Egypt. J. Phys. **Vol. 48,** pp. 45-52 (2020)

Egyptian Journal of Physics
https://ejphysics.journals.ekb.eg/

The Mechanical Properties of the Ordinary Portland Cement Containing Nano Metakaolin

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IN THIS work, the crystalline kaolin granules were grinded in a ball milling machine until reached to a nano scale size. The nano- kaolin was activated by thermal treatment. The nano- kaolin was heated for 2 hr at 750 °C to transfer the nano kaolin from the crystalline phase into the amorphous phase. The activated amorphous nano- metakaoline was investigated by differential thermal analysis (DTA), X- ray diffraction (XRD) and scanning electron microscope (SEM).

The activated nano- metakaoline was replaced by different ratios of (OPC) as 2%, 4%, 6%, 8%, and 10% by weight of cement. The compressive strength was measured at different curing times 3, 7, 14, 21 and 28 days. The thermal treatment of nano kaoline leads to the change from crystal into amorphous phase, as the decreasing of the grain size. It obvious that the compressive strength is increased, and at the concentration of 10% of nano metakaolin by 31%.

Introduction

The addition of nano materials to the ordinary Portland cement (OPC) paste has many evidence, not only the increasing of compressive strength, but also decreasing the curing time until reaches to the optimum compressive strength and reducing the cost, as a result of decreasing the amount of cement [1].

The nanotechnology can modify the concrete, such as compressive strength, volume, and stability [2]. The fire or high temperature has a harmful effect on the concrete, the deterioration processes affected on the durability of concrete structure. It is possible to minimize the harmful effect of high temperatures on concrete by taking preventive measures, such as aggregate , cement paste bond, and thermal compatibility between aggregate and cement paste [3].

Recently many researches interested in the possibility of developing concrete that has better fire resistance which can improve in different method. The cement mixed with slag, fly ash, silica fume, or metakaoline [4]. Also the addition of polypropylene fibers to concrete mix is useful [5].

The cementitious compounds are produced from the reaction of lime and siliceous materials [pozzolanas] [6]. So the clay can be considered as pozzolanic material [7].

Experimental

Materials

The materials used in this investigation were Ordinary Portland Cement (O.P.C) , and the nano- clay. The nano- clay used is prepared from the thermal activation of the nano- clay at a temperature of 750 °C for 2 hr to get activated nano- metakaolin (NMK) in amorphous state.

Samples preparation and testing

The cement pastes samples were prepared by the ratio 30% water- cement ratio (W/C), the nano-metakaoline is partially replaced by the ratios 2%, 4%, 6%, 8%, and 10%. To get a homogenous paste, the materials were mixed in an electric mixer. The paste was casted as cube 5x5x5 cm

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Received : 26/11/2019 ; accepted : 19/12/2019
DOI :10.21608/ejphysics.2019.20154.1029

for compressive strength test. The molds filled with cement paste were vibrated for one minute to remove any air bubbles. The samples were kept in molds at 100% relative humidity for 24 hr, and then cured in water for 28 days. After the periods of curing (3,7,14, and 28 days). The compressive strength measurements were carried out using five tones. German pressing machine with a loading rate of 100 kg/min. Scanning electron microscope (SEM) was used for identification of the changes occurred in the microstructure the formed and decomposed phases with a resolution of SEM was 1µm. Differential thermal analysis (DTA), thermal analyzer at a heating rate of 200 °C /min. The samples chamber was purged with nitrogen at a flow rate of 30 CC/min.

Results and Discussion

Blended nanometakaolin and cement pastes

NMK were added to the cement with a ratio of 2%, 4%, 6%, 8%, and 10% by weight of O.P.C. The water cement ratio (W/C) 30% by weight and hydrated for 3,7,14, and 28 days.

