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Effects of Nanometakaolin on the Physico-Chemical Characteristics of Various Blended Cement Pastes

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Abstract

The physico-chemical characteristics of various blended cement pastes are studied in presence of nanometakaolin (2, 4, 6 and 8%) and in absence and presence of some industrial wastes such as ground clay bricks, cement kiln dust and silica fume. These characteristics are investigated at different hydration times (1-180 days) via determination of the compressive strength, total porosity and chemically combined water content. In addition, the phase composition of the formed hydrates is investigated through X-ray diffraction analysis. The IR spectra of hardened cement pastes are also recorded. The results indicate that the presence of nanometakaolin accelerates the hydration of the pastes which exhibit higher compressive strength than the neat ordinary Portland cement, and improves some other characteristics of the different pastes.

Keywords: Nanometakaolin, Blended cement, Pysico-chemical characteristics of blended cement.

1. Introduction

Nanotechnology is one of the most promising areas of science. The development in nano-science can have a great impact on the field of construction materials. Ordinary Portland cement (OPC), is obviously the product with great, but not completely explored potential. It is known that chemical activation is an effective way to improve the strength of cement-based materials. Many trials are performed to partially replace OPC by some other cheaper additives which result as by-products from some industries and may improve the cement-pastes characteristics. Examples of these materials are metakaolin, nanometakaolin, ground clay bricks

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cement kiln dust and silica fume. These by-products may also be used to reduce costs, energy and pollution.

The effect of these additives on the mechanical and chemical properties of plain cement pastes is investigated and reported in the literature.

Metakaolin (MK), a valuable pozzolanic material, can react with the lime liberated from the hydration of OPC forming calcium silicate hydrate. It is a thermally activated aluminosilicate material obtained by calcining kaolin clay within the temperature range of 700–850^{o}C ***[*****Ambroise, 1994, Sabir et al., 1996 and Shebl et al., 2009]**. MK, when ground in a high speed grinding mill, is reduced to the nano scale (NMK) **[Aiswarya et al., 2013]**. An increase in compressive, flexural strength and improvement of mechanical properties of mortars containing nano-particles are observed by **Sobolev et al. (2006)**.

Ground clay bricks (GCB), known as homra, is a solid material produced from the manufacture of clay bricks. In Egypt about 5-10% of homra is a waste product. It is constituted mainly of silica quartz, aluminosilicates, anhydrite and hematite. Therefore, it acts as a good pozzolanic material which shows a slow rate and heat of hydration. Homra also shows good resistance to aggressive media such as sulphate and chlorides ***[*****Heikal et al., 2000 and Heikal and El-Didamony, 1999]**. **Bektas et al. (2008 and 2009)** showed that when it is finely ground, clay brick, obtained from demolished masonry, can be recycled as a pozzolanic cementitious material in concrete. It was found that 10% replacement of cement by GCB gave the highest compressive strength **[Kartini, 2012]**.

Cement kiln dust (CKD), a by-pass dust, is generated in large quantities during the production of PC. It is a fine powdery material of gray to tan in color [similar in appearance to OPC], highly alkaline powder and relatively uniform in size. **Al-Harthy et al. (2003)** reported that there was generally a decrease in compressive strength with an increase in CKD replacement for cement. ***Pavia and Regan (2010)*** observed that the compressive strength increases with the CKD content with the exception of 5% CKD mortar.

Silica fume (SF) is an amorphous mineral material composed of extremely small and chemically active particles of SiO_2 that appear as a by-product in silicon or ferrosilicon industries. More than 95% of SF particles are finer than 1µm. **Almusallam et al. (2004) and**

Mazloom et al. (2004) concluded that the higher compressive strength noted in the SF cement concrete, compared to plain cement concrete, may be attributed to the reaction of the SF with calcium hydroxide liberated during the hydration of cement. **Rang et al. (2014)** indicated that the hydration process could be accelerated at the beginning by addition of SF, and fly ash. However, retarded hydration may occur mainly in the dormant and acceleration periods.

So the present study aims to investigating the effect of the presence of NMK on the mechanical properties and microstructure of some different blended-Portland cement pastes as will be mentioned below.

