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Alkali-Activation of a High MgO GGBS – Fresh and Hardened Properties

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Abstract

In this study, concretes and pastes were produced from a high MgO ground granulated blast furnace slag (MgO content 16.1 wt.%) by alkali activation with various amounts and combinations of sodium carbonate and sodium silicate. Sodium carbonate activators tended to reduce slump compared to sodium silicate at the same dose, and, in contrast to the literature for many blast furnace slags with more moderate MgO, to shorten the initial and final setting times in comparison with concretes activated by sodium silicate for dosages less than 10 wt.%. Higher heat curing temperatures and the use of larger dosages of alkali activators resulted in higher early age cube compressive strength values. The XRD analysis of 7 and 28 days old pastes activated with sodium carbonates revealed formation of gaylussite, calcite, nahcolite and C-(A)-S-H gel. Curing at 20˚C appeared to promote dissolution of gaylussite and calcite, while heat curing promoted their replacement with C-(A)-S-H, which also resulted in higher ultimate cube compressive strength values. Conversely, mixes activated with sodium silicate contained less crystalline phases and more disordered gel which strengthened the binder matrix.

Keywords: Alkali activated slag (AAS); alkali activators; workability; curing; compressive strength; setting properties.

1. Introduction

Alkali-activated concrete (AAC) has been developed and marketed as a low-CO_2 construction material, but its acceptance still faces several challenges (Provis 2017). Carbon dioxide emissions reduction and industrial waste valorisation could be achieved successfully by using alkali-activated slag (AAS) as an alternative to Portland cement, and potentially with lower cost in regions where blast furnace slag (BFS) is plentiful and activators can be readily sourced. BFS is a by-product of iron production and has some latent hydraulic properties, which can develop in cementitious products when hydrated either in the presence of Portland cement or in an alkaline solution (Neville 1995, Manmohan & Mehta 1981). Solidified alkali-activated materials can also show desirable mechanical, engineering and durability properties (Collins & Sanjayan 1999, Pacheco-Torgal et al. 2012, Provis & van Deventer 2014), including high early strength if adequate preparation and curing conditions are adopted.

The chemical composition of BFS and its fineness strongly influence the fresh and hardened properties of alkali activated materials (Douglas & Brandstetr 1990, Winnefeld et al. 2015, Bernal et al. 2011). Similarly, the type and the amount of alkali activator, the water/precursor ratio, mixing and curing procedures control both fresh and hardened matrix properties (Provis & van Deventer 2014). The relatively high content of calcium in BFS compared to most other precursor used in alkali activation promotes formation of Al-substituted C-S-H (C-(A)-S-H) gel (Bernal & Provis 2014, Wang & Scrivener 1995), though rapid dissolution and release of Ca, Si and Al. Activation of MgO-rich BFS by alkali silicate (Bernal 2016) tends to produce C-A-S-H type gel as the main binding phase and Mg-Al layered double hydroxide as the secondary phase; if an alkali carbonate alkali activator is used, calcium-rich carbonate phases are also observed. In general, the tendency of BFS to dissolve and the consequent reaction intensity increase with alkali concentration up to a certain point but not beyond this, (Nath & Sarker 2014) and excessive alkali dose can cause a reduction in 28-day compressive strength of alkali activated concretes. Sodium hydroxide (NaOH), sodium silicate ($Na_2O{\cdot}rSiO_2$), sodium carbonate (Na_2CO_3) and sodium sulphate (Na_2SO_4) are the activators most commonly used for alkali activation of BFS. The use of sodium silicate to activate blast furnace slags of normal composition (Fernández-Jiménez et al. 2003) tends to yield a denser microstructure and results in better mechanical properties, particularly at early age, in comparison with mixes activated with NaOH or Na_2CO_3. Alkali activation of BFS leads to incorporation of Al ions by the C-S-H gel, which forms the main binder phase in these

cements, and results in longer chains of C-A-S-H with two dimensional structures and periodic inter-chain cross-links. Aluminium uptake depends on the concentration of alkalis, and has been reported to be more extensive at higher temperature and humidity (Schilling et al. 1994). XRD analysis of BFS activated with sodium hydroxide and sodium silicate (Brough & Atkinson 2002) shows formation of low quantities of crystalline phases. The silica to alkali ratio of the activator (SiO_2/Na_2O ratio, denoted Ms, and with numerically very similar values whether defined as molar or mass ratio) is one of the key parameters defining the properties of alkali activated BFS. M_s values between 0.75-1.5 often produce the best mechanical properties across a range of curing conditions slag compositions, although the details of the mix formulation do play a significant role in defining the optimal activator modulus.

Sodium carbonate as an activator can raise the pH value of the pore solution of AAS concrete (Bernal et al. 2015) to similar values as observed in Portland cement concrete. It can also be considered more environmentally friendly, is cheaper and more widely available (Provis & van Deventer 2014, Bernal 2016), either as a secondary product from industrial processes or by mining alkali carbonate deposits followed by moderate temperature thermal treatment. The sodium carbonate is considered a weaker activator mainly because the reaction of Ca^{2+} from slag with CO_3^{2-} from the activator, which favours formation of calcite, must take place before the pH increases to the level (Bernal et al. 2015), which will induce silica dissolution and the formation of C-A-S-H. Blending of sodium silicate with sodium carbonate as activators increased the pore solution pH and induced faster reaction of slag, compared to when using sodium carbonate. However, these results have largely been obtained for slags with MgO contents < 10% (Ke et al. 2016), and it is known that high-magnesia slags can behave very differently from those in the low-MgO compositional rang during alkali activation, so it is important to investigate their response in the presence of different activators.

Research on fresh concrete properties for AAS activated is rather limited compared to the understanding of the material in the hardened state, but in general the available test results show that workability and the setting time are strongly affected by alkali content, modulus and the amount of slag (Qureshi & Ghosh 2013, Lee & Lee 2013).

The setting process of AAS concretes tends to show progressive and gradual stiffening, evidenced in engineering tests as a rather fast slump loss but not always rapidly reaching a fully set condition. An increased activator dosage (Živica 2007) tends to shorten the setting time.

