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Review Paper

An Overview on the Influence of Nano Silica in Concrete and a Research Initiative

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Abstract

Nano science and technology is a new field of emergence in materials science and engineering, which forms the basis for evolution of novel technological materials. Nano technology finds application in various fields of science and technology. This article presents a critical review of the literature on the influence of nano silica in concrete and its application for the development of sustainable materials in the construction industry and to study the pore filling effect and its pozzolanic activity with cement towards improvement of mechanical properties and durability aspects. Thus, there is a scope for development of crack free concrete towards sustainable construction.

Keywords: Nano silica, cement paste, cement hydration, concrete, flowability, mechanical properties

Introduction

Concrete is a highly heterogeneous material produced by mixture of finely powdered cement, aggregates of various sizes and water with inherent physical, chemical and mechanical properties. A reaction between the cement and water yields calcium silicate hydrate, which gives concrete strength and other mechanical properties of concrete, as well as some by-products including calcium hydroxide [CH], 'gel pores' etc. Despite the hydrated cement and their by-product materials are available everywhere in the concrete, the reactions within the concrete as it sets and strengthens are difficult to control and this is an ongoing problem in the concrete industry. The major issue in the concrete at the fresh and hardened state is the crack and its consequent problems. The cracks in concrete structures and premature erosion are mainly due to alkali silica reaction, which is a chemical reaction that causes fissures in the concrete. Apart from the above, permeability of gases through pores and micro-cracks in the concrete, which leads to corrosion problem in the reinforcement of concrete causes further deterioration. Moreover, the expansion and shrinkage in concrete, which are also cause for crack in concrete at later ages, are mainly due to the sulphate attack, which causes disintegration in concrete, chemical leaching and both the events are mainly due to the excess calcium hydroxide [CH], the by-product during cement hydration as per the following chemical equations.

$$2C_3S + 6H \rightarrow C_3S_2H_3 + 3CH$$

$$2C_2S + 4H \rightarrow C_3S_2H_3 + CH$$

[Cement chemistry notation: C = CaO; S = SiO_2; H = H_2O]. With reference to the above equations, it is learnt that the C-S-H is the strength phase, whereas the by-product, CH is not having any cementitious properties, easily be leached out, prone to chemical attack. With the addition of suitable cementitious materials, mostly siliceous or aluminous, with cement which will react with excess CH and produce additional C-S-H with the replacement of porous CH and refines the pore structure and reduces permeability of gases and water in concrete. The reduction of the CH content during cement hydration associated with the possibilities of sulphate attack and chemical leaching can be reduced further, which will tackle to remediate the concrete cracking to some extent. The researchers worldwide have attempted to tackle the above problem with various methods such as pozzolanic reactions of cement using cementitious materials, by means of chemical reactions of the by-product CH to get additional C-S-H materials or by pore filling mechanism by using cementitious materials. The supplementary cementitious materials such as pulverized fly ash, ground granulated blast furnace slag, condensed micro silica fume, rice husk ash, metakaoline etc have been studied extensively in concrete as pozzolanic materials to enact the CH and get the additional C-S-H. The addition of supplementary cementitious materials in the concrete will not only improve the mechanical properties of concrete, but also its workability, alteration in setting times and durability.

At this juncture, the researchers are capitalizing on nanotechnology to innovate a new generation of concrete materials that overcome the above drawbacks and trying to achieve the sustainable concrete structures. Evolution of materials is need of the day for improved or better performance for special engineering applications and modifying the bulk state of materials in terms of composition or microstructure or nanostructure has been the established route for synthesizing new materials. The newer materials can also be obtained by intelligent and intermixing of existing materials at element level. With the advancement of nano technology, nano materials have been developed that can be applied to concrete mix designs to study the physical, chemical and enhanced mechanical properties of concrete. Among the various developed or manufactured nano materials such as nano silica, nano alumina, nano titania, nano zirconia, nano Fe_2O_3 etc, carbon nano tubes [CNT] or wires etc., the addition of nano silica (NS) enhances the possibility for the reaction with calcium hydroxide (CH) to develop more strength carrying structure of cement: calcium silica hydrate (C-S-H) and also pore filling effect of nano silica in the concrete. Hence, in this paper, a critical review on the influencing factors of nano silica in concrete in detail and the research initiative towards the above task in the future have been provided. Through the use of characterization tools, the ability to gain a better understanding of the materials under study for their size, shape and morphology of crystalline or amorphous nature of those materials have been discussed.