2. Materials and Methods

2. 1. Materials

The materials used in this investigation are: ordinary Portland cement supplied from Helwan cement factory with blain surface area of 2945 cm^2/g, nano metakaolin prepared from kaolinite by burning at 800° C for 2 hours, ground clay bricks, cement kiln dust and silica fume (SF) obtained from Toura factory and El Nasr Pharmaceutical chemical company, respectively. The chemical analysis of the utilized materials are given in Table (1),

2. 2. Preparation of the cement pastes

Cement and the different additives are first mixed in the dry state to attain complete homogeneity. The pastes are prepared using W/S ratio = 0.27. Table (2) shows the designation and the percentage composition of the prepared mixes. After complete mixing, the resultant pastes are molded into specimens by using one-inch cubic moulds. The moulds containing the pastes are cured in 100% relative humidity for 24 hours, then the specimens are removed from the moulds and cured under water for different hydration periods, namely; 3, 7, 28, 90 and 180 days. Some of the hardened cement pastes are tested by XRD (Cu Kα) and FT-IR spectra to investigate their structure. Also, the compressive strength, porosity and combined water content are determined for some chosen samples.

Materials	Oxide (%)									Ignition Loss	Insoluble residue	SM	AM
	SiO_2	Al_2O_3	Fe_2O_3	CaO	NaO	K_2O	Cl	MgO	SO_3				
OPC	20.1	4.72	3.94	62.98	--	0.27	0.04	1.77	2.67	2.95	0.91	--	--
NMK	61.24	20.89	6.38	0.16	1.61	0.71	--	0.38	0.17	13.62	--	--	--
GCB	74.8	14.03	5.04	1.25	--	--	--	1.3	0.8	2.58	--	--	--
CKD	12.11	3.05	1.92	46.86	0.29	4.44	4.15	2.04	10.89	7.31	--	2.44	1.61
SF	96.8	0.2	0.4	0.16	--	0.03	--	0.09	0.3	1.98	--	--	--

Table (1): The chemical composition of the used materials.

Mixes	Composition %				
	OPC	NMK	GCB	CKD	SF
Mo	100	0	0	0	0
MIA	98	2	0	0	0
MIB	96	4	0	0	0
MIC	94	6	0	0	0
MID	92	8	0	0	0
MIIA	80	2	10	8	0
MIIB	70	4	20	6	0
MIIC	60	6	30	4	0
MIID	50	8	40	2	0
MIIIA	80	2	10	0	8
MIIIB	70	4	20	0	6
MIIIC	60	6	30	0	4
MIIID	50	8	40	0	2

Table (2): The designation and composition of the prepared mixes.

3. Results and discussion

3. 1. Compressive strength

The values of compressive strength of the control mix (M_o) and blended cement mixes containing various percentages of nanometakaolin (MIA→MID) are given in Table (3). Generally, it is found that the compressive strength increases on increasing the percentage of NMK up to 8%at all hydration ages. Similar finding are reported by **Morsy et al. (2008, 2011 and 2012)** and by **Petal (2012).** Also **Aiswarya et al. (2013)** found that partial replacement of cement with NMK (2-20%) has a greater influence on the strength of concrete than control concrete. Hence, the obtained results are explained on the basis that ultra-fine particles of NMK may fill the voids in cement pastes, thus making its microstructure somewhat denser. Also, the pozzolanic reaction of NMK with the free lime and calcium hydroxide (released during the hydration process) may produce excess calcium silicate hydrate that gets deposited in the pore system. This deposited Ca-silicate hydrate may cause the improvement of the compressive strength.

Mix	Compressive strength (Kg/cm^2)					
	1 day	**3 days**	**7 days**	**28 days**	**90 days**	**180 days**
Mo	158.1	213.4	260.9	332	379.4	424.5
MIA	189.7	237.2	316.2	419	442.7	474.3
MIB	213.4	268.8	363.6	442.7	474.3	490.1
MIC	237.2	284.6	395.3	474.3	505.9	545.4
MID	268.8	316.2	411.1	505.9	537.5	584.9

Table (3): Compressive strength at different hydration ages of the cement mixes containing NMK only.

Tables (4& 5) show the change of the compressive strength of the cement mixes at different hydration ages on adding different percentages of GCB (10-40%) in presence of very small percentages (2-8%) of either CKD or SF, respectively. It can be shown that the increase in the compressive strength of the cement pastes reaches its optimum value at 4% NMK, 20% GCB and 6% CKD or SF, and hence 70% OPC. Then the values of the compressive strength are decreased as the percentage of GCB increases and those of CKD or SF are decreased. It is to be noticed also that the optimum compressive strength value is almost the same on using either CKD or SF. The increase in the compressive strength at 10 and 20 % of GCB may be due to the pozzolanic reaction of GCB and SF. However, the decrease in the strength at higher GCB concentration may be attributed to two factors; the dilution of OPC resulting in the decrease of C_3S and β-C_2S phases in the blended cement pastes and agglomeration of GCB around the OPC grains thus hindering the hydration process. Accordingly, the amount of hydration products is decreased leading to a decrease in the compressive strength as also reported by **Kartini (2012)**. The slight increase in the compressive strength values in presence of SF than in case of CKD may be due to the pozzolanic reaction of SF with the librated lime leading to more production of (the binder agent) CSH **[El Alfi, 2004]**.