The main objective of this study is to evaluate the effect of alkali activator type on the workability and setting time of alkali activated concretes based on a high-MgO blast furnace slag. Most previous studies were performed using low-MgO slag which appeared to produce mixes with significantly different properties from those produced from the high-Mg slag used here. The secondary objective is to determine the effects of curing temperature on the mechanical properties of these mixes, especially at early age. This will provide valuable information to underpin the full-scale deployment of alkali-activated concretes based on a wider range of slag chemistries than in currently the case in practice.

Materials and Methods

Ground granulated blast furnace slag (GGBFS, Merit 5000) provided by MEROX, Sweden was used in this study. The chemical composition of the slag shown in Table 1 was determined using a Panalytical Zetium XRF spectrometer. The physical properties were provided by the supplier. The mix proportions of the concretes studied are shown in Table 2. All concretes contained 450 kg/m^3 of GGBFS, and all the samples had water/slag w/s mass ratios of 0.45 for concretes, and 0.36 for pastes. The alkali activators used were: powdered anhydrous sodium carbonate (abbreviated SC) provided by CEICH SA (Poland), and liquid sodium silicate (SS) provided by PQ Corporation. The SS had alkali modulus Ms (by mass) SiO_2/Na_2O=2.2, with 34.37 wt.% SiO_2, 15.6 wt.% Na_2O and a solids content of 49.97 wt.%. Some mixes were activated with sodium silicate solutions of modulus 1 and 1.5, adjusted from the commercial SS by addition of sodium hydroxide pellets. The sodium hydroxide pellets had 98.5% purity and were also provided by PQ Corporation. The alkali activator doses used were 3, 5, 10 and 14 wt.% by mass of binder. In addition, a mixture of 5 wt.% sodium silicate and 5 wt.% sodium carbonate (by binder mass) was used, Table 2. The total aggregate content was 1663 kg/m^3 in all concretes. The all-in aggregates used were natural granite from Piteå, Sweden, and had particle size 0-8 mm, with a fine content of 70 wt.%.

The mixing procedure commenced with mixing of all dry ingredients for 3 minutes. Powdered sodium carbonate and/or liquid sodium silicate were combined with any additional mix water and left to stand

for one hour (to dissolve or equilibrate, respectively)before being added to the mixed dry materials, and then mixed for another 4 minutes. The slump was determined following ASTM C143 (ASTM International 2015). Cube specimens for compressive strength testing were casted into alkali-resistant polymer 100 mm cube moulds. Immediately after casting, the samples were sealed with plastic bags. Curing was carried out by two procedures. In the first procedure specimens were sealed and kept at 20±2˚C and 40-45% relative humidity (RH) until testing, while in the second procedure sealed specimens were heat cured at 65˚C for 24 hours then stored and kept sealed at 20±2˚C and 40% RH until testing. No weight changes were recorded. Sealing of specimens was done by tight wrapping in a plastic foil.

Initial and final setting times were determined following the IS 4031 implementation of the Vicat test (IS: 4031-PART 5-1988), measuring penetration using a 1-mm diameter needle and a plunger mass of 300 g. Initial set is defined to be reached when the penetration is 5-7mm from the bottom, and final set when there is no longer any visible penetration of the outer circle. Cube compressive strength was determined at 1, 7 and 28_days after casting, following SS-EN 12390-3 (SS-EN 12390-3). The loading rate was set to 10 kN/sec. The pH values of activator solutions were measured using a digital pH meter (HANNA-HI208). The XRD analysis was conducted on 7 and 28-day old powdered paste samples using a PANalytical Empyrean XRD unit operating with Cu Kα radiation, with step size 0.0262˚ 2Ɵ and total scanning time for each sample 16 minutes, with results evaluated using the HighScore Plus software.

2. Results and discussion

3.1 *Fresh state properties*

The type and amount of alkali activators strongly affected the fresh concrete properties. Mixes activated with SS generally had higher slump in comparison with SC-activated mixes, most probably due to the lubricating and dispersant effect of dissolved silicate, as also observed in the literature (Fernández-Jiménez et al. 2003, Rajesh et al. 2013), Figures 1-A and 2. Similar behaviour occurred in the mix activated with 5wt.% SS + 5wt.% SC. Higher dosages tended to strengthen this effect but also resulted in later rapid loss of workability (Figures 1 and 2), also consistent with previous studies (Fernández-

Jiménez et al. 2003, Rajesh et al. 2013). All mixes showed a progressive loss of workability (slump loss) which was more pronounced when 14wt.% of SS or SC were used, Figure 1-B.

A decrease in the SS alkali modulus from 2.2 to 1 resulted in an increase in both slump and measured pH values, which can be related to the effective increase of the Na_2O content which initiates the dissolution of slag grains, Figure 3, and also the lower viscosity of a lower-modulus silicate solution (Vail 1952). A higher pH value of the alkaline solution is known to enhance the dissolution rate of slag and thus to accelerate this step of the hydration process (Kovtun et al. 2015), this effect is sufficient to overcome the reduction in the content of dissolved silicate (which has a plasticising action as mentioned above), and so gives an increased slump, Figure 1-A. Fresh concrete properties, and especially the loss of workability, are directly linked to the recorded initial and final setting times, Figure 4-A and B.

Generally, mixes activated with 3 and 5 wt.% SS, showed the longest initial setting times, exceeding 230 minutes, independent of Ms, although these concretes also showed very low slump (Figure 1-A). Concretes activated with SC and concretes activated with higher amounts of SS (10 and 15 wt.%) had short initial setting times of between 25 and 50 minutes. The increase of the modulus Ms appeared to shorten the initial setting time, Figure 4A, which can be related to more intense dissolution of silica and alumina (Chang 2003). However, a decrease of the modulus of the SS from 2.2 to 1.0, at a dose of 3 wt.% caused the shortening of the final setting time from 120 to 78 hours, but these values were the longest final setting times among all the mixes tested. A decrease in the amount of SC resulted in a longer final setting time, and the combination of SC and SS in mix SC5-SS5 gave the initial setting after 100 minutes and the final setting after 50 hours.

