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Effect of Grinding on Physico-Mechanical Properties of Ultra-Fine Micro-Silica

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ABSTRACT

The present study explains elaborately the use of ground and unground micro-silica with concrete in 5, 10 and 15% as a partial substitute to cement and results were compared with conventional concrete. Micro-Silica was ground in ball mill using iron balls of 15 mm diameter and the sizes of the ground samples were checked after 4, 6 and 8 h of grinding by Particle Size Analyzer (PSA). Concrete specimens containing ground and unground micro-silica were cast in the laboratory and tested for compressive and tensile strengths at different ages of concrete. Permeability test was conducted after subjecting the specimens to 28 days curing. The morphology of the unground micro-silica and micro-silica ground for different time durations were analyzed using Scanning Electron Microscope (SEM). More irregular shapes were observed after 6 h of grinding so the grinding h were restricted to a maximum of 8 h. From the results it was observed that the specimens cast with micro-silica ground for 6 h, added 5% as a partial replacement for cement showed strength improvement up to 30% compared to the specimens cast with unground micro-silica. The resistance to permeability was also higher in concrete specimens cast using micro-silica ground for 6 h as a partial replacement for cement. So, it is inferred from the results that for an optimum grinding period of 6 h, ultra-fine micro-silica can be obtained. Optimum percentage of partial substitution for cement was found to be 5% in terms of strength and durability characteristics of concrete.

Key words: Ultra fine, micro-silica, grinding, compressive strength, tensile strength and permeability

INTRODUCTION

Cement can be partially replaced by a number of mineral admixtures such as fly ash, micro-silica, metakaolin which have certain properties related to that of cement. To reduce the environmental pollution created by the cement industries, the usage of cement must be limited which automatically controls the manufacture of cement. This can be done by using mineral admixtures as partial replacement for cement in concrete. Though the long term strength of concrete made using the mineral admixtures is more or high, their strength when checked initially will be less than that of control concrete. The short term strength of concrete made using mineral admixtures can be improved either by increasing the fineness of mineral admixture or by using chemical activators or by elevating the curing temperature. In this study an attempt has been made to study the improvement in the performance of concrete made by ground silica for three different duration. The mineral admixture micro-silica is used for the present work in its ground state i.e., increasing its fineness by subjecting it to grinding for several hour. Micro-silica is a byproduct obtained from Ferro silicon industries. The particle size of micro-silica is found to be between 100-500 nm.

Collepardi *et al.* (2004) stressed the importance of ultra-fine amorphous colloidal silica over micron sized silica. They found that permeable and durable characteristics of ultrafine particles are much better than micron sized particles and also concluded that 1/3rd-1/4th weight of nano silica compared to micro silica sufficient for satisfying similar strength requirements. Li (2004) found from his study that using nano-silica could improve the compressive strength of concrete which contains higher volume of fly ash even at early age considerably and was achieved by modifying microstructure. Addition of nano-silica was evaluated with different concrete mixes with nano-silica particle sizes ranging from 10-20 nm. The other components such as fly ash, gravel and plasticizer were also used to obtain similar the slump in a similar way as obtained in ordinary concrete. The properties like concrete water permeability and microstructure were studied and found that addition of nano-silica improved the microstructure and reduced water permeability in the hardened concrete. It was due to reaction of nano-silica with Calcium hydroxide and making the interfacial transition zone of aggregates and binding cement paste denser (Ji, 2005).

Balaguru and Chong (2006) highlighted the promising areas in the nano technology applications in concrete construction. They discussed the advantages of using nano sized particles in concrete like molding of components etc., with complex shapes and thermal curing. An overview of carbon nano tubes and its mechanical properties, nano composites and nano sensors for concrete structures is described in this study. They also listed out the chances of using nano sized particles and the challenges to be faced during the manufacturing process. Aiu and Huang (2006) emphasized the difficulties in manufacturing nano-sized cement particles. However, they found that production of nano sized particles through chemical vapor deposition shows good encouragement and other possibility was high tech grinding. The authors specified the significance of developing special organic and in organic admixtures that are to be mixed with concrete to control heat of hydration and setting. Though these two aspects are risky and tough to deal with, the authors believe that it is worth taking such risks. Mann (2006) mentioned in his report that the compressive and tensile strength of concrete can be improved considerably by the addition of even a very small amount of carbon nano tube say 1% by weight.

