TECHNICAL PAPER

JOURNAL OF THE SOUTH AFRICAN INSTITUTION OF CIVIL ENGINEERING
ISSN 1021-2019
Vol 64 No 1, March 2022, Pages 37–47, Paper 1315

Assessment of local sewage sludge ash as a supplementary cementitious material – effects of incineration temperature and cooling rate of the ash

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This paper reports on the possible use of sewage sludge ash as a pozzolanic supplementary cementitious material to Portland cement. Samples of sewage sludge were incinerated at 700°C, 800°C and 900°C and these were then cooled in the furnace (F_{ISSA}), in air (A_{ISSA}) or by quenching in water. The resulting ashes were ground to suitable fineness and used to prepare cement pastes and mortars in which the binder consisted of 30% ash and 70% Portland cement. The paste samples were used for microscopic and chemical assessment of the evolution of hydration products, while the mortars were used to assess the effects of the ashes on workability and compressive strength of laboratory-prepared samples using a water/binder ratio of 0.5. Fly ash was used as a reference pozzolanic material to assess the performance of sewage sludge ashes. Analysis of the sewage sludge ashes showed the presence of cementitious compounds and hydration products that suggest that this material can be used as a partial replacement of Portland cement. However, sewage sludge ash reduces the workability of the mortar. Compressive strength results indicate that the highest strength is obtained when the sewage sludge is incinerated at 900°C and then quenched in water.

INTRODUCTION

Waste disposal is a recognised global health problem and, with the human population growing, the demands for safe waste disposal are increasing. Sewage sludge, which is the residue from a wastewater treatment plant, is one of the largest and particularly problematic contributors to the burden of waste disposal worldwide. The sludge is of a semi-solid nature produced as a by-product from treatment of municipal sewage and industrial wastewater. The global estimate is that between 70 and 105 million tons of sewage sludge is produced annually (Grobelak *et al* 2019). Waste recycling in the construction industry is one of the best ways to provide new, safe and renewable resources that contribute to economic development and thus protect the environment.

Fleishman *et al* (2014) argue that South Africa is one of the countries facing difficulties in the disposal of wastewater sludge. Environmental and public health concerns of sewage sludge disposal have historically been centred around the presence of harmful pollutants such as pathogens (e.g. viruses, parasites and bacteria); organic contaminants (e.g. dioxins, surfactants and pharmaceuticals) and inorganic contaminants (e.g. metal trace elements) (National Research Council 2002; Lewis & Gattie 2002). There is clearly a need for much further assessment and analysis of alternative use possibilities to develop the evidence base that will improve sewage sludge disposal practices around the world.

In its turn, the concrete industry has long advocated for increased use of supplementary cementitious materials (SCM) such as ground-granulated blast furnace slag and fly ash (FA) to replace a part of the Portland cement used in concrete and so reduce the general carbon dioxide footprint of concrete construction (Scrivener *et al* 2017). Incinerated sewage sludge ash (ISSA) reportedly contains oxides such as Al_2O_3, CaO and SiO_2 which are the primary compounds found in Portland cement (PC)

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Keywords: sewage sludge ash, pyro-processing, pozzolanicity, mortar strength, workability

Juala R, Ballim Y, Mulopo J. Assessment of local sewage sludge ash as a supplementary cementitious material – effects of incineration temperature and cooling rate of the ash. J. S. Afr. Inst. Civ. Eng. 2022:64(1), Art. #1315, 11 pages. http://dx.doi.org/10.17159/2309-8775/2022/v64no1a4

(Tenza-Abril *et al* 2015), which makes ISSA a potential SCM or pozzolanic material that can be used in cement-based materials (Tay 1987; Gomes *et al* 2019; Payá *et al* 2019).

Previous studies (Monzó *et al* 1997; Tantawy *et al* 2012; Dyer *et al* 2011; Pavlik *et al* 2016) have shown evidence that ISSA exhibits a certain level of pozzolanic activity, as is the case with fly ash and silica fume. Pozzolans are materials that contain aluminous and siliceous material, which when finely divided react with calcium hydroxide $Ca(OH)_2$ to form cementing compounds found in hardened cement paste (Massazza 1998). The use of these materials in concrete brings durability benefits while reducing the CO_2 footprint of the binder and reducing the amounts of waste material sent to landfill (Scrivener *et al* 2017).

The decomposition of sewage sludge with increasing temperature occurs at different stages. The four principal stages are described below (Tantawy *et al* 2012):

- The loss of moisture and absorbed water at 50°C – 130°C
- The emission of volatile organic matter at 200°C – 320°C
- The combustion of organic content due to the varying boiling points of hydrocarbons in the sewage sludge at 330°C – 390°C
- The thermal decomposition of fixed carbon captured by inorganic matter, loss of structural water of the clay minerals in the sewage sludge and the elimination of carbonaceous matter in the sewage sludge at 400°C – 670°C.

To ensure that all organic matter is fully decomposed before the material is used as an SCM in concrete, sewage sludge should therefore be incinerated at a minimum temperature of 700°C.

Figure 1 shows a calcium, silica and alumina oxide ternary diagram of cementitious materials commonly used in concrete. The chemical analysis of materials in this study was used to locate ISSA on the diagram. It can be seen that ISSA overlaps strongly with FA in terms of the CaO, SiO_2 and Al_2O_3 content.

Previous studies have shown that the partial replacement of PC by ISSA affects both the strength and workability of binder pastes (Pan *et al* 2003; Monzó *et al* 2003). Other studies have shown that using ISSA as an SCM in mortar extends the setting time and reduces the workability of the mortar (Vouk *et al* 2016; Naamane *et al*

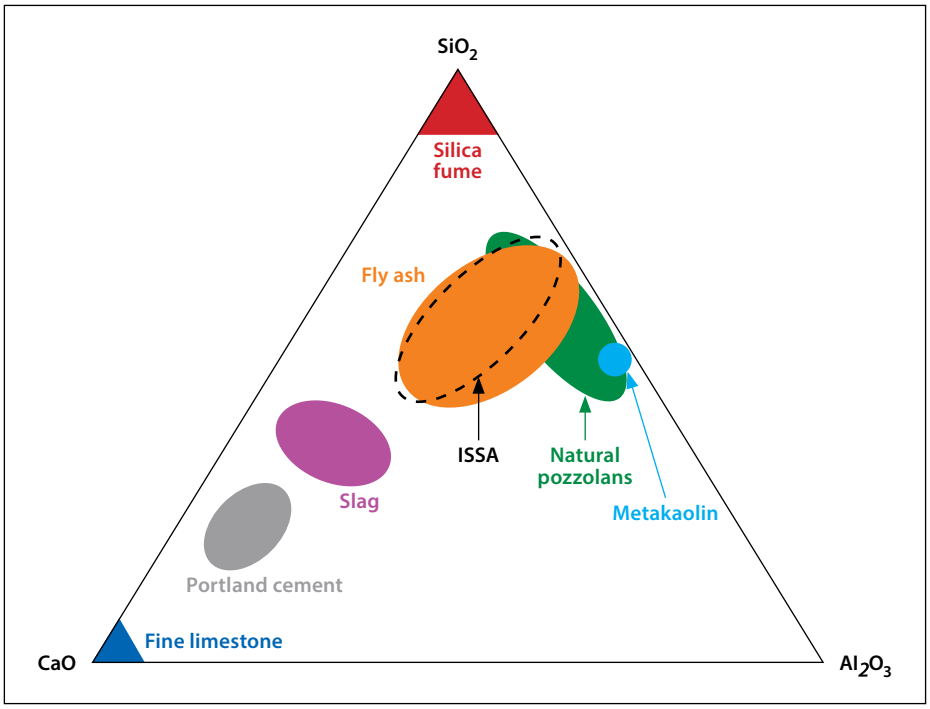

Figure 1 Ternary diagram of silica, alumina and calcium oxides showing commonly used cementitious materials and the relative position of ISSA used in this study (adapted from Rakhimov *et al* 2015)

2016). The increased setting times hav been attributed to the high amounts of P_2O_5 and SO_3 found in the ISSA (Vouk *et al* 2016; Naamane *et al* 2016).

