

The Performance of Portland Cement Pastes (OPC) Incorporated with Ceramic Sanitary Ware Powder Waste (CSPW) at Ambient Temperature

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Abstract

The physical and mechanical performance of Portland cement pastes (OPC) incorporated waste ceramic sanitary ware powder waste (CSPW) at ambient temperature has been investigated. Cement mixes were prepared by replacing the cement with CSPW at proportions of 0, 4, 8, 12, 16 and 20 wt. %. The results showed that the water of consistency and setting times (initial and final) were increased with the gradual addition of CSP, whereas the water absorption and total porosity were decreased, while the bulk density slightly was enhanced but only up till 16 wt. % CSPW. The same trend was displayed with mechanical properties, where the flexural (FS) and compressive strengths (CS) of the cement specimens were also improved and enhanced till 16 wt. %, but then all were decreased with further addition of CSPW. The experimental results indicated that the CSPW has a potential to be successfully recycled in the OPC pastes as a partial replacement merely up to 16 wt. % which had a better performance than the blank at ambient temperature. The FT-IR spectra illustrated that the amount of CSHs was increased, while that of free lime content decreased. This was conformed by SEM microscopy. Moreover, the recovery of CSPW contributes to both reducing environmental pollution and CO₂ emissions.

1. Introduction

The increase in the various solid wastes or residues in the cities particularly those do not decompose easily neither in nature, nor by weathering factors for thousands of years created the growth of both ground and air pollutions. The recycling of these solid wastes is so important for environmental and economical alternative solutions that it is becoming of a great importance. So, worldwide environmental concerns are becoming increasingly more significant [1]. The shortage of natural resources and the increasing levels of greenhouse gas emissions into the atmosphere scope a scenario that certainly forces mankind to create new sustainable alternatives for the industrial sector [2-4]. The use of different kinds of industrial waste and residues as alternative materials in the construction industry can contribute in a very effective way to such sustainability [1]. However, recycling of non-biodegradable wastes is especially too hard as ceramic sanitaryware powder waste (CSPW). Because CSPW biodegrades slowly and makes up a large part of total production, it's important to reuse it [5]. This would help the economy due to its reduction of harmful environmental effects. In Egypt, various kinds of sanitary wares were produced. These products are vitrified products of a mixture of clay, quartz, and feldspar that fired at temperature range 1100-1300 °C [6-12].

On the other hand, researchers are often working on the possible use of CSPW in the construction sector [3,5,13]. The reuse of CSPW as a cement replacing material in cementitious composites can be considered as an environmentally friendly alternative. Additionally, Ordinary Portland cement (OPC) is known as the primary reason for the insufficient environmental sensitivity of concrete. This is due to that the manufacture of OPC is responsible for > 5% of the global CO₂ emissions [14,15]. The recycling of CSPW as a cement replacing material can be considered as an alternative to restrict the environmental impacts and depletion of resources. There are very few previously published works about the reuse of CSPW as a substituting material for OPC [16-21]. Mortars incorporated up to 25 wt. % CSPW achieved mechanical strength results similar to that of other cement replacement materials [16], i.e. granulated blast furnace slag (GbfS) and fly ash (Fa). In addition, the compressive strength of mortars incorporated CSPW by 25% was slightly higher than that of the control mortars. Many investigators studied the reuse of concrete demolition waste and CSPW as substituting material for cement [17-23]. Some researchers have stated that though CSPW significantly reduced the cement hydration, it influenced the rheology of cement pastes. Therefore, it could be employed at a low level that acts as a pozzolan [24-32].

The essential purpose of the current investigation is to study and evaluate on the recycling of ceramic sanitaryware waste (CSPW) and its effect as a sustainable replacing material in Portland cement pastes (OPC). Therefore, this study investigated the effect of CSPW substitution of up to 30 wt. % by cement on the physical and mechanical properties comparing with the control (OPC) without CSPW.

2. Experimental Procedure

2.1 Raw Materials

The used raw materials in the present 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 of 3500 cm²/g and its commercial name is known as “Asmant El-Momtaaz”. 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 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/cm³, 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, wt. %

Materials	Oxide								
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	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	C ₃ S	β-C ₂ S	C ₃ A	C ₄ AF
OPC	46.81	28.43	5.90	12.56

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

Properties	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 are 6 cement batches from OPC and CSPW as 100:0, 96:4, 92:8, 88:12, 84:16, 80:20 and 76:24 having the symbols: W0, W1, W2, W3, W4, W5 and W6, respectively. The blending process of the various cement blends was done in a porcelain ball mill using 2-4 balls for two hours to assure the complete homogeneity of all cement blends. During casting, 1 % polycarboxylic ether as a high reducing water superplasticizer admixture was added to the mixing water which in turn added to the prepared cement mixes so as to avoid the agglomeration of the

nanoparticles of the used CSPW or OPC. It was applied due to its higher activity than other conventional ones, which contains several free carboxylic groups that helps greatly to improve cement dispersion.