Song *et al.* (2007) studied the effectiveness of using nano particles on compressive strength of concrete and compared the results with concrete made with commercially available Ordinary Portland Cement (OPC). They used Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD) to analyse the morphology and structure of synthesized C_3S and C_2S components and found that both the components were present in the synthesized cement. It is also found that hydration rate of nano cement was faster than OPC. While comparing the compressive strength, they found that strength of nano cement was less than strength of OPC and was justified with reasons like aggregation of particles, faster rate of hydration, high water to cement ratio and lack of gypsum etc. Liu *et al.* (2007) developed nano and micro electrical mechanical systems sensors to be used in construction industry meant for studying the performance of either material or structure. These sensors were used for monitoring and controlling the environment condition. He also highlighted that the size of the sensor was the advantage and ranges between 10^{-9} and 10^{-5} m, whereas micro sensor ranges between 10^{-4} and 10^{-2} m.

Ge and Gao (2008) examined and documented the applications of nano technology based products and hoped that it will improve the overall competitiveness of construction industry. They focused on the research areas like structural composites with light in weight and at the same time better strength, relatively lesser maintenance, deriving of better properties, reduction in thermal rate of fire retardant etc. Early age concrete properties like moisture, temperature, relative humidity and early age strength development, concrete corrosion and cracking can be monitored by using smart aggregate as a low cost piezoceramic-based multi-functional device (Saafi and Romine, 2005; Song *et al*., 2008).

Particle size distribution (grading of mix and particle packing) governs the flow properties and workability in fresh concrete, strength and durability in hardened concrete (Husken and Brouwers, 2008). One possibility of arriving improvised packing of particles is to increase the solid size range with inclusion of particles preferably below 300 nm sizes. At present silica flavor, silica fume and nano-silica are the materials readily available for the use and these products are not feasible in construction industry because of its very high cost (Husken and Brouwers, 2008; Dunster, 2009). Nano-silica was developed using olivine and correspondingly mix design was done for self compacting concrete to take care of particles with nano size (Lieftink, 1997; Jonckbloedt, 1997; Brouwers and Radix, 2005; Hunger and Brouwers, 2008; Lazaro and Brouwers, 2010). It is also found from the earlier studies that adding 1 kg of silica fume results in reducing 4 kg of cement in a mix and will further be reduced if nano-silica used and is due to high surface area (Husken and Brouwers, 2008; Dunster, 2009; Qing *et al*., 2007; Senff *et al*., 2009). Hence, in the present work, the results of the study carried out to investigate the effectiveness of grinding on physico-mechanical properties of concrete cubes cast with micro-silica are presented. The present research work aims at developing ultra-fine sized particles from micro-silica to use as mineral admixtures in concrete and to study the physical and mechanical properties of concrete when ultra fine composites are mixed with cement in different percentages.

Significance of the present research work: Several research works are going on all over the world to study the use of nano silica to be used as admixture in concrete with very little amount and very good improvement in strength characteristics. Though nano silica is readily available in market, the cost incurred to procure such materials is relatively very high and hence use of locally produced ultra fine silica by a conventional method of grinding by a ball mill is a good option. Hence, it is tried in the present work. It is believed that making of ultrafine micro-silica can very well be obtained by means of grinding to an optimal duration which improves compressive and tensile strength considerably and also economically feasible compared to commercially available one.

EXPERIMENTAL INVESTIGATIONS

Material properties: Materials used for structural concrete of grade M25 (with a characteristic compressive strength of 25 N mm^{-2}) are discussed here. The cement used for the present study was 43 grade Ordinary Portland cement (with a characteristic compressive strength of 43 N mm^{-2}) with a specific gravity of 3.12. River sand was used as the fine aggregate and coarse aggregate used was broken stone of 12.5 and 20 mm size. The specific gravity of fine and coarse aggregates was 2.63 and 2.65, respectively. The mineral admixture added with cement was micro-silica which is manufactured by M/s Oriental Treim PVT Ltd., Navi Mumbai, India and its chemical properties tested in the laboratory are given in Table 1.

