absent. The boundaries of elemental occurrence in the mortar are clearly marked, suggesting that in 7-day-old mortar these elements have not yet migrated across the interfacial zone. Previous studies also indicate that copper slag is mainly composed of relatively stable phases and can therefore be successfully used in cement-based composites without causing detrimental volumetric changes [30][31]. The TG/DTG curves after 7 days (Fig. 8) show clear differences in the thermal decomposition between the GR, GR22 and GR50 samples. GR is characterised by the lowest mass loss and the weakest DTG peaks, indicating a lower amount of hydration products. GR22 shows moderate thermal activity, while GR50 is characterised by the greatest weight loss and the most pronounced DTG peaks, indicating the highest degree of hydration and the highest content of chemically bound water. Increasing the proportion of the additive in the mixture therefore leads to a more intense hydration reaction and a greater number of products susceptible to thermal decomposition. The TG/DTG analysis was performed on hardened mortars; therefore, the observed mass loss reflects both the carbonation of cement hydration products and carbonate phases formed during slag mineralization. Consequently, it is not possible to precisely quantify the CO_2 uptake attributable solely to the slag. Future studies should include direct TG analysis of slag before and after mineralization. Based on the research approach described in article [32], the degree of carbonation of copper slag was calculated (Eq. 2 and Table 4) where $\Delta m_{550\text{-}800)}$ is the mass loss in the temperature range 550–800 °C associated with carbonate decomposition, and m_{850} is the sample mass at 850 °C. The analysis showed that the amount of captured CO_2 depends on the temperature at which carbonation takes place. The carbonation degree should be considered together with the mechanical and microstructural performance of the composite. Although relatively low values were obtained, the observed improvements in strength and microstructure indicate that even limited carbonation can positively influence the properties of cement mortars. Researchers [32] have shown that at higher temperatures, aragonite crystals form instead of calcite crystals. It can therefore be assumed that, in terms of the use of carbonated materials in cement composites, the formation of calcite is more desirable at the expense of reducing the content of bound CO_2.

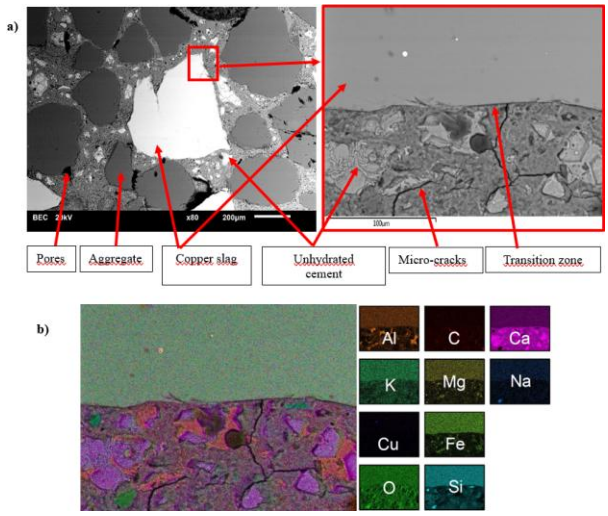

Fig.5. SEM imaging of a 7-day-old GR mortar specimen: a) mortar components and slag–cement paste interface b) chemical composition.

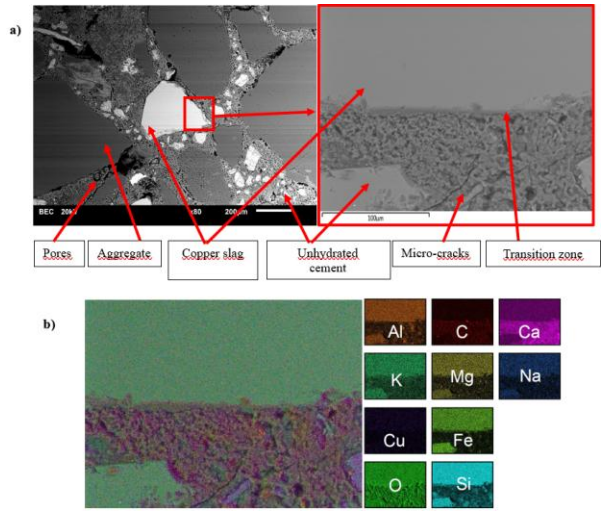

Fig.6. SEM imaging of a 7-day-old GR22 mortar specimen: a) mortar components and slag–cement paste interface b) chemical composition

