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Chemistry of Composite Cements; Interaction between Blast Furnace Slag and Natural Pozzolan in Portland Cement Mixtures

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Abstract: Composite cement has several technical, environmental, and economic advantages. Thus, we investigated the properties of a new composite cement composed of four components, including ground granulated blast-furnace slag (GGBFS) from Isfahan Steel Co., pozzolan from Rafsanjan Pozzolan Mine, Portland cement type II from Sepahan Cement Co., and limestone filler. At first, the chemical and physical properties and activity of the above components were determined. Eight composite cements were prepared and their physical and chemical properties were characterized. After compressive strength test (1 to 90 days), measurement of flowability and setting time, a composite cement made of 20% slag, 10% pozzolan, and 2.5% limestone was selected for additional investigations. After 120 days of curing, SEM, XRD, and TGA testing on cement pastes made of the proposed blend, the control cement, Portland-blast-furnace slag cement (30% slag), and Portland-pozzolan cement (30% pozzolan) were investigated. The blast-furnace slag improved the long-term compressive strength of the composite cement, provided a greater flow to the mortar, and displayed less permeability. In contrast, the pozzolan offered essential benefits, including proper initial strength, suitable setting time, and good grind-ability. The standard quality tests do not evaluate the durability of concrete containing a composite cement, while our study is based on the hydration reactions that occurred in the composite cement for four months. The microscopic investigation and TGA tests further revealed that the pozzolan additive reduces the Portlandite content of the composite cement paste. The XRD results indicated that the slag had few crystals, whereas numerous crystals were observed in the pozzolan sample, potentially reducing the compressive strength in the composite cement. The SEM images displayed that the pozzolan crystals have not reacted with water after 120 days in the composite cement. The experimental results revealed that the slag and pozzolan positively affect the properties of composite cement and improve its capabilities.

Keywords: Portland cement, Composite cement, Slag, Natural pozzolan, Concrete, Limestone

1. INTRODUCTION

Cement is the second largest CO_2 emitting industry and the third-largest energy-consuming industry globally. Direct carbon emissions from the cement industry are expected to rise by 4% globally by 2050 under the IEA Reference Technology Scenario (RTS). The production of composite cements is an effective strategy to reduce greenhouse gases emission and energy consumption, especially thermal energy. For example, in a study with 30% replacement of slag, energy consumption was reduced by about 24%, and if the proportion of slag in cement reaches 70%, energy consumption will be reduced by 60%.[1-6] In addition to environmental benefits and energy consumption reduction, the use of supplementary cementing materials (SCMs) reduces the production cost of cement and improves the performance of concrete. In mass concrete, blended cement slows down the heat release. When used at appropriate rates, SCMs can increase durability and even improve compressive strength. Using SCMs in concrete mixtures has been growing in North America since the 1970s.[7-15] Many of these materials are byproducts of other industrial processes. Their judicious use is desirable for their sustainability, given the environmental and energy conservation; and for the performance benefits they provide concrete. SCMs are used in at least 60% of ready mixed concrete in the USA.[16-24] The final strength, durability, setting time, and concrete workability change under the influence of these materials.[25-37]

Binary blended cement has been produced for many years as slag Portland cement and pozzolan Portland cement. Four groups have two or more components of the five cement groups defined in EN 197-1.[37-43] Composite cement has been the subject of interest for researchers, such as improving the properties of geopolymer cement by combining slag and pozzolan.[9-11] Salazar et al. have made a repair mortar by adding slag (30%) to natural pozzolan (70%) and using alkaline activators (e.g., soda and glass water) that can help the structure durability by coating on mortar.[10-14] The results show that the ternary composition of composite cement at higher percentages of slag replacement or moderate amounts of fly ash replacement with a small amount of micro-silica has beneficial effect on the alkali-silica reaction (ASR). Also, long-term electrical resistance, shrinkage, and compressive strength are more desirable. However, short-term compressive strength decreases, which was recommended to reduce this shortcoming by lowering the water- to-cement ratio, due to the more suitable slump of three-component cement compared to Portland cement.[11-15] In another study, combination of three components of type II Portland cement, Rafsanjan pozzolan, and limestone in concrete provided a reduction in setting time of concrete containing composite cement and less expansion than Portland cement. In all samples, the compressive strength (at the ages of three to 90 days) of composite cement concrete was lower compared to the Portland cement. Increasing the percentage of the pozzolan (when the percentage of lime was about 10%) reduced the capillary water absorption of concrete.[44-58]

