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Pozzolanic action of nanoparticles of ceramic sludge ash with Portland cement pastes

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ABSTRACT

Use of the highly fineness ceramic sludge ash (CSA) has a high importance due to its CO_2 reduction by its substitution into cement. Current research studies the expected changes in the characteristics of blended cement pastes with nanograin size particles of CSA. Results showed that the high fineness of CSA was the main reason to increase w/c-ratio and to delay the setting time. The CSA influenced the strength of cement pastes after 3 days of hydration more than after 28 days. Quartz and feldspar in CSA may be the essential reason for the early hydration retardation. After 28 days, physical and mechanical properties were used to limit both free lime contents and rate of hydration. Pozzolanic character of CSA could produce calcite, which enhanced the rate of hydration and improved the strength up to 90 days. SEM microscopic analysis proved the formation of amorphous hydration products, CSHs and portlandite that were smolder the surfaces and sides of CSA forming a robust structure due to the normal hydration and pozzolanic activity of CSA.

Keywords: Ceramic sludge ash, Cement paste, Consistency, Setting, Water absorption, Strength, Portlandite, Heat of hydration, SEM.

INTRODUCTION

Carbon dioxide emission (CO_2) during the manufacture of cement is the main anthropogenic contributor to global warming[1-3]. Global warming may lead to human casualties and substantial economic losses[3,4]. To minimize CO_2 emissions from cement industry, the ceramic sludge ash (CSA) could be used as a supplementary cementitious material (SCM), which is partially replacing to cement to form suitable cement pastes particularly as Portland cement clinker (PCC) that has similar constituents with CSA[5-12]. Recently, several studies had investigated the utilization of CSA. The fineness of CSA waste particles is too high to be nanoparticles that facilitate its reutilization it blended cement[13]. The porous matrix caused by the addition of CSA demonstrated its lower fluidity in cementitious mortar[14-16]. Some studies showed that CSA increased the setting of cement pastes[17-21]. The cement dilution influences the early strength of cementitious mortar, and with a lower effect on the long-term strength[22-24]. When the hydration proccedes, the pozzolanic reactions of CSA with portlandite started and form more C-S-H-gel that compensated the loss of initial strength which is mainly attributed to decline of early hydration reaction by orthophosphate from CSA in the pore solution[25-27]. Existence of more inert materials in CSA could be negatively effective. This is the main cause that influencing the early hydration, Pozzolanic reactions during the later ages of hydration showed that the high SiO_2 materials affected the amount and type of the formed C-S-H. This influenced the characteristics of cements, i.e. fluidity, porosity and strength. It is essentially attributed to the easy reaction of SiO_2 when present in alkaline media with $Ca\ (OH)_2$. Thus, CSA has more SiO_2, Al_2O_3 and CaO contents[28-31]. So, CSA was shown to be effective and necessary and the investigation of CSA effect on the long run strength of cement paste by determining of the pozzolanic reaction is too important to study[32-36]. Hence, the pozzolanic reactions comprise heat release, bound water, and/or portlandite consumption serves as a reliable indicator[29-33]. Relation among heat release and $Ca\ (OH)_2$ consumption depends on the chemical composition of the pozzolan itself. The objective of the current research is to show the effect of the CSA waste material on the various properties of the OPC pastes. Results are confirmed by the free lime content, heat of hydration and scanning electron microscopy (SEM).

Test procedures

Used raw materials in the research article are Ordinary Portland cement which was supplied from Sakkara cement factory, Egypt. Elementary phases of the OPC are tabulated in Table 1, while oxide ratios of OPC and ceramic sludge ash, as analyzed by an X-ray fluorescence are summarized in Table 2. Basically physical features of the OPC cement and CSA waste material are shown in Table 3. At first, CSA was dried and kept at 100°C for two days. CSA waste was calcined in a furnace up to 800 for 2 hours. Then, it was put into a ball mill for 2 min to obtain CSA[24-26]. Particle size distribution of both OPC and SSA are shown in Fig. 1, where the CSA is the higher fineness, whereas the OPC is the lower. Table 4 shows the constitutions of the various cement mixtures. The CSA was substituted for cement to prepare cement mixtures.

