**PERFORMANCE OF CONCRETE BY INCORPORATING NANO-SIZED PARTICLES**

 **MID SEMESTER REPORT**

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Thesis

By

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**ABSTRACT**

Concrete can be nano-engineered by incorporating nano-sized particles to modify its properties reducing the production cost of construction materials to provide concrete with superior performance. The nanoparticle is the elementary building block in nanotechnology and is comprised of up to thousands of atoms combined into a cluster of 1-100 nm. Reduction in the size provides an exceptional surface area-to-volume ratio thereby changing its basic properties and reactivity. Inclusion of nano-particles significantly enhances the mechanical performance of a variety of materials, including metals, polymers, ceramic and concrete composites. Nano-silica (silicon dioxide nanoparticles), for example, has been shown to improve workability and strength in concrete. Nano-alumina (aluminium trioxide nanoparticles) and Nano-titania (titanium dioxide nanoparticles) has shown better performance towards durability of concrete. In the present work concrete of M40 grade induced with both nano-silica (NS) and nano-alumina (NA), nano-silica and nano-titania (NT) are to be tested for fresh and dry properties of concrete including durability. Also to report the combine optimum dosage of both materials as a percentage weight of cement for better performance concrete.

Keywords: Concrete; Nano-silica; Nano-alumina; Optimum dosage

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CHAPTER 1

INTRODUCTION

Concrete is a building material composed of cement as well as some percentage of other cementitious materials such as slag and flyash, coarse aggregate such as gravel and granite, fine aggregate such as sand and crush rock fines(CRF), water and admixtures. Concrete is mostly used than other man-made material in the world. The ability of concrete to resist the action of water without serious deterioration makes it an ideal material for building structures to control, store and transport water. In spite of all these it has some serious deficiencies which, but for its remarkable qualities of flexibility, resilience and ability to redistribute stress, would have prevented its use as a building material. Efforts to improve the properties of concrete are continuously being made by researchers which led to the development of fiber reinforced concrete, Ferro-cement concrete etc. in recent years, improvement in concrete properties have been achieved by the invention of high performance concrete (HPC). Improvements involving a combination of improved compaction, improved paste characteristics an aggregate-matrix bond and reduced porosity are achieved through the use of super plasticizers. Further enhancements of some properties have been obtained through the addition of mineral admixtures such as metakaolin, silica fume and fly ash.

 A concrete structure is built so as to last and maintenance free service as far as possible during the life of structure. Therefore apart from strength, long term behavior under service conditions and environmental effects has also become important consideration in evaluating performance. To overcome various problems encountered in the field and to achieve better performance even in aggressive environments, use of high performance concrete is becoming a more popular solution.

High performance concrete is a concrete in which certain characteristics are developed for a particular application and environment, so that it will give excellent performance in the structure in which it will be placed in the environment to which it will be exposed, and with the loads to which it will be subjected during the design life. It includes concrete that provides either substantially improved resistance to environmental influences (durability in service) or substantially increased structural capacity while maintaining adequate durability. It may also include concrete, which significantly reduces construction without compromising long term serviceability.

 Examples of characteristics that may be considered critical in an application requiring performance enhancements are, ease of placement and compaction without segregation, early age strength, long-term mechanical properties, permeability, and density, heat of hydration, toughness, volume stability and durability. Concrete possessing many of these characteristics often achieve high strength. Therefore HPC is often of higher strength, but high strength concrete may not be of high performance. HPC is usually contains additional ingredients such as mineral and chemical admixtures compared to conventional concrete. Thus, in practical applications of this type of concrete, the emphasis has in many cases gradually shifted from compressive strength to the other properties of the materials such as high tensile strength, high modulus of elasticity, high density, low permeability and high resistance to some forms of attacks. The cost and other benefits derived may include less material, light weight and fewer structural elements, reduced maintenance, extended life cycle.

 An admixture can be defined as a chemical product, which is added to the concrete mix in quantities no larger than 5% by weight of cement. It is added to batch immediately before or during mixing for the purpose of achieving a specific modification or modifications to normal properties of concrete. Admixtures may be organic or inorganic in composition but their chemical character is an essential feature. Certain organic compounds (admixtures) are used in the concrete as workability agents. A new admixture called Fenugreek comes under this category is used as workability agents to the mix designed with different percentages of admixture, and then its effects are observed in improving workability of concrete and simultaneously behavior of concrete during compressive strength test.

