CHARACTERIZATION OF SEWAGE SLUDGE ASH (SSA) IN CEMENT MORTAR

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ABSTRACT

Sewage sludge ash (SSA) is a waste material obtained from the incineration of wastewater sludge. Hence, an investigation was carried out to study the potential use of SSA in cement based materials. The chemical and mineralogical characteristics of SSA in cement mortar are presented in this paper. Effect of incineration temperature and percentage of SSA as partial cement replacement in cement mortar were examined. The percentage of SSA considered in this study was 10% replacement of the mass of cement whereas the incineration temperatures investigated were at 600°C and 800°C. The tests conducted in this study consists of X-ray Diffraction (XRD), X-ray Fluorescence (XRF) and Field Emission Scanning Electronic Microscope (FESEM) as well as compressive strength test. Results show that a significant amount of SiO_2, Al_2O_3 and CaO was traced after the incineration process. Mortar samples with 10% cement replacement of 800°C burnt SSA improves the compressive strength up to 1.14% and 5.06% at the ages of 28 and 90 days, respectively. The FESEM test results show that SSA samples burnt at 600°C exhibited needle-shaped particles whereas a smooth structure was found in SSA burnt at 800°C due to the pozzolanic reaction which filled the void and pores in the mortar. This bonding also provides additional strength to the mortar where the compressive strength has increased after 28 and 90 days.

Keywords: characteristics, incineration, percentage replacement, sewage sludge ash, temperature.

INTRODUCTION

Sewage is the collection of discharge from domestic, medical, commercial and industrial establishments as well as rain water. The purpose of sewage treatment is to produce treated sewage water which is suitable for safe discharge into the environment and for re-use purposes (Tantawy *et al.* 2012; Tantawy *et al.* 2013). Sewage sludge is usually treated by several common methods which include anaerobic digestion, aerobic digestion and composting. Sewage sludge tends to accumulate heavy metals existing in the wastewater. The origin of sludge and treatment options affect the composition of sewage sludge and its content of heavy metals. In the past decades, sewage sludge was mainly disposed of in landfills and the seas. However, space limitations on existing landfills as well as increasing environmental concerns such as groundwater pollution from landfill leachate, odour emission and soil contamination have urged further investigation on various disposal methods. According to the Working document of sewage sludge, two main types of pollution by sewage sludge were identified; heavy metals and certain groups of organic compound (Rizzardini and Goi, 2014). In general, Cd, Pb and Zn represent the biggest concentrations of heavy metals in sewage sludge from domestic wastewaters which can be a source of pollution for soils. Faeces contribute 60% - 70% of the Cd, Zn, Cu and Ni load in domestic wastewater and >20% of the input of these elements in mixed wastewater from domestic and industrial premises. Other principal sources of metals in domestic wastewater include body care products, pharmaceuticals, cleaning products as well as liquid wastes.

Sewage sludge has been used in agriculture and its effect on plant and soil (Ahmed *et al.* 2010) as well as the implications on the environmental have been studied (Usman *et al.* 2012). However, the presence of heavy metals such as Zn, Cu, Ni, Cd, Pb, Hg and Cr is restricting the use of sludge for agricultural purposes. Similarly, sewage sludge in Malaysia has acidic properties and contain high amount of heavy metal (Rosenani *et al.* 2004; Roslan *et al.* 2013). Application of sewage sludge in agriculture has become difficult in Malaysia as the fertilizer quality is difficult to standardize. Application of sewage sludge without proper management may have adverse impacts on human health and the environment (Tantawy *et al.* 2012).

One of the most common sewage sludge disposal alternative is incineration (Sahu *et al.* 2013). Sewage sludge ash has been used as additive in cement based materials (Cyr *et al.* 2007; Garcés *et al.* 2008), mortar (Agrawal *et al.* 2014; Lin *et al.* 2008; Monzó *et al.* 2003), blocks (Baeza-brotons *et al.* 2014), and bricks (Cusidó and Cremades, 2012). One of the major concern of using sewage sludge ash is the content of heavy metal which can cause deterioration to the cement based material. SSA may contain high amount of sulfur and the presence of toxic metals such as Cr, As, Hg, Ni, Pb and Zn (Barbosa and Filho, 2004).The result from the chemical test shows that the sewage sludge ash consists of high amount of sulphur. Hence, concrete degradation due to sulfur attack need to be given more concern. Meanwhile, Monzó *et al.* (1999) carried out an investigation on Portland cement that applied increasing percentages of C3A to study the influence of sulfur on the mortar. However, from the study, the sulfur in the sample was not soluble since no significant reaction with C3A was traced. The content of sulfur did not affect the strength development of the SSA mortar.

