

INFLUENCE OF CURING CONDITIONS ON THE EARLY STRENGTH OF LOW TEMPERATURE BELITE CEMENTS

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ABSTRACT: Low temperature belite cements are produced using techniques that stabilize the more reactive high temperature polymorphs of dicalcium silicate (C_2S) to improve early strength, but the effect of curing conditions on them is not well studied. The focus of this work is to assess the improvement of their early strength in different curing conditions. During the synthesis of the cements at 1000 °C, the more reactive polymorphs, α' - C_2S and β - C_2S , were stabilized using gypsum and hydrothermal treatment with potassium hydroxide. The phase composition of the synthesized cements was analysed using X-ray powder diffraction. The morphology and elemental composition of the C_2S crystals and hydrated pastes were determined using a scanning electron microscope equipped with an energy-dispersive X-ray system. Mortar samples were cured in different conditions that include hot air and hot water curing at 60 °C and 90 °C. The 28-day strength development, capillary water porosity, water absorption, and ultrasonic pulse velocity were tested. The formation of hydration products and strength was dependent on the type of C_2S polymorph. Curing at elevated temperatures improved the transport properties of mortars. Samples cured at 90 °C in hot air obtained the highest early strength. The presence of α' - C_2S and elevated curing temperatures significantly improve the early strength of the mortar samples.

ABSTRAK: Simen belite suhu rendah dihasilkan melalui teknik menstabilkan reaktif polimorf dikalsium silikat (C_2S) bersuhu tinggi bagi meningkatkan kekuatan awal, tetapi kesan keadaan pengawetan ke atasnya tidak dikaji dengan baik. Fokus kerja ini adalah bagi menilai peningkatan kekuatan awal pada keadaan pengawetan berbeza. Sintesis simen pada suhu 1000 °C, iaitu pada polimorf lebih reaktif, α' - C_2S dan β - C_2S , telah distabilkan menggunakan rawatan gipsum dan hidroterma dengan kalium hidroksida. Fasa komposisi simen tersintesis dianalisa menggunakan pembelauan serbuk sinar-X. Komposisi morfologi dan unsur kristal C_2S dan pes terhidrat ditentukan menggunakan pengimbas mikroskop elektron yang dilengkapi sistem sinar-X penyebar tenaga. Sampel mortar telah diawetkan dalam keadaan berbeza termasuk pengawetan udara panas dan air panas pada suhu 60 °C dan 90 °C. Perkembangan kekuatan keliangan kapilari air, penyerapan air, dan halaju nadi ultrasonik telah diuji pada hari ke 28. Pembentukan produk penghidratan dan kekuatan adalah bergantung kepada jenis polimorf C_2S . Pengawetan pada suhu tinggi meningkatkan sifat pengangkutan mortar. Sampel yang diawet pada 90 °C dalam udara panas memperoleh kekuatan awal tertinggi. Kehadiran α' - C_2S dan suhu pengawetan tinggi dengan ketara meningkatkan kekuatan awal sampel mortar.

KEYWORDS: *belite cements; dicalcium silicate; thermal curing; transport properties; porosity*

1. INTRODUCTION

Portland cement is the predominantly used binder in construction with an annual production of 4.1 gigatons in 2020, partly due to its utilization in concrete, the most used synthetic material in the world [1]. It is estimated that the global concrete stock is around 315 gigatons [2]. The vast usage of cement can be attributed to its high early strength and the availability of its primary raw materials (limestone and clay) in economic quantities across the continents. However, despite efforts to improve sustainability, the production of cement accounts for about 12 percent of total industrial energy consumption. It also consumes about 1.7 tonnes of raw materials and emits around 0.84 tonnes of carbon dioxide for every tonne of cement produced [3-5]. These are among the factors necessitating research in alternative binders such as belite cements.

Belite cements have similar raw materials and production processes as Portland cements but with lower environmental consequences. They both have similar compounds with the major difference being the percentage of the calcium silicates i.e., tricalcium silicate (C_3S) and dicalcium silicate (C_2S). Clinkers of Portland cements contain an average of 60 percent C_3S and 10 percent C_2S while those of belite cements are composed of 40–60 percent C_2S and 10–30 percent C_3S [2]. Producing belite clinkers emits 10–12 percent less carbon dioxide due to lesser demand for limestone. The average burning temperature of 1300 °C is also used which is almost 200 °C less than the clinkering temperature for Portland cements [6]. Furthermore, belite cements can be produced in a Portland cement factory, thus, making their large-scale production feasible [7]. They can also be covered by existing codes of practice such as EN 197-1 if they satisfy the requirements that include $C_3S + C_2S > 66$ percent of total clinker [2].

Recent studies assessed the characteristics of C_3S -free belite cements that are produced at temperatures between 800 and 1200 °C [8-13]. Burning at low temperatures produces C_2S with smaller grain size that can accelerate hydration, and a porous crystalline structure capable of incorporating by-products [14]. The by-products are introduced by adding compounds such as gypsum and sodium fluoride as mineralizers or through hydrothermal treatment with alkaline solutions e.g., sodium hydroxide and potassium hydroxide [9,12,15]. The introduction of ions into the crystalline structure of the C_2S from these by-products and the fast cooling assists in stabilizing the more reactive high-temperature polymorphs of C_2S . There are five (α , α'_h , α'_l , β , γ) polymorphs of C_2S that are stable at different temperatures with distinct crystalline structures [16]. These polymorphs have different reactivity, with the α' - C_2S being the most hydraulic. The α - C_2S and β - C_2S have moderate reactivity while the γ - C_2S is the least reactive and the most stable polymorph [10].