The standard water of consistency (WC) as well as setting time (initial and final) of the various cement pastes were directly determined using Vicat Apparatus [33-37]. The cement pastes were then cast using the predetermined water of consistency, moulded into one-inch cubic stainless steel moulds ($2.5 \times 2.5 \times 2.5 \text{ cm}^3$) 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 moulds was smoothed using a suitable spatula. Thereafter, the moulds were kept in a humidity chamber for 24 hours at $95 \pm 2 \text{ RH}$ and room temperature ($22 \pm 1 \text{ }^\circ\text{C}$), demoulded in the following day and soon immersed in water till the time of testing at 1, 3, 7, 28 and 90 days. The water absorption (WA), bulk density (BD) and total porosity (δ) of the hardened cement pastes were determined [35-37].

The mechanical properties in terms of flexural strength (FS) and compressive strength (CS) of the various hardened cement pastes [38,39] were measured. The FS could be carried out using the three-point adjustments system (Fig. 1).

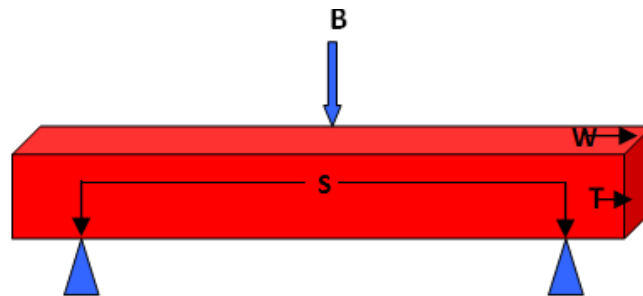


Fig. 1 Schematic diagram of bending strength, *B*: Beam or loading of rupture, *S*: Span, *W*: Width and *T*: Thickness

Thereafter, about 10 grams of the broken specimens from the compressive strength were first well ground, dried at $105 \text{ }^\circ\text{C}$ for an hour, and then were placed in a solution mixture of 1:1 methanol: acetone to stop the hydration [40,41]. The chemically-bound water content was measured, where about one gram of the sample was first dried at 105°C for 24h, and the chemically-bound water content (BW_n) at each hydration interval was determined on the basis of ignition loss at $1000 \text{ }^\circ\text{C}$ for 30 min. soaking [34,37,42].

The pozzolanic activity was detected by measuring the free lime content (FL_n) of the hydrated samples pre-dried at $105 \text{ }^\circ\text{C}$ for 24h. About 0.5 g of the sample + 40 ml ethylene glycol → heating to about 20 minutes without boiling. About 1–2 drops of pH indicator were added to the filtrate and then titrated against freshly prepared 0.1N HCl until the pink colour disappeared. The 0.1N HCl was prepared using the following equation: Where, W_n , W_1 and W_2 are combined water content, weight of sample before and after ignition, respectively. The free lime content of the hydrated samples pre-dried at 105°C for 24h was also determined. About 0.5g sample +40 ml ethylene glycol → heating to about 20 minutes without boiling. About 1–2 drops of pH indicator were added to the filtrate and then titrated against freshly prepared 0.1N HCl until the pink colour disappeared. The heating and titration were repeated several times until the pink colour did not appear on heating [34,40,41].

The obtained results are 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 $4000\text{-}400 \text{ cm}^{-1}$ 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- α 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 of Consistency and Setting Time

Fig. 2 shows the water of consistency and setting times (initial and final) of the various cement pastes containing different ratios of CSPW (W0-W6). It is obvious that the water of consistency enhanced as the CSPW content increased. This is mainly contributed to that the CSPW gradually absorbs water too much to form standard cement pastes [34-38]. On the other side, as the water of consistency increases, the setting time increases too. Therefore, the CSPW is very voracious for water. The measured water of consistency was always used in all tests.

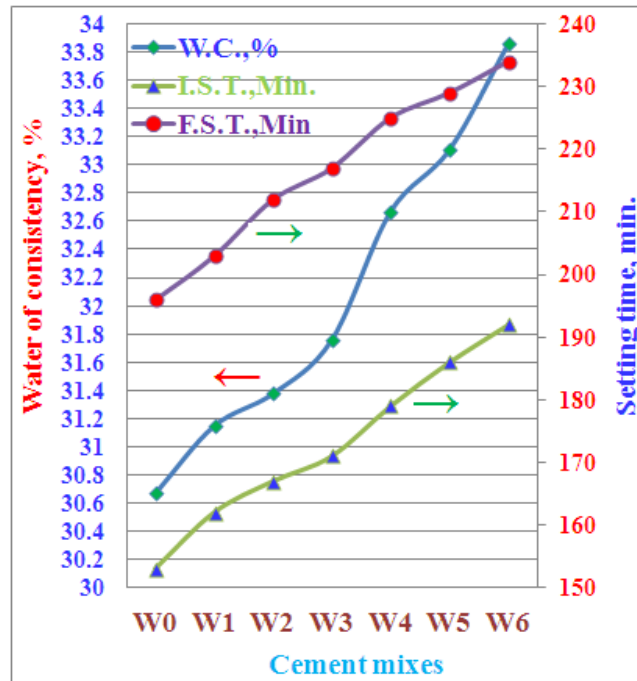


Fig. 2 Water of consistency and setting times (initial and final) of the various cement pastes with and without CSPW

