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Study on Portland Cement Pastes Containing Sanitary Ware Ceramic Wastes at Elevated Temperatures

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Abstract

Resistance of Portland cement pastes incorporated 20 wt. % sanitary ware ceramic nano-powder wastes to firing temperatures from 100 up to 600°C was investigated. Results revealed that all physical and mechanical properties were improved and gradually enhanced with firing temperatures, but only up to 400 °C, and then adversely affected with any further increase of firing temperatures than 400°C, i.e. water absorption and total porosity decreased, whereas bulk density was enhanced. Water absorption and total porosity were decreased by 10 and 4 %, respectively, while bulk density was increased by 2.25 %. Furthermore, flexural and compressive strengths were also improved and increased by 1.69 and 1.2 %, respectively. Fourier transform infrared spectra (FT-IR) showed the disappearance of free lime and ettringite on firing. Scanning electron microscopy (SEM) showed that the crystal phase growth of the formed hydration products as a result of both normal hydration and pozzolanic reactions at ambient temperature were developed and modified due to the temperature exposure. The hardened cement pastes can withstand and resist only up to 400°C.

1. Introduction

Existence of waste from the construction sector and due to its adverse environmental effects, it is desirable to reutilize this waste and to promote a sustainable waste recycling. This has encouraged many researchers to creat some solutions to evaluate the construction and demolition wastes [1–5]. This is often applied to materials as asphalt, brick, concrete, ferrous metal, ceramics and glass [6]. However, construction demolition waste such as concrete, ceramics and brick, is usually dumped into the ground without being reused [7]. In this concern, the environment has been deteriorated while the lands are occupied with debris [5-8].

Construction materials especially conventional cement, concrete, and/or mortar employed in structures can be exposed to high temperatures during fire [9]. The strength of these materials is significantly affected after being subjected to elevated temperature [10]. The fire resistance of aggregates is affected by elevated temperature, moisture content, mineralogical composition, and pore structure. Ca $(OH)_2$ begins to disintegrate into its components at 400 °C and continues until the substantial dissociation of calcium silicate hydrate (CSH) gel at about 800°C [11,12]. Therefore, researchers tended to decrease the hazardous impact of elevated temperature on these construction composites [13,14]. Selection of the most suitable raw sources for the manufacture of these composites is one of the precautions to reduce the damage of fire [15]. In recent years, to improve and increase the fire resistance of these cementitious composites, several industrial wastes are being utilized in the cementitious composites, such as fly ash [16-18], silica fume [19,20], and blast furnace slag [21,22] have been used to improve the fire resistance of cementitious composites. However, the effect of these

materials on the performance of Portland cement mortars at elevated temperatures needs to be further investigated.

Many studies [23-29] had investigated the pozzolanic properties of Portland cement pastes (OPC), mortar and concrete produced with different waste types such as slag, fly ash, and silica fume. However, the influence of ceramic sanitary ware powder waste (CSPW) on the physical and mechanical properties of Portland cement pastes subjected to high temperature has not yet been investigated sufficiently. The primary target of the current research is to investigate the recycling of ceramic sanitary ware powder waste (CSPW) and evaluate its effect as a sustainable replacing material in Portland cement pastes. Therefore, the present study intends to investigate the effect of elevated temperature on the performance strength of OPC with 20 wt. % CSPW at the expense of cement. Therefore, the physical and mechanical properties of OPC pastes incorporating 20 wt. % CSPW at 100, 200, 300, 400, 500 and 600°C have been researched. The obtained results are confirmed with Fourier transform infrared spectra (FT-IR) and scanning electron microscopy (SEM).

2. Experimental Procedure

2.1 Raw Materials

The used raw materials in the current study are ordinary Portland cement (OPC) and ceramic sanitary ware waste (CSW). The OPC sample (OPC Type I- CEM I 42.5 R) was delivered from Sakkara cement factory, Giza, Egypt having the surface area or fineness of 3500 cm^2/g. The surface area was measured by Air Permeability Apparatus [30]. The broken pieces of sanitary wares were crushed using a suitable crusher. These crushed ceramic wastes were then let to grind in a ball mill (many balls of various sizes and diameters) for only 60 minutes till pass from a 75 µm sieve. The resulting powder is called ceramic sanitary powder waste (CSPW). The specific gravities of OPC and CSPW as measured with a Le Chatelier flask were 3.15 and 2.73 g/cm^3, respectively. The chemical analysis of OPC and CSPW using X-ray fluorescence technique (XRF) is shown in Table 1. To achieve the pre-established reference consistency, it was necessary to add 1 % polycarboxylic ether as a high reducing water superplasticizer admixture to the mixing water. Table 2 shows the Mineralogical composition of OPC sample, while Table 3 indicates the physical properties of the raw materials.

