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Colour, compressive strength and workability of mortars with an iron rich sewage sludge ash

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Abstract

This paper reports a study of the colour, compressive strength and workability of mortar when cement is partly replaced by sewage sludge ash (SSA). In the study, an iron rich SSA was dry milled into six different fractions. The results showed that the colour, compressive strength and workability parallel to one another gradually changed when the particle sizes of the SSA decreased. The milling of the SSA altered the performance of mortars to the extent that the compressive strength and workability were comparable to the performance of ordinary mortar. At the same time, the colour also changed from grey to a reddish colour. As the change in colour may be of importance for application, it is suggested to include colour as experimental parameter in future work.

Keywords: mortar, sewage sludge ash, colour, compressive strength, workability

1. Introduction

Negative environmental effects and overexploitation of available resources, due to a growing human population, is a problem faced by the construction industry. Not only has the construction industry a high demand for materials, but 10% of the global emission of CO_2 is due to provision of construction materials of which cement alone is accountable for approximately 85% [1]. The growing demand for reduced emissions of CO_2 is urging the cement and concrete industry to find new less CO_2 intensive materials, which allows substituting cement in blended cement and concrete.

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The cement and concrete industry have for a long period of time played the role of scavengers by retrieving waste from other sectors to be utilised in blended cement and concrete production [2]. This has led to conditions where 20 -70 % of cement is replaceable with these silico-aluminate materials, which have filled out the function as supplementary cementing material (SCM) [3]. But in order to achieve higher rates of cement substitution it is, as suggested by Scrivner et al [4], necessary to develop and use new SCM, which also are locally available. Sewage sludge ash (SSA) could be such a local available resource suitable for SCM. Sewage sludge ash derives form incineration of sewage sludge, which is a waste handling option used at water treatment plants to deal with large volumes of sludge.

Extensive research has investigated the possibilities of utilizing SSA not only as SCM in blended cement but in a wide range of building materials such as bricks, tiles, pavers, light aggregates but also for substitution for cement in concrete and mortar [5,6]. The majority studies conducted, which have tested the effect of SSA in mortar, have reported that the compressive strength decreases, when using SSA as partial cement replacement [5-7]. In order to be qualified as SCM a key question of interest, which have been addressed in previous works, is whether SSA possess pozzolanic activity and therefore belongs to the group of pozzolans like other residues such as blast furnace slag, silica fume and coal fly ash [7-11]. In the review by Cyr [7] the oxide content of SSA found in literature were compared. The analyses of the content taken from 46 studies showed that content of SiO_2 and Al_2O_3 were in average less than 50 %. This means that material parameters important for the reactivity in general are lower in SSA than in other known pozzolans. In two studies by Pan et al. [12] and Donatello et al. [11] the SSA was milled to obtain finer particle sizes. The pozzolanic activity of un-milled and milled SSA was determined by Strength activity Index (SAI) [11,12] and Frattini Test [11]. In the Frattini test the reactivity of the pozzolane is measured by monitoring the removal of $Ca(OH)_2$ from a solution of cement and the pozzolane in focus, whereas the SAI is an indirect method that determines the activity of the pozzolane by determination of the compressive strength of the test mortar. The results of the studies showed that the pozzolanic activity of SSA increased as the SSA was milled. However, Donatello et al. [11] also showed that the assessment method used to determine the pozzolanic activity provided different results. With the Frattini test pozzolanic activity was only detected for milled SSA, whereas the SAI showed

pozzolanic activity in both un-milled and milled SSA. Another important parameter for the performance of mortars with SSA is the morphology and fineness of the SSA. Results from the studies by Pan et al. [12] and Donatello et al. [11] showed a connection between decreasing particle sizes of SSA and improved compressive strength development of mortars with milled SSA. Both studies also found that even though the particles sizes of the SSA decreased due to the milling, the specific surface area of the SSA did not increase to the same extent. Therefore it was found that the SSA's were characterised by having many open pores trapping water, which effected the workability of the mortar and thereby also the hydration process due to lower available water in the system [11].

Despite of numerous studies investigating the use of SSA as SCM in mortar and concrete, research has not led to application of cement based materials with SSA in construction. This might be due to the uncertainty to what extent SSA belongs to the group of pozzolans. However, in a Danish demonstration project *Bio Crete* supported by EU LIFE program [13] utilisation of SSA in concrete production was tested on a larger scale. During a three year project period app 28.000 m^3 concrete was produced using app 2000 t of SSA in total [14]. In project it was found that a high content of Fe in the SSA affected the colour of the concrete, which changed from the normal grey to red tones depending on the quantities of SSA in the mix [14,15]. Even though the concrete produced with SSA met the technical requirements for materials to be used in construction, the change of the colour was seen as an obstacle, because it restricted the application of the concrete mainly to be used for hidden structures if not intentionally used aesthetically. The colour and possible colouration of mortar and concrete with SSA is relevant to address and include as parameter into the experimental framework in order to anticipate obstacles for its application in the build environment. The focus of present study was therefore jointly to examine how the milling of SSA affected the colour, compressive strength, and workability of mortar when 20 % of cement was replaced with processed SSA.

