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Effect of nano-metakaolin addition on the hydration characteristics of fly ash blended cement mortar

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Abstract The effect of nano-metakaolin (NMK) addition on hydration characteristics of fly ash (FA) blended cement mortar was experimentally investigated. The amorphous or glassy silica, which is the major component of a pozzolan, reacts with the calcium hydroxide liberated during calcium silicate hydration. It is believable to add FA and NMK particles in order to make high performance concrete. The physico-mechanical properties of FA blended cement mortars made with different percentages of NMK were investigated. The experimental results showed that the compressive and flexural strengths of mortars containing NMK are higher than those of FA blended cement mortar at 60 days of hydration age. It is demonstrated that the nanoparticles enhances strength than FA. In addition, the hydration process was monitored using scanning electron microscopy and thermal gravimetric analysis (TG). The results of these examinations indicate that NMK behaves not only as a filler to improve microstructure, but also as an activator to promote the pozzolanic reaction.

Keywords Nano-metakaolin · Fly ash · Compressive strength · Flexural strength · Thermal analyses · Microstructure

Introduction

Portland cement-based binders are the primary active components of cementitious composites used in most modern construction. Production of cement emits high levels of carbon dioxide (CO_2) which is one of the primary greenhouse contributors to global warming. Therefore, the use of supplementary cementitious material (SCMs) in cement, mortar, or concrete resulted in the reduction of CO_2 emission.

Fly ash (FA) is a waste material produced from the electrical power plant. FAs are classified into two basic types; Class F (low-calcium FA) and Class C (high-calcium FA) [1]. Blended cements can be produced through the replacement of ordinary Portland cement (OPC) by pozzolanic materials like FA [2]. Using FA in blended cement is useful for a number of purposes. FA in blended cement enhances workability and chemical resistance. It is known that, it reduces the heat of hydration and block the alkali-silica reactions [3]. The use of FA as a mineral admixture in high performance concrete improves both strength and durability properties of concrete [4–6].

Nowadays, nanotechnology has attracted a considerable scientific interest due to the new potential uses of particles in the nanometer (10^{-9} m) scale.

The nanoscale size of particles may result in radically improved properties from conventional grain-size materials of the same chemical composition. Thus, industries may be able to re-engineer many existing products and to design new and novel products that function at unprecedented levels. In fact the nanotechnology is already applied in the fields of concrete, steel, and glass. Concretes produced with this technology are stronger, more durable and have a higher workability [7].

When nanoparticles are used as SCMs in concrete, various improvements can be attained, as microstructure of the cement matrix, which leads to improved permeability and strength. The nanoparticles improve the cement structure by pozzolanic reaction [8] as well as by filling the

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voids between cement grains [8–11]. Pozzolans react chemically with free lime liberated during OPC hydration to form additional amounts of calcium silicate hydrate (CSH) hydrated product [12, 13]. Moreover, the large surface area of nanoparticles and their small size can accelerate the chemical reactions necessary to produce a dense cement matrix with more CSH content. However, this enhances the overall concrete performance. In general, the smaller size of nanoparticles than that commonly used as SCMs, making them more reactive and effective. Nanosilica (NS) and nanoclays (NC) have been reported to increase compressive strength, reduce permeability, and develop a denser microstructure of cement paste [14, 15].

It has been reported that, the replacement of OPC by 5 % NMK in cement mortar hydrated at ambient temperature increases the compressive and flexural strengths of cement mortar [15].

The appropriate percentage of nanosilica was studied by many authors [16–19] and they conclude that the small amount (1–5 mass%) of NS is suitable for cementitious pastes due to the agglomeration caused by the lack of particle dispersion during the mix. On the other hand, some researcher showed that the improvement in properties can be achieved with higher dosages, of about 10 mass%. They also showed that, the proper adjustments are made to avoid excessive self-desiccation and microcracking that could hinder the strength development [20]. Other researchers showed that the improvement in properties can be achieved with higher dosages of about 10 mass%, if proper adjustments are made to the formulation in order to avoid excessive self-desiccation and microcracking that could hinder the strength [20]. The addition of fine particles to cement leads to a strong tendency to form agglomerates when present in contact with water. The fine particle agglomerates phenomena affect to rheological behavior [21] and the ultimate hardening properties of cement pastes [22].

This paper aimed to study the effects of nano-sized amorphous metakaolin addition on the hydration characteristics of FA blended cement mortars. The hydration characteristics include the mechanical properties, phase composition, microstructure, and thermal analysis of the hardened cement mortar.