Table 1 *Chemical oxide composition of the raw materials, %*

Material	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	LOI
OPC	20.12	5.25	1.29	63.13	1.53	0.55	0.3	2.54	2.64
CSPW	30.56	8.31	3.68	51.73	3.67	0.02	1.09	0.07	0.71

Table 2 *Mineralogical composition of OPC sample, wt. %*

Phase / Material	C_3S	β-C_2S	C_3A	C_4AF
OPC	46.81	28.43	5.90	12.56

Table 3 *Physical properties of the raw materials, wt. %*

Properties / Materials	Specific gravity	Density, g/cm^3	Blaine surface area, cm^2/g
OPC	3.15	1445	3500
CSPW	2.66	1248	5950

2.2 Preparation and Methods

There is one cement batch from OPC and CSPW as 80:20 which is the optimum batch in a previous study [31] and it was considered the control cement batch having the symbol W0. Blending process of the various cement blends was done in a porcelain ball mill using 2-4 balls (1 cm diameter and of 50 g weight) for two hours to assure the complete homogeneity of the cement blend. During casting, 1 % polycarboxylic ether as a high reducing water superplasticizer admixture was added to mixing water which in turn added to the prepared cement mix so as to avoid the agglomeration of the nanoparticles of the used CSPW or OPC. It was applied to improve cement dispersion.

The standard water of consistency (WC) of the prepared cement mix was directly determined using Vicat Apparatus which was 33.11 % [32-35]. Cement pastes were then cast using the predetermined water of consistency (33.11 %), moulded into one-inch cubic stainless steel mould (2.5 x 2.5 x 2.5 cm^3) using about 500 g

cement mix, vibrated manually for three minutes, and then on a mechanical vibrator for another three minutes to eliminate all air bubbles. The surface of the mould was smoothed using a suitable spatula. Thereafter, the mould was kept in a humidity chamber for 24 hours at 95 ± 2 relative humidity (RH) and room temperature (22±1°C), demoulded in the following day and soon immersed in water till 90 days. The hydrated cement pastes were then exposed to elevated or firing temperatures from 100 up to 600°C. Water absorption (WA), bulk density (BD) and total porosity (δ) of the fired hardened cement pastes were determined [33-36].

The mechanical properties in terms of flexural strength (FS) and compressive strength (CS) of the various fired hardened cement pastes [36-38] were measured. The FS could be carried out using the three-point adjustments system (Fig. 1).

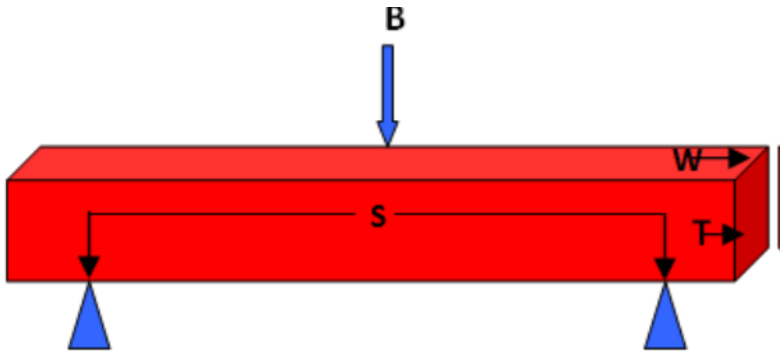

Fig. 1 *Schematic diagram of bending strength, B: Beam or loading of rupture, S: Span, W: Width and T: Thickness*

The obtained results were confirmed with fourier transform infrared spectra (FT-IR) and scanning electron microscopic (SEM) analysis. The FT-IR was performed by Pye-Unicum SP-1100 in the range of 4000-400 cm^{-1} and a resolution of 500 cm^{-1}. The FT-IR analysis was done in the National Research Centre, Dokki, Cairo, Egypt. The SEM microscopy was conducted for some selected samples by using JEOL–JXA–840 electron analyzer at accelerating voltage of 30 KV. The fractured surfaces were fixed on Cu-kα stubs by carbon paste and then coated with a thin layer of gold.