Literature Review

Recently nano technology is being used or considered for use in many applications and it has received increasing attention in building materials, with potential advantages and drawbacks being underlined[1]. At present, a significant number of RandD works dealing with the use of NS in cement based materials are available in the literature. However, there is a limited knowledge about the mechanism by which NS affects the flow properties, setting times, consistency, workability, rheological, micro structural, mechanical properties etc of cementitious mixes. Furthermore, the literature appear to be contradictory about the influence of NS on the development of such materials. A contribution to the development of building materials comprises adding synthetic silica to concrete and cement mortars, whereby the resulting product displays improved aging properties with regard to strength gain, sulphate attack and alkali silica reaction. Due to the high specific surface area for nano material sized colloidal nano silica (CNS) particles, they contribute a highly reactive siliceous material. However, it has not been established whether the more rapid hydration of cement in the presence of NS is due to its chemical reactivity upon dissolution (Pozzolanic activity) or to a considerable surface activity. Bjornstrom et al investigated the hydration process of tricalcium silicate (C_3S) cement and established the accelerating effects of colloidal silica and role of water during hydration[2]. From their study, it was observed that CNS accelerate dissolution of C_3S phase, thereby renders the rapid formation of C-S-H phase. If the nano particles are integrated with cement based materials, the new materials might posses some outstanding properties. The pozzolanic activity of NS is more obvious than that of silica fume counterpart. NS can react with CH crystals, which are arrayed in the interfacial transition zone (ITZ) between hardened cement paste and aggregates and produce C-S-H gel. Thus, the size and amount of CH crystals are significantly decreased and the early age strength of hardened cement paste in increased. Ji studied the water permeability resistant behaviour and micro structure of concrete with NS[3]. The water permeability tests showed that NS concrete has better water resistant permeability than control sample, and microstructure of the NS revealed that the uniform and more compaction of NS in concrete. Similar kind of study was carried out by Ye Qing et al, but with comparison of micro silica [SF] and with influence of NS, they found that the setting times and consistency for SF and NS incorporated concrete were different, but NS makes the cement paste thicker and accelerated cement hydration[4]. Compare to SF concrete, NS showed improved compressive strength and bond strength too. Jo et al studied the properties of cement mortar with NS particles and reported the importance of NS addition towards strength characteristics, hydration progress and calorimetric investigations[5]. Observations were also made from the heat of hydration values, which depicted the amount of CH formed by the addition of NS could increase the amount of heat evolved during setting and hardening of the cement. The reduced calcium leaching behaviour of cement paste by the addition of NS were reported by Gaitero, et al and revealed that the calcium leaching was a degradation process that consisted in the progressive dissolution of the cement paste as a consequence of the migration of Ca^{2+} ions to the aggressive solution[6]. The results obtained showed that NS increases the strength of the cement paste by about 30% in the ore samples and ultimately the observed results highlighted the introduction of NS particles modified the cement paste in three different ways viz. reduced porosity; transforming Portlandite (CH) into C-S-H gel by means of pozzolanic reaction; and modified the internal structure of the C-S-H gel increasing the average chain length of the silicate chains.

