EFFECT OF FINE GRINDING ON THE PROPERTIES OF ALKALI ACTIVATED LADLE SLAG

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Introduction

Ladle slag (LS) is an industrial residue generated in steel-making processes. Air-cooled or slowly cooled ladle slags are made of fine particles due to the conversion of the thermally unstable β-C_2S to γ-C_2S during the cooling process[1]. Thus, the mineralogy of ladle slag is majorly crystalline with phases including calcium aluminate (mayenite) and dicalcium silicate. Ladle slag generation in Europe alone is estimated to be between 1.9-2.4 Mt yearly[2], and depending on the policies in different countries, it can be tagged as either waste or by-product. Previously, the valorisation of finely ground ladle slag in alkali activation was studied by the authors and promising results for ladle slag as a sole binder obtained[2]. Based on the hydration kinetics, it is apparent that slag made up of finely distributed particles reacts faster than coarse particles due to the high specific surface area of the former. Therefore, in this study the effect of particle size distribution on the alkali activated ladle slag properties was studied. The aim was to study the effect of grinding time on the properties of alkali activation ladle slag.

Materials and Methods

The as-received LS was milled with tumbling ball mill and the particle size distribution analysis was done by laser diffraction technique in isopropanol medium using the Fraunhofer diffraction model. Chemical composition was determined through XRF and shown in Table 1. Five samples of 1 kg each were milled at different times: 0, 10, 30, 60 and 120 minutes with the sample name represented as BM0, BM10, BM30, BM60 and BM120 respectively. The median particle sizes are shown in Table 2. Activating solution used comprises sodium silicate (Na-Sil) solution with a modulus of 3.5 mixed and pellets of potassium hydroxide (KOH) prepared one day before mixing with the binder.

Table 1. Chemical composition of the slag used in this study

CaO	Al_2O_3	SiO_2	MgO	Fe_2O_3	Others	LOI
46.3	28.3	8.6	7.4	5.0	4.4	-1.3

Standard sand according to EN 196-1 specifications was used as aggregates and the mix composition was done as reported in Table 2. Each sample was then prepared by mixing the binder with the alkali solution. The mortar sample was then casted in rectangular prisms and cured at 60°C for 24 hrs before they were unmoulded and kept

in a sealed bag until compressive testing. The samples for shrinkage test were kept at relative humidity of 50±5% at 22±2°C and length change was calculated at different curing times. Initial and final setting time was determined using automated Vicat needle apparatus conforming EN 196-3. Compressive strength was done at 64 days using an Instron testing machine with a loading rate of 2.4 kN/s. Duplicate samples of each mix were analysed for freeze thaw (F-T) resistance modified according to EN 14617-5. The mortars were subjected to 25 F-T cycles of freezing the sample in air at -20°C for 4 hours and then thawing in water at 20°C for at least 2 hours. The microstructural analysis was done using a Zeiss Ultra Plus microscope with an accelerating voltage of 15 keV engaged on polished surfaces which were carbon-coated.

Results and Discussion

Setting time

The milling time influences the setting time of the AALS samples (Table 2). It can be seen that both initial and final setting times are reduced with increased milling time ranging between 50 to 15 min and 80 to 30 min respectively. This is attributed to the increased surface area of the slags, which are readily available for dissolution and the increased degree of reaction. Thereby, finer slag particles will require less time to achieve set compared to coarser slag particles which showed to require more time to achieve set.

