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Effect of curing temperatures on the hydration of cement pastes containing nanograin size particles of sanitary ware ceramic powder waste

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Abstract

Effect of curing temperatures on the hydration, physical and mechanical properties of the cement pastes incorporated 20% ceramic sanitary ware powder waste (CSPW) was investigated. Results showed that the curing temperatures initiated and activated the hydration process of the cement pastes. Total porosity was decreased till 60 °C. This was reflected positively on water absorption and bulk density. The same trend was displayed with mechanical properties. The specimens of cement pastes that were hydrated at curing temperature 60 °C recorded the best results, where the total porosity (15.13%) and water absorption (14.97%) are the lowest, while the bulk density (2.0903 g/cm³) was the highest. Also, the flexural and compressive strengths achieved the highest values as 46.83 and 73.42 MPa, respectively. Suddenly, all the characteristic properties were slightly adversely affected with any further increase of curing temperature > 60 °C. Consequently, the optimum water curing temperature is 60 °C, and any higher curing temperature over than 60 °C must be avoided.

Keywords: Cement, sanitary ware powder waste, temperature, hydration, water absorption, density, porosity, strength

Introduction

Manufacturing process of Ordinary Portland cement (OPC) produces large emission amounts of CO_2 . Emission of CO_2 is due to the decomposition of limestone during firing inside the rotary kiln at \geq 1400 °C, into CaO and CO₂, where about 60% of CO₂ emissions were coming. Cement industry is responsible for about 8% of the total CO₂ emissions worldwide ^[1-2]. This created the most serious environmental problem as global warming. So, it is hoped to reduce this rate of CO₂ emissions as possible and hence, alternative materials to cement are needed. Blended cements, in which a portion of the cement is replaced by industrial wastes or by-products, i.e. granulated blast furnace slag (GbfS), fly ash (FA), silica fume (SF), agro/wastes as rice husk ash (RHA), sugar cane bagasse ash (SCBA), wheat strew ash (SWA), sun flower ash (SFA), saw dust (SD) and many other agricultural ashes as well as concrete waste [3-20]. These eco-friendly binders are very important because it can produce very safe cementing materials to environment that linked to conventional Portland cement. In recent years, the incorporation of ceramic powder wastes (CPW) in OPC as a pozzolanic material has gained popularity because the use of CPW can improve the properties of cementing materials and reduce the total embodied carbon. Several studies [21-38] have shown that the incorporation of CPW in cementing materials has a positive effect on the properties of the hardened cement pastes, concretes and mortars, as heat of hydration, density, absorption, porosity, mechanical properties, durability and resistance to firing. It is well known that the variation in temperature between day and night, and also between seasons have a vital effect on the rate of hydration of the major phases of the cement [24-30]. Therefore, the influence of curing temperatures on hydration of cementing materials was investigated. It could be occurred during the precast process where the high temperatures accelerated the hardening of the concrete and its activation energy [37-39]. To activate this energy, several techniques could be used as thermal activation ^[40], mechanical activation by increasing the specific surface area ^[11-13] and chemical activation by adding activators as Ca (OH)₂ and Na₂SO₄^[41]. Though the positive effect of pozzolanic materials on the mechanical properties has already been demonstrated at 20 °C ^[18, 28-30], the effect of temperature on the

hydration mechanism, microstructure and resistance to the compression

of cement pastes is rarely discussed. Effect of curing temperature on the hydration process of the main phases of clinker as alite (C₃S), belite (β -C₂S), ferrite (C₄AF) and aluminate (C₃A)) for the pure cement ^[42] and also for blended cements ^[43]. Hydration of these phases at temperatures of 10-60 °C was evaluated. During the early ages, the compressive strength of the various mixes was directly proportional to the curing temperature, where the highest compressive strength was obtained after one year when were cured at low temperatures (10-20 °C). This inversion is due to the deposition of more compact hydration products around the grains of the hydrates which slows down the diffusion of the reagents. So, the hydration was delayed ^[44].