According to Sololev et al, the role of nano particles of silica act as fillers in the voids or empty spaces[7]. The well dispersed NS act as a nucleation or crystallization centres of the hydrated products, thereby increasing the hydration rate, that is, NS assisted towards the formation of smaller size CH crystals and homogeneous clusters of C-S-H composition. Moreover, they found that NS improved the structure of the transition zone between aggregates and paste. The drawback in using NS in concrete is self - desiccation due to increased surface area and it will lead to autogenous shrinkage at high concentration and thereby produces cracks in concrete. However, it can be controlled by carefully adding superplasticisers and by appropriate curing methods[2,8].

An indirect method for measuring viscosity change such as rheological study of cement paste and mortars will be helpful to predict the accelerating effect of inclusion of NS[8,9]. From the result it was proved that cement paste and mortar with NS addition requires more water in order to keep the workability of mixtures constant, and it was concluded that NS shows stronger tendency for adsorption of ionic species in aqueous medium and the formation of agglomerates was also as expected. Moreover, it is important to note that it was necessary to use a dispersing additive or plasticizer to reduce the agglomeration effect. Khanzadi et al reported the influence of NS particles on the mechanical properties and durability of concrete through measurement of compressive and tensile strength, water absorption and the depth of chloride penetration[10]. It was observed that the compressive and tensile strength increased in presence of nano SiO_2, which indicates the pozzolanic activity of NS. Improvement in interfacial transition zone was noted and also water absorption, capillary absorption and distribution of chloride ion test results indicate the nano-silica concrete has better permeability resistance than the normal concrete.

With advent of supplementary cementitious materials and other siliceous and aluminous materials, today's concrete technology has achieved enormous potential applications, by the way of reduction in cement consumption, enhanced properties and reduced carbon foot print. In concrete, for example, the micro silica fume works in the form of chemical reaction with calcium hydroxide [CH] form more C-S-H gel at final stage and also fill the voids and pores in the fresh and hardened cement paste, thereby increasing the concrete's density. Some researchers found that the addition of 1 kg of silica fume [SF] permits a reduction of 4 kg of cement, and this can be more if NS is used[4,9,11]. Lin et al. observed the effect of NS addition on permeability and compressive strength of fly ash cement mortar[11]. From the pore analysis study, it was reported that the relative permeability and pore sizes of concrete were decreased, whereas the compressive strength increased by adding more NS. Li demonstrated the effect of addition of NS in high volume fly ash concrete (more of CaO content) and the results compared and reported that the pozzolanic activity of fly ash based concrete with NS were increased considerably and found that decrements in permeability of concrete gained high strength in the early and later stage[12]. Lin et al. described the improvements in the compressive strength on sludge / fly ash mortar in the presence of NS[13]. Yazdi et al. investigated the effect of NS on high volume fly ash concrete [HFC], and reported that due to the low pozzolanic reactions of fly ash, early strength of HFC reduced considerably, but with the addition of NS promoted the pozzolanic activity reaction which enabled the enhancement of strength of HFC, especially in the early ages[14]. The experimental investigations conducted by Sadrmontazi Barzegar concluded that the properties of self compacting concrete with and without Rice Husk Ash [RHA], an agro-industry waste, and exhibited improvement in the physical and mechanical properties of concrete with addition of NS[15].

The splitting tensile strength assessments, thermal behaviour and microstructure of concrete containing different amounts of ground granulated blast furnace slag and SiO_2 nanoparticles as binder were investigated by Nazari and Riahia[16]. The cement replaced with 45% of ground granulated blast furnace slag and up to 3.0 wt% SiO_2 nano particles increased compressive strength due to the presence of increased crystalline $Ca(OH)_2$ by formation of dense C-S-H at early stage and improvement in resistance of water permeability was discussed. It was emphasized that beyond the percentage level of 3% SiO_2 replacement would cause reduction in splitting tensile strength. The nature of pores present inside the concrete was also discussed.