3. Results and Discussion

3.1 Physical Properties

3.1.1 Water Absorption

Results of water absorption (WA) of the optimum hardened cement pastes incorporating 20 wt. % CSPW hydrated up till 90 days at the ambient temperature (23±1 °C) [31] that was considered as the control (C0) [39-41] was subjected to different firing temperatures (200, 300, 400, 500 and 600 °C) are illustrated in Fig. 2. The WA of the blank sample (W0) hydrated up to 90 days was 16.89 %. This value was decreased with increasing the firing temperature only up to 400 °C, and then slightly increased with the further increase of firing temperature. The decrease of WA is firstly attributed to the higher compaction effect resulting from the high nanograin-size particles or fineness of both cement and CSPW which reflected positively on the physical properties of the hardened cement pastes, where it reduced the pore structure of the hardened cement pastes, in addition to the activation and initiation influence of firing temperature that promoted, improved and modified the crystal growth of the fired phases. This in turn decreased more the WA of the hardened cement pastes [42,43]. The WA is also related to surface porosity as well as structural pores [44]. But, the little increase of WA at 500-600 °C firing temperature is often due to that the higher firing temperature caused the break down or slight damage to some CSH phases [40-42]. Hence, the larger firing temperature (> 400 °C) must be avoided due to its adverse action.

3.1.2 Total Porosity

Results of the total porosity (TP) of the optimum hardened cement pastes incorporating 20 wt. % CSPW hydrated up till 90 days at the ambient temperature [31] which subjected to different firing temperatures (200, 300, 400, 500 and 600 °C) are shown in Fig. 3. The TP of the control (C0) hydrated up to 90 days was 17.07 %. This value was decreased with the increase of firing temperature till 400 °C, but it re-increased with any further increasing of firing temperature (› 400 °C). The decrease of TP is mainly due to the normal activation action of the firing temperatures [18,42], which initiated and improved the crystal growth of the formed phases [18,45,46]. This continued till 400 °C, and then increased with any rising of firing temperature (> 400 °C). The re-increased values of the TP with higher firing temperature are mainly attributed to the larger deficiency and

break down of the formed CSHs. So, the higher firing temperature than › 400 °C must be prevented, i.e. the optimum firing temperature at which the best results were achieved and the hardened cement pastes could be resisted is 400 °C.

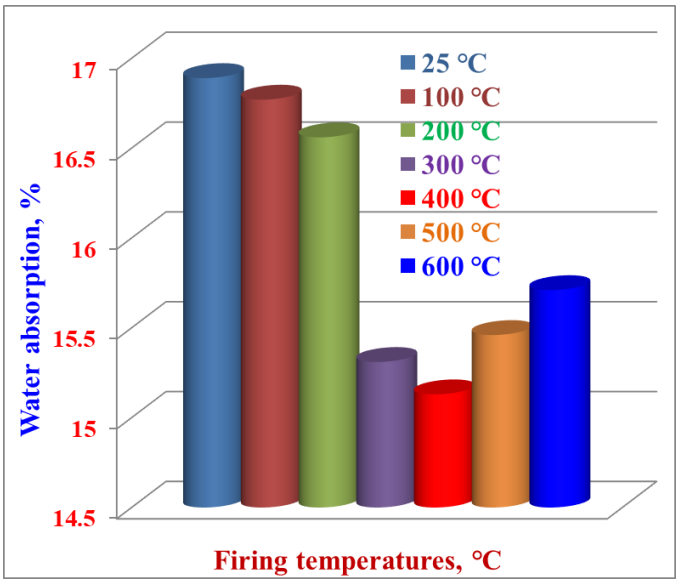

Fig. 2 *Water absorption of the optimum cement pastes containing 20 % CSPW hydrated up to 90 days and subjected to different firing temperatures*