CHAPTER 2

LITERATURE REVIEW

1. Anwar M. Mohamed et al.(2016) “Influence of nano materials on flexural behavior and compressive strength of concrete”

This paper investigates the effect of nano particles on the mechanical properties at dif-ferent ages of concrete. Different mixtures have been studied including nano-silica (NS), nano-clay (NC) or both NS and NC together with different percentages. Mechanical properties have been investigated such as compressive and flexure strength through testing concrete prisms 40, 40 and 160 mm at 7, 28 and 90 days in order to explore the influence of these nano particles on the mechan-ical properties of concrete. Results of this study showed that nano particles can be very effective in improving mechanical properties of concrete, nano-silica is more effective than nano clay in mechanical properties and wet mix gives higher efficiency than dry mix. Exceeding a certain percent-age of nano particles in concrete negatively affects the mechanical properties. Also, binary usage of nano particles; (NS + NC) had a remarkable improvement appearing in concrete compressive strength than using the same percentage of single type of nano particles. This improvement can be attributed to the reaction of nano materials with calcium hydroxide Ca(OH)2 crystals, which are arrayed in the interfacial zone (ITZ) between hardened cement paste and aggregates, and produce C- S-H gel and the filling action of nano particles which cause more densified microstructure.

Conclusions

1- Nano Silica in wet condition and nano Clay in dry con-dition have remarkable improvement on the compres-sive strength of high performance concrete till 18% days enhancement for nano silica and 11% enhancement for nano clay at 90 days, due to the reaction of nano materials with calcium hydroxide Ca(OH)2 crystals, which are arrayed in the interfacial zone (ITZ) between hardened cement paste and aggregates, and produce C-S-H gel and the filling action of nano particles which cause more densified micro structure.

2- An improvement for flexure strength due to use of nano particles can be achieved till 4% and 8% at 90 days for NS and NC respectively, due to the filler action of nano particles which increases the bond between the aggregate and the cement matrix.

3- The optimum percentage for replacement of cement with nano particles which was 0.75% for NS and 3% for NC in this study and additional percentage may reflect negatively on the compressive and flexure strength of concrete due to agglomeration of nano particles, also to achieve economical mix since increasing the cost of mix without major effect on the mechanical properties is noticed.

4- The wet mix for nano clay is more efficient than dry mix with approximately 24% and 32% improvement in com-pressive and flexure strength, respectively at 90 days due to the good uniform dispersion of the nano particles in concrete mix and suitable reaction conditions.

5- The hybrid nano particles had a remarkable improve-ment on the compressive strength of high performance concrete than the same percentage of the nano particles used separately. This improvement can be referred to the filler action of nano silica and nano clay and proper mix percentage. The optimum Hybrid ratio in this study can be noticed with 3% nano particles consists of 25% NS and 75% NC which can easily be produced in Egypt and which gives best results in both compressive and flexure strength, in addition this percentage is nearly the same of the optimum percentage of each nano type used alone.

6- The wet mix hybrid nano particles are more efficient than dry mix hybrid with approximately 6% improve-ment in compressive strength at 90 days due to the good uniform dispersion of the nano particles in concrete mix.

7- The dry mix hybrid nano particles are more efficient than wet mix hybrid with approximately 28% and 52% improvement in flexure resistance at 90 days for 1% and 3% hybrid nano particles respectively, due to the increase in the bond between aggregate and cement paste. Nano-particles improved the quality of the inter-facial transition zone due to the precipitation of stronger C- S-H and the accelerated rate of hydration.

1. Saloma et al(2015) “Improvement of concrete durability by nanomaterials”

The green concrete capable for sustainable development is characterized by application of industrial wastes to reduce pollution of the environment. Fly ash processed with nanotechnology developed by Indonesia Center for Ceramics using Polishing Liquid Milling Technology. Nanomaterial concrete is new generation concrete formed of materials of the grain size of nanoscale. The materials used in this research were cement type I, nanosilica 10 - 150 nm, quartz powder in 0.3 - 25.0 m, fine sand (quartz of sand) size of 50 - 650 m, coarse aggregate in 5 - 10 mm, and super plasticizer. In this paper, mechanisms are discussed by which the incorporation of nanomaterials in concrete enhances durability to sulfate attack. Application of nanotechnology is an effective way to reduce environment pollution and improve durability of concrete.

Based on the research conducted on the concrete nanomaterial can be summarized as follows:

1. Nanosilica is capable of improving the performance of concrete.
2. Based on the test results of concrete compressive strength at 28 days, a maximum compressive strength value is 129.48 MPa.
3. The improvement of 10% nano silica replacement in the resistance to sulfate attack is better than that of 0% nano silica.
4. Spiesz et al. (2014) “Effect of nano-silica on the hydration and microstructure development of Ultra-High Performance Concrete (UHPC) with a low binder amount”

This paper deals with the effect of nano-silica on the microstructure development and hydration of Ultra-High Performance Concrete (UHPC) with a low binder amount. The design of UHPC is based on the Andersen and modified Andreasen particle packing model. The results show that by utilizing this model - a dense and homogeneous skeleton of UHPC can be obtained with relatively low binder amount (about 440 kg/m3). Due to the high amount of superplasticizer utilized to produce UHPC in the present study, the dormant period of the cement hydration is extended. However, due to the nucleation effect of nano-silica, the retardation effect from superplasticizer can be significantly compensated. Additionally, with the addition of nano-silica, the viscosity of UHPC significantly increases, which causes that more air to be entrapped in the fresh mixtures and the porosity of the hardened concrete correspondingly increases. In contrary, due to the nucleation effect of nano-silica, the hydration of cement can be promoted and more C–S–H gel can be generated.