The positive effect of slag in combination with pozzolan in geopolymer cements has been investigated. In another study, the synergistic effect of slag (30%) on natural pozzolan (70%) in geopolymer cements was studied. Allahverdi et al. carried out an investigation to optimize the strength of pozzolan of Taftan geopolymer cement by adding slag from Isfahan Steel Co.[12-16]

The previous studies often focused on the composition of fly ash and slag or on the properties of pozzolan and slag in concrete (especially geopolymer concretes). In the present research, the chemical aspects and hydration reactions were considered, and new methods were used to study the microstructure of composite cements, which were cured for extended periods. We also discussed here the role of pozzolan in reducing the Portlandite phase of the composite cement (especially in slag cements), which has received less attention. This feature is effective in lowering the concrete efflorescence.

In this study, composite cements were prepared using granulated blast-furnace slag from Isfahan Steel Co., and pozzolan from Rafsanjan mine (northwest of Kerman province, Iran) to investigate the behavior of these two materials in cement mortar. The chemical and physical properties of the cement samples were measured according to the ASTM standard test methods.[38-57]

2. EXPERIMENTAL

Materials and methods

The materials were prepared from the following sources:

A- Type II Portland cement (base cement) from mill number 3 of Sepahan Cement Co. (closed circuit ball mill), including 95.5% of type II clinker and 4.5% of natural gypsum.

B-Ground granulated blast-furnace slag from Isfahan Steel Co. (GGBFS) (a by-product from Isfahan Steel Co., Furnace No. 3) which was cooled quickly with high pressure water, then dried in Sepahan Cement Company, and ground with Ball Mill No. 2 of this company Blaine fineness of 4300 cm^2/g.

C- Pozzolan from Rafsanjan Pozzolan Mine was pulverized in a laboratory mill to a Blaine fineness of about 4200 cm^2/g.

D- Esteghlal Mine limestone (as the filler) was ground with a laboratory ball mill to a fineness similar to the Portland cement.

Chemical analyses of the raw materials were performed according to ASTM C114, and the results are presented in Table 1. The results of base cement (Portland type II) comply with the requirements of ASTM C150, slag with EN 197-1 and ASTM C595, limestone filler with EN 197-1, and the natural pozzolan with ASTM C618.

The physical properties of four materials are given in Table 2. The activity of slag powder (GGBFS) according to ASTM C989 and pozzolan according to ASTM C618 were tested, which satisfied the requirements of these standards (Table 2). The slag grade is 80, according to ASTM C989. Portland cement was prepared from a mixture of the base cement and limestone powder (2.5%). The physical properties of this cement were measured according to the ASTM methods which are presented in Table 3.

Mixing proportions

The composite cement in this study complies with the requirements of CEM II/BM cement in EN 197-1 and is close to the specifications of CEM V/A (slag-pozzolan composite cement) in EN 197-1.

For the two materials milled in the laboratory, it was tried to adjust the Blaine fineness of the natural pozzolan to fineness as close as possible to the slag and to adjust the Blaine fines of the limestone filler to a fineness as close as possible to the Portland cement, to minimize the effect of fineness (Table 2). These powders were mixed vigorously in a laboratory mixer with the proportions shown in Table 4, and then the tests of compressive strength, flow resistance, setting, and permeability were performed on the fresh and hardened cement pastes.

Table 1. Chemical compositions of the materials (% Wt.)