Experimental

Materials

The OPC, ASTM C-150 Type I [23] used in this investigation was supplied by Yamama Cement Company, Saudi Arabia.

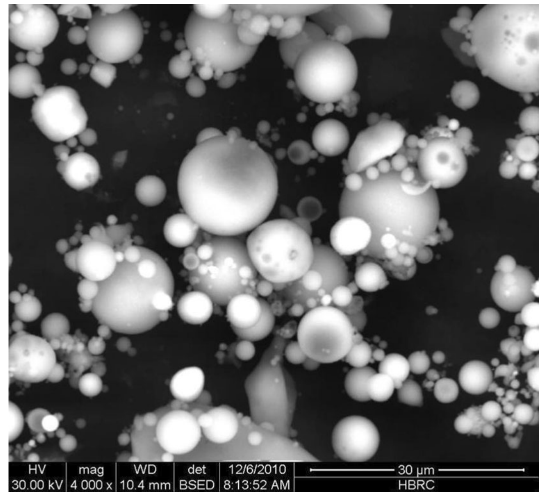

Fig. 1 SEM micrograph of fly ash

Table 1 Chemical composition of materials by XRF, mass/%

Oxide composition	OPC	FA	NK
CaO	63.85	4.29	0.01
SiO_2	19.83	52.87	48
Al_2O_3	5.29	33.08	36.5
Fe_2O_3	3.53	3.58	0.2
MgO	0.52	0.9	0.02
SO_3	2.43	0.38	–
Na_2O	0.21	0.3	0.03
K_2O	0.07	0.79	0.07
TiO_2	–	1.89	1.3
P_2O_5	–	0.55	–
MnO	–	0.05	–
Total	95.73	98.68	86.13
Ignition loss	2.82	0.97	13.87

Class F FA of Blaine surface area $\approx$3,800 cm^2 g^{-1} and average diameter $\leq$10 µm was used in this investigation [2]. Figure 1 shows the scanning electron microscopy (SEM) micrograph of raw FA particles. The mineral and chemical compositions of FA used in the investigation are shown in Fig. 2 and Table 1, respectively. It consists of a minor amount of glassy phase with major amount of crystalline inclusions of mullite, hematite, and quartz as shown in Fig. 2. Nano-kaolin (NK) with average dimensions 200 nm $\times$ 100 nm $\times$ 10 nm and Blaine surface area of $\approx$48 m^2 g^{-1} was supplied by the Middle East Mining Investments Company (MEMCO), Cairo, Egypt. The TEM micrograph of nano-kaolin is shown in Fig. 3. The raw NK consists of kaolinite, illite, and quartz as major mineral phases, whereas NMK contains only illite and quartz. The heat treatment of raw nano-kaolinite at 750 °C resulted in the disappearance of its crystalline structure of kaolinite