Conclusions drawn out are :- Using the modified Andreasen and Andersen particle packing model, a dense and homogeneous skeleton of UHPC can be obtained with a relatively low binder amount (about 440 kg/ m3 ). When mixed with about 2.5% (vol.) of steel fibers, the flexural and compressive strength of the reinforced UHPC are around 25 and 135 MPa. An optimal amount of the utilized nano-silica (3.74% by the mass of the binder amount found here) corresponds to the highest mechanical properties of UHPC. In this study, due to a large amount of superplasticizer utilized to produce UHPC, the hydration of cement is obviously retarded. However, the addition of nano-silica can significantly compensate this retardation effect. With the addition of nano-silica, the viscosity of UHPC signifi- cantly increases, which causes that more air voids are entrapped in the fresh mixtures and the porosity of the hardened concrete correspondingly increase. However, in contrary, due to the nucleation effect of nano-silica, the hydration of cement can be promoted and more C–S–H gel can be generated. Hence, there is an optimal nano-silica amount for the production of UHPC with the lowest porosity, at which the positive effect of the nucleation and the negative influence of the entrapped air can be well balanced.

1. Aref Sadeghi Nik et al (2013)-“Estimation of compressive strength of self-compacted concrete with fibers consisting nano-SiO2 using ultrasonic pulse velocity”

In this research the performance of ultrasonic pulse velocity in concrete is examined as a nondestructive experiment, in order to estimate compressive strength of fiber-reinforced self-compacted concrete with nano particles. For this purpose there were 40 mix plans including four groups of A, B, C and D in which cement was replaced with 0, 2, 4 and 6 vol.% of nano-SiO2 respectively. In this experiment a comparison was made between the four groups which included three types of fibers (steel: 0.2, 0.3 and 0.5 vol.%, poly-propylene: 0.1, 0.15 and 0.2 vol.% and glass: 0.15, 0.2 and 0.3 vol.%). Cube specimens were tested in order to determine ultrasonic velocity. The compressive strength was also tested. According to the results, rela-tions were established between ultrasonic velocity in the specimens and the compressive strength at dif-ferent ages and so the range of the velocity of the waves was also determined for this kind of concrete.

The results of the study are as follow:

Proposed formula by former researchers which were an exponential relationship between compressive strength and ultrasonic pulse transmission velocity in concrete, has a good regression with the experimental results from this study. It seems that increasing the volumetric percentage of steel fiber up to 0.3% will cause an increase in pulse velocity. While in case of polypropylene fibers, the increase of the volumetric percent-age will cause a decrease in the velocity, for the case of glass fiber there is first an increase in velocity and then a decrease. Also when the percentage of nano-SiO2 is increased up to 4% of the cement weight in the specimens, there is first an increase in both compressive strength and pulse velocity but then they almost decrease. This is due to filling and pozzolan effect of nano-SiO2 in reinforcing fiber zone and cement matrix. It’s afterward decline is due to lumping of the silica which is itself because of its high specific surface. A physical reaction causes the silica to get together and make unstable lumps.

1. Kiachehr Behfarnia et al.(2013) -“The effects of nano-silica and nano-alumina on frost resistance of normal concrete”

In this study frost resistance and mechanical properties of concrete containing nano-silica and nano-alu-mina are studied. Nano-particles were employed as a partial substitute of cement. The specimens were subjected to cycles of freezing and thawing in water according to ASTM C666A. The reduction in com-pressive strength, loss of mass, change in length and water absorption of specimens was measured after specified number of freeze and thaw cycles. Experimental results showed that the frost resistance of con-crete containing nano-particles were considerably improved, as result of a more compacted microstruc-ture. It was also concluded that the frost resistance of concrete containing nano-Al2O3 was better than that containing the same amount of nano-SiO2. Compressive strength of normal concrete containing nano-SiO2 was higher than that containing the same amount of nano-Al2O3.

The following primary conclusions can be drawn:

1. The compressive strength of concrete specimens increased by using nano-Al2O3 as cement replacement. Compressive strength of concrete specimens increased by increasing the nano-alumina content.
2. Replacement of cement content with nano-SiO2 improved the compressive strength of concrete mixes used in this research. In this study, the optimum content of nano-SiO2 in concrete in order to increase its compressive strength was 5 wt%.
3. Frost resistance of concrete mixes can be considerably improved by the addition of nano-Al2O3 and nano-SiO2. These nano-materials behave not only as promoters of poz-zolanic reaction but also as fillers improving the pore struc-ture of concrete and densifying the microstructure of cement paste.
4. The frost resistance of concrete containing nano-Al2O3 is better than that containing the same amount of nano-SiO2.

1. Alaa M. Rashad (2013) “A synopsis about the effect of nano-Al2O3, nano-Fe2O3, nano-Fe3O4 and nano-clay on some properties of cementitious materials.”