High temperatures caused the formation of large pores and the increase of the accumulated volume which has a negative effect on the mechanical properties of the specimen. The great reactivity of pozzolanic materials with Ca (OH)₂ makes it a good replacing material for cement due to its pozzolanicity ^[13, 45-48]. It was reported that the presence of certain activators as lime, chlorides, sulphates, etc. in the pozzolanic reactions can modify the kinetics of the pozzolanic reactions as well as the development of hydrates crystals ^[26, 49-53]. Accordingly, the effect of curing temperatures (25, 30, 40, 50 and 60 °C) on the hydration of Portland cement pastes incorporated 20 wt. % CSPW that was hydrated at ambient temperature (T0) was evaluated. To assess the effect of curing temperature, the heat of hydration, absorption, porosity, density and mechanical characteristics are evaluated. The obtained results are confirmed with fourier transform infrared spectra (FT-IR) as well as scanning electron microscopy (SEM) were conducted to characterize the hydration and microstructure of the cement pastes.

2. Experimental

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 cm²/g. T was measured by Air Permeability Apparatus ^[54].

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 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. %

Oxides Material	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na2O	K ₂ O	SO3	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 ₃ S	β-C ₂ S	СзА	C4AF
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, cm²/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 ^[55] and it was considered the control cement batch having the symbol T0. 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 the cement blend. During casting, 1% polycarboxylic ether as a high reducing water superplasticizer admixture was added to mixing water which in tuern 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% ^[56]. Cement pastes were then cast using the predetermined water of consistency, moulded into one inch cubic stainless steel moulds (2.5 x 2.5 x 2.5 cm³) 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±2 RH and room temperature (22±1 °C), demoulded in the following day and soon immersed in water till 90 days. The temperature of curing water was 25, 30, 35, 40, 45, 50, 55 and 60 °C. Then, water absorption (WA), bulk density (BD) and total porosity (δ) of the cured hardened cement pastes were determined ^[57]. Also, the mechanical properties in terms of flexural strength (FS) and compressive strength (CS) of the various hardened cement pastes [58, 59] 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.

3. Results and Discussions

3.1. Total porosity

The total porosity (TP) of the optimum hardened cement pastes (T0) incorporating 20 wt. % CSPW hydrated up till 90 days at the ambient temperature $(25 \text{ °C})^{[55]}$ which was let to hydrate at different curing temperatures (30, 35, 40, 45, 50, 55, 60 and 65 °C) are shown in Fig. 2. The TP of the

control (T0) hydrated up to 90 days at 25 °C was 17.07%. This value was decreased with the increase of water curing temperature till 60 °C, but it suddenly increased with the increase of water curing temperature (> 60 °C). The gradual decrease of TP is mainly due to the energy activation effect of the higher temperatures ^[60-61], which initiated, promoted and accelerated the hardening. This in turn improved the crystal growth of the formed phases ^[60-63]. This continued till 60 °C, and then TP increased again with any increase of

curing temperature (> 60 °C). The increased values of TP with higher curing temperature are mainly contributed to unsuitable environment for cement phases to hydrate normally and also may be the slight break down of the formed CSHs which created more and large pores ^[39-44]. So, the higher curing temperature than > 60 °C must be eliminated, i.e. the optimum curing temperature at which the lowest values of TP were achieved is 60 °C.



Fig 2: Total porosity of the cement pastes containing 20% CSPW hydrated at various water curing temperatures.

3.2. 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 ^[55] that was considered as the control (T0) was subjected to different water curing temperatures (25, 30, 35, 40, 45, 50, 55, 60 and 65 °C) are illustrated in Fig. 3. The WA of the control sample (T0) hydrated up to 90 days at 25 °C was 16.89%. This value was slightly decreased with increasing of the curing temperature but only up to 60 °C, and then slightly increased with any further increase of curing temperature (65 °C). 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. This reduced the pore structure of the hardened cement pastes. In addition, the activation and initiation influence of curing temperature that improved and decreased the total porosity which modified the crystal growth of the cured phases. This in turn decreased more the WA of the hardened cement pastes ^[61, 64]. But, the little increase of WA at > 60 °C curing temperature is often due to that the higher curing temperature caused the break down or slight damage to some CSH phases which led to the formation of more pores ^[55, 59-60]. Hence, the higher curing temperature (> 60 °C) must be avoided due to its adverse action.



Fig 3: Water absorption of the cement pastes containing 20% CSPW hydrated at various water curing temperatures.