3.1.2 Water Absorption

The results of water absorption (WA) of the various hardened cement pastes at all water curing ages are shown in Fig. 3. The WA of the blank sample (W0) was 28.25 %. This value was decreased with the increase of CSPW content only up to 16 % (W5), and then slightly increased with the further increase of CSPW content (W6). The decrease of WA is essentially attributed to the higher compaction effect resulting from the higher grain 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 [38,39]. The increase of WA is often due to the large deficiency of the main binding material of the cement [40-43]. Hence, the larger ratios of this CSPW must be avoided due to its adverse action.

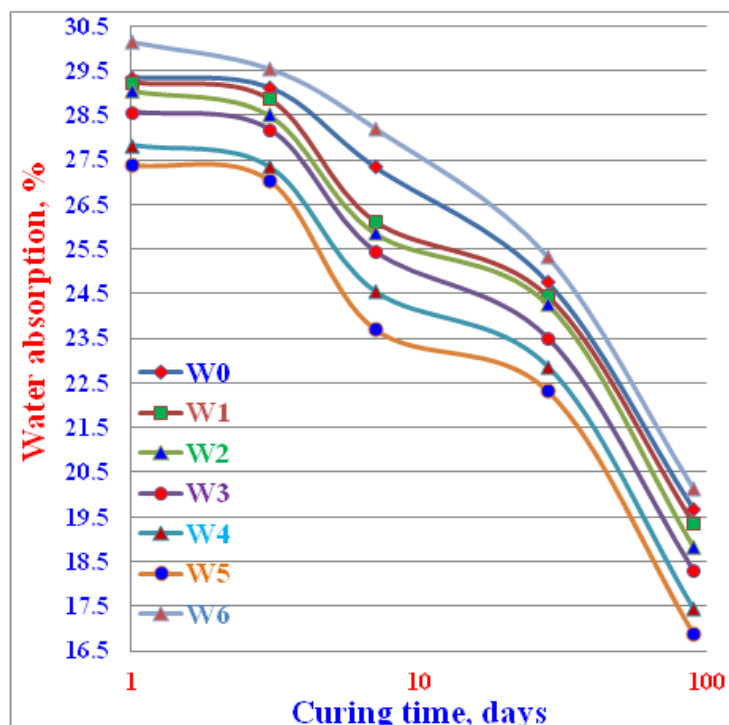


Fig. 3 Water absorption of the various cement pastes with and without CSPW hydrated up to 90 days

3.1.3 Total Porosity

The results of total porosity of the various hardened cement pastes with and without CSPW (W0-W5) cured up to 90 days at ambient temperature are illustrated in Fig. 4. The total porosity of the blank (W0) at any hydration age was decreased with the increase of CSPW content, but only up to 16 %, and then reincreased with any further addition of CSPW (W5). The decrease of total porosity is mainly due to the normal hydration of the main four phases of the cement [44-47]. Moreover, the pozzolanic reactivity of CSPW with the resulting $\text{Ca}(\text{OH})_2$ coming from the hydration process of the silicate phases of the cement producing additional CSH that precipitated into the pore structure. This reduced the total porosity [40,46-49]. The reincreased values of the total porosity with higher content of CSPW than 16 % (W5) is mainly attributed to the larger deficiency of the main binding material which is responsible for the hydration process [35,35,49-51]. So, the higher content of CSPW than 16 % must be avoided, i.e. the optimum content of CSPW is only 16 wt. %.

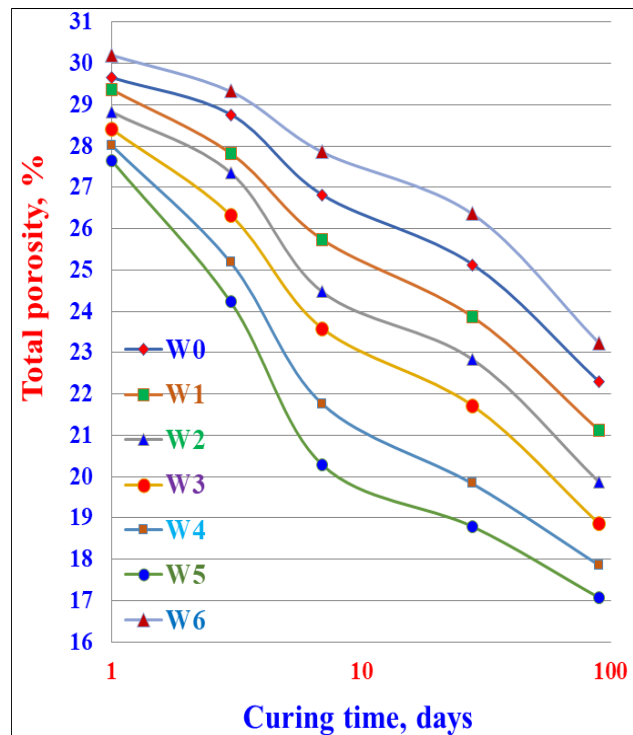


Fig. 4 Total porosity of the various cement pastes with and without CSPW hydrated up to 90 days