Oxide	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**CaO**	**MgO**	**SO_3**	**Na_2O**	**K_2O**	**IR**	**LOI***	
Portland Cement (type II)	21.00	4.69	3.84	64.40	2.32	2.01	0.21	0.58	0.20	1.16	$C_3S^{**} = 58.80$ $C_3A^{***} = 6.17$
GGBFS	35.80	11.78	0.48	37.38	8.80	0.72	0.60	0.66	0.21	0.27	$S^{2-} = 0.75$ $(CaO+MgO)/SiO_2 = 1.29 > 1$
Natural pozzolan	60.1	16.97	3.21	6.22	2.49	0.33	1.34	1.80	74.55	7.72	
Limestone	0.31	0.30	0.12	55.60	0.24	0.28	0.10	0.10	0.05	43.73	

*Loss on ignition. **$C_3S = 3CaO.SiO_2$. ***$C_3A = 3CaO.Al_2O_3$.

Table 2. Physical properties of the materials

Materials	Portland cement (type II)	GGBFS	Limestone	Natural pozzolan	Standard method (ASTM)
Blaine (cm^2/g)	3590	4310	3650	4180	C 204
% Residue on sieve 45 micron	5.4	3.2	17.0	24.0	C 430
Density (g/cm^3)	3.12	2.99	2.66	2.51	C 188
Activity at 7 days (%)		60%			C349
Activity at 28 days (%)		85%		87%	C349

Table 3. Physical properties of the Portland cement (Portland cement + 2.5% lime)

	% Autoclave expansion	% Normal consistency	Initial setting time(min)	Final setting time(min)
Standard method (ASTM)	C151	C187	C191	C191
Content	0.01	24	199	275

Compressive Strength (kg/cm^2)- EN 196-1

Day	1	3	7	28	56	90
Content	139	322	449	552	608	645

Table 4. Sample code and composition percentage of the pozzolan and slag mixtures

ID	Portland cement (type II)	Limestone	GGBFS	Natural pozzolan
S0P0	97.5	2.5	0	0
S30P0	67.5	2.5	30	0
S25P5	67.5	2.5	25	5
S20P10	67.5	2.5	20	10
S15P15	67.5	2.5	15	15
S10P20	67.5	2.5	10	20
S5P25	67.5	2.5	5	25
S0P30	67.5	2.5	0	30

Characterization

The instrumental analyses were carried out using a Quanta 200 scanning electron microscope (SEM) equipped with a dispersive X-ray analysis (EDS) detector, SETARAM thermal gravity analyzer (TGA), and X-ray diffraction (XRD) by Bruker mode D8ADVANCE.

3. RESULTS AND DISCUSSION

Compressive strengths of the composite cement

The compressive strength of the composite cement in 4 × 4 × 16 cm^3 prisms according to ASTM C349 (Figs. 1 and 2) was improved in 7 to 90 days with higher amounts of the slag in the composite cement. However, the early- age compressive strength of the composite cement containing pozzolan was slightly higher in the initial strength. The ratio of water to cement was kept constant.

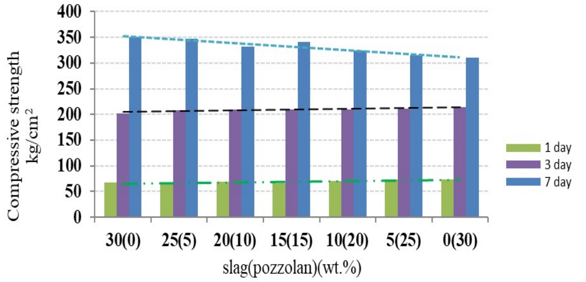

Fig. 1. Compressive strength of 1 to 7 days of the composite cement.

The hardening reaction of a composite cement mortar depends on its components.[15] The slag participates in the hardening reaction after seven days, while the pozzolanic reaction initiates with a delay of four weeks (Figs. 1 and 2). The results of compressive strength of the composite and mixed cement during three months in Fig. 2 show that the rate of hardening reactions of the composite cement pastes containing more slag is greater. For example, the compressive strength of a mixed cement mortar which contained 30% slag was thirty percent greater than the strength of the blended cement which contained 30% pozzolan. While after 90 days, the difference between both pastes was 26% (Fig. 2). The hardening reaction of blended and composite cements continued after 28 days. After eight weeks, the compressive strength of mixed cement which contained 30 % slag became greater than the Portland cement mortar (Fig. 3).