Generally, mixes with higher alkali contents, showed shorter initial and final setting times, whether activated with SC or SS. This could be directly related to the higher alkalinity of these systems which caused faster and more extensive dissolution of the slag followed by a more intense precipitation of reaction products at early stages (Chang 2003, Bernal et al. 2011). The observed rapid loss of workability in the case of mix SC14 can be related to the initiation of C-A-S-H formation. Earlier studies indicate that even the usage of a retarder did not prevent loss workability of similar mixes after 30 min (Collins & Sanjayan 1998), a similar timeframe to the results shown here in Figure 1-B. Formation of hydrotalcite within the reaction products is controlled significantly by the MgO content of the slag; due to the high concentration of MgO (16.1 wt.%) in the ground granulated blast furnace slag used in this

work, much more extensive formation of this phase at early age may have contributed to the short initial setting time and accelerated the kinetics of reaction, particularly when SC was the activator (Ke et al. 2016).

3.2 *Hardened state properties*

The compressive strength results are shown in Figures 5-7. In general, a higher dose of alkali activators led to increased 1, 7 and 28-day cube strength values. The 1-day cube strength was higher for heat cured specimens, Figure 5, due to the increased dissolution of BFS and acceleration of the formation of strength-giving reaction products (Bakharev et al. 1999a). The lab cured specimens (20±2˚C) activated with 14 wt.% SS having Ms=1, and those with <14 wt.% SC, were not yet hard after one day of curing. After 7 days of lab curing cube strength values varied between 10 to 48 MPa, and between 16 and 60 MPa after 28 days. Longer curing times are known to promote further strength progress in ambient- cured specimens activated with both silicate and carbonate solution, as the slag continuous to progressively react with highly alkaline pore solution (Bernal et al. 2016).

The 28 day old concretes cured at 20˚C and activated with SC, or with 3 and 5 wt.% SS, showed higher final (28 day) cube strength values than heat cured mixes. Similar trends are common for OPC concretes (Verbeck & Helmuth 1968), as the lower curing temperature allows the development of a more mature final microstructure. Conversely, mixes activated with 10 and 14 wt.% of SS showed higher strength values when heat cured. Similar observations in the past for alkali-activated slags have been related to the decreased crystallinity of the C-A-S-H (Wang & Scrivener 2003), and also an inferred higher amount of aluminosilicate gel formation (Wang et al. 1994). The literature also indicates that heat curing tends to coarsen the pore structure due to a higher reaction rate for C-A-S-H, in comparison with the rate of diffusion, resulting in formation of interstitial pores in between denser precipitates to form a barrier that prevents further dissolution and dispersion of ions (Bakharev et al. 1999b). The present test results indicate that this possible coarsening of the microstructure could be overcome, at least at high activator doses, by the formation of stronger reaction products (and potentially related also to higher solubility in the more alkaline pore solution) which yield higher ultimate cube strength values.

The type and the relative amounts of phases formed by alkali-activation of BFS depend largely on the composition of the alkali activator and the BFS (Myers et al. 2017). The high MgO content of over 16

169 wt.%, present in the BFS used in this study BFS accelerates the kinetics of reaction and promotes
170 formation of hydrotalcite group minerals (Bernal 2016) as detected by XRD here, Figures 8 and 9. Other
171 reaction product phases detected by XRD include tobermorite-like C-A-S-H gels, as well as the
172 carbonate phases gaylussite ($Na_2Ca(CO_3)_2.5H_2O$), nahcolite ($NaHCO_3$) and calcite ($CaCO_3$). The HT
173 was observable as a crystalline reaction product only in heat cured mixes activated with SS, and in
174 mixes activated with 14 wt.% SC (both heat and lab cured), but may have been present in a disordered
175 form in other samples. The XRD results show the presence of C-(A)-S-H gels in all mixes at all ages
176 studied, where aluminium is incorporated into the silicate chains of the C–S–H structure (Wang &
177 Scrivener 2003, Richardson et al. 1994). This incorporation, even at early ages, results in longer chain
178 length in sodium silicate-activated slags than in hydrated C_3S or Portland cement systems (Wang &
179 Scrivener 2003, Myers et al. 2013). Mixes activated with high dosages of sodium carbonate showed
180 the predominant formation of nahcolite, calcite and gaylussite which seem to be favoured over
181 development of ordered structures within the C-(A)-S-H gel. This can be related to the reaction of Ca^{2+}
182 from slag with CO_3^{2-} from SC leading to its consumption from the solution, resulting in a lower degree
183 of formation as it is thus not available to react initially with dissolved silica (Bernal et al. 2015).

184 The type of gel formed is affected by the pH of the activating solution and the calcium content in the
185 system. Systems with pH over 12 and a high calcium content binder (i.e. those based on BFS, as is the
186 case here) tend to show formation of C-A-S-H in preference to N-A-S-H gel (Garcia-Lodeiro et al. 2011,
187 Palomo et al. 2014). Here, the mixes activated with sodium silicate had higher pH values than those
188 activated with sodium carbonate, Table 2. As shown in Table 1 the slag used here had a relatively high
189 content of MgO (16.1 wt.%). The MgO drives the removal of carbonate species from the activator, and
190 then from the pore solution if any remains after setting, through formation of hydrotalcite-group phases
191 and therefore makes the calcium available for formation of the dominant C-(A)-S-H gel (Ke et al. 2016).

192 The observed gaylussite tends to form at a very early age, even prior to initiation of C-A-S-H formation
193 (Fernández-Jiménez & Puertas 2001), due to the easier breakage of O-Ca bonds at lower pH in
194 comparison with O-Al and O-Si bonds (Ke et al. 2016, Bernal et al. 2015). As shown in Eq. (1), a high
195 dosage of sodium carbonate will provide an excess of Na ions, which will support gaylussite formation.
196 From Figures 8 and 9, gaylussite showed a weaker presence in the XRD data with increasing time of
197 curing (7 and 28 days), as it is considered a transient phase in sodium-carbonate activated slag binders,

forming initially but then being consumed, and its consumption is related to the development of more stable carbonates (Ke et al. 2016). Usually, once the C-A-S-H starts to form the gaylussite will tend to dissolve. However, the results presented here indicate that the combination of a higher dosage of sodium carbonate and laboratory curing at 20˚C could slow down or limit the dissolution process of gaylussite, some of which could still remain in the hardened binder matrix even after 28 days of curing.

$$2Na^{+} + Ca^{2+} + 2CO_3^{2-} + 5H_2O \rightarrow Na_2Ca(CO_3)_2 \cdot 5H_2O \quad \text{(gaylussite)} \qquad \text{……..eq. (1)}$$

Gaylussite was detected in 7 and 28-day old mixes activated with 14 wt.% SC, and with the combination of 5wt.% SS and 5wt.% SC under both lab and heat curing. In the previous study of Bernal (Bernal et al. 2016) using a mixed carbonate-silicate activator, pirssonite (a less-hydrous sodium-calcium mixed carbonate) was formed instead of gaylussite, but the observation of a carbonate double salt here is nonetheless consistent with that previous work.