Nanotechnology is one of the most active research areas with both novel science and useful applications that has gradually established itself in the last two decades. Nanoparticles belong to be prospective mate-rials in the field of civil engineering. Some researchers have employed nanoparticles into cementitious materials-based on Portland cement (PC) aiming to modify some properties of this system. This paper presents an overview of the previous works carried out on the effect of using nano-Al2O3, nano-Fe2O3, nano-Fe3O4 and nano-clay into the cementitious materials. Some properties of the modified composites as heat of hydration, workability, setting time, mechanical strength, water absorption and durability were reviewed.

1. The inclusion of NA into the cementitious matrix-based on PC accelerated the peak times, decreased workability and decreased the initial and final setting times.
2. The inclusion of NA (0.5–5% in mortars and 0.5–3% in con-cretes) into the matrix increased the compressive strength, splitting tensile strength, flexural strength and elastic mod-ulus. The optimum NA content that gave the highest strength is still different from one author to another, but 1–2% seemed to be the optimum.
3. The abrasion resistance of concrete increased with increas-ing NA content up to 2%.
4. The inclusion of NA into the matrix reduced the percentage of water absorption at later ages. 0.5% seemed to be the opti-mum content, but it depends on curing condition and other cementitious materials blended with PC, where adversely effect of NA on the percentage of water absorption was obtained in mortar containing FA.
5. The inclusion of NF into the matrix accelerated the peak times and dropped the heat rate values. The workability decreased with increasing NF content.
6. The inclusion of NF (0.5–10% in mortars and 0.5–5% in con-cretes) into the matrix increased the mechanical strength. Some authors reported 3% is the optimum, other reported 4%, other reported 1% and other reported 2%; this may depend on curing condition and type of pozzolan blended with PC. 0.5% NF showed the optimum content in mortar containing either 5% SF or 5% FA.
7. The inclusion of NF (up to 5%) into the matrix increased the percentage of water absorption at early ages (up to 7 days), but reduced it at later ages. Some authors reported that 0.5% NF is the optimum content and other reported 4%. In matrix blended with pozzolan, 0.5% NF is the optimum content in mortar containing 5% SF, whilst 1.25% is the optimum con-tent in mortar containing 5% FA.
8. The inclusion of nano-Fe3O4 (up to 0.3%) into the matrix increased the compressive strength, whilst the inclusion of nano-Fe3O4 (up to 1.5%) into concrete containing 15% MK increased the compressive strength as well as reduced the percentage of water absorption and the chloride penetration.
9. The inclusion of NC into the matrix increased the mechanical strength. Some authors reported 1% is the optimum, other reported 6%, but most authors used NC up to 2.5%. The addition of NC up to 2.5%, in mortars, improved the chloride penetration resistance, reduced ASR and improved strength after wet/dry cycles, whilst the addition of 0.6% and 0.8% NC, in pastes, almost increased the permeability coefficient. On the contrary, the addition of NC up to 0.4%, in pastes, reduced the permeability coefficient.
10. L P Sing et al.(2013) “Beneficial role of nanosilica in cement based materials”

Nanomaterials are gaining widespread attention to be used in construction sector so as to exhibit enhanced performance of materials in terms of smart functions and sustainable features. During the last one decade a number of nanomaterials such as nanosilica, nanotitania, carbon nanotubes and nanoalu-mina have been explored and among them nanosilica has been used most extensively. A number of pub-lications appeared towards the use of nanosilica in cementitious system is mainly due to the fact that concrete remains the most complex material and its hydration mechanism is still not completely under-stood. Consequently, researchers are focusing on the basic science of this material at nano/atomic level. Further, researchers are continuing to improve the durability and sustainability of concrete, and they have realized significant increment in mechanical properties of cementitious materials by incorporating nanosilica.Physical state and dispersion of nanosilica into the concrete is a major issue. Although various dispersing agents are in action, their feasibility in field is still questionable. A thorough study on dispersion mechanism is required.

The optimum quantity of nanosilica for concrete or cement paste cannot be fixed with certain percentage. It all depends on the type of nanosilica (colloidal, dry powder, etc.) and the average particle size of nanosilica which can be expressed in terms of surface area to mass ratio. In this aspect a relationship should be established between optimum quantity and characteristics of nanosilica.

Most of the research works have been done with cement pastes and mortars, only a few researchers worked extensively on mechanical properties and permeability of the nanosilica added concrete. Other durability properties are still not been investigated. Due to incorporation of the finer materials shrinkage behavior could possibly be getting altered, which need to be studied. Further investigations on carbonation, corrosion resistance, acid resistance, sulfate resistance, etc. are to be assessed.

Even though researchers investigated some properties of concrete, they are not enough in confidence building for their use in concrete. It is high time to carry out a complete systematic experimental study to assess the properties of nanosilica incorporated concrete. Optimization, fresh, mechanical, microstructural and durability properties of concrete should be investigated in full thrust.