Flow-table test of the composite cement

To investigate the effect of the composite cement composition on the flow, the water-to-cement ratio was kept constant at 0.485 according to the minimum ratio specified in ASTM C230 and ASTM C1437.[16-17] The flow rate of the base cement in this test is 90 mm, and the results of other cements are plotted in Fig. 4. The composite cements with more than 15% slag showed an increase in the flow rate.

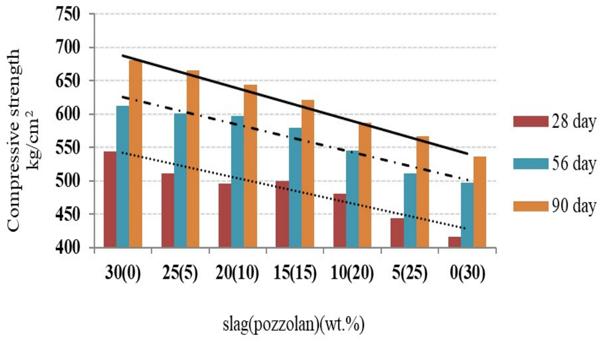

Fig. 2. Compressive strength of 28 to 90 days of the composite.

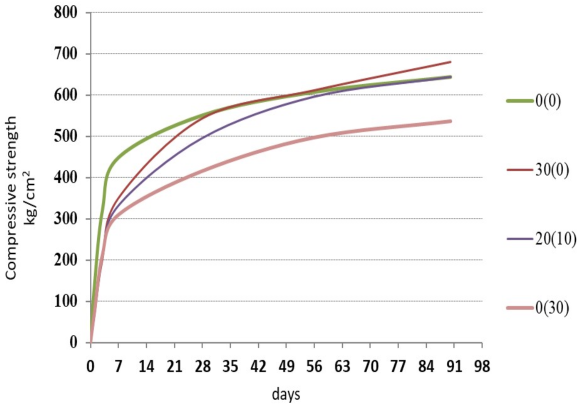

Fig. 3. Comparison of the growth of compressive strength of the selected base cement 20 (10) containing 20% slag (10% pozzolan) with Portland cement 0 (0), Portland slag cement 30 (0), and Portland pozzolan cement 0 (30) up to 90 days.

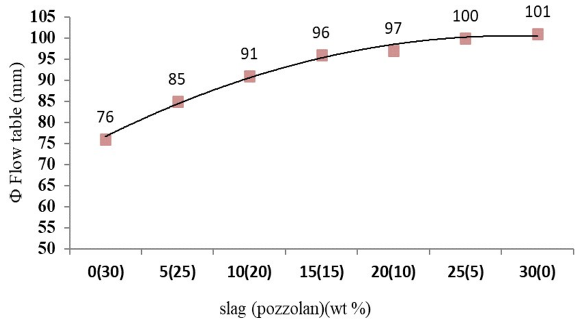

Fig. 4. Flow of the composite cements with a fixed water-to-cement ratio.

The shape and dimensions of the particles forming the composite cement affect the cement mortar and concrete flow behavior;[15] therefore, the change in the flow can be attributed to the shape and dimension of the slag and the natural pozzolan. The porous structure of natural pozzolan remains intact even after pulverization. Therefore, in the first minutes when water becomes in contact with the powder, the porous structure of the natural pozzolan absorbs a portion of the water in the mixture. In contrast, the slag particles are powder shaped, glassy, and irregular. So in the first minutes, they do not absorb water as much as the natural pozzolan particles and allow the flowability of the mortar. Slag has a beneficial effect on concrete workability.[15]

Setting time of the composite cement

The water content was measured according to ASTM C187, and the initial and final setting times were performed according to ASTM C191. In this study, the amount of water was adjusted to about 25.5% to minimize its impact on the setting time. Pozzolan and slag delay the setting time of cement and concrete.[18] Several other factors, including temperature, cement fineness, and sulfate content affect the setting time.[19] The initial and final setting time for the base cement were measured to be 170 and 195 minutes, respectively. The setting times of the composite cements in Fig. 5 show that with the fixed water-to-cement ratio, the setting time of control cement increases with increasing the slag percentage. Whereas higher percentages of pozzolan decrease the setting time. This difference can be explained by considering the microstructure of slag and pozzolan powder. If the amount of water is constant, a substantial portion of water is absorbed through the pores of pozzolan so that less water is available for cement. In contrast, slag does not absorb water; thus, more water is exposed to the cement, and more time will be taken to cause hardening in the slag cement. This excess water causes the setting time of slag-containing cement to be longer, and the gap between the initial setting time and the final setting time (surface hardening) is extended.