3.1.4 Bulk Density

The results of bulk density of the various hardened cement pastes with and without CSPW (B0-B5) hydrated up to 90 days of curing time are presented in Fig. 5. Generally, the bulk density slightly increased with the incorporation of CSPW till 12 % (B4), and then decreased by the further increase of CSPW (B5). The increase of bulk density is mainly due to the deposition of the formed CSH from the hydration of the main phases of the cement and also the additional CSH that resulted from the pozzolanic reactions of CSPW particles with the free lime, $\text{Ca}(\text{OH})_2$ coming from the hydration of the calcium silicate phases of the cement [39,40,45]. The slight reduction in the bulk density is due to the lower specific gravity of the CSPW compared to that of OPC [34,44,45,51]. Additionally, the hardened cement pastes with CSPW showed lower water absorption and lower porosity especially at 90 days. 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 water absorption and total porosity were resulted [35,37,43-46]. It is good mention that the rate of the normal hydration process of the OPC is often decreased due to the addition of CSPW at the expense of the OPC material. So, the formed amount of CSH was reduced, This was compensated by the pozzolanic reactions among CSPW and free lime of $\text{Ca}(\text{OH})_2$ resulting from the hydration of calcium silicate phases of the cement.

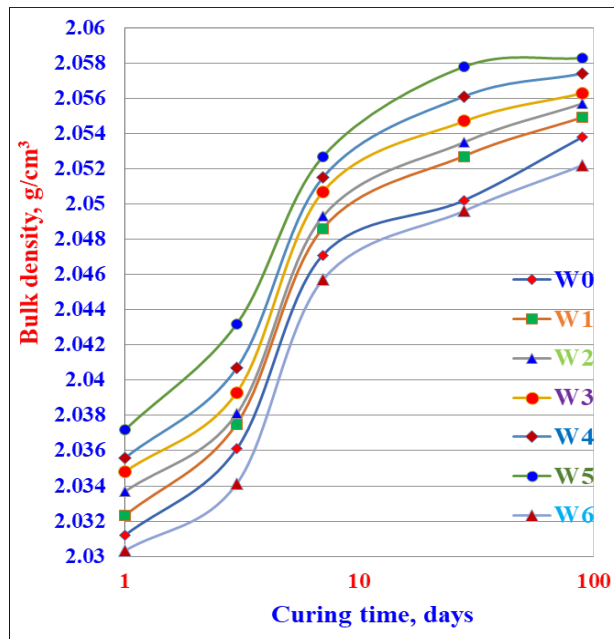


Fig. 5 Bulk density of the various cement pastes with and without CSPW hydrated up to 90 days

3.2 Chemical Properties

3.2.1 Chemically Bound Water Content

Fig. 6 shows the chemically-bound water contents of the various cement pastes with and without CSPW (W0-W5) are drawn versus curing time. The bound water content of the blank (W0) was improved and enhanced as the hydration time progressed up to 90 days. This is principally due to the occurrence of the normal hydration process of the major phases of the cement [39,40]. With the substitution of CSPW, the bound water content was further improved and increased till 16 % CSPW content (W4), and then was suddenly decreased (W6). The increase of bound water contents was contributed to both normal hydration of cement phases and pozzolanic reactivity of CSPW with the resulting $\text{Ca}(\text{OH})_2$ from the first process [13,52]. The decrease of bound water content was distinctly due to the large deficiency of the main binding material of the cement [28,53,54]. As a result, the optimum CSPW content is only 16 % (W4). Hence, the higher CSPW content more than 16 % is undesirable.