The compressive strength evaluation of cement mortar with nano SiO_2 and with silica fume were discussed for different w/c ratio[17,18]. The experimental results confirm that the compressive strength of mortars with NS were higher than those of mortars containing silica fume at 7 and 28 days. It was proved from this study that the enhancement of strength mainly depends on NS addition rather than addition of silica fume. The hydration progress was continuously monitored from SEM observation, residual quantity test for $Ca(OH)_2$, and rate of heat evolution It was confirmed based on the above conducted tests, the SiO2 in nano scale behave not only as a filler to improve the microstructure, but also as an activator to promote pozzolanic reactions. The influences and importance of super plasticizer while mixing cement with nano particles for mortar or concrete preparation were addressed. The reaction mechanism of NS with cement based on their available high surface area towards the strength improvement by strengthening the C-S-H gel was also discussed.

Min-Hang Zhang et al found that the incorporation of NS by about 2% weight of cement with 50% GGBS cement mixture, not only altered the setting time, but also increased the compressive strength by about 22% and 18% for 3rd and 7th days respectively compared to the reference slag concrete[19]. They also reported that the large capillary porosity decreased and medium porosity increased in the slag cement pastes at 28 days. However, the incorporation of 2% NS by mass of cementitious materials densify the pastes-aggregates interface compared with slag concrete without NS addition. Further reduction of size of NS appeared to be more effective in increasing the rate of cement hydration and reaction compared with SF. In another study, the same authors used NS to reduce setting times and increase early strength of concrete with high volumes of fly ash or slag[20]. Based on the experimental results by using NS in pastes, mortars and concretes with about 50% of fly ash or slag, the incorporation of 2% NS by weight of cementitious materials reduced initial and final setting times and increased 3- and 7- days compressive strengths of high volume fly ash by 30% and 25% respectively in comparison to the reference concrete with 50% fly ash and the similar trends were observed in high volume slag concrete too. It was noted that later strength for NS with fly ash increased, but not in the case of slag cement. Similar action of chloride permeability of the NS with slag and fly ash concrete were reported compared with control concrete.

Another interesting study was reported by Mastafa Jalal et al for the mechanical, rheological, durability and micro structural properties of high performance self compacting concrete [HPSCC] containing silica of micro and nano size and with blended NS and SF[21]. The addition of NS alone up to 2% weight of cement enhanced both the compressive and split strengths by about 62% and 25% respectively, whereas 2% NS blended with 10% SF with control concrete, there was an additional strength improvement of 9% and 8% respectively. They described that the enhancement of strength was not only because of pore filling effect, but also by the accelerated cement hydration due to their higher reactivity of NS. Moreover, the water and capillary absorption results revealed significant decrease by the addition of blended NS and SF for the binder content. According to SEM microstructure studies, more refined microstructure and smaller pores were achieved by the addition of NS and SF, which can led to enhancement of mechanical, durability and micro structural properties of HPSCC.

The effect of colloidal NS [CNS] on the cement hydration process in comparison with SF, as well as its influence on the gel structure and nano scale mechanical properties of cement paste were studied by Hou et al and showed that the pozzolanic activity of colloidal NS (instead of NS powder) was higher than that of SF and its hydration acceleration effect was also higher than SF in the early age, but this effect was comparable to that of SF in the later stage[22]. The strength enhancing effect of CNS and SF mix of about 5% revealed that there was about 16% and 45% improvement in strength for 3rd and 7th day respectively, whereas SF showed less than 10% improvement. At the same time, they found that there were considerable reduction is the CH content in the cement paste with CNS addition, but not the case in SF addition.

The influence of NS with different dosages were studied by Stefanidou, and Papayannis and reported that the addition of NS tends to primarily increase the mechanical response and caused 20 -25% strength improvement [23]. At the same time, with the addition of superplasticizers in 1% w/w of cement reduced the water demand and the strength increase varied from 30% to 35%. Impressive changes were also recorded in the structure of nano-modified samples as the calcium silicate crystal size is larger in samples with high NS content and micro structure observation also recorded a denser structure in nano-modified samples. In a similar line, the effect of NS addition with Portland cement pastes on the workability and compressive strength were studied by Berra, et al[24]. They found that due to the instantaneous interactions between NS and the liquid phase of the cementitious mixes (mainly dissolved alkalis), the formation of gels characterized by high water retention capacities produced a remarkable reduction of the mix workability, without changing water / binder ratio and /or addition of superplasticizers.