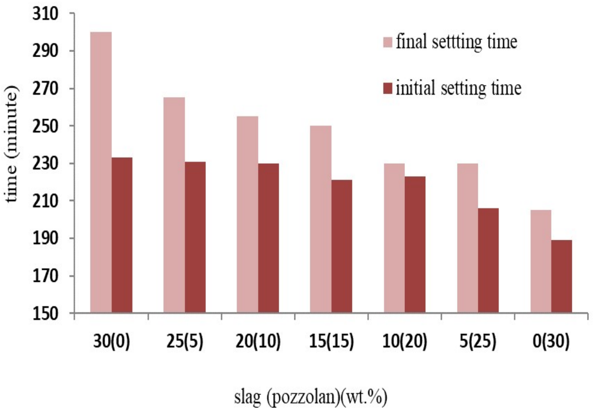

Fig. 5. Setting time of the composite cements using the Vicat needle method.

Density of the hardened composite cementpaste

The bulk density, absorption, and voids of the hardened mortar of composite and Portland cement were measured according to ASTM C642[20] after 120 days of curing. The mortar was similar to the mortar used in the compressive strength test. The bulk density diagram is presented in Fig. 6. The mortar made with the Portland cement had the lowest water absorption and the greatest density. After the Portland cement, the composite cement containing a higher proportion of slag possesses higher density and lower water absorption. But why is there a decreasing trend in the density of hardened mortar in Fig. 6 as the slag percentage decreases? The reason for this difference is initially due to the higher density of slag powder (2.99 g/cm^3) than the density of pozzolan powder (2.5 g/cm^3). The lower density of the slag and pozzolan compared to the Portland cement can also be an advantage for production of light-weight concrete.

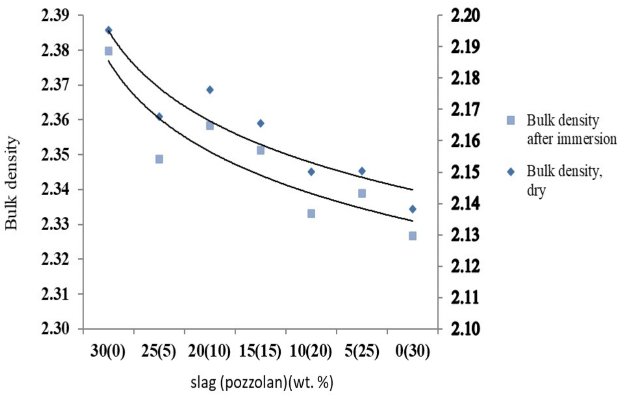

Fig. 6. Percentage of the volume of permeable cavities of hardened mortar after 120 days.

For the following studies, a sample containing 20% slag and 10% pozzolan was selected from the composite cement. The reason for choosing this sample was the better performance of this composite cement in the other tests. Additional tests were performed on four samples, including slag-Portland cement (with 30% slag), Portland-pozzolan cement (with 30% pozzolan), Portland cement, and a blend (containing 10% pozzolan and 20% slag). For this purpose, the cement pastes were prepared with 25% water. The samples were then cured for 120 days at 20° C and at a humidity above 90%.

SEM/EDX study

Figure 7 compares the microscopic structure of the four hardened cement pastes. The SEM images were taken with an electron microscope at 400 to 600x magnification. The unreacted particles were observed in three samples, including paste made with Portland cement, slag, and natural pozzolan. In the Portland cement paste, the unreacted particles were coarse Alite and Bilite. The glass grains and coarse slag were observed in the slag cement paste, while in the pozzolan cement paste, calcite and inactive clay particles were seen. All of these features could also be seen in the composite cement paste. This is one of the reasons for the strength difference observed in the samples made with these three cements mentioned above.

