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INFLUENCE OF POLYPROPYLENE FIBERS ON THE PERFORMANCE OF NANO-SIO_2-INCORPORATED MORTAR*

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Abstract– Although Polypropylene (PP) fibers have advantageous characteristics, the weak bond with the cement matrix as a result of their smooth surface and chemical inertness remains a large limitation. It has been demonstrated that the fiber-matrix bond strongly affects the ability of fibers to stabilize crack propagation in the matrix. As the bond between fiber and matrix is mainly mechanical, it seems that incorporating nano-SiO_2 into fiber reinforced cement composites provides a better bond with the matrix through pore refinement and better distribution of the hydration products. Hence, in this paper an effort was made to study the effect of PP fibers on the mechanical and physical properties of mortars incorporating nano-SiO_2. Four fiber volume fractions, 0, 0.1%, 0.3% and 0.5% were considered. Compressive and flexural strength, water absorption and shrinkage of mortars were reported. SEM observation was also conducted to evaluate the effect of nano-SiO_2 on the microstructure of cement paste. Results showed that nano-SiO_2 significantly improved the mechanical and water absorption characteristics of mortars. It was observed that the microstructural characteristics of cement paste can be effectively improved by incorporating nano-SiO_2, as the presence of nano-SiO_2 enhanced the PP fibers effectiveness in strengthening mortar properties.

Keywords– Cement mortar, nano-SiO_2, silica fume, mechanical properties, shrinkage, SEM

1. INTRODUCTION

It seems that in the near future significant developments will be observed in concrete technology thanks to the introduction of nano-SiO_2 with high purity and a finer particle size. Studies have shown that the application of nano-SiO_2 into the production of mortar and concrete can lead to improvement in compressive strength, flexural behavior and abrasion resistance [1-4]. Therefore nano-SiO_2 can be applied in the production of high performance concrete (HPC), which has gradually been replacing normal strength concrete. The rate of pozzolanic reaction is proportional to the amount of surface available for reaction and owing to the high specific surface of nano particles. They possess high pozzolanic activity and consume calcium hydroxide (CH) which arrays in the interfacial transition zone between hardened cement paste and aggregates, and produce hydrated calcium silicate (CSH) which enhances the strength of cement paste [5]. In addition, due to the nano scale size of the particles, nano-SiO_2 can fill the ultra fine pores in the cement matrix. This physical effect of the finer grains leads to a reduction in the porosity of the transition zone in the fresh concrete. This mechanism strengthens the bond between the matrix and the aggregates and improves the concrete properties. Furthermore, it has been found that when the small particles of nano-SiO_2 uniformly disperse in the paste, due to their high activity, a large number of nucleation sites for the precipitation of the hydration products are generated, accelerating cement hydration[6].

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Polypropylene (PP) fibers have been widely used for the reinforcement of cementitious materials to improve the toughness and energy absorption capability of the matrix [7-8]. They were found to be extremely effective in reducing free plastic shrinkage, in retarding first crack appearance and in controlling crack development [9-10]. Although the effectiveness of PP fibers in shrinkage cracking, impact resistance and the ductility of cement matrices has been proved by many researchers, the effect of PP fibers on compressive and flexural strength is not quite clear [11]. Studies have shown that there can be little or no chemical adhesion between the fiber and matrix as a result of their chemical inertness [12]. It seems that the smooth surface of the PP fibers intensifies this effect. Moreover, it has been suggested that the presence of PP fibers in cement paste results in the formation of a water film at the interface of the fiber and matrix called wall effect. Due to the greater mobility of calcium ions in a water environment, portlandite (calcium hydroxide) macro crystals can easily grow and make the transition zone more porous [13]. This phenomenon has a negative impact on the bond between fiber and matrix. It is clear that in order to utilize the maximum strength of the fiber and improve the composite properties, it is essential to enhance the interfacial bond of pp fibers. It seems that the physical and chemical effects of nano particles can be useful in the reduction of the wall effect between the fiber and matrix. Accordingly, the present study focuses on the effect of PP fibers on the mechanical and physical properties of cement composite mortar with nano-SiO_2. The results of this investigation are important for clarifying the effect of interface densification on the effectiveness of fiber reinforcement.

