EFFECT OF TEMPERATURE ON THE STRUCTURE AND STRENGTH PROPERTIES OF CEMENT PASTES CONTAINING FLY ASH ALONE OR IN COMBINATION WITH LIMESTONE

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This study aimed to investigate the effect of heat treatment on microstructure and phase composition of pastes containing fly ash (FA) and limestone (LS). Two groups of cement pastes were prepared. In the first group, Portland cement (OPC) has been partially replaced by 10, 20 and 30 wt.% FA (M.0, M.10, M.20, M.30). The second group of specimens is constituted by samples derived from M.20, in which, FA has been replaced by 5, 10, 15 and 20 wt.% LS (L.5, L.10, L.15 and L.20). After 28 and 366 days of water curing, the hardened cement pastes were dried at 105°C for 24 hours, subjected to thermal treatment for 3 hours at 250, 450, 650 and 800°C, then cooled to room temperature in the furnace switched off. The compressive strength and weight loss were determined. Scanning electron microscopy, differential scanning calorimetry and X-ray diffraction were used for identification of the changes occurring in the microstructure of the formed or decomposed phases. The results show that additional hydration of unhydrated cement grains, recrystallization, deformation and transformation of CSH phases were occurred. The changes in physical state of cement pastes were studied by measuring deterioration in the compressive strength with temperature.

INTRODUCTION

Concrete subjected to high temperature undergoes a series of severe changes in its chemical composition and physical structure. The effect of temperature on hydrated cement paste depends on the degree of hydration and moisture state. It undergoes physical transformations and chemical reactions, thereby causing progressive breakdown of cement gel structure. This change in structure has, as consequent loss in its load bearing capacity, reduced durability, increased of drying shrinkage and structural cracking. A sequence of events takes place during boating being the release of water vapour the main consequence, coming from vaporization of moisture. The transformation of C-S-H, dehydration of CH and ettringite, induced by high temperatures, affects the microstructure of cement paste. The capillary and total porosity increase and the nanoporosity associated with C-S-H gel decomposes [1-3]. In the temperature range 100-400°C, the interlayer C-S-H water and some chemically bound water of C-S-H and sulphoaluminate hydrates should be lost [4,5]. These changes occur primarily in the hardened cement paste starting from the dissociation of CH at 400-600°C, CaCO₃ dissociation to CaO and CO₂ accompanied with the re-crystallisation of non-binding phases from hydrated cement are dominant processes between 600 and 800°C and continue until the complete destruction of C–S–H gel at around 900°C. As a result of these changes, concrete gradually and sometimes sharply loses its mechanical strength and durability [6-8].

The addition of pozzolanic or supplementary cementing materials as partial replacements is one effective method of preparing high-performance concrete [9-11]. In general, these blending materials enhance the performance of cementing materials through pozzolanic reaction with filling effect.

The use of limestone aggregates has been common and elsewhere for many decades. Indeed in recent years it has become common practice to incorporate fine limestone dust as a minor addition constituent in the cement paste, where or not a coarse limestone aggregate is also used to make concrete. The Canadian Standard Association, under CAN3-M83, allows up to 5% addition of limestone for normal Portland cement, so long as the limestone is of a good quality for the manufacture of the cement [12]. In the USA there is a proposal to modify ASTM specification for Portland cement (C150) to allow up to 5% limestone to be interground with cement clinker into Portland cement. The European standard allows the use of the term "Ordinary Portland cement" with incorporation of up to 5% or less limestone, while the term (Portland limestone cement) is used for cement containing 6-35% limestone replacement of Portland cement.

Limestone (LS) plays an important role in the hydration of C_3A , and also in the hydration of C_3S or β -C₂S in the presence of CaSO₄ and CH. Fine particles of LS fill the space between the cement particles by the formation of carboaluminate phases [13]. LS filler accelerate the hydration of OPC at early ages with the formation of calcium carboaluminate hydrates. In addition, the presence of $C\overline{C}$ modifies the course of initial hydration reaction of C₃A owing to the rapid formation of a barrier of hydrated calcium carboaluminate (C₃A–CaCO₃–xH₂O) developed on the surface of C₃A grains [14].

