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CHARACTERIZATION OF SEWAGE SLUDGE ASH AS AFFECTED BY DIFFERENT INCINERATION TEMPERATURE AND TIME

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ABSTRACT

Sewage sludge is a public environmental concern. Incineration of sewage sludge significantly reduces its volume, resulting in a powder material (i.e., sewage sludge ash (SSA)). This study investigates the effect of different incineration temperature and time on the characteristics of the produced SSA. The raw sewage sludge was incinerated at different temperatures (700 ^{o}C, 900 ^{o}C, and 1100 ^{o}C) for 2 and 4 hours. The chemical and mineral compositions and morphology were assessed using X-ray fluorescence, X-ray diffraction, and scanning electron microscopy, respectively. The pozzolanic activity of SSA particles was evaluated using Frattini and strength activity index tests. Also, the effect of incorporating SSA as a partial replacement on cement paste workability and workability retention was examined. Test results showed that SSA exhibits some pozzolanic properties, suggesting the feasibility of using it as a partial replacement of ordinary Portland cement in making sustainable and environmentally-friendly concrete.
Keywords: Sewage sludge ash, incineration temperature, Frattini activity, pozzolanic properties, ordinary portland cement.

INTRODUCTION

Due to the current innovations and development worldwide, the rate of producing solid waste has increased considerably. Many research studies are being directed to investigate the influence of waste materials on the environment and also on examining potential applications for recycling and reusing such waste. The improving economic, social and environmental indicators of sustainable development are attractive to the construction industry, which is a globally emerging sector, and an extremely active industry in both developed and developing countries (Ortiz et al., 2009). Concrete is the most used building material worldwide, and several billions of cubic meters are produced annually. Furthermore, cement industry, which is a main ingredient of concrete, has always been among the largest CO_2 emission industries (Benhelal et al., 2013; Chindaprasit et al., 2007; Pacheco-Torgal et al., 2012). As a result, the use of waste in concrete-making became the main focus of many investigators. In the UAE, the growing development resulted in the production of large amounts of sewage sludge. More than one million ton (on a dry-weight basis) of sewage sludge is produced yearly (Alshankiti.et al. 2018). Most of the produced sewage sludge is dumped in the landfills, causing environmental problems aside from occupying a large area of the landfill. Incineration of sewage sludge reduces the volume of the produced sewage sludge by converting it to a small stabilized ash known as sewage sludge ash (SSA) achieving up to 80–90% volume reduction (Yusuf et al., 2012). However, the disposal of SSA in landfills is not eliminated. It was reported that the main oxides composing SSA are SiO_2, CaO, Al_2O_3, and P_2O_5. Oxides such as Fe_2O_3, Na_2O, MgO, P_2O, SO_3, and others are present in lesser amounts (Lynn et al., 2015; Paris et al., 2016). Besides, SSA was found to include heavy metals (i.e., zinc (Zn), lead (Pb), copper (Cu), cadmium (Cd), chromium (Cr), arsenic (As), and nickel (Ni)) (Lin et al., 2006; Wang et al., 2008). Although these metals are present in very small amounts, the leaching of such metals is an important issue if SSA will be considered for use in the construction industry. Previous researches have revealed that sewage sludge ash may be used as to substitute cement in mortars and concrete (Lin and Lin, 2005; Yusuf et al., 2012) Monzó et al. (1996) and Ingunza et. al (2018) concluded that the incinerated SSA could replace part of cement in mortar as long as that level of replacement was below 20%. Pan et al. (2003), Donatello et al. (2010) and Kappel et al. (2017) investigated the effect of grinding SSA on the performance of cement mortars including SSA as partial cement replacement. It was concluded that increasing the SSA fineness improved the mechanical strengths and workability of mortars. Few investigators studied the effect of sintering temperature on the characteristics of produced SSA (Lin et al., 2006; Xu et al., 2008; Wang et al., 2008). This study examines the characteristics of the produced SSA as affected by incineration temperature and time for the possibility of using SSA in making concrete mixtures. Also, the effect of using SSA incinerated at different temperatures and time on the compressive strength of cement mortar, and the workability and workability retention of cement paste was evaluated.

