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Microstructure and Strength Development of Sodium Carbonate-Activated Blast Furnace Slags

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ABSTRACT

This paper presents the study of alkali activated slags where sodium carbonate acts as a primary activator. The slow activation mechanism of sodium carbonate is accelerated by sodium hydroxide and with traces of calcium hydroxide. Strength development and the progress of hydration of the mixes are studied with the phase transformation and development of microstructural features through quantitative techniques such as thermogravimetric analysis and phase-identification techniques such as Fourier transform infrared spectroscopy and X-ray diffraction. Sodium carbonate replacement with sodium hydroxide and the presence of calcium hydroxide in the binder as a replacement of the slag enhances the rate of dissolution of slag leading to a faster strength development. Calcium hydroxide is significantly increased the compressive strength even at early age. On the other hand, sodium hydroxide substitution is effective at later ages of the reaction when used at high dosages (e.g. 40%). Formation of strength giving phases such as hydrotalcite and calcium aluminum silicate hydrate are confirmed with the microstructure analysis and explains the strength development.

Keywords: Alkali activated materials; compressive strength development; microstructure; slag; sodium carbonate.

1. INTRODUCTION

Portland cement (PC) production is responsible for 5-8% of global CO₂ emissions (Scrivener and Kirkpatrick, 2008). Thus, in the past two decades, alkali activated materials (AAM) have gained increased attention as an environment friendly alternative to PC in concrete and are now available in several countries as a commercial cementitious product (van Deventer et al., 2010; Provis and van Deventer, 2014; Provis, 2014). It has been found that the production of AAM leads to 55%-80% reduction in CO₂ emissions compared to PC (Yang et al., 2013; Duxson et al. 2007) and is less energy intensive when compared to PC production (Abdalqader et al., 2016).

AAM are produced by the dissolution of aluminosilicates from pozzolanic waste materials in alkaline environment which subsequently leads to the precipitation of hydration products (Abdalqader et al., 2016; Shi et al. 2006). There are several factors such as fineness, composition, mineralogy of the precursor used, the type and concentration of the alkali activator, the mixing procedure, and the curing conditions that affect the microstructure of AAM (Ke et al., 2016; Winnefeld et al., 2014). Especially, the type of activator and raw material are two of the most important factors as they determine the fresh and hardened state properties of AAM.

The most common activators used in alkali activation are sodium/potassium hydroxides and sodium silicate as they have the dual effect of raising the pH as well as facilitating the dissolution (Provis and van Deventer, 2014). However, they have several disadvantages such as they are expensive and are caustic in nature (Ke et al., 2016; Turner and Collins, 2013; Abdalqader et al., 2016). Therefore, naturally available sodium carbonate (NC) is gaining attention as the cost effective and environmentally friendly alternative to synthetic alkali hydroxides and silicates (Ke et al., 2016; Luukkonen et al., 2018). Turkey has the second largest NC reserves with 840 million tons (Marcu et al., 2015) out of the more than 25 billion tons worldwide. Utilization of this great potential in AAM production would contribute to the sustainability. However, NC activators suffer from prolonged setting time and delayed strength development (Ke et al., 2016). It is of vital importance that the pH level of the solution should be at desired level to facilitate dissolution of the precursor which in turn leads to the formation of calcium aluminum silicate hydrate (C-(A)-S-H) type gels which provide the strength (Huanhani et al., 1993).

Since the pH of the NC solution is below 12, the dissolution process is slow (Ke et al., 2016). The slow reaction mechanism of NC-activated slag was explained in different studies. Jimenez and Puertas (2001) reported that the reason of the prolonged stiffening of NC-activated slags is the initial formation of carbonate salts. Bernal et al. (2014) indicated that the CO_3^{2-} ion concentration in the pore solution and the initially precipitated calcium carbonate govern the activation process to a significant extent. It is known that dissolved Ca^{2+} ions from the slag first react with CO_3^{2-} from NC solution and generate carbonate salts such as calcite (CaCO_3) and gaylussite ($\text{Na}_2\text{Ca}(\text{CO}_3)_2 \cdot 5\text{H}_2\text{O}$) and increase pH through the release of hydroxide ions. This reaction appears before the precipitation of C-(A)-S-H gel which consumes Ca^{2+} , hinders the increase of pH and causes dissolution deceleration of silicate species. Therefore, both the hardening and stiffening processes are considerably low due to the lack of Ca^{2+} ions and the presence of the CO_3^{2-} ions in the system.