Mix	Compressive strength (Kg/cm²)					
	1 day	**3 days**	**7 days**	**28 days**	**90 days**	**180 days**
Mo	158.1	213.4	260.9	332	379.4	424.5
MIIA	181.8	230	268.8	347.8	395.3	439
MIIB	189.7	258	297	371.5	424	461
MIIC	160	200	250	311	359	391
MIID	145	176	227	291	346	360.6

Table (4): Compressive strength at different hydration ages of the cement mixes containing NMK, GCB and CKD.

Mix	Compressive Strength (Kg/cm²)					
	1 day	**3 days**	**7 days**	**28 days**	**90 days**	**180 days**
Mo	158.1	213.4	260.9	332	379.4	424.5
MIIIA	189.7	253	284.6	363.6	400	441.1
MIIIB	197.6	268.8	300.4	379.4	420	465.3
MIIIC	170	225.7	272	345.3	389.9	432.6
MIIID	154	200.1	250.7	315	363	395.3

Table (5): Compressive strength at different hydration ages of the cement mixes containing NMK, GCB and SF.

3. 2 . The porosity

Table (6) shows the total porosity of all mixes at the two hydration ages 7 and 180 days as representative examples. It is obvious that the total porosity of all the investigated mixes decreases with increasing the hydration ages. This may be due to the progress of the hydration reaction and formation of more hydration products (CSH) which fill the pores.

Such results agree with those of the compressive strength. Table (6) also shows that the values of porosity decrease in presence of CKD and SF compared to that of the control mix. This can be explained as being due to the retardation effect caused by filling the pores by these admixtures.

Hydration Time	Cement Mixes												
	Mo	MIA	MIB	MIC	MID	MIIA	MIIB	MIIC	MIID	MIIIA	MIIIB	MIIIC	MIIID
7 days	**33**	**28.8**	**28.2**	**27.8**	**27**	**32.3**	**31.9**	**33.4**	**33.8**	**29**	**28.5**	**29.4**	**30.1**
180 days	**31**	**26.1**	**24.2**	**23.8**	**22.9**	**28.8**	**28**	**29.1**	**30.9**	**26**	**25**	**25.4**	**26.6**

Table (6): The porosity values of blended cement pastes at the hydration ages 7 and 180 days.

3. 3. The chemically combined water

The values of the combined water content are found to increase with increasing hydration time for all the blended cement pastes as shown in Tables (7, 8 & 9). This is due to the continuous hydration and accumulation of the hydration products. Table (7) indicates that the addition of the different percentages of NMK (2,4,6 and 8%) increases the combined water content at all the hydration ages compared to that of the control mix (Mo). A similar conclusion is reported by **Abd El-Aleem et al. (2005)**. However, the presence of the other additives GCB/CKD and GCB/SF (Table 8 and 9, respectively) lowers the values of the combined water content relative to those of Mo and MIA-MID mixes. This decrease in the values becomes more considerable as the percentages of GCB is increased from 10 to 40 %. This may be accounted for the high hydraulic properties of cement phases rather than other additives which are characterized by low or no hydraulic properties **[Shoaib et al., 1999]**. Also, it has been shown recently that the increased hydration products due to pozzolanic reactions of metakaolin result in reduced values of water sorptivity **[Aly et al., 2011]**.

Mix	Combined water (%)

	1 day	3 days	7 days	28 days	90 days	180 days
Mo	9.8	12.7	14.2	15.8	18.1	19.4
MIA	10.0	13.0	15.0	17.0	18.9	19.8
MIB	10.5	13.5	15.6	17.7	19.5	20.8
MIC	10.9	14.0	16.3	18.2	20.1	21.7
MID	11.3	14.4	17.0	18.9	20.7	22.4

Table (7): Combined water at different hydration ages of the cement mixes containing NMK only.