4. Conclusions

The effects of carbonate activators and curing temperatures on alkali activated slag concretes produced from a high-MgO slag were studied. Key findings include:

- Concrete mixes activated with sodium silicate had a generally higher slump than with mixes activated with sodium carbonate due to the plasticity effect of the sodium silicate.

- Workability loss was more rapid for sodium carbonate activated mixes produced using this slag; this differs from results for lower-Mg slags where sodium carbonate-activated mixes often remain fluid for a much longer period, and then set only slowly. The workability of the AAS concrete mix activated by 14% SC has been lost after 12 minutes of mixing with water. It would be impossible to use that concrete mix for in-situ concrete produced in the factory and transported over a long distance. However, 12 minutes may be a sufficiently long working time for some precast concrete production of small size products, e.g. tiles or bricks, in a rapid process.

• AAS pastes activated by sodium carbonate have shorter initial and final setting time than pastes activated by sodium silicate; the setting times decrease with increasing activator dosage, and with decreasing SiO_2/Na_2O ratio in sodium silicate.

• The combination of 5 wt.% sodium silicate + 5 wt.% sodium carbonate as activator appeared to increase the slump, initial and final setting time with no substantial loss of early or later compressive strength.

• Heat curing and higher dosages of alkali activators produced higher early age and ultimate cube strength values, and appeared to mitigate the loss of final strength that was induced by heat curing of concrete with lower activator dose.

• 28-day old pastes contain C-(A)-S-H, hydrotalcite-group phases and carbonates including gaylussite, nahcolite and calcite as reaction products.

• Curing at 20˚C promotes dissolution of gaylussite and calcite; heat curing promotes the replacement of gaylussite with C-(A)-S-H, which resulted in higher ultimate cube strength values.

• Mixes activated with sodium silicate contain less crystalline phases and more disordered C-A-H gel, which strengthened the binder matrix. Some traces of ordered C-A-S-H phases were observed, but only at lower dosages of sodium silicate or when sodium carbonate was used as a co-activator.

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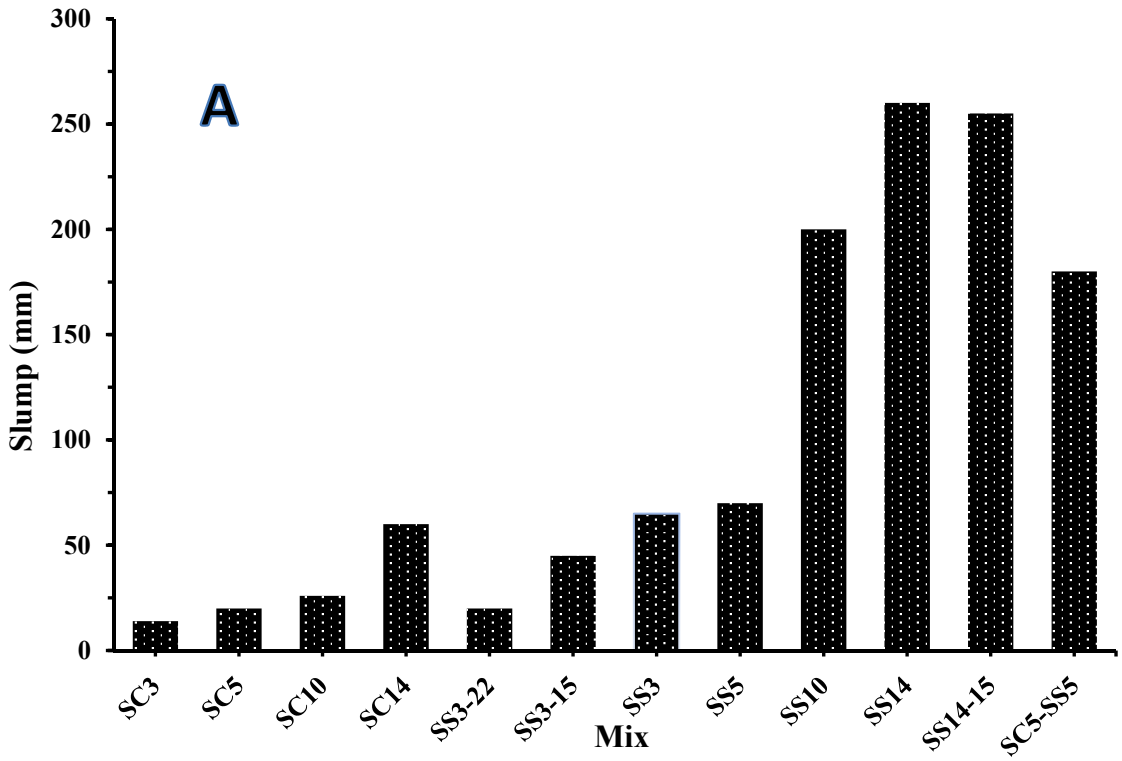
List of Figure and Table Captions