In previous studies, acceleration methods of NC-activation mechanism is investigated with different accelerators. Kovtun et al. (2015) reported that NC activation of slag can be accelerated using ordinary PC combined with mixture of silica fume and slaked lime as admixtures. It was also noticed that replacing NC with sodium hydroxide (NH) increases the pH further and facilitates faster dissolution of the slag which will result in noticeable acceleration of strength development. Temuujin et al. (2009) observed the improvement in mechanical properties of fly ash based geopolymers cured at room temperature with the addition of calcium compounds. In that study, calcium hydroxide is found to be more effective than calcium oxide and it was reported that calcium compound addition negatively affected the mechanical properties of geopolymer cured at 70°C. On the other hand, according to Kim et al. (2013) calcium oxide was found to be more effective than calcium hydroxide in activating slag. In another research (Abdalqader et al., 2015), the effect of combining MgO and NC on the reaction kinetics of slag/fly ash activation was examined and it was concluded that MgO incorporation significantly influenced the reaction rate. Increasing the alkali content resulted in an increase of the strength at all ages as well. Alternative accelerators like calcined layered double hydroxides (Ke et. al (2016)), reactive magnesia (Abdalqader et al. (2015)) and reactive MgO (Jin F. et. al (2015)) are also reported as an effective means of NC activation. Atis et al. (2009) studied the influence of activator type on the strength development of alkali-activated slag mortar and found that NC activated slag mortar exhibited

comparable setting time and strength with respect to PC mortar. The prolonged formation of sodium calcium carbonate results in a longer setting time for slag activated by NC in contrast to quicker setting systems with sodium silicate activation. NC activation proceeds by pozzolanic reactions that account for compressive strengths similar to PC mortars which increase with Na content.

The studies mentioned above have predominantly focused on the methods to accelerate the reaction kinetics of NC activated slag by either increasing the alkali content or incorporating with reactive admixtures. However, there are very limited research studies which investigate and compare the effect of the alkali concentration and reactive admixtures on the strength development and microstructure analysis of NC activated slag in detail. The aim of the current study is to study the effect of accelerators such as NH, calcium hydroxide and combination of these accelerators on the strength development and microstructure of alkali activated slags cured at ambient temperature.

2. MATERIALS and METHODS

2.1 Materials and Mix Design

Ground granulated blast furnace slag (GGBS), with a basicity coefficient ($[\text{CaO}+\text{MgO}]/\text{SiO}_2$) of 1.02 and a hydration modulus ($[\text{CaO}+\text{MgO}+\text{Al}_2\text{O}_3]/\text{SiO}_2$) of 1.33, was supplied by Eregli iron and steel plant, Turkey. Slaked lime ($\text{Ca}(\text{OH})_2$) was used as carbonate binding agent. The physico-chemical properties of GGBS and slaked lime (CH) are shown in Table 1.

Table 1. Physico-chemical composition of GGBS and CH

Materials	GGBS	CH
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Chemical composition (%)	SiO ₂	40.6	0.8
	Al ₂ O ₃	12.6	0.4
	Fe ₂ O ₃	1.2	1.2
	MgO	5.8	1.3
	SO ₃	0.1	-
	Na ₂ O	0.8	-
	CaO	35.7	69.0
	K ₂ O	0.7	0.13
	TiO ₂	0.7	< 0.1
	Mn ₂ O ₃	0.8	-
	S	0.7	0.22
	Cl (ppm)	187	226
	LOI (%)	0.13	26.03
Specific surface area (cm ² /g)		5136	-
	Specific gravity (g/cm ³)	2.93	2.32

The particle size distribution was determined by laser diffraction and the median particle sizes (d_{50}) of GGBS and CH were found to be 8.0 μm and 2.2 μm respectively, as shown in Fig. 1.

Fig. 1. Particle size distribution of GGBS and CH

Analytical grades of NC and NH were used as alkaline activators at 6% and 10% by weight of the binder and were prepared by pre-dissolving in tap water. NC was selected as the primary activator, while NH was used as secondary activator, replaced with NC at 20% and 40% by weight of the total activator amount. CH was used to accelerate the hydration process of the NC-activated slag. The replacement ratio of CH was chosen as 3% by weight of slag, which demonstrated the optimum amount to accelerate the stiffening sufficiently among a series of trial mixtures. The water to binder ratio was set as 0.35 and

0.45 for paste and mortar mixes, respectively. In mortar production, CEN Standard sand was used as aggregate proportioned with a volume of 50%.

Table 2 provides the proportions of the mixes. A total of 12 mixtures were prepared and each mixture was coded as shown in Table 2, where NC refers to Na_2CO_3 , the first number refers to NC percentage in the total activator concentration, the second number refers to total activator amount of mix by weight of the binder and “L” refers to slaked lime.

After the production, all samples were exposed to laboratory conditions ($20\pm 2^\circ\text{C}$ and $55\pm 5\%$ RH) until the test day.

Table 2. Mix proportions

Mixes	Activator concentration (%)	Binder types and proportions (%)		Activator types and percentages by weight (%)	
		GGBS	Ca(OH) ₂	Na ₂ CO ₃	NaOH
NC100-6	6	100	0	100	0
NC80-6				80	20
NC60-6				60	40
NC100-10	100			0	
NC80-10	10			80	20
NC60-10	60			40	
LNC100-6	6	97	3	100	0
LNC80-6				80	20
LNC60-6				60	40
LNC100-10	100			0	
LNC80-10	10			80	20
LNC60-10	60			40	

2.2 Methods

2.2.1 Compressive strength development

Compressive strength was determined according to the ASTM C109 (2016). The mortar samples were cast into 50 mm cubic molds. Afterwards, the molds were covered with a plastic sheet to prevent

any moisture losses. All specimens were kept in laboratory conditions ($20\pm 2^{\circ}\text{C}$ and RH $55\pm 5\%$) until the test day. The compressive strength test was carried out at 3, 7, 28 and 90 days and the averages of three repetitions were considered as the compressive strength for each age.