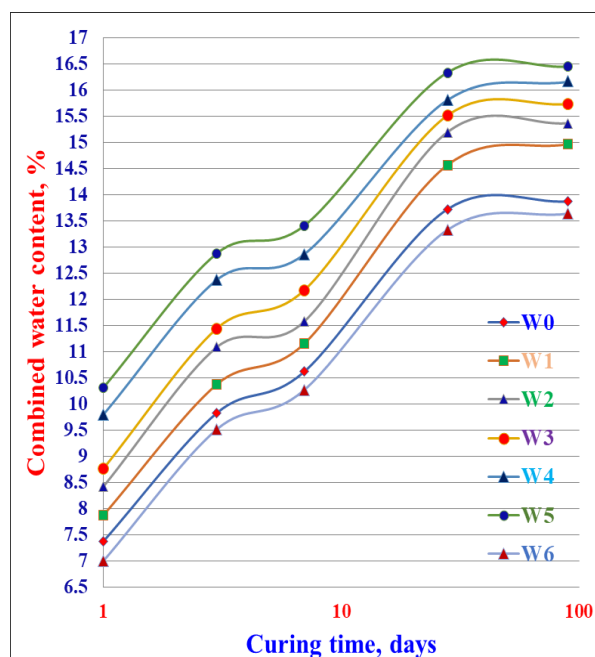
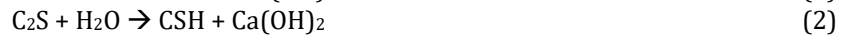
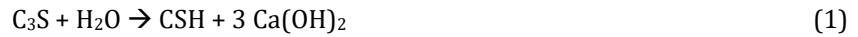


Fig. 6 Chemically-bound water content of the various cement pastes with and without CSPW cured up to 90 days

3.2.2 Free Lime Content

The free lime contents of the various cement pastes containing different level of CSPW (W_0 - W_5) cured up to 90 days, are drawn as a function of curing time in Fig. 7. The free lime content of the reference control (W_0) gradually increased with the hydration time up to 90 days. This is mainly attributed to the normal hydration of the di- and tricalcium silicates of the cement (β - C_2S and C_3S) to form CSH [39,40] as follows:



With the incorporation of CSPW at the expense of the cement, the free lime content slightly decreased with the time of hydration till 90 days. This is essentially contributed to the pozzolanic reactivity of the CSPW with the resulting free lime, $Ca(OH)_2$ forming additional CSHs [1,10,37,43,55].

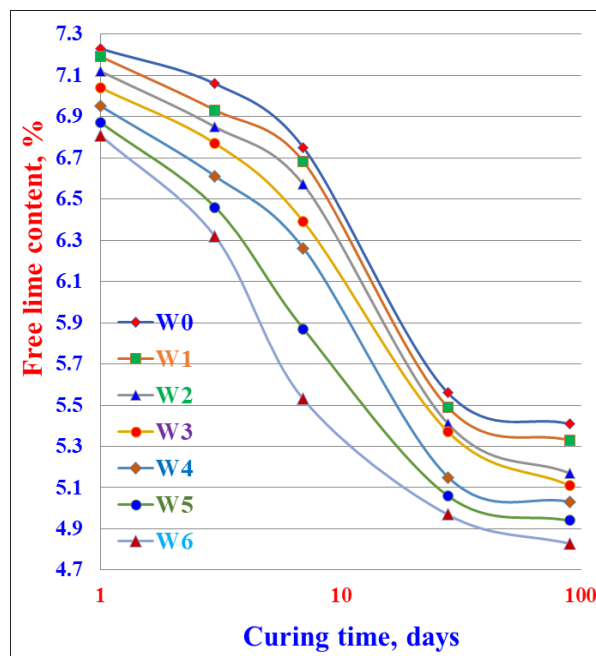


Fig. 7 Free lime contents of the various cement pastes with and without CSPW cured up to 90 days

3.3 Mechanical Properties

3.3.1 Flexural Strength

Fig. 8 illustrates the results of flexural or bending strength of the various cement pastes incorporating different levels of CSPW (W_0 - W_5) cured and hydrated in water at normal or room temperature up to 90 days. Generally, the flexural strength of the various hardened cement pastes improved and enhanced with the hydration time up to 90 days. This is mainly due to the normal hydration process of the main phases of the cement [39,40]. The flexural strength was further increased as the CSPW content increased, but only up to 16% (W_4), and then decreased with any further increase of CSPW content (W_5). The increase of flexural strength with the curing ages could be contributed to the slow pozzolanic reaction of the CSPW during the early hydration ages (1-7 days), along with the ongoing hydration of the cement. This often resulted in a more densification of the microstructure of the cement [1,13,56-59]. The increase in the flexural strength of the different mixes up to 16% replacement of cement with CSPW can be explained by its possible pozzolanic reaction coupled with the pore filling effect of the CSPW. The decrease of the flexural strength at higher inclusion of CSPW (>16%) could be attributed to the dilution effect of the CSPW on cement, which was created in a lower $Ca(OH)_2$ available for the formation CSHs. This level is related to the $Ca(OH)_2$ amount in the matrixes of cementitious materials. At the optimum level, almost all the $Ca(OH)_2$ could be reacted with the SiO_2 in the pozzolans. When the pozzolans are used more than the optimum level, there will be extra pozzolan available in the matrix freely that does not react with $Ca(OH)_2$ and water. Therefore, the flexural strength may become stable or little decreased [52,60-66]. Consequently, the optimum content of CSPW was 16% at all ages of hydration because at this content a larger level of pozzolanic reactivity and microfill effect was created.