2. EXPERIMENTAL PROCEDURE

a) Materials and mix proportions

The cement used in all mortars was ordinary Portland cement which corresponds to ASTM type 1. The chemical analysis of Portland cement is shown in Table 1. Nano-SiO_2 in liquid form with an average particle size of 50 nm was used in this study. In order to achieve the desired fluidity and better dispersion of nano particles, a polycarboxylate ether based superplastisizer was utilized. The content of superplastisizer was adjusted for each mixture to keep the fluidity of the mortars constant. Ottawa sand confirming to ASTM-C778 [14] was used for mortar preparation. Table 2 reveals the physical properties of PP fibers. All specimens were fabricated with the water/binder and sand/binder ratios of 0.5 and 2.75 respectively. The weight of cementitious materials was considered equal to the sum of the weight of the cement and nano-SiO_2.

Table 1. Chemical compositions of cement

Items	Chemical compositions (%)
SiO_2	21.5
Al_2O_3	3.68
Fe_2O_3	2.76
CaO	61.5
MgO	4.8
SO_3	-
L.O.I	1.35

In the initial stage of the present study a total of six batches of mortars were prepared to find out the optimum amount of nano-SiO_2 in ordinary cement mortar (Table3). According to the initial stage results, in the second stage 0.1%, 0.3%, and 0.5% PP fibers (compared with the total mortar volume) were added to the ordinary and the optimum mixtures selected in the initial stage with the purpose of evaluating the influence of the PP fibers on the strength and shrinkage properties (Table 4). In all tests, specimens without fiber were considered as the reference materials.

Table 2. Properties of polypropylene fiber

Property	Polypropylene
Unit weight (gr/cm^3)	0.9-0.91
Reaction with water	Hydrophobic
Tensile strength (MPa)	300-400
Elongation at break (%)	100-600
Melting point (∘C)	175
Thermal conductivity (W/m/K)	0.12
Length (mm)	6

Table 3. Mix proportion of the specimens (initial stage)

Batch No	Sand/Binder	Water/Binder	% Content (by weight)	
			O.P.C.	N.S.
NS0	2.75	0.5	100	0
NS1	2.75	0.5	99	1
NS3	2.75	0.5	97	3
NS5	2.75	0.5	95	5
NS7	2.75	0.5	93	7
NS9	2.75	0.5	91	9

Table 4. Mix proportion of the specimens (second stage)

No	S^a/B^b	W^c/B^b	%Content (by weight)		%PP (Vol)	No	S^a/B^b	W^c/B^b	% Content (by weight)		%PP (Vol)
			O.P.C	N.S					O.C	N.S	
1	2.75	0.5	100	-	0	5	2.75	0.5	100	-	0.3
2	2.75	0.5	93	7	0	6	2.75	0.5	93	7	0.3
3	2.75	0.5	100	-	0.1	7	2.75	0.5	100	-	0.5
4	2.75	0.5	93	7	0.1	8	2.75	0.5	93	7	0.5

a: Sand b: Binder (Cement +Nano-SiO_2) c: Water

b) Test method

The procedures for mixing the fiber-reinforced mortar involved the following. First, the nano-SiO_2 particles were stirred with 90% of mixing water at high speed for about 1 min. Then, the specified amount of fiber was distributed and mixed for 2 min at medium speed. Next, cement was added to the mixture. After adding, the mixer was allowed to run for 1 min at medium speed. The sand was then gradually added over a period of 30s while the mixer was running at medium speed. Finally, the superplastisizer and remaining water were added and stirred at high speed for 30s.The mixture was allowed to rest for 90s, then mixing was continued for 1 min at high speed.