3.3. 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 [55] which subjected to different curing temperatures (25, 30, 35, 40, 45, 50, 55, 60 and 65 °C) are represented in Fig. 4. The BD of the optimum hardened cement pastes hydrated up to 90 days was 2.0583 g/cm³. This value was slightly enhanced with curing temperature till 60 °C, and then decreased by further increase of curing temperature > 60 °C. Increase of BD is mainly due to the slight energy activation and initiation of curing temperatures which accelerated the rate of hydration and hardening that modified the microstructure and crystal growth of the formed CSHs ^[39-42, 60-62, 64], i.e. the exposure of the hardened cement pastes to curing temperatures activated, initiated and also increased the formed CSHs which precipitated in the pore volume of the samples. This decreased the micropores, improved and enhanced the BD ^[60, 62]. The slight reduction in the BD is due to the lower specific gravity of the CSPW and the breakdown some of some CSHs ^[60, 64, 65], the hardened cement pastes with CSPW showed both lower water absorption, lower porosity and higher bulk density especially up to 60 °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 water absorption and total porosity were resulted. So, the optimum curing temperature is 60 °C at which the best values of BD were achieved, and the higher ones > 60 °C must be prevented ^{[39-} 44]



Fig 4: Bulk density of the cement pastes containing 20% CSPW hydrated at various water curing temperatures.

3.4. Flexural strength

Figure 5 shows 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 (T0) ^[55] was subjected to different curing temperatures (25, 30, 35, 40, 45, 50, 55, 60 and 65 °C) to explore its effect on the hydration process. FS of the blank hardened cement pastes was 44.13 MPa. This value was improved and enhanced with curing temperatures only up to 60 °C, but then decreased with any further rise of curing temperature > 60°C. The increase of FS is mainly due to that the heating of curing water activated, initiated and promoted the thermal interactions between the different

ingredients of the samples so that it improved, enhanced and modified the formed CSHs. On the other side, the decrease of FS is essentially contributed to the decomposition of Ca $(OH)_2$ or may be due to the significant dehydration within the cement matrix modifying the physical properties and the dehydration of both calcium silicate hydrates (CSHs) that occurred with high heated curing water ^[67-70]. This caused the weakening of bonds of pastes, which in turn resulted in the destruction of micro-structural arrangements in the hardened cement pastes. So, the high temperature of curing water must be prevented.



Fig 5: Flexural strength of the cement pastes containing 20% CSPW hydrated at various water curing temperatures.

3.5. Compressive strength

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 (T0) ^[55] was subjected to varying curing temperatures (25, 30, 35, 45, 50, 55, 60 and 65 °C) are demonstrated in Fig. 6. CS of the various hardened cement pastes was first increased with curing temperatures, but only up to 60 °C, and then decreased gradually by rising the curing temperature. Increase of CS is principally due to the initiating effect of the heating temperatures that activated, promoted and modified the crystal growth structures of the formed CSHs ^[60-64, 67]. Decrease of CS at curing temperature > 60 °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 ^[68] by > 60 °C onward could be due to the decomposition of Ca (OH)₂ and dehydration of both calcium silicate hydrates (CSHs) that occurred at temperature beyond 60 °C^[60-62, 64]. 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 [69-72]

The sharp decline in CS at 65 °C curing temperature may be due to the decomposition of Ca(OH)₂ and dehydration of both calcium silicate hydrates (CSHs) that took place at temperature beyond 60 °C ^[60, 73]. This always caused the breaking down of bonds between particles of cement pastes that caused the destruction of 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 60 °C. The improved resistance against high temperatures with CSPW inclusion can be mainly attributed to the decreased amount of Ca (OH)₂ which in turn lowered its pozzolanic activity with CSPW ^[68-74]. Consequently, the higher curing temperature must be avoided



Fig 6: Compressive strength of the cement pastes containing 20% CSPW hydrated at various water curing temperatures.

4. Conclusion

Total porosity was gradually decreased with water curing temperature, i.e. as the temperature of curing water increased, the total porosity decreased. This was continued up to 60 °C. This in turn was positively reflected on water absorption and bulk density. Any further increase of curing temperature (> 60 °C), the total porosity and also all physical properties were slightly affected negatively. Mechanical properties in terms of flexural and compressive strengths also improved and enhanced, and always achieved the same trend as physical properties. Therefore, the optimum water curing temperature was 60 °C, and any more increase of curing temperatures > 60 °C was reflected adversely on the whole characteristics. So, the higher water curing temperature is undesirable. It can be recommended that during of casting cement pastes, mortars or even concretes, the mixing water must be previously heated up to 50-60°C before adding to the cement mixes.

Declaration of Competing 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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