Vouk *et al* (2016) investigated the effect of ISSA on the workability of cement mortar. The results showed that 10% replacement of cement by ISSA caused a 19% reduction of the mortar workability. Similar results were obtained by Monzó *et al* (1996) who used ashes with three different fineness levels, at 15% replacement of PC. This reduction in workability of the cement mortar was attributed to the irregular morphology and the increased water absorption of ISSA. Vimonsatit *et al* (2015) point out that the significantly higher water demand of ISSA can be resolved by adding coal fly ash.

Naamane *et al* (2016) investigated the effect of sewage sludge incineration temperatures on the compressive strength of mortar produced by partially replacing cement with ISSA. They found that an incineration temperature of 800°C for a period of 2.5 hours yielded the highest compressive strength of the PC/ISSA mortars after 90 days.

Vouk *et al* (2017) found that, at 72 days, mortars containing 90% PC and 10% ISSA had a compressive strength that was about 7% lower than that of companion mortars made with 100% PC. In a study conducted by Cyr *et al* (2007), the effect of the curing time on the compressive strength of mortars containing ISSA at replacement levels of 25% and 50% was considered. It was found that the rate of compressive strength gain was lower at early ages, but accelerated after 28 days. Also, the compressive strength of mortar containing 25% ISSA was higher than that of mortar containing 50% ISSA.

The study reported in this paper was aimed at evaluating the feasibility of ISSA from a wastewater treatment plant in the Gauteng area to be used as an SCM in cement-based materials for construction. In particular, the study was focused on the effects of the post-incineration cooling rate of ISSA on the workability and compressive strength of mortars, when used at 30% replacement of PC. Samples of sewage sludge were incinerated at 700°C, 800°C and 900°C and then cooled in the furnace or in air or by being quenched with water. The sewage sludge and the resulting ISSA materials were characterised for their physical and chemical characteristics. Paste and mortar samples made with ISSA and PC were assessed for their microstructural development during early hydration, as well as for workability in the plastic state and for compressive strength development up to 28 days. Pastes and mortars prepared with plain PC and with a 70/30 blend of PC and fly ash (FA) were used as reference materials for comparison of the overall performance of ISSA as a possible SCM.

Table 1 Chemical composition (in %) of PC, FA (Ballim & Graham 2009) and DSS

Oxide	PC	FA	DSS
CaO	64.00	4.20	3.88
SiO_2	22.90	55.10	21.42
Al_2O_3	4.60	33.30	5.89
Fe_2O_3	2.58	3.15	4.79
MgO	1.60	1.20	1.12
Na_2O	0.12	0.00	0.22
P_2O_5	0.06	0.39	3.95
K_2O	0.30	0.69	0.51
TiO_2	0.46	1.67	0.44
Mn_2O_3	0.12	0.02	0.13
SO_3	2.05	0.12	0.00
LOI	2.41	0.45	56.64

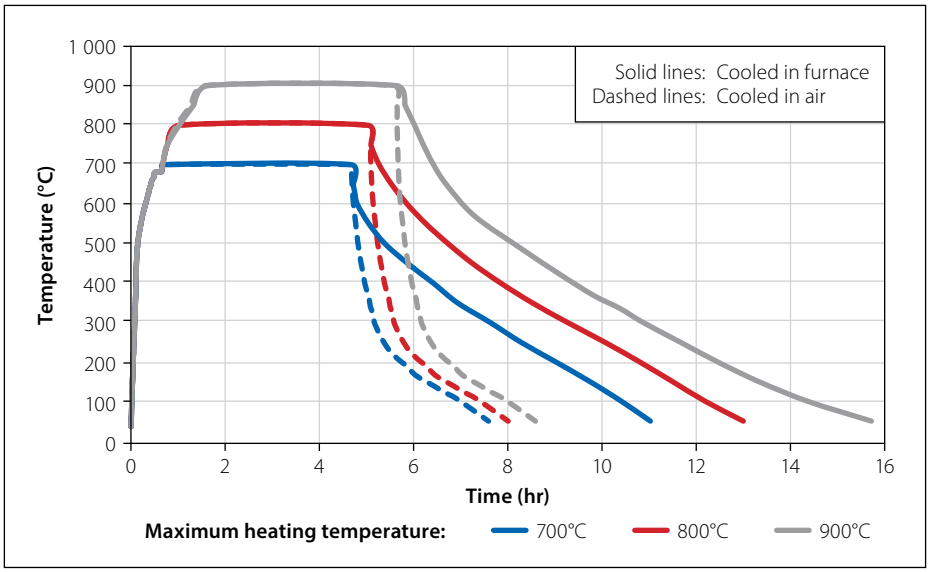

Figure 2 Measured heating and cooling curves for the samples cooled inside the furnace and in laboratory air

MATERIALS AND TEST METHODS

Cementitious materials

In this study PC classified as CEM I, 52,5 N and FA from a local supplier were used to prepare paste and mortar samples. Sewage sludge with 95% solids and 5% water was collected from a wastewater treatment plant in Gauteng, after it had been dried in the sun for some time. The collected sewage sludge was further dried in an oven at 100°C for 24 hours to produce dried sewage sludge (DSS). X-ray fluorescence (XRF) analysis was then conducted using a PANalytical Axios instrument on the DSS to determine its chemical composition, and the results are shown in Table 1. The loss on ignition (LOI) was determined by roasting the sample at 1 050°C until constant weight.

As a basis for comparison, the analyses of samples of PC and FA, similar to that used in this study and derived from the same sources, and determined by Ballim and Graham (2009), are also shown in Table 1. It is interesting to note that the DSS used in this study contained no measurable SO_3 which Vouk *et al* (2016) and Naamane *et al* (2016) noted as contributing to increased setting times of the ISSA materials that they studied. The high LOI of the DSS is common with organic materials and is mainly caused by a loss of carbon and bound water.

Separate samples of DSS were then incinerated at 700°C, 800°C and 900°C in a closed-lid furnace. The furnace was heated at an average rate of 17°C/minute until 700°C and thereafter, for the 800°C and 900°C samples, at an average rate of 4°C/minute. The furnace temperature was then held at the intended incineration temperature for four hours, after which the furnace heater was turned off. Three samples of DSS were prepared at each of the incineration temperatures and these were cooled as follows:

- One sample was left to cool inside the furnace.
- One sample was immediately removed from the furnace and left to cool in still laboratory air.
- One sample was immediately removed from the furnace and quenched by pouring the ash into a bucket of water at room temperature. This sample was then placed in a ventilated oven at 100°C to dry.

Figure 2 shows the measured heating and cooling rates of the samples left to cool in the furnace and in air. The temperature was monitored by placing a type K thermocouple in the sample, connected to an external temperature recording unit. The cooling rate of the quenched sample was not measured as the temperature drop occurred too rapidly.

Table 2 shows the yield of ash for the different incineration temperatures, as a proportion of the mass of DSS incinerated, as well as the average cooling rates for the furnace and air samples, when cooled to 50°C. This table also indicates the identification codes for each of the nine ashes produced for this study.