During hydration, both C_3S and C_2S produce the same compounds, calcium silicate hydrates (C–S–H) gel and calcium hydroxide. However, C_3S hydrates faster than C_2S , and it is responsible for the high early strength and heat of hydration in Portland cements. Furthermore, C_3S can achieve about 78–80 percent hydration at 28 days under normal curing conditions while C_2S can achieve only 20–50 percent. But at full hydration C_3S produces 72.5 and 27.5 percent volume of C–S–H gel and calcium hydroxide respectively while C_2S produces 94.4 percent C–S–H gel and only 5.6 percent calcium hydroxide. This makes C_2S more durable and resistant to chemical attacks by producing more C–S–H gel that contributes to the strength and less calcium hydroxide which is the more reactive hydration product [2]. The low hydration heat in belite cements limits the possibilities of thermal cracking and increases their suitability in mass concrete production.

The major drawback in the wide acceptance of belite cements is their low early strength due to their slowed hydration. Accelerating the hydration process and hardening is necessary

for obtaining cement composites with sufficient early strength. The techniques used include elevating the hydration temperature in different curing processes such as steam curing, hot water curing, hot air curing, and autoclaving [17]. There is no agreement regarding which method is the most effective and the optimum curing temperature. Similarly, opinions on the delay time (the period between casting and application of heat) differ among the researchers. From the reviewed literature, three delay times were suggested: immediately after casting; after the initial set; and after a 24-hour delay [18].

Thermal acceleration of hydration in Portland cements is extensively studied but there is not enough data on belite cements. It was discovered that elevated curing temperatures can have a detrimental effect on Portland cements hydration that can cause a loss of long-term strength, a phenomenon called the crossover effect [19]. This is widely agreed to be caused by the non-uniform distribution of hydration products that creates cracks and a porous microstructure. This might vary between belite cements and Portland cements, as a study by Thomas et al. [20] reported a 25 percent increase and 21 percent decrease in compressive strength for belite cements and Portland cements, respectively, at 28 days when curing temperature was raised from 20 °C to 70 °C.

Although there are recent studies that were conducted to assess the effects of curing conditions on belite cements, they were conducted on cements produced at high temperatures that contained C₃S [17,19]. The objective of this study is to produce C₃S-free belite cements at low temperatures using waste materials and assess the effect of six curing conditions on early strength development. They include curing in saturated lime water, curing at room temperature (RT) and 85±5 percent relative humidity (RH), and hot water and hot air curing at 60 °C and 90 °C.

2. MATERIALS AND METHODS

2.1 Materials

The raw materials used in this study were cockle shells, rice husk ash (RHA), and calcined clay. They were all oven dried at 105 °C to constant mass, ground in a ball mill grinder, and screened through a sieve with 150 µm aperture [9,10,12,13,21]. The calcined clay was obtained by burning at 800 °C in an electric furnace with a 5 °C heating rate for 120 minutes. Figures 1 and 2 show the raw materials used in the study and particle size distribution of the fine aggregate used in mortar preparation, respectively. The chemical composition of the materials that was determined using X-ray fluorescence (XRF) is shown in Table 1.

2.2 Synthesis of Belite Cement

The raw meals were prepared to have a lime/silica (CaO/ SiO₂) ratio of 2. The first group contained 55.18 percent cockle shell and 44.82 percent calcined clay with a lime saturation factor (LSF) of 0.61 while the second group was composed of 65.20 percent cockle shell and 34.80 percent RHA, and an LSF of 0.71. The chemical composition of the raw meals is shown in Table 2. Three batches of raw meals were prepared from each group with different treatment methods as shown in Table 3. A total of six batches of binary raw meals were prepared for this study.

The CH and RH precursors from the hydrothermal treatment were prepared from a dry mix of raw meals. The ground raw materials were added to a 0.6 M potassium hydroxide (KOH) solution at a liquid-to-solid ratio of 5:1. The mixture was stirred and heated at 100 °C using a magnetic stirrer at atmospheric pressure for four hours. The solution was then filtered through a Whatman filter paper to remove excess liquid before drying in an oven at 105 ± 5 °C

for three hours. The precursors were crushed and sieved to a particle size of less than 150 μm before burning.

