

Impacts of Thermal Heating Phosphogypsum on the Physiochemical Properties of Blast Furnace Slag Cement

Bassam Ahmed (✉ ch_bassam66@yahoo.com)

Al-Azhar University Faculty of Science <https://orcid.org/0000-0001-8801-1094>

Mahmoud Ahmed Taher

Al-Azhar University Faculty of Science

Adel Mohamed Amine

Housing and Building National Research Center

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Abstract

Within approximately 50 years, 1.0×10^8 tons of Phosphogypsum have already been produced and collected. Nearly 85 percent PG byproduct has been stored, only 15 percent has been reprocessed. Lowering disposal of waste materials offers both environmental and economic advantages. Physical and chemical characteristics of Blast furnace slag cement after partial and full replacing of raw gypsum with samples prepared from BFSC-PG at various temperatures (200–1000°C) are formed by blending different proportions with PG have been studied. The results validated the application of calcined Phosphogypsum at 800 and 1000 degrees Celsius rather than raw gypsum in cement manufacturing.

1. Introduction

Phosphogypsum (PG) is considering an acidic by-pass material from the fertilizer manufactory, annually huge amounts are manufactured in the world with calculated that by the year 2000 more than 280 million tones [1]. Although Phosphogypsum (PG) is a residue of the phosphoric acid manufacturing, for every ton of phosphoric acid synthesized, roughly four- six tons of PG are created, only 15 percent is reprocessed, and 85 percent of the global PG byproducts are stockpiled [2–3]. According to the standard PG management principles, recovery is the major method for reducing storage costs and minimizing the waste damaging public health and environmental ramifications [1]. One of the most effective experiments was conducted in Turkey [4], where PG was combined with natural Gypsum NG at varying percentages to produce extra gypsum. Based on the findings of the increasing percentage of PG in August 1995, the management of Barricksill Cement Plant decided to use PG as a replacement of raw gypsum in Ordinary Portland cement. But they found that Phosphogypsum has high moisture content and impurities, which interfere with the consistency of the cement [5]. The impurities in PG such as Phosphorus, Sulfate, Fluoride, and organic may all interact in unexpected ways leading to an increase in the setting time period of cement and a decrease in the mechanical strength development [6]. The utilization of PG instead of raw gypsum in cement industry has both economic and environmental benefits [7]. On the other hand, Blast furnace slag Cement (BFSC) is the most essential and valuable resource for the cement industry, which has been the subject of much research [8–10]. The purpose of this study is to investigate the physico-chemical properties of Blast Furnace Slag cement paste after partial substitution of gypsum with thermally treated Phosphogypsum at different temperatures (200, 400, 600, 800, and 1000 °C). The setting time, compressive strength, bulk density, and total porosity of wound healing after being maintained in water up to (3 days, 7 days, 28 days, and 90 days) examined. Combining water determination over a storage time of up to 90 days with free lime assessment after 90 days of curing was used to study hydration properties. The structure of various mortuaries was investigated using FTIR spectroscopic research. The microstructure of the hardened paste studied using a scanning electron microscope (SEM). It was observed that Phosphogypsum might well be utilized up to 5 percent as a portion in BFSC. This will decrease a significant source of pollution in the environment. Furthermore, it will lower the cost of cement manufacturing.

2. Materials And Examination Methods

2.1. Raw materials

Phosphogypsum (PG) utilized in the current examination purchased from a local supplier (Abyad Fertilizers Company's industrial plant (North of Amman, Jordan). Manaseer Cement Company provided Portland clinker cement (PCC), Raw Gypsum (RG) and Blast furnace slag (BFS) materials (Amman, Jordan). Raw material's chemistry and composition content examined using X-Ray Florescent in Table 1.

2.2. Preperation of BFSC- PG specimens

Phosphogypsum was calcined by burning it inside a Muffle, this was achieved by setting up the muffle at temperatures starting from 200 and up to 1000 °C with a heating rate 10 °C/min for 2 hours. Various BFSC-PG pastes were produced by heat treatment in varied proportions at different temperatures (200–1000°C) using Phosphogypsum to partially replace the raw gypsum in the weight-to-volume ratios. Samples were produced into a mold with a diameter of (1*1 inch). Usually kept in a humid room at 20 ± 1 °C for 24 hours to cure. Afterwards, it got de-molded and submerged in tap water until this was tested. Vicat equipment was used to measure the setting periods of cement mixed in various quantities according to EN-196-3 [11]. After the hydration was completed, the combined water content was determined by incinerating samples around 1000°C for 1 hour in a Muffle furnace [12, 13]. Blast furnace slag cement was made from 60 % Portland cement clinker (PCC), 35 % blast furnace slag (BFS), and 5 % raw gypsum (blank sample). Also, samples were made by mixing PCC, blast furnace slag (BFS) and raw gypsum (RG) with thermally treated PG at temperatures (200- 1000°C) in the weight ratios of 60:35:4:1 (SI), 60:35:3:2 (SII), 60:35:2:3 (SIII), 60:35:1:4 (SIV), 60:35:0:5 (SV) and blank sample 60:35:5:0 (S), respectively.