Table 1: Phase constituents of Cement spacemen, %

Phase Material	C_3S	β-C_2S	C_3A	C_4AF
OPC	49.47	29.19	4.83	11.85

Table 2: Chemical composition of materials (%)

Oxides Materials	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	LOI	Total
OPC	21.24	4.68	2.87	63.32	2.01	1.23	0.68	2.06	1.76	
CSA	56.12	17.26	7.88	9.22	3.29	.0.96	2.98	1.60	1.65	

Table 3: Physical properties of raw materials, %

Properties materials	Spec. gravity	Density, g/cm^3	Fineness, cm^2/g
OPC	3.15	3.12	3564
CSA	2.66	2.87	5683

Table 4: Composition of cement mixtures, wt. %

Mixtures materials	S0	S1	S2	S3	S4	S5	S6	S7
OPC	100	95	90	85	80	75	70	65
CSA	0	5	10	15	20	25	30	35

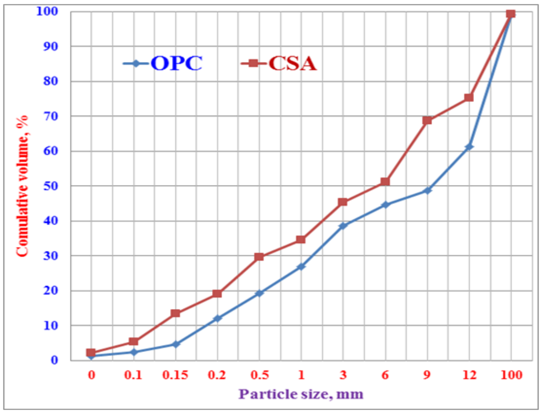

Fig. 1. Particle size distribution of raw OPC and CSA

During the preparation of cement mixtures, different dosages of CSA (0, 5, 10, 15, 20, 25, 30 and 35 wt.%) were added to the OPC. These OPC/CSA mixtures were categorized into nine groups as C0, C1, C2, C3, C4, C5, C6 and C7, respectively. Mixing of the various cement blends has done in a porcelain ball mill containing 3-5 balls for one hour to assure the complete homogeneity of blends. Firstly, the w/c-ratio and setting time are measured using Vicat apparatus[37-39]. Pastes of the various blends were prepared with the determined w/c-ratio, i.e. the right w/c-ratio was added to the cement powder in the mixer. Then, the mixer was run for about 5 min at an average speed of 10rpm to have a perfect homogenous mixture. Cement pastes were then molded into one inch cubic stainless steel molds (2.5 cm^3) using about 500 g cement mix, vibrated manually for two min. and then on a mechanical vibrator for another two min.[32] The surface of the molds was smoothed using a suitable spatula. The molds were kept in a humidity cabinet for 24 hours. on 95±2 RH and R.T of 20±1°C for curing until the corresponding days. In the following day, the molds were demolded and the samples were immediately placed in water till the time of testing. Water absorption, bulk density and total porosity[40,41] of the hardened cement pastes could be calculated.

Flexural strength was calculated[42,43], whereas the samples were marked at three points adjusting to place them on the correct point of contact (Fig. 2). The compressive strength of the different cement pastes also was measured[44,45].

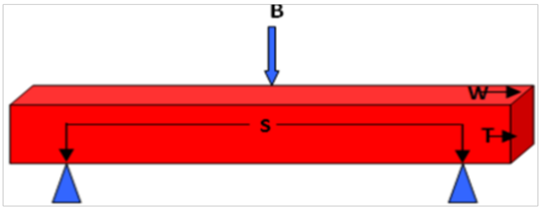

Fig. 2. Schematic diagram of bending strength, B: Beam or loading of rupture, S: Span, W: Width and T: Thickness

Free lime (FLn) of the hydrated specimens pre-dried at 105°C for 24 h was also measured[32,45-49]. Heat of hydration has been studied to assure the obtained results[50-53]. Ultra-sonic pulse velocity was essentially done. Based on the measured free lime contents, the degree of cement hydration was explored the influence of CSA on the long-term strength of cement pastes.

