We are IntechOpen, the world's leading publisher of Open Access books Built by scientists, for scientists

6,300

Open access books available

172,000

International authors and editors

190M

Downloads

Our authors are among the

154

Countries delivered to

TOP 1%

most cited scientists

12.2%

Contributors from top 500 universities

WEB OF SCIENCE™

Selection of our books indexed in the Book Citation Index
in Web of Science™ Core Collection (BKCI)

Interested in publishing with us?
Contact book.department@intechopen.com

Numbers displayed above are based on latest data collected.
For more information visit www.intechopen.com

Compressive Strength of Concrete with Nano Cement

Jemimah Carmichael Milton and Prince Arulraj Gnanaraj

Abstract

Nano technology plays a very vital role in all the areas of research. The incorporation of nano materials in concrete offers many advantages and improves the workability, the strength and durability properties of concrete. In this study an attempt has been made to carry out an experimental investigation on concrete in which cement was replaced with nano sized cement. Ordinary Portland cement of 53 grade was ground in a ball grinding mill to produce nano cement. The characterization of nano cement was studied using Scanning Electron Microscope (SEM), Brunauer Emmett–Teller (BET), Energy Dispersive X ray microanalysis (EDAX) and Fourier Transform Infrared Spectroscopy (FTIR). From the characterization studies, it was confirmed that particles were converted to nano size, the specific surface area increased and the chemical composition remained almost the same. The properties of cement paste with and without nano cement were found. For the experimental study, cement was replaced with 10%, 20%, 30%, 40% and 50% of nano cement. Cement mortar of ratio 1:3 and concrete of grades M20, M30, M40 and M50 were used. Compressive strength of cement mortar and concrete with different percentages of nano cement was found. The cement mortar was also subjected to micro structural study. It was found that the strength increased even up to the replacement level of 50%. Further increase in the replacement is not possible since the addition of nano cement reduces the initial and final setting time of cement paste. At 50% replacement level, the initial setting time got reduced to 30 minutes which the least permitted value as per IS 12269: 2013. The increase in strength was due to the fact that nano cement acts not only as a filler material but also the reactivity increased due to the higher specific surface area. The SEM image shows the formation of additional C-S-H gel. The percentage increase in compressive strength was found to increase up to 32%. The workability of concrete with nano cement was found to be significantly more than that of the normal cement concrete.

Keywords: compressive strength, nano cement, normal cement concrete, scanning electron microscope (SEM), energy dispersive X ray microanalysis (EDAX), Fourier transform infrared spectroscopy (FTIR)

1. Introduction

Nano technology is a new emerging area in field of engineering. Development of nanotechnology in the field of material science and evolution of advanced instrumentation have paved way for application of nanotechnology in the construction field. Incorporation of nano sized particles in cement composites makes a significant

change in structural and nonstructural properties of cement paste, mortar and concrete. The particles converted from micron size to nano size results in more specific surface area. The increase in surface area leads to changes in morphology, increase in the chemical reactivity, structural modification of cement hydrates and enhancement of the properties of concrete. Nano particles are produced by two approaches. In “top down” approach, larger particles are reduced to smaller particles without altering the original properties and in “bottom down” approach very small nanoscopic molecules and atoms combine together to form bigger structures wherein the particles properties can be altered. The nano scale particles can result in vividly improved properties from conventional grain size materials of the same composition. Nano materials show unique physical and chemical properties that can lead to the development of more effective materials than the ones which are currently available. The use of nano materials in concrete, results in stronger and more durable concrete with desired stress-strain behaviour.

The structure of nano materials can be studied using the various sophisticated non-destructive techniques. The scaled down particles are to be checked for their size and some of the equipment available to determine particle size are scanning electron microscope, atomic force microscope and transmission electron microscope. Many techniques like Energy dispersive X-ray analysis, X-ray diffraction, X-ray absorption spectroscopy, Fourier transform infrared spectroscopy, Nuclear magnetic resonance *spectroscopy*, Thermal gravimetric analysis, Low-energy ion scattering spectroscopy, UV-V's spectroscopy, Photoluminescence spectroscopy, Dynamic light scattering are available for the surface chemical analysis and characterization of nano materials.

Nowadays application of nanotechnology can be widely seen in medical, car manufacturing, pharmaceutical, chemical and other industries. Nano particles are used for the manufacturing of medicines, bio medical instruments, paints, coatings, glass, plastics and rubber. In the construction field, nano titanium oxide, nano silica, nano aluminum oxide, nano zirconium oxide, carbon nano tubes, carbon nano fibers and nano fly ash are commonly used nano materials. According to Konstantin Sobolev [1], nano particles improve the ductility, thermal resistance and hence can be used in refractory concrete. Hanus [2] reported that nano particles produce anti-microbial surfaces and can be adopted in hospital buildings. It was also suggested that nano particles can be used in pavement as it possess high thermal resistance and abrasion property. According to Gann [3], shells and bones contain crystals of calcium and can be used in nano form to arrest crack and to dissipate energy.

Nano particles are added to concrete to improve its material properties. Perumalsamy Balaguru and Ken Chong [4] expressed that particle size upto 500 nm can be used in concrete whereas Surinder Mann [5], Florence Sanchez and Konstanin Sobolev [6] Bhuvaneshwari et al., [7], reported that the size of nano particles used in concrete has to be restricted to 200 nm. Hui li et al., [8], Maile Aiu and Huang [9] studied the effect of nano materials on the compressive strength of cement mortar. Tao Ji [10] and Ali Nazari and Shadi Riahi [11] studied the permeability and microstructure of concrete containing nano particles. Thomas.K.paul et al., [12] reported that nano materials can be produced using different techniques and gave an outline of producing nano fly ash. Research proves that there will be considerable changes in the chemical reactivity and mechanical properties, when the particles are converted to nano size. When the size is reduced, more atoms will be found at the surface of particle which significantly imparts a change in the morphology and energy at the surface. The changes in the chemical reactivity will improve the catalytic ability in paints and pigments which impart self-cleaning and self-healing properties. Nano titanium oxide is used as a self-cleaning material in

glass and as anti-corrosive element in steel. Carbon nano tubes (both single walled and multi walled) and carbon nano fibers are used in concrete to prevent cracks and to improve ductility. Nano ferric oxide due to its super para magnetic property can improve the strength property of concrete. Nano aluminum oxide is used to resist abrasion of concrete in pavements. Nano fly ash acts as a promoter to enhance the pozzolanic property. Nano silica with different quality and properties are used as an additive material in concrete. It can be seen from literature that addition of nano particles significantly improves the strength and durability of concrete. In common, nano particles are used to enhance mechanical and durability properties of concrete and to develop sustainable concrete materials and structures.

