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Autogenous and drying shrinkage of sodium carbonate activated slag altered by limestone powder incorporation

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HIGHLIGHTS

- Effect of Limestone powder (LP) on autogenous and dryings shrinkage is explored.
- Pore structure modification caused by LP addition is studied.
- Correlations among LP content, reaction kinetics and autogenous shrinkage are observed.
- Drying shrinkage increases with the increase of LP addition.
- Decomposition of gaylussite and natron is partly responsible for the high drying shrinkage.

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ABSTRACT

This paper aims to study the shrinkage mechanism of sodium carbonate activated slag containing limestone powder (LP). The workability, pore structure, reaction kinetics and strength development were characterized. The results show that the autogenous shrinkage increases when the dosage of LP is low (≤30 vol%) primarily because of the intensified and accelerated reaction, but decreases when further increasing the LP content due to the dilution effect caused by the reduced binder content. A high amount of limestone powder incorporation slightly increases the pore volume in the range of mesopore and macropore, while its effect on the autogenous shrinkage is not prominent. However, the drying shrinkage of the samples is significantly increased with the increase of LP, caused by the more free water as indicated by the mass loss of the samples. In addition, the decomposition of gaylussite and natron also contributes to the enlarged drying shrinkage.

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1. Introduction

Alkali activated slags (AAS) have attracted worldwide attentions, attributed to their room temperature curing, good material properties and environmental benefits [1]. The influence of alkali activators, such as alkali nature and concentration [2–4], has been extensively studied, and the effect of supplementary materials (especially fly ash and metakaolin) on the properties of waterglass/NaOH activated slag has also been often reported [5,6]. However, only a few investigations have studied the effect of supplementary materials on the sodium carbonate activation. The effect of limestone powder on the mechanical properties [7] and its potential chemical involvement [8] have been reported recently, however, its influence on the durability, e.g. shrinkage, of sodium carbonate activated slag was rarely studied.

Limestone powder (LP) has been widely used in cementitious materials because of its economic benefits and good performances [9–13]. The physical and chemical effects of limestone powder on the hydration process of cement have been intensively investigated, e.g. as nuclei sites for hydration and/or involved in the chemical reaction with C_3S [14,15] and C_3A [15–17]. Moreover, the role of LP in the durability of Portland cement based materials was also widely studied. Most researches [18–20] found that the incorporation of LP does not significantly alter or sometimes reduces the drying and autogenous shrinkage, for instance Benachour et al. [19] reported that an addition up to 35% did not enlarge the drying shrinkage. Bouasker et al. [21] observed that the presence of limestone filler causes an acceleration of the chemical shrinkage since the very first hours of hydration, but it does not significantly modify the relationship between

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chemical shrinkage and hydration degree of the cementitious materials.

The shrinkage of alkali activated materials was found to be higher than Portland cement (PC) based materials [4,22]. Many factors can affect the shrinkage performance, e.g. alkali nature (especially activated by waterglass) [2], properties of solid precursor (e.g. slag) [23], curing condition (e.g. relative humidity) [24] and reaction products. Collins [25,26] reported that the shrinkage of alkali activated slag concrete is about two times larger than ordinary Portland cement concrete. Atis [2] found that Na_2CO_3 activated slag has a similar or lower shrinkage than PC mortar, while slag activated by waterglass generally shows a shrinkage 3–6 times higher than that of PC mortar. Neto et al. [3] studied the drying and autogenous shrinkage of waste glass activated slag cement and found that the amount of silicate contained in the activator plays an important role. It should be noted that the pore structure of the samples also significantly affects the shrinkage of the matrix. Colins and Sanjayan [27] found that AAS pastes have a much higher proportion of mesopore than OPC pastes, which may be the reason for the higher drying shrinkage of AAS pastes. Li et al. [28] also observed a linear relationship between the autogenous shrinkage and volumetric percentage of pores in the range of 5–50 nm.

In recent years, sodium carbonate activation has been research interest because of its low price and environmental benefits. Different from alkali activation using other activators [29], sodium carbonate activation is predominately controlled by the CO_3^{2-} concentration in the pore solution [30]. The main reaction products are calcium silicates hydrates gel with a small amount of Al incorporated in the structure (C-(A)-S-H), calcium carbonate, gaylussite ($Na_2Ca(CO_3)_2{\cdot}10H_2O$), and hydrotalcite ($Mg_6Al_2CO_3(OH)_{16}{\cdot}4H_2O$, depending on the MgO content in the raw materials). According to Moseson et al. [31], the CO_2 emission and energy consumption can be reduced up to 97% while a compressive strength of 65 MPa at 28 d can be achieved by a proper mix design. Rakhimova et al. [7] concluded that the incorporation of limestone powder improves the physical structure of samples activated by sodium carbonate, while Myers [8] found that the conversion of gaylussite is chemically affected by LP. It has been reported that in low calcium-containing alkali activated metakaolin system, the presence of LP can provide Ca^{2+} via the hydrolysis, leading to the formation of layered calcium carboaluminates [32]. While in sodium carbonate activated slag system, a new phase natron was formed when a high amount of LP was incorporated [8]. Both gaylussite and natron are not stable phases that will decompose with time, which can potentially affect the durability of the matrix. Moreover, methods to reduce the shrinkage of alkali activated materials have been studied by for instance applying shrinkage reducing agent [33,34]. Jin et al. [34] investigated the shrinkage of Na_2CO_3 activated slag and successfully reduced the drying shrinkage by dosing reactive MgO powder. Nevertheless, to the best of the authors' knowledge, the influence of limestone powder on the shrinkage of samples produced by sodium carbonate activation has not been reported yet.