The X-ray fluorescence was analyzed the chemical and phase composition of OPC and CSA, identifying crystalline phases of OPC cement. Constituents of some samples were studied with scanning electron microscopy. The SEM images of the fractured surfaces, coated with a thin layer of gold, were obtained by JEOL-JXA-840 electron analyzer at accelerating voltage of 30 KV. The arithmetic mean for each group could be determined the corresponding strength values, noting that any abnormal data must be excluded.

RESULTS AND DISCUSSION

Water of consistency and setting times (ST) of OPC pastes (C0) blended with various percentages of CSA (C1-C7) are shown in Fig. 2. It is clear that WC of the control (S0) was 28.83%. This value was increased as the content of CSA enhanced. Also, the ST (Initial and final) of the blank (C0) were 132 and 157 minute. respectively. These values were enhanced as the CSA content enhanced. It is mainly contributed to the high fineness of CSA material[54-57]. Furthermore, the pozzolanicity of the CSA with the produced portlandite, which is in need to more water to occur[46,56-60].

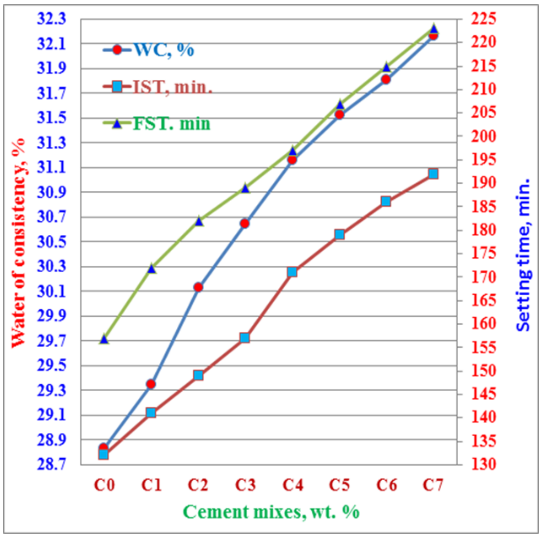

Fig. 2. Water of consistency and setting time of OPC pastes mixed with CSA

Physical characteristics

Water absorption of OPC cement pastes blended with various contents of CSA hydrated up to 90 days are represented in Fig. 3. Results illustrated that the WA of control (C0) decreased all over hydration times. It is surely attributed to the mechanism of hydration of cement phases[37,57,61]. As CSA content increased, the WA decreased. This firstly may be attributed to the increased compaction of the hardened cement pastes due to the decreased pore volume. Furthermore, the pozzolanic property of CSA with the producing portlandite that was coming from the hydration of silicates phases of the cement help to decrease the WA[45,46,62]. This was continued till 30wt.% CSA (C6). But, with any further CSA addition, the water absorption tended to increase (C7). This is essentially contributed to that the higher content of CSA at the expense of the main binding material negatively reflected on the WA results[32,46,56-58,63-65]. Accordingly, the higher content of the additive material must be refused.

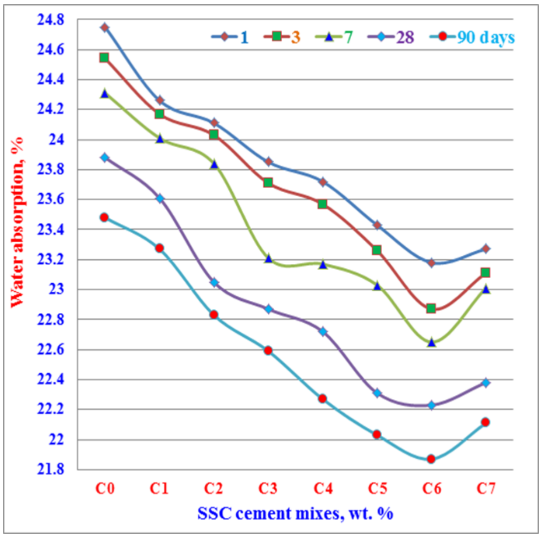

Fig. 3. Water absorption of OPC pastes mixed with CSA hydrated till 90 days

Figure 4 illustrates the bulk density (BD) of cement mixtures containing various ratios of CSA (C1-C7) compared to that of the control mixture (C0) hydrated up to 1, 7, 28 and 90 days. Results indicated that the BD increased as the content of CPSA increased[32,37,39,66]. This continued till the mix containing 30wt.% (C6). With any increased addition of CSA › 30 wt. %, the BD tended to decrease. The increase of BD is due to the increased compaction by CSA which precipitated inside the pore volume of the specimens. This resulted to decrease the total porosity[45,46,56,67]. Therefore, the higher contents of CSA must be prevented.