EXPERIMENTAL PROCEDURES

Materials

In this study, municipal sewage sludge was collected from Al Saad Wastewater Treatment Plant in Al Ain, UAE. Raw sewage sludge was homogenized before incineration. Sewage sludge was incinerated using a programmable electrical furnace at three different temperature and for two different periods as summarized in Table 1.

The produced granular particles of the incinerated SSA particles were ground using a disc-type grinder to reduce particle size and increase its fineness. A total of six samples of finely ground SSA were obtained. For mortar and paste preparation, Portland cement conforming to the specifications of ASTM C150 (2019) Type I was used. Natural crushed stone sand with a specific gravity of 2.65 and a single size (i.e., passing 1.18 mm and retained on 0.6 mm sieves) was used.

Table 1: Incineration temperature and time for sewage sludge samples

Sample I.D.	Incineration Temp. (^{o}C)	Incineration Duration (hrs.)
RSS	Raw sewage sludge	
SSA7-2	700	2
SSA9-2	900	2
SSA11-2	1100	2
SSA7-4	700	4
SSA9-4	900	4
SSA11-4	1100	4

Experimental Methods

The chemical and mineral composition of the SSA samples was assessed using X-ray fluorescence (XRF) and X-ray diffraction (XRD), respectively. The major elements of the SSA samples were found by X-ray fluorescence (XRF) spectrometer using powder samples. The X-ray diffraction (XRD) analysis was carried out using a PANalytical X'Pert PRO powder diffractometer operated at 45 kV and 40 mA, with a copper (Cu) anode ($\lambda(K\alpha1)$ =1.5406, $\lambda(K\alpha2)$ =1.5444). Scans were recorded for 2θ from 20 to 60°. The particles morphology of the SSA samples were examined using a Scanning Electron Microscope (SEM).

The pozzolanic activity of the SSA samples was evaluated by the Frattini test and the strength activity index (SAI) test. The Frattini test was conducted according to the procedures described in the BS EN 196-5 (2011). The control specimen was prepared with 100% ordinary Portland cement (OPC). The tested specimens were composed of 80% OPC and 20% by mass of SSA. The specimens were then stored in sealed containers at 40 ^{o}C for 8 days and 28 days. The pozzolanic activity of the SSA samples was evaluated by measuring the concentrations of CaO and OH^- and comparing that with the saturation isotherm of calcium hydroxide at the same temperature. The SAI test was carried out on cement mortar mixtures using cube molds (50x50x50 mm) according to the ASTM C311 (2019). Control mortar was prepared by using 100% Portland cement. Tested specimens were prepared by replacing 20% by mass of the OPC with SSA. The specimens were tested at 7 and 28 days. The average strength of three specimens was reported, and the index was calculated as the ratio of the compressive strength of the tested mix to the strength of the control mix at the same age.

Flow table test was performed to evaluate the workability of the cement paste with SSA replacing 20% by mass of the OPC. Control cement paste was 100% OPC. The test of mortar workability, i.e., flow table spread (FTS), was conducted according to the ASTM C1437 (2019) test method. The flow table spread (FTS %) was calculated as the increase in the average base diameter of the cement paste specimen. Furthermore, the workability retention (i.e., change of workability with time) of the control mix and the mixes including SSA was measured at different time intervals (i.e., 15, 30, 45, and 60 minutes). The paste samples were mixed for 30 seconds before each test.