The cooled samples of ISSA were then ground for 20 minutes in a pulveriser (0068I Fact East model). Particle size analysis of the powdered materials was carried out using a laser diffraction-based Malvern Mastersizer (MS2000) to determine the particle size distribution of the PC, FA and

Table 2 Characteristics and identification of the nine ISSA samples produced for this study

	Incineration temperature (°C)								
	700°C			800°C			900°C		
Average ash yield (% by mass)	47			44			41		
Cooling method	Furnace	Air	Quench	Furnace	Air	Quench	Furnace	Air	Quench
Average cooling rate (°C/min)	1.7	3.7	–	1.6	4.3	–	1.4	4.7	–
Sample identification	7F	7A	7Q	8F	8A	8Q	9F	9A	9Q

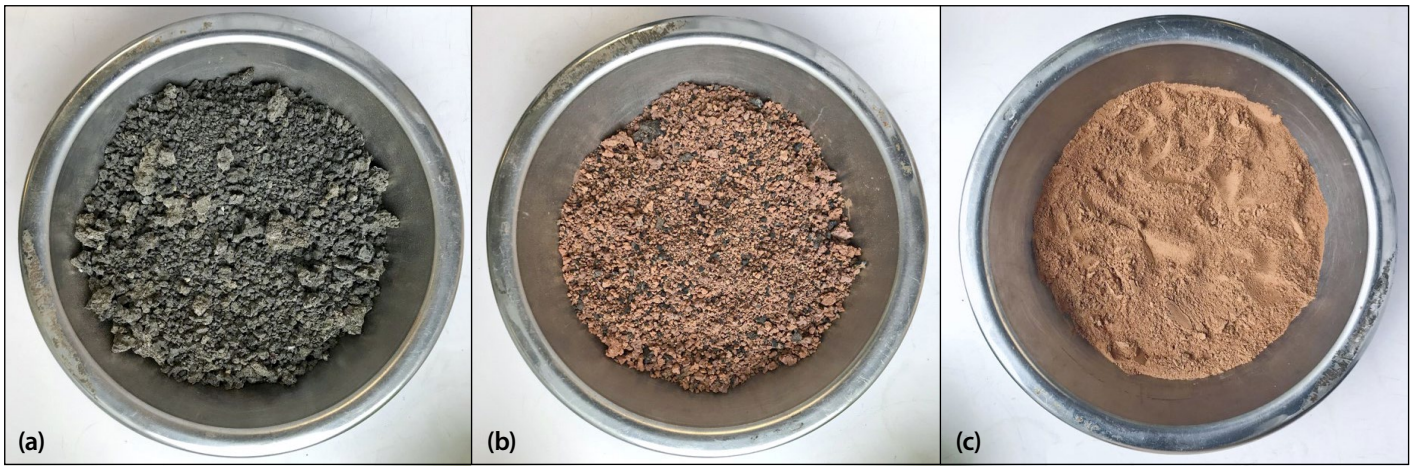

Figure 3 Change of form of DSS (a), after incineration (b) and after pulverising (c)

ISSA. Figure 3 gives a visual sense of the transformation of the DSS after incineration and pulverisation.

Atomic absorption spectroscopy (AAS) analysis

Atomic absorption spectroscopy was used to analyse the elemental composition and concentration of DSS and ISSA to determine the compositional effects of the incineration, the incineration temperatures and cooling rates. A microwave digester was used to convert pulverised DSS and ground ISSA samples in an acid suspension, into a solution prior to the analysis.

Samples for analysis were prepared using 1 g of each material suspended in a combination of 3 mL hydrochloric acid (HCl) and 10 mL nitric acid (HNO_3) standard solution. The samples were then digested for 20 minutes and filtered using filter paper. Because of the high concentrations of HNO_3 and HCl, 2 mL of the digested and filtered mixture was diluted with water to produce a standard solution of 25 mL with a suitable concentration level to produce reliable results. The samples were then analysed using an Agilent 200 Series AAS analyser. Acetylene was used as fuel gas and air as the oxidant to detect each element after excitation by hollow cathode lamps.

X-ray fluorescence (XRF) analysis

The DSS and ISSA materials were also characterised for their chemical composition using XRF analysis. Samples were initially ground to suitable maximum particle sizes and submitted to a local cement laboratory for analysis as part of their routine testing programme.

Scanning electron microscopy (SEM)

A Jeol JSM 6400 scanning electron microscope, equipped with energy dispersive spectroscopy (EDS) capacity, was used for microscopic examination of the ISSA samples. EDS allows a chemical element analysis of particular features of the sample being viewed in the SEM. The analysis was used to study the surface morphology of the ground ISSA, as well as the nature of hydration products after six hours and seven days of hydration of the binder pastes. These times were chosen to give a sense of the early and more mature stages of hydration. Because SEM analysis was intended to be largely qualitative, this part of the study considered only the ISSA samples that had been incinerated at 800°C.

Characterisation tests

A Malvern Mastersizer (MS2000) was used to determine the particle size distribution of PC, FA and ISSA. The analysis was conducted on dry powder samples. Also, the density of a ground ISSA sample was determined as 2 285 kg/m^3 using a standard pycnometer method (ASTM 2000).

Compressive strength and workability tests

Mortar samples were prepared to study the effect of ISSA on workability and compressive strength at a PC replacement level of 30%. To compare the effectiveness of ISSA, two reference mortars were prepared – one containing 100% PC and one containing a 30/70 blend of FA and PC as binders. A local crushed andesite sand was used as aggregate for the mortar. A total of 11 mortar mixes were therefore prepared – one with 100% PC as binder, one with 30% FA as part of the binder, and nine with 30% ground ISSA as part of the binder, using all the ISSAs described in Table 2, with a water/binder ratio of 0.5. The mixture proportions of the mortars are shown in Table 3. The sand content was adjusted to account for the increase in volume of the FA and ISSA in replacing a part of the PC. The amounts of materials indicated in Table 3 were sufficient to produce the nine cube samples.

Table 3 Mixture proportions (in grams) of the mortars used for workability compressive strength testing

Binder type	PC	70/30 PC/FA	70/30 PC/ISSA
Water	310	310	310
PC	620	434	434
Fly ash	–	186	–
ISSA	–	–	186
Andesite sand	2089	2027	2025

After mixing, the workability of each mortar was assessed using a standard flow table test (ASTM 2007). The mortar flow is expressed as a percentage increase in the diameter of the mortar sample over the original moulded diameter, after 25 drops of the flow table. Thereafter, nine 50 mm cube samples were prepared of each mortar mixture for compressive strength testing at 3, 7 and 28 days after casting (three samples per test age) to assess the rate and extent of strength development of the mortars over the first four weeks after mixing. The cube samples in their moulds were stored under plastic sheeting in the laboratory and were de-moulded the following day. They were then cured in water at 22 ± 2°C until the time of testing. Compressive strength tests were conducted on a Tinius-Olsen machine with a capacity of 600 kN, at a loading rate of 15 MPa/minute.