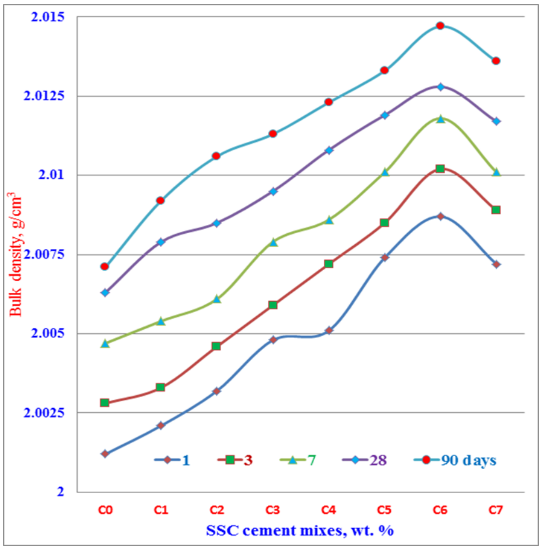

Fig. 4. Bulk density of OPC pastes mixed with CSA hydrated till 90 days

Total porosity of OPC cement pastes (C0) blended with various proportions of CSA (C1-C7) hydrated till 90 days are represented in Fig. 5. It is clearly that the TP decreased, while the hydration occurred till 90 days. Moreover, it also lowered with the increase of CSA content, but this continued merely up to 30% (C6). Then, it enhanced with any other CSA addition. Decreasing of TP is mainly due to the pozzolanic properties of CSA with the produced portlanditethat is produced from the hydration of C3S and C2S phases of the cement[22,37,47]. But, the increased values of TP is mainly contributed to stopping of hydration that was resulting from the incorporation of larger amounts of CSA (C7), which decreased not only the normal hydration process, but also the pozzo;anic activity of CSA[68-70]. So, the higher quantities of CSA must be refused due to its adverse effect. Results of TP are in a good agreement with those of BD and WA to a large extent.

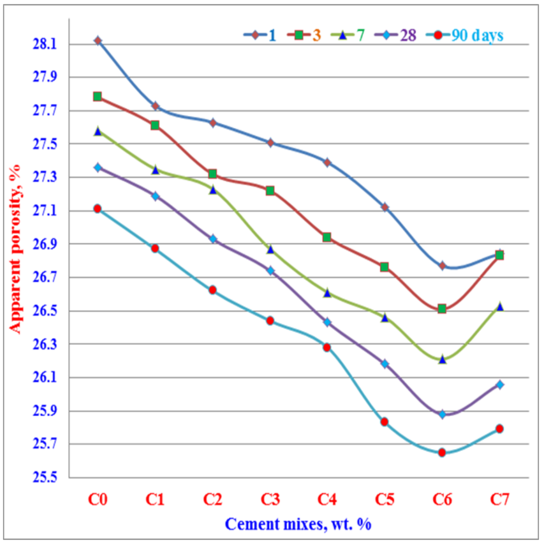

Fig. 5. Total porosity of OPC pastes mixed with CSA hydrated till 90 days

Mechanical properties

Results of flexural strength of OPC cement pastes (C0) mixed with various contents of CSA (C1-C7) hydrated till 90 days are demonstrated in Fig. 6. As shown from the figure, the FS increased as the hydration age increased up till 90 days. It is mainly due to the newly formed CSH products as a result of hydration, which improved FS of hardened cement pastes[37,69]. The FS also increased by the addition of CSA till 30wt.% (C6), and then negatively influenced by any other addition of CSA (C7). The increased FS results with CSA addition are often attributed to the pozzolanic reactivity of CSA with the formed portlandite from the hydration of di- and tricalcium silicates phases (C3S and β-C2S) of the cement. Furthermore, unreacted CSA acted as a filler that closed the pore system of samples, i.e. the total porosity decreased[57,70,71]. The adverse results with the higher addition of CSA (C7) is principally due to that the high quantities of the additive material hindered and may be ceased the hydration of cement phases and helped to open the pore structure to a large extent[45-47]. Therefore, the higher amounts of the CSA must be removed.