2. Review of literature

The review of literature on the behaviour of mortar and concrete with nano materials is reported to understand the behaviour of cement mortar and concrete with nano particles, According to Hanus and Andrew T. Harris [13], Silvestre et al. [14] and Elzbieta Horszczaruk [15], nano silica, nano titanium oxide, nano zirconium oxide, carbon nano tubes and carbon nano fibers are commonly used materials for making nano concrete. These nano materials are not cost effective and also not available in abundance compared to the supplementary cementitious materials. Balaguru Perumalsamy and Chong Ken [4] reported that nano silica in colloidal state was more efficient than the micron sized silica in improving the durability of concrete. It was also reported that nano cement and nano carbon tubes can enhance the properties of concrete compared to nano carbon fibers. Konstantin Sobolev and Miguel Ferrada Gutierrez [16], Surinder Mann [5], Zhi Ge and Zhili Gao [17] reported that introduction of nanotechnology may bring major changes in the field of concrete technology. The concept of nanotechnology can be applied to concrete, steel and glass to produce new products with new properties. It was highlighted that the structure and behaviour of concrete should be understood at micro and nano scale. Bhuvaneshwari et al. [7] reported that efforts were taken to evolve new nano material towards a green and sustainable solution in the area of cement based materials and their composites for the construction applications. It was reported that the study of cement based material at nano level will result in a new generation construction materials with enhanced strength and durability properties. Sanchez Florence and Sobolev Konstantin [6] reported that the measurement and characterization of nano structure of cement and concrete materials is nano science and use of nano materials in cement and concrete composite is nano engineering. The nano structure study of nano materials was done by atomic force microscope, nano indentation technique, nuclear techniques, neutron and X ray scattering technique. Experimental study and micro structural study on nano materials is essential to study the effect of nano particles in concrete. They reported that mechanical properties of concrete can be enhanced with incorporation of nano particles in concrete. Laila Raki et al. [18] reported that nano-sized particles modify and improve the durability of concrete.

Jafarbeglou et al. [19] reviewed the current state of nano technology in enhancing the performance of concrete by producing new sustainable advanced cement based composites. The advance instruments like Atomic Force Microscope (AFM), Transmission Electron Microscope (TEM) were used to understand the role of nano particles and to predict the life of concrete with nano materials. It was discussed that the uniform proper dispersion and compatibility of nano materials should be taken care while incorporating the nano sized materials in concrete. Guillermo Bastos et al. [20] stated that it is necessary to have a unique synthesis

method to produce nano materials in a large scale Standards should be adopted to mix efficiently nano particles in cement composites. High quality standards in production, common codes and identical terminology are needed to transfer the knowledge of research findings to global market. Implementation of concept of nano technology in construction field is difficult due to the cost involved in synthesis and dispersion of nano materials. Muhd Norhasri Muhd Sidek et al. [21] reported that particle size less than 500 nm can be used in concrete which enhances the properties of concrete. It was also stated that ultrafine particles less than 200 nm helps to reduce cement content and helps in the reduction of micro pores by acting as a filler agent and increase the density of concrete. Hosam M. Saleh et al. [22] discussed the formation of stabilized radioactive waste immobilization and construction materials from hazardous cement kiln dust. Portland cement with slag, silica fume, kiln dust along with 0.1% of nano materials were mixed and solidified. Compressive strength and porosity of the cement composite were found. It was reported that the performance with 0.1% nano silica increased the mechanical integrity by four fold. Hosam M. Saleh et al. [23] explored the possibility of improving the properties of cement by adding iron slag and titanate nano fibers to stabilize the radio active waste. The mechanical and physical characterization of the cement was enhanced. It was reported that it captured radionuclides from the contaminated aqueous solution before the immobilization process. Hosam M. Saleh et al. [24] studied the performance of cement-slag-titanate nano fibers composite immobilized radioactive waste solution. It was observed that cement nano composite was created by mixing iron slag with Portland cement which was hydrated with aqueous titanate nano fibers. SEM, FT-IR and X-ray diffraction analysis was performed to confirm the calcium silicate hydrate formation. It was observed that nano composite enhanced the mechanical and durability properties of cement and cement based materials. The chemical stability of cement-slag-titanate nano fibers composite was studied by monitoring leaching of ^{137}Cs which confirms immobilization radioactive waste and other hazardous waste. Hosam M. Saleh et al. [25] studied the influence of severe climatic variability on the structural, mechanical and chemical stability of cement kiln dust slag nano silica composite. The dust from cement kiln dust was mixed with 20% of iron slag and 0.1% nano silica, to produce modified cementitious composites which are suitable for construction and waste stabilization applications. The leachability studies showed that the impact of flooding can be reduced. Further freezing and thawing studies showed that the immobilization of radioactive waste can be enhanced.