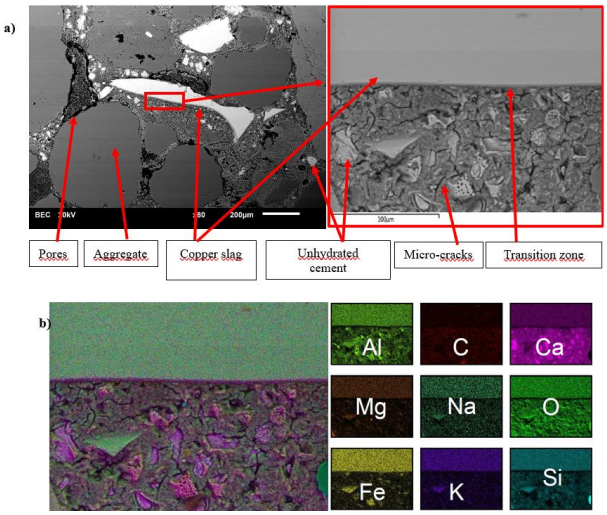

Fig.7. SEM imaging of a 7-day-old GR50 mortar specimen: a) mortar components and slag–cement paste interface b) chemical composition

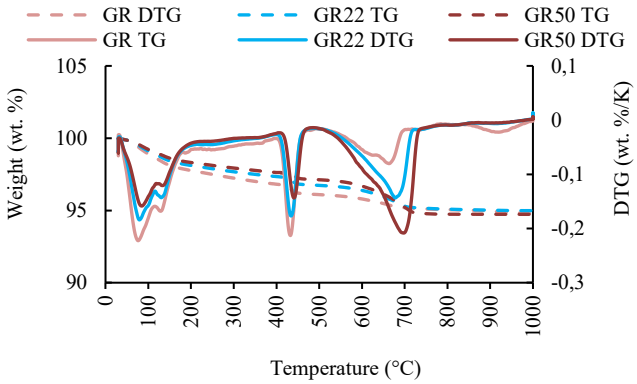

Fig.8. TG and DTG analysis after 7 days

$$Carb.degree\ of\ copper\ slag\ (\%) = \frac{\Delta m_{550-800}}{m_{850}} \qquad (2)$$

TABLE 4. Carb.degree of copper slag [%]

Aggregate	GR 22	GR 50

4. CONCLUSIONS

Based on the preliminary tests carried out, the following conclusions were formulated:

- Replacing part of the quartz sand with copper slag in mortar GR reduced its flow diameter to 120 mm compared to the reference mortar REF, for which the diameter was 150 mm. Mineralization of the slag with CO_2 at 22 °C (mortar GR22) and 50 °C (mortar GR50) did not cause a further decrease in the flow diameter compared to mortar GR. This indicates that mineralization of copper slag with CO_2 does not negatively affect the consistency of the tested cement mixes.
- Replacing part of the quartz sand with copper slag in mortars GR, GR22, and GR50 resulted in increased bulk density compared to the reference mortar REF. The highest bulk density of 2388 kg/m³ was obtained for mortar GR. Slightly lower densities were recorded for mortars GR22 and GR50, 2324 kg/m³ and 2357 kg/m³, respectively. The lowest bulk density of 2224 kg/m³ characterized the reference mortar REF.
- The flexural tensile strength of cement mortars GR, GR22, and GR50 containing copper slag in the amount used in this study was lower after 3 days of curing compared to the reference mortar REF. However, after 7 and 28 days of curing, it was higher compared to REF. The highest average values of this strength were obtained for mortar GR22, 5.94 MPa and 6.88 MPa after 7 and 28 days of curing, respectively. By comparison, the average flexural tensile strength of reference mortar REF was 5.31 MPa after 7 days and 5.94 MPa after 28 days of curing. On this basis, it can be concluded that the presence of copper slag in cement mortar positively affects its flexural tensile strength after 7 or more days of curing. As shown by the tests, the best effect in this regard was achieved with mineralization of slag using CO_2 at 22 °C.
- The compressive strength of cement mortars GR, GR22, and GR50 containing copper slag in the amount used in this study was lower compared to the reference mortar REF after 3, 7, and 28 days of curing. However, the compressive strength of mortar GR22 was higher after 3, 7, and 28 days of curing, and that of mortar GR50 after 3 and 7 days of curing, compared to mortar GR. On this basis, it can be concluded that mineralization of copper slag in cement mortar with CO_2 favourably influences compressive strength. As shown by the tests, the best effect in this regard was obtained with mineralization at 22 °C.
- The brittleness index for cement mortars GR, GR22, and GR50 containing copper slag was higher after 3, 7, and 28 days of curing compared to the reference mortar REF. For example, after 3 days it was higher by 25% for mortar GR, 12.5% for mortar GR22, and 25% for mortar GR50. After 28 days it was higher by 29% for mortar GR, 43% for mortar GR22, and 43% for mortar GR50 compared to mortar REF. This change results from the modified relationship between flexural and compressive strength in mortars containing copper slag.

- Microstructural analysis showed that copper slag used in mortars did not adversely affect the morphology of the cement matrix. Denser interfacial transition zone was observed, likely due to the presence of carbonate phases $CaCO_3$ during mineralization. The chemical composition of the slag, determined independently, indicates the presence of oxides such as SiO_2, CaO, and Fe_2O_3, which contribute to its behaviour in cement composites.

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