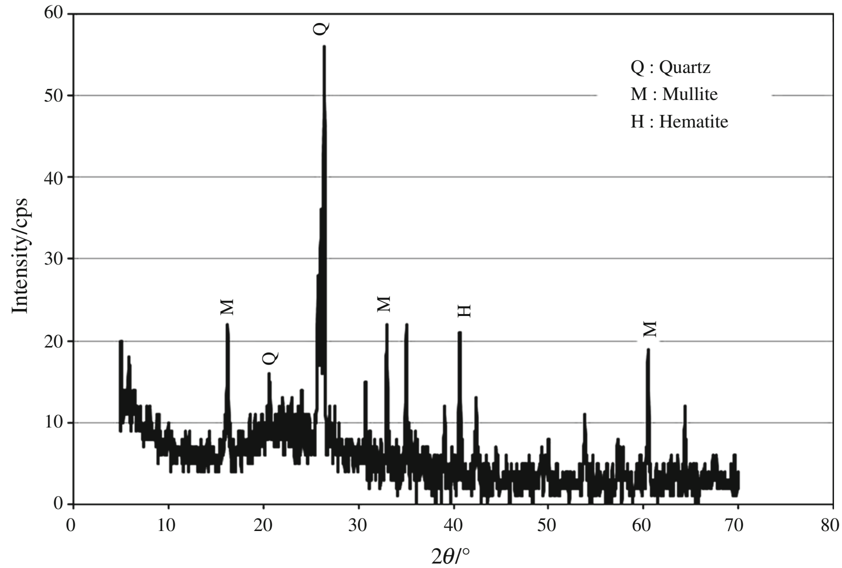

Fig. 2 XRD of fly ash

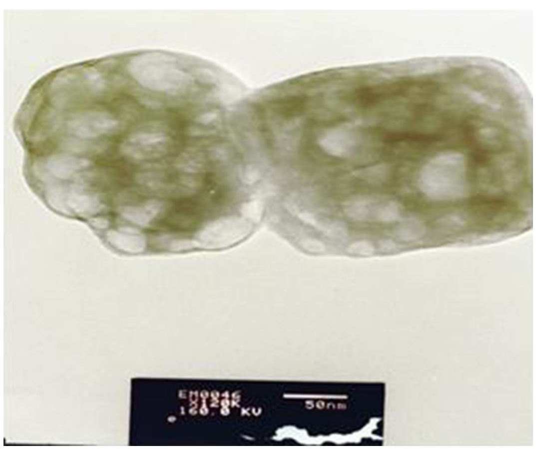

Fig. 3 TEM micrograph of NK

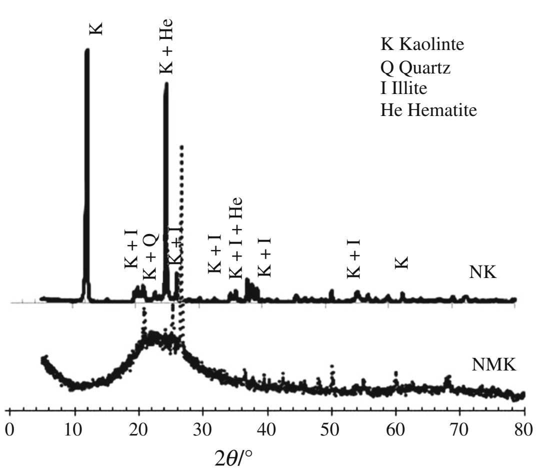

Fig. 4 XRD of NK and NMK

Table 2 Dry mix composition of blended mortar, mass/%

Mix	OPC	FA	NMK
Ml	80	20	0
M2	80	20	2.5
M3	80	20	5.0
M4	80	20	7.5
M5	80	20	10.0

and the development of an amorphous structure as shown in Fig. 4. The oxide composition of FA, NK, and OPC are summarized in Table 1. Commercial local red sand was used as a fine aggregate in mortar preparation.

Mortar preparation and identification

The composite cement used in this investigation was obtained by blending OPC, FA, and NMK. OPC was partially substituted by 20 % FA by mass and NMK was added by 0, 2.5, 5, 7.5, and 10 % by mass of OPC-FA cement blend as illustrated in Table 2.

The dry constituents were mixed in a ball mill with four balls for 2 h to attain complete homogeneity. The mortar was prepared using blended cement: sand ratio of 1:2.75 and water/binder ratio of 0.5. The composite mortar pastes were molded into 5 cm cubes for compressive strength and 4 cm × 4 cm × 16 cm prisms for flexural strength. The molds were vibrated for 1 min to remove any air bubbles. The samples were kept in molds at 100 % relative humidity for 24 h, and then cured in water till the required times of testing. Compressive and flexural strengths were determined at 3, 7, 28, and 60 days. The hardened cement mortar specimens were removed from water before testing. The compressive and flexural strength tests were performed on wetted specimens. The crushed samples, resulting from compression tests, were grounded and used for thermal and microstructural analysis. The evaporable water of the hydrated crushed samples was removed using the method described elsewhere [24]. The total porosity of the cement mortar was determined according to ASTM C-642 [25].

Thermogravimetric analysis and differential thermal analysis were conducted using a DSC-TGA SDT Q600 thermal analyzer at a heating rate of 10 °C min^{-1}. The sample chamber was purged with nitrogen at a flow rate of 100 cc min^{-1}.

The crystalline phases present in the raw materials were identified using the X-ray diffraction technique. Nickle-filtered Cu Kα radiations at 40 kV and 20 mA were used throughout in a Philips PW 1390 diffractometer. A scanning speed of 2° min^{-1} was used. The scanning electron microscope JWEL 6360A was used for the identification of the changes in the microstructure of the formed and/or decomposed phases.

The CH content was calculated using the following equation:

$$CH(\%) = WL_{CH}(\%) \times (MW_{CH}/MW_{H}), \quad (1)$$

where WL_{CH} corresponds to the mass loss in percentage attributable to CH dehydration, and MW_{CH} and MW_H are the molecular masses of CH (74.01 g mol^{-1}) and water (18 g mol^{-1}), respectively [26].