Mix	Combined Water (%)					
	1 day	3 days	7 days	28 days	90 days	180 days
Mo	9.8	12.7	14.2	15.8	18.1	19.4
MIIA	8.5	11.5	13.1	14.4	16.6	18.0
MIIB	7.9	10.6	12.4	13.6	15.9	17.2
MIIC	6.8	9.3	11.5	12.9	15.0	16.4
MIID	5.8	8.5	10.3	11.7	14.1	15.2

Table (8): Combined water at different hydration ages of the cement mixes containing NMK, GCB and CKD.

Mix	Combined water (%)					
	1 day	3 days	7 days	28 days	90 days	180 days
Mo	9.8	12.7	14.2	15.8	18.1	19.4
MIIIA	9.2	12.0	13.7	15.0	17.2	18.5
MIIIB	8.5	11.3	13.0	14.4	16.5	17.9
MIIIC	7.4	10.5	12.1	13.7	15.6	17.0
MIIID	6.3	9.2	11.0	12.5	14.7	16.1

Table (9): Combined water at different hydration ages of the cement mixes containing NMK, GCB and SF.

3. 4. Phase composition:

The X-ray diffraction (Cu Kα) partterns obtained after 90 days hydration of the control mix (Mo) and in the presence of 2, 4, 6 and 8% NMK (MIA-MID, respectively) are shown in Fig. (1). It can be observed that, the intensity of the peaks characteristic of calcium hydroxide (CH) is gradually decreased as NMK-percentage is increased. This may be due to the high pozzolanic activity of NMK.

The diffraction patterns of the cement pastes containing 2, 4, 6 and 8% of CKD and SF in addition to the considered percentages of NMK and GCB (MIIA-MIID and MIIIA-MIIID, respectively) after 90 days hydration are given in Figs. (2 and 3, respectively). The intensity of the peaks characteristic of CH is found to decrease in case of the first two mixes (MIIA, MIIIA and MIIB, MIIIB) . Then, the peak's intenisties increase in the other two mixes (MIIC, MIIIC and MIID, MIIID). Also, it can be noticed that the intensities of the CH peaks in presence of CKD are higher than those in presence of SF. This can be attributted to the higher pozzolnic activity of SF than that of CKD.

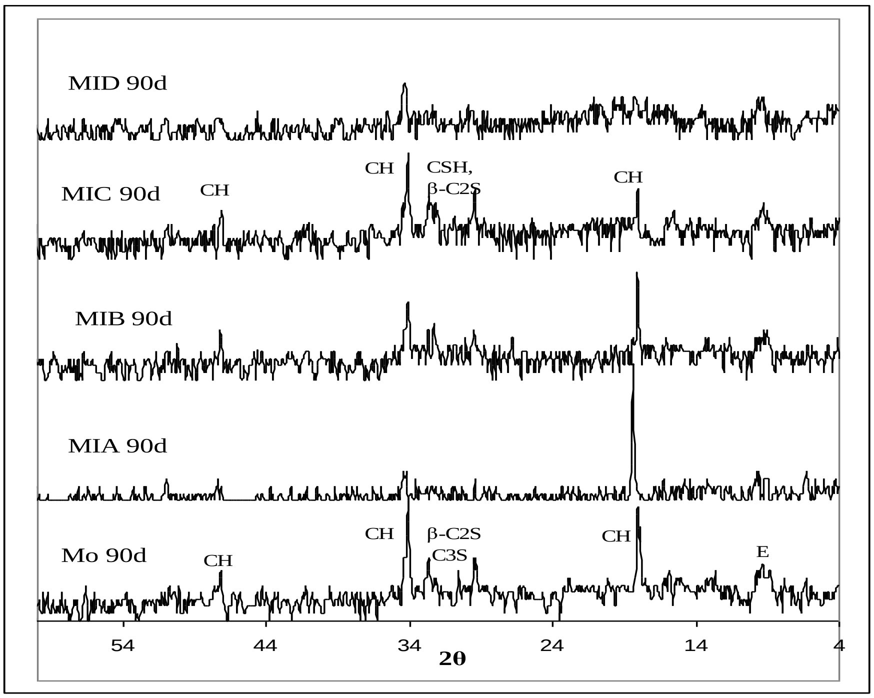

Figure (1): X-ray diffraction patterns of the control mix Mo and MIA-MID mixes at 90 days hydration time.