3. Results And Discussion

3.1. Setting time and Water Consistency (W/C)

It is well known that these combinations were hand-molded into cylinders and subsequently cured for 3,7,28, and 90 days under tap water shown in Fig. (1). The impact of partial substitution of RG with calcined PG on water consistency W/C at various temperatures decreased by increasing PG dosage. Additionally, the calcined temperature of PG raised, and the W/C value increase. According to the results that have been obtained, found SV mix possesses the lowest W/C values at all firing temperatures of PG. Furthermore, Mixes made from BFSC- PG at firing temperature 1000 °C achieved the lowest value of W/C [14].

The effect of partial and complete substitution of raw gypsum by treated PG at different temperatures on the sitting time of mixes BFSC- PG have been studied. Assessing the final and initial setting time of mixes (SI-SV) with the blank sample (S) at various temperatures revealed that the mixes had greater values than the blank sample at low PG thermal treatment temperatures. While increasing the thermal treatment of PG causing decreasing values setting time.

All these findings were due to attributed and present fluoride, phosphorus, sulfate, and organic pollutants in PG interact in an unanticipated way to prolong the setting time [15].

Table (1). Chemical examination of the raw components (percentage).

Contents	PCC	PG	PG at 1000 °C	RG	BFS
CaO %	65.00	35	40.50	39.80	46.7
SiO ₂ %	20.84	7.32	11.50	7.80	33.05
AL ₂ O ₃ %	5.42	0.15	0.25	1.1	8.3
Fe ₂ O ₃ %	2.78	0.17	0.15	0.56	0.34
MgO %	2.08	0.42	0.2	1.59	4.78
K ₂ O %	0.58	0.04	0.003	0.35	0.43
Na ₂ O %	0.58	0.74	0.01	0.23
SO ₃ %	0.89	41.06	32.1	22.18	0.27
P ₂ O ₅ %	0.33	6.79	2.35	0.39	0.04
CL %	0.002	0.43	0.02	0.013
LOI %	0.00	10.1	0.82	30.04	0.96

3.2. Compressive strength (Kg/cm²)

Figures (2, 3) show the compressive strength results produced for several blast furnace slag cement pastes, following partial replacement of RG with fired PG at varied temperatures (200–1000 °C) as a function of curing time. It was evident that the comparison of strength values of mixes (SI- SV) with blank S indicated that all mixes possessed higher values than blank S after 90 days curing at all firing temperatures of PG. Generally, the compressive strength values were measured after 90 days curing for all mixes had highest values as per hydration process. Also, SV mix owned higher values at firing temperatures 800, 1000 °C but the highest value was measured at 1000 °C. By increasing firing temperature of PG was attributed to the decreasing in P₂O₅, K₂O, SO₃ contents in PG and increasing of CaO and SiO₂ contents which leads to increasing in compressive strength [16]. Finally, complete substitution of RG with 5 percent thermally treated PG at 1000°C leading to improve the compressive strength of BFSC.

3.3. Bulk density and total porosity

Generally, density is an essential component in evaluated porosity. Based on the hydration process, hydration products fill part of the holes which leads to reduce in the total porosity and increase in bulk density at all mixes formed from BFSC- PG. Therefore, the bulk density and total porosity results got by the replacement of RG with burned PG at varied temperatures 200, 400, 600, 800, and 1000°C via curing time were graphically represented in Figs. (4, 5). The values for all mixtures (SI-SV and blank S) increasing with increasing curing time. Put differently, the porosity reduced as the age of curing rose as a part of the hydration processes. This was attributed to the hydration of part of the accessible pore volume. In pastes and mixtures BFSC-PG, the porosity decreased when the temperature of PG increased. Bulk density values of cement pastes of SV mixes gave the high values at all curing age and firing temperatures of PG. The highest value was obtained at 1000°C firing temperature of PG, which is in a good agreement with the compressive strength values. On the Other side, the porosity decreased with increasing firing temperature of PG in the all pastes. Finally, found SIV &SV mixes possessed lower values of porosity at all firing temperatures of RG and mix SV possessed the lowest value after 90 days of curing time which is in harmony with compressive strength and bulk density results [17].