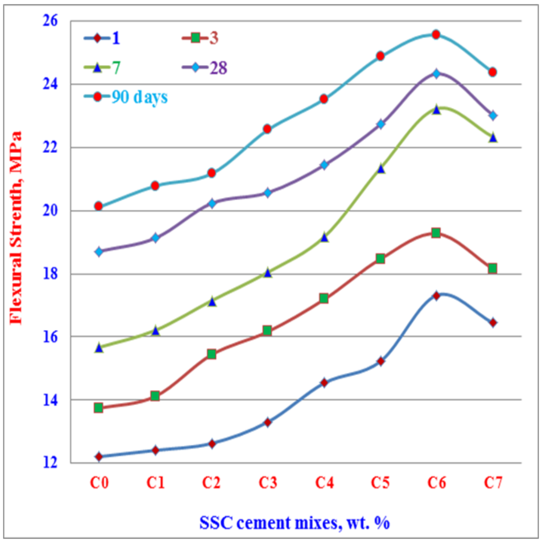

Fig. 6. Flexural strength of OPC pastes mixed with CSA hydrated till 90 days

Compressive strength of hardened blank pastes (C0) mixed with different proportions of CSA (C1-C7) hydrated up 90 days is represented in Fig, 7. CS of hardened pastes enhanced with as the hydration age till 90 days. It is mainly due to the newly resulting CSH, which are precipitated into the pore volume, i.e. this decreased the TP and increased the BD. So, this could be inverted well on the CS of the cement pastes[37,45,67]. Also, crystal growth of the formed CSH was gradually improved and enhanced. The improvements of CS often enhanced with CSA addition, but merely till 30wt.% (C6), and then adversely affected by any other addition of CSA (C7). Improved CS results with CSA addition are often due to the pozzolanic reactivity of CSA with the resulingportlandite from the normal hydration process of di- and tricalcium silicates (β-C2S and C3S) of cement, Furthermore, CSA improved the compaction of the constituents together, i.e. the pore structure was partially or completely closed. Unreacted CSA particles were acted as a filler that closed the pore system of samples[55,70,71]. The adverse results with the higher content of CSA (C7) were principally contributed to that the higher contents of the additive material largely stopped the hydration of cement phases. This helped to open more-pore structure to a large extent[32,45-47]. Therefore, the higher contents of CSA must be illuminated.