Kontoleontos et al studied the influence of CNS on ultrafine cement hydration in terms of physiochemical and micro-structural characterization [25]. CNS behaved not only as a filler to improve cement micro-structure (decrement in porosity), but also a promoter of pozzolanic reaction by transforming CH in to C-S-H gel and reported that the optimized value of NS was 4%, which presented the higher compressive strength of 64% over control mix. They found that the enhancement of the compressive strength at later ages was due to the consumption of CH by NS. At the same time, the setting time and workability decreased, because of rapid agglomeration of the slurry from the suspending state and also NS improved the packing of particles, which contributes to the growth of paste viscosity. Also NS was proved as an agent that improved the micro-structure of the ultrafine cement pastes and showed the cement with NS presented a denser microstructure. The pore size refinement at later ages, as a result of pozzolanic reaction, also led to a significant enhancement of the compressive strength.

Research Initiative

Taking advantage of nanostructure characterization tools and materials, the optimal use of nano silica will create a new concrete mixture that will result in long lasting concrete structures in the future.

Generally, concrete is two phase system with cement paste and aggregate, but the aggregates are inert in nature. The hydrated cement paste is composed of capillary pores and the hydration product - 'gel' pores, C-S-H, CH, Aft [Ettringite], AFm [Monosulfates] etc and one third of the pore space is comprised of gel pores and the rest are capillary pores. There are various indications that confirm the layered nature of C-S-H. Study conducted by Feldman and Sereda indicated that the cement paste inflow increases as water is removed until a point, at which the flow decreases[26]. This point is the indication of a possible collapse in the nano-structure of hydration products and the C-S-H that is produced during the hydration of Portland cement has the microstructure as shown in figure 1.

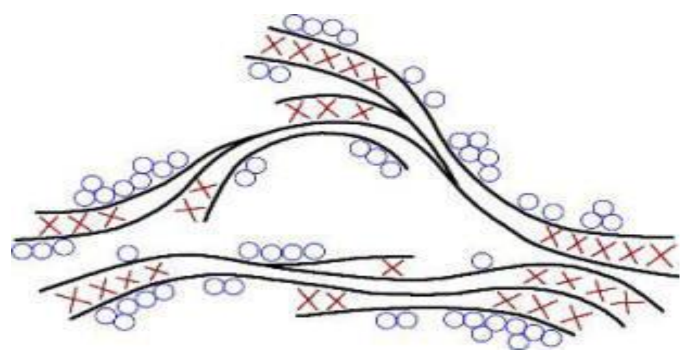

Figure-1
Feldman-Sereda model for the microstructure of C-S-H Black lines: C-S-H sheet, Circles: Adsorbed water, Crosses: Interlayer water

Table-1
Ranges of pore sizes in concrete[26]

Item	Size
Interparticle spacing between CSH sheets	1-4 nm
Capillary voids	10-1000 nm (0.01-µm)
Hexagonal crystals of $Ca(OH)_2$	0.7 - 3µm
Aggregation of CSH particles	0.7 - 3µm
Entrained iar bubbles	60-1000 µm (0.06-1 mm)
Entrapped air voids	1-5 mm

Table-1 depicts the relative sizes of pores in concrete. At one end of the scale are entrapped air voids, while on the lower extreme are the inter-particle spaces between sheets of C-S-H.