One can expect that CaO resulting from the rehydrated of $Ca(OH)_2$ is, after exposure in moist atmosphere, rehydrated to $Ca(OH)_2$ with a volume expansion of 97 % and with the formation of cracks [15].

The pozzolanic materials (i.e. FA) can react with liberated calcium hydroxide from the hydration of OPC forming additional amounts of C–S–H. The fire resistance is improved when FA–OPC is employed, that the overall CH amount it the hydrated material becomes reduced. The incorporation of FA and LS in the cement blends results in a decrease in the heat of hydration and an increase in the resistance of the resulting durability towards aggressive media.

The present work is aimed to investigate the effects of thermal treatment temperatures (250, 450, 650 and 800°C) on the physico-mechanical properties, phase composition as well as on the microstructure of cement pastes containing fly ash and limestone using X-ray diffraction (XRD), differential scanning calorimetry (DSC) and scanning electron microscopy (SEM).

Table 1. Chemical analysis of the starting materials (wt.%).

	OPC	FA	LS
SiO ₂	20.12	47.49	4.37
Al_2O_3	5.25	23.75	0.36
Fe_2O_3	3.58	7.96	0.29
CaO	62.13	4.81	52.99
MgO	1.53	2.44	0.90
SO_3	2.54	0.54	0.10
L.O. I	2.64	3.98	41.20
K ₂ O		4.02	
Na ₂ O		1.24	
Cl-		0.40	
TiO ₂		1.90	
P_2O_5		0.35	
Cr_2O_3		0.34	
MnO		0.26	
C ₃ S	51.79		
β -C ₂ S	18.61		
C ₃ A	7.81		
C ₄ AF	10.92		
Blaine surface area (m ² /g)	310	350	350

EXPERIMENTAL

The materials used in this investigation were Ordinary Portland cement and limestone from Helwan Portland Cement Company, Helwan, Egypt, pulverized fly ash from Toushki, Project Aswan, Egypt. The chemical compositions of starting materials are shown in table 1. The specimens used in this investigation were prepared from OPC that has been partially replaced by FA with 0, 10, 20 and 30 wt.%. These samples are denoted M.0, M.10, M.20 and M.30 and constitute the first group. The second group is constituted by samples obtained by replacing FA with LS in M.20 at 5, 10, 15 and 20 wt.%. They are denoted L.5, L.10, L.15 and L.20 (table 2). The cement pastes were prepared using the amount of water required to obtain the standard water of normal consistency according to ASTM C187 in table 2. The initial and final setting times and degree of hydration after 28 days of unfired cements are given as shown also in table 2. The cement pastes were mixed and moulded into 25.4×25.4×25.4 mm cubes, vibrated for 1 minute to remove any air bubbles. Immediately after molding, the cement pastes were cured in a 100% relative humidity chamber at 25°C for 24 hours, then demoulded and cured under water until the desired curing time. After 28 and 366 days of water curing, the hardened cement pastes were dried at 105°C for 24 hours. The specimens (25.4 25.4 25.4 mm cubes) were heated at a rate of 10°C/min and then kept for 3 hours at the peak exposure temperature to establish a stable temperature as shown in figure 1. The pastes were kept for 3 hours [8] at 250, 450, 650 and 800°C. Specimens are cooled to room temperature in the furnace switched off and then taken out for testing. Once cooling process finalized, the specimens were covered with plastic films and kept into a desiccator to order to avoid the influence of humidity and the carbonization of thermally treated pastes.