2.2.2 X-ray diffraction (XRD)

The XRD analysis was performed at 3, 7 and 28 days on powder of the paste samples and the patterns were collected in the range of 5° to 55° (2θ) with a step size of 0.02° (2θ) and a measuring time of 1 s/step.

2.2.3 Fourier transform infrared spectroscopy (FTIR)

In the current study, the FTIR spectra for the reaction products were recorded using a Bruker Tensor 27 FTIR Spectrometer. All spectra were obtained with 32 scans from 4000 to 400 cm^{-1} per spectrum. The band between 600 and 1800 cm^{-1} was considered for analyzing the results, since the reaction products were determined at this range. XRD and FTIR analysis were used to describe and assess the hydration products of the paste samples for 3, 7 and 28 days of curing.

2.2.4 Scanning electron microscopy

The microstructural analysis of the accompanying paste samples were performed after a 28 day period. Small, broken paste specimens were placed on brass stubs using carbon tape, coated with Au, and then analyzed at an accelerating voltage of 15 kV with a ZEISS EVO LS10 SEM. For examinations by the SEM, secondary electron imaging were used. Atomic ratios were determined using EDS analyses of 80 spots per sample.

2.2.5 Thermogravimetric analysis

The reaction products of the NC-activated slag pastes were analyzed by thermogravimetric and derived thermogravimetric analyses (TG-DTG) at the heating rate of $15^{\circ}\text{C}/\text{min}$ from 20°C to 900°C using a platinum holder and nitrogen as the carrier gas. About 20-25 mg powder of the paste samples were used and TGA-DTG analysis was conducted at the curing age of 28 days. The mass loss at different temperatures was used to quantify the amounts of reaction products.

3. RESULTS and DISCUSSION

3.1 Compressive Strength Development

Figs. 2 and 3 show the strength development of the GGBS mortars with and without CH replacement, activated with only NC and combinations of NC+NH solutions with varying ratios. The 3, 7, 28 and 90 days compressive strengths of mixtures range from 0.5 to 20.4 MPa, 1.2 to 32.5 MPa, 3.1 to 44.4 MPa and 3.4 to 44.5 MPa (Table 3), respectively, which shows that some of these mixes may use in constructions with different strength requirements.

Fig. 2. Compressive strength of NC-6 and LNC-6 mixes

Fig. 3. Compressive strength of NC-10 and LNC-10 mixes
Table 3. Compressive strength results and standard deviation values

Mixes	Compressive strength (MPa) / (Standard deviation)			
	3 days	7 days	28 days	90 days
NC100-6	0.6 (0.1)	1.4 (0.3)	3.1 (0.4)	3.4 (0.1)
NC80-6	0.5 (0.0)	1.2 (0.1)	4.6 (0.2)	4.8 (0.9)
NC60-6	1.0 (0.0)	16.6 (0.3)	19.0 (1.0)	19.6 (0.3)
LNC100-6	9.4 (0.1)	13.9 (0.6)	15.0 (1.5)	15.5 (0.8)
LNC80-6	13.8 (0.2)	23.0 (0.4)	25.3 (1.0)	28.2 (0.3)
LNC60-6	14.3 (0.2)	23.6 (0.4)	30.5 (0.4)	32.4 (0.3)
NC100-10	0.9 (0.0)	2.7 (0.3)	6.9 (1.0)	7.5 (1.0)
NC80-10	0.7 (0.0)	9.0 (0.6)	20.7 (0.9)	21.0 (0.8)
NC60-10	2.1 (0.1)	27.4 (0.5)	42.4 (0.5)	43.5 (4.0)
LNC100-10	14.2 (0.6)	22.3 (0.8)	28.1 (1.0)	30.8 (1.7)
LNC80-10	15.7 (0.3)	24.3 (0.8)	26.9 (2.3)	31.8 (2.9)
LNC60-10	20.4 (0.4)	32.5 (1.1)	44.4 (1.3)	44.5 (0.2)

Activation with only NC, for both 6% and 10% activator dosages, shows the lowest strength for both the early age and the long term period of curing, in comparison to other mixes (Fig. 2 and 3), which

confirms the results in (Kovtun et al., 2015). The NC100-6 mixes exhibited hardly any strength throughout the entire curing time. The compressive strength reached only 3.1 MPa at 28 days for this mix (Fig. 2). Increase in activator dosage, from 6% to 10%, did not cause any change for 3 days, while an increase was seen for further curing ages (Figs. 2 and 3). Recently, the slow activation mechanism of NC-activated slag was explained in detail by Bernal et al. (2014). It was reported that the activation occurred in three steps starting with the dissolution of the ions such as Ca, Si and Al in GGBS and the formation of gaylussite ($\text{Na}_2\text{Ca}(\text{CO}_3)_2 \cdot 5\text{H}_2\text{O}$), and zeolite NaA ($\text{Na}_{12}\text{Al}_{12}\text{Si}_{12}\text{O}_{48} \cdot 18\text{H}_2\text{O}$) in the first 24 hours. Then, the reaction might continue with an extended induction period of 4–6 days with the conversion of gaylussite to CaCO_3 and the formation of hydrotalcite ($\text{Mg}_6\text{Al}_2\text{CO}_3(\text{OH})_{16} \cdot 4\text{H}_2\text{O}$). Finally, the precipitation of C-(A)-S-H gel started. Thus, the reaction mechanism in NC activated slag leads to formation of relatively weaker phases in the initial stages of curing. Consequently, the NC samples fail to develop measurable compressive strengths during the period.