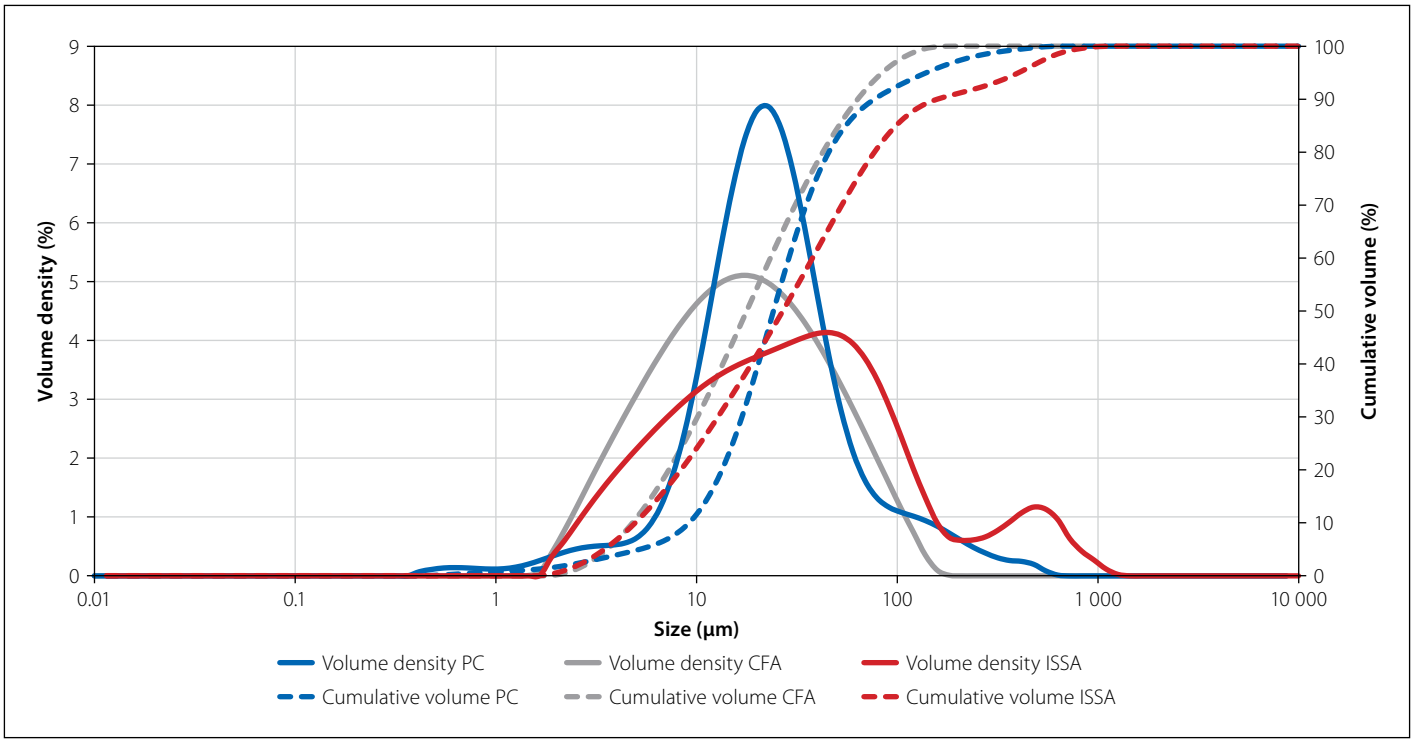

Figure 4 Particle size distributions of the PC, FA and ISSA

RESULTS AND DISCUSSION

Particle size distribution

Figure 4 shows the particles size distributions of the PC, FA and ground ISSA used in this study. FA particles were in the range of 1.7 µm to 180 µm, while ISSA particles ranged from 1.7 µm to 180 µm, with a small cluster of particles in the size range 180 µm to 1250 µm. It is thought that this is a cluster of particles that are harder and more difficult to grind and that, at this size range, is unlikely to contribute significantly to pozzolanic activity of the ISSA. The size distribution of the PC seems unusually large, as it ranges from 0.48 µm to 630 µm, when the maximum particle size for cement is typically 100 µm (Osbaeck & Johansen 1989). It is likely that some agglomeration of particles occurred during the analysis.

The results in Figure 4 were used to determine the median particle size, or D_{50} value, for the three materials as follows:

- FA: D_{50} = 17.37 µm
- PC: D_{50} = 24.60 µm
- ISSA: D_{50} = 28.3 µm

The particle size distribution of the ISSA is skewed to the coarse side and is generally coarser than the FA. This may well influence the observable reactivity of the ISSA and further studies will be needed to consider the effects of fineness of grinding of ISSA. In other studies, on ISSA, the material was ground to maximum particle sizes that ranged from 600 µm to 100 µm (Halliday *et al* 2012; Garcés *et al* 2008; Coutand *et al* 2006).

Atomic absorption spectroscopy (AAS) – elemental analysis

Figure 5 shows the AAS results obtained from an elemental analysis of the DSS and the nine ISSA samples. The results show that DSS has a significant amount of the

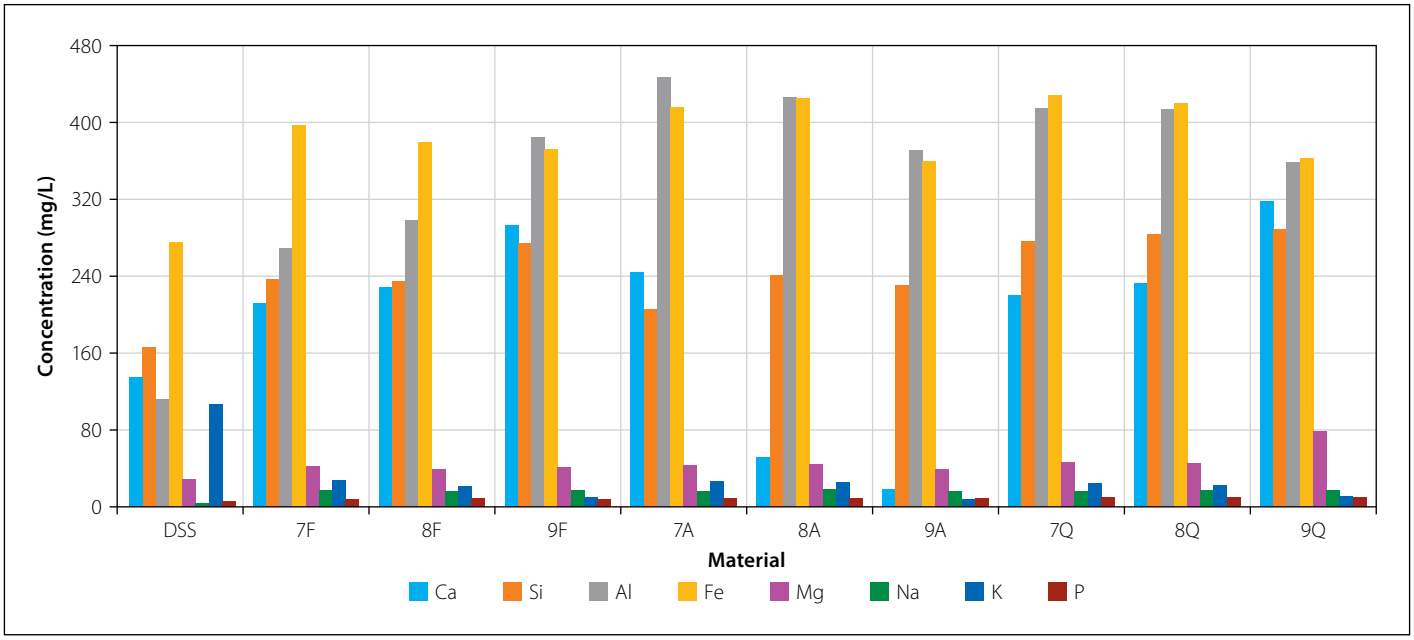

Figure 5 Atomic absorption spectroscopy results of DSS and the nine ISSA samples subjected to different pyro-processing regimes