Sewage sludge waste is one of the largest contributors of waste material in Malaysia, which indirectly elevates local environmental problems (Abbas, 2011). In addition, Malaysia has limited landfill sites (Idrus, 2008). Each person produces about 1 kg of solid waste everyday and the waste production rate is increasing at 15% per year due to urbanization and population growth. The disposal rate of municipal waste is far higher than the decomposition rate at landfills. In a short period, the current landfills in Malaysia will reach their design capacity. Although the volume of sludge is reduced after the incineration process, the sewage sludge ash produced from the incineration process must still be disposed. In the meantime, Malaysia has limited research about the properties and characteristics of sewage sludge.

Various researches had been carried out to determine the potential use of sewage sludge ash (SSA) as partial cement replacement materials, in bricks, clay and ceramic materials. However, further research in order to determine the potential use of SSA in cement and concrete based material in Malaysia seems to be lacking. Hence, the aim of this study is to characterize the raw sewage sludge obtained in Kuantan, Malaysia and incinerated sewage sludge at 600°C and 800°C with 10% replacement in cement mortar.

MATERIALS

Sewage Sludge

Sewage sludge used in this research was collected from one of the IWK Kuantan's treatment plants. The sewerage treatment plant is surrounded by mostly residential and commercial areas; hence, the sludge collected is categorized as domestic waste sludge. At the sewerage treatment plant, the sewage sludge was sun dried in the sludge bed. Only the top part of the sludge in the sludge bed was collected using shovel since underneath the sewage sludge is fine sand which functions to filter the sewage sludge. The sewage sludge was placed in plastic bags and stored in a dry store room. Although the sewage sludge was sun dried at the treatment plant; it was still high in moisture content. To further remove the moisture content, the sewage sludge sample was oven dried. Excess moisture content will affect the incineration process. The sewage sludge was oven dried in a universal oven at 100°C for 24 hours to reduce the moisture content of the sample before the incineration process. The removal of water is important to ensure the reaction during the incineration process is constant. Next, the oven dried sewage sludge was incinerated using a furnace. The dried sewage sludge was placed in crucibles before being inserted into the furnace for the incineration process. This was to ensure the burning process of sewage sludge is completed and to make it more convenient for the sample handling after the burning process. The sewage sludge was burnt at 600°C for 4 hours. The ash was then ground into fine particles in powder form before it was mixed with the mortar. The fine SSA was sieved using a sieve shaker with a 150 μm sieve. Only the SSA that passed through the 150 μm sieve was used to prepare the mortar mix in this research.

Cement

The cement that was selected for the mixing of mortar in this research is YTL ORANG KUAT ordinary Portland cement, OPC. YTL ORANG KUAT is certified by MS 522-1: 2007 (EN 197-1: 2000), CEM I 42.5 N/ 52.5 N and MS 522: Part 1:2003. Only the same batch of cement was used to improve the precision. The cement was ordered and stored in a dry place in the laboratory.

Sand

The fine aggregate used in this research was natural fine aggregate from Panching, Kuantan. According to ASTM, aggregates that passed through the 4.75 mm sieve is considered as fine aggregate. Hence, the fine aggregate was prepared by passing through a 4.75 mm sieve. After the sieving process, the fine aggregate was stored in an air dry condition to prevent excess water content and impurities from happening before the mix.

Mortar Mix

The mortar mix was designed, according to the standard ASTM C1329-05, as a type N mortar (2.75:1:0.6). Water cement ratio considered in this study is 0.6. The mortars were cast in 50 mm x 50 mm x 50 mm engineering steel moulds. The mortar paste was cured for 24 hours in the steel mould and immersed in water at room temperature for curing.