3.4. Hydration Kinetics

Kinetics of hydration for the various blast furnace cement pastes (SI-SV) after partial and complete substitution of RG with calcined PG at different temperatures (200–1000 °C) were studied by the determination of chemically-combined (non-evaporable) water contents Wn% show in Fig. (6) and Free lime content show in Fig. (7). Generally, increases the combined water content by curing time due to the progress and formation of hydration products which have high combined water content such as CSH, C₄AH₁₃ and C₂ASH₈. Additionally, the change of hydration of mixes BFSC- PG by curing time causing decrease in combined water content. Chemically combined water contents Wn revealed that mixes SI (contain PG treated at 200 °C) had the lowest combined water contents in BFSC-PG mixes while mix SV (contain PG treated at 1000 °C) have the highest combined water contents and highest effective hydrated phases. These results totally agreement with the compressive strength, bulk density and total porosity results. Free lime contents result of blank sample (S) which contains 5 % raw gypsum decreased with curing time due to attributed the accumulation of Ca(OH)₂ with silica and alumina phases. On the other contract, samples contain calcined PG (SI-SV) exhibited an increase in free lime content at all temperatures by curing times. This was attributed to the presence of P₂O₅ in fired PG which affected on the formation of tricalcium silicate. Higher amounts of P₂O₅ inhibit C₃S crystallization and lead to the preferential formation of

phosphate solid solution of dicalcium silicate. Finally, increasing rates of P_2O_5 decompose C_3S crystallization and favor the development of phosphate solid to dicalcium silicate solutions and free lime which lead to an increase in free lime content at all mixes [18].

3.6. FTIR Analysis

As seen in Fig. (8) explains FTIR spectroscopic of blank sample S (contains 5 % RG). At intensities of 3643, 3445, 1650, 1460, 967, 875, and 458 cm^{-1} , distinct bands appeared. The production of C_2S was accompanied by the release of lime, resulting in absorption bands at 3643 cm^{-1} and 1460 cm^{-1} for OH group vibrations and Ca-O stretching vibrations. Due to the vibration of HOH groups and mixed water a sharp band appears at 3445 cm^{-1} . Calcite formed when unreacted lime is partially carbonated has absorption bands at 875 cm^{-1} . Si-O bending belongs to the weak intensity band at 458 cm^{-1} [19]. Some bands exist at 3646, 3417, 1656, 1473, 977, 870, and 477 cm^{-1} in Fig. (9), which were previously described from pastes SV which contain PG treated at 1000°C. Bands at 3646 cm^{-1} and 1473 cm^{-1} are due to the C_2S phase accompanied. Lime liberation at 977 cm^{-1} is due to the formation of intermediate calcium silicate hydrate, and the intensity of the 3646 cm^{-1} band decreased as curing time increased due to more lime consumption and high formation of hydrated compound CSH, resulting in high compressive strength, while the 1656 and 870 cm^{-1} intensity decreased as curing time increased due to more lime consumption and high formation of hydrated compound [20].

3.7. Morphology and microstructure

Figure (10) show the effects of calcined Phosphogypsum at various temperatures on the microstructure of BFSC-PG pastes which are clearly visible in SEM. The porosity of the pastes reduces after 28 days of hydration causing amorphous and ill-crystalline phases to arise. After 90 days, the amorphous and ill-crystalline phases increase with the creation of a closed texture phase, indicating a significant increase in the pastes' hydraulic capabilities. It was also clear, by increasing the firing temperature of PG has a significant impact on the morphology and crystalline structure of pastes made from mix SV by forming ill-crystallized and well-crystallized hydration products, which include massive calcium silicate hydrates and significant amounts of calcium aluminate hydrates (C_3AH_13). An acquired physico-chemical measurement of BFSC-PG pastes containing 5% PG thermally treated at 1000°C after 90 days curing is in good accord with these findings [21].

