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THE ROLE OF NANO-SIO_2 IN THE MECHANICAL PROPERTIES OF RHA COMPOSITE CEMENT MORTARS

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Abstract- This paper presents a study on the properties of rice husk ash (RHA) cement composite mortars incorporating nano-SiO_2. Different amounts of nano-SiO_2 (0%, 1%, 3% and 5%) were incorporated into the mortars with 20% replacement of RHA. The compressive and flexural strengths of mortars were tested at 7, 28, 60 and 90 days. The water absorption test was carried out at 28 days. The drying shrinkage of mortars was measured up to the age of 42 days. Scanning electron microscopy (SEM) observation was conducted to evaluate the effect of nano-SiO_2 on microstructure of cement paste containing RHA. Incorporating nano-SiO_2 into RHA mortars significantly improved the mechanical strength and water absorption properties of the products. Nano-SiO_2 increased the drying shrinkage of RHA mortars. SEM analysis showed that nano-SiO_2 improved the microstructure of RHA pastes.
Key Words- Rice Husk Ash, Nano-SiO_2, Mechanical Properties, Shrinkage, Water Absorption, SEM

Introduction

The use of nano particles in developing materials has been introduced in many fields [1]. The extremely fine nano particles strongly affect their physical and chemical properties and integrate with existing cement-based building materials which lead to formation of new products with some outstanding properties [1, 2]. Among the nano particles, nano silica was used to improve the properties of cement based materials due to its pozzolanic activity. The excellent mechanical properties and microstructure of cement composite with nano-SiO_2 were also reported [3]. Li [4] reported that incorporating nano particles into high-volume fly ash concrete increase significantly the initial pozzolanic activity of fly ash. Nano-SiO2 enhances the short-term and Long-term strength of high-volume fly ash concrete. According to Lin [5] nano-SiO_2 particles improve the negative influences (caused by sludge-fly ash on the early strength) of mortar and based on the research of Jo *et al*. [6] nano-SiO_2 increases the compressive and flexural strength of concrete and mortar. Nano particles possess high pozzolanic activity because their rate of pozzolanic reaction is proportional to the amount of specific surface. These particles consume calcium hydroxide (CH) in the interfacial transition zone (between hardened cement paste and aggregates) and produce hydrated calcium silicate (CSH) which increases the strength of cement paste [7]. Nano-SiO_2 particles can fill the ultra fine pores in cement matrix due to their scale size. This physical effect reduces the porosity of transition zone in the fresh concrete which strengthens the bond between matrix and aggregates, and improves the cement microstructure and properties. The uniformly dispersed nano-SiO_2 particles generate a large number of nucleation sites for the precipitation of the hydrated products which accelerates cement hydration [8]. The rice husk ash (RHA) was added to the concrete mixtures for substituting more expensive Portland cement and reducing the construction cost. Regarding to the properties and amount of SiO_2, rice husk ash is similar to silica fume [9]. Hence it is not just a cheap alternative but a well burn, a well-ground RHA with most of its silica in an amorphous form and with enough specific surfaces is very active and considerably improves the strength and durability of concrete [10]. The concrete containing RHA has a higher sulfate and acid resistance and lower chloride permeability [11]. The main aim of this research is to study the effect of nano-SiO_2 (as an innovative approach) on mechanical and microstructural characteristics of RHA mortar.

Materials and Methods

In this study, ordinary Portland cement type I, standard graded sand, rice husk ash, nano-SiO_2 particles and tap water were used.

Rice Husk Ash

RHA has been used in many countries as a low cost concrete admixture because of its role as a pozzolan [12]. It has been also used for producing high performance concrete (HPC) or achieving other desire properties. RHA used in this experiment contained 92.1% SiO_2 with average particle size of 15.83 μm. The chemical compositions and physical properties of RHA and Portland cement are listed in Table 1.

Superplastisizer

Superplastisizers are widely used as additives in concrete with high rheological requirements. The use of superplastisizers allows the reduction of water / cement ratio (w/c) of mortar and concrete, without significant change in their flow properties. The sodium salts of formaldehyde condensates disperse the cement particles by electrostatic repulsion which results from the adsorption on cement surface.

Due to high specific surface of nano-SiO_2 (which needs more water for complete hydration) workability of mortar decreases as the nano-SiO_2 content increases. In order to achieve desire fluidity and better dispersion of nano particles, polycarboxylate ether based superplastisizer was incorporated into all mixes. The content of superplastisizer was adjusted for each mixture to keep constant the fluidity of mortars.

Nano-SiO_2

The percentage of calcium hydroxide consumed by a pozzolan indicates the pozzolanic activity index. Among available pozzolanic materials, nano-SiO_2 has the highest pozzolanic activity index that explains its high purity and high specific surface. Basic material properties of nano-SiO_2 are given in Table 2.

Aggregates

Natural river sand was used with the fraction of sand passing through 1.18 mm sieve and retaining on 0.2mm (confirming to ASTM C778) [13]. The specific gravity of sand was 2.61 gr/cm^3 and the absorption capacity was 3.4%.