Results and discussion

Figure 5 illustrates the variations of the compressive strength of blended cement mortars containing FA and NMK versus curing age. Evidently, the compressive strength values of the composite cement mortars increase with the increase of curing age up to 60 days. However, the compressive strength increases with increasing additions of NMK up to 7.5 % and decreases at 10 % ratio. Obviously, the addition of 7.5 % NMK to the blended cement mortar containing 20 % FA (M1) leads to enhancement of the compressive strength of M4 by 17.2 % as compared to M1 at the curing age of 60 days. The enhancement in the compressive strengths of hardened blended cement mortar due to the addition of NMK can be attributed to the pozzolanic effect of NMK which improves the interfacial zone. The pozzolanic effect combines glass-like silica and alumina elements in FA with the free lime liberated during OPC hydration which adds to the bonding strength and solid volume. Obviously, the platelet particles of NMK have an average dimension of 100 nm × 50 nm × 20 nm, which is finer than the average cement particles, resulting

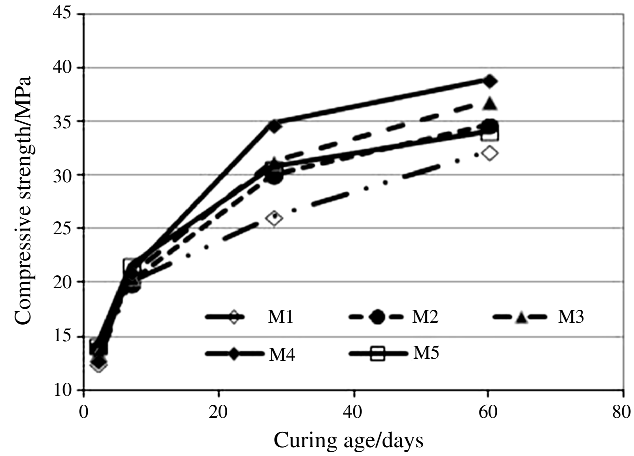

Fig. 5 Compressive strength of composite cement mortar containing fly ash and NMK

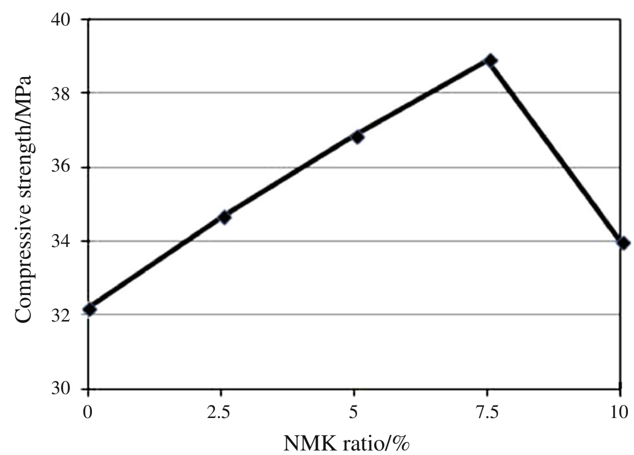

Fig. 6 Compressive strength of composite cement mortar containing fly ash and NMK at 60 days

in an extremely high surface area. The NMK reacts very rapidly with the free calcium hydroxide, liberated from OPC hydration, to form CSHs in an alkaline environment like the pore solution of the fresh Portland cement paste. In addition, the improvement in strength with the addition of NMK loaded up to 7.5 % is attributed to the cross-linking of NMK platelets with hydration products with a consequent resistance of microcracks formation due to shrinkage. The reduction in compressive strength at 10 % NMK addition is due to agglomeration of NMK particles around cement grains

Figure 6 shows the variation of the compressive strength of blended cement mortars containing FA and NMK hydrated at 60 days versus NMK addition. Obviously, the compressive strength increases with increasing addition of NMK up to 7.5 % and then decreases with further additions up to 10 % by mass. At 7.5 % NMK addition, the increase in the compressive strength was 1.2-fold. The physical filling of NMK particles inside interstitial spaces of FA