2. Materials and methods

2.1. Materials

SSA was collected at the wastewater treatment plant Biofos in Copenhagen, Denmark (February, 2013). The SSA is incinerated in a fluidized bed combustor at about 850°C. The plant treats wastewater from 255.000 person equivalents (PE) with a subsequent ash production of app. 2500 tons annually. Phosphorous is removed from the wastewater by chemical precipitation with Fe. The resulting ash therefore has a characteristic red iron-oxide colour. The SSA is a mixture of at least 95 % fly ash captured in the electrostatic filter and not more than 5% Air pollution control residues taken form the bag filter. It is according to the European waste catalogue classified as fly ash with a hazardous substance (EWC code 19 01 13) [16].The SSA was collected directly from the process line and stored in sealed plastic containers at room temperature. The SSA was both used as-received and milled, and these samples are named SSA and SSA_{Xmin}, respectively, where x is the duration of the milling.

For mortar preparation a Portland cement (CEM II/A-LL 52.5R) was used. This cement type has a content of less than 20% limestone filler. The sand used in the experiment was a natural sea sand 0-4mm with technical specification following DS/EN 12620 [17] and DS 2426 [18] In this paper, the term “test material” covers SSA (as-received and milled) and test binder (cement and SSA).

2.2 Drying and milling procedures

The impact of drying the SSA on particle size distribution of the ash was investigated. The SSA was dried for 24 h at 50˚ or 105˚ respectively. Two samples were dried at 50˚ or 105˚ before they were milled, and compared with two samples which also were dried at 50˚ or 105˚ but after the milling, SSA as-received and cement. A vibratory cup mill (FRITSCH - pulverisette 9) with the capacity to process a batch size of 250 ml was used for milling the SSA. The SSA was placed in the grinding set which consisted of a container with a ring and a puck inside. The grinding set was placed on a vibrating plate and the sample was milled by

centrifugal force. The samples were dried milled for 30 sec. The particle size distribution of cement, SSA as -received and four milled SSA samples were analysed by laser diffractometry.

For the remaining part of the work, the procedure for drying and milling was: drying at 50 °C for 24 h before milling. Milled SSA samples from 6 different durations were produced: 0 sec, 10 sec, 30 sec, 3min, 6 min, and 10 min. Particle size distribution was measured for each fraction and they were compared to the particle size distribution of cement.

2.3 Analytical procedures

The concentrations of the trace elements Ni, Cr, Cu, Zn and Pb in the test samples were measured after the pre-treatment procedure described in DS/EN 259 [19]: 1.0 g material and 20.0 ml (1:1) HNO_3 was digested at 200 kPa (120 °C) for 30 min. The digested suspension was filtered through 0.45 µm filter paper, and the filtrate analysed by ICP–OES (Induced coupled plasma – optical emission spectrometry). The equipment used for the analyses was a Varian 720-ES.

The water content of the test samples were measured as weight loss by drying at 105°C for 24 hours. The pH was measured by suspending 10.0 g of test material in 25 ml distilled water. After 1 h agitation pH was measured directly in the suspension. Loss on ignition (LoI) was determined as weight loss after 30 minutes at 950 ˚C. Solubility in water was evaluated by suspending 100 g test material in 500 ml distilled water. After agitation for 1 min and settling, the water was decanted and 500 ml new distilled water was added. This was repeated and the ash was washed three times. Finally the suspension was filtered, dried and weighed, and the solubility expressed as weight loss by this procedure.

The buffering capacity of the test materials was determined by firstly preparing a suspension of the test material mixed in water (6.7 % w/v) secondly stirring the suspension for 30 min before pH was measured. Successive 10 ml of concentrated HCl were made every 30 min and pH was measured thereafter. This was repeated every 30 min until pH was below 2 [20]. The analyses conducted comprised, besides a

characterisation of SSA as received (SSA) and cement, also an analysis of Test Binder (80 wt % cement and 20 wt % SSA.)

Major oxide composition and Cl content in SSA and cement was found by X-ray fluorescence (XRF) on powder samples by an external laboratory. Images of particle morphology were made using a scanning electron microscopy (SEM) of a small sample placed directly on carbon tape. The SEM apparatus used was a FEI Quanta 200. It was equipped with a large field detector and x-ray cone and the accelerating voltage of the SEM was 15 kV. Particle size distribution of the test materials was determined by a laser diffractometry and the apparatus a Malvern Mastersizer 2000.