The phase compositions of the hydration products were determined by X-ray diffraction using a Philips diffractormeter with a scanning speed of $1^{\circ}2\theta/min$ (Ni-filtered CuK α radiation). Differential scanning colorimetry runs were conducted using a Shimadzu type



Figure 1. Heating process subjected to the cement paste vs. time.

50-thermal analyzer at a heating rate of 10°C/min. The microstructure of the selected samples was examined using a high-resolution scanning electron microscope.

The compressive strength and weight loss were determined. As strengths vary with FA and LS content, the term relative strength was used to analyze the experimental results. Relative compressive strength =

residual compressive strength (postheated) original compressive strength (preheated)

RESULTS AND DISCUSSION

X-ray Diffraction Analysis

Figure 2 shows XRD patterns of unfired specimens (25°C) and thermally treated cement pastes fired at 250, 450 and 800°C. All thermally treated samples having typical reflection associated to hydrated phases (C–S–H, CH, $C\overline{C}$ and unhydrated fraction (C₃S and β -C₂S). Some reflections observed in XRD patterns of unfired specimens disappear after their thermal treatment at 100-450°C, mainly those corresponding to ettringite and C–S–H. A progressive reduction in the peak intensity of calcium hydroxide is observed with increase of temperature of thermal treatment of above 450°C up to 800°C. The presence of $C\overline{C}$ is detected in samples containing LS and every specimen even increases in intensity up to

450°C. At higher temperature (800°C), the peaks of $C\overline{C}$ disappeared. CaO is well detected in samples subjected to temperature at 800°C due to the decomposition of CH and $C\overline{C}$.



Figure 2. XRD patterns of M.0, M.20 and L.5 thermally treated at 250, 450 and 800°C.

Table 2. Mix composition of the prepared batches, required water of standard consistency, setting times and degree of hydration after 28 days.

	OPC		LS	W/C ratio	Setting time, Min		Degree of hydration	
Mix No.		FA			Initial	Final	after 28 days (%)	
M.0	100	00	00	0.275	142	231	69.21	
M.10	90	10	00	0.273	204	275	63.96	
M.20	80	20	00	0.270	243	310	61.72	
M.30	70	30	00	0.265	280	347	61.10	
L.5	80	15	05	0.280	225	315	63.13	
L.10	80	10	10	0.283	220	280	67.36	
L.15	80	05	15	0.285	213	274	69.29	
L.20	80	00	20	0.290	194	262	76.34	

Table 3.	The enthalpy va	lues corresponding to en	dothermic peaks at	t 100-135°C and 470-49	0°C (J∕g).
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Temperature of treatment	Mix. No.	100-165°C	280-310°C	380°C	430-470°C
	M.0	73.32	1.36	0.271	92.67
	M.20	61.18	0.872	1.29	73.11
25°C	L.5	54.95	0.897	1.65	76.69
	L.15	53.22	1.04	1.47	85.17
	M.0	1.52	1.52	8.80	99.73
	M.20	4.66	4.66	13.17	81.74
250°C	L.5	5.00	0.117	17.85	85.67
	L.15	1.80	0.131	9.89	88.01
	M.0				93.11
	M.20				61.31
450°C	L.5				62.69
	L.15				77.43

At temperature of thermal treatment above 250° C, an increase of CH was observed due to the progress in hydration of anhydrous cement phases resulting in higher content of C–S–H at 450°C. Cement specimens subjected to firing indicate the structure modification of C–S–H gel [16]. C–S–H gel looses water molecule and OH-groups from the interlayer space, at 750-800°C C–S–H gel has completely disappeared, this is mainly replaced by crystalline phase with structure similar to C₂S. Figure 2 reveals that the main products of the C–S–H decomposition were C₂S and C₃S consistent with previous reports [17].