Table 1: Chemical and physical properties of micro-silica

Parameters	Test value	As per ASTM C1240-00
SiO_2 (%)	90.3	85 (min)
Moisture content (%)	0.6	3.0 (max)
Carbon (%)	0.8	2.5 (max)
Retained on 45 micron (%)	0.4	10 (max)
Bulk density (kg mm^{-3})	640.0	500-700

Specimen details: The Concrete mix design was made using IS method for M25 grade concrete to obtain a characteristic compressive strength of 25 N mm^{-2} (Target mean strength of 33.75 N mm^{-2}) and based on the design, the mix proportion arrived was 1: 1.2: 2.4 [Cement: Fine Aggregate: Coarse Aggregate] with 0.43 as its water cement ratio. Specimens were prepared separately to study the strength characteristics such as compressive strength and tensile strength. Concrete cubes of size 100×100×100 mm were cast for testing compressive strength, cylinders of size 150×300 mm were cast to study the tensile strength. Specimens were subjected to desired periods of curing as 7, 14 and 28 days before testing. Compressive strength of cube specimens was tested in a 300 kN capacity automatic compression testing machine. Specimens were subjected to stress controlled loading. Tension test on cylindrical specimens were also tested in same compression testing machine by split tension test.

RESULTS AND DISCUSSION

General: The variations of mechanical properties viz., compressive strength and tensile strength of specimens at different ages of concrete and permeability characteristics are discussed in the following paragraphs. A total of seven combinations were prepared for the present research work to assess the above three parameters. The combinations are: conventional concrete, concrete with unground micro-silica and concrete ground micro-silica. In the unground and ground categories micro-silica with 5, 10 and 15% substituted for cement. Grinding of micro-silica was done for three different durations in 4, 6 and 8 h. From the above parameters, the effect of unground and ground micro-silica and grinding duration on strength and permeability characteristics were studied.

Effect of unground and ground micro-silica on strength characteristics: The variation of compressive strength of concrete with respect to age of concrete due to the replacement of cement with different percentages of micro-silica at different ages of concrete is shown in Fig. 1. The results of tests conducted by Bouzoubaa *et al.* (1997) reported the behavior of concrete cast with fly ash ground for 2, 4 and 10 h and their results show that water requirement of fly ashes first decreased for the grinding period between 2 and 4 h and increased after 4 up to 10 h due the increase in the irregular-shaped particles. They also showed that the same trend was followed considering the strength activity concrete made with ground fly ash which showed that the 7 day strength of concrete cubes cast with fly ash ground for 2-4 h was higher than that of the cubes cast with fly ash ground after 4 h. There was not much change in the 28 day strength. From the results of the present work it is inferred that at the age of 28 days conventional concrete gives a compressive strength of 36.98 N mm^{-2} which is 10% more than the target mean strength of 33.75 N mm^{-2}. Hence, it is ensured that the quality control during preparation of concrete is up to the desired standard. Compressive strength of concrete increases with the age of concrete irrespective

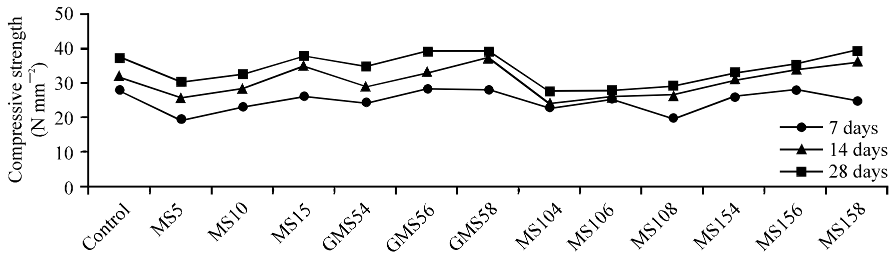

Fig. 1: Compressive strength of control concrete, unground, ground silica fume added concrete. MS: Micro-Silica, MS5: Concrete with 5% unground micro-silica, MS10: Concrete with 10% unground micro-silica, MS15: Concrete with 15% unground micro-silica, GMS: Ground micro-silica, GMS54: Concrete with 5% ground micro-silica for 4 h, GMS56: Concrete with 5% ground micro-silica for 6 h, GMS58: Concrete with 5% ground micro-silica for 8 h, GMS104: Concrete with 10% ground micro-silica for 4 h, GMS106: Concrete with 10% ground micro-silica for 6 h, GMS108: Concrete with 10% ground micro-silica for 8 h, GMS154: Concrete with 15% ground micro-silica for 4 h, GMS156: Concrete with 15% ground micro-silica for 6 h and GMS158: Concrete with 15% ground micro-silica for 8 h