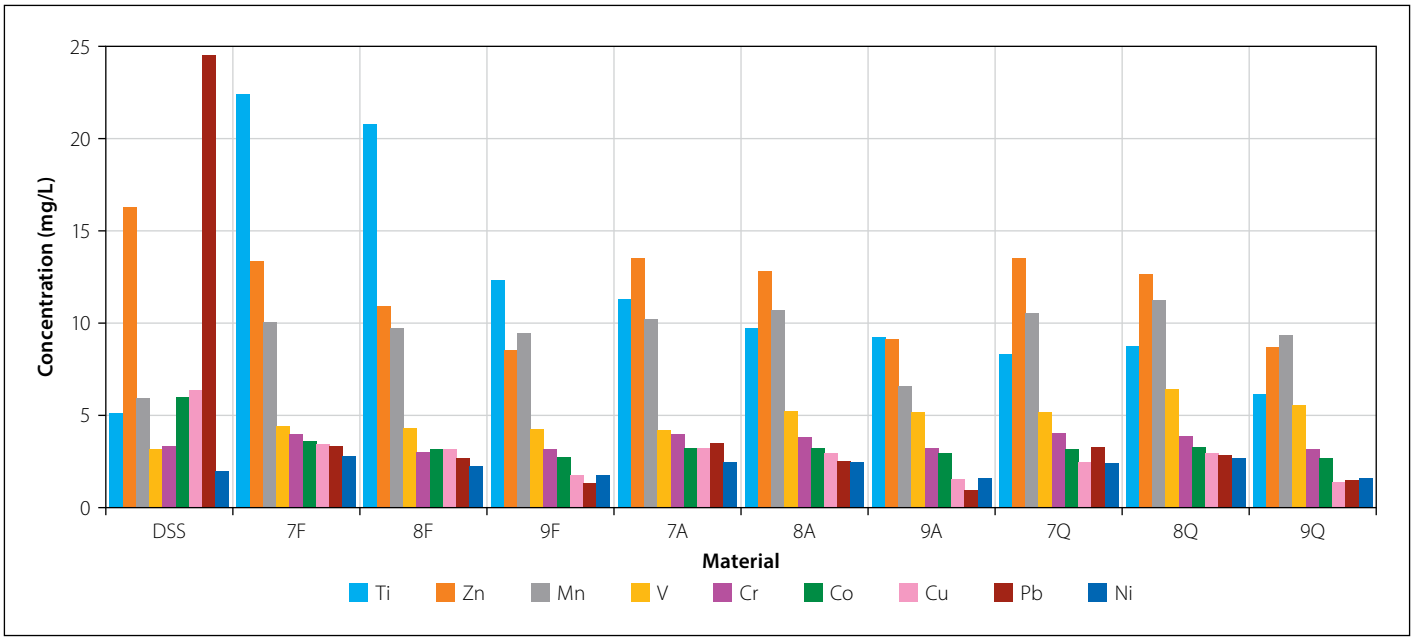

Figure 6 Heavy metal elements detected in DSS and ISSA

chemical elements found in PC (Ca, Si, Al, Fe, Mg and Na). As expected, incineration had a significant effect in increasing the concentration of elements in the ISSA, since water and volatile materials are removed from the sample during heating. Similar results were noted in previous studies (Halliday *et al* 2012; Chen & Lin 2009; Coutand *et al* 2006), varying in relation to the composition of the DSS used. Also, the lowest concentrations were noted for the furnace-cooled samples, while the air-cooled and quenched samples showed similar concentration results. It is possible that this is the result of morphological changes that occur during cooling, as this may be influenced by the cooling rate of the ash.

Potassium and phosphorus were also detected in small amounts in both the DSS and the ISSA samples. The potassium concentration was reduced as a result of incineration, whereas the phosphorus content showed a slight increase after incineration.

In consideration of the toxicity and pollution potential of DSS and ISSA, AAS was also used to detect the presence of the following heavy metals: lead (Pb), chromium (Cr), copper (Cu), nickel (Ni) zinc (Zn), cobalt (Co), Titanium (Ti), vanadium (V) and manganese (Mn). These results are shown in Figure 6.

While the concentrations of heavy metals in the samples are much smaller than those of the elements shown in Figure 5, they are large enough to warrant concern. Notable increases in concentrations of Ti and Mn were observed after incineration. The concentrations of Ni, V and Cr were largely unaffected by incineration.

After incineration, there were significant reductions in Pb, Zn, Cu, and Co concentrations because these metals are volatilised as the temperature approaches their boiling points. The presence of heavy metals in DSS tends to limit its industrial application, and the possibility of binding or encapsulating the elements makes its use in concrete attractive. However, releasing some of the elemental content into the atmosphere presents a serious concern and points to the need for active removal of

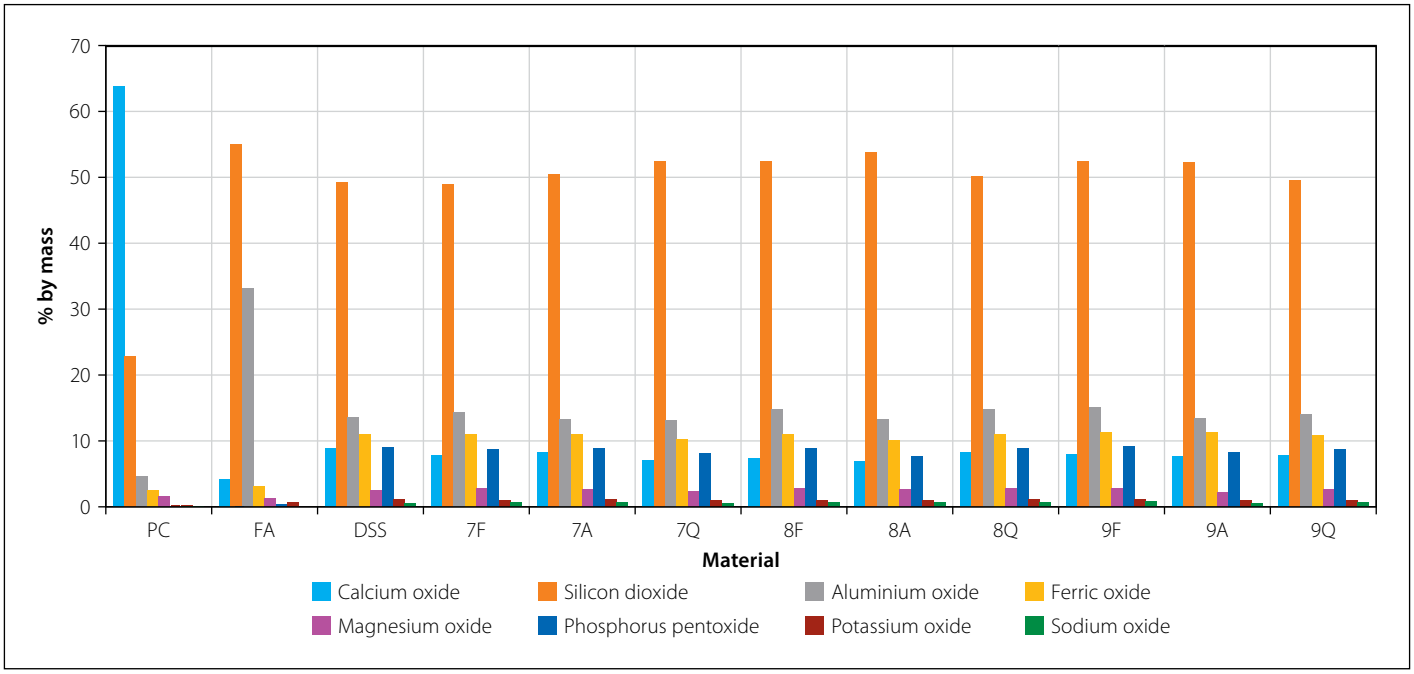

Figure 7 XRF results of PC and FA compared to DSS and ISSA materials

these elements from the flue gasses during incineration to reduce the impact of such metals on the environment.