Most of the mixes, almost reach their ultimate strength at 28 days. Only slight improvement was observed at 90 days (Figs. 2 and 3). It can be seen that, there is an increase in the strength-giving phase formation such as hydrotalcite and C-(A)-S-H, from 3 to 7 days, especially for NC-60 mixes, with a related strength increment. Thereafter, the compressive strength increased gradually up to 44 MPa with extended curing.

Substitution of NC with NH did not cause any considerable change in compressive strength at the early age (3 days) for both activator dosages. At further ages, for NC-6 mixes, only a slight increase was seen with 20% NH replacement, while with 40% NH replacement compressive strength increased substantially for both activator dosages (NC60-6 and NC60-10). Thus, it can be deduced that NH replacement was effective at the further ages of the reaction. Kovtun et al. (2015) studied the acceleration of NC-activated GGBS by replacing NC with NH at 20% and 50% by weight. They found that replacing NC with NH at low dosage does not increase the compressive strength at 3 days. On the other hand, for both NH replacement dosage compressive strength reaches higher values than only NC-activated mixes at 28 days.

Compressive strength increased notably when the NC+NH activator dosage was raised to 10% (Fig. 3). Among the mixes without CH, maximum compressive strength was achieved with NC60-10 mixes

which reached 2.1 MPa and 43.5 MPa at 3 and 28 days, respectively. Despite the fact that very low early age strength was observed, this mixture exhibited satisfactory compressive strength over time. According to Li and Sun (2000) the compressive strength of 10% NC activated slag mortar was found as 0 MPa at 3 days and increased up to 60 MPa at 28 days substantiating the combined effect of NC and NH being more distinct at later stages. The low early age compressive strength of NC mixes was due to the lower initial OH^- and higher CO_3^{2-} concentration. With the replacement of NH, initial OH^- concentration and the pH value increase which facilitates the dissolution of the slag and improves the degree of hydration and the strength (Abdalqader et al., 2016; Bernal et al., 2014). On the other hand, lower amount of CO_3^{2-} exists in the medium when NC replaced by NH. The dissolution of reactive species such as SiO_2 , Al_2O_3 increases and higher amount of Ca^{2+} exists in the medium at higher pH levels. The increased dissolution and reduced CO_3^{2-} concentration provide more calcium to participate in C-A-S-H formation which leads to increase in the compressive strength.

With the inclusion of CH, especially at the early age, significant increase in compressive strength was seen for all mixes irrespective of the NH usage and/or activator concentration (Figs. 2 and 3). This increase was more distinct for LNC80 mixes. For LNC80-6 and LNC80-10 mixes, 3 d compressive strength values were 27 and 22 times higher than those of NC80-6 and NC80-10 mixes, respectively. Only one exception is that NC60-10 mixes reached almost the same strength value with LNC60-10 mixes at 28 and 90 days. These mixes had the maximum compressive strength value with approximately 44 MPa at 28 days among all mixes. Jeong et al. (2015) studied activation of GGBS with using mainly CH and substituting or adding four different accelerators such as NH, NC, Na_2SO_4 and $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$. They investigated the compressive strength development on paste mixes and concluded that adding NC into the CH activated mixes gives higher values compared to replacing CH with NC. It is obvious that CH plays a significant role in increasing the early and later age compressive strength. The enhanced early alkalinity due to NaOH generation from $\text{Ca}(\text{OH})_2$ and Na_2CO_3 (Eq. 1) accelerates slag dissolution (Yuan et al., 2017). However, at later ages CH replacement is not as effective for higher activator dosages and NH incorporation.

The increase in compressive strength with CH can be attributed to a few main reasons: Firstly, dissolved Ca^{2+} ions from CH consume the CO_3^{2-} from NC and form calcite and gaylussite. Removing CO_3^{2-} ions from pore solution increases the pH thereby facilitating increased ionic dissolution from slag. These dissolved ions (Ca^{2+} , Si^{4+} and Al^{3+}) lead to increased concentration of reaction products. In addition, CH introduces higher amount of Ca^{2+} required for C-(A)-S-H formation and eventually accelerates the reaction of NC-activated slag. Another reason for the high early age strength might be the filling effect of the CaCO_3 leading to reduced porosity (Wang et al., 2018).

To understand the strength development mechanism in detail, it is necessary to investigate the time-dependent microstructure properties of the NC-activated mixes, including NC and NH combinations and also CH replacement. The following sections are thus focused on the characterization of various aspects of NC-activated GGBS and the effects of the accelerators such as NH and CH.