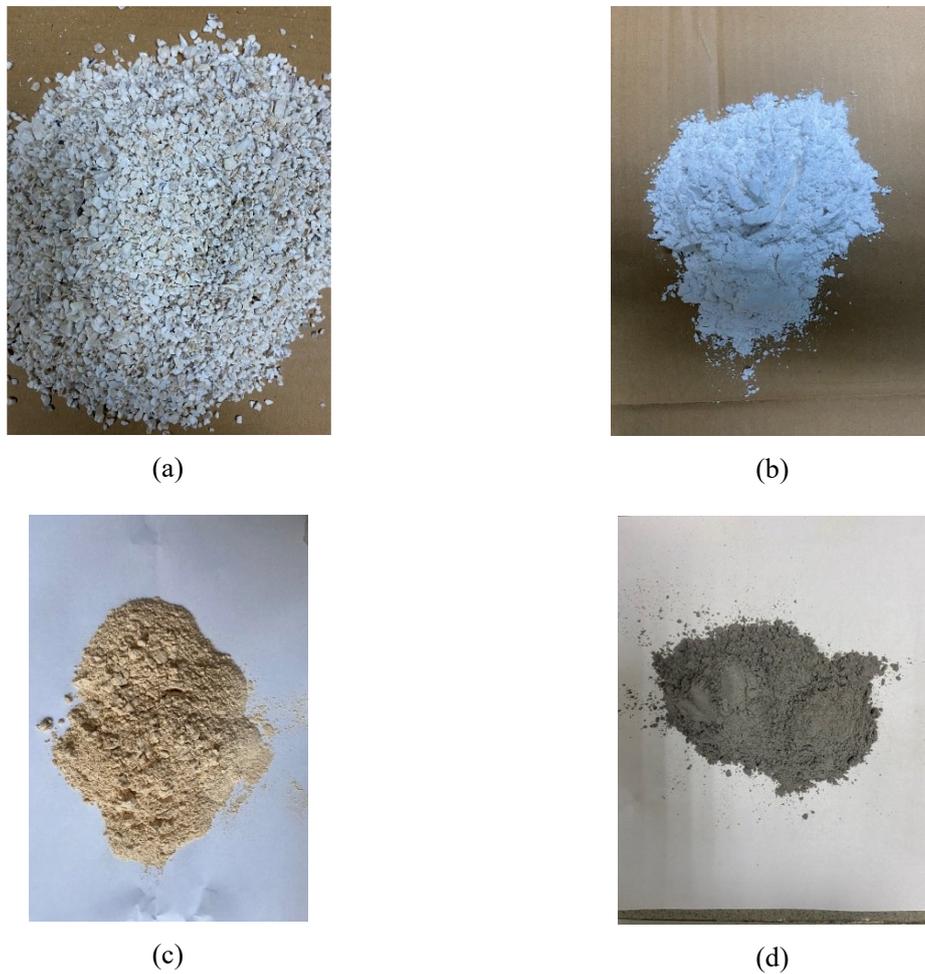


Fig. 1: Samples of (a) crushed cockle shell (b) ground cockle shell (c) calcined clay (d) rice husk ash.

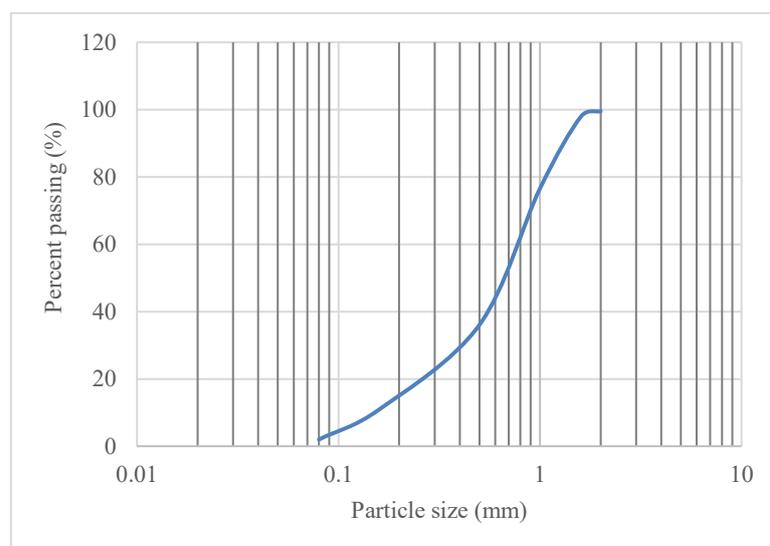


Fig. 2: Particle size distribution of fine aggregate.

Table 1: Chemical composition of raw materials (percent)

Oxide	Cockle shell	Clay	RHA
SiO ₂	-	60.07	92.40
Fe ₂ O ₃	0.34	8.68	0.44
Al ₂ O ₃	-	20.69	-
CaO	97.59	0.21	1.11
K ₂ O	0.26	3.34	5.12
MnO	-	-	0.12
TiO ₂	-	1.02	-
BaO	-	4.35	-
ZnO	-	-	0.08
SrO	0.54	-	-
LOI	1.27	1.64	0.73

The raw meals were calcined in alumina-based crucibles in a laboratory furnace at 1000 °C for three hours followed by rapid air cooling down to room temperature. The products were ground in a ball mill grinder and sieved to a particle size of less than 53 µm.

Table 2: Chemical composition of raw meals (percent)

Oxide	Clay based	RHA based
SiO ₂	26.92	32.16
Fe ₂ O ₃	4.08	0.37
Al ₂ O ₃	9.27	-
CaO	53.94	64.02
K ₂ O	1.64	1.95
MnO	-	0.04
TiO ₂	0.46	-
BaO	1.95	-
ZnO	-	0.03
SrO	0.30	0.35
LOI	1.44	1.10

Table 3: Binders and their treatment method

Binder ID	Raw materials	Treatment
CN	Cockle shells and clay	No treatment (control)
CG	Cockle shells and clay	2% dihydrate gypsum (CaSO ₄ ·2H ₂ O)
CH	Cockle shells and clay	Hydrothermal treatment in 0.6 M KOH
RN	Cockle shells and RHA	No treatment (control)
RG	Cockle shells and RHA	2% dihydrate gypsum (CaSO ₄ ·2H ₂ O)
RH	Cockle shells and RHA	Hydrothermal treatment in 0.6 M KOH

2.3 Testing Methods

The X-ray diffraction (XRD) patterns of all the synthesized products were obtained using an Empyrean PANalytical XRD diffractometer (Cu K α 1.54060Å, 5–70° 2 θ range, 0.0260

step). The microstructure, morphology, and elemental analysis of the belite crystals and pastes were examined using a Quanta FEG 650 scanning electron microscope (SEM) equipped with an energy-dispersive X-ray (EDX) system. The pastes were prepared with water-to-binder ratio of 0.4, and the samples were coated using gold sputtering. The mortars were prepared according to EN 196-1 [22] using sand-to-cement ratio of 3 and water-binder ratios ranging from 0.66–0.75. They were cast in 50 mm cube moulds and demoulded after 24 hours. The mortars were cured in water and tested at 28 days for compressive strength. Compressive strength test was conducted using a 2000 kN compression machine and a loading rate of 0.4 kN/s. The pulse velocities through the mortars were measured using a direct transmission method between the transmitter and receiver transducers. The test was conducted on the samples before the compressive strength test.