EXPERIMENTAL TECHNIQUES

X-ray diffraction (XRD) and X-ray fluorescence (XRF)

In this research, X-ray diffraction (XRD) was performed by using an XRD machine with the method of 2θ scanning ranging between 5° and 50°. The analysis scan was set to run at 0.02° steps, with 1 second counting time. The samples considered for XRD test in this research were raw sewage sludge as well as 600°C and 800°C SSA in the form of powder.

X-ray fluorescence (XRF) analysis was to determine the chemical composition of the specimens in a powdery form which consist of fine grains of single crystalline material. Raw sewage sludge, 600°C SSA and 800°C SSA was sent for testing. XRF was carried out by using a Bruker S8 XRF spectrometer.

Compressive Strength Test

Compressive strength test was carried out according to the standard of compressive strength test (BS1881: Part 116:1983).

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Table-1. Mix proportions of SSA mortar (kg/m3).

Label	SSA	Cement	Sand	Water
0 SSA	0	1417.51	515.46	309.27
A10 SSA	141.75	1275.76	515.46	309.27
B10 SSA	141.75	1275.76	515.46	309.27

**A10SSA represents 600 °C 10 % SSA replacement mortar*
**B10SSA represents 800 °C 10 % SSA replacement mortar*

RESULTS & DISCUSSION

X-ray Diffraction (XRD)

Figure-1 presents the X-ray diffraction patterns of raw sewage sludge. The patterns from the XRD test show that sewage sludge consist mainly of quartz (silicon dioxide, SiO2), phosphorus pentoxide (P2O5) and iron oxide (Fe2O3). The peak of XRD patterns is at 27° with an intensity of 92 cps, which is silicon dioxide, followed by silicon dioxide and phosphorus pentoxide at 21° with 33.21 cps. Raw sewage sludge sample in this research consist of high amount of quartz. Tantawy *et al.* (2012) and Jamshidi *et al.* (2010) both indicated that the main phase of sewage sludge is quartz.

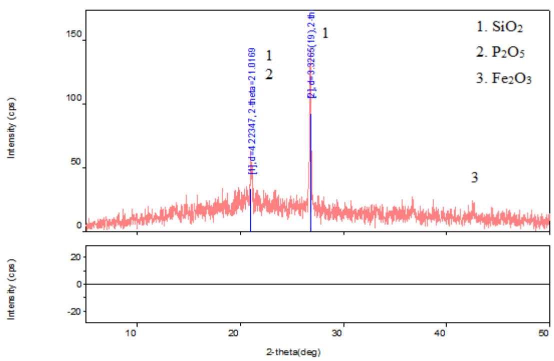
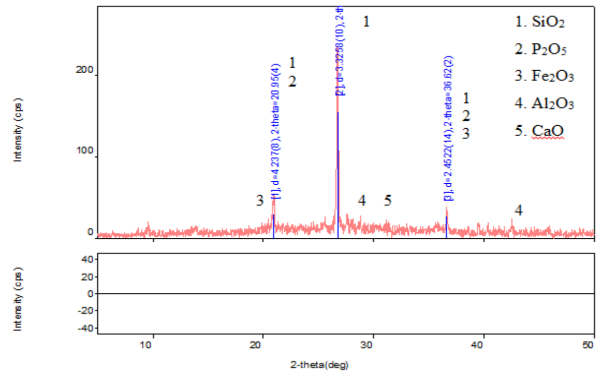
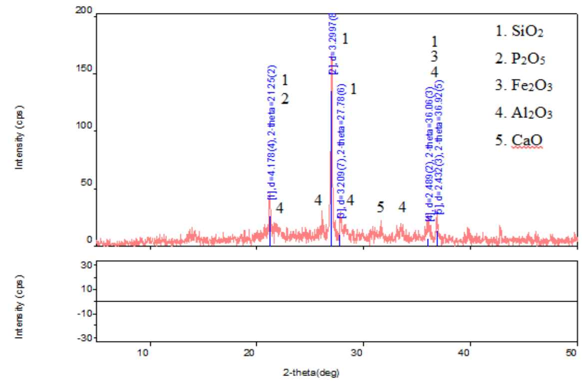


Figure-1. XRD pattern of raw sewage sludge.