Maile Aiu and Huang [9] synthesized the components of portland cement type I nano particles using sol gel process and compared the properties with that of commercial cement. Scanning Electron Microscope study revealed that nano cement particles were of size between 40 nm to 100 nm and appeared to be conglomerated and spherical. Energy Dispersive X-Ray Analysis test showed that the calcium to silica ratio was 3:1 or 2:1 and X-ray powder diffraction (XRD) result showed that nano cement contains C_3S , C_2S and copper oxide. Thomas Paul et al. [12] explained about the preparation, characterization of nano structured fly ash. The class F fly ash was ground in a high energy ball milling and converted into nano structured material. The nano structured fly ash was characterized for its particle size using particle size analyzer. Specific surface area was found using Brunauer-Emmet-Teller (BET) surface area apparatus. Fourier Transform Infrared Raman analysis (FTIR), SEM and TEM were used to study the particle aggregation and shape of the particles. On ball milling, the particle size got reduced from 60 μm to 148 nm i.e., by 405 times and the surface area increased from 0.249 m^2/g to 25.53 m^2/g i.e. by more than 100%. Surface of the nano structured fly ash was

found to be more active as was evident from the FTIR studies. Morphological studies revealed that the surface of the nano structured fly ash was more uneven and rough and shape is irregular as compared to fresh fly ashes which are mostly spherical in shape. Narasimha Murthy Inampudi et al. [26] studied the crystallite size and lattice strain using XRD when micro sized fly ash was converted to nano sized fly ash using high energy ball milling process. The characterization was done after every 5 hours and at the end of 30 hours, the size of nano fly ash was found to be 83 nm. It was observed that the crystallite size was decreased and lattice strain was increased. It was also observed that the spherical shaped fly ash was converted to irregular shaped nano fly ash particle. The nano fly ash particles increased the hardness of composite and improved the compressive strength of cement composite. Gujjala Raghavendra et al. [27] presented the method of converting uneven micro size fly ash in to smooth glassy nano sized fly ash by planetary ball milling. From BET and XRD studies, it was noted that after 16 hours of milling, the surface area increased from $0.31\text{m}^2/\text{g}$ to $24.65\text{m}^2/\text{g}$, the crystalline structure reduced from 59–26% and the particle size converted from $11\text{ }\mu\text{m}$ to 148 nm . Sada Abdal khaliq Hasan Alyasri et al. [28] investigated the economic feasibility of producing nano cement in a large scale through the cement factories. The mineral admixtures such as fly ash and slag should be added with crushed clinker where the moisture content should be maintained below 3%. The mixture was ground for 30 to 40 min to get nano cement. The specific gravity of nano cement was found to be 2.11 and specific surface area was $3,582,400\text{cm}^2/\text{g}$. Bickbau and Shykun [29] explained that the Russian federal agency on technical regulating and metrology formulated the national standard on nano modified portland cement. Nano cement was produced by grinding process in ball mills of the clinker. Silicate minerals and gypsum were also added. The properties of nano cement were checked for its consistency. The specific surface area found by Blaine's apparatus should be below $400\text{m}^2/\text{kg}$ and grain size should be 10-100 nm. The mineral supplements were added in clinker to get economical nano cement, to reduce cost of fuel, reduce CO_2 emissions and to improve the quality of concrete with nano cement.

Parang Sabdono et al. [30] studied the effect of nano cement on the compressive strength of cement mortar. It was proved that use of nano cement in mortar helps in the reduction of the micro voids present in cement mortar, enhances the pozzolanic activity and increase the hydration rate of cement. Ordinary Portland Cement (OPC) and Portland Pozzolana Cement (PPC) were converted to nano particles of size 50 nm and 47 nm size. The experimental result showed that the initial setting time reduced from 138 min to 75 min with nano OPC and 123 min to 45 min with nano PPC. The compression test was conducted on 28 days cement mortar specimens of size 50 mm x 50 mm x 50mm. The compressive strengths of mortar with nano OPC and nano PPC were found to be 68.493 N/mm^2 and 65.286 N/mm^2 respectively. It was reported that the nano cement improved the hydration reaction, lowered the initial setting time and increased the compressive strength. Ikhlef Bualem [31] studied the properties of cement and cement mortar with two combination of nano particles. First combination used was grinding 100% OPC to nano size without mineral additives and the second combination was grinding 50% of OPC together with 50% of silica sand (it is equal parts of granulated blast furnace slag and quartz sand) to nano size. It was found that the specific surface area of nano cement without mineral additive was $4900\text{ cm}^2/\text{g}$ and nano cement with 50% of mineral additive, the specific surface area was found to be $5000\text{ cm}^2/\text{g}$. It was also found that the 90th day compressive strength of nano cement mortar was 51.4 MPa. for cement mortar with 100% nano cement, the strength was 114.3 MPa and for mortar with nano cement and mineral additive the strength was 77.8 MPa.

Gengying Li [32] compared the properties of normal cement concrete, high-volume fly ash high-strength concrete (SHFAC) incorporating nano silica and high-volume fly ash high-strength concrete (HFAC). Compressive strength was found from 3 days to 2 years. It was found that HFAC showed a 10% less compressive strength upto 56 days compared to normal cement concrete but increased to 21% higher than that of normal cement concrete at 2 years. The compressive strength of SHFAC showed that addition of 4% nano silica helped to gain early age strength by about 81% and also helped in gaining later strength upto 47% compared with normal cement concrete. The pore size of SHFAC was found to be less than that of NCC and HFAC. Zaki and Khaled Ragab [33] studied the influence of nano silica on the properties of fresh and hardened normal cement concrete. In this study 18% of silica fume and 0.5%, 0.7% and 1% of nano silica were used. It was found that the workability of concrete improved with addition of super plasticizers when nano silica was added. It was reported that concrete with nano silica had a higher compressive strength, since nano silica not only acts as filler but helps in rapid formation of C-S-H gel. It was also reported that the efficiency of nano silica depends on its morphology, size and method of production. It was stated that 0.5% nano silica was found to perform better with and without silica fume compared to normal cement concrete. Nili et al. [34] discussed the performance of concrete with using nano silica and micro silica. It was reported that due to the high pozzolanic property, the compressive strength of concrete with 1.5, 3, and 4.5% of nano silica and 3, 4.5, 6 and 7.5% of micro silica gave a higher value than the normal cement concrete. It was found that the compressive strength of concrete with 1.5% nano silica and 6% of micro silica gave optimum values. Praveen and Janagan [35] experimentally studied the strength of concrete with nano particles. In this study, cement was replaced with 30% nano fly ash along with 3% nano Ground Granulated Blast Furnace slag (GGBS), 40% nano fly ash along with 4% nano GGBS and 50% nano fly ash along with 5% nano GGBS. The compressive strength and tensile strength of concrete were investigated on 7th and 28th day. It was reported that the strength was more when cement was replaced with 30% of nano fly ash along with 3% nano GGBS. Harihanandh and Sivaraja [36] experimentally studied the compressive strength, tensile strength and flexural strength of M20 grade concrete with nano fly ash. In this study class F calcium fly ash was converted into nano size in a ball grinding mill and its size was confirmed by SEM analysis. It was found that concrete with cement replaced with 23% nano fly ash gave 34% more compressive strength, 58.57% more tensile strength and 33.07% more flexural strength than the normal cement concrete. It was reported that the nano fly ash filled the pores and made the concrete denser.