2.4 Mortar preparation and compressive strength test

The mortar preparation followed the procedures as described in DS/EN 191-3+A3 [21] except for the sand, where the 0-2 mm sand prescribed was replaced by coarser sand with a grain size distribution between 2- 4 mm. This sand was chosen to have a coarser consistency, closer to that of concrete, but still manageable at laboratory scale.

In the experimental 20 wt % of the cement was replaced by SSA. This percentage was chosen in order to obtain results that would clearly show what effect milled SSA had on colour, compressive strength development and workability. The same percentage of cement replacement was used in two studies in which milled SSA was tested for pozzolanic activity [11], setting time and workability [12]. 20 % of cement replacement can also be seen as an appropriate starting point for dealing with the environmental implications of cement production. Seven experimental mortars were produced, five with substitution of milled SSA, one with substitution of un-milled SSA (0sec)and one control sample without SSA (Ref) No additional water was added to any of the test samples. A description of the different mortars is shown in Table 1. The mixing, casting procedures and the moulds used were as prescribed by DS/EN 191-3+A3[21]. The mortar samples were removed from the moulds after 24 h, placed vertically in a water bath 20 °C and cured for 28 days. Each prismatic mould produced 3 specimens measuring 160mm x 40mm x 40mm which after curing were

cut into 6 equal test samples measuring 80mm x 40mm x 40mm. A Toni 3000 compression machine was used for the determination of the compressive strength. The halved samples were loaded side faced with a momentum of 2400 N/s. Detection of fracture was set at 2%.

Table 1 Recipe for reference and test mortars

Mortar sample	SSA duration of milling	cement	SSA	sand	water
REF	÷	450 g	÷	1350 g	225 g
0SEC	0 sec	360 g	90 g	1350 g	225 g
10SEC	10 sec	360 g	90 g	1350 g	225 g
30SEC	30 sec	360 g	90 g	1350 g	225 g
3MIN	3 min	360 g	90 g	1350 g	225 g
6MIN	6 min	360 g	90 g	1350 g	225 g
10MIN	10 min	360 g	90 g	1350 g	225 g

2.5 Workability

The flow value expresses the workability of mortar with un-treated and milled SSA. Preparation of mortars followed DS/EN 191-3+A3(DS 2009) and the tested mortars are those listed in Table 1. The flow value was determined according to DS/EN 1015-3[22]. A truncated conical mould (50 mm high, internal diameter 100 mm at the bottom and 70 mm at the top) was uniformly filled with mortar. The mould was removed, and the mortar exposed to jolting by slowly raising the mould 2 cm vertically and dropping it, 15 times at a rate of one pr. second at a flow table. The mean diameter (d_{mean}) from two measurements of the subsequent mortar diameter in two directions at right angles was found. The procedure was repeated twice for each mixture. The flow value is defined as D_{mean} of second measurement and accepted, if D_{mean} differs less than 10 % between the two mixtures.2.6 Colour samples

The seven mortars in Table1 were prepared for the production of samples for colour evaluation. The mixing followed the same procedures as for compressive strength testing. However, the moulds used were three

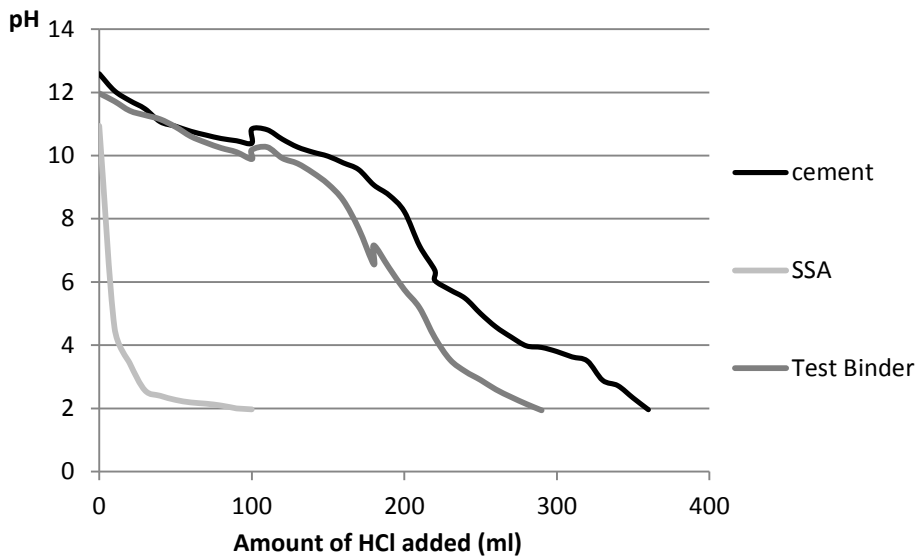
compartment moulds made from film faced ply wood where each compartment had the internal dimensions 100x100x30mm. The mortar was uniformly distributed in the mould by means of a vibrator table, covered in plastic and kept in the wooden mould for 24 h. The samples were ejected and stored at room temperature without any exposer to daylight.