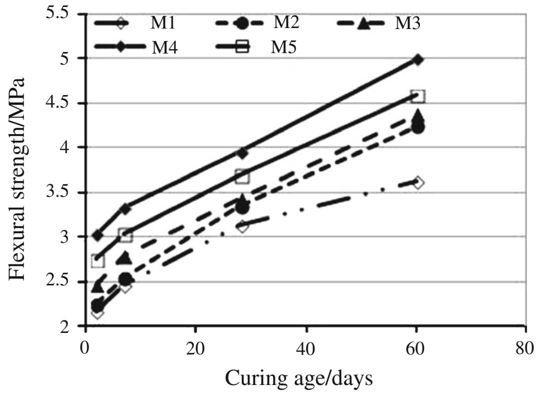

Fig. 7 Flexural strength of composite cement mortar containing fly ash and NMK

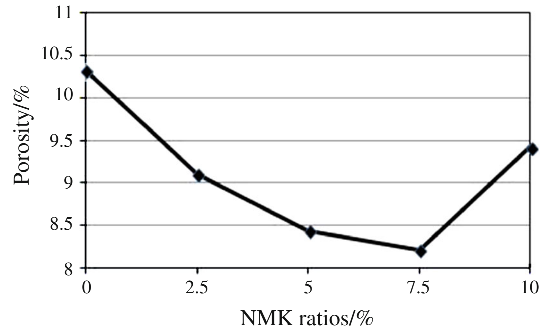

Fig. 8 Total porosity of composite cement mortar containing fly ash and NMK at 60 days of hydration

cement skeleton leads to increase in its density as well as its strength. Moreover, the pozzolanic reaction, between NMK and the free CH liberated during OPC hydration, which produces the additional CSH hydrated product. The reduction in the compressive and flexural strengths at higher NMK addition may be due to the NMK agglomerates around the OPC grains which hinder the hydration process. Moreover, the formed crystalline hydrates in pore system lead to a sort of the pore opening. The sort of opening and/or formation of microcracks lead to the development of fewer points of contact which act as binding centers between cement grains.

Figure 7 shows the variation of the flexural strength of blended cement mortars containing FA and NMK versus curing age. It is clear that the flexural strength increases as the curing age increases and also as the NMK addition increases. Moreover, at 60 days of hydration, the flexural strength increases as the NMK addition increases up to 7.5 % and then decreases at 10 %. The increase of flexural strength is due to pozzolanic reaction of FA and NMK with free lime librated during OPC hydration and also due to physical filling of the NMK platelet particles inside the interstitial spaces of FA–cement skeleton. However, the NMK platelet particles act as nano-size enhanced as a result of interfacial zone. At 7.5 % NMK addition, the increase in the flexural strength was 2.3-fold. The reduction of flexural strength at later age and 10 % NMK addition may be due to agglomeration of NMK particles around cement grains.

Total porosity of blended cement mortars containing FA and NMK at 60 days of hydration versus NMK ratios is presented in Fig. 8. It is clear that the total porosity decreases with the increase of NMK addition up to 7.5 mass%, then increases as the NMK increases up to 10 mass%. The decrease of total porosity is attributed to pozzolanic reaction of FA and NMK with CH liberated during OPC hydration to form additional hydration products that fill some available pores, whereas the increase of