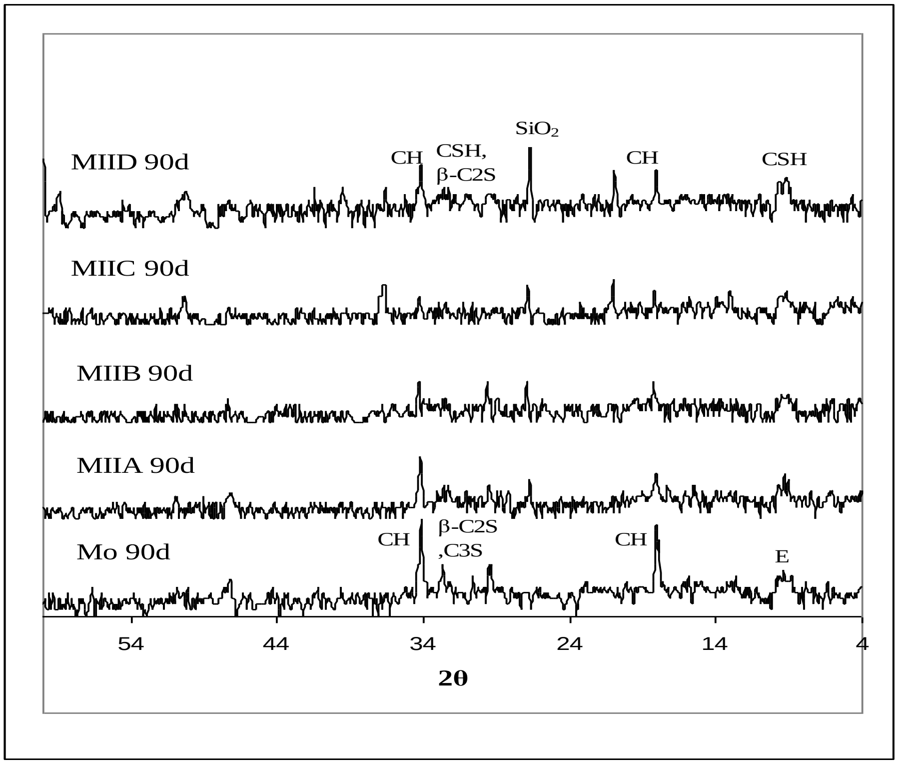

Figure (2): X-ray diffraction patterns of the control mix Mo and MIIA-MIID mixes at 90 days hydration time.

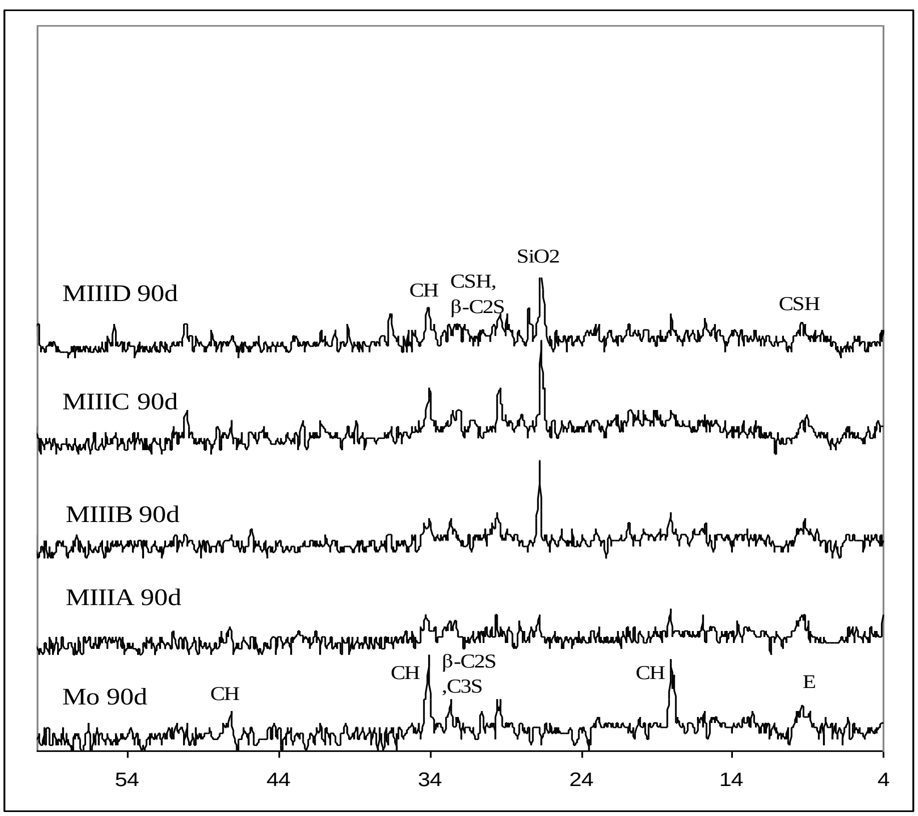

Figure (3): X-ray diffraction patterns of the control mix Mo and MIIIA-MIIID mixes at 90 days hydration time.