3. Results

3.1 Material characteristics

The characteristics of the test materials: SSA, SSA_{10min}, cement, Test Binder (80 wt % cement and 20wt % SSA) are shown in Table 2. The results show that all test materials were alkaline. However, the pH of SSA was 9.9 against pH 12.6 for both cement and test binder (Table 2). This finding was supported by findings provided by the determination of the buffering capacity (Figure 1).The graphs monitoring the buffering capacity of cement displays a high resistance against acidification as the pH dropped slowly. Even though the buffering capacity of SSA was significantly lower in comparison to cement, the buffering capacity of the test binder was relatively high and to some extends comparable to the buffering capacity of cement.

Figure 1 Buffer capacity of SSA, SSA10min, cement and Test Binder



The SSA had a water soluble fraction of about 1.5% per weight (Table 2). Determination of water solubility gave negative values for cement and for the test binder. The negative values represent an increase in mass due to the hydration process. The values found display that the hydration process of the test binders was less reactive as these values were less negative than for cement.

The chemical analysis showed that the concentration of trace elements Cu, Zn and Pb were significantly higher in the test binders than in cement due to the higher concentration levels in the SSA. The concentrations of Cr and Ni, on the other hand, were only slightly elevated in the test binder compared to cement, as the concentration of these trace elements were less than half and half, respectively, in the SSA compared to cement.

Table 2 Characterisation of SSA, SSA10min, Cement and Test Binder

	SSA	Other SSA[a] Mean	cement	Test Binder
water content %	0.63 ± 0.13	-	0.28 ± 0.11	0.24 ± 0.09
water solubility %	1.27	-	− 3.56	− 1.93
pH	9.9 ± 0.00	-	12.6 ± 0.02	12.6 ± 0.01
Loss on ignition (%)	1.35 ± 0.04	-	7.04 ± 0.09	5.81 ± 0.05
Major oxides (%)				
Al_2O_3	5.1	14.2	4.91	4.95[b]
CaO	23.8	14.8	65.7	57.5[b]
Fe_2O_3	15.7	9.2	5.43	7.48[b]
K_2O	1.57	1.3	0.81	0.96[b]
MgO	2.32	2.4	0.53	0.89[b]
MnO	0.09	0.3	0.04	0.05[b]
Na_2O	1.15	0.9	0.67	0.77[b]
P_2O_5	20.2	11.6	0.23	4.22[b]
SiO_2	17.1	36.1	20.1	19.5[b]
SO_3	2.02	2.8	4.74	4.2[b]
TiO_2	0.83	1.1	0.35	0.45[b]
Cl	0.01	-	0.1	0.08[b]
trace elements (mg/kg)				
Ni	57.5 ± 1.53	671	27.0 ± 5.55	35.6 ± 1.15
Cr	38.7 ± 0.76	452	26.0 ± 4.85	30.7 ± 2.11
Cu	688 ± 17.3	1962	67.5 ± 13.1	183 ± 8.12
Zn	1930 ± 26.8	3512	115 ± 22.0	415 ± 17.0
Pb	144 ± 2.00	600	21.6 ± 4.49	46.3 ± 1.56

[a] Mean values of oxide and heavy metal content in SSA samples reported in Cyr et al
[b] calculated oxide content on basis of the detected content values of SSA and cement

The distribution from XRF of the major oxides for SSA was: CaO > P_2O_5 > SiO_2 > Fe_2O_3 > Al_2O_3 (Table 2). In comparison to mean values reported in the study Cyr et al [7] the SSA of present study had a high content of P_2O_5, relatively low content of SiO_2 and Al_2O_3.The content of P_2O_5 was 20 % and was at the same level as CaO (23.8 %) and Fe_2O_3 (17.5 %) %. When SSA and the content of four main constituents of cement: CaO, SiO_2, Fe_2O_3 and Al_2O_3, are compared, only the content of SiO_2 was at a comparable level to the content found in cement. MgO and MnO were found to be between 2- 4 times higher in SSA than the levels found in cement. Only the content of SO_3 was higher in cement than in SSA which was 2.02% against 4.74% for cement. The major oxides composition for Test Binder was calculated on basis of the measured compositions for the two parts SSA and cement (Table 2) and it had quite similar composition as cement. The concentration of P_2O_5 was however, much higher (4.2% against 0.2%) for Test Binder due to the high content in SSA. The content of heavy metals Ni, Cr, Cu, Zn and Pb were much lower than the mean values reported in the study by Cyr et al [7].