The EDX elemental analysis of the four composite cements revealed that slag and pozzolan changed the chemical composition of cement pastes (see Figs. S1-S9 and Tables S1-S5 in SI). Furthermore, the ratio of calcium to silica changed in all four cement pastes. The lowest and highest ratio of calcium to silica belonged to the slag cement and Portland cement, respectively.[15] Thus, the microscopic investigation suggests that in the composite cement, the combined presence of pozzolan, slag, and Portland cement leads to the formation of a binder paste in the cement, which is called calcium silicate hydrate (CSH) gel.[4] Slag has a latent hydraulic property and can produce a CSH gel when an activating material such as quick lime, gypsum, cement, and alkaline metal sulfates is available. Cement is often used as a slag activator.[17] The reaction of Portlandite in Portland cement with pozzolan also produces a CSH gel.[15,19]

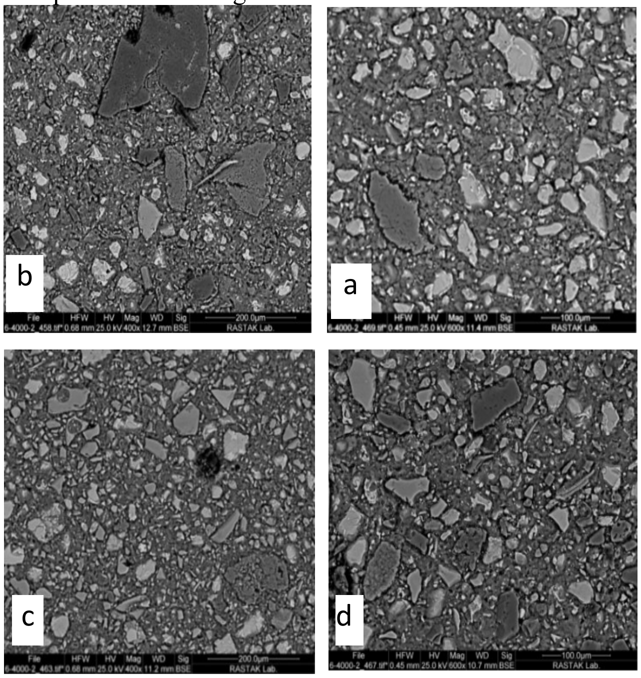

Fig. 7. SEM images of the microstructure of hardened paste, a) Portland cement with 600x magnification, b) slag cement (30% slag) with 400x magnification, c) pozzolan cement (30% pozzolan) 400x magnification, and d) composite cement (20% pozzolan and 10% slag) with 600x magnification - after 120 days of curing.

XRD study

Figure 8 shows the XRD pattern of hardened composite cement, slag powder, and pozzolan powder. In the composite cement, the source of Albite and quartz crystals is the pozzolan, while the source of Portlandite $Ca(OH)_2$ and calcium silicates (coarse crystals of Alite and Bilite that have no chance of reacting) is the Portland cement. The presence of $CaCO_3$ (calcite) in the composite cement is due to limestone or pozzolan. Gehlenite (related to slag) and Richterite phases (related to pozzolan) are not observed due to the dilution effect and reduction of their percentage in the composite cement. A broad XRD pattern is observed for the slag powder, which indicates its glass structure. Several relatively sharp peaks resulting from the crystalline phases in the pozzolan powder can also be seen. The broad peaks in the composite cement, especially at 2θ = 25-35 degrees, are attributed to the hydrated materials, which is mainly the CSH gel. The formation of CSH gel is the main contributor to strength development.