Figure-2 shows the XRD pattern for the 600°C and 800°C incinerated sewage sludge ash in this research. According to the XRD patterns, the major components that were identified in 600 °C SSA are quartz (silicon dioxide, SiO_2), phosphorus pentoxide (P_2O_5) and iron oxide (Fe_2O_3). The XRD patterns show the peak of the sample at 26.8°, where it consists of mainly quartz. The intensity at peak of 26.8° is 155 cps. The next peak is at 20.9° with 29 cps, consisting of quartz and P_2O_5. Meanwhile the peak at 36.6 ° with 26 cps shows that 600 °C SSA also comprise of iron oxide (Fe_2O_3). The minor component show at the 31.2°, 29.2° and 42.4° are aluminium oxide (Al_2O_3) and calcium oxide (CaO). Similar to the sewage sludge, SSA also had quartz as the major component. However, the 600°C SSA shows the presence of different components such as aluminium oxide (Al_2O_3) and calcium oxide (CaO). This indicates that the incineration of SSA at 600 °C promotes the formation of Al_2O_3 and CaO. Similarly, quartz or silicon dioxide (SiO_2), phosphorus pentoxide (P_2O_5) and iron oxide (Fe_2O_3) were identified as the main component of 800°C SSA. The XRD result indicated that more quartz and aluminium oxide were formed at 800°C compared to the raw sewage sludge.



(a) 600°C SSA



(b) 800°C SSA

Figure-2. XRD pattern of (a) 600°C SSA (b) 800°C.

X-ray Fluorescence (XRF)

Table-2 summarizes the content of elements in sewage sludge (SS), 600 °C SSA and 800 °C SSA. Sewage sludge and sewage sludge ash contain primarily the elements silicon (Si), iron (Fe), aluminium (Al), phosphorus (P) followed by heavy metal such as Sulphur (S), titanium (Ti), zinc (Zn), barium (Ba), copper (Cu) and manganese (Mn). There was a very high content of silicon in the SSA samples after the incineration process. Raw sewage sludge consist of 4.82% silicon. After incineration at the temperatures of 600 °C and 800 °C, the content of silicon increased to 18.49% and 18.66% which is about 4 times the silicon content in raw sewage sludge. This shows that the burning of sewage sludge up to 800 °C triggers the formation of silicon (S) in the SSA. The process of incineration is able to produce more silicon which is the main component that is responsible for pozzolanic activity in cementatious material. However, the content of silicon in 800°C SSA is almost the same as in 600 °C SSA. By comparing the 600 °C SSA and 800 °C SSA, the sulphur content of 600 °C SSA is higher than 800 °C SSA. 600°C SSA and 800 °C comprises of 1.44% and 0.69% sulphur, respectively. This may be one of the reason that caused

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better performance of 800 °C SSA as compared to 600 °C SSA where the presence of sulphur can cause deterioration in mechanical strength of the mortar. Furthermore, the burning of sewage sludge at 600 °C shows higher content of heavy metal such as potassium, titanium, magnesium and zinc as compared to 800 °C SSA. The content of heavy metal slightly decreased when the burning temperature reached 800 °C.

Table-2. XRF test for oxide content in sewage sludge, 600°C and 800°C SSA.

Sample	SS	600SSA	800SSA
Element	Percentages (%)		
Silicon (Si)	4.82	18.49	18.66
Iron (Fe)	12.07	14.44	14.30
Aluminium (Al)	1.59	7.95	7.55
Phosphorus (P)	4.85	6.56	6.32
Calcium (Ca)	2.76	4.20	4.12
Sulphur (S)	1.35	1.44	0.69
Potassium (K)	0.49	1.13	1.08
Titanium (Ti)	0.36	0.99	0.96
Magnesium (Mg)	0.17	0.91	0.82
Zinc (Zn)	0.40	0.81	0.79
Barium (Ba)	0.12	0.13	0.13
Copper (Cu)	0.05	0.13	0.11
Manganese (Mn)	0.05	0.09	0.09
Tin (Sn)	-	0.06	0.06
Zirconium (Zr)	0.03	0.05	0.04
Chlorine (Cl)	0.14	0.05	-
Lead (Pb)	0.04	0.03	0.03
Bromine (Br)	0.04	0.02	-
Chromium (Cr)	0.03	0.02	0.02
Strontium (Sr)	0.03	0.02	0.02
Nickel (Ni)	0.01	0.02	-
Vanadium (Nb)	-	-	0.01
Rubidium (Rb)	0.01	-	-