Compressive strength

The compressive strength and different ratios of NMK by 2%, 4%, 6%, 8%, and 10% by the weight of (O.P.C.) at different curing times 3,7,14, and 28 days as shown in Fig. 1. It is found that the compressive strength is increased with increasing the ratio of NMK. The compressive strength is increased approximately by 31% at 28 days of curing time for the concentration of 10% nanometakaolin. The increasing in compressive strength is mainly due to the thermal treatment of NMK produces an hydrous alumino- silicate (Al_2O_3 . $2SiO_2$) which is mainly amorphous material and behaves as a highly reactive artificial pozzolan; Also the reaction of alumino – silicate in NMK with free lime liberated during cement hydration. As well as the packing effect of NMK as filter into interstitial spaces inside the skeleton of hardened microstructure of cement paste [8].

The pozzolanic reaction between the calcium hydroxide and amorphous silica is usually slow during a prolonged period of mosit curing but it reacts rapidly in alkaline environment such as pore solution of fresh Portland cement mortar. It is clear that the reaction of NMK with calcium hydroxide is formed the additional calcium silicate hydrates (C.S.H) to those obtained from the hydration of Portland cement. The addition of NMK can improve the nucleation of CH, so NMK can activate the hydrate reaction to produce additional hydrated products, as seen in SEM micrographs.

NMK particles fill the microcraks in the matrix and increasing the resistance of crack propagation and crack opening [10].

Thermal treatment of NMK

Figure 2 illustrated the x-ray diffraction of the NMK at different concentrations 0%, 2%, 4%, 6%, 8%, and 10% .

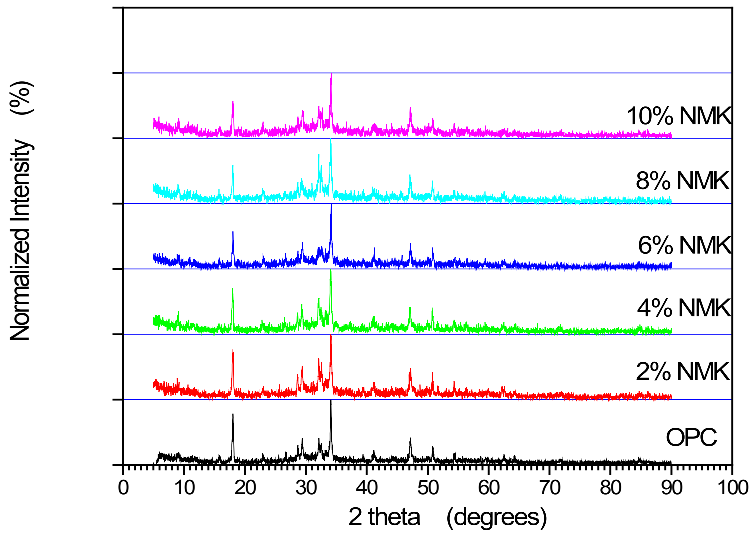


Fig. 1. The variation of change the compressive strength with different ratios of NMK at different curing ages

The interpretation led to the existence of mineral phases, quartz, kaolinite and illite. The sharp intense peak indicate the crystalline nature of nano kaolin. As in Fig. 3. The amorphous phase is clearly shown in Fig. 4.

To transfer the nano kaoline from the crystal phase into nano metakaoline (NMK) must be trated thermally at a high temperature. The differential thermal analysis (DTA) was performed for the nano kaolin to specify the decomposition/ calcinations temperature as illustrated in Fig. 5 . The nano kaolin exhibited at peak ≈ 580 °C corresponding to its dehydroxylation. This means that the conversion of kaolinite into metakaolin.

It is clear that (CH) and (CSH) ratios are higher in the ratio 6 % and 10 % respectively than OPC. To get active amorphous nanometakaolin. The nano kaoline was thermally treated at 750 °C for 2 hr. To assure that the ingredients were complete homogeneity, the nanometakaolin were rolled by porcelain ball mill for 1 hr [9].