It is obvious from the review of literature, that the application of nanotechnology in concrete is one of the promising areas of research. Studies were done on cement paste, cement mortar and cement concrete with incorporation of small percentage of nano materials. Nano particles of silicon dioxide, cement, fly ash, clay, metakaolin, iron oxide, aluminum oxide, zirconium oxide, carbon nano tubes, carbon nano fibers and nano carbonates are considered by many researchers in which nano silica was commonly used. Syntheses of nano materials were done by various methods and the properties of the nano materials were found to be dependent on the method of production. Characterization is done to understand the property of nano materials. Since the particle is of nano size with high specific surface area, the behaviour of cement paste, mortar and concrete with nano materials may be different from that of the normal cement paste, mortar and concrete. Normal consistency and setting time tests are usually carried out with cement paste, strength and durability studies are done with mortar and concrete. Few studies were also carried out with the addition of admixtures.

The special focus of the present investigation intends to highlight the replacement of cement with nano cement in larger percentages. Though many methods of production of nano materials are given in literature, ball milling method is adopted by which a large amount of nano materials can be produced economically without chemical change. Hence an attempt has been made to study the properties of nano cement, properties of cement paste with nano cement, properties of cement mortar with nano cement and compressive strength of concrete with nano cement.

3. Properties of materials

Concrete is considered to be a composite material containing a binder medium within which aggregate particles are embedded. The properties of materials used in the experimental investigation are cement, fine aggregate, coarse aggregate, and water. The properties are presented in this session.

3.1 Cement

Ordinary Portland Cement (OPC) of 53 grade conforming to IS: 12269-2013 [37] and procured from a single source was used for this investigation. The chemical and physical properties of the cement used are given in **Table 1**.

Particulars	Results (%)	Requirements of IS:12269
SiO ₂	21.8	—
Al ₂ O ₃	4.8	—
Fe ₂ O ₃	3.8	—
CaO	63.3	—
SO ₃	2.2	—
MgO	0.9	Maximum6
Na ₂ O	0.21	—
K ₂ O	0.46	—
Cl	0.04	Maximum0.1
P ₂ O ₅	<0.04	—
Loss of ignition	2.0	Maximum4
Insoluble residue	0.4	Maximum3
Specific surface area, m ² /kg	370	Minimum225
Initial setting time, minutes	50	Minimum30
Final setting time, minutes	510	Maximum600
Standard consistency, %	34	—
Soundness, Le-chatelier, mm	1.0	Maximum10
Compressive strength, MPa		
3-days	42.5	Minimum27
7-days	48.0	Minimum37
28-days	63.5	Minimum53
Specific gravity	3.15	—

Table 1.
Chemical and physical properties of 53 grade OPC.

3.2 Fine aggregate

The locally available clean and dry natural sand from Cauvery river basin, Karur, India free from debris was used as fine aggregate. The specific gravity of fine aggregate was found to be 2.65. From sieve analysis, it was confirmed that the sand belongs to Zone II grading. Bulk density of fine aggregate was found to be 1520 kg/m³. Fineness modulus of sand was found to be 2.32. The properties of fine aggregate were found to confirm with IS: 383-2016 [38].

3.3 Coarse aggregate

The coarse aggregate used was natural hard broken granite stones. Crushed granite metals of size 20 mm were used. The specific gravity of coarse aggregate was 2.79 and it was confirming to IS: 383-2016 [38].

3.4 Water

Potable water available in laboratory was used for casting and curing all specimens in this investigation. The quality of water was found to satisfy the requirements of IS: 456-2000 [39].

4. Synthesis and characterization of nano cement

4.1 Production of nano cement

Nano cement was produced by grinding ordinary Portland cement of 53grade in a high intensity ball grinder for 12 hours. In high energy ball grinding milling machine, high impact collisions were used to reduce microcrystalline materials down to nano crystalline structure without chemical change. Care was taken to avoid balling effect and agglomeration.

4.2 Microstructure analysis of nano cement

The particles size of the nano cement were found by surface morphology studies, the specific surface area was found by Brunauer Emmett Teller theory, and the elemental compositions were found by X-ray spectroscopic method.

4.2.1 Particle size determination of nano particles

Surface morphology studies were carried out using a Scanning Electron Microscope (JEOL, JSM 35 CF, Japan) shown in **Figure 1**.

53grade OPC was ground in the ball grinder mill for 12 hours to produce nano cement which was taken for morphological study using SEM. The SEM images of cement and nano cement are shown in **Figures 2 and 3** respectively.

From the SEM image, it can be seen that the cement particles have been ground to nano size. The size varies between 45 nm to 86 nm. It was also found that the shape of the particles was not altered due to grinding and agglomeration of cement particles did not take place.

4.2.2 Specific surface area of nano materials

The specific surface area is the total surface area of the exposed surface in square centimeter per unit mass. It was found by Brunauer, Emmett and Teller

Figure 1.
Scanning electron microscope.

