Influence of Nano-Silica on the Properties of High Free Lime Ordinary Portland Cements

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ABSTRACT

In the recent years, there is a great interest in replacing nanomaterials (NMs) in concrete structure to produce concrete with novel function and better performance. NMs are used either to replace part of cement, producing ecological profile concrete or as admixtures in cement pastes. A number of NMs such as nano silica (NS) have been explored and extensively used. This work aims to study, the effect of replacing 2, 4 and 6 mass % (NS) on physico-mechanical properties of OPC containing high CaO. The hydration behavior of cement pastes was followed by the determination of (CH) and combined water (Wn) contents with curing time. The water of consistency (W/C), setting times (IST&FST), penetration of chloride and compressive as well as flexural strengths were also estimated. The hydration products were analyzed using XRD technique. The results showed that, NS improves the hydration behavior and the physico-mechanical properties of Pozzolanic cements. This is due to that, NS not only acts as filler to improve the microstructure, but also as activator to promote pozzolanic reaction with CH, which enhances the formation of excessive hydration products especially C-S-H. The higher beneficial role of NS is mainly due to its   high surface area, filling effect and pozzolanic activity. The blended cement with 2.0 % NS gave the optimum mechanical properties at early ages while at later ages all cement pastes are similar and better than the blank sample.

Key Words: Hydration; High free lime; Mechanical properties; Nanosilica; Mortar; Curing time

1. INTRODUCTION

Nowadays, the micro-level does not provide enough insights into building materials. Therefore, allover the world, increasing amounts of funding are being directed to research material properties on the nano-level, which is claimed to have a tremendous potential for the future (Quercia, et al., 2014). Nanotechnology (NT) has become an important key in the field of construction and building materials which can be considered as the most modern aspect in every domain of science and technology (Singh, et al., 2013 & Said, et al., 2012).

The evolution of NT provides materials with new properties and over the last years a lot of efforts have been put to introduce nano-materials (NMs) into cement pastes, mortars and concretes in order to improve their properties and produce new materials with novel function as well as better performance at unprecedented levels (Stefanidou, et al., 2012). Actually, NMs can change the concrete world, due to their unique physicochemical properties, which differ from those of the traditional conventional materials (Xiao, et al., 2004).

Nano engineering of cement-based materials can result in outstanding or smart properties. Introduction of (NT) in cement industry has the potential to address some of the challenges such as CO_2 emissions, poor crack resistance, low tensile strength, high water absorption, low ductility and many other mechanical performances. An improvement in the mechanical properties and durability of cementitious materials can be observed with incorporation of nano-SiO_2, ZnO_2, Al_2O_3, TiO_2, carbon nanotubes, NMK, carbon nano fibers and others (Jo, et al., 2007).

NMs were also used either to replace part of cement, producing ecological profile concrete or as admixtures in cement pastes (Ridha, et al., 2007). In both cases, their addition improves the performance of cement pastes (Senff, et al., 2007). When using NMs, three main advantages are considered in fresh and hardened states i) Production of high strength concrete for specific applications, ii) Reduction of cement consumption for specific grade of concrete, and iii) The reduction of the construction period, because NMs can produce high strength concrete (HSC) at short curing time (Stefanidou, et al., 2012). These advantages will decrease the national energy consumption, the overall cost of the structure, and the environmental pollution to a great extent (Olivier, et al., 2012 & Quercia, et al., 2010).

Due to their sizes, some researchers have recorded an increased water demand for mixtures with NMs of the same workability (Sobolev, et al., 2009). Also, their tendency to agglomerate can be restricted by using dispersing admixtures or by applying different techniques during mixing (Porro, et al., 2005). The fundamental processes that govern the concrete properties are affected by the performance of the material on nano-scale. The main hydration product of cement-based materials, the CSH gel, is a nano-structured material (Sanchez, et al., 2010). The nano-scale revealed that, the nano-crystallized CSH and also nano-particles (NPs) have been found to act as nuclei for, promoting their hydration (Zyganitidis, et al., 2011).

The mechanical properties of concrete depend mainly on the refinement of the microstructure of hardened cement paste and improvement of the paste aggregate interfacial zone (ITZ) (Quercia, et al., 2014). The role of NPs can be summarized as follow: i) NPs act as fillers in the empty spaces; ii) well dispersed NPs act as crystallization centers of hydrated products, increasing hydration rate of cement phases, and iii) NPs assist towards the formation of small sized CH crystals as well as homogeneous clusters of C-S-H (Quercia, et al., 2014, & Xiao, et al., 2004 & Quercia, et al., 2010).