RESULTS AND DISCUSSIONS

SSA Chemical, Mineral Composition and Morphology

Several aspects of mortar casting, such as hydration degree and its rate, workability, or compressive strength will be affected by the chemical compounds of the cement or SSA (Krejcirikova et al., 2019). Table 2 summaries the XRF analysis results of the raw sewage sludge and the SSA at incinerated at different temperatures and times. The major constituents of SSA were SiO_2 (11.4–31.2%), CaO (10.6–23.5%), and P_2O_5 (9.2–21.3%). The next most abundant components were Al_2O_3 (4.2–9.7%), Fe_2O_3 (3.5–6.4%), MgO (3.41–7.2%), SO_3 (5.6 –0.3%), and K_2O (0.5–1.2%). Furthermore, it was notable that increasing the incineration temperature and duration resulted in higher SiO_2 content. The highest SiO_2 content (31.2%) was found when SSA was incinerated at 1100 ^oC for 4 hours. Cyr et al. (2007) reported that the CaO content in SSAs ranges from 1.1% to 40.1%, so all the SSA samples examined here were within the average range of other SSAs. The CaO content in SSA may be considered high enough to provide pozzolanic properties to the ash, hence supporting their feasibility to be used as a partial substitute of cement (Pekmezci and Akyüz, 2004). Hewlett (2004) stated that P_2O_5 is occasionally present in small amounts in the raw materials of cement manufacture and it passes into the clinker. If present in quantities of 1–2% in Portland cement clinker it slows the rate of hardening of the cement. Since, P_2O_5 one of the main components of SSA particles, the addition of SSA to the concrete may attribute to the increase of its setting time, hardening and so may reduce its early compressive strength. The mineralogical configuration of SSA was investigated using X-Ray diffraction (XRD) analysis. The XRD results illustrated in Figure 1 noted the predominant peaks at 2θ value 26.5^o for quartz (SiO_2). This verified the XRF results which revealed that the SiO_2 constituted the highest percentage in the SSA composition. Other minerals were detected including Mullite $Al(Al_{1.272}Si_{9.728}O_{4.864})$, Quartz ($SiO_2$), Fluorspar ($CaF_2$), Calcite ($CaCO_3$), Whitlockite ($Ca_7Mg_2P_6O_{24}$) and Dolomite ($CaMgCO_3$). The amount of quartz increased when SSA was incinerated at 1100 ^oC for 4 hours. Additionally, the presence of humps in the range between 2θ values of 20 and 30^o as well as the unleveled graph trend from 2θ values between 0 to 40^o indicated the occurrence of some amorphous phases in SSA materials which as mentioned earlier, will play a major role in increasing the tendency of SSA to exhibit pozzolanic activities and the potential of SSA to be used as a partial substitute of cement. Figure 2 shows the microstructure of the incinerated SSA. It is observable that, SSA particles were irregular with rough surfaces confirming the previous report by Donatello and Cheeseman, (2013). Moreover, Figure 2 clearly showed that the pore structure of the particles became more compact as a result of increasing the incineration temperature from 700 ^{o}C to 1100 ^{o}C. This might be due to expansion or bloating of materials at high temperature, and this is thought to be due to gas evolving decomposition. Also, it was noticeable from Figure 2 (e) the existing of some spherical particles with some fussed material on the surface. To further understand the composition of the fused material on the surface, an EDS (i.e., energy dispersive spectrometer) analysis was carried out. It was indicated the presence of heavy metals including zinc (Zn), lead (Pb), copper (Cu), nickel (Ni). cadmium (Cd), chromium (Cr), and arsenic (As).

Table 2: XRF analysis of sewage sludge ash

Oxide	RSS	SSA7-2	SSA9-2	SSA11-2	SSA7-4	SSA9-4	SSA11-4
SiO_2	11.44	26.57	25.15	30.52	29.19	27.74	31.23
CaO	10.59	22.69	23.53	23.20	21.78	22.66	23.07
P_2O_5	9.26	20.28	21.29	20.01	19.09	20.20	19.81
SO_3	5.61	4.99	4.54	0.47	4.95	4.22	0.30
Al_2O_3	4.22	9.30	9.63	9.74	9.10	9.54	9.54
Fe_2O_3	3.53	6.17	6.17	6.41	5.99	6.01	6.36
MgO	3.43	7.13	7.02	7.12	7.15	6.95	7.22
K_2O	0.53	1.24	1.20	1.18	1.23	1.20	1.14
TiO_2	0.34	0.70	0.70	0.74	0.67	0.68	0.72
ZnO	0.16	31.80	0.25	0.07	0.30	0.30	0.07
SrO	0.07	0.16	0.15	0.16	0.15	0.15	0.16
Br	0.07	0.04	0.01	0.00	0.03	0.01	0.00
MnO	0.06	0.13	0.13	0.13	0.12	0.12	0.13
CuO	0.04	0.11	0.10	0.09	0.10	0.11	0.09
Cr_2O_3	0.03	0.07	0.07	0.08	0.07	0.06	0.08
NiO	0.02	0.04	0.04	0.04	0.00	0.04	0.04
ZrO_2	0.01	0.02	0.02	0.03	0.02	0.02	0.02
Sum of SiO_2, Al_2O_3 & Fe_2O_3	19.19	42.04	40.95	46.67	44.29	43.29	47.13