3.2 XRD for Phase-Identification

Figs. 4-6 show the XRD patterns of the selected NC/NC+NH-activated slag pastes at different curing ages such as 3, 7 and 28 days. The selected mixes are NC100-6, NC60-6, NC60-10, LNC100-6 and LNC60-10 which represents the effect of combination of NC with NH and replacement of CH with GGBS. It can be seen from Figs. 4-6, the main reaction products are C-(A)-S-H, hydrotalcite ($\text{Mg}_6\text{Al}_2\text{CO}_3(\text{OH})_{16}\cdot 4\text{H}_2\text{O}$), calcite (CaCO_3) and gaylussite ($\text{Na}_2\text{Ca}(\text{CO}_3)_2\cdot 5\text{H}_2\text{O}$), along with portlandite ($\text{Ca}(\text{OH})_2$) and dolomite ($\text{MgCa}(\text{CO}_3)_2$).

Fig. 4. XRD patterns of selected mixes for 3 days. C: calcite; CSH: calcium alumino silicate hydrate; D: dolomite; G: gaylussite; Ht: hydrotalcite; P: portlandite

Fig. 5. XRD patterns of selected mixes for 7 days

Fig. 6. XRD patterns of selected mixes for 28 days

The main binding phase C-(A)-S-H reflected at approximately 30° (2θ) (Rashad et al., 2016; Lopez and Garcia, 2016) was found in all specimens throughout the all XRD measurements. This characteristic peak overlapped with the strong peak for calcite at the same position (Kovtun et al., 2015; Puertas and Carrasco, 2014). In the previous studies, the reason of the overlapping was explained with the semi-amorphous nature of the C-S-H and mentioned that this peak appear with the background hump in the 2θ range of 25° – 35° (Gu et al., 2014; Ben Haha et al., 2011, Garcia et al., 2003; Ben Haha et al., 2012). Calcite was also identified at approximately 43° (2θ) and 47° (2θ) for all of the mixes at all curing days, which was due to the cation exchange reaction between slag and NC (Ca^{2+} from slag and CO_3^{2-} from NC), as well as atmospheric carbonation. In NC100 mixes, due to the high concentration of CO_3^{2-} the calcite precipitation occurs at very early stage. Although the addition of CH was expected to yield enhanced peaks, this assumption was not proved by XRD results (Figs. 4-6). The contradiction can be resolved by considering the formation of gaylussite instead of calcite. The more intensive reflections of gaylussite and hydrotalcite are presented by the incorporation of CH at the curing age of 3 d (Fig. 4).

Gaylussite was identified at three different diffraction angles; approximately at 28° , 32° and 36° (2θ) but only the gaylussite peak at 32° (2θ) was seen for all mixes and all curing ages. The number of gaylussite peaks decreased in mixes containing CH, especially after 7 days (Figs. 4-6) since gaylussite converts to more stable products such as calcite and hydrotalcite (Bernal et al., 2014). According to

Kovtun et al. (2015), the intensity of gaylussite decreased in the pastes produced using with accelerators such as NH and cement.

The characteristic peaks of hydrotalcite was identified at approximately 11.5° , 23° , 34° and 39° (2θ), the same diffraction peaks were reported in the previous studies (Bernal et al., 2014; Rashad et al., 2016; Puertas and Carrasco, 2014; Gu et al., 2014). In the CH blended mixes, the intensities of hydrotalcite was higher than the NC mixes (Figs. 4-6). At the early ages, hydrotalcite was observed only with poor reflections but as a result of the ongoing reactions its intensity increased. A special example; the peak at 11.5° (2θ) was not observed for any of the mixes at 3 days of the reaction but it formed at 7 days and finally its intensity increased for all mixes at 28 days. This increased intensity of hydrotalcite peak might be one of the reason for the strength development from 3 to 7 days.

Diffraction peaks for portlandite ($\sim 18^\circ$ 2θ) was only identified in LNC mixes. In LNC60-10 mixes, intensity of the peak was decreased from 3 to 7 days and disappeared at 28 days (Figs. 4-6). On the other hand, in the LNC100-6 mixes portlandite continued to appear at all reaction days, only its intensity decreased. Jeon et al. (2015) reported that CH turns into the calcite and CSH at further ages of the reaction in alkali activated systems. The rapid consumption of CH in LNC60-10 mixes could be due to the formation of CaCO_3 from the initial reaction of CH with NC and the facilitated C-(A)-S-H formation from the reaction between the CH and the amorphous phase of the slag (Shi and Day, 2000). This assumption can be supported by the Figs. 2-6. It can be seen that LNC100-6 mixes did not gain a substantial compressive strength increase from 3 to 28 days, while LNC60-10 mixes presented a significant increase during the same period. A longer curing duration, 28 d (Fig. 6), promotes the development of a more crystalline structure compared with the 3 and 7-day samples (Figs. 4 and 5). Increasing the curing time did not generate new phases, but it increased the crystallinity of C-(A)-S-H type structure, especially in the series of NC60-10 and LNC60-10, visible by sharpening of the main peak at around $\sim 29.5^\circ$ (2θ) (Bernal et al., 2013).