4. Conclusion

The main conclusions derived from this study will be summarized as follows: The addition of treated PG to Blast Furnace Slag Cement at various temperatures improved the physio-chemical characteristics of the cement according to results from current research, while found by increasing thermal treatment of PG the W/C decreased at all temperatures. The bulk density values increased, and porosity values decreased for all types of BFSC-PG mixes at all curing time. Compressive strength values increased continuously with the increasing age of hydration due to the increasing in the amounts of hydration products. The SEM micrographs obtained for all specimens of mix SV after different firing temperatures of PG and after different curing times were studied. These results are in good agreement with the obtained physico-chemical measurements of BFSC-PG pastes containing 5% PG thermally treated at 1000°C after 90 days curing. Finally, it is recommended to use purified Phosphogypsum obtained by thermal processing at 800 and 1000 degrees Celsius which can be used instead of raw gypsum in cement manufacturing, avoiding severe negative environmental consequences caused by the dispersion of hazardous compounds present in PG into the environment and reducing cement manufacturing costs.

5. Declarations

1. **Availability of data and materials**, all data of manuscript are available and saved for any required from all the reviewers.
2. **Competing interests**, the authors declare that they have no identified competing financial interested or personal dealings that could have appeared to affect the work reported in this manuscript.
3. **Funding**, this manuscript is personally funding. (not applicable)
4. **Authors' contributions, Bassam following and** Conception or design of the work, data collection, Dr. Mahmoud Taher did Data analysis and interpretation, Drafting the article, and Dr. Adel did Critical revision of the article and Final approval of the version to be published.
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6. **Authors' information (optional)**, all available in manuscript.

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Figures

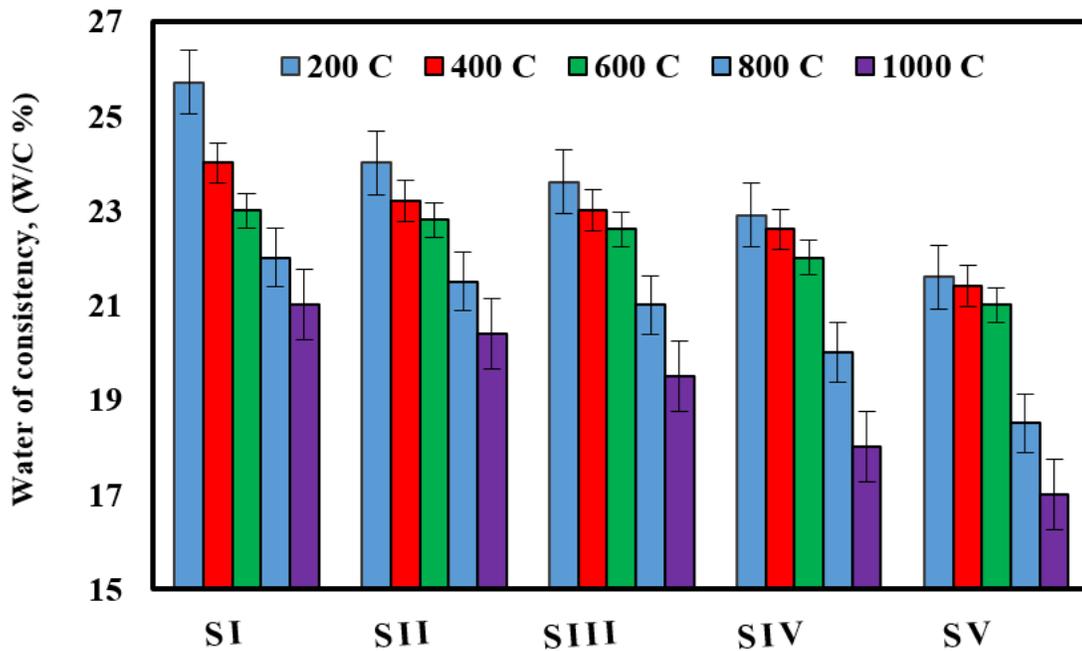


Figure 1

W/C of mixes (SI-SV) containing calcined PG at different temperatures (200 – 1000 °C)