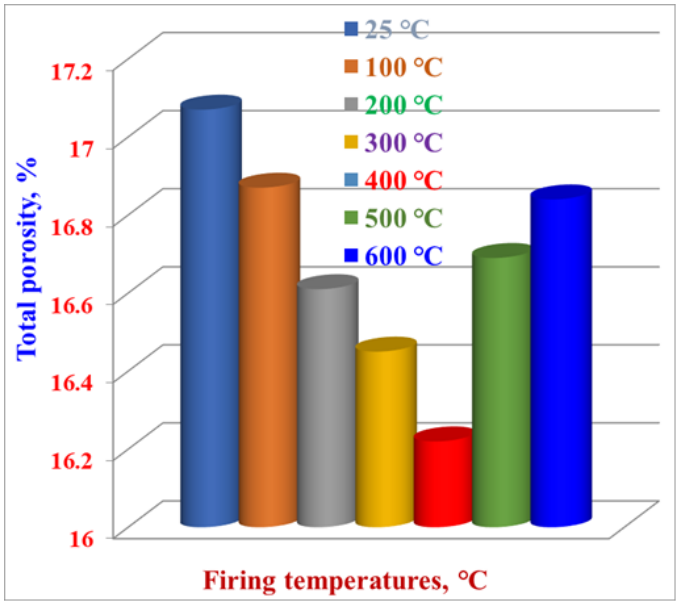

Fig. 3 *Total porosity of the optimum cement pastes containing 20 % CSPW hydrated up to 90 days and subjected to different firing temperatures*

3.1.3 Bulk Density

The bulk density (BD) of the optimum hardened cement pastes containing 20 wt. % CSPW hydrated up till 90 days at the ambient temperature [31] which subjected to different firing temperatures (200, 300, 400, 500 and 600 °C) are shown in Fig. 4. The BD of the optimum hardened cement pastes hydrated up to 90 days was 2.0583 g/cm^3. This value was slightly increased with the increase of firing temperature till 400 °C, and then decreased by further increase of firing temperature › 400 °C. The increase of BD is mainly due to the slight activation and initiation influence of the firing temperatures which modified the crystal growth of the formed CSHs [18,42-45], i.e. the exposure of the hardened cement pastes to firing temperatures activated, initiated and also increased the formed CSHs which precipitated in the pore volume of the samples. This decreased the porosity, improved and enhanced the BD [18,45]. The slight reduction in the bulk density is due to the lower specific gravity of the CSPW and the breakdown some of the formed CSHs [18,43,45]. It means that the hardened cement pastes with

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CSPW showed lower WA, lower TP and higher BD especially up to 400 °C. The better performance of the cement pastes with CSPW is due to the combined effect of pozzolanic activity and the filler effect of the CSPW, resulting in the refinement of the pores of the cement pastes. Therefore, the reduction of WA and TP were resulted. So, the optimum firing temperature is 400 °C at which the best resistance results were achieved, and the higher ones > 400 °C must be eliminated.

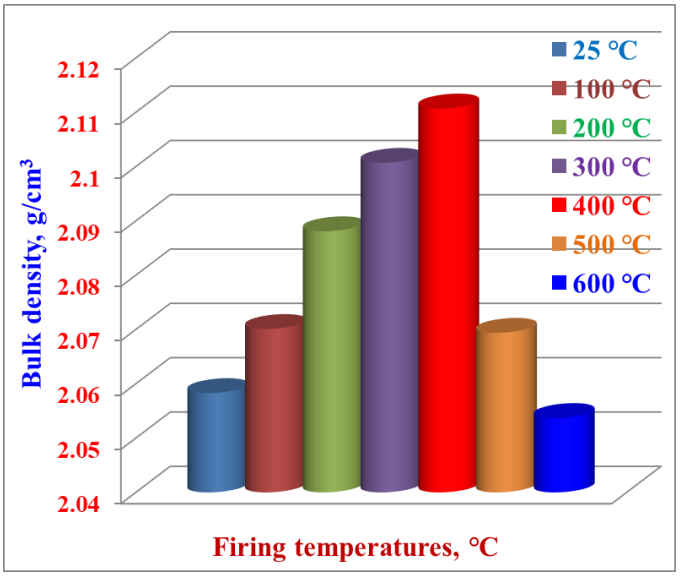

Fig. 4 *Bulk density of the optimum cement pastes containing 20 % CSPW hydrated up to 90 days and subjected to different firing temperatures*