Nano-SiO_2 has a significant role to increase the compressive strength and reduce the overall permeability of hardened concrete. This is attributed to the pozzolanic reaction of the amorphous NS with the liberated CH during the hydration of cement, forming finer hydrated phases (CSH gel), densified microstructure (nano-filler and anti-leaching effects), reduced permeability, and enhanced mechanical properties (Belkowitz, et al., 2010 & Heikal, et al., 2013).

This work aims to study the physico-mechanical and chemical properties of OPC pastes prepared from substitution of different percentages of High free lime OPC by NS up to 6.0 mass, %. The water of consistency, initial and final setting times were determined for each cement paste. Also, the combined water, free lime contents, and compressive as well as flexural strengths of hardened mortars were measured up to 90 days. In addition, XRD, and penetration of chloride were investigated.

2. Materials and Experimental Techniques

2. 1. Materials

The starting materials used in this study were the ASTM Type (I) high free lime (HFL.OPC) and nano-silica (NS). HFL.OPC with Blain surface area of 3020 ± 50.0 cm^2/g was provided from Lafargeholcim Cement Company, Egypt. Their chemical and mineralogical analyses are given in Tables (1, 2) and Nano-silica (NS) with average particle size, Blaine surface area and of purity about 15.0-50.0 nm, 50.0 m^2/g and 99.9 %.

2. 2. Experimental Techniques

Nano-silica was prepared from a desired amount of Na$_2$SiO$_3$ solution was diluted with distilled water; then stirred for 15.0 min, and precipitated using dil HCl. The precipitate was filtered and washed several times with distilled water till free from chloride, and then dried overnight and milled by ball mill (600 rpm) for 10.0hrs. The amorphous nature of NS-particles was verified using XRD, and TEM techniques, Figs.1, and 2 respectively. The hump in the XRD is mainly due to the amorphous structure of NS.

HFL.OPC was partially substituted with NS 2, 4 and 6 mass; %. Each dry mix was blended in a steel ball mill for 1.0 h and mixed in a rotary mixer in complete order to attain high degree of homogeneity. The required water of standard consistency gives a paste which permitted the settlement of the Vicat plunger (10.0 mm in diameter) to a point 5.0-7.0 mm from the bottom of the Vicat moulds. It was measured to get all specimens having the same workability. The required water of consistency and setting times for each mix were determined according to ASTM specification (ASTM, C191. 2008). Each dry blend was mixed with the required amount of water consistency. The hydration product of some selected cement pastes were analyzed using XRD technique. On other side, the compressive and flexural strengths were carried out on mortar prisms and the chloride penetration was carried out on concrete.

Table 1: Chemical oxide analysis of HFL.OPC (mass, %)

Oxides	SiO$_2$	Al$_2$O$_3$	Fe$_2$O$_3$	CaO	MgO	SO$_3$	Na$_2$O	K$_2$O	F.L	LOI	LOI
mass,%	18.18	4.20	4.14	62.39	2.02	2.91	0.43	0.40	5.00	3.80	103.47

Table 2: Mineralogical composition of HFL.OPC

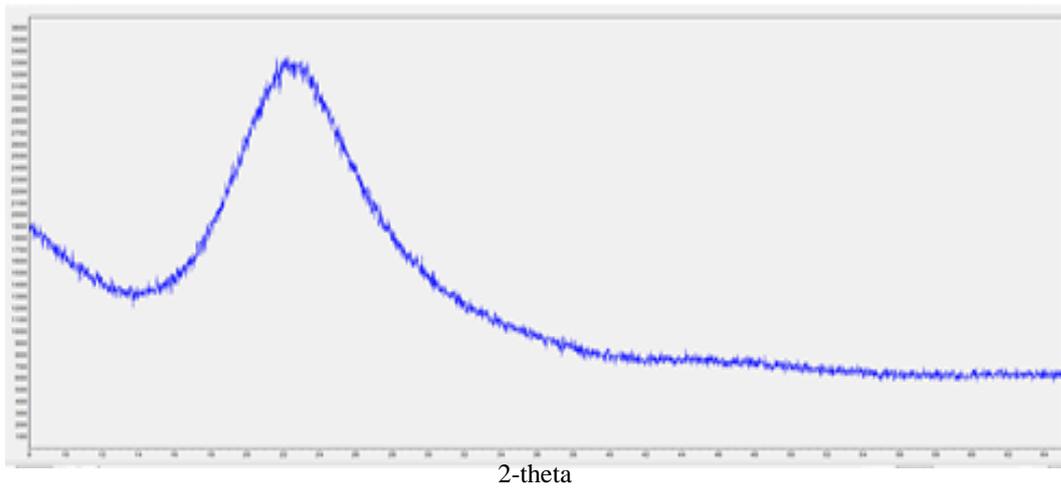| Compound	Abbreviation	Chemical formula	Content, %
Tri-calcium silicate	C$_3$S	3CaO.SiO$_2$	47.10
Di-calcium silicate	C$_2$S	2CaO.SiO$_2$	22.95
Tri-calcium aluminate	C$_3$A	3CaO.Al$_2$O$_3$	2.49
Tetra-calcium aluminoferrite	C$_4$AF	4CaO.Al$_2$O$_3$.Fe$_2$O$_3$	13.59