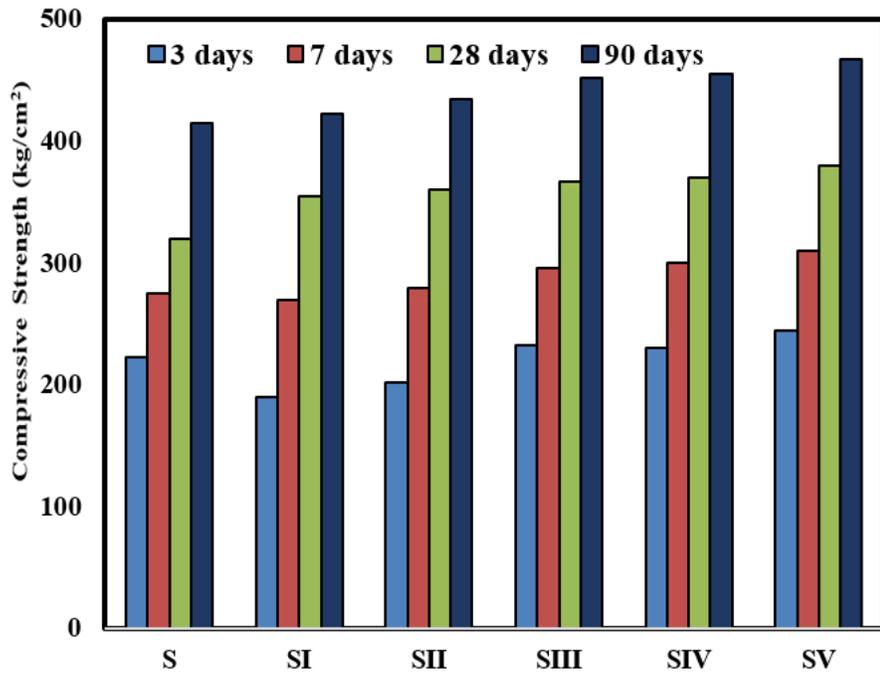


Figure 2

Strength of (SI-SV) mixes with calcined Phosphogypsum under 800 °C after 3,7,28, and 90 days of curing.

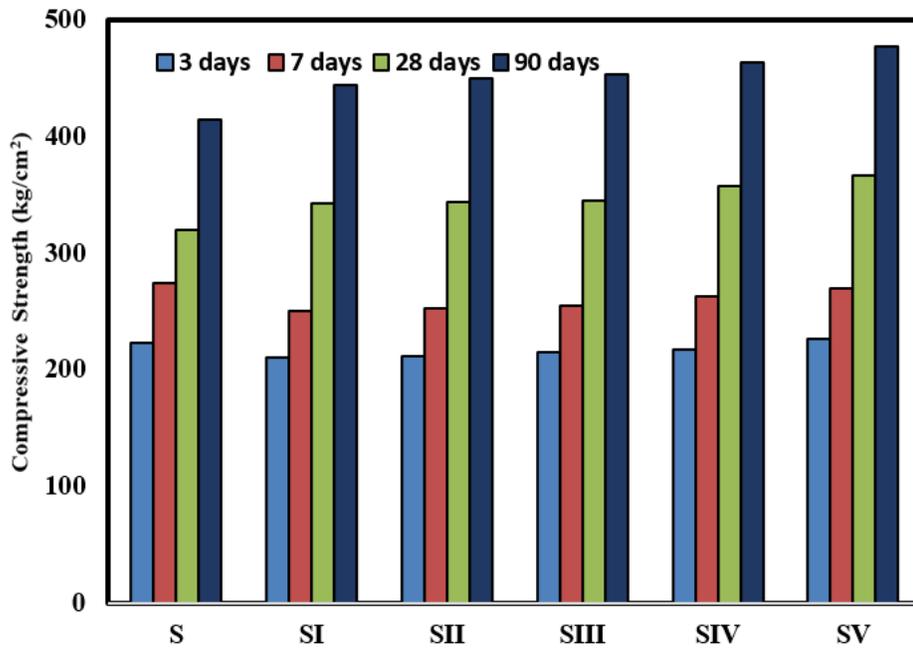


Figure 3

Strength of (SI-SV) mixes with calcined Phosphogypsum under 1000 °C after 3,7,28, and 90 days of curing.