Differential Scanning Calorimetry

Figures 3-5 show the DSC thermograms of unfired specimens (25°C) and thermal treatment specimens at 250 and 450°C. There are four peaks located at 100-200, 280-310, 380 and 430-470°C. The endotherms below 200°C are mainly due to the dehydration of interlayer of C-S-H (tobermorite-like phase) with various composition and crystalline state as well as ettringite or monosulphate and carboaluminate hydrates. The peak located in the range of 230-260°C is attributed to the decomposition of gehlenite hydrates. The third peak located at 380°C is due to the decomposition of hydrogarnet phase, which has been formed as a result of steam effect under the condition of internal autoclaving at temperature of thermal treatment (250°C). The peak located at 430-470°C is due to the decomposition of CH. The peak intensity is mainly related to the amounts of hydrated decomposed.

For specimens thermally treated at 250°C, it was shown that mix L.5 has higher enthalpy values corresponding to peaks located at 100-165°C and 380°C than other mixes as shown in table 3 and figures 3-5. This result is agreed with the compressive strength values as shown below. The enthalpy values corresponding to the dehydroxylation of peak located at 470°C were 99.73, 81.74, 85.67 and 88.01 J/g. M.20, L.5 and L.15 have lower values than M.0. The decrease of the enthalpy of peak corresponding to dehydroxylation of CH is actually the result of consumption of Ca(OH)₂ formed during cement hydration by FA to form additional hydrated phases, leading to an increase of the peak area of C-S-H. Also it is evident that with increasing LS in specimens increases the enthalpy values at 470°C. The enthalpy values of peak corresponding to CH decreases; this is due to the liberation of water from the cement specimens from 250°C up to 450°C, which accelerate carbonation kinetics of Ca(OH)₂ [1,18] This is indicated by a partial decrease in the enthalpy values of CH peak at 450°C than the enthalpy values at 250°C, which tends to increase CC content. The enthalpy values of C-S-H decrease as the temperature of thermal treatment increases up to 450°C as shown in figure 5.

The weak endotherms peaks at 100 and 260-280°C are due to the removal of strongly adsorbed water and thermal decomposition of further formation of small amounts of C–S–H; these two endotherms are mainly due to slight exposure of thermally treated specimens with humidity of the air during handling. The results show that even if the dehydroxylation reaction is reversible, CH formed during the cooling has an onset temperature of decomposition lower than the original CH and can thus be considered as a criterion for determining the temperature history after a fire exposure.



Figure 3. DSC thermograms of unfired specimens M.0, M.20, L.5 and L.15 cured under tap water up to 28 days at 25°C.



Figure 4. DSC thermograms of M.0, M.20, L.5 and L.15 thermally treated at 250°C up to 3 hours.



Figure 5. DSC thermograms of M.0, M.20, L.5 and L.15 thermally treated at 250°C up to 3 hours at 450°C.

Weight Loss

The variations of weight loss of cement pastes cured up to 28 and 366 days with temperature are shown in figure 6 and 7. In general, the weight loss increases with temperature of thermally treatment. This is attributed to the amount of bound water removed with the increase in temperature. The evaporable water was removed at 105°C, the decomposition of C-S-H and C-A-H hydrates occurred up to 400°C and the dehydroxylation of CH was occurred at 400-600°C. The decarbonation of $C\overline{C}$ occurred between 650-800°C. Addition of FA shows a small decrease in the weight loss; due to the relatively lower water required for normal consistency of the pastes containing FA, which results in a lower rate of accumulation of hydration products due to the limited pore space available for these hydration. This cause a reduction in bound water as indicated by the weight loss. 20 wt.% FA shows an increase in the weight loss than 10 and 30 wt.% FA (figure 6).

Figure 7 depicts the weight loss of cement pastes containing LS cured at 28 and 366 days as a function of temperature of thermal treatment. The weight loss of specimens M.20 and L.5 show an increase in its values than the other mixes at temperature of 250-450°C; It was reported that the addition of 5 wt.% LS acts as a nucleating agents which activates FA to react with liberated CH, forming thus an additional C–S–H, calcium carboaluminate and carboaluminate ferrite hydrated products [13,19,20]. As the temperature increases up to 650-800°C, the weight loss increases with the increase of LS content due the decarbonization of $C\overline{C}$.