The results of the particle size distribution analysis' seen in Figure 2 show that finer particles are obtained if SSA is dried before milling regardless the applied temperature. Samples of SSA were dried either before (Bef) or after (Aft) milling at 50°C and 105°C. At 50 % of volume for $Aft_{50°C}$ the accumulated volume had increased by approximately 10 % in comparison with $Bef_{:50°C}$. Thus finer particles were obtained when the SSA was dried before the milling. The temperature applied did not affect the particles size distribution. Based on this result, the SSA was dried at 50°C before it was milled in order to obtain the smallest possible particle sizes in the remaining experiments.

Figure 2 Particle size distribution of cement, SSA as-received, and milled (30 Sec) SSA dried either before (Bef) or After (Aft) the milling

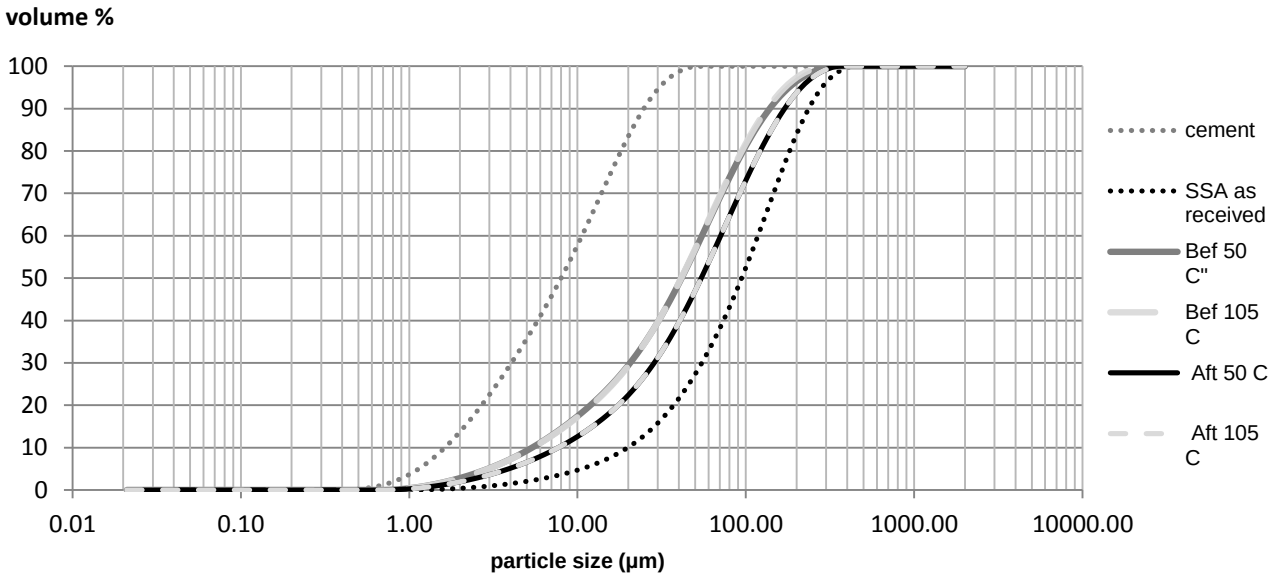
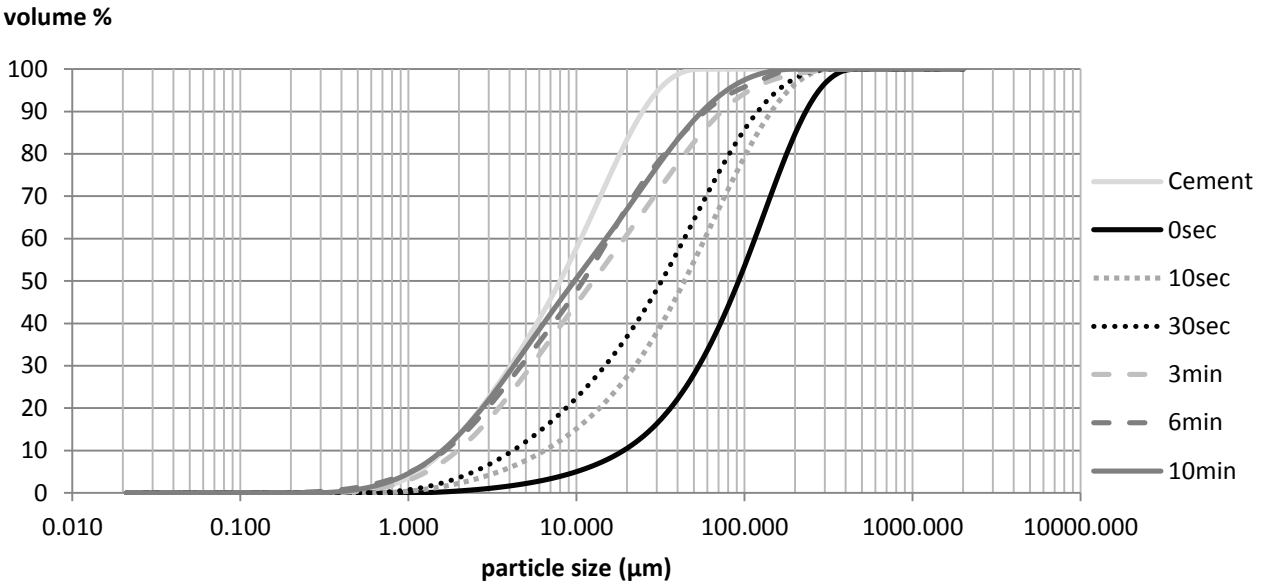