3. 6. Infrared Spectroscopy (IR)

FT-IR technique is used to investigate the IR spectra of the cement pastes in presence of NMK and the additives: GCB, CKD and SF. The IR spectra of some of the tested cement mixes, namely Mo, MIA, MID, MIIA, MIID, MIIIA and MIIID are presented in Figs. (5 and 6). Absorption bands at 3641- 3642 nm due to the stretching vibration of the (OH) group of $Ca(OH)_2$ in additiom to strong bands at 3450 and 1650 nm attributed to –OH or H_2O stretching and bending vibrations, respectively, clearly appear in the spectra **[Theophile, 2012 and Ylmen et al., 2009]**. The bands at 1424, 875 and 712 nm are attributed to ύ (CO_3). The strong bands at 970 and 515 nm may be accounted for the (Si-O) absorption bands of the C-S-H phase. Absorptions at 2360 and 2337 nm are supported to be due to the stretching vibration of ύ(Si-H) of C-S-H.

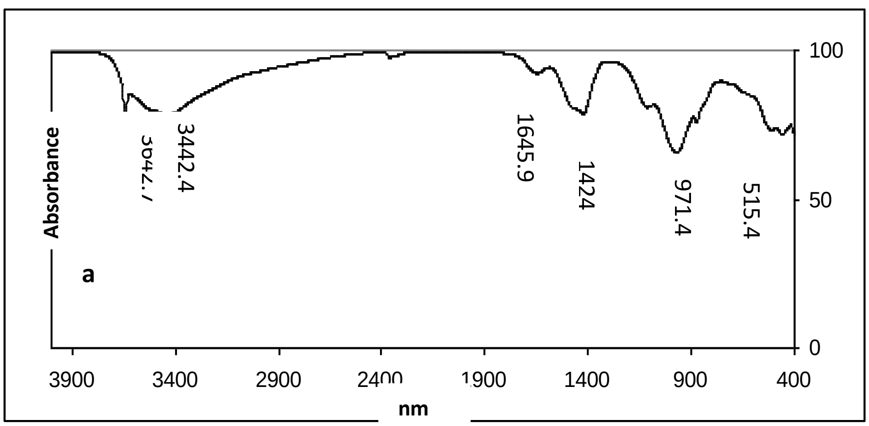

Figure (5 a): IR spectra for blank cement pastes hydrated for 90 days.

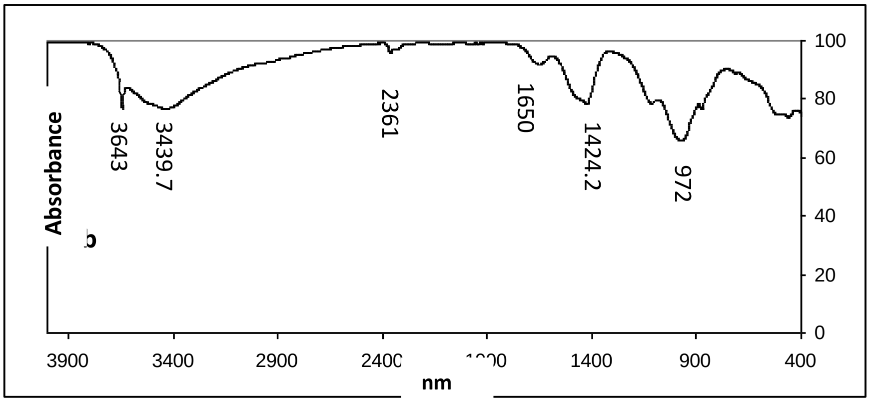

Figure (5b): IR spectra for blended cement paste MIA hydrated for 90 days.