2-theta

Fig. 1: XRD pattern of nano-silica (NS)
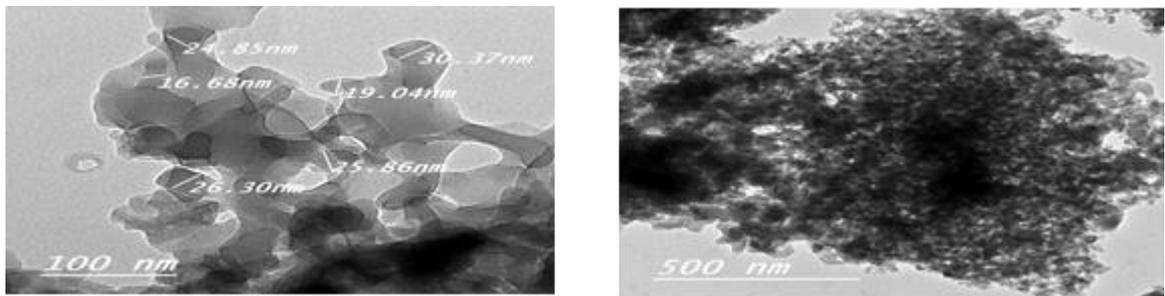
Fig. 2: TEM of nano-silica

Table 3: Mix composition of blended cements, (mass, %)

Mix No.	M0	M1	M2	M3
HFL.OPC,%	100	98	96	94
N.S,%	0	2	4	6

The cement blend mortars were prepared by mixing of cement and Lafarge standard sand proportions with water. Freshly prepared cement mortars were placed in $40\times40\times160$ mm prisms into two approximately equal layers manually compacted and pressed until a homogeneous specimen was obtained. The prisms were vibrated for a few minutes to remove any air bubbles to give a better compaction. The mix composition of different cement blends (M0, M1, M2 and M3) containing 0, 2,4 and 6 wt% NS is seen in table [3].

The cement pastes was done by using water of consistency (W/S) cured in a humidifier at about (100% R.H) at room temperature 23.0 ± 2.0ºC for 24.0 h, then immersed in tap water until the time of testing (2, 7, 28 and 90 days). After the predetermined curing time, the hydration of cement pastes was stopped by pulverizing 10.0 g of representative sample in a beaker containing methanol-acetone mixture (1:1), and mechanically stirred for 1 h. The mixture was filtered through a gouch crucible, G4 and washed several times with the stopping solution then with ether. The solid was dried at 70.0^{o}C for 1.0h to complete evaporation of alcohol, and then collected in polyethylene bottles sealed and stored in desiccators for analysis (Abd-El.Aziz, et al., 2012).

The Wn, % is considered as the ignition loss of the dried sample (on the ignited weight basis). Approximately 2.0 g of the after dried sample were ignited at 1000^oC for 1h (El-Didamony, et al., 2005).

The free lime content of each hydrated cement paste was estimated by glycerol-ethanol method. (El-Didamony, et al., 2005).

The powder method of XRD was adopted in the present study. For this, a Philips diffractometer PW 1730.0 with X-ray source of Cu kα radiation (λ=1.5418Å) was used. The scan step size was 2θ, the collection time 1s, and in the range of 2θ from 5.0^o to 55.0^o. The X-ray tube voltage and current were fixed at 40.0 KV and 40.0 mA respectively. An on-line search of a standard database (JCPDS database) for X-ray powder diffraction pattern enables phase identification for a large variety of crystalline phases in a sample (Ramachandran, 2001).

Compressive and flexural strengths were determined according to EN (197-1:2011& ES 4756-1:2013). A set of three prelims were tested on a compressive strength machine (3R), Germany, with maximum capacity of 150.0 MPa force.

The resistance to chloride ion penetration is an important aspect that needs a better definition in structural materials. Accelerated chloride permeability test was conducted on all the concretes and the total charge passing in 6 h as a measure of the chloride permeability. The chloride ion penetrability limits suggested by ASTM C1202 was also superimposed. The Penetration chloride test was done in Industrial Expert Services - Product & Laboratory IPC-CAI Laboratory (Lafargeholcim cement Company).