X-ray fluorescence – oxide compound analysis

The compositions of the ISSA samples used in this study were determined using XRF analysis and are compared to the reference PC and FA as determined by Ballim & Graham (2009) in Figure 7. The principal compounds in the ISSA samples were SiO_2 (49.08% to 53.88%), CaO (6.89% to 8.94%), Al_2O_3 (13.26% to 15.13%) and Fe_2O_3 (10.19% to 11.38%). The CaO/SiO_2 ratio of the ISSA materials ranges from 0.12 to 0.16, which is slightly higher than that of the FA (0.08), indicating that ISSA may be suitable for use amended as an SCM to partially replace PC. The significant presence of Al_2O_3, Fe_2O_3 and CaO can be linked to the use of ferric salts, aluminum and lime as flocculators in the secondary sludge conditioning at wastewater treatment plants (Halliday *et al* 2012; Tantawy *et al* 2012), as well as the oxidation of some of the elements on exposure to air.

In general, incineration had a minor impact on the component composition of the DSS. The ISSA samples that were burned at 800°C and 900°C revealed the most significant, albeit modest, increases in chemical concentrations. However, no clear relation exists between the compound composition of ISSA materials and the incineration temperature or cooling rates used. It should also be noted that the DSS and all the ISSA materials contain a significant amount of P_2O_5 (phosphorus pentoxide), varying from 7.74% to 9.24%. This may be attributed to the domestic detergents either in the waste or those used at the wastewater treatment plant. Also, although not noted on Figure 7, LOI from the XRF analyses of the ISSA materials was in the range of 1.16% to 5.90%, increasing with decreasing incineration temperature of the original ashes, and this was noted for all the cooling methods.

Morphology and composition of ISSA

Figures 8 to 10 show the morphology of ISSA incinerated at 800°C and cooled in the furnace (Figure 8), in air (Figure 9) and quenched (Figure 10) after incineration. These figures also show the EDS trace analyses of the grains indicated. The results show that ISSA has an irregular morphology and size of grains with well-developed surfaces and a few traces of platy, rectangular or near-spherical grains. These results are similar to research findings on ISSA in other parts of the world (Monzó *et al* 1996; Tantawy *et al* 2012; Naamane *et al* 2016). Fine particles were also observed in all ashes incinerated at different temperatures, and these often appear to adhere to the surfaces of larger particles (see particle C in Figure 8, particle A in Figure 9, and particles A and B in Figure 10). Some platy or more prismatic particles with well-defined edges were noted in the samples (see particle B in Figure 8 and particle C in Figure 9).

EDS traces of the particles shown on the SEM micrographs indicate high peaks of silicon (Si) as the main constituent of the particles assessed, with some portions of the ISSA containing aluminum (Al), calcium (Ca), phosphorus (P) and iron (Fe). These results correlate with the

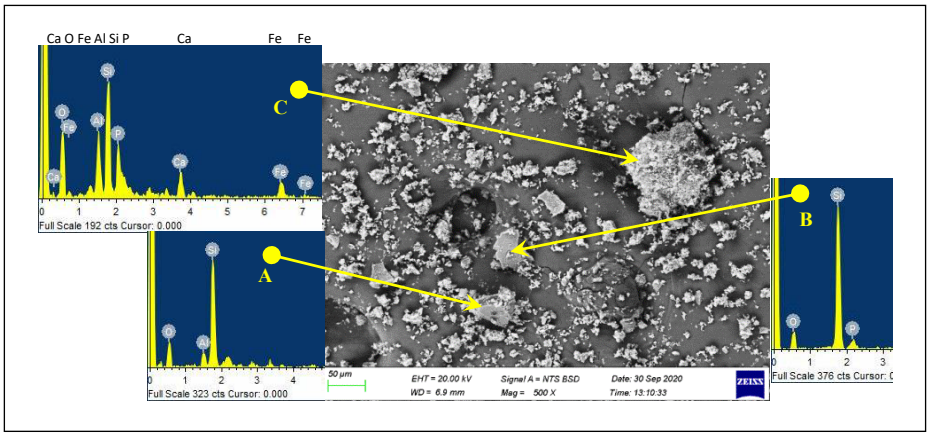

Figure 8 Micrographs and EDS of ISSA incinerated at 800°C and furnace-cooled (8F – element identification indicated on top left EDS for clarity)

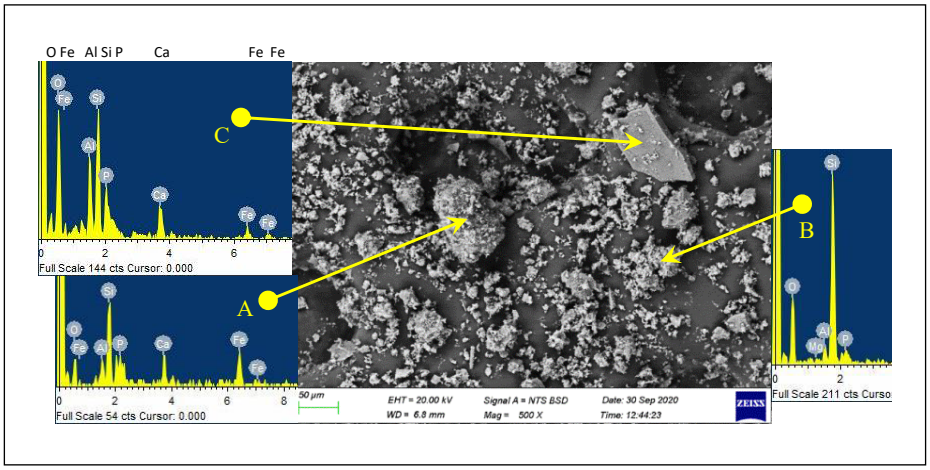

Figure 9 Micrographs and EDS of ISSA incinerated at 800°C and air-cooled (8A – element identification indicated on top left EDS for clarity)

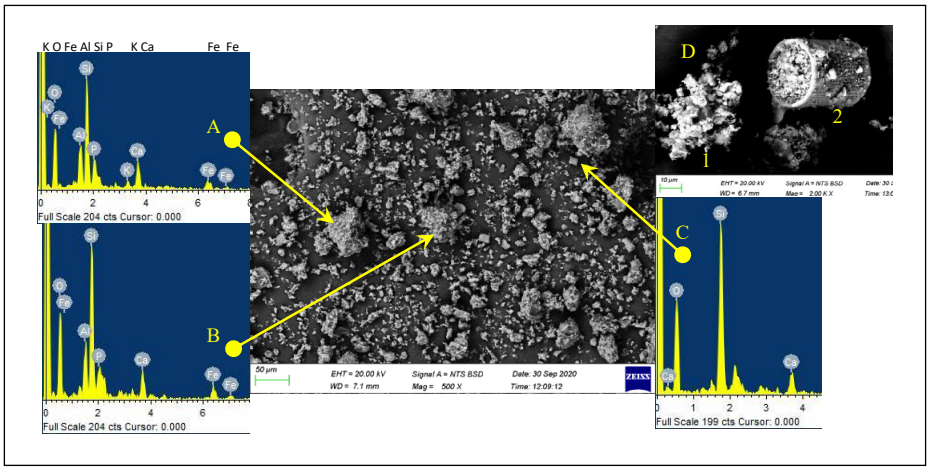

Figure 10 Micrographs and EDS of ISSA incinerated at 800°C and quenched in water (8Q)

composition of the ISSA noted with the AAS and XRF results.