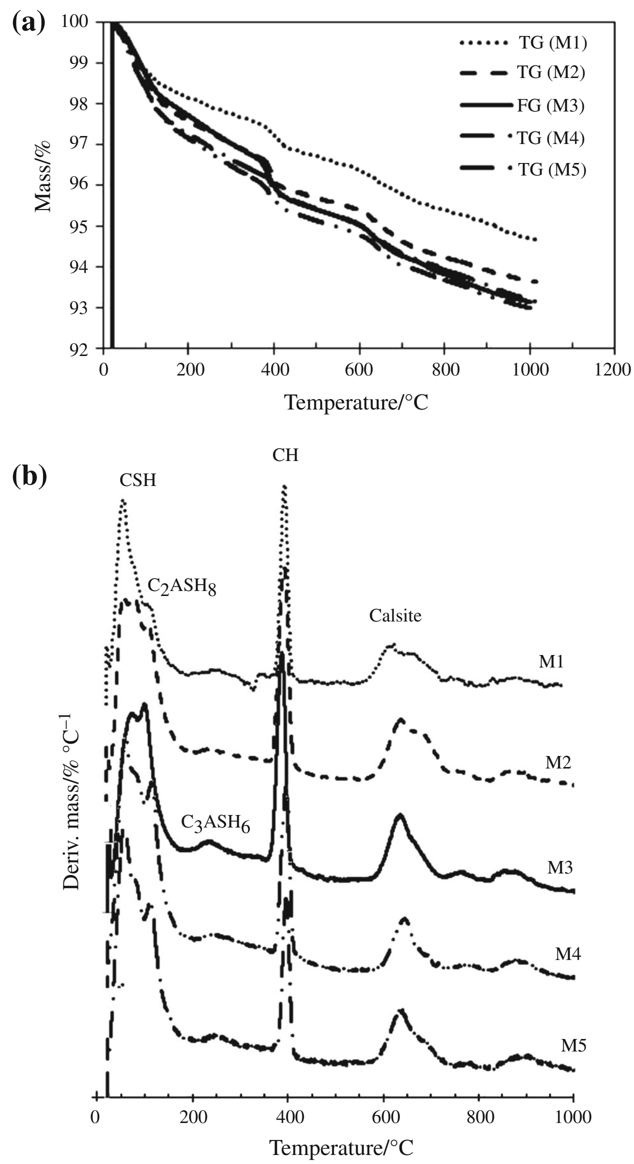

Fig. 9 TG/DTG curve of composite cement mortar containing fly ash and NMK at 60 days of hydration

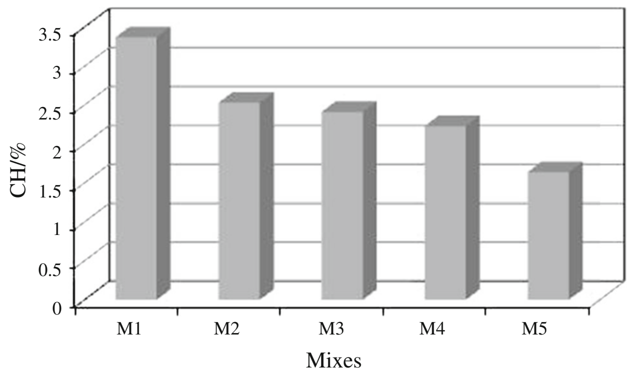

Fig. 10 Free lime content of composite cement mortar containing fly ash and NMK at 60 days of hydration

total porosity with NMK addition is due to the formation and enlargement of microcracks and/or the increase of degree of crystallinity of the formed hydrates leading to a sort of opening of the pore system of the hardened cement mortars pastes [27].

Figure 9 displays the thermal gravimetric analysis (TG) results, mass loss, and corresponding derivative curves presented as differential mass of composite cement mortars containing FA and NMK hydrated for 60 days. It can be seen that the hardened cement mortar showed a reduction in mass up to 250 °C which is attributed to the loss of adsorbed surface water as well as the loss of water from CSH gel layers of gehlenite hydrate (C_2ASH_8) and hydrogarnet (C_3ASH_6). The second mass loss was observed at about 410 °C which represents the thermal decomposition of CH. Also, the third mass loss at about 650 °C is attributed to the decomposition of calcite. The main features of the thermograms are characterized by a decrease in the peak areas of CH and an increase in the peak area of CSH, C_2ASH_8, and C_3ASH_6 phases as the NMK addition increases.

Figure 10 illustrates the variation of residual calcium hydroxide (CH) in the composite cement mortars containing FA and NMK hydrated for 60 days. The amount of calcium hydroxide present in the composite blended cement mortars was calculated using Eq. 1. It is clear that, the FA blended cement mortar contains 3.37 % CH. The addition of NMK resulted in the consumption of CH content which decreases with increasing NMK addition due to the higher pozzolanic activity of NMK. At 7.5 % NMK the CH content decreases to 2.23 %. It was also observed that with the increasing of NMK to 10 %, the CH content is also decreases to 1.64 %. It is also clear that the FA blended cement mortar is less in reactivity than FA-NMK blended cement mortar. The increase of pozzolanic activity can be attributed to nano-size of amorphous NMK particles which facilitate the pozzolanic reactions necessary to produce a dense matrix with more additional CSH and less calcium hydroxide content.

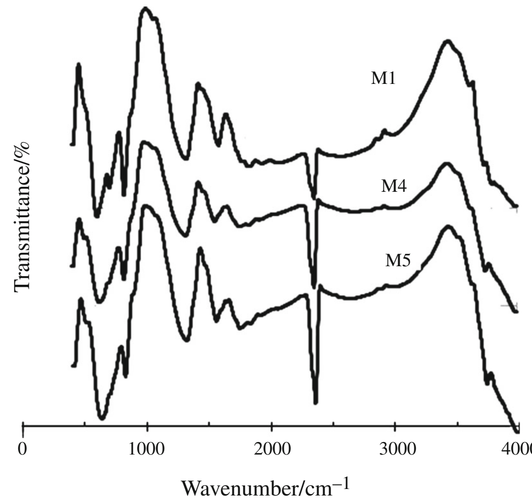

Fig. 11 FTIR spectra of composite cement mortar containing fly ash and NMK (M1, M4, and M5) at 60 days of hydration