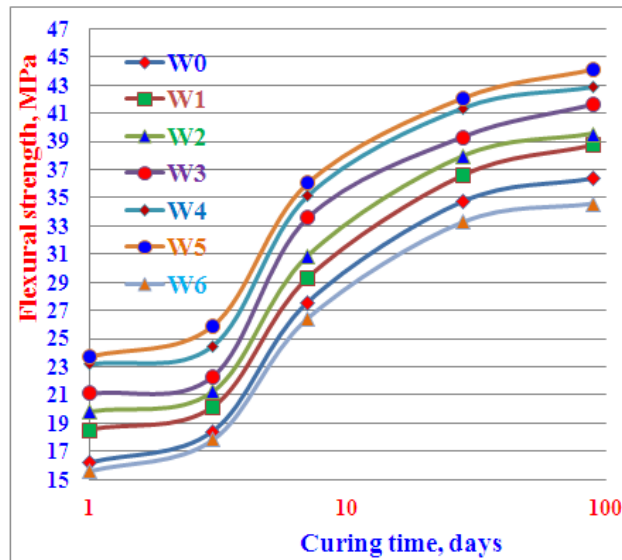


Fig. 8 Flexural strength of the various cement pastes with and without CSPW cured up to 90 days

3.3.2 Compressive Strength

The results of compressive strength of the various cement batches containing CSPW (W₀-W₅) cured and hydrated at the ambient or room temperature up to 90 days are shown in Fig. 9. The compressive strength of the various hardened cement pastes increased with curing or hydration time up to 90 days. This is principally due to the normal hydration process of the main major phases of the cement [34,39,40,67-69].

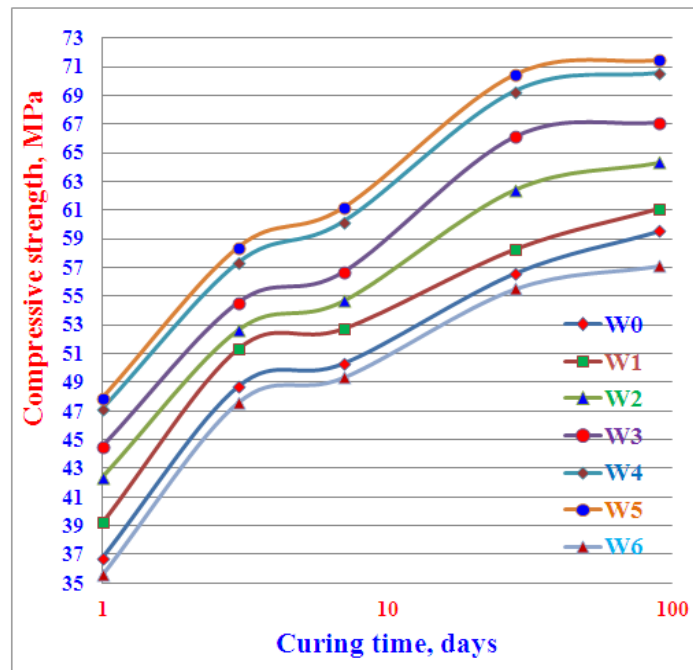


Fig. 9 Compressive strength of the various cement pastes with and without CSPW cured up to 90 days

The compressive strength was also increased with the increase of CSPW content, but only up to 16 % (W₄), and then decreased with any further increase of CSPW content (W₅). The increase of the compressive strength is mainly attributed to the filling performance and pozzolanic reactivity of CSPW material [43-46,53,68-70], whereas the decrease of compressive strength is due to the detrimental characteristics of the higher specific surface area of CSPW that created the agglomeration of the cement particles. This poor performance effect is associated with the lower cement content that influences the direct reduction of the primary hydration products responsible for the compressive strength (CSH). This was negatively reflected on the rate of Ca²⁺ release which was delaying the release of Ca (OH)₂ [28,57]. Therefore, it reduces its availability for the development of

pozzolanic reactions with the CSPW. Hence, the ideal limit of CSPW to be incorporated in the OPC (W_0) is 16 %. The higher percentages (W_5), the filler effect and the availability of $\text{Ca}(\text{OH})_2$ in the hydration reaction are reduced [64,71], i.e. the higher substitution of CSPW is undesirable. The larger specific surface area of CSPW causes a microfilling effect resulting in the creation of additional nucleation sites and higher effective proportion of water for cement hydration [27,34,65]. Also, the low crystallinity of CSPW favours the development of pozzolanic reactions, allowing additional CSHs gel to be formed. So, the combination of these effects, the porous structure of the hardened cement pastes is refined [34,46,56-59,71]. Consequently, an improvement in compressive strength is noted. It could be concluded that the rate of the normal hydration of the OPC (W_0) is often declined due to the replacement of higher quantities of CSPW at the expense of the OPC, and therefore the quantity of the formed CSHs was reduced. This was compensated by the pozzolanic reactions among CSPW and free lime, $\text{Ca}(\text{OH})_2$ coming from the hydration of di- and tricalcium silicate phases of the cement (Equations 1 and 2). The unreacted mineral particles may fill up the voids and increase the density of the cement paste [39,40]. The decrease of mechanical properties may be due to the incomplete filled voids and the reduction of pozzolanic reactions, whose amount depends on the cement dosage [13,58,59,66]. Also, the CSPW caused a compact microstructure, which is considered to be the main cause for the highest compressive strength [28,43-46,67,71]. An upper limit of 16% CSPW content was proposed to be incorporated in OPC for the sustainable production.