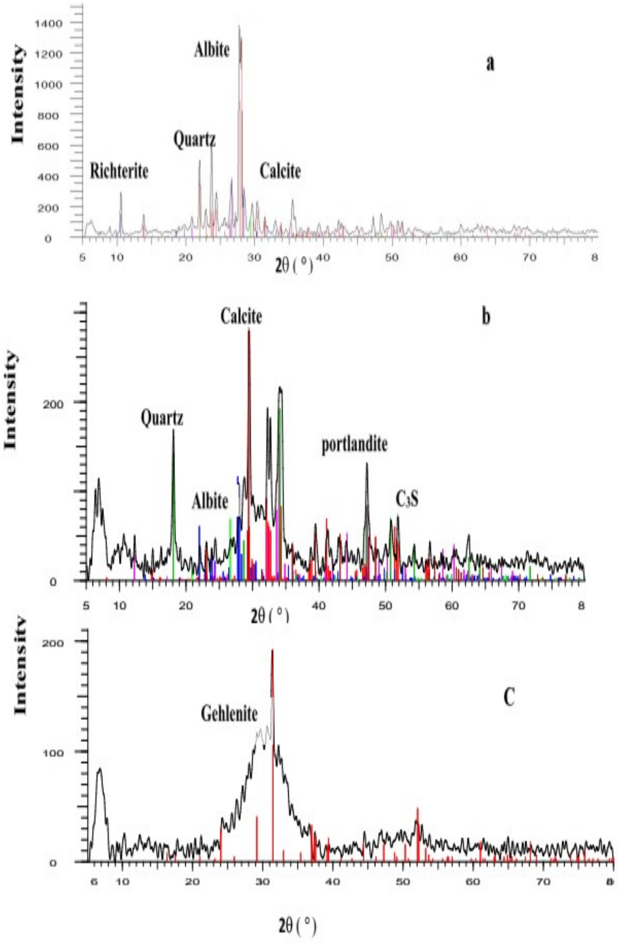

Fig. 8. XRD patterns of a) pozzolan powder, b) composite cement (10% pozzolan and 20% slag) after 120 days of curing, and c) slag powder.

TGA study

After 120 days of curing, the hardened cements were pulverized, and the weight loss of the samples was evaluated using TGA up to 1100 °C. The TGA and DTA diagrams are displayed in Fig. 9. Four major steps can be traced. The first step in the temperature range of 25 to 100 °C is due to the evaporation of physically adsorbed water. The second step in the range of 100 to 350 °C is due to the dehydration of the CSH gel, ettringite, and calcium aluminate-hydrate.[23] The dehydration process for the cement and the CSH gel is seen as a gentle slope and the temperature required for water loss depends on the CaO/SiO_2 ratio of the hydrated cement matrix. The third step in the range of 430 to 460 °C is characteristic of the Portlandite decomposition. Finally, in the fourth step, $CaCO_3$ decomposes at 790 °C.[21-22]

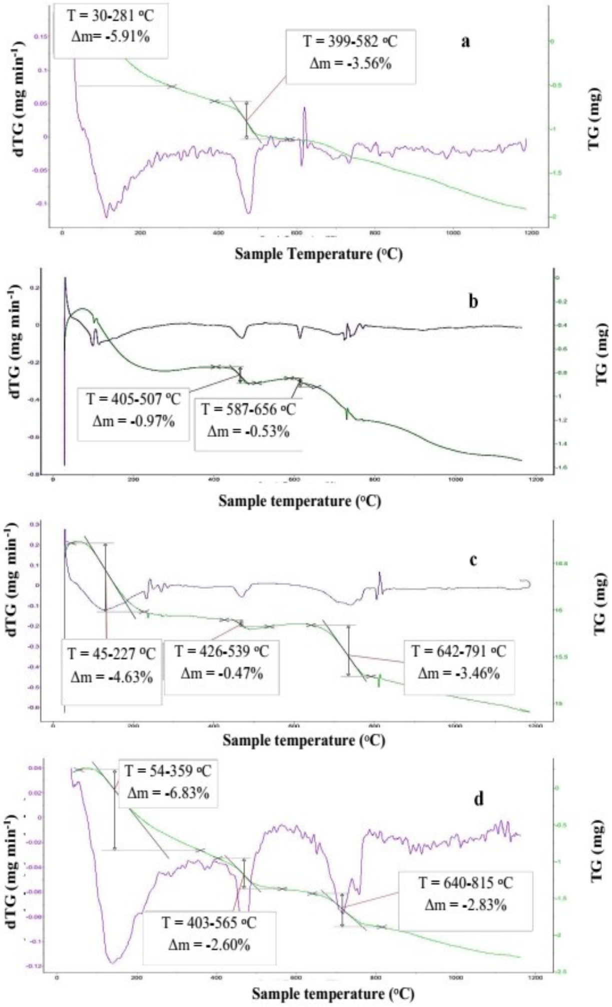

Fig. 9. TGA of the hardened pastes; a) Portland cement, b) slag cement (30% slags), c) pozzolan cement (30% pozzolan), and d) composite cement (10% pozzolan and 20% slag) after 120 days of curing.