The samples containing calcined clay (CN, CG, and CH) were selected and further tested for their early compressive strength and transport properties in different curing conditions, including elevated temperature curing. The elevated temperature curing was for a period of 48 hours after demoulding and then subsequent curing in saturated conditions until testing. The curing ID was assigned based on the name of the binder used, curing temperature, and curing method. The curing age was added as a suffix, with 7 and 28 signifying testing at 7 and 28 days, respectively. Table 4 shows the curing IDs and conditions for the samples prepared using the CN binder, and Fig. 3 shows the curing conditions of the samples.

Table 4: Curing conditions of samples

Curing ID	Curing condition
CNRLW7, CNRLW28	Saturated lime water at RT
CNRST7, CNRST28	RT and 85±5% RH
CN60W7, CN60W28	60 °C in hot water
CN60A7, CN60A28	60 °C in hot air
CN90W7, CN90W28	90 °C in hot water
CN90A7, CN90A28	90 °C in hot air

The capillary water porosity was determined using the water displacement method. After curing, the mass of the saturated samples was measured both in air (m_{sat}) and while fully submerged in water (m_{sub}). The samples were oven dried at 105 °C until constant mass and the mass was measured again (m_{dry}). The capillary water porosity was measured using Eq. (1). The water absorption coefficient was obtained using the partial immersion method in accordance with EN ISO 15148 [23]. The three sides of the dried samples from the capillary water porosity were coated with hot paraffin wax and placed on supports with the water at a 5mm level from the bottom of the sample. The increase in mass of the samples was measured at different time intervals until the difference between two successive measurements was less than 1%. The increase in mass per face area (in kg/m²) was plotted against the square root of the weighing time (in seconds), and the water absorption coefficient was calculated from the graph.

$$\text{capillary water porosity (\%)} = \frac{m_{sat} - m_{dry}}{m_{sat} - m_{sub}} \times 100 \quad (1)$$



Fig. 3: Curing conditions in (a) saturated conditions, (b) saturated lime water, (c) hot air and (d) hot water.

3. RESULTS AND DISCUSSION

3.1 XRD Analysis

All the obtained belite cements contained different polymorphs of C_2S due to the influence of their treatment method. The XRD patterns of the belite cements in Fig. 4 and Fig. 5 show that β - C_2S is the most common hydraulic phase in all the binders as was obtained by several previous researchers [8-12]. The diffractograms show that α' - C_2S peaks are only present in the belite cements that were produced from the precursors that were hydrothermally treated with KOH i.e., in Fig. 4c and Fig. 5c for CH and RH, respectively. The stabilization of α' - C_2S can be attributed to the presence of potassium ions introduced from the treatment.

Belite cements from raw meals without any treatment and those containing 2 percent gypsum only had peaks of β - C_2S . The XRD patterns of CG and RG, in Fig. 4b and Fig. 5b, respectively, indicate that they contain more peaks of β - C_2S than CN and RN (Fig. 4a and Fig. 5a respectively). This is due to the presence of SO_3 ions from the addition of gypsum that favours the stabilization of β - C_2S . It was reported by Zhao et al. [15] that at a high percentage of gypsum (up to 4 percent), the SO_3 ions stabilize the higher temperature polymorphs such as

α' -C₂S. However, Mazouzi et al. [9] suggest the presence of SO₃ ions in high quantities favours only β -C₂S and can sometimes prevent the stabilization of other polymorphs.