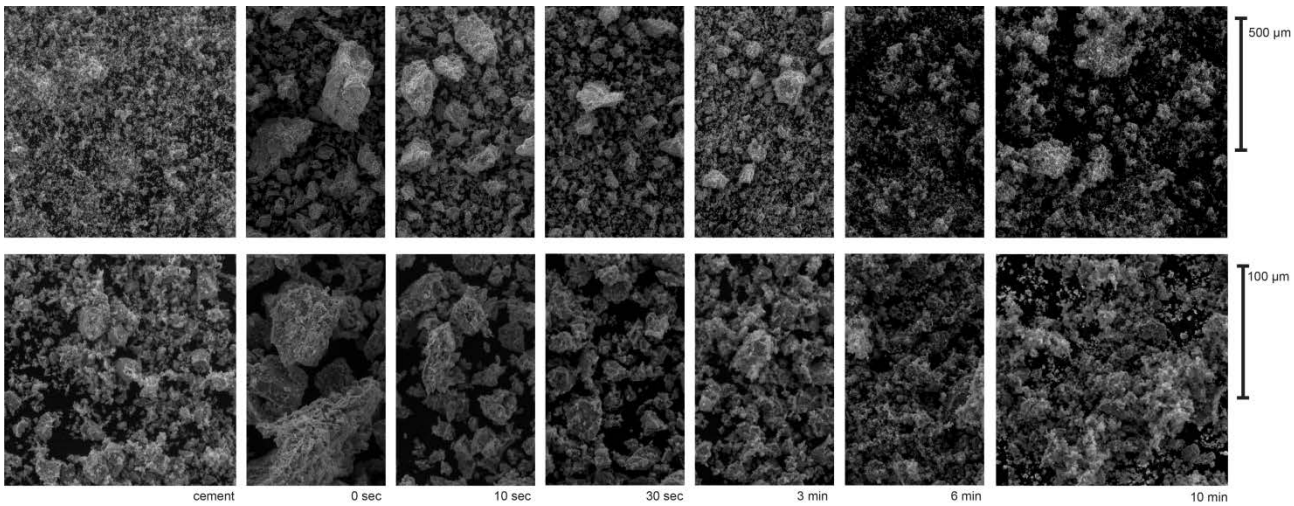
Figure 3 Particle size distribution of cement and SSA milled in interval between 0 sec and 10 min



The effect of the milling process for different durations was analysed by comparing the particle size distribution and morphology of the milled SSA with the particle size distribution and morphology of cement

(Figures 3 and 4). The effect of milling SSA can be seen in Figure 3. A comparison of the particle size distribution for the milled SSA shows that the slopes of the curves and the medium size particles (d_{50}) move closer to that of cement when milling time reached intervals between 3- 10 min, and further decrease of the particle sizes was minor between this intervals (Figure 3). SEM images of the morphology of as-received, milled SSA and cement (Figure 4) support the findings from Figure 3. The effect of the milling on the coarse particles of as-received SSA, which were steadily crushed as the duration of the milling increased, can easily be observed in Figure 4.