This research aims to study the effect of limestone powder on the drying and autogenous shrinkage of sodium carbonate activated slag. The reaction kinetics and strength development of the specimens were characterized. The pore structure of the selected mixtures was characterized employing the Brunauer, Emmett and Teller (BET) method. The autogenous shrinkage and drying shrinkage were measured up to 56 days of curing. A correlation between the autogenous shrinkage and reaction kinetics was derived. Furthermore, the effect of pore structure modification by limestone powder on shrinkage was discussed.

2. Materials and experiments

2.1. Materials

The raw materials applied were ground granulated blast furnace slags (GGBS) (provided by ENCI, the Netherlands) and limestone powder (LP). Table 1 and Fig. 1 show the chemical compositions and particle size distributions of the GGBS and limestone powder measured by an X-ray Fluorescence spectrometer and a particle size analyser applying laser diffraction (Mastersizer 2000), respectively. The used GGBS has a basicity coefficient (K_b, $(CaO + MgO)/(SiO_2 + Al_2O_3)$) of 1.5. The designed activators were prepared by dissolving sodium carbonate (powder form, analytical grade) in water and then cooled down to room temperature (20 ± 1 °C) prior to application.

2.2. Experimental methods

The reference paste sample was sodium carbonate activated slag with the dosage of sodium carbonate 4% (equivalent Na_2O by mass of slag) and water to solid ratio 0.4 (solids include slag and sodium carbonate powder), while the other mixtures were designed with different contents of limestone powder (up to 50 vol%). The name of the mixtures was designated as the replacement level of limestone powder, e.g. Mixture LP 10% and Mixture LP 50% mean that 10% and 50% volume of slag were replaced by LP, respectively.

2.2.1. Flowability and strength

A Hägermann cone (top diameter = 70 mm, base diameter = 100 mm, height = 60 mm and volume = 344 ml) was used to measure the spread flow of the pastes, according to the suggestion of EN 196–1. The flowability of the pastes is interpreted by the mean of two perpendicular diameters of the measured spread. Afterward the flowability measurement, the fresh pastes were cast into plastic moulds (40 × 40 × 160 mm^3). Plastic foil was applied to cover the specimens to prevent the moisture loss and then the samples were stored in a chamber at room temperature (20 ± 1 °C) with a stable relative humidity of >99% till the ages of testing. The strength of the samples were determined according to EN 196–1.

2.2.2. Reaction kinetics

An isothermal conduction calorimeter (TAM AIR Calorimeter, set at 20 °C) was used to measure the heat evolution of the samples. The fresh pastes were prepared at room temperature and mixed using a vibrating device to ensure the homogeneity. The prepared pastes were filled into an ampoule which is then loaded to the calorimeter, which means that the sampling time (4–6 min) was not recorded. Moreover, due to the interruption caused by sample preparation, the recorded data in the initial 45 min might not be reliable.

2.2.3. Shrinkage

Three paste samples per mixture were prepared for the drying shrinkage with the dimension of 40 × 40 × 160 mm^3. A climate chamber with the controlled temperature and relative humidity was used for the drying shrinkage measurement. Different from previous investigations, a slightly higher relative humidity of 65% was selected for the experiments based on DIN 52450: 1985. A digital length comparator (±0.001 mm) was adopted to measure the linear dimension variation of the samples along the longitudinal axis. The autogenous shrinkage was determined with standardized samples using corrugated polyethylene tubes (∅ 29 mm × 420 mm) as suggested by ASTM C 1698-09 and all paste samples were stored in a climate room (20 ± 1 °C and RH 50%).

Table 1
Chemical composition of the GGBS determined by XRF.

Chemical composition	GGBS (%)	Limestone powder (%)
SiO_2	30.48	0.84
CaO	40.05	53.96
Al_2O_3	12.92	0.24
MgO	9.49	1.01
Fe_2O_3	0.57	0.32
SO_3	3.48	–
K_2O	0.39	0.34
TiO_2	1.48	–
Cl	0.02	–
L.O.I		43.01
Density (kg/m^3)	2930	2710
BET surface area (m^2/g)	1.87	1.33

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