Compressive Strength

Compressive strength results of the SSA mortar are summarized in Table-3 and plotted in Figure-3. The compressive strength of control mortar (0% SSA) is 7.43 MPa which is the highest among all the other SSA mortar. The mortar samples A10 SSA, B10 SSA, are recorded as 2.32 MPa, 2.11 MPa, respectively. This shows that the early strength of the SSA mortars are very weak as compared to the control mortar. Wang *et al.* (2009) stated that the early strength of mortar with SSA replacement is reduced because the early hydration process is delayed. The formation of calcium silicate hydrate, C-S-H at the early stage becomes retarded as the replacement of SSA increases where C-S-H is responsible for the early strength development in cement based materials.

The compressive strength of the A10 SSA and B10 SSA increased drastically to 21.36 MPa and 22.75 MPa, respectively at the age of 7 days. On the other hand, the control mortar recorded 23.39 MPa which is 9.50% and 2.74% higher than the A10 SSA and B10 SSA. The mortar reached its designed strength at the age of 28 days. The compressive strength of A10 SSA at 28 days is 33.28 MPa whereas the compressive strength decreases 5.23% after the SSA replacement. Meanwhile B10 SSA recorded a 35.53 MPa compressive strength which is 1.44% higher than the control mortar strength, 35.02 MPa at the age of 28 days. The compressive strength result at 28 days shows that mortar with 10% SSA replacement started to reduce the gap with control mortar. Cyr *et al.* (2007) suggested that this might be due to the pozzolanic activities that are triggered by SSA after the early hydration process of cement.

As the curing days reached 90 days, the compressive strength of A10 SSA was 40 MPa which is slightly lower than the control mortar strength of 42.06 MPa. However, the compressive strength of B10 SSA increases to 44.30 MPa which is 5.06% higher than the control mortar strength.

Table-3. Compressive strength of mortar with SSA partial replacement.

Specimens	Compressive Strength, MPa			
	1 Day	7 Days	28 Days	90 Days
0 SSA	7.43	23.39	35.02	42.06
A10 SSA	2.32	21.36	33.28	40.00
B10 SSA	2.11	22.75	35.53	44.30

*A10SSA represents 600 °C 10 % SSA replacement mortar
*B10SSA represents 800 °C 10 % SSA replacement mortar

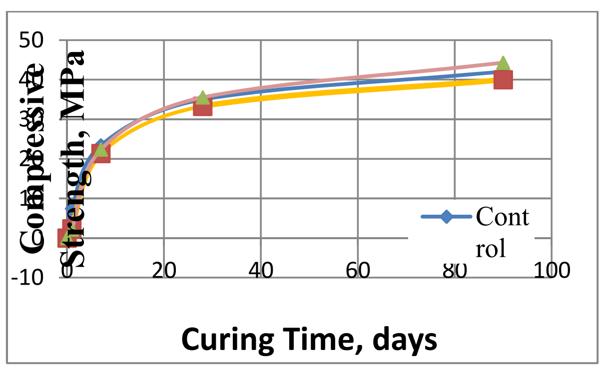

Figure-3. Compressive strength versus curing time of 10% SSA at 600°C and 800°C.

Field Emissions Scanning Electronic Miscroscope (FESEM)

Figures-4 to 6 show the FESEM micrographs of the control mortar with SSA. FESEM of the micrograph obtained as depicted in Figure-4 shows that the shape of the particles in the control mortar are mostly angular.

Spherical particles were also found which indicates the bonding between the angular particles. The spherical particles were possibly produced from the hydration of cement, resulting in C-S-H. The formation of the C-S-H bond shows the hydration process of cement occurs in the mortar. The C-S-H bond holds the particles together and provides primary strength to the mortar. Figure-4 also shows pores between the sand particles in the control mortar.

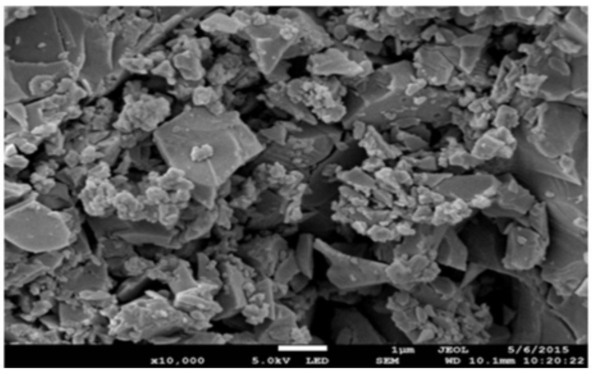

Figure-4. FESEM image of 0 SSA control mortar 10 kx.