Figure 2.
SEM image of cement.

Figure 3.
SEM image of nano cement.

method for cement and nano cement. The specific surface area of cement was found to be $3700 \text{ cm}^2/\text{g}$ and for nano cement, the specific surface area was found to be $485,000 \text{ cm}^2/\text{g}$. It can be seen that the particles in nano size have much higher specific surface area compared to particles in micro size.

4.2.3 Energy dispersive X-ray analysis of materials

EDAX is an x-ray spectroscopic method for determining elemental compositions. EDAX studies were carried out using a Scanning Electron Microscope. EDAX analysis

Figure 4.
EDAX of cement.

Figure 5.
EDAX of nano cement.

Element	Atomic weight percentage of chemical elements (%)						
	C	O	Al	Si	K	Ca	Fe
Cement	21.55	58.27	6.84	12.17	0.20	0.28	0.69
Nano Cement	19.80	59.95	8.00	11.07	0.24	0.19	0.75

Table 2.
EDAX analysis of particles.

was done in conjunction with SEM analysis. In EDAX analysis, X rays are emitted from the sample due to bombardment of electron beam from a spot, an area, a line profile or a 2D map. These X-rays are detected to characterize the elemental composition. In the EDAX images, the X axis represents energy and Y axis represents intensity. EDAX of cement and nano cement are shown in **Figures 4** and **5** respectively.

The details of EDAX analysis are given in **Table 2**.

From the EDAX study it was noted that the chemical composition of elements does not vary much when ground to nano particles.

5. Tests on cement paste with nano cement: normal consistency and setting time

The consistency test and setting time test on cement mortar with nano cement were carried out using the Vicat’s apparatus conforming to IS: 5513-1996 [40].

Particulars	% Replacement of Cement with Nano Cement					
	0	10	20	30	40	50
Normal Consistency	34	33	33	34	35	33
Initial setting time	50	45	40	38	35	30

Table 3.
Normal consistency and setting time of cement paste with nano cement.

The values of consistency, initial setting time and final setting time of the cement paste with 0%, 10%, 20%, 30%, 40% and 50% of nano cement are given in **Table 3**.

From **Table 3**, it can be seen that the consistency of cement pastes with nano cement was almost the same but the initial and final setting times of are found to decrease as the replacement percentage increases.

The initial setting time of cement paste with nano cement was found to decrease to 30 minutes with 50% replacement whereas the initial setting time of normal cement paste is found to be 50 minutes. As per the IS 12269-2013 [37], the initial setting time of cement should not be less than 30mins. Hence the replacement level of cement with nano cement should not exceed 50%. The final setting time of cement paste with nano cement was found to decrease to 245 minutes at a replacement level of 50%.

6. Tests on cement mortar with nano materials

The compressive strength of cement mortar was determined on the 3rd, 7th, 21st and 28th days. After the 28th day test, the powder of the tested cement mortar cubes was taken for micro structural studies. The properties were evaluated by SEM, EDAX and FTIR test.

6.1 Compressive strength of cement mortar by experiment

The compressive strength of hardened mortar cubes of size 70.6 mm x 70.6 mm x 70.6 mm with and without nano cement were found using a compression testing machine of capacity 2000kN. The load was applied with a uniform rate of 35 N/mm²/min after the specimen had been centered in the testing machine. The compressive strengths of cement mortar cubes are shown in **Table 4**.

The compressive strength of cement mortar was found to increase upto 50% replacement of cement with nano cement. The percentage increase in strength was found to vary between 34.36 and 77.85 for nano cement.

% replacement of cement with nano cement	Average Compressive Strength of Cement mortar (N/mm ²)			
	3 rd day	7 th day	21 st day	28 th day
0	42.50	48.00	57.00	63.50
10	58.98	74.48	83.23	85.32
20	62.98	79.50	88.42	90.66
30	65.50	81.20	90.96	93.24
40	67.88	85.37	92.44	95.68
50	70.00	75.53	94.00	98.00

Table 4.
Compressive strength of cement mortar with nano cement.

6.2 Microstructure of cement mortar with nano cement

Scanning Electron Microscope image of the crushed cement mortar particles cured for 28th days is shown in **Figure 6**.

It can be seen from **Figure 6** that the textures of particles consists of standalone clusters which indicates less formation of C-S-H gel. Hence the strength will be less than the mortar containing nano cement.

SEM images of cement mortar cubes in which cement was replaced with 10%, 20%, 30%, 40% and 50% of nano cement are shown in **Figure 7**.

From **Figure 7**, it can be seen that with 10% nano cement, the texture of hydrate particles are standalone clusters and with 50% nano cement, the texture of particles are colloidal due to the formation of C-S-H gel and hence the strength of mortar cubes with 50% nano cement is higher.

6.3 Energy dispersive X-ray spectroscopy study on cement mortar with nano materials

The EDAX study was carried out using the same sample used for SEM study. The EDAX of crushed cement mortar is shown in **Figure 8**.

The EDAX images of cement mortar in which cement was replaced with NC are shown in **Figure 9**.

From the EDAX analysis, the chemical elements present in the mortar with nano cement were found and the details are given in **Table 5**.

From the chemical composition, Ca/Si ratio was found. The strength of the mortar depends on Ca/Si ratio. Wolfgang Kunther et al. [41] reported that the strength of mortar cubes decreases as the Ca/Si ratio increases. It can be seen from **Table 5** that the ratio of Ca/Si decreases for replacement levels and hence maximum strength is obtained at 50% replacement level.

6.4 FTIR Spectrum for 28 days cement mortar with nano materials

Fourier transform infrared spectroscopy is a technique used to obtain an infrared spectrum of absorption or emission of a solid, liquid or gas. an Fourier transform infrared spectroscopy spectrometer simultaneously collects

Figure 6.
SEM image of cement mortar cured for 28th days.