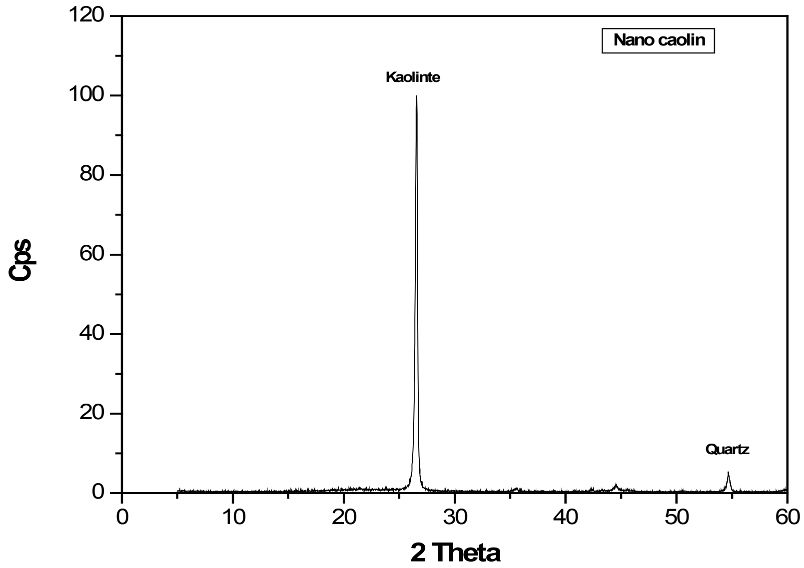


Fig. 2. X- ray diffraction pattern on nano kaolin

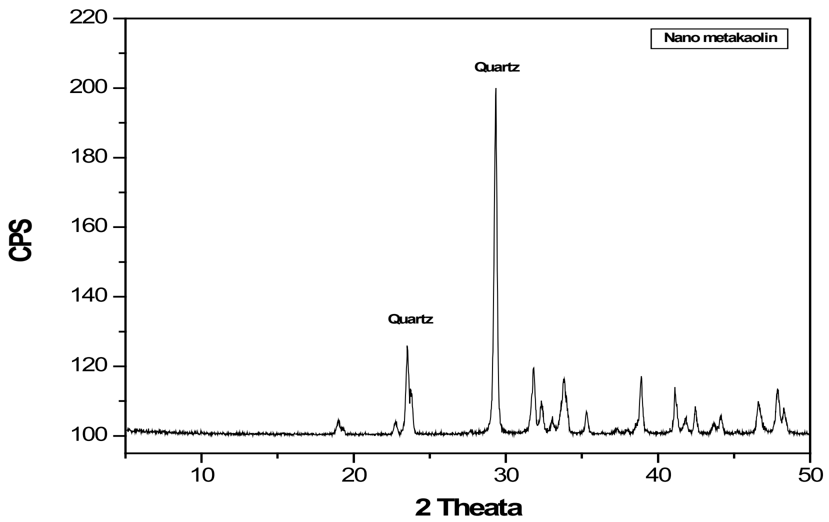


Fig. 3. X- ray diffraction pattern on nano metakaolin

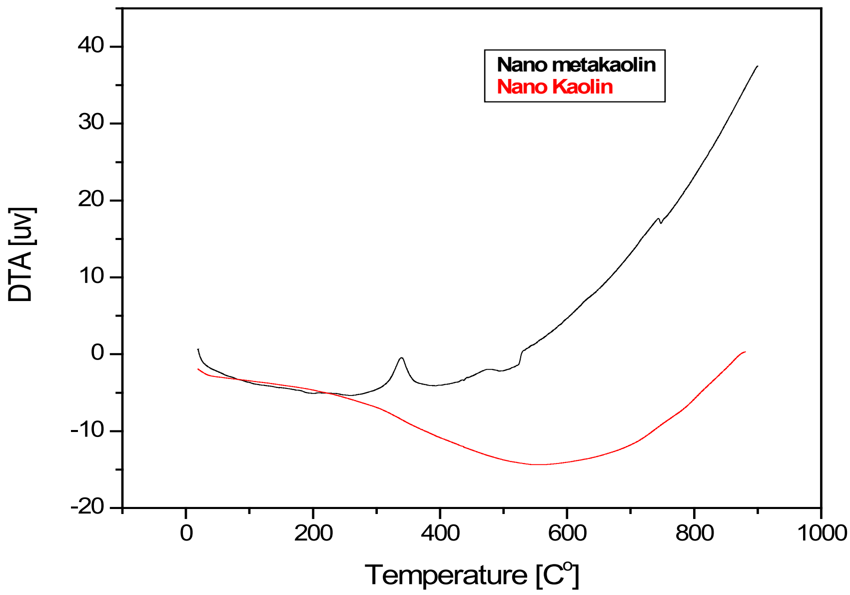


Fig. 4. DTA thermogram of nano kaolin and nano metakaolin

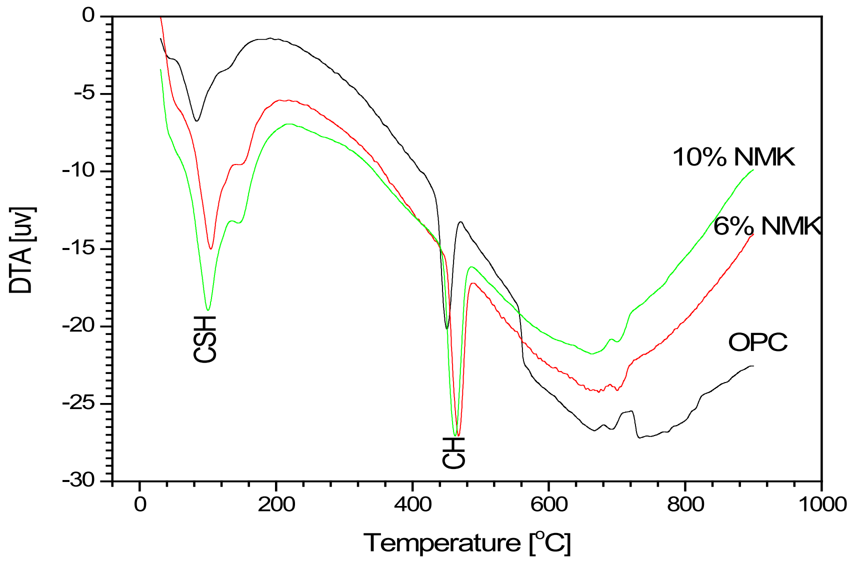


Fig. 5. DTA thermogram of OPC, OPC+ 6% NMK, and OPC+ 10% NMK .

X- ray diffraction (XRD)

The XRD patterns for (O.P.C.) and NMK with different ratios 2%, 4%, 6%, 8%, and 10% respectively and hydrated for 28 days, as in Fig. 2.

Obviously, the main compounds detected are calcium silicate hydrate (C-S-H), calcium hydroxide (CH), and calcium aluminate hydrate (CAH). It is clear that (CH) peaks decreases with increasing NMK content while the peaks associated with (C-S-H) increases slightly with increasing NMK ratios.

Evidently, the increase of the CH intensity phases in nano metakaolin clay cement pastes, hydrated for 28 days is attributed to pozzolanic reaction of the nano metakaolin clay with the free lime liberated during hydration.

Scanning electron microscopy (SEM)

Figure 6-b Shows the microstructure of neat cement paste without NMK. It was found that CSH existed in gel- like form, also SEM micrograph shows needle like monosulfate. Rather more, CH crystals were distributed in the cement paste.