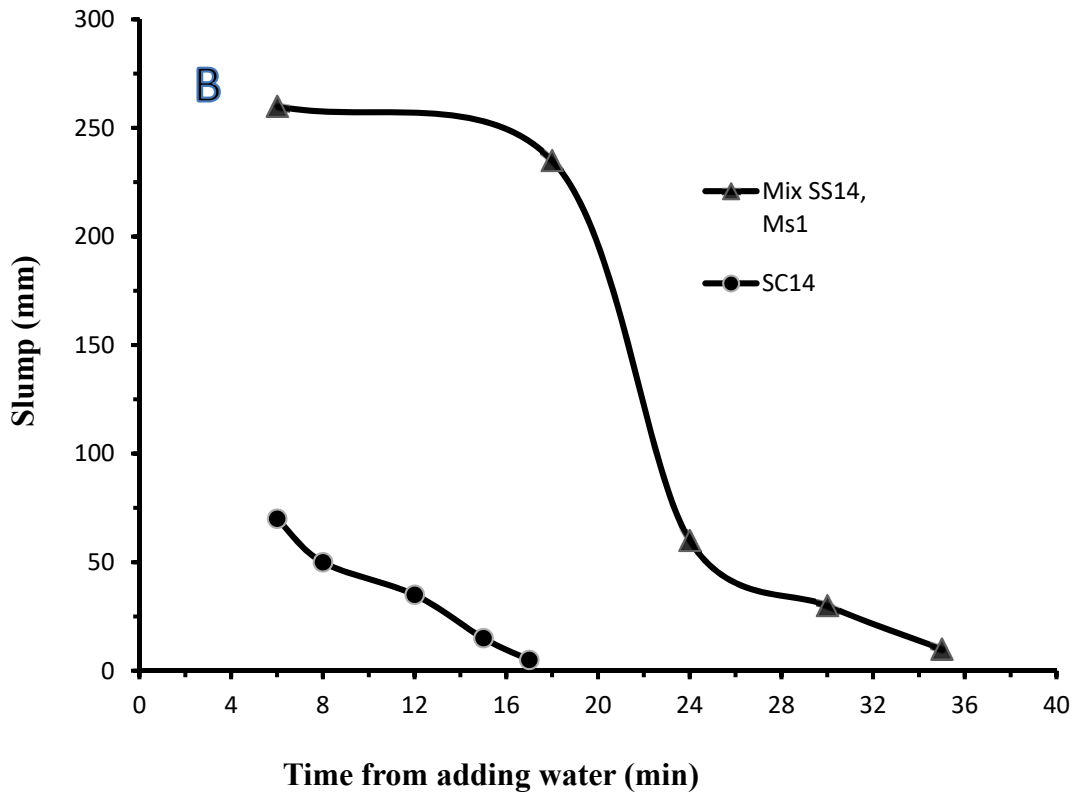


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Figure 1: Effect of activator type and its proportion on workability, A: Slump test results after mixing about 6 min, B: workability loss of mixes with 14% sodium silicate SS and 14% sodium carbonate SC.

Figure 2: Results of slump testing of mixes with sodium silicate activator.

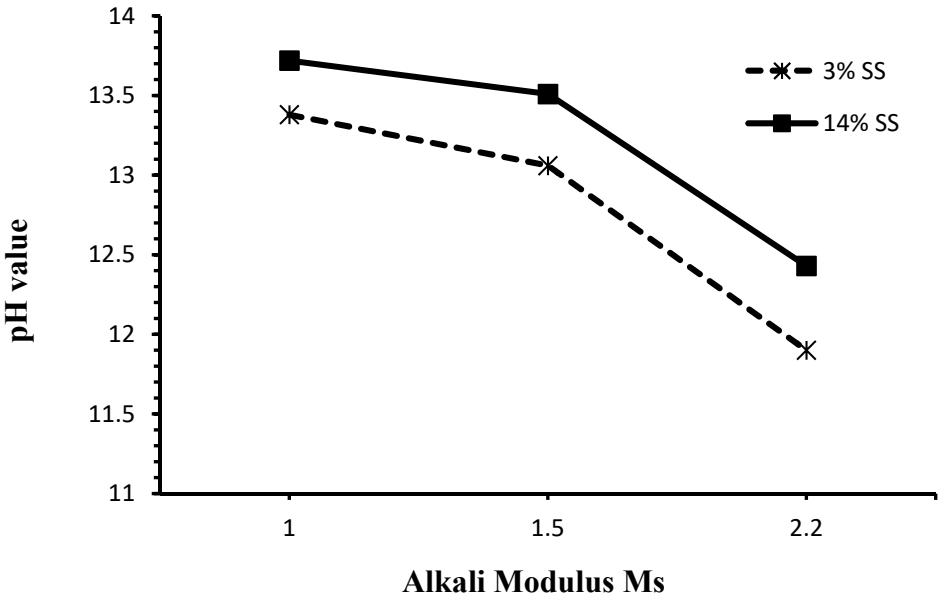
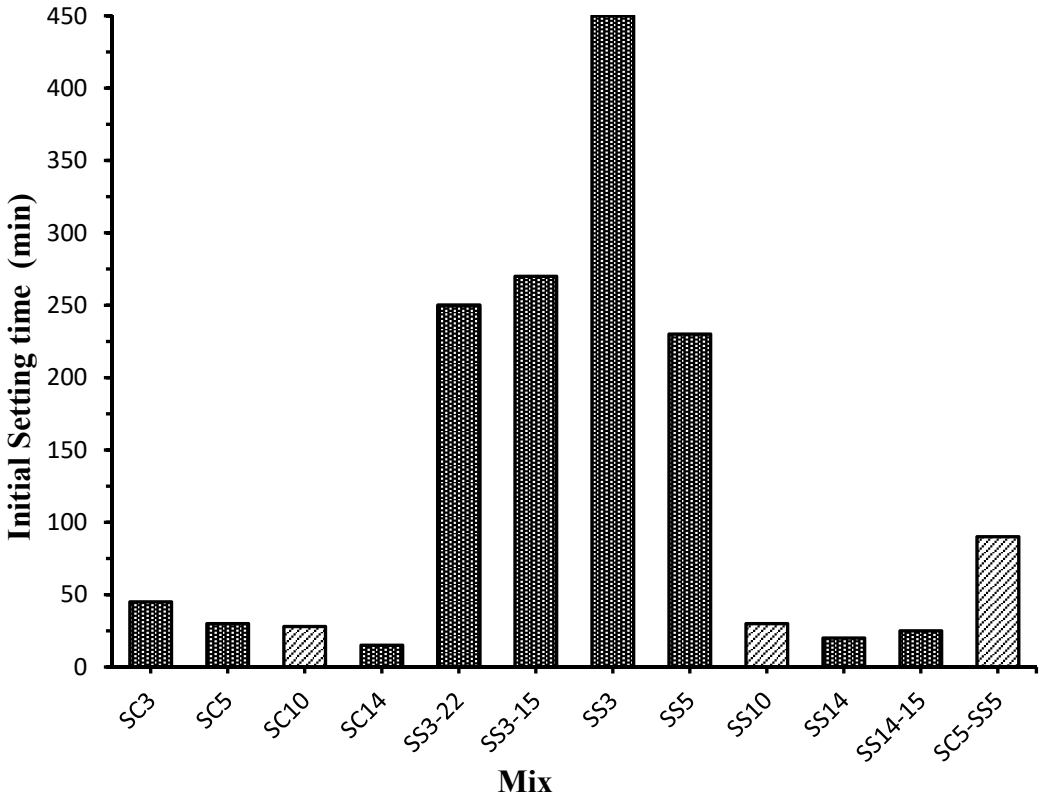


Figure 3: Relationship between M_s and the measured pH of the sodium silicate activator solution.