Mix Proportions

Ten different combinations were cast (Table 3). Mixtures NS0 to NS9 were used to find out the optimum content of nano-SiO_2 in ordinary cement mortar. Mixtures RHN0 to RHN5 were made to study the effect of nano-SiO_2 particles on properties of cement mortar containing RHA. The amount of RHA replacement in mortar was 20% by weight of cement which is an acceptable range and is used frequently. The water/binder ratio for all mixtures was 0.5 where the binder weight is the total weight of cement, RHA and nano-SiO_2. The sand/cement ratio was 2.75 for all the mixtures.

Test Procedure

In order to achieve desire properties, it is essential to disperse nano-SiO_2 particles uniformly. Accordingly mixing was carried out in a rotary mixer as follows:

1. The nano-SiO_2 particles were stirred with the 90% of mixing water at high speed and 1 min.
2. Dry mixed cement and RHA was added to the mixture. Then the mixer was allowed to run for 1 min at medium speed.
3. The sand was gradually added at 30s while the mixer was running at medium speed.
4. The superplastisizer and remaining water were added and stirred at high speed for 30s.
5. The mixture was allowed to rest for 90s. Then mixing was continued for another 1 min at high speed.

At the end of mixing, the samples were cast into the 50×50×50 mm cubes for compressive and water absorption tests and into 50×50×200 mm prismatic specimens for flexural and shrinkage tests. The compressive samples were placed in two layers. Each layer was temped 32 times in about 10s following the procedure of ASTM C 109 [14]. The flexural samples also were placed in two layers and each layer was tamped 12 times in 4 rounds following the procedure of ASTM C 348 [15]. After 24 hours the specimens were removed from the moulds and cured in water at 23±2 $^{\circ C}$ for 7, 28, 60 and 90 days. The samples were tested using hydraulic testing machine under load control at 1350 N/sec for compressive test and 44N/ sec for flexural test. The absorption test was carried out on two 50 mm cubes. Saturated surface dry specimens were kept in an oven at 110$^{\circ C}$ for 72 h. After measuring the initial weight, specimens were immersed in water for 72h. Then the final weight was measured and the final absorption was reported to assess the mortar permeability.

Results and Discussion

Compressive Strength

The compressive strength of cement mortars are shown in Fig. 1. It can be seen that the compressive strength of ordinary cement mortar gradually increased with an increase in the amount of nano-SiO_2. It is obvious that increase in the nano-SiO_2 content beyond 7% did not change the compressive strength considerably. It is clear that the nano-SiO_2 particles are more effective in developing compressive strength than that of RHA. According to the results integrating nano-SiO_2 with RHA in mortar improved the compressive strength of the products.

Flexural Strength

Fig. 2 reveals the flexural strength of different mortars at four ages. It shows the improvement of flexural strength of ordinary cement mortars by nano-SiO_2. The greatest increase among all ages was observed in mixture NS5. High amount of nano-SiO_2 (especially in excess of 7% in ordinary mortar) had an adverse effect on the flexural strength. Poor dispersion of nano-SiO_2 particles in cement matrix is due to self desiccation and formation of micro cracks.

The results show that nano-SiO_2 particles are more effective in developing flexural strength than RHA. Incorporating nano-SiO_2 into cement mortars containing RHA cause further increase in mechanical strength of the products. The optimum nano-SiO_2 content in ordinary and RHA mortars were 7% and 3% respectively. Two fundamental mechanisms influence the strength enhancement in presence of nano-SiO_2:

1. Matrix densification and paste-aggregate interfacial zone refinement.
2. Reduction in the content of $Ca(OH)_2$.

The first strengthening mechanism is the filler effect which is one of the important factors for the development of dense concrete/mortar with very high strength. The ultra fine particles of nano-SiO_2 fills the interstitial spaces of the hardened microstructure skeleton of cement paste and increase its density and strength.
Rice husk ash reduces the volume of large pores, increases the mortar strength, because the size ratio between filler and the aggregates is one of the main parameters that affects strongly the strength. The results showed that integrating nano-SiO_2 with RHA in mortar improves more effectively the mechanical strength. This is due to increase in packing density of binder.
Kaufmann et al [16] claimed that packing density of a powder is improved by adding a fine powder to a coarse one. Integrating nano-SiO_2 particles with RHA in cement composite mortar improve the packing density and reduce the porosity of cement matrix which play an important part in mechanical strength.
It was reported that the microstructure of the transition zone between aggregates and cement paste strongly influence the strength and durability of concrete. Presence of nano-SiO_2 particles in mortar reduces the wall effect in the transition zone between the paste and the aggregates, and strengthens the weaker zone due to the higher bond between two phases [5, 17 and 18]
The second strengthening mechanism is the pozzolanic activity. Pozzolans are defined as siliceous or siliceous and aluminous materials which in themselves possess little or no cementing property but in finely dispersed form in the presence of moisture chemically react with calcium hydroxide at ordinary temperature to form compound possessing cementitious properties [19].
Two major products of cement hydration are calcium silicate hydrate (CSH) and calcium hydroxide (CH) respectively. CSH is produced by hydration of C_3S and C_2S, plays a vital role in mechanical characteristics of cement paste. CH is formed by hydration of cement, has low cementing property and contains about 20-25% of the hydration products. Calcium hydrates are morphologically weak and brittle therefore cracks can easily propagate through the regions especially at the aggregate-paste interface. Nano-SiO_2 particles react with CH rapidly and produce calcium silicate hydrate with cementitious properties which increase the strength of concrete/mortar. Nano-SiO_2 and RHA are pozzolanic materials, but the pozzolanic activity of the first one is greater than that of the second. Because of high specific surface, Nano-SiO_2 serves as additional nucleation sites for the precipitation of the hydrated products which accelerates the chemical reactions [20 & 21].