micro-silica percentage added and whether it is ground or unground. For addition of unground and ground micro-silica, characteristic compressive strength of 25 N mm^{-2} was achieved even at the age of 14 days itself. At the age of 28 days the compressive strength micro-silica admixed concrete exceeded the value of characteristic compressive strength to an extent of 25-50% for unground condition. Similar trend was observed for ground micro-silica with 5 and 15% addition, whereas for concrete with 10% ground micro-silica this value ranges only from 9-15%. Grinding of micro-silica makes changes in the microstructure of its particles and compressive strength was considerably increased after grinding. The effect of grinding for 5% micro-silica added concrete on compressive strength increased to 14, 30 and 29% for 4, 6 and 8 h duration, respectively. There is a reverse trend for 10% micro-silica added concrete in compressive strength and reduction of 14, 15 and 10% compared to unground micro-silica. For concrete with 15% added micro-silica there is a reduction of 13 and 2% in compressive strength for 4 h grinding and 8 h of grinding. For 6 h of the compressive strength increases to 7%. Hence, it is inferred from the results that 6 h of grinding is effective in modifying the microstructure of micro-silica added concrete. It is also understood that ground micro-silica with 5% addition gives better strength. The variation of tensile strength of conventional concrete, concrete with unground and ground micro-silica is shown in Fig. 2. Similar trend as observed in compressive strength is seen here also. But none of the tensile strength values of ground micro-silica added concrete is less than the unground micro-silica added concrete except for 15% micro-silica added concrete. Even in tensile strength 6 h of grinding of micro-silica found to be effective.

Effect of grinding on permeability characteristics: From the results of Teng *et al.* (2013) it is understood that the use of ultra fine mineral admixtures improved the durability properties of the concrete even when tested after a short period of 3 days curing. The effect of grinding on permeability characteristics in terms of sorptivity, an ability to permit water through the bottom surface of concrete called as capillary suction. Figure 3-6 depicts the values of sorptivity with

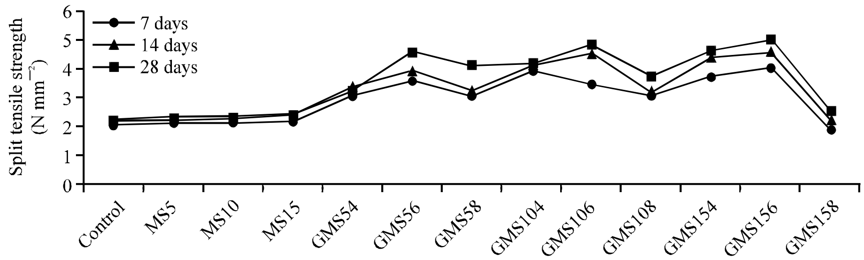

Fig. 2: Tensile strength of control concrete, unground, ground silica fume added concrete. MS: Micro-Silica, MS5: Concrete with 5% unground micro-silica, MS10: Concrete with 10% unground micro-silica, MS15: Concrete with 15% unground micro-silica, GMS: Ground micro-silica, GMS54: Concrete with 5% ground micro-silica for 4 h, GMS56: Concrete with 5% ground micro-silica for 6 h, GMS58: Concrete with 5% ground micro-silica for 8 h, GMS104: Concrete with 10% ground micro-silica for 4 h, GMS106: Concrete with 10% ground micro-silica for 6 h, GMS108: Concrete with 10% ground micro-silica for 8 h, GMS154: Concrete with 15% ground micro-silica for 4 h, GMS156: Concrete with 15% ground micro-silica for 6 h and GMS158: Concrete with 15% ground micro-silica for 8 h