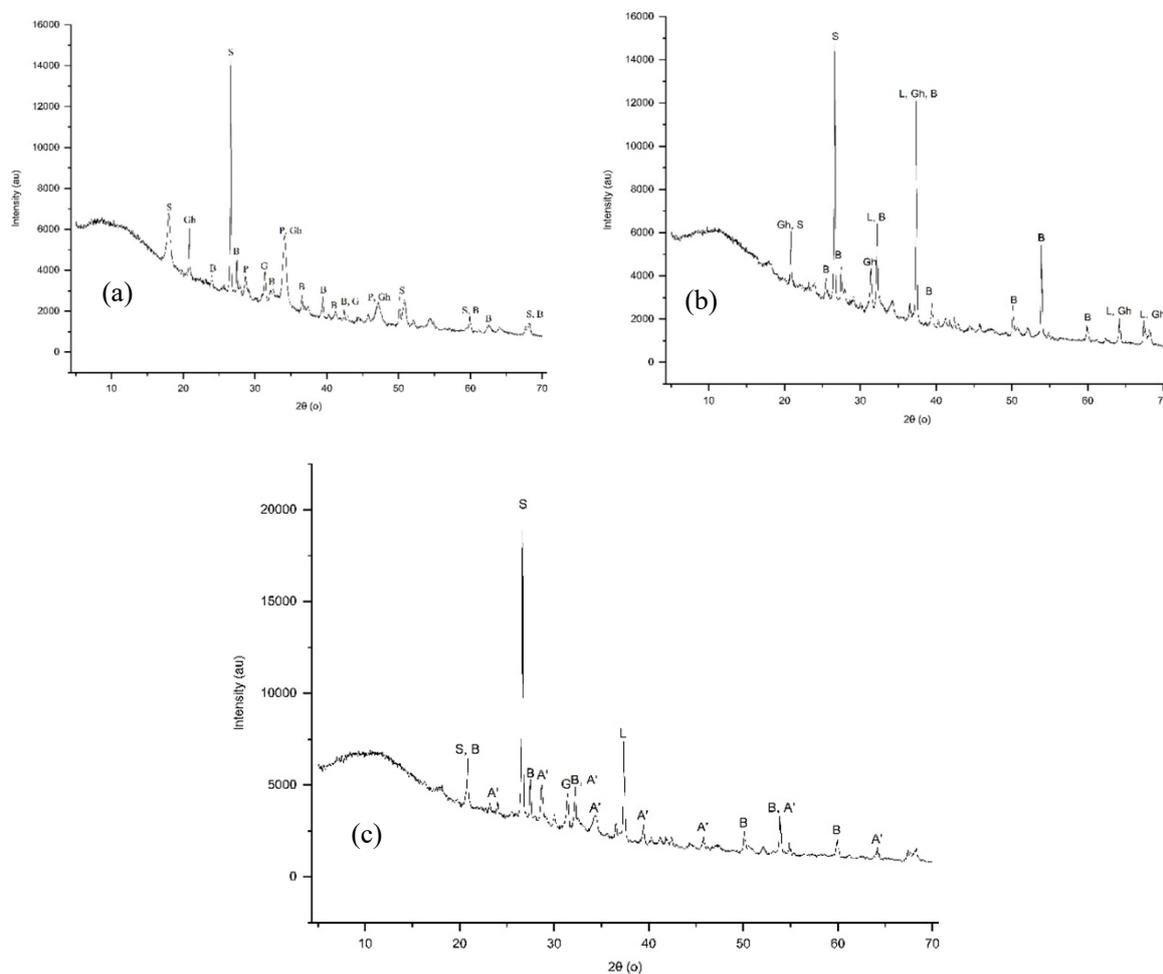


Fig. 4: XRD patterns of belite cements (a) CN (b) CG (c) CH (A': α' -C₂S; B: β -C₂S; G: γ -C₂S; S: SiO₂; L: lime; Gh: gehlenite; P: portlandite).

There were also other compounds that were formed in the belite cements that include γ -C₂S, wollastonite, and gehlenite that are undesirable. Although γ -C₂S can be activated by grinding, it is largely regarded as an inert compound when compared to α' -C₂S and β -C₂S. The presence of wollastonite signifies the incomplete reaction of calcium, and it has very poor hydraulic properties. Gehlenite is obtained through a reaction between calcium oxide and dehydroxylated clay minerals. It poorly reacts with water, and thereby has little hydraulic value.

3.2 Scanning Electron Microscopy (SEM) and Energy-dispersive X-ray (EDX) Analysis

The SEM analysis of CN and CH binders in Fig. 6a and Fig. 6c, respectively, show that the belite crystals obtained have an irregular shape with a particle size of less than 10 μ m which is in agreement with the findings of other studies [8,9]. The binders are expected to have improved hydraulic reactivity due to their small particle size. The microstructures of the hydrated pastes cured at 60 °C in hot air are shown in Fig. 7a and Fig. 7c for CN and CH, respectively. The C-S-H obtained in both pastes has a flake-like morphology. However, the CH paste produced a denser microstructure compared to the CN paste, which can be attributed

ions stabilizing more β -C₂S. The increase in compressive strength of the CNRST28 sample with respect to CHRST28 and CGRST28 samples is 213 percent and 110 percent respectively.