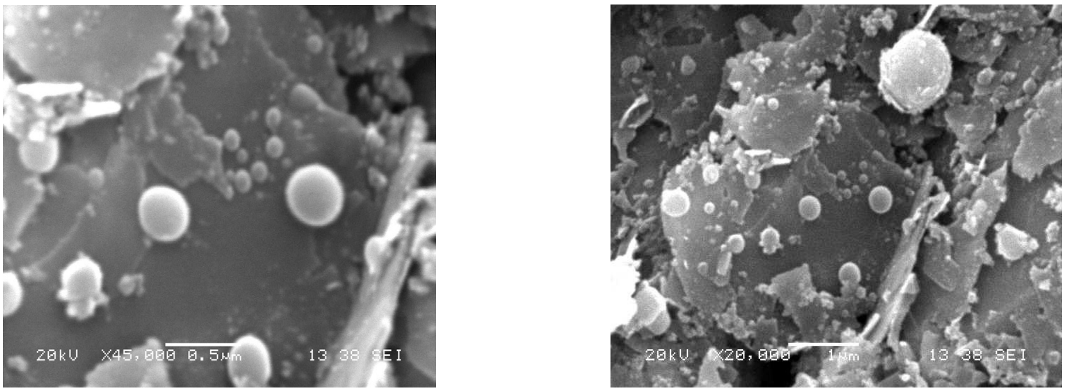
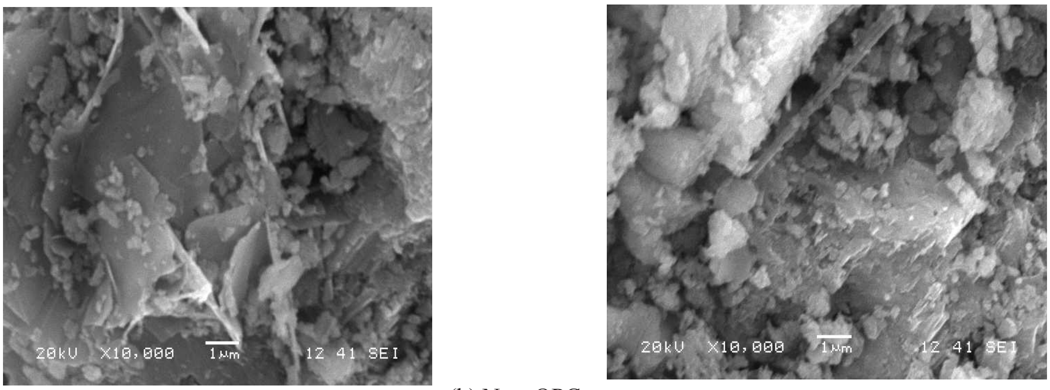
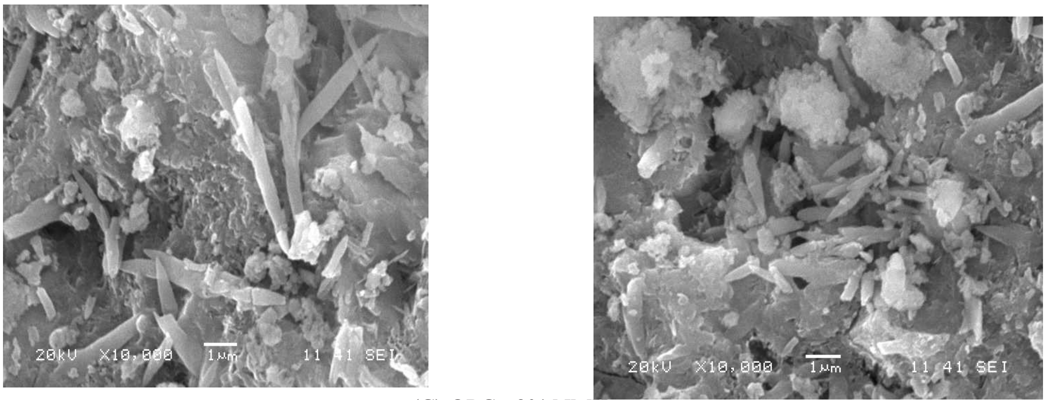