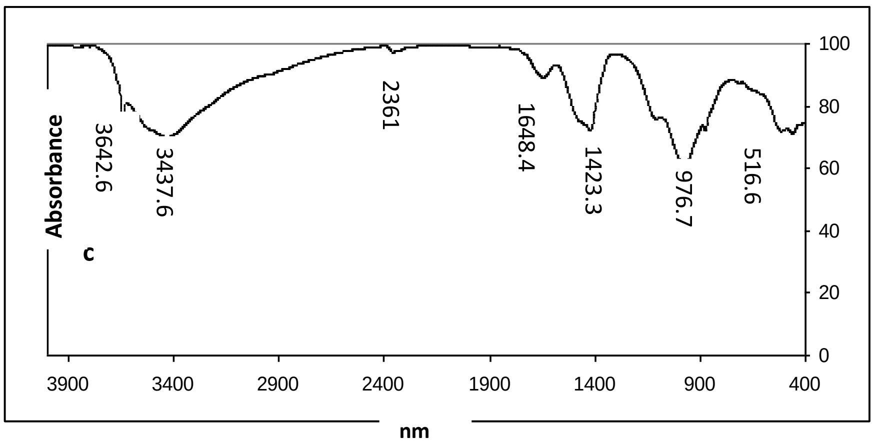

Figure (5c): IR spectra for blended cement paste MID hydrated for 90 days.

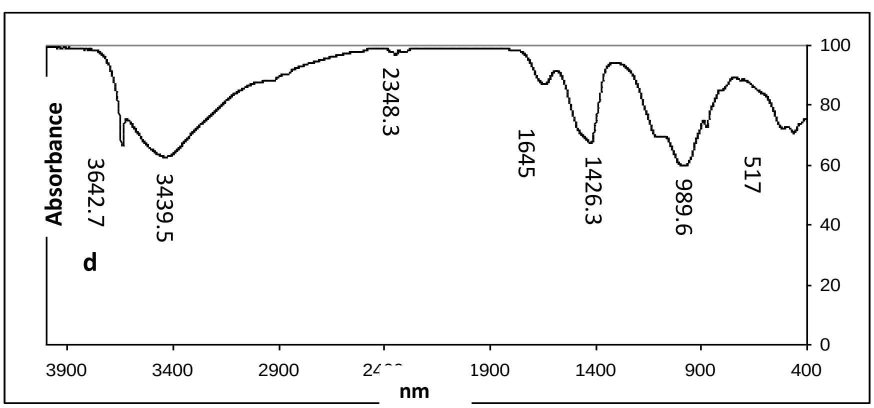

Figure (6d): IR spectra of blended cement paste MIIA hydrated for 90 days.

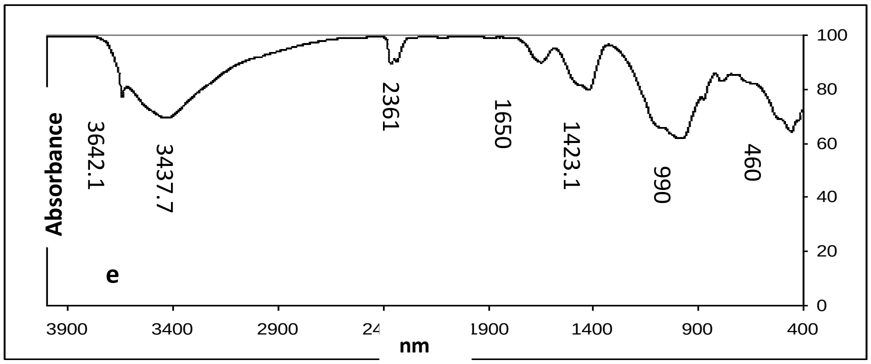

Figure (6e): IR spectra of blended cement paste MIID hydrated for 90 days.

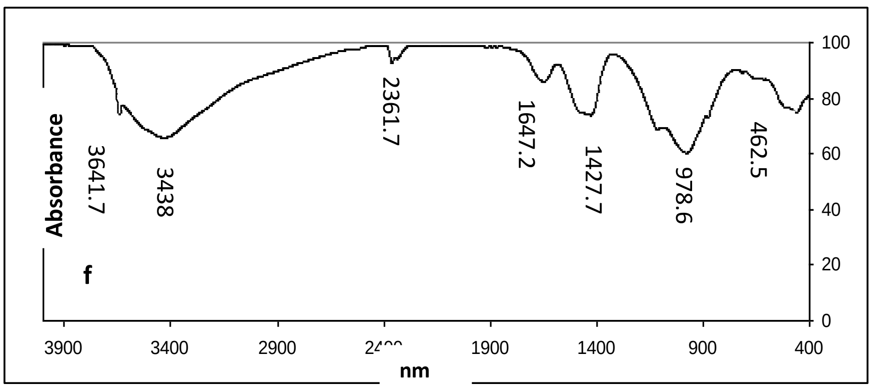

Figure (6f): IR spectra of blended cement paste MIIIA hydrated for 90 days.