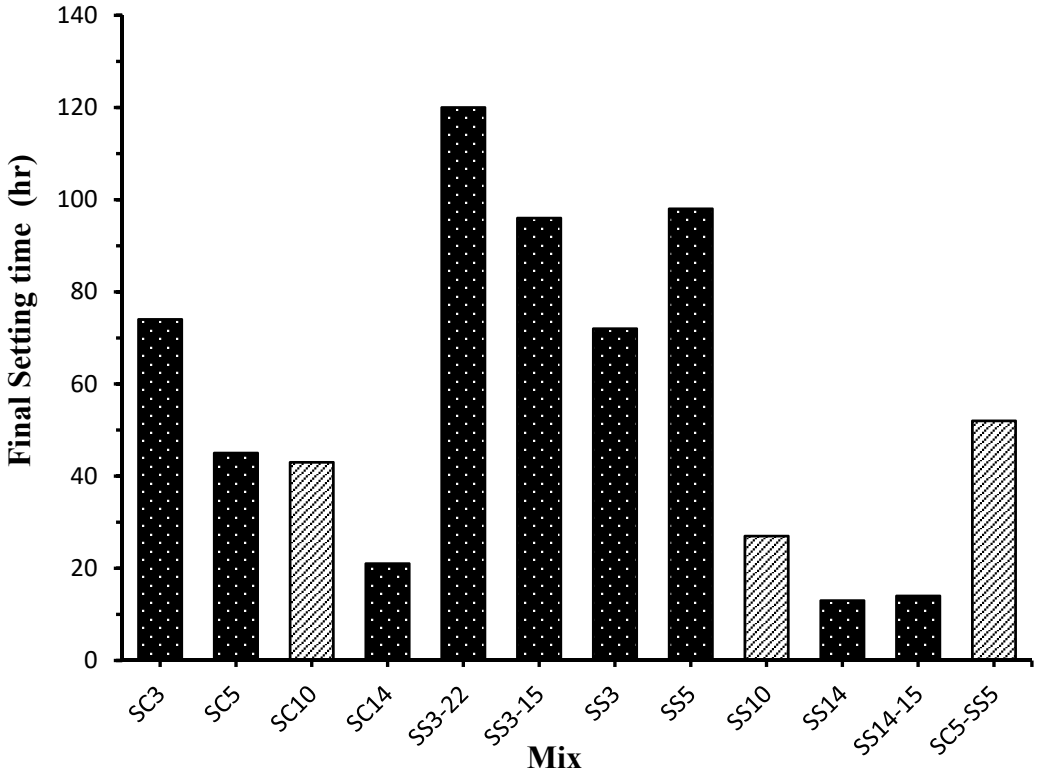


Figure 4: A: initial setting times for AAS pastes in minutes; B: final setting times for AAS pastes in hours.

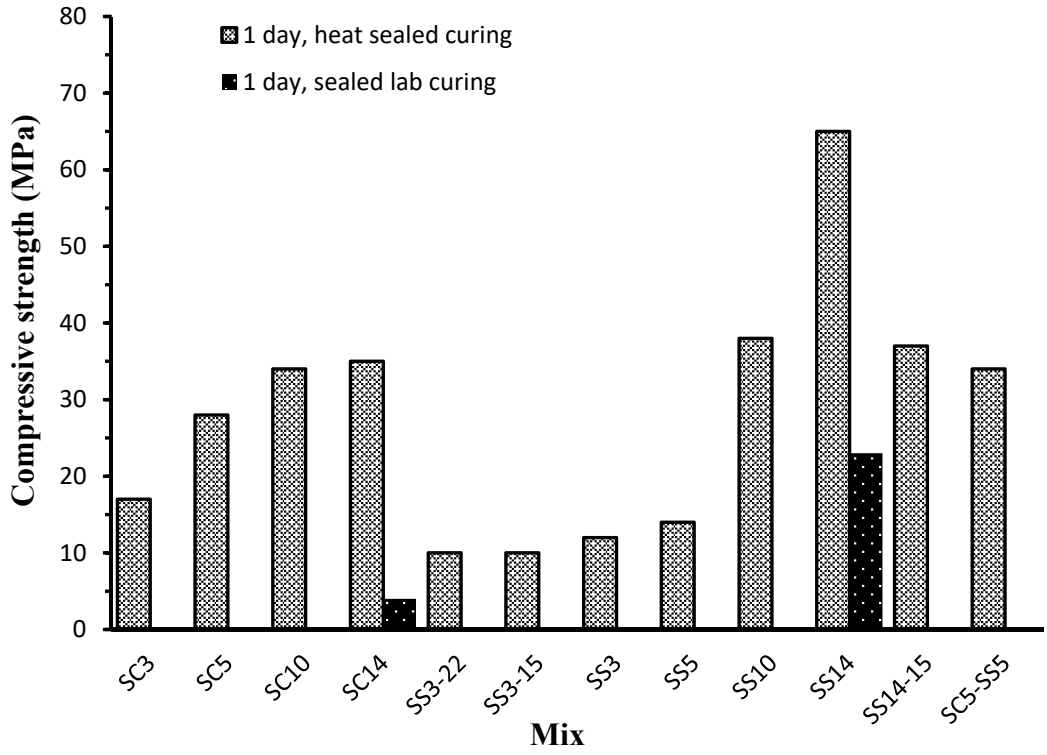


Figure 5: 1 day compressive cube strength results for concretes.