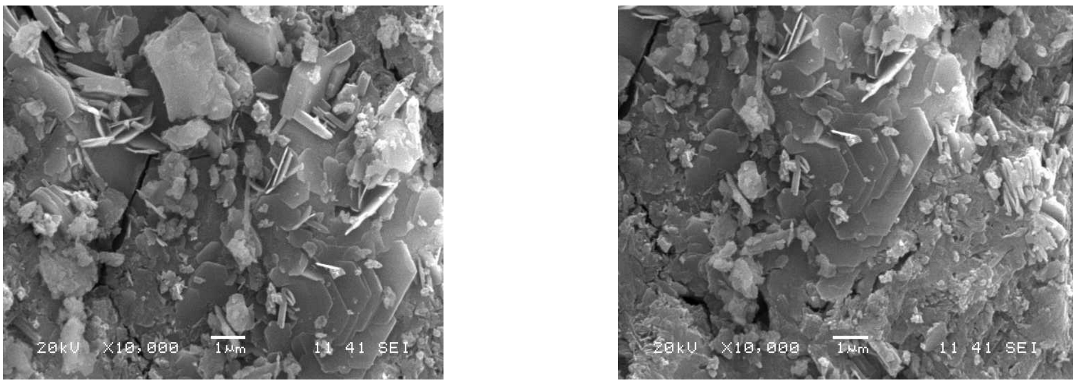
Fig. 6-a shows the SEM micrographs of the activated nano metakaolin.