Microstructure

SEM investigations on hardened cement pastes show distinct changes in the morphology as a consequence of exposure to elevated temperatures. SEM micrograph shows a mixture of hydration products having well crystallized calcium hydroxide appeared as sheets and ill-crystallized calcium silicate hydrate representing the microstructure of thermally treated OPC pastes at 250°C; the sheets of CH are stacked as parallel layers as shown in figure 8 a. The morphology of hydrates is characterized as well-formed crystals deposited in the pore system with no apparent cracks after exposure at 250°C as shown in figure 8a and 8b.

The micrograph as shown in figure 8c and 8d of thermally treated M.0 pastes indicated that; microcracks were detected in the structure at 650°C. Also, the micrograph of thermally treated OPC paste at 650°C shows microcracks intermingled with voids due to the increase in porosity. The formation of microcracks leads to a destruction of binding forces as represented by the low compressive strength and high porosity of OPC at 650°C. Loss of strength at higher temperature of thermal treated specimens may be related to the loss of bound water, increased porosity, and consequently increased permeability, which makes the OPC cement specimens progressively more susceptible to further destruction.

Figure 9 shows the SEM micrographs of cement paste made with 20 wt.% FA. The micrograph shows the formation of C–S–H, which is produced as result of consumption of hydrated lime by silica and alumina containing FA, which are deposited within the pore system and around the cement grains, which can accommodate larger amounts of hydration products especially C–S–H leading to relatively high compressive strength values as compared with those of OPC paste (figure 9a). The microstructure obtained at higher temperature (650°C) displayed the absence of microcracks as shown in figure 9 b. Evidently, the pore spaces are available for the deposition of hydration products

Compressive Strength

Figures 10-13 indicate the compressive strength of all cement pastes at different thermal treatment temperatures as well as the relative increase or decrease in the compressive strength of each specimen as compared to its original compressive strength before heating. Figure 10 shows the compressive strength of specimens containing FA initially water cured for 28 and 366 days. The compressive strength of OPC paste, curing up to 28 days suffers a significant loss of strength subsequently up to 450°C. This is due to the loss of crystal water, besides the changes in the morphology and microcrack formation. The compressive strength of OPC pastes declines sharply above 450°C. The reduction in the compressive strength with temperature shows distinct decreases beyond 650 and 800°C.

The compressive strength increases with the addition of FA with temperature of thermal treatment up to 250°C then decreases. The higher compressive strength of cement pastes containing FA is found at 250°C, due to the formation of additional hydration of unhydrous cement grains as a result of steam effect under the condition of steam of self (internal) autoclaving; leading to the formation of additional amounts of C-S-H phases 250°C, which deposit in the pore system [1, 8]. Internal autoclaving takes place within the temperature rang 105 250°C. The addition of FA or slag enhances the fire resistance of concrete [21]. Also, it was shown that the compressive strength values of cement pastes containing FA at 250°C were higher than the original (prefired) compressive strength values. Moreover, the OPC pastes containing FA retained higher strengths than the plain OPC pastes at higher temperatures up to 650°C.



Figure 6. Weight loss of cement pastes containing FA cured for 28 and 366 days as a function of treatment temperatures.



Figure 7. Weight loss of cement pastes containing LS instead of FA cured for 28 and 366 days as a function of treatment temperatures.

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Figure 8. SEM micrographs of thermally treated OPC paste, a) and b) thermally treated at 250°C, c) and d) thermally treated at 650°C.



Figure 9. SEM micrographs of thermally treated cement paste containing 20% FA. a) thermally treated at 250°C, b) thermally treated at 650°C.



Figure 10. Variations of compressive strength of cement pastes containing FA with temperature after 28 and 366 days of curing.