Figure 11 illustrates the FTIR patterns of blended cement mortar containing FA and NMK hydrated for 60 days. It is clear that, the presence of a band of CO_3^{2-} at 1,429 cm^{-1} (asymmetric stretching vibration of CO_3^{2-}) and 893 cm^{-1} (out-of-plane bending vibration of CO_3^{2-}) decrease as the NMK addition increases up to 7.5 % and then increases as the NMK increases to 10 %. However, the decrease of transmittance bands as the NMK increases up to 7.5 % is due to the decrease of the total porosity which leads to reduction of carbonation. However, the increase of transmittance bands as the NMK increases up to 10 % is due to the increase of total porosity which in turn may increase the rate of carbonation of hydrated cement mortar. The vibration band of CSH at 978 cm^{-1} (stretching vibration of SiO_4^{4-}) shifts to higher wave-number values with increasing NMK content. This may be due to that the CSH formed by the pozzolanic action of FA and NMK differs in its nature from that than formed by the hydration of OPC. The transmittance band of silica at 462 cm^{-1} (bending vibration of O–Si–O) arises from the replacement of OPC with FA and NMK. The ettringite band at 1,124 cm^{-1} disappeared because SO_4^{2-} ions were probably replaced by CO_3^{2-} ions in case of FA blended cement mortars. The vibration band at 3,640 cm^{-1} corresponds to OH stretching characteristic for CH. Furthermore, other bands located at 3436, 1641 cm^{-1} indicate the presence of gypsum.

Figure 12 shows the SEM micrographs of composite cement mortar containing FA and NMK hydrated for 60 days. Obviously, the microstructures of mix M1 (FA blended cement mortar) are porous with the presence of many voids (Fig. 12a); some of the surfaces of FA particles were found to be coated with layers of small amounts of hydration products. Some of the hexagonal calcium hydroxide and ettringite needles, which grow in vacant areas in paste, were also