Table 2: Mix formula for the AALS mortars and correspondent slag median size (d_{50})

Sample name	BM0	BM10	BM30	BM60	BM120
LS (g)	300	300	300	300	300
Na-Sil (g)	245	245	245	245	245
KOH (g)	15	15	15	15	15
Sand (g)	560	560	560	560	560
d_{50} (µm)	33.84	24.27	23.55	20.80	14.19
Initial setting time (min)	50	35	30	20	15
Final setting time (min)	80	60	35	35	30

Mechanical and durability properties

In Figure 1, the mechanical strength of the frosted and non-frosted samples at 64 days are presented. BM60 seems to have the highest compressive strength with 48 MPa and BM120 with the lowest at 30 MPa. It should be noted that the compressive strength can be redefined with optimised parameters; water-to-binder ratio, increased silicate content using supplementary materials such as silica fume and diatomaceous earth[2]. In this analysis, no supplementary silica was added to the mix and water to binder ratio was fixed at 0.40. The frost resistance of the various specimens at 64 days age of the samples shows that all mortar samples having

considerable frost resistance with similar or even higher strength to control or non-frosted samples.

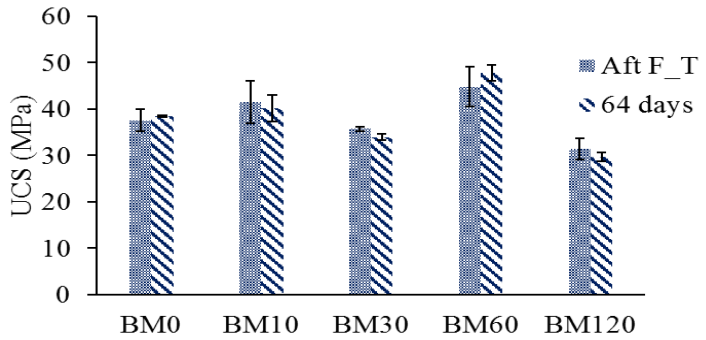

Figure 1: Compressive strength of the various mortars after freeze-thaw and without freeze thaw at the same age

In Figure 2, the drying shrinkage expressed as the length change of the mortars at 3, 7, 14, 21, 28, 45, 56 and 90 curing days is presented. The drying shrinkage of the AALS mortars was more pronounced at the early ages of the samples up to 21 days, after which existed nominal changes in length. Overall, BM0 exhibited the lowest drying shrinkage of the AALS samples. The smaller drying shrinkage of the sample is partly attributed to the coarsest particles of slag which do not wholly hydrate, thereafter becoming dense bodies covered by hydrated gel as can be seen in Figure 3a. These dense structure offers restraint similar to aggregates, which ensures bridging of crack propagation. Overall, the drying shrinkage of the AALS samples increases from 2 to 3 times that of OPC mortar which has a higher sand to cement ratio.

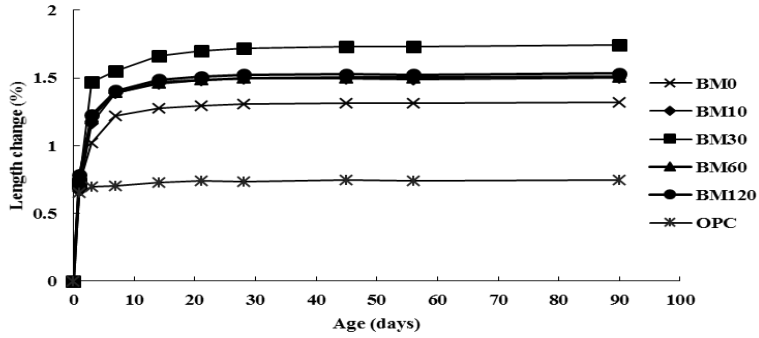

Figure 2: Drying shrinkage of AALS mortars for the activated slag

Microstructure

The effect of particle size distribution after different milling time on microstructure of activated LS is shown using SEM-Backscattered Electron (BSE) images in Figure 3. With BM0, the effect of the partly unreacted coarse slag particle can be observed forming dense bodies with the reacted slag particles (Figure 3a). This correlates with the discussion of the sample's drying shrinkage. The cracks seem to increase with reduction in particle size of the slag particles.