Figure 11. Relative compressive strength of cement pastes containing FA with temperature after 28 and 366 days of curing.



Figure 12. Variations of compressive strength of cement pastes containing FA and LS with temperature after 28 and 366 days of curing.



Figure 13. Relative compressive strength of cement pastes containing FA and LS with temperature after 28 and 366 days of curing.

For specimens cured up to 366 days, the values of compressive strength of M.0 and M.10 decrease with increase of temperatures of thermal treatment up to 800°C. While, the compressive strength increases with temperature up to 450°C for samples M.20 and M.30 compared to unfired specimens.

Figure 11 presents the relative compressive strength of cement pastes containing FA cured at 28 and 366 days. The relative compressive strengths were 95.86-135.35, 91.2-134.08, 38.2-98.17 and 6.33-51.55 for samples cured at 28 days and thermally treated at 250, 450, 650 and 800°C. On the other hand, the relative compressive strengths for samples cured at 366 days were 90-127.96, 73.13-112.72, 54.4-95.88 and 30.6-45.7 respectively. The addition of FA increases the relative compressive strength. The relative compressive strength of specimens cured at 28 days are relatively higher than those cured at 366 days; this may be probably due to the hydration of unhydrated parts of cement grains, which were activated as a result of temperature rise; due to the formation of C–S–H phase.

Figure 12 shows the compressive strength of cement pastes containing FA and LS cured at 28 and 366 days. For specimens cured at 25°C for 28 days, the compressive strength of all specimens increases with temperature up to 250°C. The compressive strength of cement pastes containing 5 wt.% LS was higher than M.20 cement pastes at all temperatures [13]; this is due to that LS activates the pozzolanic reaction of FA with the liberated CH to produce additional amounts of calcium silicate, aluminosilicate and calcium carboaluminate hydrates as well as additionally calcium silicocarbonate hydrates was formed during the hydration of C₃S in the presence of LS which enhance the compressive strength. The amount of CaCO₃ incorporating into the cement hydration for formation of carboaluminate depends on the amount of aluminate and ferrite phases available, as well as the presence of free lime [22-24].

Increase the LS content (10 wt.% LS), the values of compressive strength of L.10 has nearly the same values of the reference samples (M.20; 80 wt.% OPC+20 wt.% FA). The decrease of compressive strength of mix L.15 and L.20 is due to the decrease of the amount of FA content [20]; LS dilutes the pozzolanic reaction. The compressive strength development depends primarily on the formation of the hydrated calcium silicates and its crystallization states. The compressive strength decreases with LS content due to the dilution effect. The lower strength of cement pastes made with 15-20 wt.% LS was found especially at a temperature of 650-800°C, compared with the cement pastes containing 0-10 wt.% LS. The decompositions of CH and $C\overline{C}$ at 430-600°C and 650-800°C respectively have an additional effect on the strength loss [18].

For specimens prefired, the compressive strength decreases with LS content. Increase of temperatures of thermal treatment up to 250°C, the compressive strength increases (figure 12). The increase of compressive strength is higher than specimens cured at 25°C for 28 days in the temperature range from 250-450°C.

The relative compressive strength values of cement pastes containing FA and LS cured up to 28 and 366 days are graphically represented in figure 13. The relative compressive strengths were 129.1-149.44, 112.82-137.56, 81.79-61.77 and 17.03-27.31 for samples cured at 28 days and thermally treated at 250, 450, 650 and 800°C. Whereas, the relative compressive strengths for samples cured at 366 days were 105-158.11, 92.43-139.14, 81.27-86.93 and 39.44-62.19 respectively. The relative compressive strength of specimens subjected to the temperature increases in the range 250-450°C, whereas it decreases in the range 650-800°C. It was shown also that the relative compressive strength values of specimens containing FA and LS were higher than specimens containing FA only in the temperature range of 250-450°C.