The XRD test results may explain the compressive strength development of NC-activated slag mortars. At the early age of the reaction (3 days), NH replacement and activator dosage did not change compressive strength significantly. However, for 7 days and later on, substantial increase was seen. This increment can be explained by the increase in the number and intensity of the peaks such as hydrotalcite

and C-(A)-S-H. This trend was observed for CH blended mixes from 3 days. In LNC mixes, the peaks at higher degrees seen in XRD patterns were more intense than NC mixes which resulted in decrease in interlayer distance explaining the gain in compressive strength (Sikander et al., 2017). At 28 days of curing, the intensities of the peaks of main reaction products, calcite/C-(A)-S-H and especially hydrotalcite diffracted at 11.5° and 23° (2θ), show increased values for all mixes signifying a higher concentration of reaction products thus pointing to higher compressive strength. It is seen from Fig. 2 and 3 that although NC60-10 exhibited lower compressive strength values than LNC60-10 at 3 days, both mixes were presented almost the same values at 28 days. Thus, different XRD diffractograms for 3 days and similar XRD diffractograms for 28 days can be expected for these mixes. At 3 days, while high amount of gaylussite and C-(A)-S-H and moderate amount of hydrotalcite exist for NC60-10; substantially high amount of hydrotalcite and less amount of gaylussite exist for LNC60-10 mixes. Thus, it can be deduced that hydrotalcite, diffracts at approximately 39° (2θ), might have more contribution for compressive strength, compared to other phases. On the other hand, both mixes present the same peaks at the same diffraction angles with similar intensities at 28 days.

3.3 Evaluation of Chemical Phases of the Binder Using FTIR

The FTIR spectra of the anhydrous slag used in this experimental study and selected paste samples at 3, 7 and 28 day-age are presented in Figs. 7-10. Although the analysis was conducted between 400 and 4000 cm^{-1} , only the wavelength between 600 and 1800 cm^{-1} were presented as there is no significant feature beyond this wavelength.

For the anhydrous slag (Fig. 7), the distinct intensity band centered at around 901 cm^{-1} was related to the asymmetric stretching vibration mode of Si-O-T bonds, where T represents Si or Al, and the band position depended on the glass structures of the raw material and another small intense band at approximate 710 cm^{-1} represents the functional group of AlO_2 (Abdalqader et al., 2016; Ben Haha et al., 2011). Moreover, the band at 1423 cm^{-1} identified in the GGBS is attributed to the symmetric stretching mode of the O-C-O bonds of CO_3^{2-} groups regarded as weathering of the GGBS (Abdalqader et al., 2016).

Fig. 7. FTIR spectra of anhydrous slag

NC100-6, NC60-6, NC60-10, LNC100-6 and LNC60-10 mixes were chosen for further analysis, which represents the reaction process and NH replacement and CH effect on NC activated slag. All the paste mixes exhibited similar bands, suggesting similar nature of the hydration products, mainly containing CO_3^{2-} and C-(A)-S-H gel, irrespective of the activator dosage, NH replacement or inclusion of CH. Major bands were identified at approximately 1430, 945, 870, 665 cm^{-1} for all mixes (Figs. 8-10). The band at about 665-712 cm^{-1} is associated with the stretching vibrations of Al-O bonds in the AlO_4 groups which could probably be related to the formation of hydrotalcite and C-(A)-S-H gel (Puertas and Carrasco, 2014).

Fig. 8. FTIR spectra of selected mixes at 3 days

Fig. 9. FTIR spectra of selected mixes at 7 days

Fig. 10. FTIR spectra of selected mixes at 28 days

The bands at around 1420 and 870 cm^{-1} suggests the presence of CO_3^{2-} , which trace of the presence of gaylussite, calcite, hydrotalcite or dolomite (Puertas and Carrasco, 2014), as detected by both XRD (Figs. 4-6) and TGA (Figs. 17-18). Generally, the vibration of CO_3^{2-} groups is indicated to the activator used (NC). One of the reasons of the reflection at this range may also be due to the carbonation or weathering of the samples.

The main binder gel band, at approximately 945 cm^{-1} , corresponds to the Si-O-T asymmetric stretching bands in the SiO_4 tetrahedra (Abdalqader et al., 2016). This finding is in accordance with the formation of C-(A)-S-H of the slag activation, determined by XRD and also confirms in (Garcia et al., 2003; Ben Haha et al., 2012).

In all mixes, the Si-O-T band shifted slightly towards higher wavenumbers at further ages (Figs. 8-10). This is possibly due to the ongoing activation of GGBS, which increases the formation of more polymerized gel (Puertas and Jimenez, 2003). Moreover, in the case of pastes accelerated with CH and with the combination of CH and NH, the Si-O-T band also shifts towards higher wave numbers of 939 cm^{-1} . This behavior again attributed to the higher degree of polymerization. Abdalqader et al. (2016) reported that replacement of slag with the fly ash causes slight shifting of Si-O-T band to a higher wavenumbers due to activation of fly ash. Since only slight shifting are seen on the Si-O-T band values of mixes, the gel structure of C-(A)-S-H represents only slight differences with the CH and/or NH replacement and also activator dosage.

The bands representing CO_3^{2-} ranged between approximately 1410 cm^{-1} and 1464 cm^{-1} . In the presence of NH and/or CH replacement, this band observed at higher wavenumbers, which representing the stronger bonds (Figs. 8-10) and might be indicating higher compressive strength.

3.4 Microstructural Characterization

The selected SEM images of the mixes cured for 28 days are shown in Figs. 11-13, and the EDS quantifications of the atomic ratios are given in Table 4.

Higher amount of the micro cracks which negatively affect the strength are seen on the matrix phase of NC mixes compared to LNC mixes (Fig. 11).