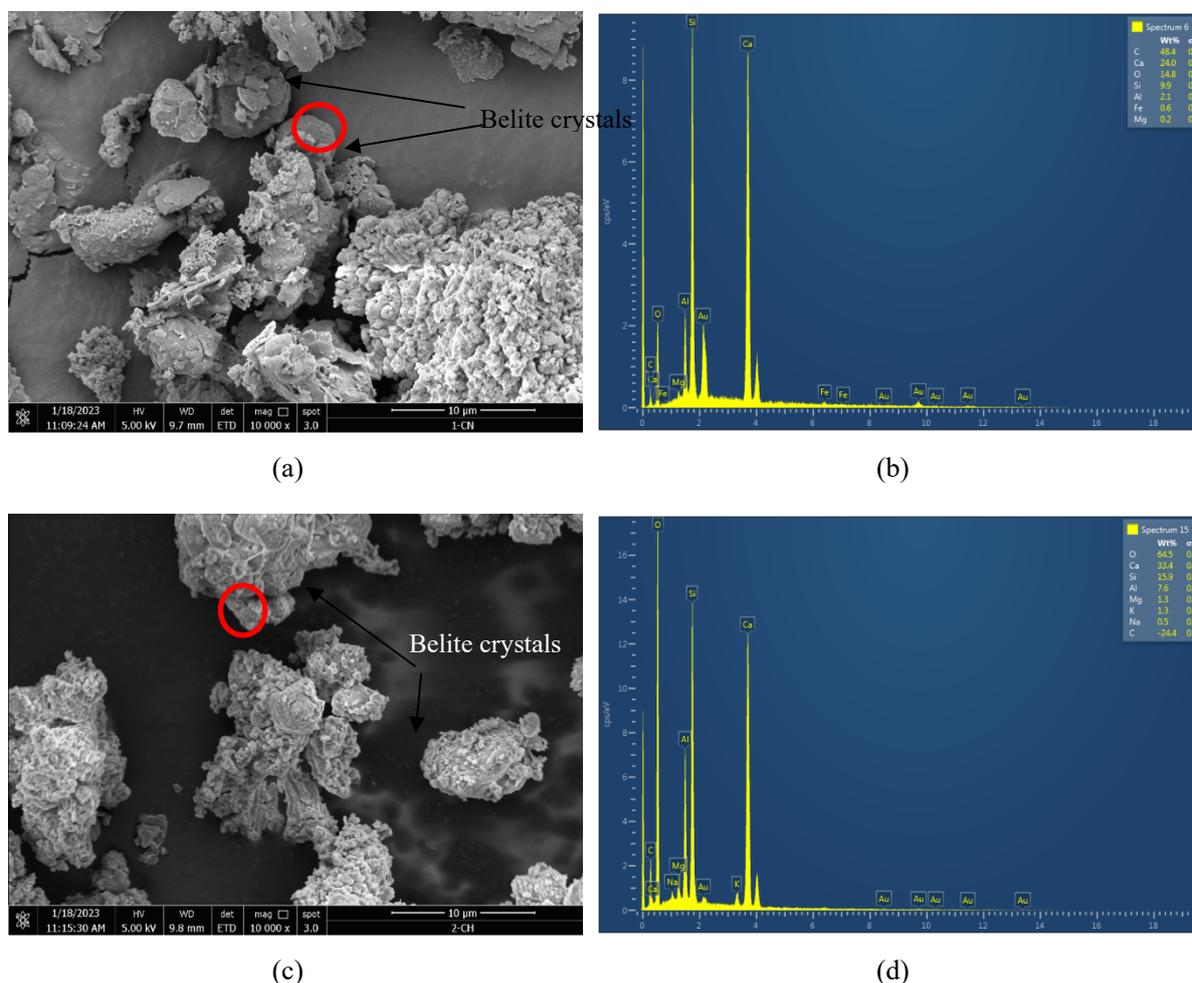


Fig. 6: SEM and EDX results of synthesized cements (a and b) CN (c and d) CH.

The strength after 7 days of curing increased with increasing curing temperatures. The CH90A7 obtained the highest 7-day strength due to the high reactivity of the α' -C₂S and curing temperature. However, the presence of a crossover effect was observed at 28 days in all samples cured at 90 °C, and there was a strength decrease of 9.8 percent between CH90A7 and CH90A28. From the compressive strengths obtained, the most preferred method of elevated temperature curing cannot be deduced between hot water and hot air curing methods. However, it can be concluded that a curing temperature of 90 °C is detrimental to the long-term strength of low temperature belite cements.

3.4 Ultrasonic Pulse Velocity (UPV) Test

The results of the UPV test conducted on the samples at 28 days are presented in Fig. 9. It can be observed that samples cured at room temperature obtained values between 3.11 and 3.91 km/s. The CHRLW28 and RHRLW28 samples had the highest UPV values of 3.91 and 3.84 km/s respectively which can be classified as ‘good’. This can be attributed to the faster hydration of α' -C₂S that produces a compact structure that reduced porosity and water absorption.