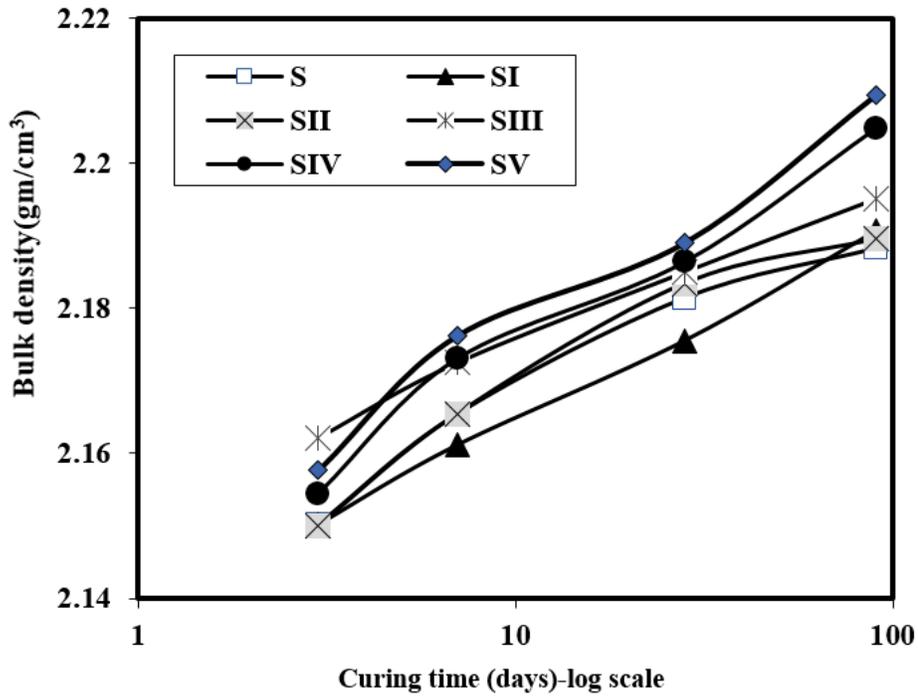


Figure 4

Bulk density (g/cm³) of mixes (SI-SV) with calcined PG under 1000 °C after 3,7,28 and 90 days curing.

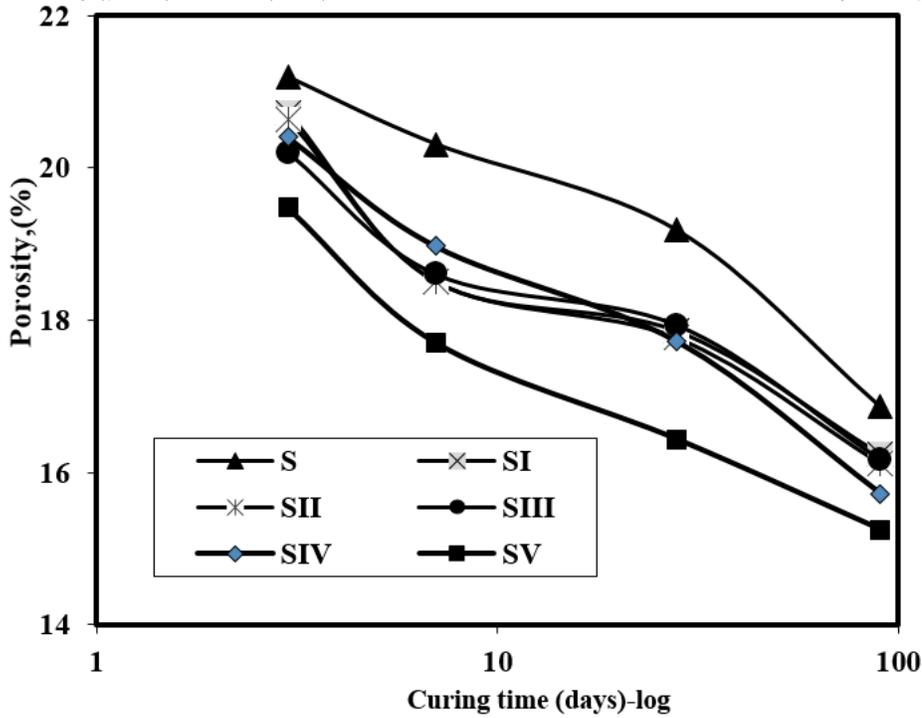


Figure 5

Porosity (%) of mixes (SI-SV) with calcined PG under 1000 °C after 3,7,28 and 90 days curing.