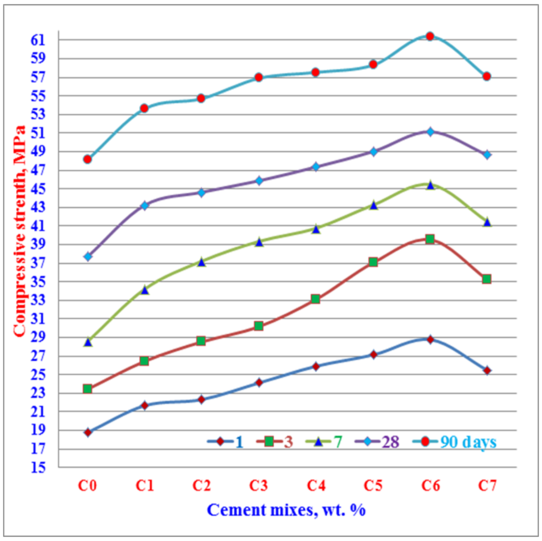

Fig. 7. Compressive strength of OPC pastes mixed with CSA hydrated till 90 days

Free lime contents (FLn) or portlandite of blank cement pastes (C0) blended with various ratios of CSA (C1-C8) hydrated till 90 days are shown in Fig. 8. FLn of blank (C0) was enhanced little by little as the hydration period was going till 90 days. It is essentially contributed to the hydration of the main silicate phases of the cement[37,58,72-74]. Additionally, FLn of other blended cements incorporated different ratios of CSA increased till 7 days of hydration, but then it began to reduce down to 90 days. It is fundamentally contributed to the pozzolanic phenomenon which could be occurred among the resulting free line and CSA particles[64-66,73]. The consumption of FLn by its reaction with the constituents of CSA led to the formation of extra C-S-H phases which were precipitated in the pore system of cement pastes. This in turn had improved and supported the physical, chemical and mechanical properties. This was contributed to the improvements of the densification parameters and the microstructure due to the reduction of voids, porosity, and water permeability[32,45,46,72]. The FLn or portlandite of cement pastes incorporated

CSA›30wt.% (C7) continued to decrease due to that the high content of CSA at the expense of the OPC hindered its hydration, i.e. the hydration process was completely ceased.

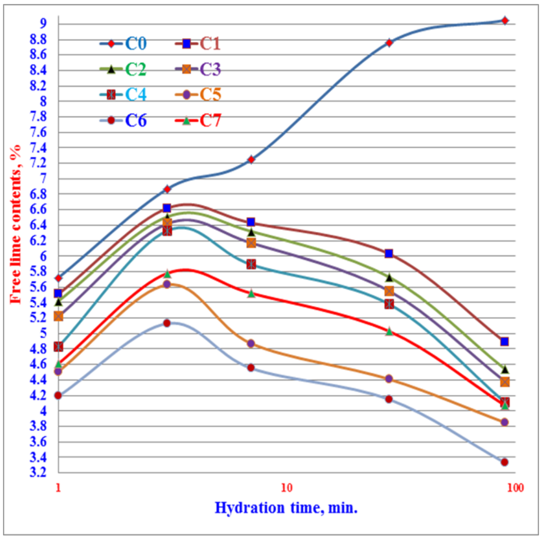

Fig. 8. Free lime contents of OPC pastes mixed with CSA hydrated till 90 days

Figure 9 shows the heat of hydration (HH) of OPC pastes (C0) blended with different ratios of CSA (C1-C7) hydrated till 90 days. When water was poured on cement powder, heat due to hydration starts to generate at once. HH of cement pastes was generally increased with the hydration age till 28 days, but at later ages it was so slightly that it seemed to be constant. The same behavior was done by all cement pastes incorporated different ratios of CSA[75-78]. This is principally contributed to increasing the rate of hydration of cement phases that was coming from the pozzolanic reactions of CSA. This was accompanied by a gradual generation of heat[79-81]. Moreover, the rate of the produced HH was largely increased at early stages from 3 to 7 days. It is due to the initiation action on the hydration mechanism of C3S by the finer particles of CSA. During older periods (28-90 days), the rate of hydration and the released HH increased so little that it seemed to be unchanged or stable. This may be due to the non-activation action of C3S and the slight activation action mechanism of β-C2S at later ages. Also, the HH enhanced little as the CSA addition enhanced, but only till 30% (C6). The increased values of HH are mainly attributed to two hydration mechanisms. The first is the hydration of cement phases and the second is the pozzolanic reactions of CSA with the evolving or formed free lime from the first process[75,76,79,80]. The HH was then unchanged with any further increase of CSA addition (C7). It is principally due to the dilution action of CSA on the cementitious compounds and the high SiO_2 content of CSA. So, the high CSA content must be avoid due to its adverse action[77-81].