Water Absorption

The water absorption test results are given in Table 4. Mortars with nano-SiO_2 showed lower absorption values. The water absorption values decreased as the nano-SiO_2 content increased. It is clear that nano-SiO_2 is more effective in reduction of permeability than RHA. Incorporating nano-SiO_2 into RHA mortar had a positive effect on water absorption of the products. The water absorption of the RHA mortars significantly decreased when nano-SiO_2 particles were incorporated. The impermeability enhancement attributed to two phenomena:

1. Nano-SiO_2 particles generated a large number of the nucleation sites for precipitation of the hydration products and induced more homogenous distribution of CSH and hence less pore structure.
2. Nano-SiO_2 particles blocked the passages connecting capillary pores and water channels in cement paste.

Shrinkage

Shrinkage is a common phenomenon in every cementitous product due to contraction of total mass upon loss of moisture. It is sometimes accompanied with development of cracks especially in members that surface area to volume ratio is high. These cracks serve as conduits for salt and water. The saline solution comes in contact with reinforcing steel and promotes corrosion. Corrosion causes expansion of steel and inevitably pop-outs occur in the concrete cover, thereby reducing the strength and service life of the concrete. To study the influence of nano-SiO_2 on the drying shrinkage of ordinary and RHA mortars, prismatic specimens with 50×50×200 mm dimensions were prepared. The first measurement was taken using a length comparator with a precision of 2µm after 24 h of mixing and then it is followed at 3, 7, 14, 21, 28, 35 and 42 days of ages. The specimens were cured in the laboratory environment with the average temperature of 26±3 °C. The shrinkage behavior of mortars containing nano-SiO_2 is presented in Fig. 3. The drying shrinkage of mortars with nano-SiO_2 is higher than that of control mortar and it augments with increase in nano-SiO_2 content. The influence of nano-SiO_2 on the drying shrinkage of RHA mortar is shown in Fig. 3(b). Incorporating nano-SiO_2 into RHA mortar increases the drying shrinkage of the products. The increase in the drying shrinkage of mortar containing nano-SiO_2 is mainly because of pore size refinement and increase in mesopores, which is directly related to the shrinkage caused by self desiccation.

Microstructure

The physical properties of concrete, (especially strength and permeability), depend up on its pore structure. To evaluate the effect of supplementary cementing materials on the strength and durability of the products, scanning electron microscopy (SEM) analysis is carried out.
Several researches were devoted to evaluate the influence of nano-SiO_2 on microstructure of plain cement mortar. [3, 7 & 8]. The results showed that nano-SiO_2 particles formed compact texture of the hydrated products and decreased the size of crystals (e.g. $Ca(OH)_2$). The effect of nano-SiO_2 on the microstructure of RHA paste was evaluated. Cement pastes containing 20% RHA with 0% and 3% nano-SiO_2 with the same water/ cement ratio were prepared. Samples were cured

under standard condition until they were tested. The RHA pastes microstructures with 0 and 3% nano-SiO_2 are presented in figures 4 & 5. RHA pastes show a porous microstructure, but when 3% nano-SiO_2 by weight of cement was added, it changed significantly. The nano-SiO_2 particles improved dense and compact RHA pastes microstructures to homogenous distributed hydrated products.

Conclusions

Important improvement was observed in compressive and flexural strength of ordinary cement mortars by adding nano-SiO_2. The compressive and flexural strength of RHA mortars increased with the incorporation of nano-SiO_2 particles. According to the results there was a significant deference between the water absorption of the mortar with and without nano-SiO_2. Nano-SiO_2 particles decreased the water absorption of the cement mortars by pore filling and pozzolanic effects. Nano-SiO_2 particles were more effective in reduction of permeability than RHA. Integrating nano-SiO_2 with RHA in mortar had a positive effect on water absorption of the products. Nano-SiO_2 had a remarkable impact on the drying shrinkage of mortars. Ordinary mortar containing nano-SiO_2 showed higher shrinkage, comparing with the control mortar. This effect was more for larger amount of nano-SiO_2. Nano-SiO_2 increased the drying shrinkage of RHA mortars as well. Incorporating nano-SiO_2 into RHA cement paste, improved compact form of microstructure.

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