3. Results and Discussion

3.1. Water of standard consistency and setting times

Figure (3) show the variations of water of consistency (W/C, %) and setting times of the cement pastes with various NS, %. The results show that, the water of consistency increases with NS content up to 6.0 mass,%, which is mainly attributed to the increase of surface area and decrease of crystal lattice (Bjornstrom, 2004). Therefore, the paste containing NS require more water to rapid forming of hydrated products (Nazari, et al., 2011). The initial and final setting times are elongated by replacement of OPC with 2.0% NS due to the formation of excessive amount of CSH, which fill up some of open pores originally filled with water that accelerates the setting. At 4.0-6.0% NS, the setting times are slightly elongated, due to either the increase of water of consistency or the coating effect of some NS particles on the cement paste (Jo, et al., 2007).

3.2. Chemically combined water contents (Wn, %)

The variation of Wn %, of hydrated OPC and blended cement pastes as a function of NS contents and curing time are graphically represented in Fig. (4). It is apparent that, Wn, %, increases with curing time for all hydrated cement pastes. This mainly due to the continuous hydration of cement phases as well as NS-pozzolanic reaction, leading to the formation of more hydrated products. It is obvious that, the values of Wn increase with the amount of NS content from 2.0 to 6.0 mass %, up to 90 days. This means that the effect of NS is mainly effecting at early ages of hydration. The increase of Wn, % with NS content is mainly due to two factors. The first is the high-water demand and the second is the pozzolanic activity of NS. This reacts with the liberated lime during the hydration of cement, leading the formation of additional CSH and the combined water contents increase.
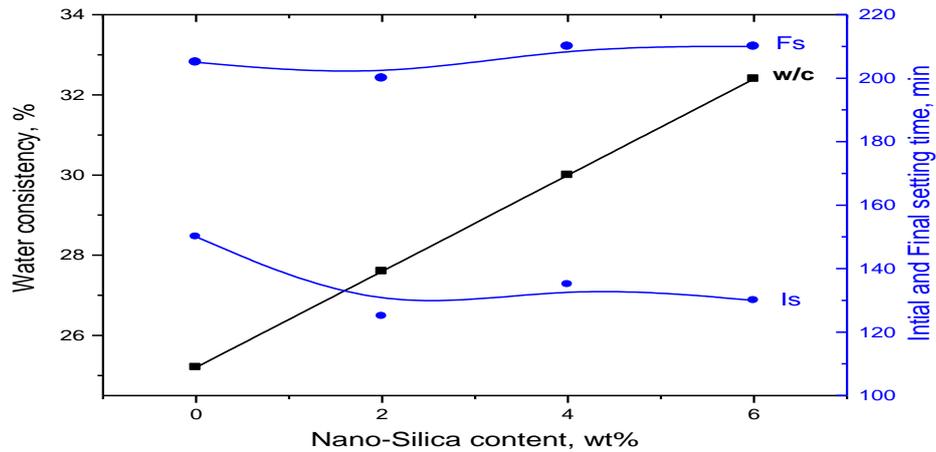
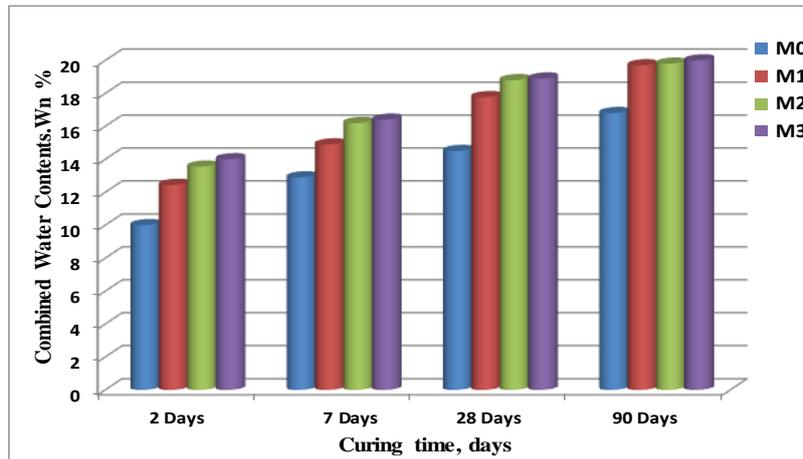Fig. 3: Water of consistency initial and final setting times of HFL.OPC and NS-cement pastes

Fig. 4: Combined water contents of hydrated HFL.OPC and NS-cement pastes up to 90 days

The results also show that, NS accelerates the hydration of cement phases, especially at early ages of hydration beginning from the time of mixing up to 28 days (Qing, et al., 2007). It is clear that, NS gives higher Wn contents than OPC up to 90 days.