1. Nilofar Salemi et al.(2012) “Effect of nano-particles on durability of fiber-reinforced concrete pavement”

In this paper mechanical properties and frost resistance of fiber-reinforced concrete pavement containing nano-particles are studied. Nano-particles are employed to be as substitute of a portion of cement. For comparison the mechanical properties and frost resistance of plain concrete (control concrete), concrete containing polypropylene fibers and concrete containing nano-particles (without fibers) are also experimentally studied separately in this work. The specimens were subjected to cycles of thawing and freezing in water according to ASTM C666A.

Following are the primary conclusions from the paper (a) The compressive strength of concrete can slightly be increased by using nano-alumina as a substitute to cement materials partially. Compressive strength of concrete was found to increase as much as 8% by replacing cementitious materials by nano-alumina. (b) Nano-silica considerably improves the compressive strength of concrete. The optimum content of nano-silica in concrete in order to increase its compressive strength is 5% (by the weight of cementitious materials). The compressive strength of concrete improves as much as 30% by using 5% nano-silica (by the weight of cementitious materials). (c) Using nano-particles in concrete lower the workability of the concrete. (d) Frost resistance of concrete can be considerably improved by the addition of nano-particles. Nano-alumina and nano-silica behave not only as promoters of pozzolanic reaction (because of high surface area to volume ratio), but also as fillers, improving the pore structure of concrete and densifying the microstructure of cement paste. The strength loss of concrete containing 5% nano-silica was 16% while the strength loss of control concrete was 100% after 300 cycles. Using 5% nano-silica results in 84% reduction in deterioration of concrete after freezing and thawing cycles (in terms of compressive strength). Also 3% nano-alumina can improve the durability of concrete against freezing and thawing cycles as much as 82%. (e) PP fibers increase the frost resistance of concrete slightly. However the enhanced extent of frost resistance of concrete containing nano-particles is much larger than that of concrete containing PP fibers; so nano-particles are more favorable to improve the frost resistance of concrete than PP fibers. (f) The concrete containing both nano-particles and PP fibers showed to be the most frost resistant among the studied samples.

1. Shekari A & Razzaghi M (2011) “Influence of nano particles on durability and mechanical properties of high performance concrete.”

The influence of Nano-ZrO2, Nano-Fe3O4, Nano-TiO2 and Nano-Al2O3 on durability and mechanical properties of high performance concrete was experimentally investigated. For this purpose compressive tests and indirect tensile tests were conducted in order to investigate the effects of nano-particles on mechanical properties of high performance concrete. Moreover water absorption and chloride penetration tests were conducted to explore the effect of nano particles on durability of concrete. Results of this study showed that: All of the examined nano-particles can improve durability and mechanical properties of high performance concrete. The contribution of NA on improvement of mechanical properties of high performance concrete was more than the other nano-particles. All of the examined nano-particles had noticeable influence on improvement of durability parameters.

1. Aref et al(2011) “Nano-particles in Concrete and Cement Mixtures, Applied Mechanics and Materials”

Titanium nano particles and silicon nano particles were used in mixtures. Nano particles are synthesized using sol-gel method at 500oC in ambient media and the nano structures are studied using Scanning Electron Microscopy technique. In the experiments conducted silicon nano particles proven to have stable structure of concrete and cement mixtures.

Sol-gel Method:- the **sol-gel** process is a method for producing solid materials from small molecules. The method is used for the [fabrication](https://en.wikipedia.org/wiki/Manufacturing) of [metal oxides](https://en.wikipedia.org/wiki/Metal_oxide), especially the oxides of silicon and titanium. The process involves conversion of monomers into a colloidal solution (*sol*) that acts as the precursor for an integrated network (or *gel*) of either discrete particles or network [polymers](https://en.wikipedia.org/wiki/Polymer).

1. C. Lan et al.(2009) “Ductility of high strength concrete containing nano-particles”

The ductility of high strength concrete containing nano-TiO2 were experimentally studied and compared with that of plain concrete and concrete containing silica fume by stress-strain relationship. The results showed that the ductility of high strength concrete containing nano-TiO2 were better than that of plain concrete and concrete containing silica fume, which demonstrated that it is an available and effective way to improve ductility of high strength concrete by means of mixing nanophase materials into concrete. The origin of nano-particles improving ductility of high strengthen concrete was also preliminary interpreted.

1. Weiguo et al.(2007) -“Nano Particle Modified Bonding Agent for Concrete Repair”

The adhesive force at new-old concrete interface of the repairing concrete structure is usually very weak, a new type of nano particle modified new-old concrete interface agent was prepared in this paper to enhance the interfacial bond of the new-old concrete basing on the huge surface area and high activity of nano particles. The results indicate that the splitting strengths on the top surface or fracture surfaces treating with the nano particle modified new-old concrete interface agent between the new and old concretes are much higher than those treating with ordinary fly ash interface agent. The modification effect of nano particle on the bond of fracture surface is much more remarkable than that of top surface. The surface agent paste is more effective to repair the top surface while the surface agent mortar is more suitable to repair the fracture surface. The hydration product portlandite is depleted by nano particle so the directional crystallization of the portlandite is baffled, the nano particle can form some chemical bonds at the interfacial zone so the adhesive force is enhanced, the new-old concrete become an integral bulk with the treatment of nano particle modified interface bonding agent.