Figure 9. EDAX images of cement mortar cubes with nano cement cured for 28th days (a) 10% replacement (b) 20% replacement (c) 30% replacement (d) 40% replacement (e) 50% replacement.

The Y-axis is labeled as Transmittance in % and ranges from 0 at the bottom to 100 at the top. The characteristic peaks in the infrared spectrum were determined. All infra-red spectra contain many peaks. However, the large peaks on the spectrum will provide the data necessary to read the spectrum. The regions of the spectrum

in which the characteristic peaks exist were determined. The characteristic peak is compared to IR spectrum and the compounds were identified. an Fourier transform infrared spectroscopy transmission spectrum of the normal cement mortar is shown in **Figure 10**.

According to Hasan Biricika and Nihal Sarierb [42], Xu et al. [43] and Varas et al. [44], the band of the spectra between 3640 cm^{-1} to 3400 cm^{-1} corresponds to the structural -OH group formed during the hydration of C_2S and C_3S and free -OH group from water molecules present in the mixture. The spectral band between 2500 cm^{-1} and 1500 cm^{-1} corresponds to the H-O-H absorbed water molecule group which indicates the decrease in the free water and between 1500 cm^{-1} to 400 cm^{-1} and it corresponds to Si-O and Si-O-Si silicate group which indicate the formation C-S-H.

The FTIR transmission spectra of cement mortar in which cement was replaced with nano cement are shown in **Figure 11**. From the FTIR transmission spectra, the peaks attained by the mortar with nano cement are given in **Table 6**.

From **Figure 10** and **Table 6**, it can be seen that the band value of 3429.43 cm^{-1} diminishes to 462.92 cm^{-1} which implies the formation of C-S-H gel.

Figure 11 and **Table 6**, it can be seen that the band value between 3635.74 cm^{-1} and 3410.28 cm^{-1} diminishes to 488.34 cm^{-1} and 455.87 cm^{-1} which implies that strength has been achieved. It can be seen that the lowest band value is seen in 50% replacement of cement with nano cement and the decrease in the band value implies a decrease in free water and increase in strength.

S.No	Element	Atomic percentage of elements					
		% Replacement of cement with nano cement					
		0%	10%	20%	30%	40%	50%
1	O	53.9	78.3	71.7	73.7	71.7	69.1
2	Al	5.80	2	3.3	3.9	1.5	0.43
3	Si	12.7	7.9	9.1	10.2	17.9	29.4
4	Ca	26.9	10.3	14.1	11.3	8.3	1.5
5	Fe	0.70	0.6	1	0.7	0.6	0.12
6	Ca/Si	2.12	1.3	1.5	1.1	0.5	0.1

Table 5.
Results of EDAX analysis of cement mortar with nano cement.

Figure 10.
FTIR transmission spectrum of cement mortar cured for 28th days.

Figure 11. FTIR transmission spectra of cement mortar on 28th day with nano cement (a) 10% replacement (b) 20% replacement (c) 30% replacement (d) 40% replacement (e) 50% replacement.

FTIR spectra peak of mortar with NC in cm ⁻¹					
%RCNP					
0	10	20	30	40	50
3429.43	3635.74	3634.87	3639.35	3410.28	3410.28
2626.61	3427.98	3425.87	3412.84	1408.37	2510.47
1423.47	1617.48	2715.38	1409.85	1058.76	1417.38
1012.63	1387.87	1625.47	1055.38	778.24	1055.87
462.92	1002.57	1419.87	779.34	460.84	455.87
	773.27	1004.87	460.87		
	488.34	778.37			
		470.63			

Table 6. FTIR transmission spectrum peak of cement mortar with nano cement.

From both the experimental and micro structural studies, it was noted that the compressive strength of cement mortar increases as the replacement level of cement with nano cement increases. From the SEM analysis, it was seen that the nano particles filled the pores and made the concrete denser. Additional formation

of C-S-H gel can be seen from SEM analysis, reduction of Ca/Si ratio from EDAX analysis and the reduction of band spectra values from FTIR analysis which indicate the increase in strength of cement mortar with nano particles.

7. Compressive strength of concrete with nano materials

The compressive strength of M20, M30, M40 and M50 grades of concrete with and without nano cement are presented in **Table 7**.

Figure 12 shows the compressive strength of concrete with respect to the percentage replacement of cement with NC.

From the **Table 7** and **Figure 12**, it can be seen that the compressive strength increases with the increase in percentage replacement of cement with nano cement. It can be seen that the compressive strength increases up to 50% replacement of cement with nano cement. The correlation coefficient between the compressive strength and percentage replacement of cement with nano cement were found to be 0.9971, 0.9946, 0.9717 and 0.9928 for M20, M30, M40 and M50 grades of concrete respectively.

It can be seen that the compressive strength increases as the replacement of cement with nano materials, curing days and grade of concrete increase. The increase in compressive strength was found to range between 0.71% and 31.5%. The percentage increase in the compressive strength of M20 grade was found to be more than that of M50 grade of concrete. Saloma et al. [45] reported that the rapid development of the compressive strength of concrete with nano materials is due to the fact that nano materials serve as a filler to increase the density and as an activator

Average compressive strength of concrete with nano cement N/mm ²						
GC		M20			M30	
%RCNC	28 th day	56 th day	90 th day	28 th day	56 th day	90 th day
0	25	27.3	33.1	32	34.7	42
10	26.1	28.7	36.5	32.6	35.6	43.4
20	27.6	29.9	37.3	33.7	36.7	44.6
30	28.4	32.2	38.9	35.1	37.9	46.3
40	29.5	34	41.1	36.4	38.9	47.7
50	31	35.9	42.8	37.4	39.7	48.8
GC		M40			M50	
%RCNC	28th day	56th day	90th day	28th day	56th day	90th day
0	42	44.3	48	49.9	51.4	59.3
10	44.3	46.6	49.6	52.3	55.1	62.6
20	46	47.6	52.7	53.2	56.7	64.2
30	45.7	48.3	54	55.3	58.6	66.8
40	47.8	49.8	55.5	57.2	60.9	69.4
50	49.2	50.8	56.6	59.9	63.9	70.9

GC is grade of concrete.
%RCNC is percentage replacement of cement with nano cement.