3.3.3 FT-IR Spectra

The FT-IR spectra of the cement pastes W_0 , W_2 and W_4 hydrated up to 90 days are illustrated in Fig. 10. The sharp absorption band at wave number $3640\text{-}3639\text{ cm}^{-1}$ is related to the free OH^{-1} group coordinated to Ca^{2+} , i.e. free lime, $\text{Ca}(\text{OH})_2$ or (CH). The intensity of the free lime absorption band of the control (W_0) is detected obviously, which was gradually decreased with the incorporation of CSPW (W_3 and W_4). This is mainly attributed to the active pozzolanic effect of CSPW that initiates and improves the rate of hydration [43-45,72-75]. The broad absorption band intensity at wave number $3785\text{-}3048\text{ cm}^{-1}$ which is due to the OH^{-1} group associated to H^+ bond (H_2O) increased with the incorporation of CSPW due to the absorption of large quantity of water to form CSH. The intensity of the absorption band of CSH was improved and increased with CSPW content. The two absorption bands nearly at $1722\text{-}1679$ and $1568\text{-}1145\text{ cm}^{-1}$ are related to the main silicate band involve Si-O stretching vibration bands of CSH. The three absorption bands at $1127\text{-}685\text{ cm}^{-1}$ that are characterizing CO_3^{2-} and SO_4^{2-} , enhanced with CSPW content. This may be due to the rate of carbonation and sulfonation of CSH and /or CSAH.

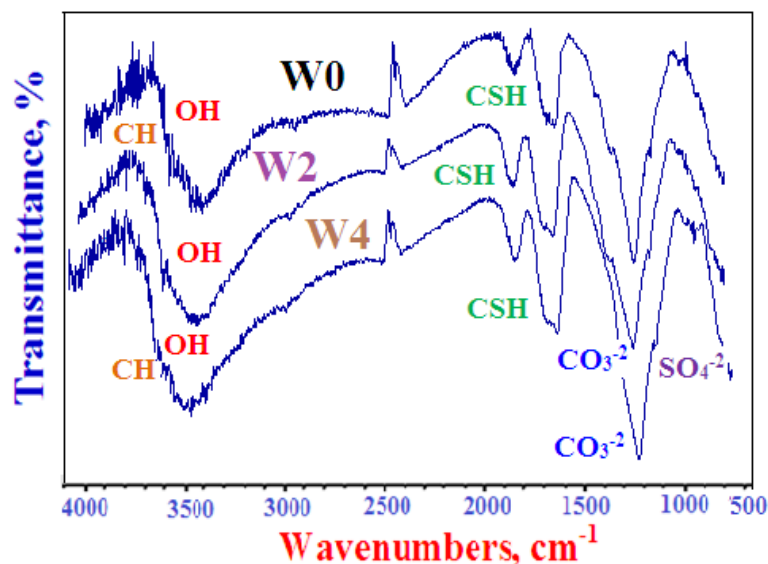


Fig. 10 FT-IR spectra of the cement pastes W_0 , W_2 and W_4 cured at 90 days

3.3.4 SEM Images

The SEM images of the various cement batches containing CSPW ($W_0\text{-}W_5$) cured and hydrated at the ambient or room temperature up to 90 days are represented in Fig. 11. The hydrated phases of cement paste as trisulphoaluminate hydrate ($\text{C}_3\text{A} \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$) that known as ettringite (AFt) which appeared as needle-like crystals as shown with W_0 (A) and W_1 (B), the calcium hydroxide (CH) as in W_0 (A), W_1 (B) and W_2 (C) and the calcium silicate hydrate (CSH) are clearly observed in W_0 (A), W_1 (B), W_2 (C), W_3 (D), W_4 (E) and W_5 (F),

respectively. The size and amount of CSHs gradually increased with the addition of CSPW. The AFt crystals as well as CH content are clearly diminished and reduced till completely disappeared with (W₄), while CSH increased, i.e. the disappearance of AFt and CH was compensated by the formation of additional CSH. The SEM image of W₄ showed a higher densification due to the lower voids or porosity and the higher formation of CSHs, while that of W₅ (F) showed minor cracks due to the existence of more porosity. This is supported by the obtained results of physical, chemical and mechanical properties as bulk density, porosity, flexural and compressive strengths. The occurrence of cracks in W₅ (F) is mainly due to the higher amounts of the CSPW leading to more porosity which adversely affected on the properties of the cement pastes.