1. Hui Lui et al (2004)-“Microstructure of cement mortar with nano-particles”

The mechanical properties of nano-Fe2O3 and nano-SiO2 cement mortars were experimentally studied. The experimental results showed that the compressive and flexural strengths measured at the 7th day and 28th day of the cement mortars mixed with the nano-particles were higher than that of a plain cement mortar. Therefore, it is feasible to add nano-particles to improve the mechanical properties of concrete. The SEM study of the microstructures between the cement mortar mixed with the nano-particles and the plain cement mortar showed that the nano-Fe2O3 and nano-SiO2 filled up the pores and reduced CaOH2 compound among the hydrates. These mechanisms explained the supreme mechanical performance of the cement mortars with nano-particles.

The compressive and flexural strength of the cement mortars with nano-SiO2 and with nano-Fe2O3 were both higher than that of the plain cement mortar with the same w/b. The SEM observations also revealed that the nano-particles were not only acting as a filler, but also as an activator to promote hydration proves and to improve the microstructure of the cement paste if the nano-particles were uniformly dispersed. The optimum mixing volume of different nano-particles was not the same due to different functions. Study in this direction is required.

CHAPTER 3
MIXING PROCEDURE OF NANO MATERIALS IN CONCRETE

**Cement And Concrete Mixing**

When it comes to improvements in concrete properties, mixing technology is as important as concrete composition. Mixing is an essential step in the production of uniform, high quality concrete. Although numerous guidelines and regulations, e.g. DIN EN 206 cover the composition of concrete and its components, the actual process of cement mixing and concrete mixing is left to the user. It is decisive, that water, cement and admixtures are evenly dispersed and distributed down to a fine scale and that agglomerates are sufficiently dispersed. Insufficient dispersing or deagglomeration results in inferior concrete properties. Due to the low water content and the high dosage of admixtures, the mixing of self-compacting concrete (SCC) and ultra high strength concrete (UHPC) requires a longer mixing time or a more effective mixing technology.

Nanomaterials In Concrete During the hydration of the cement nanoscale hydration products, such as calcium hydrates form in the hardening concrete. Nano particles of silica or nanotubes turn into nano particles of cement during the solidification of the concrete. Smaller particles lead to shorter particle distance and a denser and less porous material. This increases the compressive strength and reduces the permeability. A major disadvantage of nanosize powder and materials, though, is the tendency to form agglomerates during wetting and mixing. Unless the individual particles are well dispersed, agglomeration reduces the exposed particle surface leading to inferior concrete properties. Click here to learn more about the benefits of ultrasonic mixing of cement pastes for precast, drycast and concrete plants.

**Ultrasonic Mixing Of Nanomaterials**

Ultrasonication is a very effective means for the mixing, dispersing and deagglomeration. The picture below shows a typical result of ultrasonic dispersing of fumed silica in water. Starting (green curve) at an agglomerate particle size of more than 200 micron (D50) most of the particles were reduced to less than 200 nanometers. Ultrasonic Mixing At Any Scale Hielscher offers ultrasonic mixing devices for use in research and full scale processing. Laboratory Research And Development Hielscher ultrasonic laboratory devices are the perfect mixing tool for laboratory R&D. The lab devices are typically used for the ultrasonic mixing of small batches. Hielscher ultrasonic devices offer an accurate parameter control and excellent reproducibility for the preparation of the scale up. This makes it easy to mix different formulations and determine the impact of ultrasonication intensity and duration of sonication. Ultrasonic Mixing In Production The ultrasonic mixing equipment needed for scale up can be determined exactly based on the laboratory test. The table below shows general device recommendations depending on the batch volume or flow rate to be processed.

 **Batch Volume Flow Rate Recommended Devices**

10 to 2000mL 20 to 400mL/min UP200Ht, UP400S

 0.1 to 20L 0.2 to 4L/min UIP1000hd, UIP2000hd

 10 to 100L 2 to 10L/min UIP4000

 n.a. 10 to 100L/min UIP16000

 n.a. larger cluster of UIP16000

Inline Mixing Hielscher ultrasonic mixers are typically installed in-line. The material is pumped into the ultrasonic reactor vessel. There it is exposed to intense ultrasonic cavitation. Inline sonication eliminates by-passing as all particles pass the mixing chamber following a defined path. Therefore, ultrasonication typically shifts the particle size distribution curve rather than widening it. Robust And Easy To Clean An ultrasonic mixing reactor consists of the flow cell and the sonotrode. No bearings are needed. Flow cell reactors (stainless steel) are have simple geometries and can easily be disassembled and cleaned. There are no small orifices or hidden corners. Other Applications Of Ultrasonics For Cement And Concrete The use of Hielscher ultrasonic devices in the preparation of cements and concretes is not limited to the mixing and dispersing of cement premixes or concretes. Ultrasound is a very effective means for the degassing of liquids and slurries. This reduces the number and volume of gas bubbles entrapped in the concrete after hardening. Ultrasonic sieving devices improve the throughput and quality of powder sieving for small particles. Hielscher offers ultrasonically agitated sieves for laboratory and industrial application.