The formation of needle-shaped particles in 10% replacement of SSA in cement mortar, A10 SSA can be observed in Figure-5. The pores in A10 SSA mortar are filled with small spherical particles and needle-shaped particles. The spherical and needle-shaped particles are probably the bonding formed by the SSA component. Tantawy *et al.* (2012) reported that the needle-shaped particles is calcium siliate which are formed from the pozzolanic activity of sewage sludge ash and portlandite liberated from the cement hydration. The needle-shaped particles are able to fill the pores in the mortar. The reduction of pores in the mortar can improve the structural strength of the mortar.

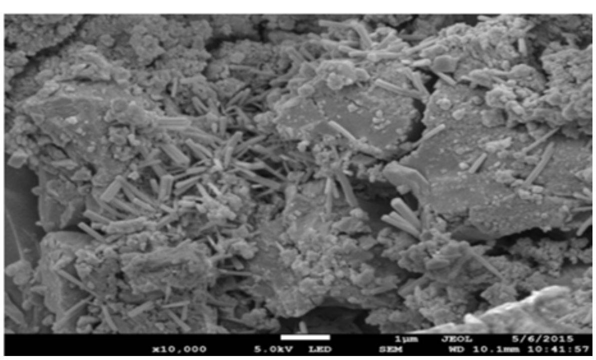

Figure-5. FESEM image of A10 SSA mortar at 10 kx.

Referring to Figure-6, there is a formation of a smooth surface structure in B10 SSA. The smooth structure shows the difference between A10 SSA mortar and B10 SSA mortar. The structure formed is probably due to the reaction of pozzolanic material from SSA with $Ca(OH)_2$ as suggested by Wang *et al. (2009).* The smooth textured particles are the product from the pozzolanic activity of SSA and cement which filled the void and pores in the mortar. The bonding also provides additional strength to the mortar. This explains the increase of compressive strength after the replacement of SSA into the mortar.

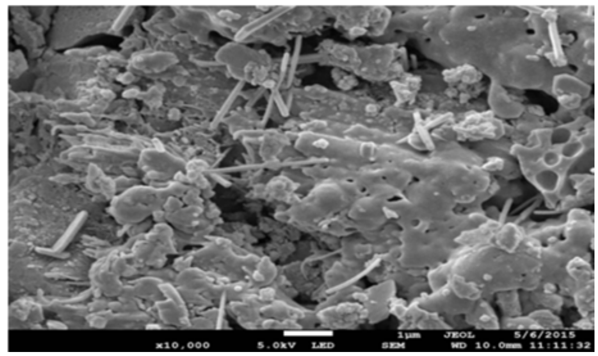

Figure-6. FESEM image of B10 SSA mortar at 10 kx.

CONCLUSIONS

From the studies carried out, the following conclusions can be drawn. Raw sewage sludge contains mainly quartz (silicon dioxide, SiO_2), phosphorus pentoxide (P_2O_5) and iron oxide (Fe_2O_3). After incineration, the major components in sewage sludge ash are quartz, aluminium oxide (Al_2O_3), iron oxide (Fe_2O_3), and phosphorus pentoxide (P_2O_5). The formation of SiO_2, Al_2O_3 and CaO occur after the incineration process in which a significant increase in the amount of the contents were traced.

On the other hand, the replacement of 10% of 600 °C and 800 °C SSA into mortar causes the deterioration of early strength of the mortar. The early hydration process is retarded with the replacement of SSA. However, mortar with 10% replacement of 800 °C burnt SSA improves the compressive strength up to 1.14% and 5.06% at the ages of 28 and 90 days, respectively. Results of compressive strength implies that the replacement of 800 °C SSA in cement mortar exhibited better performance compared to 600°C. The mortar samples with 10% replacement of SSA burned at 600 °C and 800 °C show the formation of spherical and needle-shaped particles which act to fill the gap. The needle-shaped particles are possibly the products formed from the pozzolanic activity triggered by the SSA components.

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