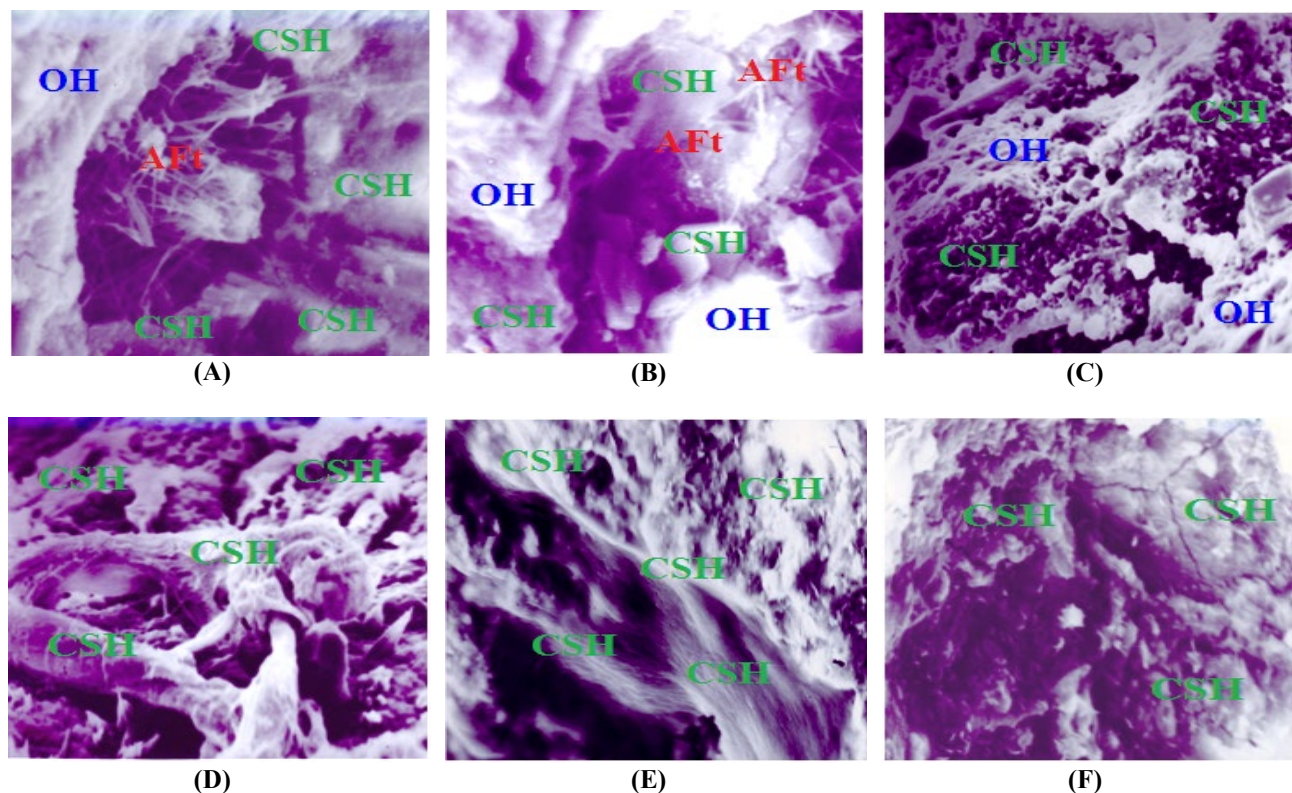


Fig. 11 SEM images of the various cement pastes with and without CSPW hydrated up to 90 days

4. Conclusions

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 $\text{Ca}(\text{OH})_2$ to form additional CSHs. Replacing of cement with CSPW up to 16 wt. % led to improve and enhance workability, bulk density and both flexural and compressive strengths, while decreased the total porosity and free lime content. Inclusion of 16 wt. % CSPW had given the better physical and mechanical properties. Therefore, it was selected to be the optimum content. The further addition of CSPW more than 16 wt. % led to an adverse effect on all properties. All of the obtained results are in a good agreement with each other. The FT-IR spectra confirmed the improving of hydration process with the replacement of CSPW and the increase of CSHs, while the free lime was decreased. The SEM images indicated the gradual increase of CSHs and the decrease of free lime content. 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.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: **study conception and design:** H.H.M. Darweesh, H. Abu-El-Naga; **data collection:** H.H.M. Darweesh; **analysis and interpretation of results:** H.H.M. Darweesh, H. Abu-El-Naga; **draft manuscript preparation:** H.H.M. Darweesh, H. Abu-El-Naga. All authors reviewed the results and approved the final version of the manuscript.

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