Table 7.
Compressive strength of concrete with nano cement.

Figure 12. Compressive strength of concrete with nano cement (a) M 20 grade of concrete (b) M 30 grade of concrete (c) M 40 grade of concrete (d) M 50 grade of concrete.

NP	Particulars	Variations in compressive strength			
		M 20	M30	M40	M50
NC	%	4.4 to 31.5	1.88 to 16.88	3.33 to 17.92	4.81 to 24.32
	ratio	1.04 to 1.32	1.02 to 1.17	1.03 to 1.18	1.06 to 1.24

Table 8. Range in the variations in compressive strength for different grades.

in the hydration reaction and reacts with free Ca(OH)_2 resulting in concrete with higher compressive strength. The ranges in the variations of compressive strength are given in **Table 8**.

From the **Table 8**, it can be seen that the nano cement is very effective in increasing the compressive strength of concrete.

8. Conclusions

8.1 Effect of grinding on materials

The micro-sized cement was converted to nano size by grinding it in a ball grinding mill for 12 hours and the particle size was found to range from 45 nm to 86 nm.

The specific surface areas of nano cement, increased by 13008.11%, when compared with that of ordinary portland cement.

The chemical properties of nano sized particles were found to be the same as the particles before grinding.

8.2 Effect of nano materials on properties of cement paste and cement mortar

The normal consistency of cement paste with nano cement was found to range between 33% and 35%.

The initial setting time of cement paste with nano cement, was found to decrease to 30 minutes at 50% replacement level when compared to the initial setting time was 50 minutes for the normal cement paste.

The final setting time of cement paste with NC, was found to decrease to 245 minutes at 50% replacement level when compared with the final setting time of 510 minutes for the normal cement paste.

The percentage increase in compressive strength of cement mortar with nano cement was found to range between 23.76 and 64.91 when compared with the compressive strength of the normal cement mortar.

The optimum replacement level of nano cement is 50%. Replacement level beyond 50% will result in the rapid setting which is not desirable.

8.3 Effect on compressive strength of concrete

The compressive strength of concrete was found to increase as the replacement level of cement with nano cement increases for all grades of concrete and for all curing days considered. The percentage increase in strength was found to vary between 2% and 29.3% for nano cement. The lower percentage of 2% was obtained for M30 mix cured for 28 days at a replacement percentage of 10%. The higher percentage of 29.3% was obtained for M20 mix cured for 90 days at a replacement percentage of 50%.

Conflict of interest

The authors declare no conflict of interest.

Author details

Jemimah Carmichael Milton^{1*} and Prince Arulraj Gnanaraj²

¹ Department of Civil Engineering, Vignan's Lara Institute of Technology and Sciences, Vadlamudi, Guntur, Andra Pradesh, India

² School of Engineering and Technology, Karunya Institute of Technology and Sciences, Coimbatore, Tamil Nadu, India

*Address all correspondence to: jemimahcarmichael@gmail.com

IntechOpen

© 2020 The Author(s). Licensee IntechOpen. This chapter is distributed under the terms of the Creative Commons Attribution License (<http://creativecommons.org/licenses/by/3.0>), which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

References

- [1] Konstantin Sobolev. Modern developments related to nanotechnology and nanoengineering of concrete. *Frontiers of Structural and Civil Engineering*. 2016;**10**(2):131-141
- [2] Hanus MJ. Nanotechnology innovations for the construction industry. *Progress in materials science*. 2008
- [3] Gann D. A Review of Nanotechnology and its Potential Applications for Construction. SPRU, University of Sussex. 2002
- [4] Perumalsamy B, Ken C. Nanotechnology of concrete: Recent developments and future perspectives, *Nanotechnology and Concrete: Proceedings of ACI Session*; 2006:15-28
- [5] Surinder M. Nanotechnology and construction. *European Nanotechnology Gateway-Nanoforum Report*, Institute of Nanotechnology; 2006:2-10
- [6] Florence S, Konstantin S. Nanotechnology in concrete—a review. *Construction and building materials*; 2010;**24**:2060-2071
- [7] Bhuvaneshwari. B, Saptarshi S, Nagesh RI. Nanoscience to Nanotechnology for Civil Engineering - Proof of Concepts. *Proceedings of the 4th WSEAS International conference on Energy and development-environment-biomedicine*, Corfu Island, Greece. 2011:230-235
- [8] Hui L, Hui-gang X, Jie Y, Jinping O. Microstructure of cement mortar with nano-particles. *Composites Part B Engineering*; 2004;**35**:185-189
- [9] Maile A, Huang CP. The chemistry and physics of nano-cement. Report submitted to NSF REU University of Delaware; 2006:1-28
- [10] Tao J. Preliminary study on the water permeability and microstructure of concrete incorporating nano-SiO₂. *Cement and concrete research*; 2005;**35**(10):1943-1947
- [11] Ali N, Shadi R. The effects of SiO₂ nano particles on physical and mechanical properties of high strength compacting concrete. *Composites Part B: Engineering*; 2011;**42**(3):570-578
- [12] Thomas PK, Satpathy SK, Manna I, Chakraborty KK, Nando GB. Preparation and characterization of nano structured materials from fly ash. *Nanoscale Research Letters*; 2007;**2**(8):397-404
- [13] Hanus MJ, Andrew TH. Nanotechnology innovations for the construction industry. *Progress in materials science*; 2013;**58**(7):1056-1102
- [14] Silverstre J, Silvestre N, Brito JD. Review on concrete nanotechnology. *European Journal of Environmental and Civil Engineering*; 2016;**20**(4):455-485
- [15] Elzbieta H. Properties of cement-based composite modified with magnetic nanoparticles: A review *Materials*; 2019:1-34
- [16] Konstantin S, Miguel FG. How nanotechnology can change the concrete world. *American ceramic Society Bulletin*; 2005;**84**:16-20
- [17] Zhi G, Zhili G. Applications of nanotechnology and nanomaterials in construction. *First International Conference on Construction in Developing Countries (ICCIDC-I), Advancing and Integrating Construction Education, Research & Practice*, Karachi, Pakistan; 2008:235-240