According to the above model of C-S-H, the cement paste is mostly having the gel pores, capillary pores, interlayer water etc. At the same time, in the concrete, there is an ITZ between the cement paste and aggregates, which is a weak link in the concrete usually the site of first occurrence of cracking. Therefore, avenues are available for further research towards the generation of crack free concrete with the possible incorporation of NS towards the promotion of cement hydration with high reactivity in the locations discussed earlier, with enhancement of the delay in development of micro crack, pore filing effect, creation of strong bond between the aggregates and cement paste and / or C-S-H. Towards these, the challenges ahead in the future are as follows: i. Characterize different grade of cement and thorough analysis such as qualitative as well as quantitative by using sophisticated software. Since the cement is one of the major role players, these analyses are required to understand the behavior with foreign moieties. ii. A detailed investigation on the clear C-S-H phases of cement without any modification and modified C-S-H phases with guest molecules, for example with nano SiO_2. iii. Studies on physical, chemical and mechanical properties of nano modified cement at different length scales towards the achievement of crack free concrete. This is one of the major tasks in our research initiative and major challenges for researchers, who have involved in this area.

The important phenomena such as crystallite size, Full Width at Half Maximum [FWHM] and arrangement of atoms or molecules need to be studied for nano modified composites as well as for cement to understand the reactions and responses. Towards this, some fundamental research work has been initiated to achieve the main objective as stated explicitly above.

Initially, we have characterized the commercially received nano silica by using Transmission Electron microscopy [TEM] and XRD to find the phases of the NS. Fig. 2 and 3 show the TEM image and XRD pattern of NS respectively, which indicate the spherical shape of NS and amorphous nature of NS particles.

XRD analysis of the ordinary Portland cement has been carried out as shown in Figure-3 and the search match was conducted by using International Crystal Diffraction Database (ICDD) PDF 4+ for phase analysis are provided in the Table 2 and the post peak analysis was conducted to find out the crystallites size and FWHM of the major peaks is provided in the Fig. 4 and Table 3, which are responsible for the development of strength as well as durability of the concrete. This is an important step to go further on the research on nano materials, particularly for nano silica.