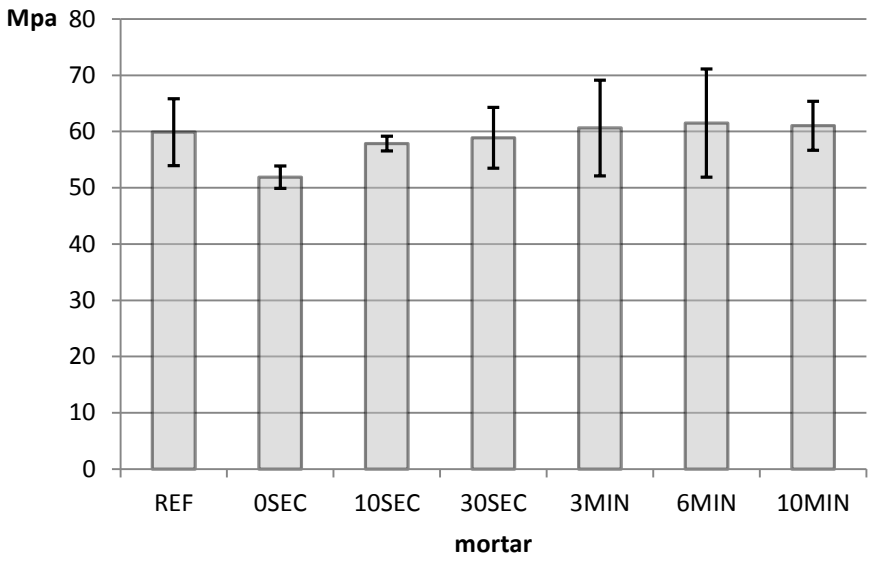
Figure 4 SEM images of cement and milled SSA in interval 0sec- 10min



3.2 Material properties: compressive strength, workability and colour

The results of the compressive strength test (Figure 5) showed a positive effect from milling the SSA. A decrease in compressive strength was found when 20 % of cement was replaced by SSA as received. The measured compressive strength of the control (REF) was around 60 MPa and decreased by 13.4% to the level of 52 MPa(0SEC) when cement was replaced by un-milled SSA. However, the compressive strength improved immediately when SSA had been milled, even for only 10 sec. The compressive strength for 10SEC was approximately 58Mpa, a decrease of only 3.4% compared to the compressive strength of REF. Test mortars containing SSA milled for 3- 10 min achieved the same level as REF.

Figure 5 Compressive strength after 28 days



The workability, evaluated by determining the flow value of test mortars, is seen in Figure 6. The particle size distribution of SSA was essential to the workability of the six test mortars. The flow value of 0SEC, where 20 % cement was replaced by SSA as received, decreased by 35% in comparison to REF . As the milling duration increased, the flow values increased correspondingly. For sample 6MIN the flow value was close to that of REF.

Figure 6 Flow value -Workability of control and test mortar

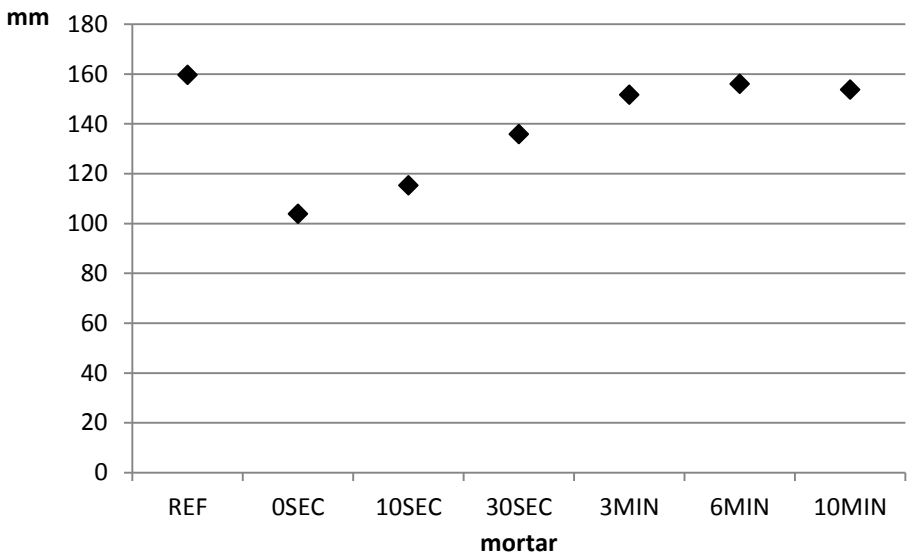


Figure 7 shows the colour samples. It shows that the colour of mortar containing milled SSA evolved simultaneously to an increased duration of the milling (Table 1). The images also display that the colour tone of 0SEC, which was grey colour with a slight red tint, was comparable to the grey colour of REF. In figure 8 the three samples: 0SEC, 10SEC and 6MIN are displayed together with REF, and it illustrates that the colour progression of the six samples containing SSA can be ordered in three step colour scale. In the colour scale each of samples has a distinct colour different from the neighbouring sample. The remaining samples which are not included in the colour scale in Figure 8 have tones which are similar to the samples: 0SEC, $10SEC_c$ and 6MIN.