- [18] Laila R, James B, Rouhollah A, Jon M, Taijiro S. Cement and concrete nanoscience and nanotechnology. *Materials*; 2010;**3**:918-942
- [19] Jafarbeglou M, Abdouss M, Ramezaniapour AA. Nanoscience and nano engineering in concrete advances a review. *International journal of nano science and nanotechnology*; 2015;**11**(4):263-273
- [20] Guillermo B, Faustino P, Faustino P, Julia A. Nano-inclusions applied in cement-matrix composites: A review. *Materials*; 2016;**9**(12):10-15
- [21] Muhd NMS, Hamidah MS, Mohd FA. Applications of using nano materials in concrete: A review, *Construction and Building Materials*; 2017;**133**:91-97
- [22] Hosam MS, Fathy A El-Saied, Taher AS, Aya AH. Macro-and nanomaterials for improvement of mechanical and physical properties of cement kiln dust-based composite materials. *Journal of Cleaner Production*; 2018a-1;**204**:532-541
- [23] Hosam M, El-Sheikh SM, Elshereafy EE, Essa AK. Mechanical and physical characterization of cement reinforced by iron slag and titanate nano ibers to produce advanced containment for radioactive waste. *Construction and Building Materials*; 2018b -2; **200**:135-145
- [24] Hosam MS, El-Saied FA, Salaheldin TA, Hezo AA. Influence of severe climatic variability on the structural, mechanical and chemical stability of cement kiln dust-slag-nanosilica composite used for radwaste solidification. *Construction and Building Materials*; 2019a -1;**218**:556-567
- [25] Hosam MS, El-Sheikh SM, Elshereafy EE, Essa AK. Performance of cement-slag-titanate nanofibers composite immobilized radioactive waste solution through frost and flooding events. *Construction and Building Materials*; 2019b -2; **223**:221-232
- [26] Narasimha MI, Venkata RD, Babu Rao J. Microstructure and mechanical properties of aluminum-fly ash nano composites made by ultrasonic method. *Materials and design*; 2011;**25**:55-65
- [27] Gujjala R, Shakuntala O, Samir KA, Pal Sk. Fabrication and characterization of nano fly ash by planetary ball milling. *International journal of material science innovations*; 2014;**2**(3):59-68
- [28] Sada AKHA, Iyad SA, Prabir KS. Feasibility of producing nano cement in a traditional cement factory in Iraq. *Case studies in construction materials*. 2017;**7**:91-101
- [29] Bickbau MY, Shykun VN. Nano-cements future of world cement industry and concrete technology. Program International conference, seminar, Dubai; 2017:3-33
- [30] Parang S, Frisky S, Dion AF. The effect of nano-cement content to the compressive strength of mortar. 2nd International Conference on Sustainable Civil Engineering Structures and Construction Materials 2014 (SCESCM 2014), *Procedia Engineering*; 2014:386-395
- [31] Ikhlef B. Test on nano cement mortar and concrete. Program International conference, Dubai; 2017:38-55
- [32] Gengying L. Properties of high-volume fly ash concrete incorporating nano-SiO₂. *Cement and Concrete Composites*; 2004;**34**(6):1043-1049
- [33] Zaki SI, Khaled SR. How nano technology can change the concrete industry. SBEIDCOH International conference on sustainable Built Environment Infrastructures in

developing countries, Oran, Algeria; 2009:407-414

[34] Nili M, Ehsani A, Shabani K. Influence of nano-SiO₂ and microsilica on concrete performance. *Construction Materials and Technologies*; 2010:1-7

[35] Praveen S, Janagan SS. Partial replacement of cement with nano flyash(class c) and nano GGBS. *International research journal of engineering and technology*; 2015;2(8):979-983

[36] Harihanandh M, Sivaraja M. Strength and mechanical properties of nano fly ash concrete. *International Journal of Advanced Engineering Technology*; 2016;7(2):596-598

[37] IS: 12269-2013. Indian Standard Specification for 53 Grade Ordinary Portland Cement. Bureau of Indian Standards, New Delhi, India

[38] IS: 383-2016. Indian Standard Specification for Coarse and Fine Aggregates from Natural Sources for Concrete. Bureau of Indian Standards, New Delhi, India

[39] IS: 456-2000. Indian Standard Code of Practice for Plain and Reinforced Concrete. Bureau of Indian Standards, New Delhi, India

[40] IS: 5513-1996. Vicat Apparatus – Specification. Bureau of Indian Standards, New Delhi, India

[41] Wolfgang K, Sergio F, Jorgen S. Influence of the Ca/Si ratio on the compressive strength of cementitious calcium–silicate–hydrate binders. *Journal of Materials Chemistry*; 2017;33:17401-17412

[42] Hasan B, Nihal S. Comparative study of the characteristics of nano silica, silica fume and fly ash incorporated cement mortars. *Materials Research*; 2014;17(3):570-582

[43] Xu P, Kikpatrick RJ, Poe B, McMillan PF, Cong X. Structure of calcium silicate hydrate (C-S-H): Near-, mid-, and far-infrared spectroscopy. *Journal of the American Ceramic Society*; 1999;82(3):742-748

[44] Varas MJ, Alvarez de Buergo M, Fort R. Natural cement as the precursor of Portland cement: Methodology for its identification. *Cement and Concrete Research*; 2005;35:2055-2065

[45] Saloma AN, Iswandi I, Mikrajuddin A. Improvement of concrete durability by nanomaterials. *Procedia Engineering*; 2015;125:608-612