3.3. Free lime contents (F.L, %)

The pozzolanic reaction of NS with the liberated $Ca(OH)_2$ during cement hydration can be followed by monitoring the decrease in F.L,% with curing time and NS contents . The CH of hydrated OPC and NS-modified cement pastes up to 90 day is graphically plotted in Fig.(5). The results show that, the F.L, % of the blank cement paste (OPC) increases with curing time as a result of the hydration of silicate phase (β-C_2S and C_3S). On the other side, the substitution of NS tends to decrease the residual protlandite (CH), due to the pozzolanic reaction between the amorphous glassy NS and liberated free CH as well as the already present free lime (Heikal, et al., 2013).

It is clear that, the cement paste with NS gives lower CH contents than OPC. This result is also in agreement with that of combined water contents. The free lime content of admixed cement pastes cured for 90 days increases due to the continues hydration of CH from C_2S and C_3S phases. The high Wn content at later ages with NS contents are mainly due to agglomeration of the filled NS in the cement pastes.
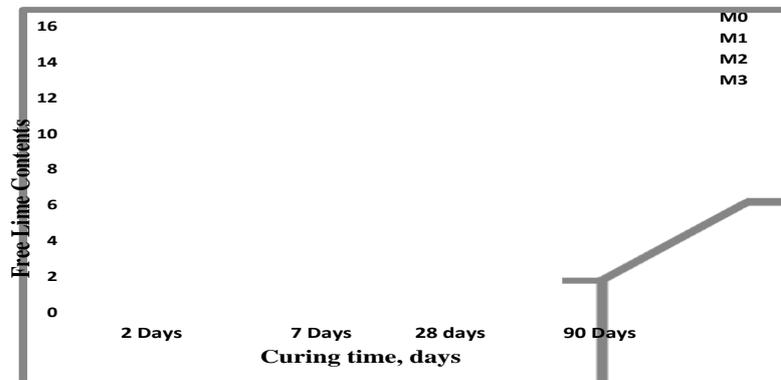

Fig. 5: Free lime contents of hydrated HFL.OPC and NS-cement pastes up to 90 days

3.4. XRD analysis

Figure 6 illustrates the XRD patterns of M0, M1 and M2 pastes cured for 7 days which show the presence of (CH), $CaCO_3$, C-S-H and unhydrated phases C_2S, C_3S. It is clear that the CH decrease with NS particles due to its reaction with CH in the pozzolanic reaction. Figure 7 show the XRD patterns of M0 and M1 hydrated for 28 days. It is clear that the CH decreases with the substitution of 2 mass % NS due the pozzolanic reaction. The CSH peak is overlapped with that of $CaCO_3$.
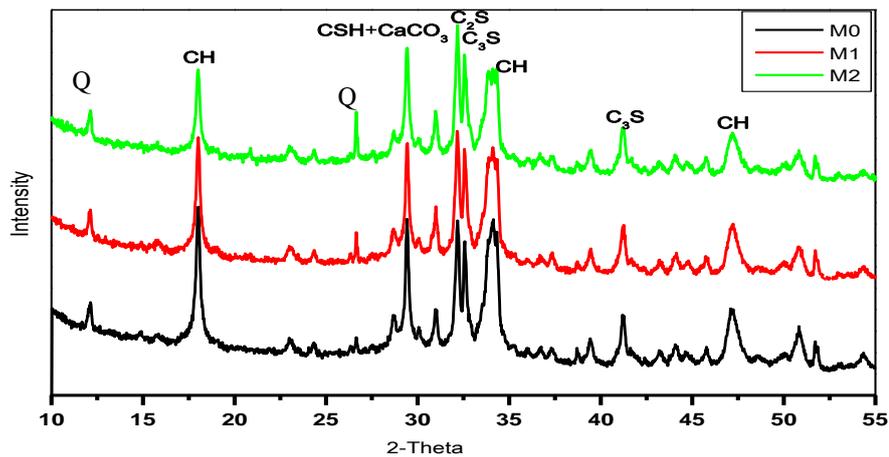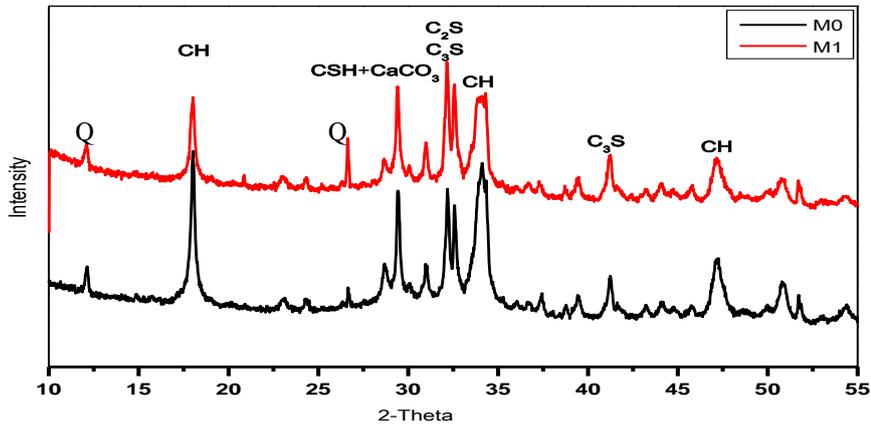