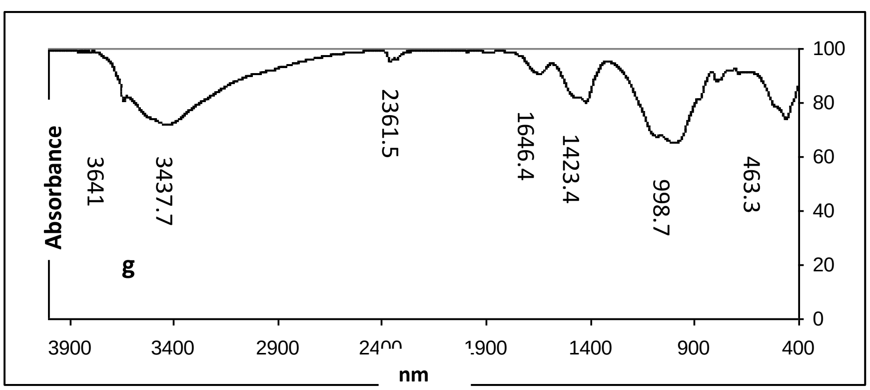

Figure (6g): IR spectra of blended cement paste MIIID hydrated for 90 days.

4. Conclusion

From the above results, it can be concluded that:

- The compressive strength of all the blended cement mixes increases on increasing the hydration time, while, the combined water content for these mixes percentages decrease on increasing hydration time.
- The compressive strength of the blended cement pastes at all hydration times is increased on adding NMK.
- The blended OPC mixes with NMK show relatively lower values of total porosity compared to that of the control mix.
- The optimum value of the compressive strength is exhibited at 4% NMK, 20% GCB and 6% SF or CKD.

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الملخص العربي

تأثير إضافة النانو ميتا كاولين على الخصائص الفيزيوكيميائية و الميكانيكية لعجائن مختلفة من الأسمنت المخلوط

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تعتبر صناعة الاسمنت من اكثر الصناعات الملوثة للبيئة و ذلك لانبعاث ثانى اكسيد الكربون و تراب الافران منها , و لذلك اتجهت الانظار الى استبدال جزء من الاسمنت ببعض الاضافات التى تعمل على تحسين الخواص الفيزيائية و الميكانيكية مثل النانو ميتاكاولين و بعض المخلفات الصناعية مثل الحمرة و السيلكا فائقة النعومة و تراب افران الاسمنت و بذلك يتم التخلص من بعض المخلفات الضارة و تقليل كمية الاسمنت المستخدمة مما يساعد على توفير الطاقة المستخدمة و كذلك التقليل من تلوث البيئة .

تهدف هذة الدراسة الى استبدال جزء من الاسمنت ببعض المخلفات الصناعية مثل الحمرا و تراب الافران و السيلكا فائقة النعومة بالاضافة الى النانو ميتاكاولين كعامل محفز للعجائن و التى تعمل على زيادة سرعة تأدرت الاسمنت و ملئ الفراغات بين الجزيئات مما يزيد من قوة تحمل الضغط الميكانيكى و تقليل المسامية للعجائن المتصلدة .

يمكن تلخيص اهم النتايج التى حصلنا عليها فى ما يلى :

- قوة التحمل للضغط الميكانيكى تزداد مع الزمن لجميع العجائن المتصلدة المحتوية على الاسمنت الصافى او الاسمنت و الاضافات الاخرى, بينما تقل كمية الماء المتحدة بزيادة ازمنة التأدرت للعينات المحتوية على الاسمنت المخلوط .
- تزداد قوة التحمل للضغط الميكانيكى للعينات المحتوية على نانوميتاكاولين حتى 8 % عن عجائن الاسمنت الصافى.
- زيادة نسبة النانوميتاكاولين تعمل على نقص المسامية فى جميع الازمنة بالمقارنة بعينات الاسمنت الصافى.
- اضافة الحمرة بنسبة 10 , 20 % تعمل على نقص المسامية للعجائن المتصلدة بالمقارنة بالعينة القياسية بينما تزداد المسامية فى العينات المحتوية على 30 , 40 % من الحمرة.
- اان اضافة 4% من النانو ميتاكاولين و 20% حمرة و 6% سيليكا فائقة النعومة او تراب افران الاسمنت تعطى اعلى نتائج فى قوة التحمل للضغط الميكانيكى.