Fig. 11. Comparison of NC and LNC mixes in terms of crack patterns

Micro pores and sharp edges particles were mostly seen in NC100-10 mixes compared to NC60-10 (Fig. 12). Similar findings were reported, in which the matrices looked rather dense with increasing NH concentration (Mustafa et al., 2011). This is attributed to the formation of higher amounts of C-(A)-S-H gels caused by the increase in pH of pore solution (Jim et al., 2015; Gebregziabiher et al., 2015).

Fig. 12. Comparison of (a) NC100-10 and (b) NC60-10 mixes

Deposition of high amount of calcium carbonates were observed with various sizes (Fig. 13) in all mixes, especially for LNC ones. They are seen on the surface of the slag particles and C-(A)-S-H gel. The filling effect of the calcite may also contributed to the denser microstructure of matrix.

Fig. 13. Depositions of calcium carbonates (a) LNC80-6 and (b) LNC60-6 mixes

Combined with SEM, X-ray microanalysis (EDS) was performed to determine the differences in the elemental composition of the hydrated phases. Difference of various atomic ratios between NC and LNC mixes changes with the activator dosage and NH replacements and designate different hydration products. The results showed that the major elements were Ca, Na and Si and the minor elements were Al and Mg (Table 4). The major elements were mainly attributed to the formation of C-(A)-S-H and also gaylussite and the minor elements were assigned to the hydrotalcite, which was proved by the XRD and FTIR results.

The Ca/Si ratios of all pastes were in the range of 0.80-1.38. LNC mixes has higher Ca/Si ratios compared to NC ones, which should be caused by the inclusion of CH. The increase of Ca/Si ratio is also seen with the replacement of NH, and that is attributed to the increased Ca^{2+} ions dissolved from GGBS and hence higher amount of Ca^{2+} ion occupied in C-(A)-S-H gel. The maximum Ca/Si ratios were 0.96 and 1.38 in 6% and 10% mixes, respectively. The higher activator dosage resulted in the higher Ca/Si ratio, which could lead to increased Ca^{2+} ion dissolution from the slag in the higher alkaline environment. Higher Ca/Si is indicative of the formation of denser, calcium rich products with increased activator (Jin et al., 2015) and CH inclusion, which was consistent with the compressive strength development. The Ca/Si ratio of C-S-H gel depends on the type of activator, curing condition and chemical composition of slag and ranges from 0.6 to 2.3 (Ben Haha et al., 2011; Richardson et al., 2010; Wang and Scrivener, 2003; Lloyd et al., 2010). The Al/Si ratio did not change significantly with changes in activator concentration and NH and CH replacement, for all mixes the ratio ranged between 0.29-0.36. Lloyd et al. (2010) announced that the more Al incorporated in the gel structure with increasing the activator concentration. Wang and Scrivener (2003) reported that actual Al/Si ratios were in the range between 0.16 and 0.22 depending on activator type and age.

The Ca/Na ratios of all pastes were in the range of 2.00-4.75. The values were higher in LNC mixes compared to NC ones and were lower for 10% activator concentration. These results are expected to be considering LNC mixes which contains CH and higher amount of Ca^{2+} and mixes contain 10% activator dosage which provides higher amount of Na^{2+} into the mixes. Na is defined in (Al Bakri et al., 2011; Jin et al., 2015; Gebregziabihier et al., 2015) as charge-balancer for negatively charged Al-O-Si monomers

in the formation of C/N-A-S-H. Magnesium and part of aluminum can be referred to the hydrotalcite which was found in all mixes by XRD analysis.

Table 4. Average atomic ratios

Mixes	Ca/Si	Ca/Na	Al/Si
NC100-6	0.86	3.00	0.32
NC80-6	0.93	3.13	0.33
NC60-6	0.93	2.78	0.33
LNC100-6	1.08	4.00	0.31
LNC80-6	1.11	4.75	0.32
LNC60-6	1.18	4.13	0.36
NC100-10	0.80	2.00	0.32
NC80-10	0.80	2.00	0.32
NC60-10	0.96	2.54	0.31
LNC100-10	0.96	2.30	0.29
LNC80-10	1.29	3.44	0.29
LNC60-10	1.38	2.78	0.29

3.5 Thermogravimetric analysis

Figs. 14 and 15 depict the representative TGA and DTG results for selected mixes at the curing age of 28 days. All mixes possess similar patterns which shows one main and three small peaks. Mass loss below 200°C was caused by the evaporation of free water and partly by the dehydration of C-(A)-S-H gel (Gebregziabiher et al., 2015; Yusuf et al., 2014) and gaylussite (Johnson and Robb, 1973). The peak between 250°C and 400°C attributed to the decomposition of hydrotalcite (Gu et al., 2014; Richardson et al., 2010), which agrees well with the XRD results. The decomposition of hydrotalcite is generally categorized in three stages (Rashad et al., 2016; Wang and Scrivener, 2003; Lloyd et al., 2010): firstly evaporation of interlayer water under 200°C; secondly, decomposition of structural hydroxyl groups around 350°C and finally decomposition of interlayer carbonate ions around 440°C. The second decomposition peak around 300°C (Fig.15) can be attributed to the dehydroxylation of brucite-like layers of hydrotalcite and the small shoulder at 400-560°C could be the decarbonation of hydrotalcite. LNC-10 mixes exhibited an additional tiny peak for Ca(OH)₂ decomposition between 400°C and 450°C

which agrees well with the results of Kim et al. (2013). This peak was more distinct in LNC60-10 among all LNC-10 mixes (Fig. 15).