3.2 Mechanical Properties

3.2.1 Flexural Strength

Fig. 5 illustrates the results of flexural strength (FS) of the optimum hardened cement pastes incorporating 20 wt. % CSPW hydrated up till 90 days at the ambient temperature that was considered as the control (C0) [31] was subjected to different firing temperatures (200, 300, 400, 500 and 600 °C) to explore its resistance. FS of the blank hardened cement pastes was 44.13 MPa. This result was improved and enhanced with firing temperatures only up to 400 °C, but then decreased with any further rise of firing temperature › 400°C. The increase of FS is mainly due to that the firing temperatures initiated and promoted the thermal interactions between the different ingredients of the samples so that it improved and increased the modification and development of the formed CSHs, while the decrease of FS is essentially contributed to the decomposition of Ca $(OH)_2$ or may be due to significant dehydration within the cement matrix modifying the physical properties and the dehydration of both calcium silicate hydrates (CSHs) that occurred at temperature beyond 400 °C [47-50]. This caused the weakening of pastes, which in turn resulted in the destruction of micro-structural arrangements in the hardened cement pastes.

3.2.2 Compressive Strength

Results of compressive strength (CS) of the optimum hardened cement pastes containing 20 wt. % CSPW hydrated up till 90 days at the ambient temperature that was considered as the control (C0) [31] was subjected to different firing temperatures (200, 300, 400, 500 and 600 °C) are illustrated in Fig. 6. The CS of the various hardened cement pastes was first increased with firing temperatures, but only up to 400 °C, and then decreased gradually by rising the firing temperature. The increase of CS is principally due to the initiating effect of the heating temperatures that promoted and modified the crystal structures of the formed CSHs [18,42-47]. The decrease in CS at firing temperature › 400 °C may be attributed to the significant dehydration within the cement matrix modifying the physical properties of the cement pastes. The sharp fall in CS [49] by > 400 °C onward could be due to the decomposition of $Ca(OH)_2$ and dehydration of both calcium silicate hydrates (CSHs) that occur at temperature beyond 400 °C [18,42-45]. This often caused the break down or at least the weakening of bonds of cement pastes. This always caused the destruction of the microstructural arrangements in constituents of cement pastes [49-52].

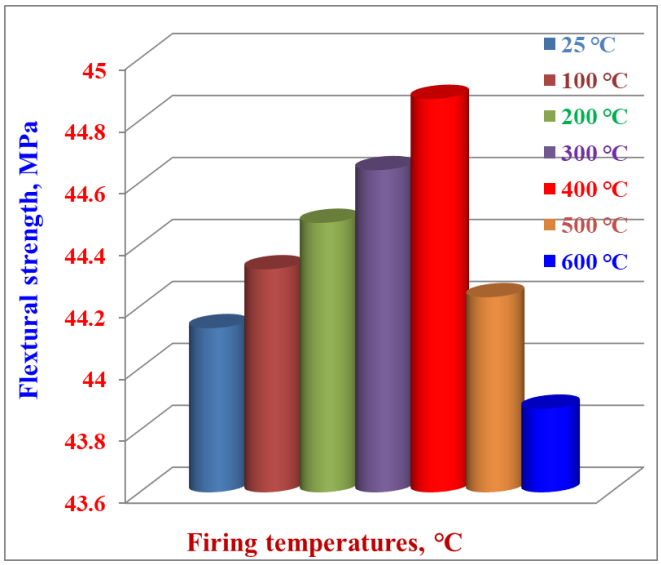

Fig. 5 Flexural strength of the optimum cement pastes containing 20 % CSPW hydrated up to 90 days and subjected to different firing temperatures

The sharp decline in CS at firing temperatures > 400 °C may be due to the decomposition of $Ca(OH)_2$ and dehydration of both calcium silicate hydrates (CSHs) that occur at temperature beyond 400 °C [18,20]. This always caused the breaking down of bonds between particles of cement pastes that caused the destruction of microstructural arrangements in the cement pastes. As a result, the presence of CSPW in the cement pastes increased the resistance of the hardened cement pastes to the high temperatures up to 400 °C. The improved resistance against high temperatures with CSPW inclusion can be mainly attributed to the decreased amount of $Ca(OH)_2$ with its pozzolanic activity [48-53].