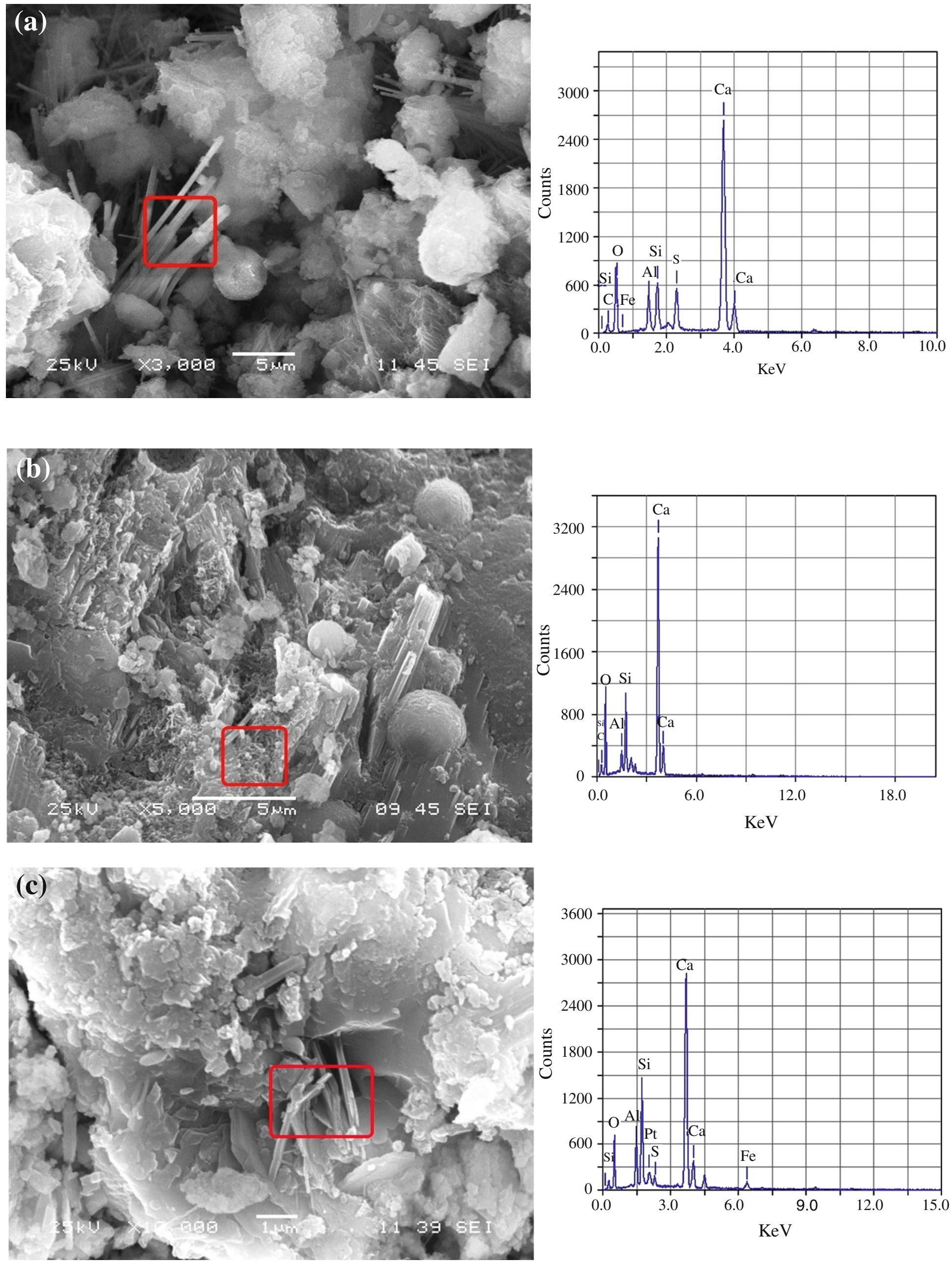

Fig. 12 SEM-EDX analysis of blended cement mortar containing FA and NMK, **a** M1; **b** M4; and **c** M5

observed. FA particles were observed in two forms: particles with a smooth surface and particles covered by layers made of hydration products and pozzolanic reaction.

In the first form, it appeared that some FA particles were still smooth, suggesting that they were unreacted or acted as an inert material with the ability to increase the packing effect and served as a precipitation nucleus for hydration compounds. The second form showed hydration and pozzolanic products around the FA particles. The corresponding EDX analysis indicates a calcium aluminate trisulfate hydrate compound. By increasing addition of NMK up to 7.5 % to FA blended cement mortar (M4 Fig. 12b), a lot of FA particles covered by hydrates appeared in the structure as the FA and NMK were activated by $Ca(OH)_2$. The pozzolanic reaction of FA and NMK was enhanced at the later ages with a marked consumption of the free $Ca(OH)_2$. The surfaces of FA particles covered by CSH were caused by the reaction of FA and NMK with $Ca(OH)_2$ and other hydration products. The corresponding EDX analysis indicates a CSH compound with associated with small amounts of aluminium. As the hydration process continued, thicker layers of hydration products on the FA and cement grains could be distinguished, although some particles still remain unreacted and acted as filling materials. Moreover, the increase of NMK addition up to 10 % (M5), leads to an increase of unreacted

NMK particles which acted as a filler material, serving as a precipitation nucleus for $Ca(OH)_2$ and CSH gel and filling the voids between cement grains (Fig. 12c); the formation of microcracks was also observed. The corresponding EDX analysis indicates a monosulphate hydrate. It is evident that the hydration products still underwent through both pozzolanic and hydration reactions. From these results, it is evident that the blended cement mortars containing FA and 7.5 % NMK produces a denser structure than that of the mortar containing FA alone.

Conclusions

Based on the results obtained in this study, the following conclusions can be drawn:

- Compressive and flexural strengths of OPC-FA-NMK blended cement mortars are higher than those of the OPC-FA blended cement mortar made with the same water/binder ratio (the same initial porosity).
- The enhancement of flexural strength at 7.5 % NMK addition was 27 % as compared to the OPC-FA blended cement (control) mortar; whereas the enhancement in compressive strength was 17.2 %.
- The total porosity of OPC-FA-NMK blended cement mortars decreases as the NMK increases up to 7.5 % and then increases as the NMK addition increases up to 10 %.
- Addition of NMK to OPC-FA blended cement mortar resulted in a marked consumption of the free lime liberated during OPC hydration.
- The SEM observations confirmed that the addition of NMK up to 7.5 % in OPC-FA-NMK mortars produces a denser structure than that of the control OPC-FA mortar. Whereas increasing the NMK addition up to 10 % leads to opening of the pore structure with the formation of microcracks.

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