Frattini Test

The pozzolanic activities of the SSA samples were evaluated by the Frattini test. The [Ca^{+2}] was expressed as equivalent [CaO]. According to the BS EN 196-5 (2011), a material is regarded as pozzolanic when the test result lies beneath the lime saturation isotherm at 40 ^{o}C. However, a result lying on or above the lime saturation isotherm indicates no pozzolanic reaction. The test was conducted at 8 days and at 28 days of curing (Figure 3). It was obvious from Figure 3 that all samples did not exhibit any pozzolanic activity at 8 days, except for SSA7-4. On the opposite, at 28 days, all SSA samples lied below the lime saturation isotherm, indicating that ground SSA incinerated at different temperature and periods could meet the criterion to be a pozzolan according to the Frattini standard. Moreover, CaO removal percentage at 8 days was negative and normalized to 0% removal. However, Figure 4 indicated that all SSA samples had a considerable percentage of CaO removal at 28 days. The highest degree of [CaO] reduction was revealed for SSA9-2 followed by SSA11-2.

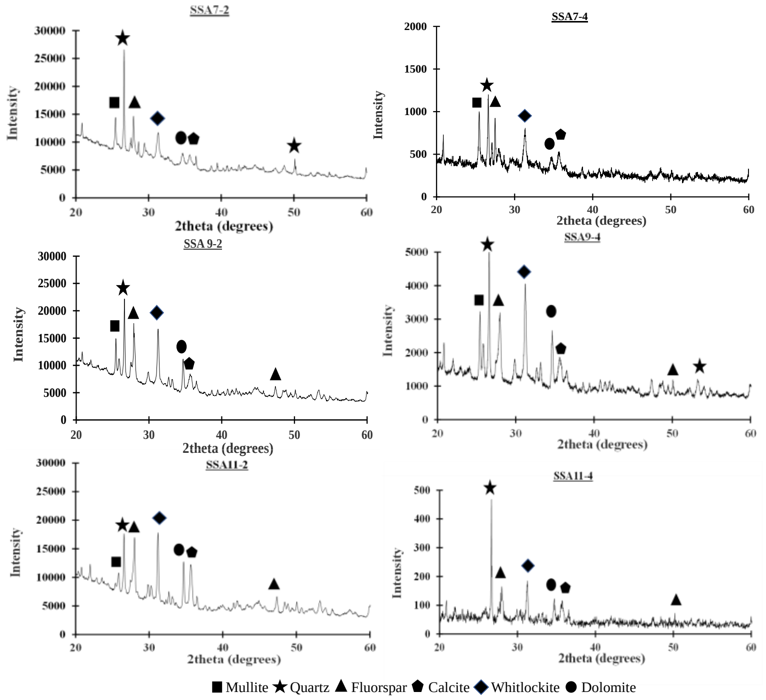

Figure 1: XRD patterns for sewage sludge ash (SSA)