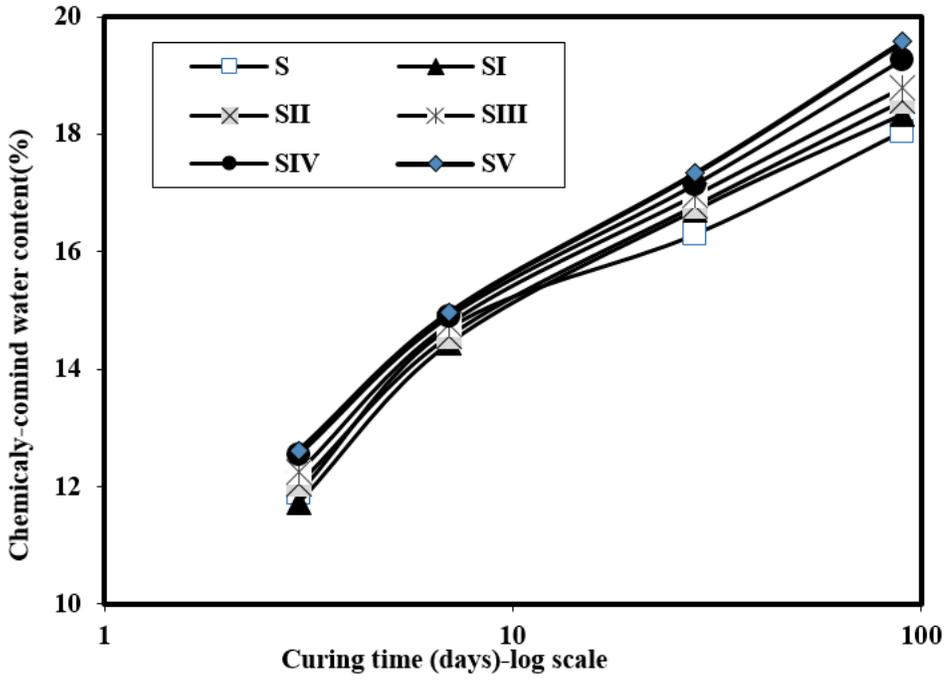


Figure 6

Combined water contents (Wn %) of mixes (SI-SV) with calcined PG under 1000 °C after 3,7,28 and 90 days curing.

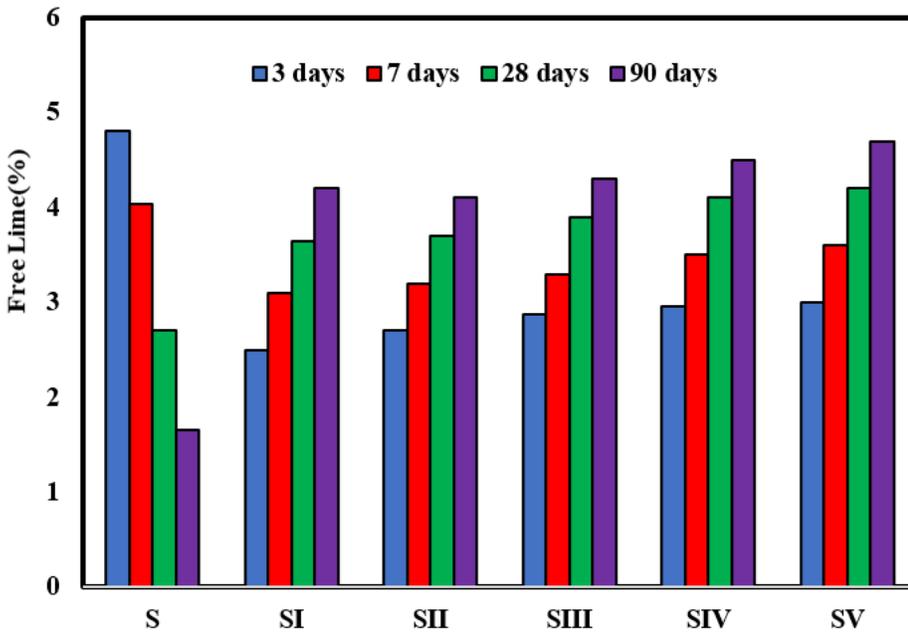


Figure 7

(CaO %) of mixes (SI-SV) with calcined PG under 800 °C after 3,7,28 and 90 days curing