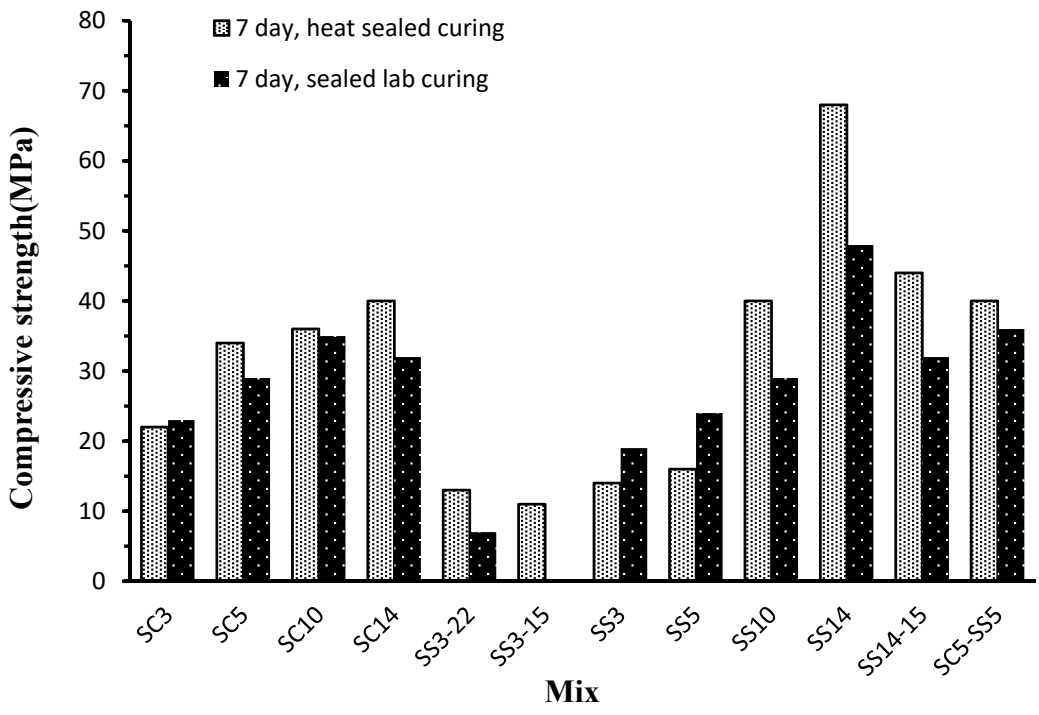


Figure 6: 7 day compressive cube strength results for concretes.

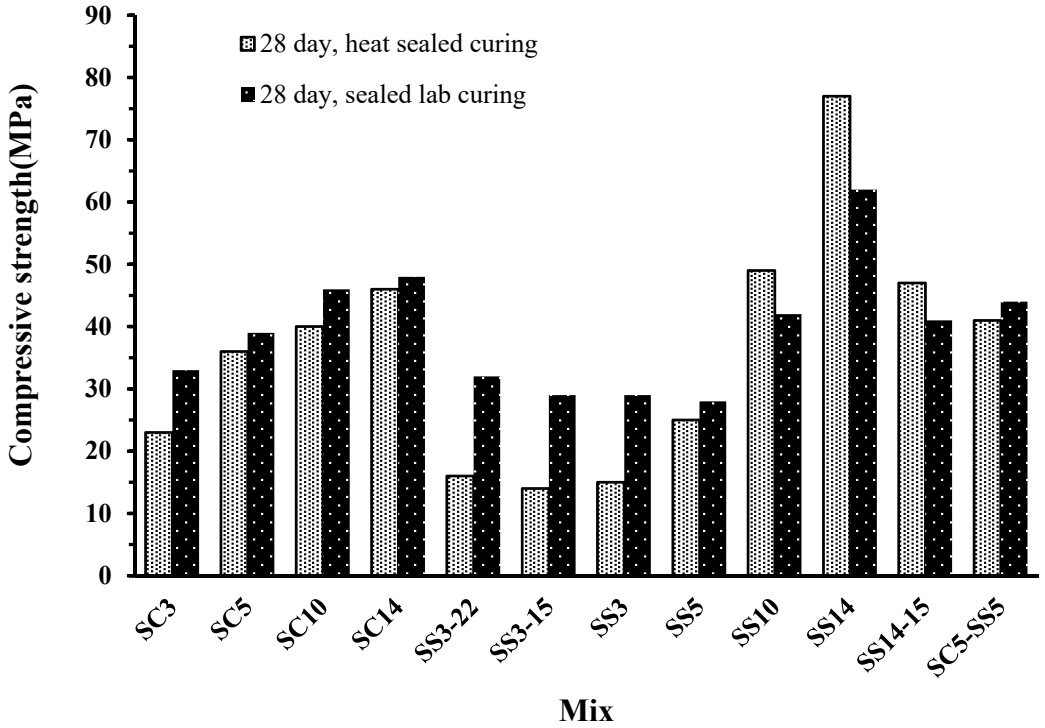


Figure 7: 28 day compressive strength results for concretes.