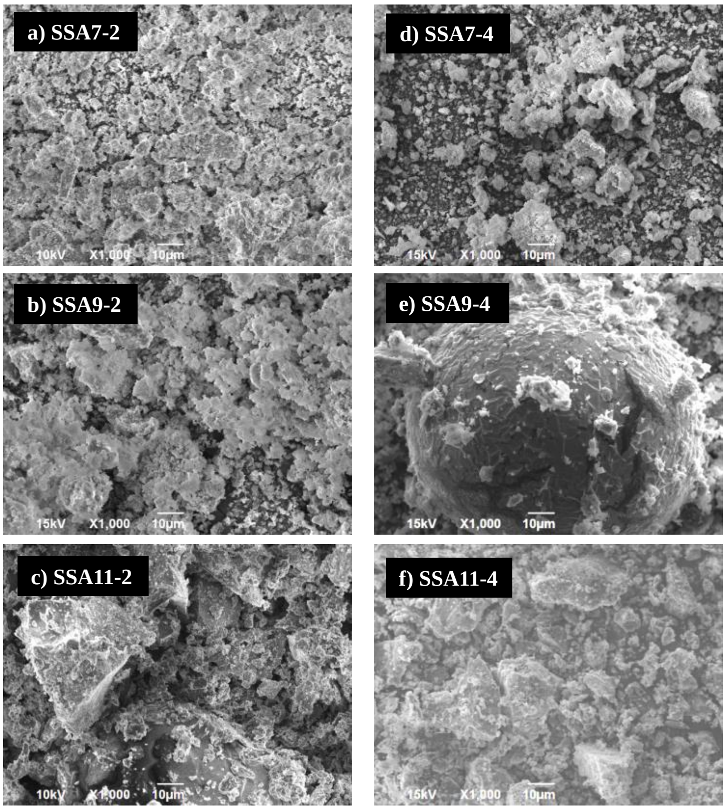

Figure 2: SEM images for SSA

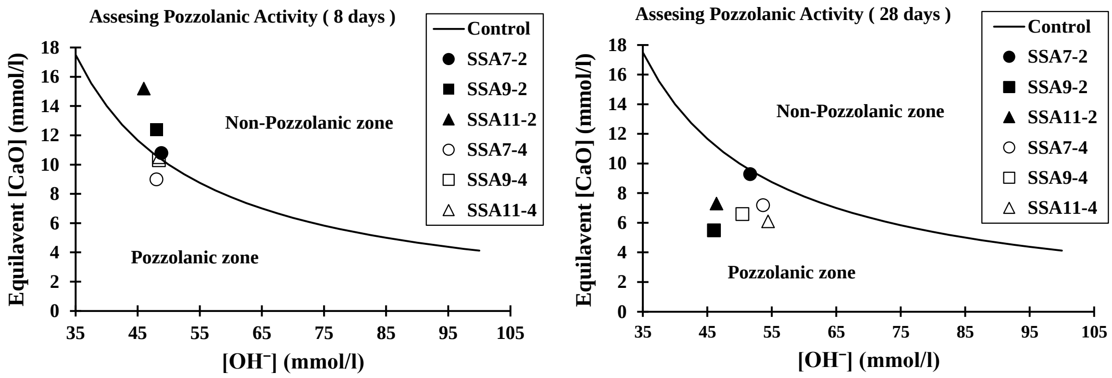

Figure 3: Frattini test results at 8 days and 28 days of curing

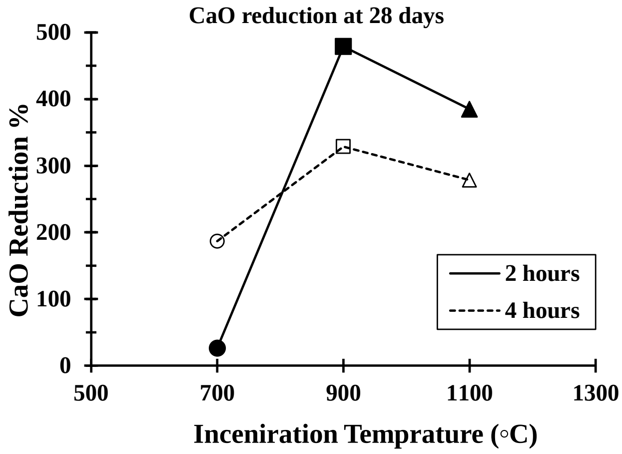

Figure 4: Percentage of CaO removal at 28 days of curing

Strength Activity Index (SAI)