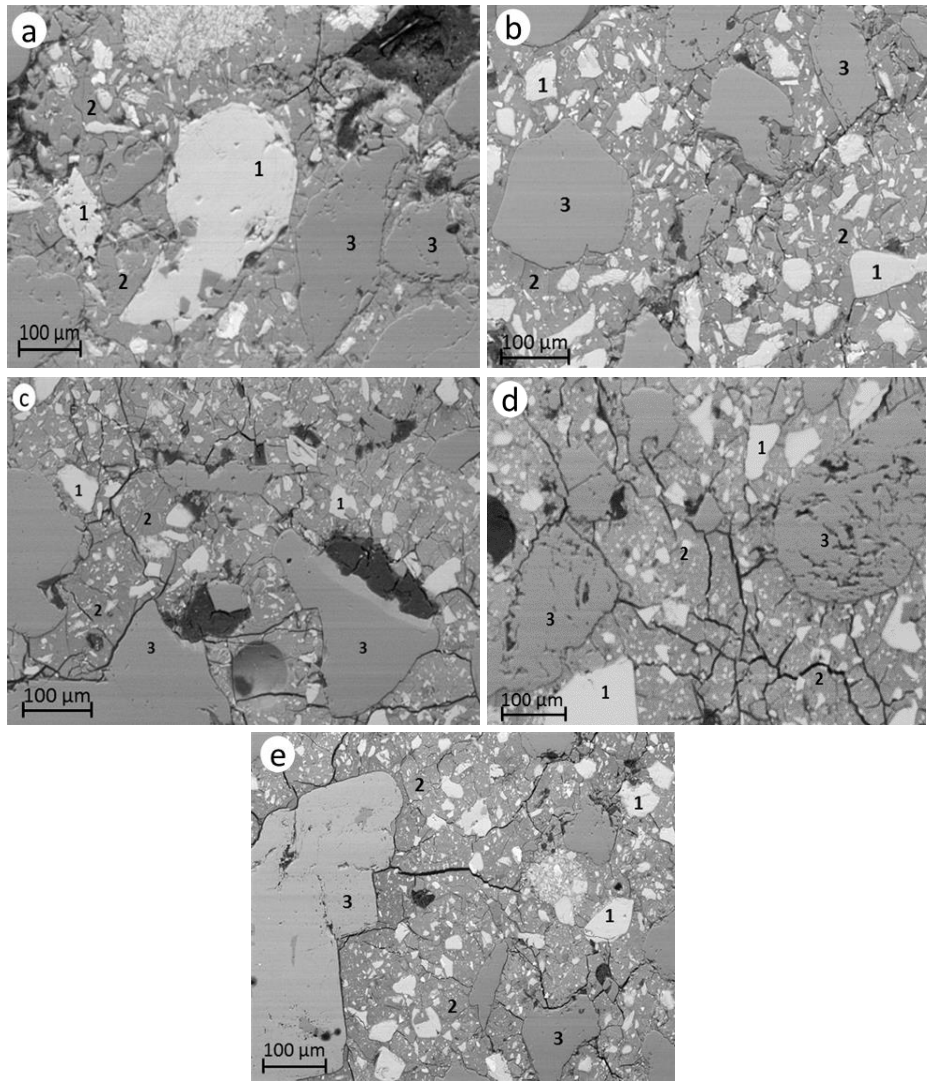

Figure 3: SEM-Backscattered images showing the microstructure of the AALS mortars (a) BM0 (b) BM10 (c) BM30 (d) BM60 (e) BM120; partially reacted LS = 1, reacted product =2 and sand aggregates =3

Conclusions

The effect of fine grinding of LS on its properties after alkali activation has been presented. The setting time, compressive strength and drying shrinkage of the AALS samples was observed to be dependent on the grinding time. The highest UCS recorded was 50 MPa for BM60. All AALS mortar showed excellent frost resistance after 25 cycles.

References

1. C. Shi, "Characteristics and cementitious properties of ladle slag fines from steel production", *Cem Concr Res*, **32** 459–462 (2002).
2. E. Adesanya, K. Ohenoja, P. Kinnunen and M. Illikainen, "Alkali Activation of Ladle Slag from Steel-Making Process", *J Sustain Metall*, 1–11 (2016).