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Hydration characteristics and compressive strength of hardened cement pastes containing nano-metakaolin

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Abstract In this study the effect of inclusion of nano-metakaolin (NMK) to ordinary Portland cement (OPC) on the hydration characteristics and microstructure of hardened OPC–NMK pastes was studied. The OPC–NMK blends were prepared by the partial substitution of OPC by NMK (4, 6, 10 and 15 weight %). The fresh pastes were made using an initial water/solid (W/S) ratio of 0.27 by weight and then hydrated for various time intervals. At the end of each hydration time, the hardened blended cement pastes were tested for compressive strength, free lime content, combined water content, X-ray diffraction (XRD) analysis, differential scanning calorimetry (DSC) and scanning electron microscopy (SEM). The compressive strength results revealed that the inclusion of nano-metakaolin into OPC improved the mechanical properties of NMK–OPC pastes during almost all ages of hydration, especially with the paste containing 10 wt% NMK. The compressive strength values obtained for OPC paste blended with 4% silica fume (SF) and 6% NMK are comparable to those of the neat OPC paste. The DSC thermograms and XRD diffractograms obtained for some selected hardened pastes indicated the formation of amorphous calcium silicate hydrates, calcium sulfoaluminate hydrates, calcium aluminate hydrate and calcium hydroxide. SEM micrographs showed the formation of a dense microstructure for the hardened OPC–NMK and OPC–NMK–SF pastes as compared to the neat OPC paste after 90 days of hydration.

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Introduction

Studies have been made to enhance the use of cementitious materials such as pozzolans as a partial substituent of Portland cement. Utilization of these materials for such purposes not only eliminates the problems of Portland cement but also results in high efficiency cement production [1,2]. Pozzolana was defined as siliceous/aluminous materials, which reacts chemically with calcium hydroxide (CH) in the presence

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of water to form compounds that possess cementitious properties. Fly ash, rice husk ash, silica fume and slag in addition to the calcined clays in the form of metakaolin are examples of pozzolanic substances [3,4].

The thermal activation process or de-hydroxylation of kaolin at a specific temperature, leads to the breakdown or partial breakdown of the crystal lattice structure, forming a phase transition (metakaolin) which has highly disordered and amorphous characters with pozzolanicity. The exposure duration to temperature above the de-hydroxylation temperature promotes recrystallization to form mullite and hence loss in pozzolanicity [5,6]. A small part of AlO_6 octahedra is maintained, while the rest is transformed into much more reactive tetra- and penta-coordinated units [7]. When nano-metakaolin particles are used as cementitious materials, various improvements can be attained, thereby leading to improved permeability and strength. The nano-metakaolin particles act as nuclei of hydration, possess pozzolanic activity, and can fill the voids in the cement matrix. Also the large surface area of nano-particles and their abundance due to their small size, can facilitate the chemical reactions necessary to produce a dense structure with more calcium silicate hydrates (CSH) and less calcium hydroxide contents [8–11].

The use of MK as a partial cement replacement material up to 10–15% in mortar and concrete leads to improvement in the pore structure and high resistance to the transportation of water, the aggressive action of organic acids and diffusion of harmful ions which lead to degradation of the matrix [12–15]. The effects of high temperatures up to 800 °C on the mechanical properties and microstructure of OPC containing nano-metakaolin (NMK) were studied. NMK was prepared by thermal activation of nano-kaolin clay at 750 °C for 2 h. It was found that after an initial increase in compressive strength at 250 °C for the mortar specimens, the strength decreased considerably at higher temperatures [16]. The mechanical and durability properties of high performance metakaolin (MK) and silica fume (SF) concretes and their microstructure characteristics were studied [14].

The object of this study is to investigate the hydration characteristics of hardened OPC pastes blended with different amounts of NMK and NMK + SF. This was done via determination of compressive strength, chemically combined water and free lime contents at different ages of hydration. In addition, phase composition was examined using XRD and DSC.

Experimental

Ordinary Portland cement (OPC) used in this investigation was obtained from South Valley Cement Company, Egypt, with Blaine surface area of $2945 \text{ cm}^2 \text{ g}^{-1}$. Its chemical oxide composition is given in Table 1. Nano-kaolin was supplied from Middle East Mineralogical (MEMCO), Egypt. The nano-kaolin was fired at 750 °C for 3 h to obtain nano-metakaolin (NMK) with a Blaine surface area of $1.8 \times 10^4 \text{ cm}^2 \text{ g}^{-1}$.

Table 2 Major oxide composition of nano-kaolin (mass %) before and after burning.

Oxide (mass %)	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	L.O.I
Before burning	52.2	33.6	0.1	1.5	12.2
After burning	53.4	34.3	0.1	1.5	10.3

Table 3 Compositions of different mixes and their designation.

Mixes	Mix proportion (mass %)		
	OPC	NMK	SF
Mo	100	–	–
M1	96	4	–
M2	94	6	–
M3	90	10	–
M4	85	15	–
MS	90	6	4

Its chemical composition before and after burning is given in Table 2. The microstructure of nano-metakaolin (NMK) was studied by means of scanning electron microscopy (SEM) and transmission electron microscopy (TEM) in an earlier publication [17]. Condensed silica fume (SF), which is a by-product of silicon or ferrosilicon alloy industries, was obtained from ferrosilicon company, Kom-Ombo, Egypt. Silica fume particles are spherical and have an average diameter of about 0.1 μm . It consists of 99% amorphous silica with a specific surface area of $20 \times 10^4 \text{ cm}^2 \text{ g}^{-1}$. These characteristics account for the substantial pozzolanic activity of silica fume in terms of both its capacity of binding lime and rate of hydration reaction.

Different blended cement pastes were prepared using W/S ratio of 0.27; only 0.1 wt% superplasticizer was added in the case of blended cements containing 10% and 15% NMK (mixes M3 and M4) and 6% NMK + 4% SF (mix MS) to maintain the same W/S ratio. Each paste was prepared by mixing the dry mixture with the required amount of water for about 3 min. After complete mixing, the resultant paste was molded into cubic specimens by using 1 inch cubic molds. Then the specimens were cured with their molds at 100% relative humidity for 24 h. The cubic specimens were removed from their molds and then cured under water at room temperature for different time intervals of 3, 7, 14, 28 and 90 days. Table 3 illustrates the sample designation and their compositions.

At each time interval, three cubes of each hardened cement paste were subjected to compressive strength test and the average value was recorded. This was accomplished using a Ton-industrie machine (West Germany) for maximum load of 60 tons. The resulting crushed specimens of the hardened cement pastes were ground and the hydration reaction was stopped [18]. The samples were then dried at 80 °C for 3 h in CO_2 -free atmosphere.

Table 1 Chemical oxide composition of OPC (mass %).

Oxide	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	Cr_2O_3	Mn_2O_3	TiO_2	L.O.I
Mass %	20.46	5.14	3.53	61.28	2.8	0.2	0.11	2.82	–	0.11	0.33	3.15

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