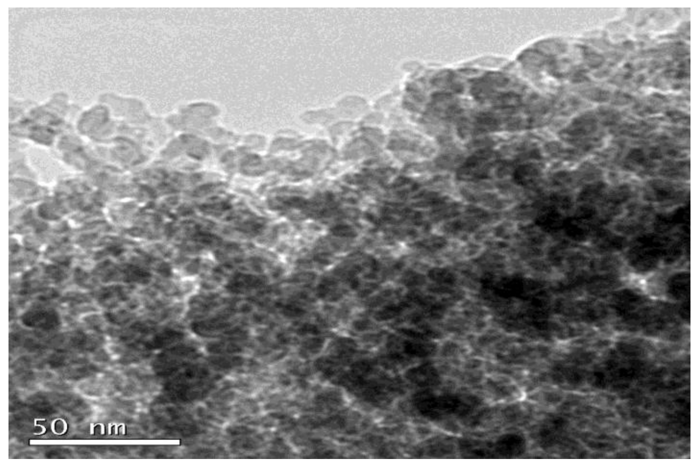

Figure- 2
TEM image of nano SiO_2

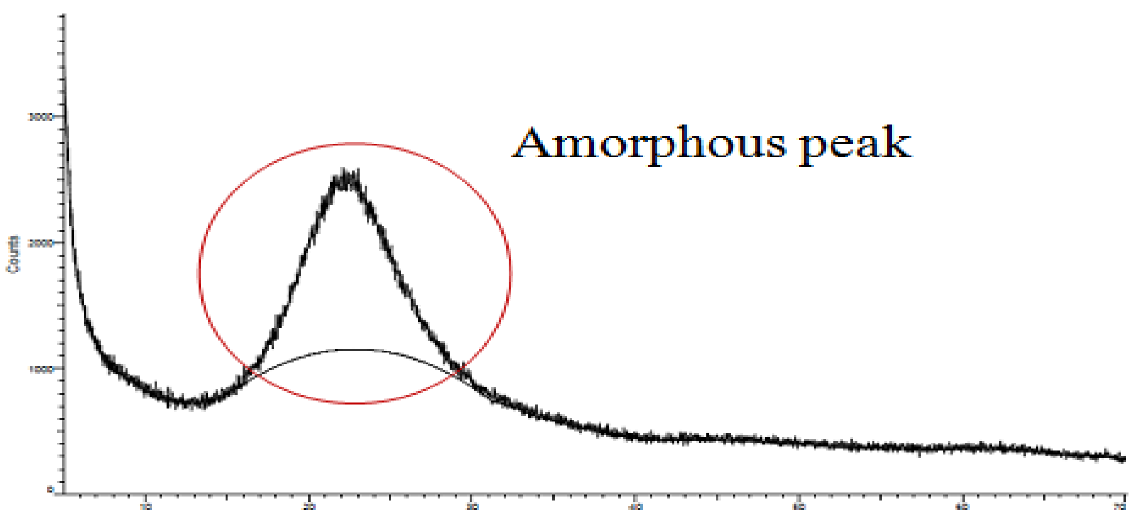

Figure-3
X-Ray Diffraction of nano SiO_2

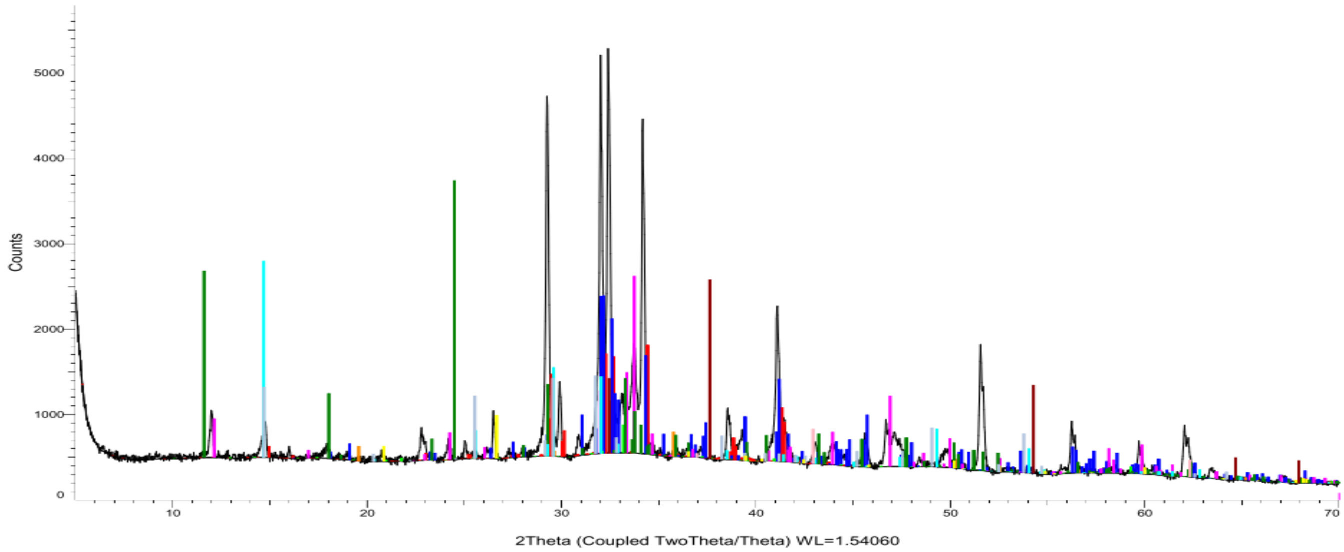

Figure-3
X-Ray Diffraction of Ordinary Portland Cement [OPC]