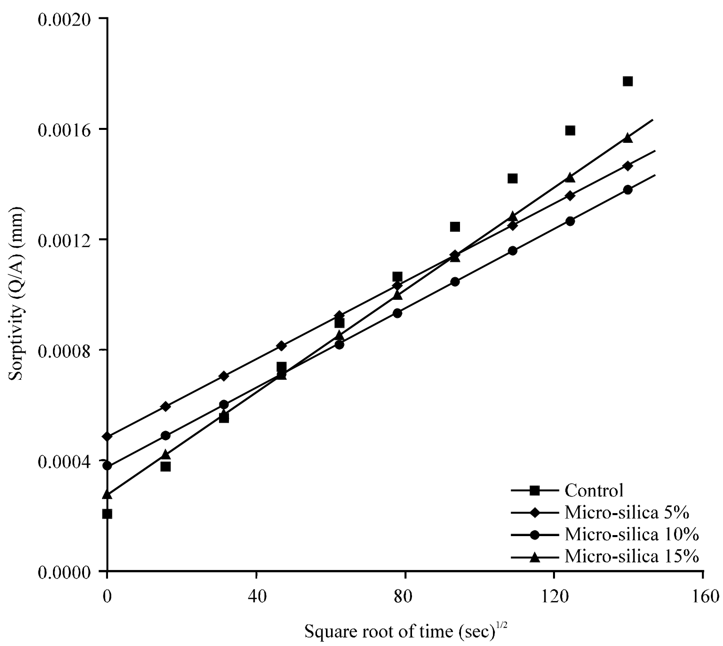

Fig. 3: Sorptivity for control concrete, unground silica fume added concrete

respect to time elapsed. By taking the slope of the line of best fit for each and every cases sorption coefficient of respective concrete was obtained. From Fig. 3 it is inferred that sorption coefficients are 1.11×10^{-5}, 6.99×10^{-6}, 7.95×10^{-6} and 9.18×10^{-6}, respectively for control concrete, concrete with unground micro-silica with 5, 10 and 15%. It is understood that capillary suction has higher value for control concrete and addition of 5% micro-silica makes heavy impact on reduction of capillary suction means that it has got higher resistance. Microstructure was effectively modified for 5%

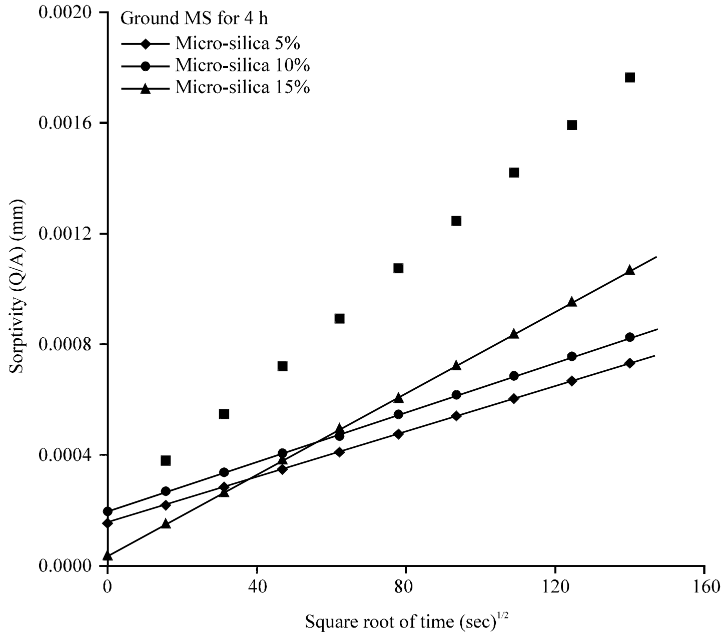

Fig. 4: Sorptivity for ground silica fume concrete for 4 h

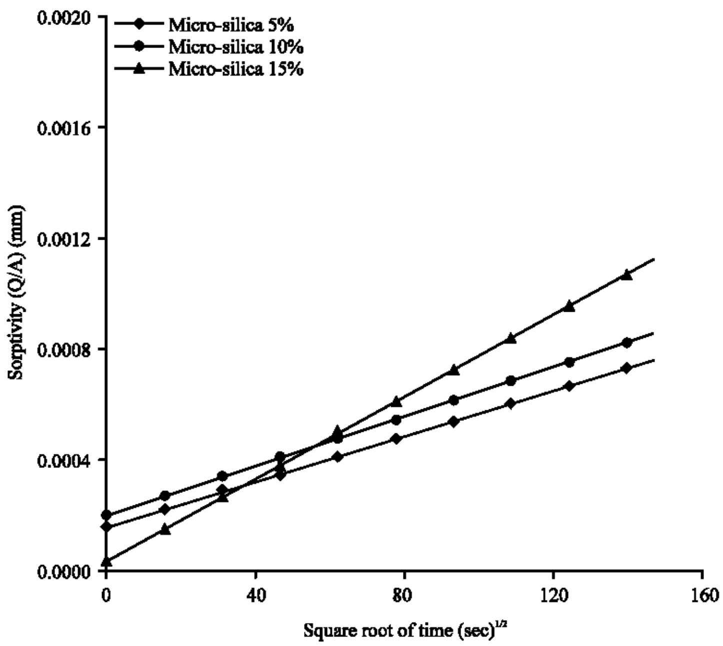

Fig. 5: Sorptivity for ground silica fume concrete for 6 h

addition and further addition of micro-silica to 10 or 15% shows reverse trend but very much less than control concrete. For ground micro-silica with 4 h grinding gives sorption coefficients of 6.39×10^{-6}, 7.15×10^{-6} and 4.86×10^{-6} for 5, 10 and 15% addition of micro-silica. The rate of reduction in sorption coefficient for the above case compared with unground case is 10% for concrete added with 5 and 10% micro-silica and 52% for concrete with 15% micro-silica. Further grinding of