Fig. 6: XRD patterns of M0, M1and M2 at 7 days

Fig. 7: XRD patterns of M0 and M1 at 28 days

3.5. Compressive strength

The effect of NS contents on the compressive strength (CS) of hydrated OPC and blended cement mortars up to 90 days is graphically represented in Fig.(8). It can be seen that, the CS increases with curing time for all hydrated cement mortars, due to the continuous hydration and formation of successive amounts of hydrated silicates, which are the main source of strength. These products accumulate in water filled pores to form a more compact body. Also, the compressive strength of the investigated cement mortars increases sharply with NS % up to 2.0 mass, %, after 2 days is due to that; nano-SiO_2 behaves not only as a filler to improve microstructure, but also as an activator to promote pozzolanic reaction. Both the nucleation and pozzolanic effects of NS lead to more accumulation and precipitation of hydration products especially CSH gel which is the main factor for strength in the open pores originally filled with water, leading to the formation of homogeneous, dense and compact microstructure. Therefore the compressive strength (CS) increases with NS up to 2.0 mass, %. (Li, et al., 2007).

3.6. Flexural strength

Finger (**9**) shows the flexural strength of admixed cement mortars with NS up to 90 days. It is clear that the substitution of OPC with (2-6 wt %) NS increases the flexural strength after 2 days of curing time. As the hydration proceeds flexural strength of admixed mortars is higher than the blank. This is mainly due to the presence of 5% wt % free lime in OPC which decrease the strength. Also, the substitution of OPC with 2 wt % (NS) gives higher adverse than those admixed with 2 wt % NS which is mainly sufficient for the consumption of $Ca(OH)_2$ then decreases the strength. This results good agreement with that of CS. Also, 2 mass % NS is sufficient to increase the flexural strength.
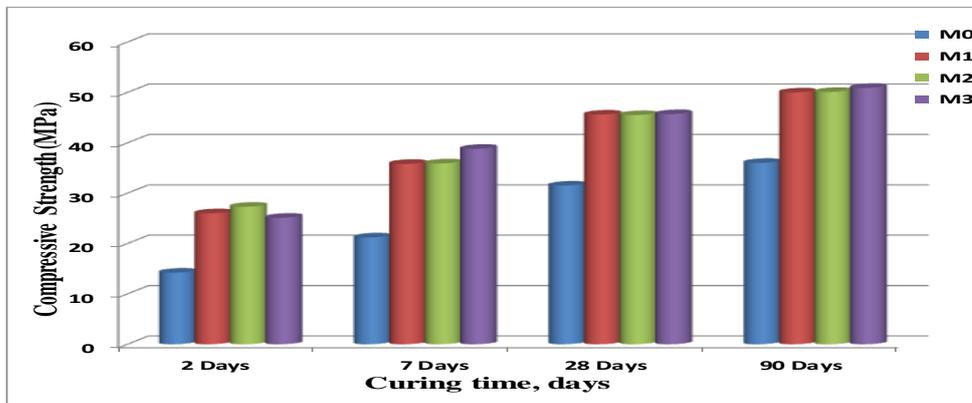