Table-2
Phase analysis of OPC

Index	Color	Formula	Pattern #	Compound Name
1		Ca3 (Si O4) O	PDF 01-070-8632	Hatrurite, syn
2		Ca2 Si O4	PDF 01-077-0409	Larnite
3		Ca3 Al2 O6	PDF 00-038-1429	Calcium Aluminum Oxide
4		Ca2 Fe Al O5	PDF 04-007-5261	Brownmillerite, syn
5		Ca O	PDF 01-074-1226	Lime, syn
6		Ca (O H)2	PDF 01-070-6447	Calcium Hydroxide
7		Ca (S O4) (H2 O)2	PDF 01-076-8129	Gypsum, syn; Gypsum
8		Ca (S O4)	PDF 01-076-6906	Anhydrite, syn
9		Ca S O4 ·0.5 H2 O	PDF 00-045-0848	Calcium Sulfate Hydrate
10		Si O2	PDF 00-005-0490	Quartz, low
11		Mg O	PDF 01-076-2583	Periclase, syn

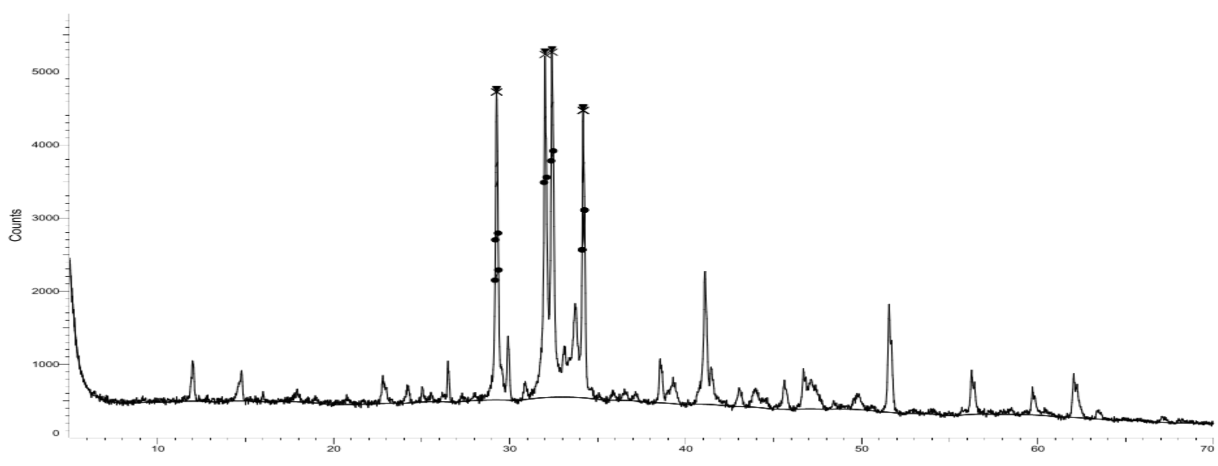

Figure-4
X-Ray Diffraction for post peak analysis of OPC

Table-3
Post peak analysis of OPC with crystallite size and FWHM

Left Int.	Right Int.	Obs. Max	d (Obs. Max)	C. Size	FWHM
0	0	0.000		0.0	0.000
16.4	16.9	29.258	3.04997	878.4	0.104
21.1	21.5	32.006	2.79408	1373.8	0.067
22.9	23.7	32.407	2.76045	1443.5	0.064
15.5	18.8	34.173	2.62170	1287.7	0.072
15.5	18.8	34.173	2.62170	1287.7	0.072
15.5	18.8	34.173	2.62170	1287.7	0.072
13.0	13.9	29.258	3.04997	765.8	0.119

Conclusion

The above discussions described that the influence of NS along with cement, cement mortars, concretes, supplementary cementitious materials and other cementitious materials. Considerable improvement in the properties of permeability, pore filling effects, reduction of CH leaching, rheological behaviour of cement pastes, heat of hydration, micro structure analysis, the pozzolanic activity or reactions and workability, strength and durability were reported. As a whole, the entire review showed the ultimatum in using the nano technology in general and nano silica in particular. All the properties studied by various researchers have not directly dealt with remediation of concrete cracking whereas the entire attempts were made indirectly towards that. However, there is a gap or room available for further research towards the fruitful application of nano silica for construction with different nano structure characterization tools, which will be enable to understand many mysteries of concrete.

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