Fig. 14. TGA curves of selected mixes

Fig. 15. DTG curves of selected mixes

The mass loss occurred at the temperatures between 400°C and 600°C can be defined as the decomposition of poorly crystalline CaCO_3 (Temuujin et al., 2009; Puertas et al., 2002) or magnesium silicate hydrates (M-S-H) (Abdalqader et al., 2016). Since M-S-H was not observed from the XRD and

the existence of CaCO_3 was proved by the both XRD and SEM, it can be deduced that the mass loss at the temperature range of 400-600°C caused by the decomposition of poorly crystalline CaCO_3 . The decomposition of the carbonate containing phases occurred between 600-800°C, including calcite (Dweck et al., 2002), gaylussite (Johnson and Robb, 1973) and hydrotalcite (Parashar et al., 2012). Gaylussite thermal behavior consists of three main features: (1) Dehydration up to 250°C; (2) Crystal transformations between 250°C and 500°C; and (3) Melting and carbonate decomposition from 500°C to a maximum of 1050°C (Johnson and Robb, 1973).

The mass loss of mixtures at different temperature ranges are given in Table 4, representing different reaction products as discussed above. In this study, the mass loss is divided into three groups and denoted as Δm_1 , Δm_2 and Δm_3 with the ranges of 50-200°C, 250-400°C and 400-800°C where C-(A)-S-H; hydrotalcite; CH and CO_3^{2-} containing phases decomposes, respectively. The maximum mass loss occurred between 50°C and 200°C (Δm_1) for all mixes and mass change between these temperatures ranged between 9.9% and 23.9%. Although the decomposition of other phases (dehydration of gaylussite and loss of interlayer water of hydrotalcite) overlaps with C-(A)-S-H within this range, their influence is supposed to be insignificant due to their relatively small amounts. Table 4 shows that the weight loss of C-(A)-S-H ranged between 9.9% and 14.9% for NC-6 and LNC-6 mixes and it gradually increased to 16.8% and 23.9% for NC-10 and LNC-10 mixes. Δm_1 increased with the increase in activator dosage, NH replacement and CH inclusion. Higher mass losses for this temperature range indicates a higher degree of reaction and this is consistent with the compressive strength results. This is expected that more slag particles are dissolved, consequently leads to a higher degree of the strength-giving phase formation. The mass loss between 250°C and 400°C (Δm_2) slightly increased with the NH replacement, CH inclusion and higher activator dosage (Table 5). It was also observed that the value of Δm_3 decreased with the NH replacement and increased with the higher activator dosage and CH inclusion, which means that the CO_3 containing phases are more at these mixes.

Table 5. Weight losses of mixtures (%)

Mixes	Δm_1	Δm_2	Δm_3
NC100-6	9.9	1.6	5.2
NC80-6	12.0	1.9	4.6
NC60-6	13.7	1.9	2.6
LNC100-6	14.3	1.9	5.0
LNC80-6	14.9	1.9	4.0
LNC60-6	13.7	1.9	2.6
NC100-10	16.8	2.1	5.8
NC80-10	23.9	2.6	5.0
NC60-10	23.1	2.3	3.4
LNC100-10	17.6	2.2	5.9
LNC80-10	23.1	2.6	5.4
LNC60-10	22.4	3.0	4.9

4. CONCLUSIONS

This experimental study has been performed to investigate the mechanisms that govern the strength development in sodium carbonate activated slags as a function of the nature of the alkaline activator. The purpose was to explore the strength gain mechanism of sodium carbonate activated slag mixes at room temperature curing. To enhance both early and ultimate compressive strength, different accelerators were used. Strength development mechanism has been clarified with the time dependent microstructure analysis. The study enables development of sustainable NC activated pozzolanic binders as feasible alternatives to OPC based systems.

The following conclusions can be drawn from this study:

- Activation of slag with only NC, exhibited hardly any strength (~7 MPa) cured at room temperature up to 90 days.
- Addition of NH as a replacement of NC enhances the rate of dissolution of slag leading to a faster strength development. Although, replacement of NC with NH did not cause any considerable change in compressive strength at the early age for both activator dosages, significant increase was seen at further ages.

- The presence of CH in the binder as a replacement of the slag led to a substantial increase in compressive strength irrespective of the NH usage and/or activator concentration. Enhanced strength development mainly caused by removing the carbonate from the medium and providing additional Ca^{2+} resources.
- Type and amount of accelerators and also activator dosage plays an important role in the strength development.
- Formation of the strength giving phases such as hydrotalcite and C-(A)-S-H plays an important role in strength development of NC activated slags.
- The detailed characterization of the microstructural development with the progress of hydration in mixes serve to an enhanced understanding of the strength development as a consequences of chemical changes.

A future perspective to the investigation can be in the form of a detailed study of the chemical reactions aided by molecular models that can effectively capture the hydration process of such alkali activated mixtures.

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