The mean values of the compressive strength (Fc) of hardened SSA mortars and control mortar at 7 and 28 days of curing was measured, and results are summarized in Tables 3. The compressive strength of all mortar specimens increased when curing age extended from 7 to 28 days. The highest compressive strength was detected for mortars containing SSA particles incinerated at 700^{o} C for 2 hours. All other SSA mortars had a lower compressive strength compared to the control mix. The highest reduction of compressive strength was observed in SSA particles incinerated at 1100 ^{o}C. Test results of the SAI at 7 and 28 days of curing for the different SSA mixes are shown in Figure 5. The SAI for SSA incinerated at 1100 ^{o} C for 2 hours and 4 hours were 66.3 % and 70.5% respectively which indicated that SSA had very low pozzolanic activity (i.e., higher than 75% as per ASTM C618 (2019)). However, the SAI for all other mortars exceeded 75% which indicated that SSA could be considered as good pozzolan material. The SAI for all other SSA mortars was between 77.1% and 95.6%. This implied that SSA could react with $Ca(OH)_2$ to produce secondary C-S-H gel. Therefore, SSA can potentially be used in concrete or mortar to partially replace Portland cement. However, previous work revealed that the pozzolanic activity of SSA is lower than that of some common pozzolans such as fly ash. As confirmed by Frattini test results, SSA exhibited pozzolanic reactivity at late ages. Tay and Show (1994) found that the strength activity index (SAI) of SSA with Portland cement was between 57.6% and 67.2%. While the SAI value of fly ash class F was between 96% and 134% (Pan et al., 2002; Bhatty and Reid, (1989). Thus, the effectiveness of using SSA in the mortar was lesser than that of fly ash. Also, on using SSA, one should not expect pozzolanic activity at an early age.

Workability and Workability Retention

The effect of SSA on the workability of cement pastes was studied by the flow table test. Results are presented in Figure 6 as the percentage increase of average base diameter (i.e., flow table spread (FTS) %). It can be seen that the addition of SSA resulted in a reduction in consistency rates for almost all cement pastes containing SSA compared to the control mix. This occurred due to the irregular morphology of SSA particles and the rough surface texture of the particles as shown in the SEM images, which adsorbed part of the mixing water, lowering the workability and changing the consistency of the mortar (Monzó et al., 1996). Thus, in order to preserve the consistency of mortars, it is necessary to increase the water content of or use workability improving admixtures for mortars containing SSA as a cement replacement (Tay, 1987; Monzó et al., 1999; Monzó et al., 2003). The highest workability was achieved when SSA particles were incinerated at 1100^{o} C. Okah and Godwin (2016) reported that much of the loss of mortar workability takes place in the first 1 hour. However, SSA did not show a notable decrease in consistency in the first hour. In conclusion, the produced SSA provided improved workability retention. The insignificant decrease of the pastes workability with time could be due to the loss of the water being utilized by the cement hydration or evaporation.

Table 3: Compressive strength at 7 days and 28 days of curing

Mortar Sample	Compressive strength (MPa)*	
	7 days	28 days
Control mix	13.9 (0.04)	18.7 (0.04)
SSA7-2	14.1 (0.02)	17.9 (0.02)
SSA92	11.5 (0.01)	14.7 (0.03)
SSA11-2	9.20 (0.06)	11.6 (0.11)
SSA7-4	11.4 (0.07)	16.6 (0.11)
SSA9-4	10.6 (0.05)	14.4 (0.07)
SSA11-4	9.8 (0.03)	13.2 (0.05)

* Values in parenthesis are the standard deviation in MPa

CONCLUSIONS

Innovation in solid waste management can have economic and environmental benefits by adding value to waste products and efficiently using them in various applications. Because direct disposal of SSA into landfills is an unsatisfactory solution both from the ecological and the economic points of view, investigation of the potential use of SSA in various applications should be undertaken. As a result of development, the demand for construction materials is growing significantly. Therefore, reusing SSA as an alternative substitute in construction materials such as concrete industry brings many benefits such as efficiently reusing a solid waste instead of being dumped in landfills, and improving the sustainability of the produced concrete. The main purpose of this study was to evaluate SSA characteristics as affected by different incineration temperature and time. From the conducted experimental work the following conclusions can be drawn:

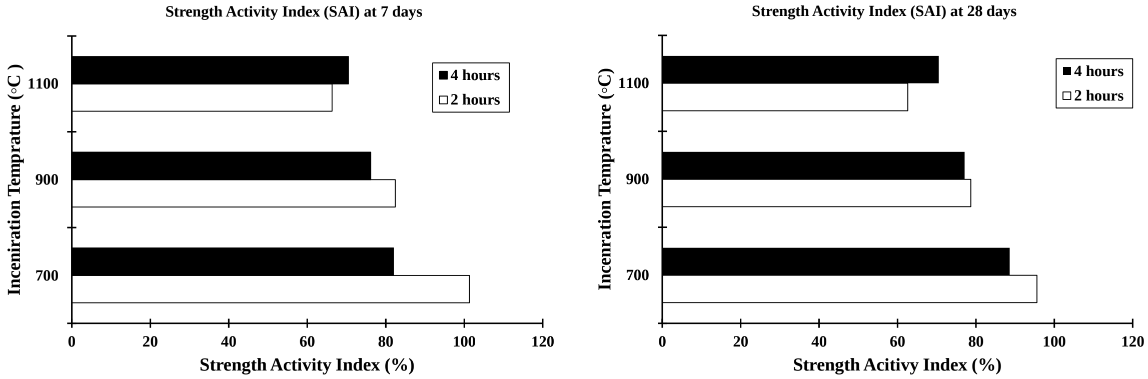

Figure 5: Strength activity index at 7 days and 28 days

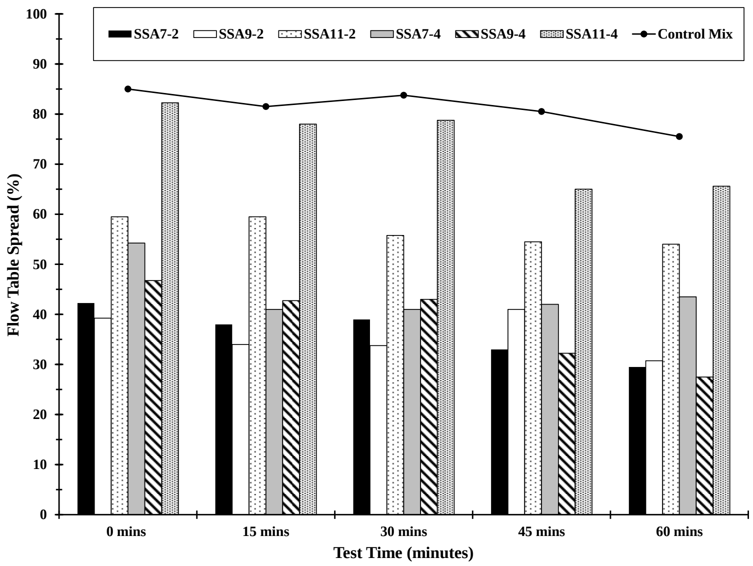

Figure 6: Workability of SSA pastes

1. The major constitutes of all SSA samples were SiO_2, CaO, and P_2O5. However, other oxides such as Al_2O_3, Fe_2O_3, MgO, SO_3, and K_2O were present in a lesser amount.
2. SSA included trace amounts of heavy metals. The leaching out of these metals should be investigated if SSA will be used in making concrete.
3. As the incineration temperature and time increases, the formation of quartz (SiO_2) increases.
4. XRD analysis indicated the occurrence of some amorphous phases in the SSA which suggested that SSA could exhibit pozzolanic activities and the potential to be used as a partial substitution of cement.
5. SEM results indicated that SSA particles were irregular with a rough surface. Nevertheless, the pore structure of the particles became more compact as a result of increasing the incineration temperature.

6. Based on the Frattini and SAI test results, all SSA samples exhibited a late pozzolanic activity, yet the highest CaO removal was observed when SSA was incinerated at 900 ^{o}C for 2 hours
7. SSA addition reduced the consistency of the mortar due to the irregular morphology and rough surface texture of SSA particles. The inclusion of SSA improved the workability retention of cement paste.

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