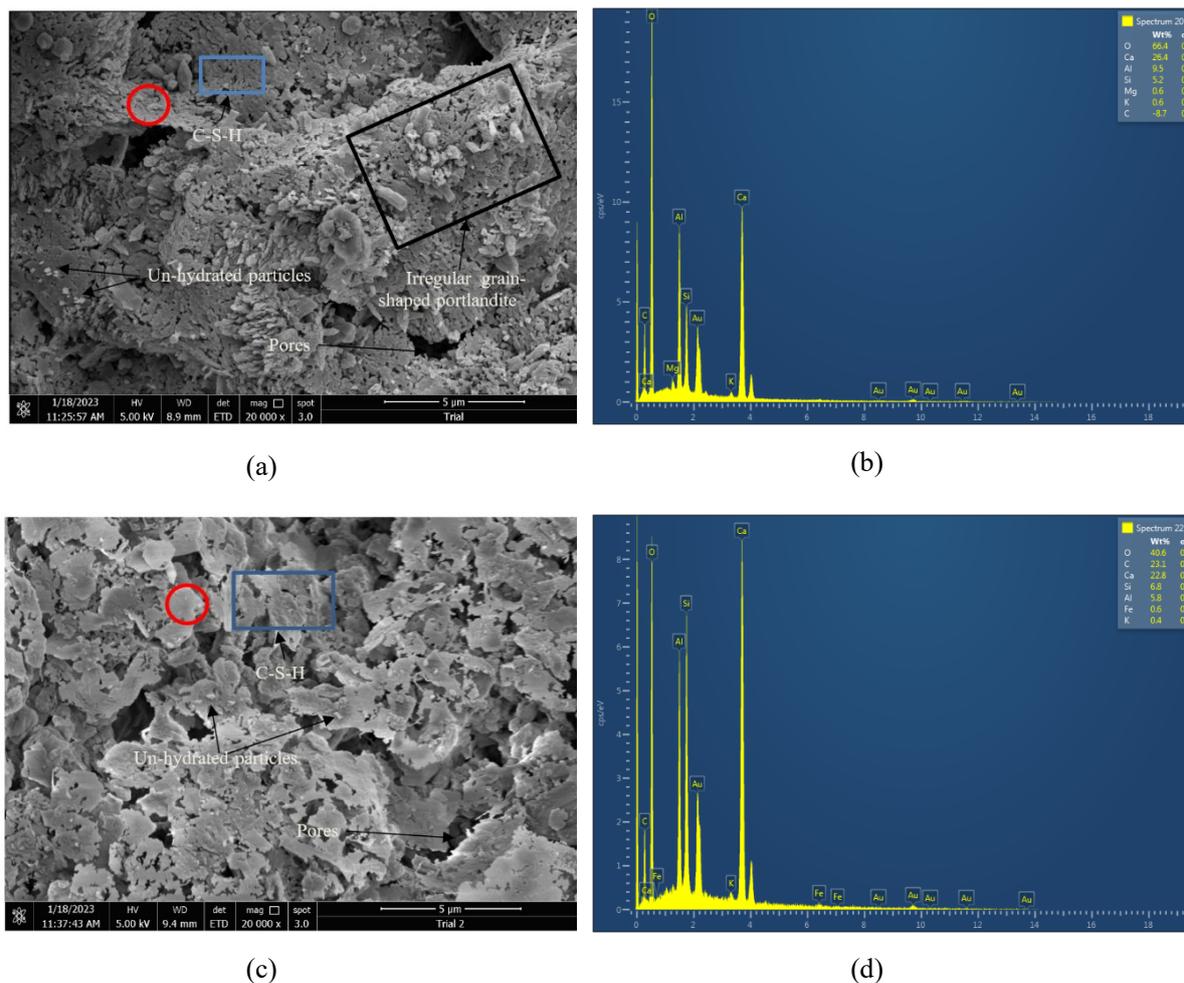


Fig. 7: SEM and EDX results of synthesized cement pastes cured at 60 °C, (a and b) CN (c and d) CH.

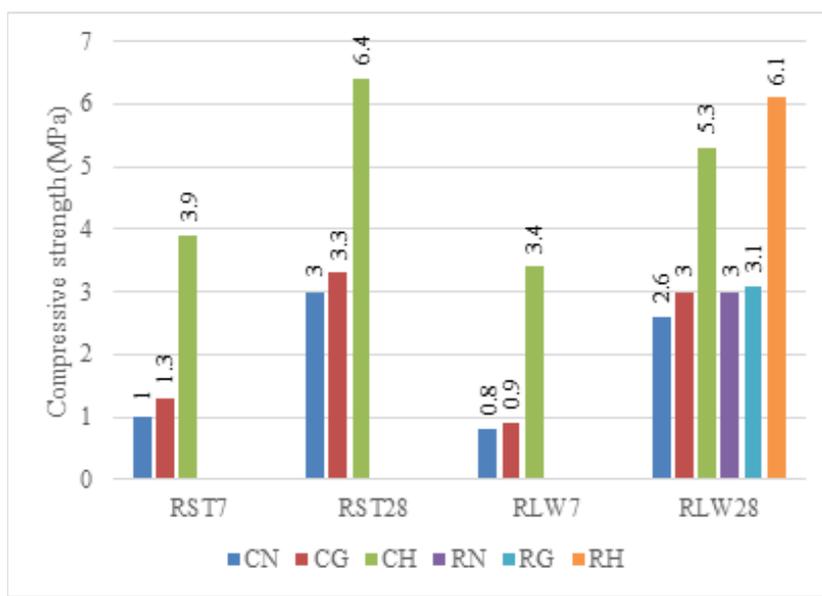
The mortar samples made from CN, CG, RN, and RG binders all had UPV values between 3.0 and 3.5 km/s which classifies them in the ‘medium’ category. This is due to the low reactivity of β -C₂S, hence, the slow hydration that led to the formation of fewer hydration products that can reduce porosity. The UPV values for all the samples cured at 90 °C, and CN60A28 and CG60A28 (samples cured in hot air at 60 °C) are all below 3.0 km/s. This can be because of the presence of flaws in the samples that may be due to the self-desiccation and rapid non-uniform formation of hydration products that resulted in internal cracking and the creation of pores.

Although other samples cured at 60 °C have values that place them in the ‘medium’ category, their long-term durability is still uncertain. The lowest values at all curing temperatures were obtained from samples cured in hot air due to more loss of internal water. Hence, curing in hot air will produce samples that will have high drying shrinkage that can negatively affect the durability of the samples.

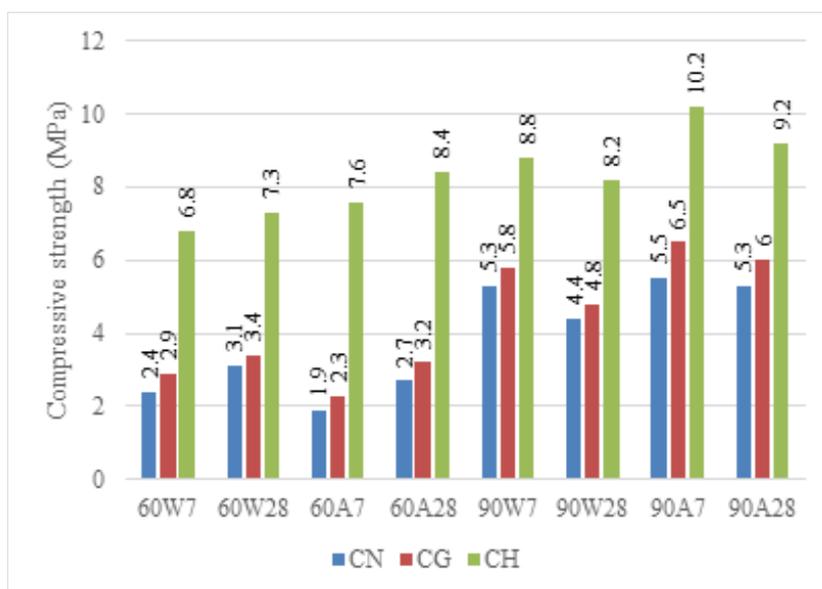
3.5 Effective Water Porosity and Water Absorption Coefficient