Traces of diatomite (or diatomaceous earth) were also noted in the ISSA samples (indicated as particle C in Figure 10). This was confirmed by both the tubular shape of the particles as well as the EDS elemental analysis. Diatomite is a low-cost material with a variety of properties commonly used as an adsorbent in wastewater treatment plants (Zhang *et al* 2009). A closer view of a cylindrical diatomite particle is shown as particle 2 in Figure 10 D, and the EDS analysis indicates that it is largely composed of silica.

Particle 1 in Figure 10 D illustrates the irregular morphology of ISSA, more characteristic of the quenched sample. Closer views of the morphology of the differently cooled ISSA samples are shown in Figure 11. This figure shows that the rate of cooling affects the particle morphology of the ISSA, with faster cooling rates producing more particles with a darker shade of grey (denser) and that are generally smaller and more nodular, rather than platy or layered. Higher cooling rates produce more glassy or amorphous forms of silica, which encourage pozzolanic reactions in combination with PC. These results are similar to previous research findings by Monzó *et al* (2003), Tantawy *et al* (2012), Horiguchi *et al* (2011) and Naamane *et al* (2016).

Microscopic assessment of hydrated PC/ISSA pastes

Figure 12 shows selected micrographs of hydrating PC and PC/ISSA pastes. Hydration products deposited on the surfaces of particles were identified by their morphology and chemical composition based on EDS analysis. At six hours after mixing, the PC paste (Figure 12(a)) showed typical deposits of outer calcium-silicate-hydrate (CSH), calcium hydroxide or Portlandite (CH) and needles of ettringite, attached to the PC grains. In the case of the ISSA pastes, a potassium gel deposit was observed in the 8F and 8A paste samples at six hours after mixing (see Figure 12(b)). This gel has a solid morphology and appears to have a retarding effect on hydration of the ISSA (Odler & Wonnemann 1983), as there was little evidence of hydration products on ISSA surfaces in these samples at this early stage of hydration. Importantly, the gel was not observed in the 8Q ISSA-blended cement paste and there was some evidence of fibrous hydration products on ISSA particles in the 8Q sample, which may be linked to early hydration of ISSA materials in the paste.

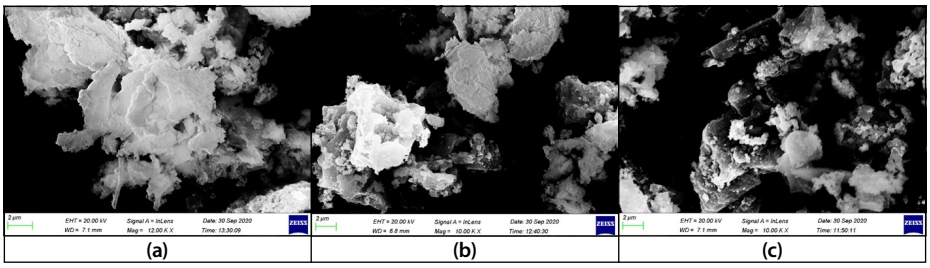

Figure 11 Micrographs of ISSA incinerated at 800°C, then (a) furnace-cooled, (b) air-cooled and (c) quenched in water

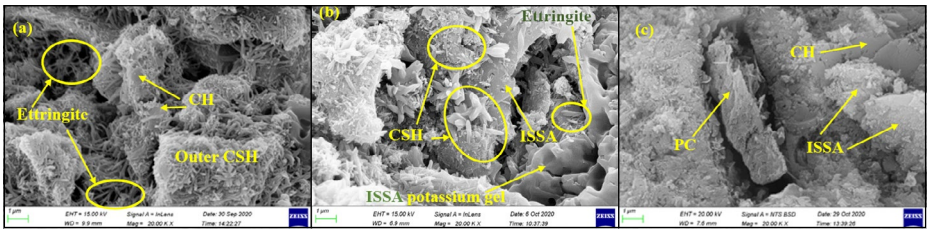

Figure 12 Samples of SEM images of hydrating binder pastes: (a) PC paste at six hours, (b) paste with 30% 8F ISSA at six hours, (c) paste with 30% 8Q ISSA at seven days

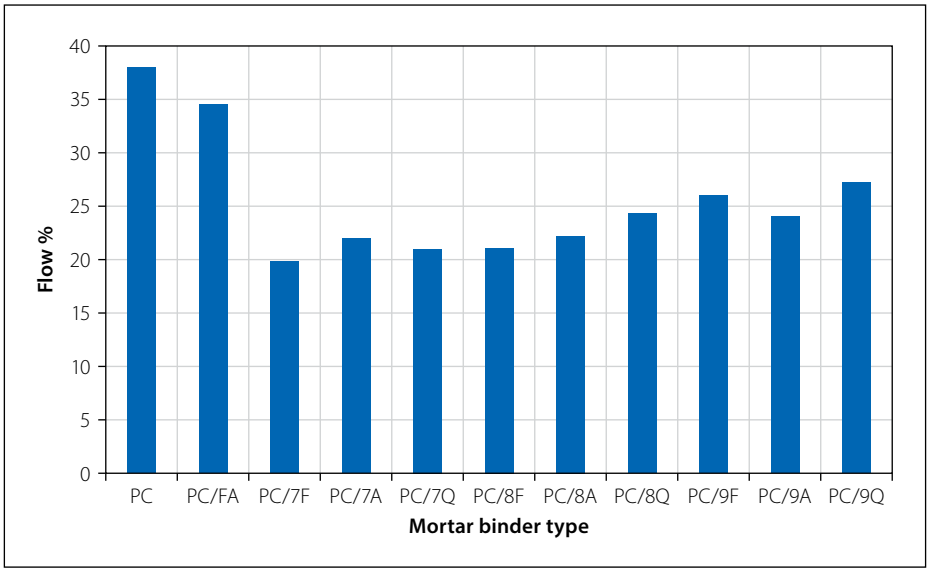

Figure 13 Effect of ISSA materials on workability of mortars when included at 30% replacement of PC in the binder, compared with equivalent replacement of FA and plain PC mortars

At seven days after mixing, there was little evidence of the potassium gel in any of the ISSA-blended pastes. All the samples showed some evidence of hydration products on the ISSA particles (see Figure 12(c)), but it was evident that the post-incineration cooling rate had an influence on the extent of hydration. The quenched ISSA sample generally showed a greater degree of hydration than the furnace-cooled or air-cooled ISSA samples.

Workability of mortars containing ISSA

The results of workability measurements conducted on each of the mortars prepared are shown in Figure 13. While the 30% FA blend caused a slight decrease in the flow of the mortar, compared to that of the plain PC mortar, all the 30% ISSA blends resulted in a significant reduction of workability. This reduction ranged from 11 percentage points for the 9Q ISSA to 18 percentage points for the 7F ISSA. Again, these results are similar to that noted by other researchers (Vouk *et al* 2016; Naamane *et al* 2016; Pan *et al* 2003; Monzó *et al* 1996). Importantly, the quenched ISSA samples generally showed the smallest reduction in workability compared with that of the furnace-cooled and air-cooled samples.