In the temperature range of 400 to 500 °C, the stoichiometric ratio of calcium hydroxide to water in the decomposition reaction of $Ca(OH)_2$ is equal to 74:18, the amount of Portlandite in the Portland cement is estimated to be 14.6%, and the Portlandite content of pozzolan cement and composite cement is 2 and 10.6%, respectively. The Portlandite content of the composite cement was measured to be between those reported for pozzolanic cement and Portland cement. This result showed that the Portlandite due to the hydration of Portland cement and the small amounts of Portlandite as the result of the slag reaction are consumed by the pozzolan in the composite cement paste. Decreasing the Portlandite content in the cement paste is a positive point of adding pozzolan because calcium hydroxide is one of the potential factors that promotes carbonation and makes concrete susceptible to attack by other salts.[3,36] In the presence of the pozzolan, the Portlandite is used to produce a CSH gel. The composite cement containing 10% pozzolan benefits from this process.

4. CONCLUSIONS

This research used two materials to produce composite cements, including GGBFS from Isfahan Steel Company and a natural pozzolan from Rafsanjan Pozzolan Mine. Each of these materials exhibits different effects on the composite cement, which are briefly listed below.

1-Materials: The results of chemical and physical tests confirm the qualification of natural pozzolan and GGBFS for the production of composite cements. Portland cement type II was used as a component for the production of composite cement. A limestone filler was added to the composite cement in small quantities. The activity of the natural pozzolan from Rafsanjan Pozzolan Mine was determined to be moderate, while the slag from Isfahan Steel Co. showed a much better potential to achieve higher final compressive strengths.

2-Mechanical properties of the composite cements: The natural Pozzolan slightly improved the initial strength; however, after 7 days, the decrease in the strength for this composite cement compared to the Portland cement and slag cement was more pronounced. Therefore, in terms of compressive strength, the cement containing slag has a better potential.

3- Flowability of the composite cements: The results showed that the cement containing slag improves workability and performs better in the flow-table test of mortar, while the natural pozzolan needs a higher water content to achieve the same workability.

4-Setting time of the composite cements: The higher setting time was achieved by a cement containing higher slag content.

5-Density of the composite cements: According to the density test after four months of curing, the mortar containing a higher slag content had a better performance.

6-Environmental benefits of the composite cements: Slag as a by-product of the iron production is considered as an industrial waste, which its release into the environment causes negative environmental consequence. However, pozzolan is a natural material with a limited availability, and its extraction in the mining process can harm the environment. Therefore, in terms of environmental considerations, the use of slag is preferable in the formulation of composite cements.

7-Improving the durability of composite cements: The TGA results showed that the natural pozzolan reacts with $Ca(OH)_2$ (Portlandite originated from cement and slag), so combining pozzolan with slag can help with durability of concrete and mortar. Although no durability testing was conducted here for the composite cement against sulfate and chloride ions, several publications have reported improvement in the durability properties of concretes made with composite cements.

8-The XRD and SEM results: The structure of slag is generally glassy and has a few crystals. In contrast, the natural pozzolan had a large amounts of clay, quartz, and calcite crystals.

9- The microscopic study showed that some coarse grains (larger than 30 microns) in cement, slag, and pozzolan did not react even after 4 months.

According to the results above, we concluded that the proper formulation should contain more slag, *i.e.*, a composite cement containing 10% pozzolan and 20% slag. In the composite cement, a higher percentage of slag was chosen because, based on the microscopic study, slag has a more complete hydration reaction than the natural pozzolan. The XRD and TGA results revealed that the natural pozzolan reduces the Portlandite phase in cement. Thus, its presence in the composite cement is essential. The natural pozzolan helps to reduce the concrete efflorescence and increases the durability of concrete.

CONFLICTS OF INTEREST

The authors declare that they have no conflict of interest.

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