The results from the capillary water porosity and water absorption coefficient tests are presented in Fig. 10a and Fig. 10b, respectively. It can be observed from Fig. 10a that mortars prepared from the CH binder had the lowest capillary porosity at all curing conditions while those from the CN binder had the highest. This is due to the formation of a dense and porous microstructure by the CH and CN binders, respectively [18]. The results for the water

absorption coefficient test in Fig. 10b show an opposite trend with mortars from the CH binders having the least coefficient of water absorption. The water absorption coefficient decreases with an increase in both curing temperature and capillary water porosity. Also, the samples cured in hot water had a lower coefficient of water absorption than those cured in hot air at the same temperature. This can be due to the difference in microstructure, as the samples cured in hot water might have a more uniform production of hydration compounds. It is evident that both properties are affected by the nature of hydration compounds formed.



(a)



(b)

Fig. 8: Compressive strength development of samples cured at (a) room temperature, and (b) elevated temperature.

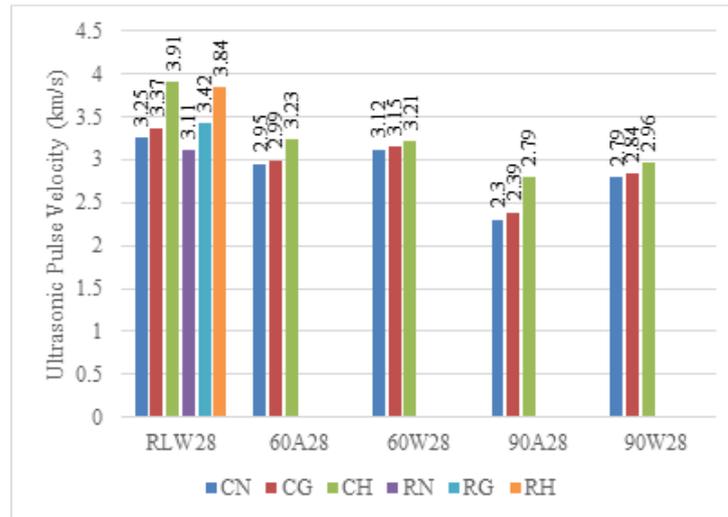
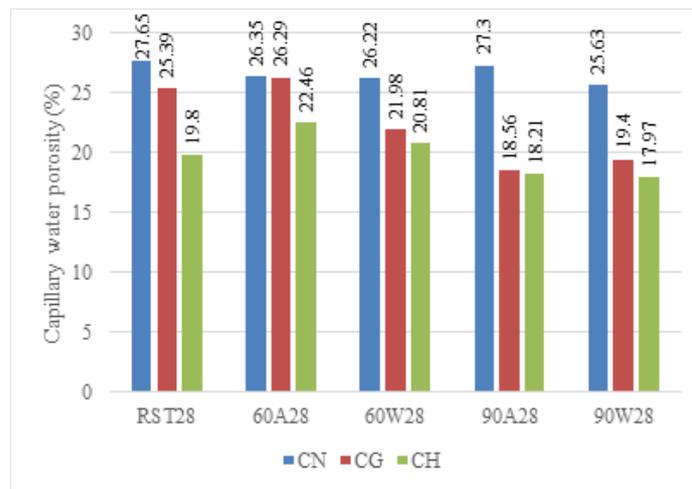
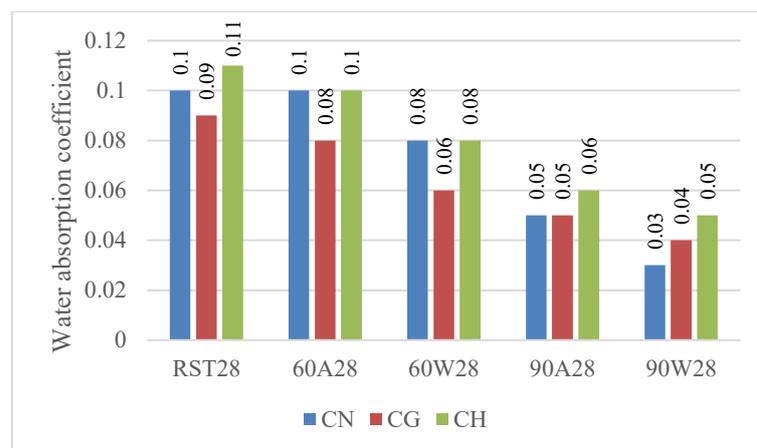


Fig. 9: UPV values of mortars with different curing conditions.



(a)



(b)

Fig. 10: Transport properties of mortars curing (a) capillary water porosity, and (b) water absorption coefficient.

4. CONCLUSION

The effect of curing conditions on the early strength of low temperature belite cements was assessed in this study from which these conclusions can be drawn:

- The development of early strength of low temperature belite cements is highly dependent on the curing temperature.
- Although the highest early strengths were obtained at a curing temperature of 90 °C, lower temperatures within the range of 60 °C are more suitable for improving the early strength of these mortars.
- The delay period of 24 hours reduced the loss of strength due to the crossover effect, and the loss is only visible in the 28 days strength of samples cured at 90 °C.
- The presence of the α' -C₂S polymorph is shown to have a beneficial effect on hydration and strength development at all curing conditions.
- Elevated temperature curing has a positive effect on the capillary water porosity and water absorption of these mortars.

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