CONCLUSION

- 1. The enthalpy values of peak corresponding to C–S–H decomposition decrease as the temperature of thermal treatment increases up to 450°C, due to looses of water molecule and OH-groups from the interlayer space and completely disappears at 750-800°C, this is mainly replaced to new crystalline silicate phase with structure similar to C_2S as shown by XRD.
- 2.M.20 and L.5 show an increase in the values of weight loss than the other mixes at temperature of 250-450°C. This is due to 5 wt.% LS which acts as a nucleating agents which activating FA to react with CH and forming additional C–S–H and calcium carboaluminate and carboaluminate ferrite hydrated products. The weight loss increases with LS content up to 650-800°C, due the decarbonization of $C\overline{C}$.
- 3. The micrograph of specimen containing FA at 250°C shows the presence of ill crystalline C–S–H, which accommodates larger amounts of hydration products leading to relatively high compressive strength values as compared with those of OPC paste.
- 4.SEM micrograph of thermally treated OPC paste at 650°C shows microcracks.
- 5.5 wt.% LS has higher compressive strength values than M.20 cement pastes at all temperatures of thermal treatment; this is due to LS which activates the pozzolanic reaction of FA with the liberated CH to produce additional amounts of C–S–H and aluminosilicate hydrates, which enhances the compressive strength.

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- 6. The relative compressive strength of specimens cured at 28 days are relatively higher than those cured at 366 days. The relative compressive strength of specimens containing FA and LS subjected to temperature increases in the range 250-450°C, whereas it decreases in the range 650-800°C. The relative compressive strength values of specimens containing FA and LS were higher than specimens containing FA only in the temperature range of 250°C.
- 7. The addition of limestone to fly ash-OPC cement has a beneficial effect, but cannot be employed at high temperature application above 450°C. The blended cements suitable for high temperature applications should contain either fly ash alone or in combination with lower limestone contents not more than 5 wt. %.

Cement Notation:

C = CaO, S = SiO₂, A= Al₂O₃, F = Fe₂O₃, H = H₂O, C-S-H = calcium silicate hydrate and CC = calcium carbonate

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ÚČINEK TEPLOT NA STRUKTURU A PEVNOSTNÍ VLASTNOSTI CEMENTOVÝCH PAST OBSAHUJÍCÍCH POLÉTAVÝ POPEL SAMOTNÝ NEBO V KOMBINACI S VÁPENCEM

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Tato studie se zaměřila na vyšetření účinku tepelného zpracování na mikrostrukturu a fázové složení pasty obsahující polétavý popel (FA) a vápenec (LS). Byly připraveny dvě skupiny cementových past. V první skupině, byl portlandský cement (OPC) částečně nahrazen 10, 20 a 30 % hmotnostních FA (M.0, M.10, M.20, M.30). Druhá skupina vzorků je tvořena vzorky odvozenými od M.20, ve kterých byla složka FA nahrazena 5, 10, 15 a 20 % hmotnostních LS (L.5, L.10, L.15 a L.20). Po 28 a 366 dnech tvrdnutí pod vodou byly vytvrdnuté cementové pasty vysušeny při teplotě 105°C po dobu 24 hodin, a pak vystaveny tepelnému zpracování po dobu 3 hodin při teplotě 250, 450, 650 a 800°C a pak ochlazeny na pokojovou teplotu ve vypnuté peci. Byla stanovena pevnost v tlaku a ztráta hmotnosti. Skenovací elektronová mikroskopie, diferenční kalorimetrie a rentgenová difrakce byly použity pro identifikaci změn, ke kterým dochází v mikrostruktuře vytvořené nebo rozložené fáze. Výsledky ukazují, že nastala dodatečná hydratizace nehydratovaných zrn cementu, překrystalizace, deformace a transformace fází CSH. Změny ve fyzickém stavu cementových past byly prostudovány změřením míry rozrušování při pevnosti v tlaku podle teploty.