Generally, as the temperature increases, the Si/Ca and (Si+Al)/Ca ratios are often decreased. These ratios in cement mixes are often higher than those of the reference (C0) after firing. The higher percentage of SiO_2 and Al_2O_3 in the material indicates its pozzolanic activity. Pozzolans had undergone thermal reactions [52-55], which suggested that the high silica content of CSPW had led to the formation of secondary or additional CSHs, i.e. the pozzolanicilty of these additive materials have a common practice for improving the durability, resistivity and mechanical properties [56]. This was in a good agreement with the obtained results of physical properties as bulk density, porosity and water absorption as well as mechanical properties like flexural and compressive strengths. The creation of more pores and some micro-cracks in C5 (F) is mainly due to the higher amounts of the CSWP that was leading to more porosity at higher firing temperatures due to water loss which adversely caused some declines and adverse actions on the features of the cement pastes [54-56].

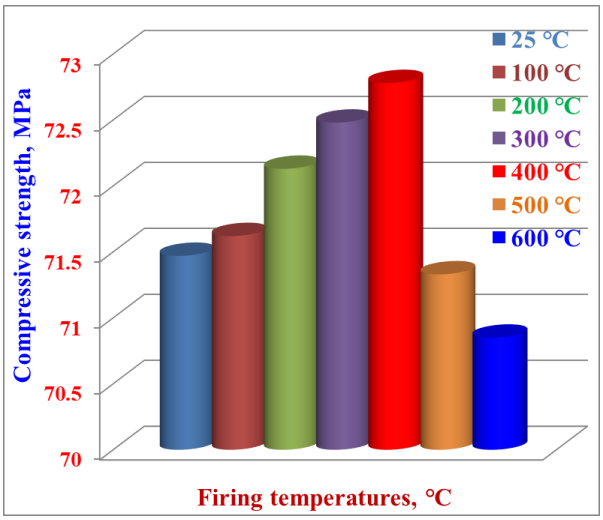

Fig. 6 *Compressive strength of the optimum cement pastes containing 20 % CSPW hydrated up to 90 days and subjected to different firing temperatures*

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3.3 FT-IR Spectra

The FT-IR spectra of the optimum hardened cement pastes containing 20 wt. % CSPW hydrated up till 90 days at the ambient temperature that was considered as the control (C0) [31] was subjected to different firing temperatures (100, 200, 300, 400, 500 and 600 °C) are illustrated in Fig. 7. The sharp absorption band of the control mix (C0) at wave number 3643-3638 cm^{-1} is related to the free OH^{-1} group coordinated to Ca^{2+}, i.e. free lime, Ca $(OH)_2$ or (CH). The intensity of the free lime absorption band of the control (C0) is detected obviously. This is mainly attributed to normal hydration process as well as the active pozzolanic effect of CSPW that initiates and improves the rate of hydration [32,46-48,53-56]. After exposure to firing, this peak was completely disappeared. The intensity of the broad absorption band at wave number 3782-3051 cm^{-1} which is due to the OH^{-1} group associated to H^+ bond (H_2O) was shrinkage with firing due to the gradual deficiency of combined water, which in turn reflected negatively on CSHs, i.e. the intensity of the absorption band of CSHs was decreased due to firing. The two absorption bands at approximately 1719-1682 and 1571-1143 cm^{-1} are related to the main silicate band involve Si-O stretching vibration bands of CSH. The three absorption bands at 1125-691 cm^{-1} characterizing CO_3^{2-} and SO_4^{2-} are slightly broken down soon after firing exposure, i.e. the rate of carbonation and sulfonation of CSH and /or CSAH was diminished.