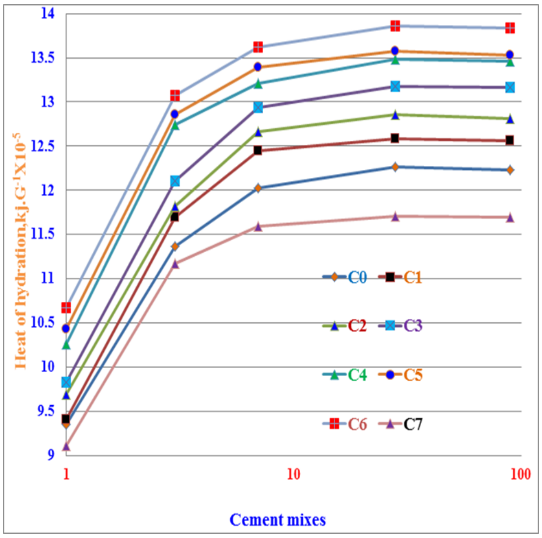

Fig. 9. Heat of hydration of OPC pastes mixed with CSA hydrated till 90 days

Scanning electron microscopy of OPC pastes blended with different ratios of CSA (A, B, C and D) or C0, C3, C6 and C7) hydrated till 90 days is demonstrated in Fig. 10. In image A of the blank (C0), ettringite phase was clearly shown as needle-like crystals and also pits of free lime covered the surfaces of CSSA particles that were resulted from the normal hydration process. Also, crystals of CSH were formed. In image B (C3), the crystals of CSH and piles of free Ca $(OH)_2$ were increased, but the ettringite phase was clearly decreased. In image C (C6), it was full of CSH and no pits of free lime, while the ettringite phase was completely disappeared. In the image D (C7), there are some internal cracks were noted and the reappearance of a very small amount of free lime which negatively reflected on the physical and mechanical properties. Moreover, the porosity was gradually decreased till C6 with the lowest degree of porosity. So, the cement batch (C6) could be considered the optimum one with well-developed crystal growth.

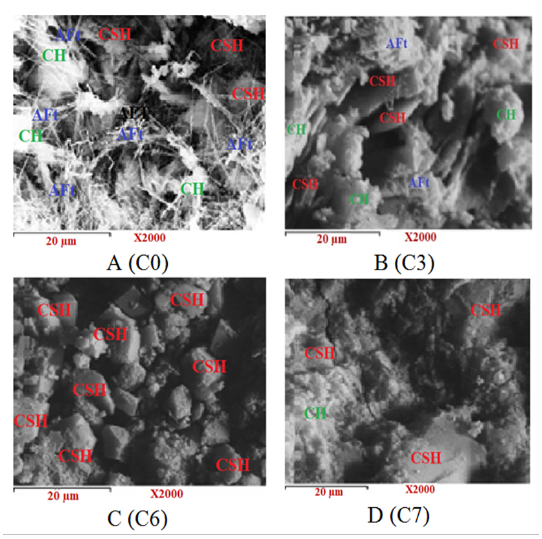

Fig. 10. SEM micrographs of OPC pastes (C0), C3, C6 and C7

CONCLUSION

The pozzolanic performance and characteristics of the nanograin sized particles CSA were assessed. The CSA needs more water to form good cement pastes, i.e. the w/c- ratio continuously increased causes. Also, it causes a gradual retardation in both initial and final setting times, i.e. as CSA addition enhanced, the retardation time increased too. The resulting free lime in the cement paste with 0% CSA (C0) on 90 days was approximately 9.05%. This result significantly decreased with increasing of both curing time and CSA content. So, CSA is a pozzolanic material. As it is clear, the added CSA › 30wt% (C7), the CH content starts to increase. It is principally contributed to the stopping or finishing of pozzolanic activity of the added material. The physical properties of cement pastes contained CSA material were improved and enhanced with curing time and CSA content, but only up to 30wt%. Flexural and compressive strengths of the blended cement improved and enhanced with the increase of CSA content till 30% (C6). Any further increase of CSA content › 30wt.%, the physical and mechanical properties would be diminished. So, the optimum CSA addition to cement without adverse effects on its characteristics is 30wt.%. SEM spectra revealed a higher quantity of CSHs in CSA paste forming a denser and more compact microstructure. Adding a large quantity of CSA caused the reduction of the heat of hydration and prolonged the time required to reach the silicate and aluminate reaction. This has led to a delay in hydration and resulted in excess setting times and reduced early and late strength development. The CSA powder contains SiO_2 and Al_2O_3 › 90% of its total mass, and therefore it has a natural of pozzolanic materials. Blending CSA with OPC could be enhanced the pozzolanic performance forming additional CSH-gel because it improves the densification parameters of the OPC pastes, i.e. it reduced the voids, porosity, and water permeability.

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Competing interest

The author declares that there are no financial or competing conflicts of interest in this work.

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