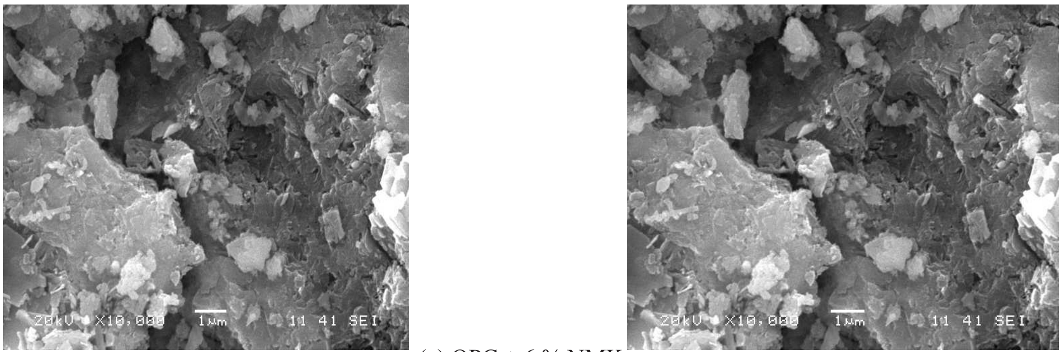
(b) Neat OPC



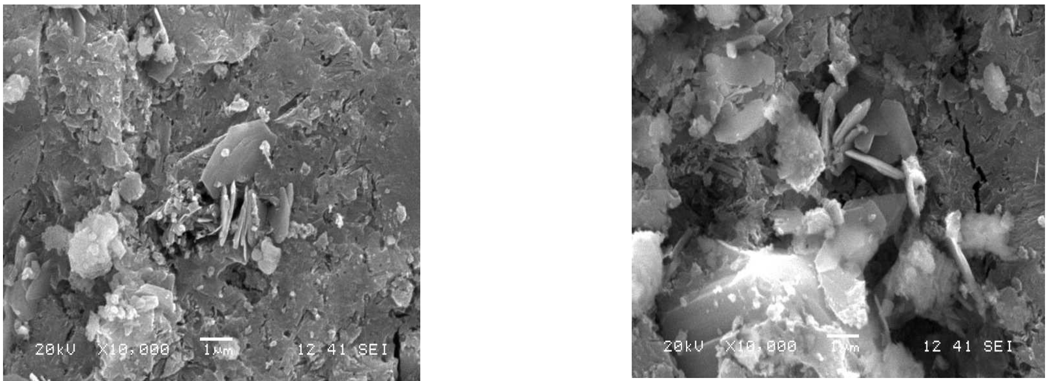
(C) OPC +2% NMK



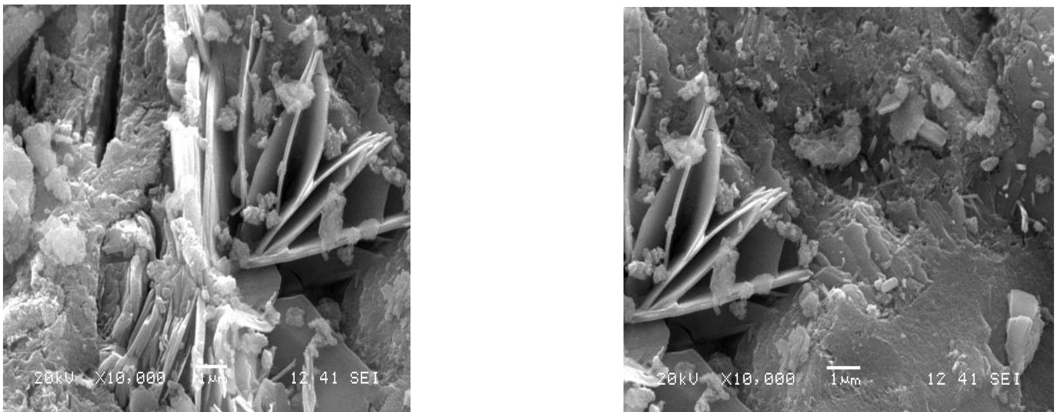
(d) OPC + 4 % NMK



(e) OPC + 6 % NMK



(f) OPC + 8 % NMK



(g) OPC + 10 % NMK

Figure 6- c, d, e, f, and g shows the microstructures of mixtures containing 2%, 4%, 6% and 8 % NMK respectively, they were compared to plain cement paste, *i.e.,* the microstructure is denser and more compact. Large CH crystals are absent. The improvement in microstructure of NMK- modified cement contributed to the enhancement in mechanical compressive strength. The NMK could improve the microstructure and strength of cement paste by a mechanism as follows; when a small quantity of the nano- particles were uniformly dispersed in the cement pastes, the hydrate products of cement will deposit on the nano- particles due to their great surface energy during hydration and grow to form conglomeration containing the nano- particles as nucleus. The nano- particles located in the cement paste as nucleus will further promote and accelerate cement hydration due to their activity. As the NMK can participate in the hydration process to generate CSH through reacting with CH, so the strength increases as the content of NMK increases even when small quantity of NMK is not very well dispersed.

The strength of the cement mortars with nano- particles has an improvement, as demonstrated in this study. Furthermore, it can be predicted that the strengthening effect of nano- particles would be further enhanced in concrete because the nano- particles improve not only the cement paste, but also the interface between paste and aggregates [10].

Conclusion

The results can be summarized as follows:

- The kaolinite transformed from crystal phase into amorphous phase, and the grain size is reduced by thermal treatment.
- The cement is partially replaced by the activated nano clay. NMK is considered as helpful tool in enhancing the compression strength of approximately 31% at the concentration of 10% of NMK at curing time of 28 dats.
- The microstructure of the hardened cement paste containing activated nano clay appeared quite dense, compact and more uniform than that of the conventional cement microstructure.

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في هذا البحث تم دراسة الخواص الميكانيكية لعجائن الأسمنت المضاف اليه الماء بنسبة **30%** من وزن الأسمنت وقد تم اضافة مادة الميتاكاولينا المعالجة حراريا عند درجة حرارة **750** درجة مئوية لمدة ساعتين . وكان اضافة المادة النانونية الميتاكولنا على حساب وزن الأسمنت بنسب **2%** و **4%** و **6%** و **8%** و **10%** . وعند قياس شدة التحمل عند أزمنة الغمر المختلفة **3** أيام و **7** أيام و **14** يوم و **28** يوم وقد لوحظ ان قوة التحمل قد زادت مع زيادة تركيزات النانو ميتاكاولينا ووصلت الى **116** ميجاباسكال عند زمن الغمر **28** يوم والتركيز **10%** من النانوميتاكاولينا**.** هذا وقد تم دراسة المادة الأمورفية النانوميتا كاولينا بأجهزة X-Ray, DSC- DTA وكذلك تم دراسة السطح عن طريق جهاز الميكروسكوب الألكتروني الماسح.