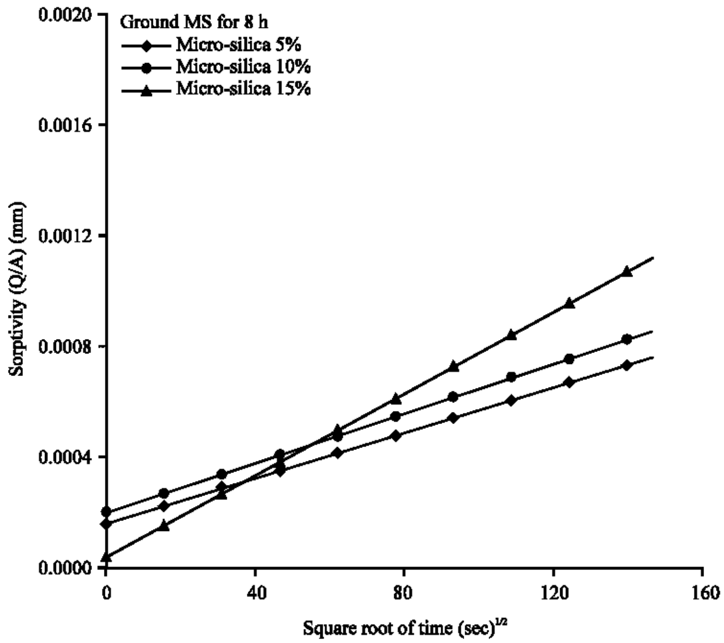

Fig. 6: Sorptivity for ground silica fume concrete for 8 h

materials to 6 h was found to be very much effective for which the sorption coefficients were 4.09×10^{-6}, 4.43×10^{-6} and 4.80×10^{-6} for 5, 10 and 15% addition of micro-silica. The rate of reduction in sorption coefficients was observed to be 58, 62 and 52% than for 5, 10 and 15% addition of unground micro-silica. This is really an encouraging trend. Though the values of sorption coefficients are less for 8 h of grinding compared to unground micro-silica for all the cases it shows a reverse trend with results of 6 h grinding (i.e., sorption coefficient of 8 h ground material was higher than 6 h ground material.

Hence, it is assessed from the results that 6 h of grinding gives better results in terms of offering resistance to permeability.

Effect of grinding on microstructure: Microstructure of micro-silica under unground condition and after grinding for specified durations was analyzed by Scanning Electron Microscope to understand the effect of grinding. Figure 7-10 depict the morphology of micro-silica for unground and ground conditions. The results indicate that the shape of the silica fume particles before grinding were spherical and after subjecting them to grinding the shapes become irregular or the size of spherical particles got reduced. When it is subjected to long periods of grinding till 6 h the spherical shape of the particles may even get reduced but for 8 h of grinding it has got less significance. It is evident from the work of Paya *et al.* (1996) that the mineral admixtures on subjecting to grinding continuously showed different shape morphologies. The authors used fly ash as the mineral admixture and the SEM images of fly ash ground for different durations of time showed shell shaped and irregular solid fragments after grinding indicating that grinding process increases poor shape particles. They also reported that increase in specific gravity was achieved and the bulk specific gravity for the fly ash decreased due to loss of optimum spherical shape