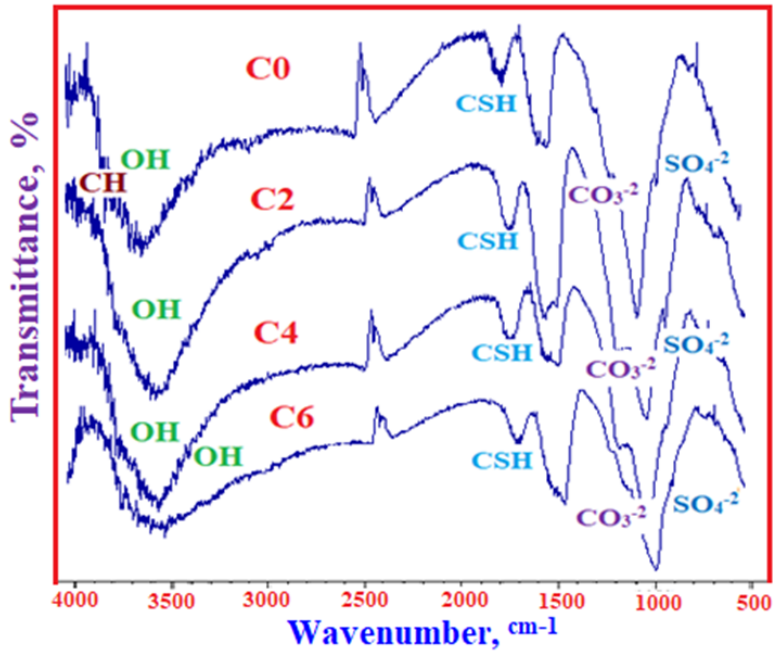

Fig. 7 *FT-IR spectra of the cement pastes C0, C2, C4 and C6 fired up to 25, 200, 400 and 600 °C*

3.4 SEM Images

The SEM images of the optimum hardened cement pastes containing 20 wt. % CSPW hydrated up till 90 days at the ambient temperature that was considered as the control (C0) [31] was subjected to different firing temperatures (200, 300, 400, 500 and 600 °C) are illustrated in Fig. 8. Hydrated phases of cement pastes as trisulphoaluminate hydrate (C_3A. 3 $CaSO_4$. 32H_2O) that known as ettringite (AFt) which appeared as needle-like crystals are still shown with C0 (A), but so little in C2 (B) after firing. Calcium hydroxide (CH) was also appeared in C0 (A) and C2 (B). Calcium silicate hydrates (CSHs) are clearly observed in C0 (A), C2 (B), C4 (C), but decreased and some were broken down after firing (C6), respectively. The size and amount of CSHs gradually decreased with firing temperatures. The AFt crystals and CH contents are clearly diminished or reduced till completely disappeared, while CSHs little improved and enhanced, i.e. the disappearance of AFt and CH was compensated by the formation of additional CSHs on firing. The SEM image of C4 fired up to 400 °C showed a higher densification on firing due to the lower voids or pores and the higher improvements of CSHs, while that of C6 (D) showed minor cracks due to the creation of more pores resulting from the breakdown of some hydro-silicate phases.