CHAPTER 4
PRECAUTIONS IN USAGE OF NANOMATERIALS

• **Hand protection**

Gloves must always be worn when handling dry nanoparticles or nanoparticles suspension in various solvents. Even though studies have demonstrated that most types of gloves (latex, nitrile, vinyl, etc.) offer a good barrier against most nanoparticles in the powder form, nitrile gloves proved to offer an increased protection when suspension in solvents are used. Recently, the IRSST examined were certain models of disposable gloves made of nitrile, latex and neoprene. A test rig was designed to deform the gloves and simulate opening and closing of the hand in the way that workers do. In addition, a physiological solution mimicking sweat was put inside the gloves while the outside of the glove was placed in contact with a solution containing nanoparticles. It was found that, in certain cases, the integrity of the materials composing them was compromised to the point of allowing nanoparticles to pass through. Double gloving is highly recommended for extensive use and gloves with gauntlets (extended sleeves) reduce risks of skin exposure and therefore offer a better protection. Disposable gloves should be changed routinely or at the first sign of contamination. Even if there are no apparent tears or scratches, the gloves should not be worn for more than two hours at a time and should never be used again once removed.

**• Eye protection**

Safety glasses are mandatory in all Concordia University laboratories and should therefore be the minimal eye protection to be worn when appropriate engineering controls (e.g. nano-cabinet or glove box) is being used. However, safety goggles offer an increased eye protection compared to safety glasses and should be favored when:

* Appropriate engineering controls are deficient or not available;
* A heavy use or large amounts of nanoparticles are being used;
* A suspension of nanoparticles in solvents is being generated and splash or aerosol generation is possible.

• Protective clothing

It was demonstrated the nanoparticles could get through most of regular woven fabrics (e.g. cotton). Therefore, regular lab coats do not offer an appropriate protection from nanoparticles. Impervious disposable clothing, such as Tyvek® lab coats, sleeves and shoe covers should rather be use. Figure 21: Skin exposure due to improper glove use Their fabrics are shown to be impermeable to nanoparticles. If a head scarf is worn by the lab user, a Tyvek® hood should be worn on top of it to avoid possible contamination of the cloth.

**• Respiratory protection**

Respiratory protection should be worn when working with dry nano-powders and when appropriate engineering controls (e.g. nano-safety cabinet) are deficient or not available. Here are descriptions of different types of respirators or masks available on the market along with their intended use; it has to be noted that these respirators cannot be used in areas of low oxygen concentration (i.e. an oxygen deficient atmosphere).

* Surgical masks Those are loose-fitting and disposable masks preventing the release of contaminants from the user to the environment. They are not meant to protect against dust or nanoparticles and should therefore not be used in laboratories using nanoparticles.
* Filtering Face-piece Respirators (FFRs) Those are the most commonly used disposable respiratory masks and they protect user from dusts and aerosols according to their NIOSH certification:
* N : not resistant to oil particles
* R : somewhat resistant to oil particles (8 hours of continuous or intermittent use)
* P : strongly resistant to oil particles(oil proof)
* 95, 99 or 100 : protection rating (%) in NIOSH test

 FFRs do not act as sieves but rather intercept nanoparticles using different types of filtering mechanisms; impaction, interception, diffusion and electrostatic attraction (when electret filter media is used). Once the particles are trapped within the FFRs filter media, they cannot be removed (not a reversible process). Several tests performed by different institutes (e.g. NIOSH, OSHA, IRSST…) demonstrated that typical N95 masks met the NIOSH filtering standards (< 5% penetration for particles less than 100 nm) offering therefore an adequate protection against nanoparticles. However, their efficacy was greatly affected by humidity and breathing rate. The main safety issue observed with the disposable FFRs resides in achieving proper seal around nose and mouth. Improper use or bad work practices by workers (e.g. putting de FFRs on top of the head) often results in a lose fit which allow nanoparticles to enter the mask (Fig. 23). Therefore, their use for work with nanoparticles is not recommended by EHS.