Figure 7 Colour samples of reference mortar, as received and milled SSA arranged in the following order. REF – 10MIN

Figure 8 Colour samples REF, 0SEC, 10SEC and 6MIN

4. Discussion

The findings of the present study supports the findings of Donatello et al. [11] and Pan et al. [12] as all three studies showed that the compressive strength and the workability improved when SSA was milled. In the present study the milling of SSA did not result in particle size distributions that exceeded the particle size distribution of cement (Fig 3). On the other hand the milling of the SSA did provide a material which could replace the cement by 20 wt % and at the same time gain compressive strengths and flow values which were comparable and reached the level of REF (figure 5 and6). In comparison, results found in the studies by Donatello et al. [11] and Pan et al. [12] showed that the compressive strength only 94 % and 77 % to the level of the compressive strength of ordinary mortar respectively. The differences in the results found between the studies may be due to differences in materials parameters. For instance, the cement used in present study was a CEM II, whereas Donatello et al. [11] used a CEM I for mortar production.

Results on compressive strength found in studies by Monzó et al. [9] and Garcés et al [23] showed that the strength development depended on the cement used in test mortars with SSA. These studies therefore exemplify the relevance of considering the type of cement when discussing the reactivity of SSA and the use of SSA as SCM. However, in order to be able to answer and understand how SSA interact with the clinker phases it may require as Lothenbach et al [24] suggest more advanced assessments methods as parametric modelling, based on profound knowledge on thermodynamics of the compounds, to determine the hydration products formed, and thus be able to predict the long term performance of the cement based material produced.

A direct correlation between increasing flow values and increasing compressive strength due to the milling of the SSA is clearly seen in figure 5 and 6. The gradual pulverisation of the coarse and angular particles (Fig 4) affected simultaneously the workability and the compressive strength. The values found in each experiment correspond to one another, as they follow the same trend. Since the flow value of the fresh mortar increased without any addition of extra water, the reason was most likely due to changes in the morphology of the SSA, and the circumstance that the availability of water in the mix was raised because, as

discussed by Donatello et al. [11], the imbibing porous particles of the SSA is crushed down without gaining larger specific surface area.

The colour samples produced in this study display that the colour changed gradually from the normal grey of ordinary mortar to increasing tones of red when the fineness of the SSA increased due to the milling (Fig.7) This finding confirms the finding of a previous study by Kappel et al. [25], which revealed that the colour did not change significantly unless the SSA was milled to obtain finer particles and thus, it was found that the milling of an iron rich SSA was a precondition for the red colour to evolve. This study showed that increasing colours of red correspond to increasing flow values and compressive strength, which means whereas the performance of mortar with milled SSA compares to performance of ordinary, it differs visually from its starting point.

In the project Biocrete [13] the colour was used as marker for the quantities to be used without affecting the colour of concrete at the same time, even though concrete with a reddish colour containing a higher amount of SSA met the technical requirements set for the concrete [14]. If the motivation for utilizing SSA is to reduce the amount of the CO_2 intensive clinker by substitution of SSA, the colour of the concrete seems less important as a measure to control the substitution rate. In general, the colour has not been included as an experimental parameter into studies on SSA containing mortar or concrete and no previous studies to the author knowledge have reported on the colour of mortar with SSA. This could be due disengagement with the importance of the subject, and the circumstance that the colour of the cement based material is not relevant for its application. However, exemplified by the Biocrete project, the colour change of concrete may be regarded as limiting factor for a general application, and therefore we believe that the colour of SSA containing mortar and concrete is relevant to address, and to unfold the colour potential of milled SSA which intentionally can be used aesthetically and/or integrated in the design solution.

5. Conclusion

The results of the study showed that milling of the SSA improved the strength development and the workability of mortar with SSA to the extent that the performance of mortars with 20 % cement substituted by $SSA_{3\text{-}10\ min}$ were comparable to the compressive strength and the workability of a ordinary reference mortar. Mortar with the iron rich SSA did not change its colour significantly unless the SSA was milled. Mortar with un-milled SSA was in a grey colour with a slightly red tint. However, the colour increased to the extent that mortar changed colour from grey to a reddish colour as the particle size of the SSA decreased due to better distribution of smaller particles in the matrix. Overall, the results of the study showed that simple pre-treatments of SSA: drying and milling have an effect on the performance of the mortar, which provide an opportunity to adjust the SSA in accordance to requirements set for application. However, the question on the reactivity of SSA and its long term performances may have to be settled before SSA may obtain the status secondary resource suitable as SCM in blended cement.

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