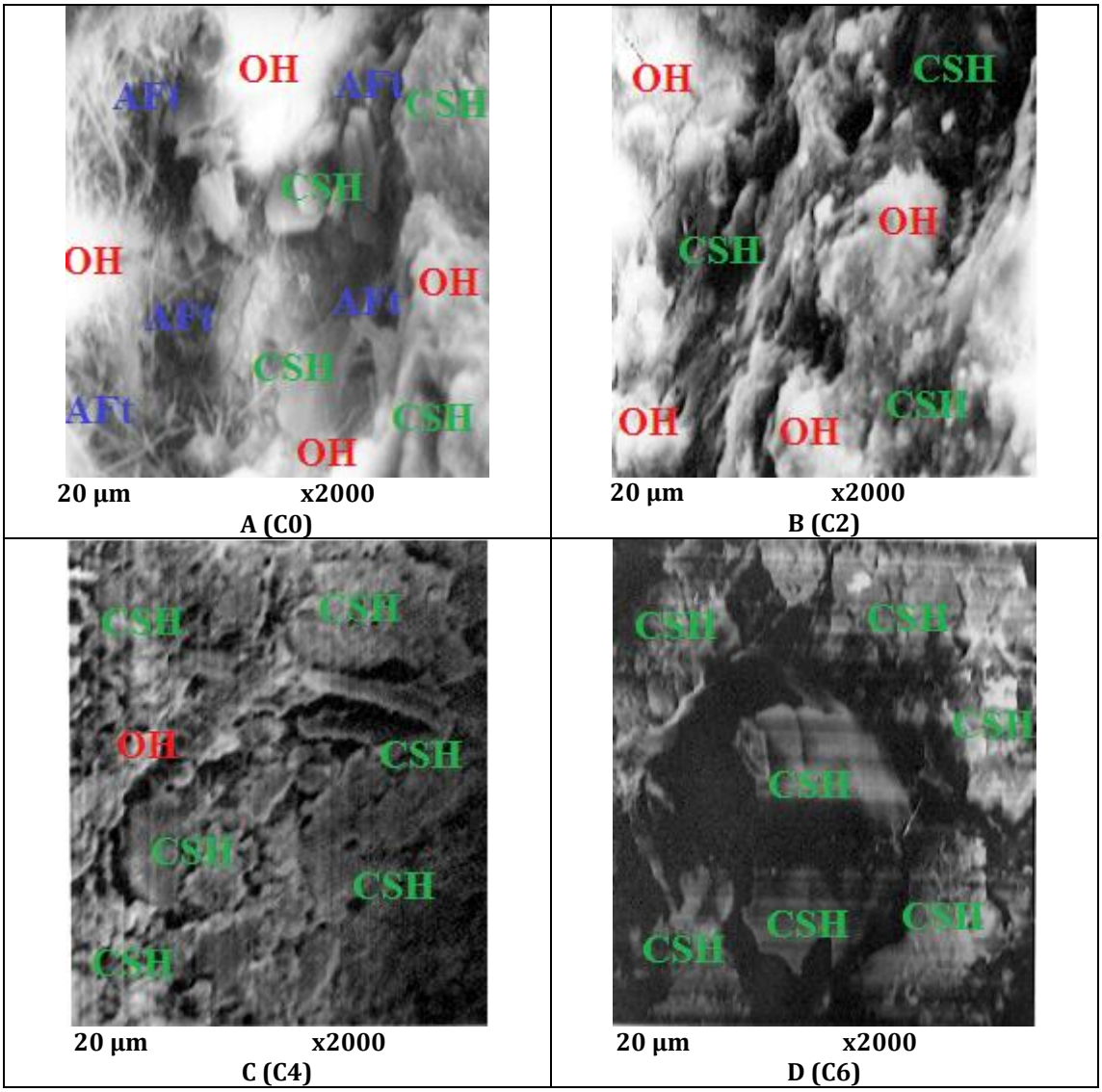

Fig. 8 *SEM images of C0, C2, C4 and C6 cement pastes with and without CSPW after the exposure to firing temperatures*

4. Conclusion

At ambient temperature, all characteristics of the various cement pastes are improved with the hydration times up to 90 days due to the normal hydration process of the major phases of the cement with water to produce CSH, and also to the active pozzolanic reactions of CSPW with Ca $(OH)_2$ to form additional CSHs. The replacing of cement with CSPW by 20 wt. % led to improve and enhance workability, bulk density and both flexural and compressive strengths, while decreased the total porosity and water absorption. Inclusion of 20 wt. % CSPW had given the better physical and mechanical properties at ambient temperature. Therefore, it was selected to be the optimum content. When the optimum cement mix was exposed to high firing temperatures up to 600 °C, all physical and mechanical properties were improved and slightly increased compared to that of the control. But this was continued only up to 400 °C and then was adversely decreased with further increase in the firing temperature more than 400 °C. All of the obtained results are in a good agreement with each other at all elevated temperatures. The FT-IR spectra demonstrated that the CSHs had improved on firing, while the free lime and Aft were disappeared completely. The SEM microscopy showed modification and improvements in the crystal phase growth of CSHs and the complete disappearance of free lime and ettringite (AFt) phase with firing temperatures. The recovery of CSPW as cement replacing material will lead to the reduction of CO_2 emission into the atmosphere. Thus, it can help to protect environment as well as to conserve natural resources. This will lead to a cleaner environment.

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Conflict of Interest

The authors declare that they do not have any commercial or associative interest that represents a conflict of interest in connection with the work submitted.

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