The reduction in mortar workability is attributed mainly to the irregular morphology of ISSA particles. However, the possible increased water absorption of the ISSA materials may also be a factor contributing

to this reduced workability. Vouk *et al* (2016) and Naamane *et al* (2016) report that an increased water absorption of the ISSA simultaneously resulted in longer setting times, and reduced the mortar workability due to the high amounts of phosphorus pentoxide (P_2O_5) and sulphur trioxide (SO_3) found in the ISSA.

Compressive strength test results

Figure 14 shows the compressive strength development of all mortar samples after 3, 7 and 28 days, each result being the average of three cube strength tests. The results show that, at a PC replacement level of 30%:

- All the ISSA materials used as an SCM caused a reduction in the measured compressive strength at all ages up to 28 days, relative to that of the mortar made with plain PC.
- For the mortars with ISSA materials incinerated at 700°C and at 800°C, the compressive strengths were significantly lower than that of the mortar with FA as SCM. However, the mortars with ISSA incinerated at 900°C showed compressive strengths that were similar to that of the FA mortar.
- The incineration temperature of the ISSA has a significant effect on mortar compressive strength. A higher cooling rate after incineration generally has a positive effect on compressive strength. For the ISSA materials incinerated at 800°C and at 900°C, the quenched samples showed marginally higher strengths than the furnace-cooled and air-cooled materials. This increased performance of the quenched ISSA may be explained by the fact that rapid cooling of the ash suppresses the formation of crystalline silica, resulting in a higher proportion of amorphous silica which is more favourable for the pozzolanic reactions in an SCM.
- Table 4 shows an assessment of the rates of early strength development for the mortars assessed. It is clear that the ISSA materials have significantly reduced the rate of strength gain at three days, to a greater extent than that caused by the addition of FA. However, there is some acceleration in strength gain thereafter and, for the period 7 to 28 days, the rate of strength gain is similar to that obtained with the PC/FA mortar samples. Importantly, all the blended cement mortars showed lower rates of strength gain between 7 and 28 days when compared with the plain PC mortar.

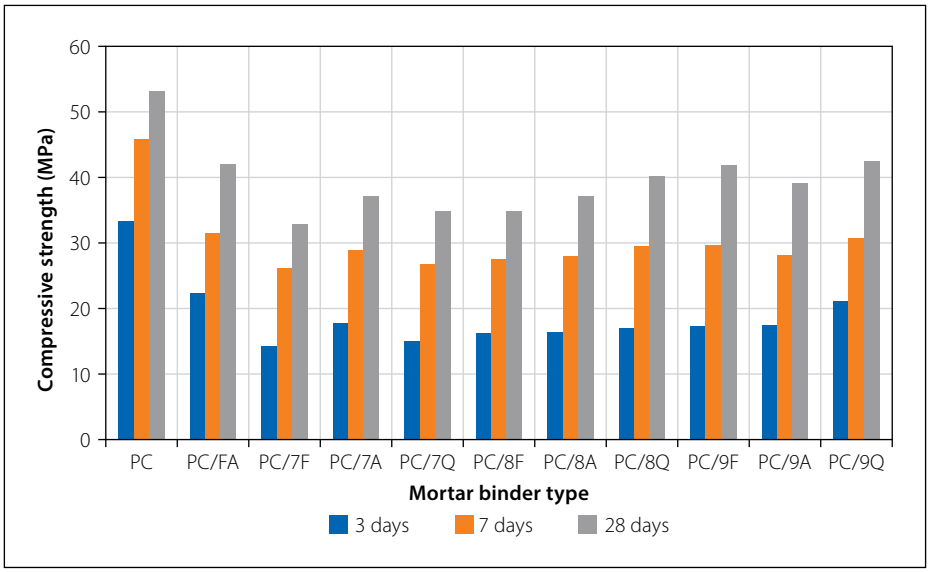

Figure 14 Compressive strength test results of mortar samples after 3, 7 and 28 days of hydration

The mortar compressive strength results shown in Figure 14 support the observations made in the SEM study of the hydrated pastes (Figure 12) that the ISSA materials do show evidence of pozzolanic activity when used as an SCM with PC. For the ISSA incinerated at 900°C, the 28-day compressive strength performance was similar to that of the FA mortar.

Of course, a factor not accounted for in this analysis was the fineness of grinding of the ISSA. Halliday *et al* (2012) showed that coarser ISSA tends to contribute to the reduction in compressive strength. The particle size distributions shown in Figure 4 indicate that the ISSA used in this study was coarser than both the PC and the FA. It is therefore possible that higher levels of pozzolanicity may be realised with more finely ground ISSA.

Manjunatha (2017) found a lower pozzolanic activity for ISSA than for FA. However, the present study has shown that under suitable pyro-processing regimes, ISSA can show similar strength performance as FA when used as an SCM. The differences in these findings may well be related to the chemical nature of the FA and the ISSA, as well as the physical processing of the ISSA after cooling.

Table 4 Rates of early mortar strength gain relative to the 28-day strength of the binder combinations assessed

Mortar binder type	3-day to 28-day strength (%)	7-day to 28-day strength (%)
PC	62.6	86.3
PC/FA	53.3	75.0
PC/7F	43.4	79.5
PC/7A	47.8	77.9
PC/7Q	43.2	77.1
PC/8F	46.6	79.2
PC/8A	44.2	75.4
PC/8Q	42.3	73.3
PC/9F	41.5	70.9
PC/9A	44.7	71.7
PC/9Q	49.7	72.2

CONCLUSIONS

This study was aimed at considering the possibility that incinerated sewage sludge ash could be used as a supplementary cementitious material in combination with Portland cement. The main conclusions to be drawn from the results are:

- The SEM assessment of paste samples, together with the mortar compressive strength results, indicates that the ISSA used in this study has pozzolanic capacity and may be used as an SCM in cement-based materials.
- DSS and ISSA consists mainly of Si, Ca, Al, Fe, Mg, Na and K, the important elements in cementitious and pozzolanic materials used in concrete. The concentrations of these elements increase with incineration and produces an ISSA with a chemical profile similar to that of FA in terms of oxide composition.
- Heavy elements that may also be toxic such as Zn, Pb, Cu, Ti, Cr, Mn, V and Ni are present as minor constituents. The amounts of these heavy elements are reduced by incineration and there may be environmental benefits to

incorporating such elements in concrete rather than in land disposal systems. However, removal of these elements from the flue gasses is also important to avoid release into the atmosphere.

- Both the maximum temperature of incineration and the subsequent cooling rate of ISSA have a significant influence on the workability, hydration and strength development of pastes and mortars made with blends of PC and ISSA. In general, increasing the incineration temperature of ISSA from 700ºC to 900ºC, together with rapid cooling by quenching in water, produced better early hydration, better workability and higher mortar strength when the ISSA is used as an SCM at 30% replacement of PC. The ISSA incinerated at 900ºC and quenched in water produced 28-day mortar strengths that are similar to that obtained with mortars made with FA at the same replacement level.
- When used as a PC replacement in paste samples, the ISSA materials that were incinerated at 700ºC and 800ºC showed deposits of potassium gel after six hours of hydration that appear to inhibit early hydration of the ISSA. This gel was not evident at later ages of hydration. In the case of the ISSA incinerated at 900ºC, the gel was not noted at any stage of assessment of hydration. This aspect of the hydration behaviour of ISSA requires further assessment to understand its implications for the use of these materials in cement and concrete.

ACKNOWLEDGEMENTS

The authors are thankful to SASOL for the financial support provided to complete the work, and to AfriSam (Pty) Ltd for their assistance with some of the chemical analyses reported in this work.

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