* Half or full-face respirators Half or full-face respirators consist of a rubber (i.e. silicone) mask, worn over the nose and mouth and held in place by adjustable straps which go over and behind the user’s head (Fig. 24). The respirators are fitted with two removable filter cartridges. These units are heavier than the disposable FFRs, but provide far better protection for the wearer, provided that they are fitted properly. Anyone requiring to wear a half or full face respirator must previously get fit-tested by EHS. The respirators require periodic maintenance and cleaning. They are washable and reusable but should not be shared among users. They require filter cartridges adapted to risk

**Storage, Waste Handling, Waste Issues and Spills**

a) Storage Nanoparticles, nanomaterials and nanopowders must be stored in closed and sealed containers at all times, in a cool and well-ventilated area clearly identified (e.g. ‘Nanomaterials storage area – ‘POTENTIALLY TOXIC’). If possible, a lab cabinet restricted to nanomaterials storage only should be dedicated. All containers with nanoparticles in should be clearly identified (e.g. Nanoscale Gold Nanoparticles). If the nanoparticles are received in plastic (Ziploc) bags from the supplier, the bag should be placed in a secondary hard sealable container. Nanoparticles and nanopowders should be stored away from acids, oxidizers and other metals.

 b) Waste Handling There are currently no specific regulation or guidelines for proper disposal of nanoparticles. It is therefore important to handle nanoparticles very cautiously since they are to be considered as toxic. Any waste containing nanoparticles or nanomaterials should never be disposed of in regular garbage and flushed down the drain. Nanoparticles should be disposed of following EHS chemical waste guidelines:

* Any contaminated objects (paper, wipes, tips) should be disposed of as solid chemical waste;
* Any contaminated disposable ppe (e.g. Gloves, disposable lab coats) should be disposed of as solid chemical waste;
* Pure nanomaterials/nanoparticles in the solid (including powder) form should also be disposed of as chemical waste;
* Nanoparticles in solution can be disposed of as liquid chemical waste. All waste containers should be properly labelled, mentioning that they contain nanoparticles and should be properly sealed once full and ready to be picked up.

 c) Spills The potential for inhalation exposure during cleanup will be influenced by the likelihood of nanoparticles becoming airborne, with powder form presenting a greater inhalation potential than the solution forms. Small nanoparticle spills that occur on non-porous surfaces (e.g. linoleum, stainless steel, hardwood flooring) can be cleaned by trained lab personnel. However, if the spill occurs on a porous surface or item (e.g. rug or mat), it might become difficult to fully decontaminate this surface and therefore the item should be discarded along with other nanomaterial waste.

CHAPTER 5
CONCLUSION

* Nano particles filled up the pores reduced CaOH2 compound among hydrates and in turn increased the strength of concrete
* The nano particle formed some chemical bonds at the interfacial zone the new-old concrete become an integral bulk with the treatment of cement mortar modified with nano particles.
* Concrete containing nano-TiO2 had better ductility than normal plain concrete when compared using stress strain relationship graphs.
* Nano-ZrO2 (NZ), Nano-Fe3O4 (NF), Nano TiO2 (NT) and Nano-Al2O3 (NA) out of it nano alumina had showed best mechanical properties with 40% more of compressive strength than normal concrete.
* Out of fibers and nano particles for high strength concrete – nano particles showed increase in 8-30% and where as fibers showed 5-10%
* Out of nano silica and nano titanium oxide – concrete with nano silica had stable structure when studied using scanning electron microscope
* There is an optimal nano-silica amount for the production of UHPC with the lowest porosity, at which the positive effect of the nucleation and the negative influence of the entrapped air can be well balanced.
* Optimum dosage of nano silica being upto 4% of cement then the strength decreases.
* With the binary usage of nano particles that is nano silica and nano clay has showed better mechanical properties compared to single nano particle.
* Nano Silica not only enhance the strength characteristics but also showed better performance for sulphate attack.
* Durability point of view when nanno alumina and nano silica are compared nano alumina showed better durability test results.
* Presence of nano silica increased residual compressive and tensile strengths, and spalling and mass loss are decreased as penetrability increased.
* Nano clay improved durability properties which are measured using chloride penetration, acid attack and sulphate attack when compared with nano alumina and nano iron oxides.
* Carbon nano fibers have decreased the strength of concrete.
* Usage of carbon nanotubes decreased the setting time and showed no sign of bleeding.
* For the purpose of Structural health monitoring concrete with nano fibres performed better where as concrete with nanotubes had significance in improvement of mechanical properties.
* Out of several mixing techniques ultrasonic dispersion technique has produced and concrete with about 30% increase in the compressice strength.
* Through proper dispersion the concrete performs better in fracture properties also.
* There should not be more dosage of CNTs as steel rebars get corroded due to their high concentration.
* By improvising the functional chain of CNTs that is carboxylated nanotubes had increased the one day strength by 30%.

CHAPTER 6
SCOPE FOR FUTURE WORK

* Usage and synthesis of these nanoparticles lead to environmental hazards which have to be taken care and methodologies must be developed with regard to this issue.
* Usage of binary nanoparticles have showed better performance but only one combination is studied therefore it is required to study different combinations and difference ratios for which set will go hand in hand.
* There is an optimum dosage for all the nano particles but only for nano silica the dosage is determined.

CHAPTER 7
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