Fig. 9: Compressive strength of hardened HFL. OPC and NS- cement mortars up to 90 days
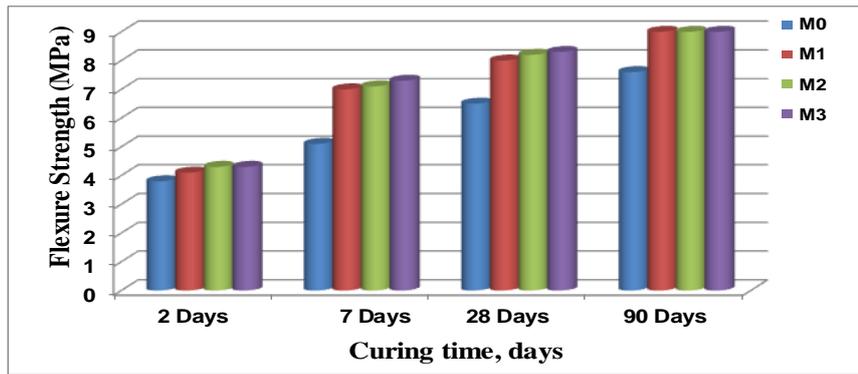
Fig. 9: Flexural strength of hardened HFL.OPC and NS-cement mortars up to 90 days

3.7. Penetration of chloride

The penetration of chloride of admixed concretes up to 28, 56, 90 days is plotted in Fig. (10). It can be seen that, all the NS concretes show very low chloride permeability due to the pozzolanic reaction of NS with CH to form CSH which is accumulated in the open pores then forming compact cement paste of the concrete and the penetration of chloride decreases. In contrast, the control concrete (M0) shows the higher chloride permeability, indicating clearly that the high reactivity of NS substantially reduced chloride ion penetration in concrete and attributed to the effect of porosity in the ions transport through the pores network, with the result of cementitious with lower permeability to chlorides. Such reductions can be expected to have a substantial impact on the service life of concrete in chloride environments. The substitution of NS increased the system's capacity to bind chloride ions, thus reducing the free chloride ion availability (Pellegrini-Cervantes, et al., 2015).The generated CSH gel due to the pozzolanic reaction and the filler effect of NS particles result in pore reduction in the cement matrix and decrease its permeability. Further, due to greater pozzolanic action and filler effect of NS particles, the permeability of the concrete incorporated with NS decreases. The mixes with 4 &6 wt % NS had a significant improvement, on the chloride resistance (Garg, et al., 2016).

It is clear that the penetration of chloride through the admixed NS concrete decreases with NS content as well as curing time which reduce the permeability of the chloride due to increase of accumulated C-S-H gel decrease total of porosity and the accumulation of the hydration products in the open pores.
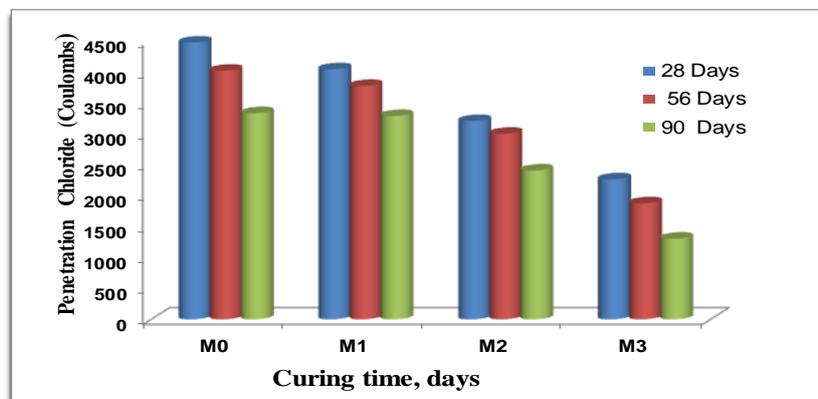

Fig. 10: Penetration chloride of the hardened HFL.OPC and NS-cement concretes as a function of curing time

4. Conclusions

From the above findings it can be concluded that:

1- The water demand increases with NS content up to 6.0 mass, %, due to the increase of surface area and decrease of crystal lattice of NS.

2- The initial and final setting times are elongated by replacement of HFL. OPC with 2.0 mass, % NS, due to the high water of consistency.

3- The chemically combined water contents (Wn, %) increase with the NS content, due to the high-water demand and the pozzolanic activity of NS, with hydration of OPC.

4- The compressive and the flexural strengths of hardened cement mortars increase sharply with NS, up to 2 mass %, this improvement is due to that; NS behaves not only as a filler to improve microstructure, but also as an activator to promote pozzolanic reaction. Both the nucleation and pozzolanic effects of NS lead to more accumulation and precipitation of hydration products in the open pores, leading to the formation of dense and compact microstructure which increase the strength.

5- The chemical and physico-mechanical tests are in a good agreement with those of XRD results.

6- The results show that, HFL.OPC can be advantageously replaced by 2.0 mass % NS and this substitution is suggested to be the most effective level for producing high-performance blended cement with a high reduction in chloride penetration.

Recommendation

The utilization of (about 2%) nano silica can be treating the high free lime Portland cement to give high-performance blended cement.