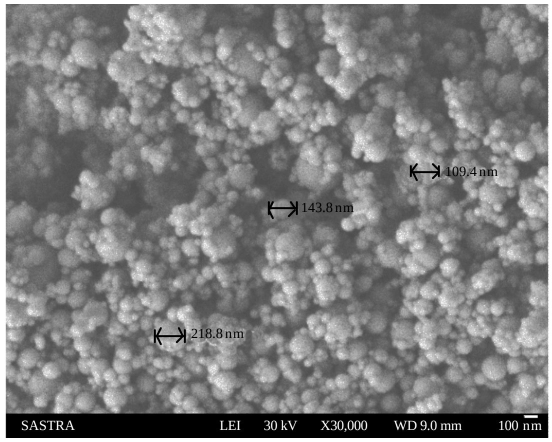

Fig. 7: Morphology of un-ground micro-silica

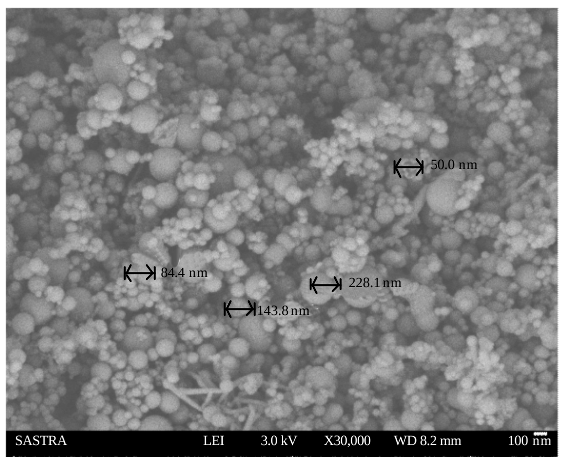

Fig. 8: Morphology of ground micro-silica for 4 h using SEM

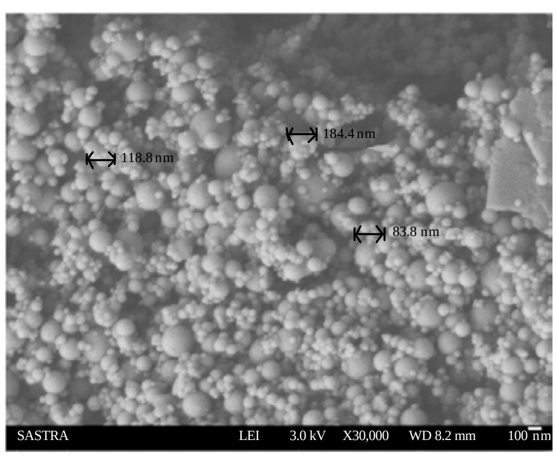

Fig. 9: Morphology of ground micro-silica for 6 h using SEM

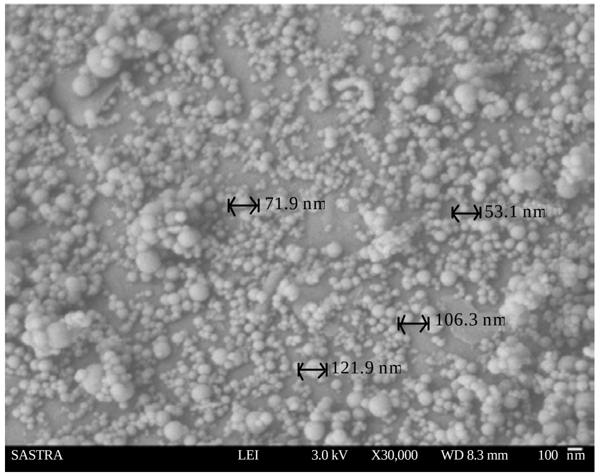

Fig. 10: Morphology of ground micro-silica for 8 h using SEM

morphology. Considering these aspects in the present work the grinding process was restricted to a maximum of 8 h as the loss of spherical shape was more even after 6 h of grinding. It is also to be noted that simply increasing the duration of grinding will lead to uneconomical project.

CONCLUSION

Experimental investigations were carried out on normal strength concrete added with unground and ground micro-silica with different duration to understand the effect of grinding on strength and permeability characteristics. From the experiments conducted, following conclusions were arrived:

- On observing the morphology of the micro-silica, considerable changes were noted as the size of the particles kept changing from regular shapes to irregular shapes. More irregular shapes were noted on ground micro-silica for 6 h
- The compressive strength of the concrete cubes cast with addition of ground micro-silica was increased to an extent of 30% when compared to concrete with unground micro-silica for 6 h of grinding. The tensile strength results also indicate that cylinders cast with ground micro-silica for 6 h showing improved strength when compared with other specimens
- Higher rate of resistance to permeability was observed in the ground micro-silica with 6 h grinding. Hence, it is concluded that grinding for 6 h is an optimal duration to get ultra fine micro-silica for better performance of concrete in terms physical and mechanical properties

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