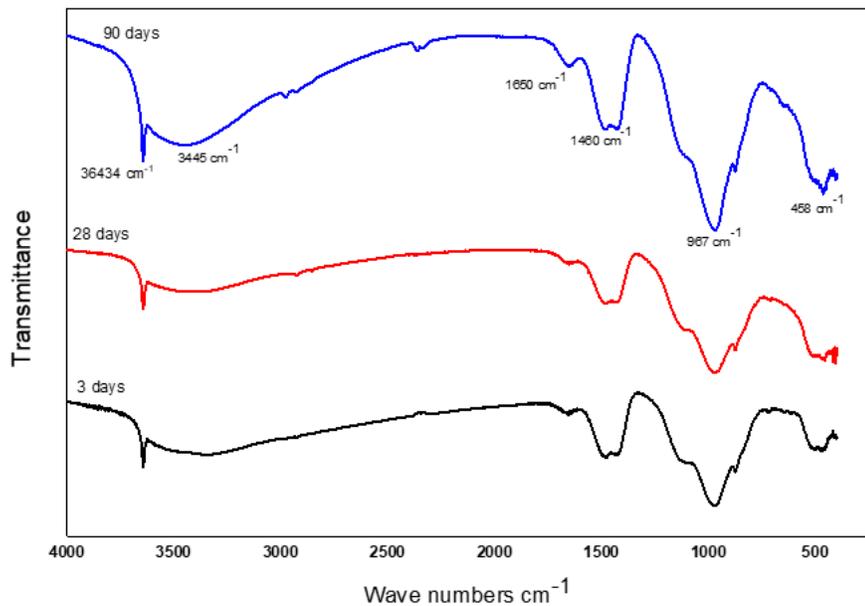


Figure 8

IR spectra of pastes made from blank sample (S) after 3, 28- and 90-days curing.

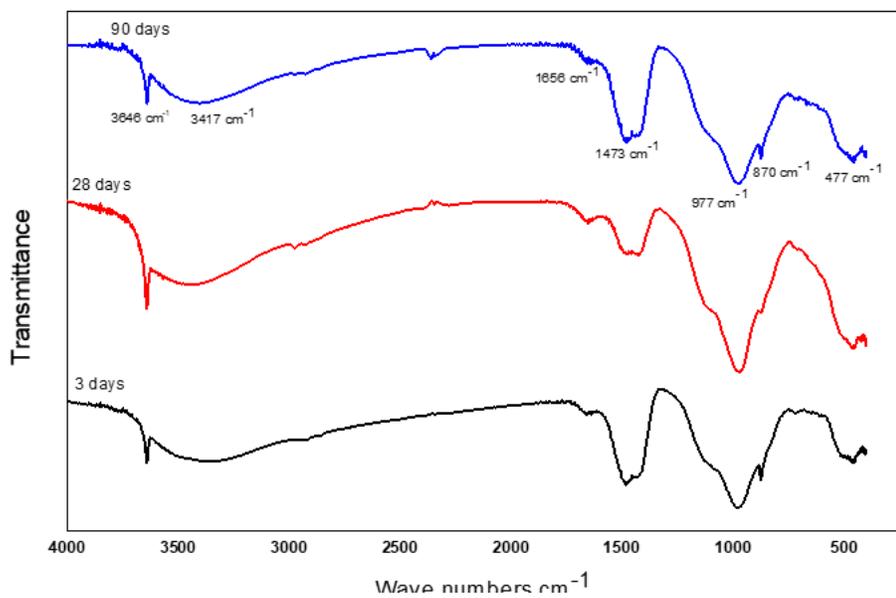


Figure 9

IR spectra of pastes made from mix SV with calcined PG under 1000 °C after 3, 28- and 90-days curing.

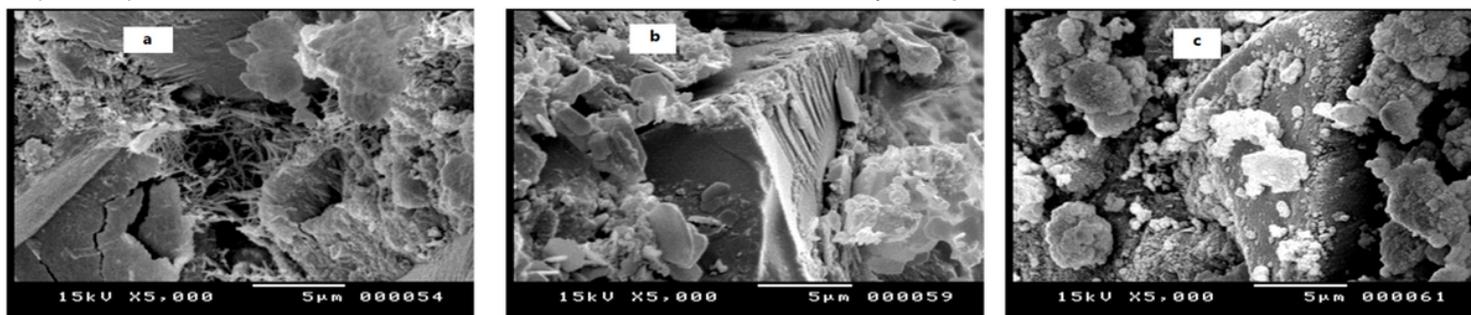


Figure 10

SEM of mixes (SV) with calcined PG under 1000 °C after 3(a), 28 (b) and 90 (c) days of curing.

Supplementary Files

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