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تأثير النانوسيليكا على خصائص الأسمنت البورتلاندي العادي المحتوي علي الجير الحر

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إن إستخدام تكنولوجيا النانو في مجال الإنشاء ومواد البناء ذو أهمية كبيرة ، ذلك لأن استخدام تلك المواد ذات الأحجام الصغيره جدًا في هذا المجال يحقق اربع فوائد رئيسية. الفائدة الأولى تتضمن إنتاج خرسانة عالية الأداء يمكن استخدامها في أغراض نوعية. الفائدة الثانية تشمل تقليل كمية الأسمنت المُستخدم ة في إعداد خرسانة لها نفس القوة و خفض تكلفة الخرسانة وكذلك خفض التلوث البيئى الناتج عن صناعة مواد البناء. الفائدة الثالثة تتضمن تقليل الوقت المُستهلك في عملية الإنشاءات المعمارية لأنها تعطي خرسانة عالية الكفاءة عند زمن أقل مقارنة بالخرسانة العادية. وأخيرا تقلل من كمية الطاقه المطلوبه لصناعة الأسمنت.

إن تقليل كمية الأسمنت المستخدمة في الخرسانة يعني ترشيد الاستهلاك المحلي للطاقة وتقليل التلوث البيئي الناتج عن الكمية المتزايدة من تصاعد ثاني أكسيد الكربون من مصانع الأسمنت .

الأسمنت المحتوي علي 5%من الجير الطليق يعتبر خارج المواصفات القياسية ونهدف في هذه الدراسة الي معالجتة .

يهدف هذا البحث الي دراسة هيدرة عجائن الأسمنت البو رتلاندي المُحضر بالاستبدال الجُزئي للأسمنت البورتلاندي العادي بنسب متفاوتة من النانو سيليكا حتي 6% بالوزن من الأسمنت المحتوي علي الكلس الحر حتي 5 %. تم تحضير عجائن الأسمنتات باستخدام المياه القياسية للخلط و تعيين زمني الشك الابتدائي والنهائي لكل عينة ثم متابعة عملية الهيدرة وذلك بتعيين كل من محتو ى الجير الطليق، الماء ال متحد كيميائيًا ، وقوى التحمل للضغط الميكانيي للعينات المتصلدرة وكذلك نفاذيه ايونات الكلور حتي 90 يوم. وتم فحص نواتج الهيدرة باستخدام حيود الآشعة السينية.

المواد المستخدمة في هذا البحث هى : الأسمنت البورتلاندي العادي المحتوي علي 5% من الجير الطليق من شركة لافارج هولسيم للأسمنت ـ مصر بمساحه سطحية 3000 ± 50 سم2/جرام، أما النانو سيليكا المُستخدمة تم تحضيرها من الصوديوم سليكات بالطريقه الكيميائية حتى حجم 15 نانومتر . بتحليل هذه الماده باستخدام جهاز حيود الأشعة السينية وجد ان النانوسيليكا المُستخدمة هي سيليكا غير متبلرة (الطور الزجاجى للسيليكا).

تتضمن نتائج هذه الدراسة مايلى:-

1- أظهرت النتائج أن ا ستبدال 2 ، 4 ، 6 % من الأسمنت البورتلاندي العادى المحتوي علي 5 % من الجير الحر بالنانوسيليكا أدى إلى زيادة كل من ماء الخلط وزمني الشك مقارنة بتلك العينات الأسمنتية .

2- إن استبدال 6-2% من الأسمنت البورتلاندي العادي المحتوي علي 5 % من الجير الحر الطليق بالنانو سيليكا كان له تاثير إيجابي علي الخواص الكيميائية والفيزيقو ميكانيكية للعينات المتصلدرة وير جع ذلك إلي النشاط البوزولاني العالي وكذلك المساحة السطحية العالية للنانو سيليكا مقارنة بالأسمنت. وجد أن جزيئات النانو سيليكا لاتعمل فقط كجزئيات مالئة للفراغات النانونية في عجينة الأسمنت ولكنها أيضا تقوم بنشاط بوزولاني عالي حيث تتفاعل مع الجير الحر الموجود وكذلك الناتج من هيدرة كلنكر الأسمنت وينتج عن ذلك هيدرات سيليكاتية ذات كثافة عالية وتُحسن التركيب الدقيق للعينات وينعكس ذلك إيجابيًا علي الخواص الميكانيكية.

3- ومما سبق يمكن القول إن إحلال 2 % من النانو سيليكا في الأسمنت البورتلاندي العادي المحتوي علي 5% من الجير الحر يعطي نتائج فيزيقو-ميكانيكية أعلى من الأسمنت البوتلاندي العادي .