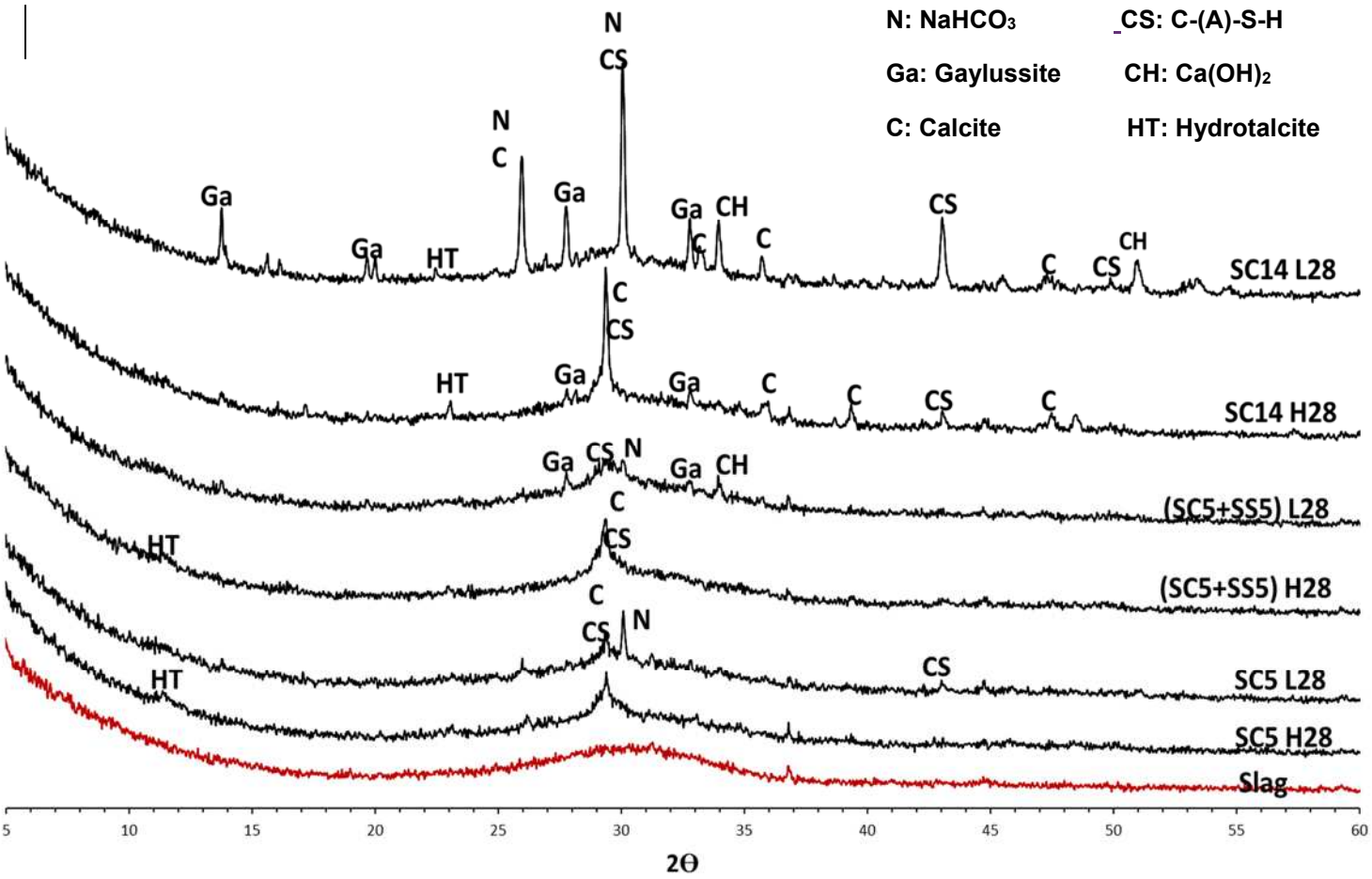


Figure 8: XRD results for 28 day old AAS pastes activated with SC. Suffix L28 denotes 28 days of lab curing and H28 denotes 28 days of heat curing.

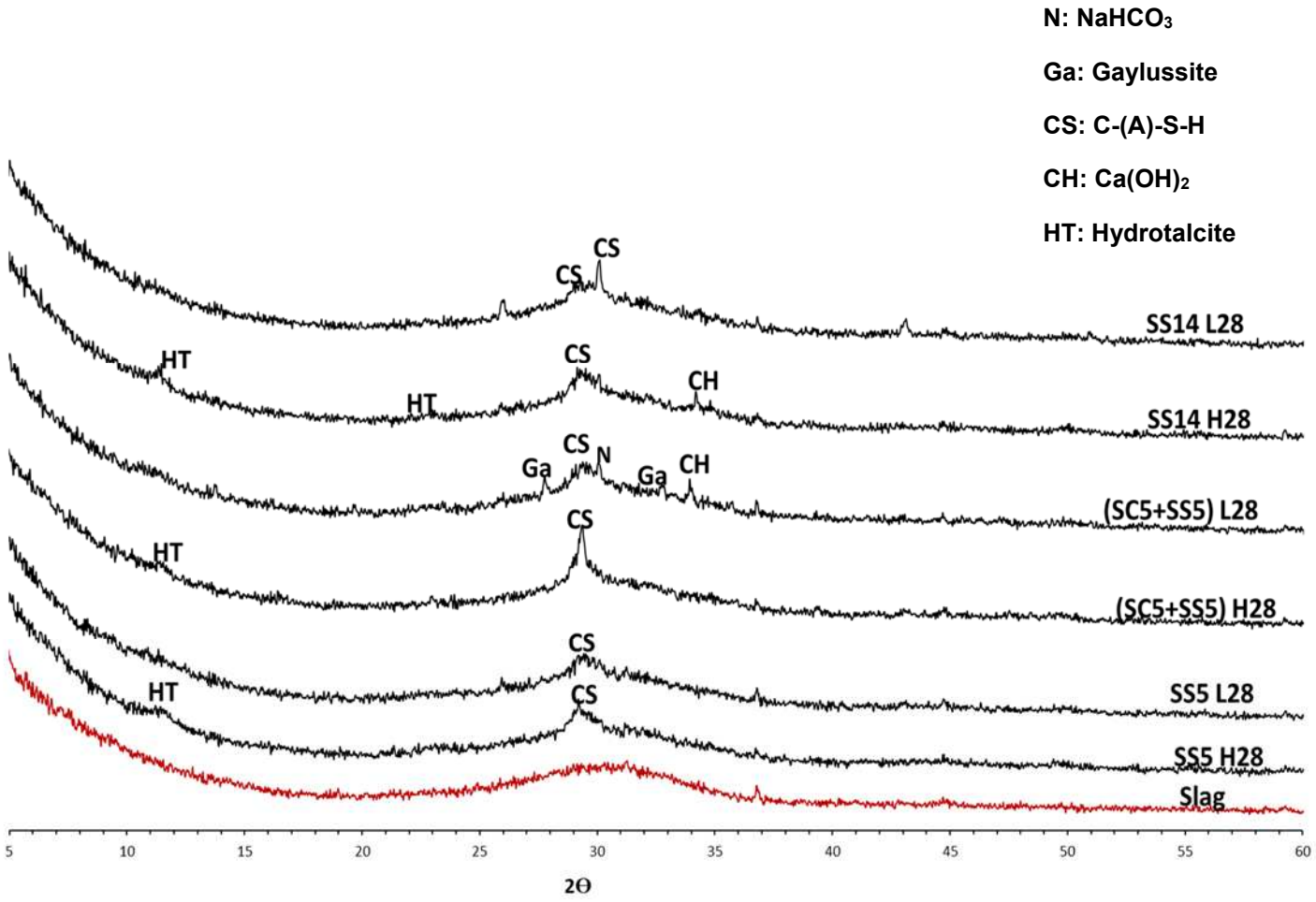


Figure 9: XRD results for 28 day old AAS pastes activated with SS. Suffix L28 denotes 28 days of lab curing and H28 denotes 28 days of heat curing.