Fresh mortar was cast into 50×50×50 mm cubes for compressive and water absorption tests and 50×50×200 steel molds for flexural and shrinkage tests. The specimens were tamped using a hard mallet

to decrease the amount of the air bulbs. After the feeding operation, each specimen was allowed to stand for 24 h. Then the specimens were demolded and kept in water at 23±3 $^{\circ C}$ until they were tested.
Compressive strength test was conducted in accordance with ASTM-C109 [15] using a hydraulic testing machine under load control at 1350N/s. The three-point (i.e. center-point) loading flexural test was carried out with the span of 180mm and at a loading rate of 44N/s. The flexural and compressive strength results were determined at 7, 28, 60 and 90 days of curing. Each test result was the average of six test specimens. Shrinkage test samples were cured in the laboratory environment at 70% RH and 27±3 $^{\circ C}$. Changes in the length of the mortar samples were measured using a length comparator with a precision of 0.002mm. The first measurement was taken after 24h of mixing, while the rest of the measurements were taken at the ages of 3, 7, 14, 21, 28, 35 and 42 days. Six shrinkage specimens were tested for each batch of mortar. The water absorption test was carried out at 28 days as follows: saturated surface dry specimens were kept in an oven at 110$^{\circ C}$ for 72 h. After measuring the initial weight, the specimens were immersed in water for 72h. The final weight was then measured and the final absorption was reported to assess the mortar permeability. For every absorption value, four specimens were tested and averaged.

3. EXPERIMENTAL INVESTIGATION AND RESULTS

a) Compressive strength

The compressive strength of cement mortars with different dosages of nano-SiO_2 at four ages are shown in Fig. 1. The standard deviation of measurements is also given in Table 5. It is clear that the compressive strength of ordinary cement mortar increases with an increase in the amount of nano-SiO_2. It can be seen that increasing the nano-SiO_2 content from 7% to 9% did not improved compressive strength significantly. It seems that a large amount of nano-SiO_2 even decreases the strength. According to Hui li [16], homogeneous hydrated microstructure, which is essential for the strength of the cement matrix, cannot be formed because nano particles cannot be well dispersed. Strength enhancement of nano-SiO_2 can be attributed to reduction in the content of $Ca(OH)_2$ which has no cementing property and produces no hydrated calcium silicate (CSH), which plays a vital role in the mechanical characteristics of cement paste [17-18]. Nano-SiO_2 particles also generate a large number of nucleation sites for cement hydration products, making the paste microstructure more homogenous and improving its strength and permeability [19]. In view of the above results, cement mortar with a substitution of cement by 7% nano-SiO_2 was selected as the optimum mixture. Figure 2 shows the compressive strength of fiber-reinforced mortars (the standard deviation of measurements is given in Table 6) .Results appearing in this figure indicate that PP fibers induce a slight modification in the compressive strength, which increased gradually at first with the increase of fiber content, but then decreased with the further increasing of fiber content. Almost all the specimens containing 0.1% PP fiber by volume exhibited an increase in compressive strength compared to the target specimens. A possible reason for this may be that PP fibers act as crack arresters. The uniformly distributed PP fibers reinforce mortar against disintegration by resisting further opening of initial cracks and disallowing the microcracks from growing into macro cracks [20].

The strength development at 0.1% PP fiber addition varied depending upon the nature of the mixtures. The mortar containing 7% nano-SiO_2 showed greater average enhancement by 6.49% compared to plain cement mortar by 3.1%. At 0.3% fiber addition, the compressive strength of plain cement mortar decreased contrary to mortar containing 7% nano-SiO_2 that still increased. It is obvious that an increase in pp dosage beyond 0.3% decreases the compressive strength. This is understandable because large contents of PP fibers are more difficult to disperse uniformly. Therefore fibers form clusters and create more micro-defects in the cement matrix, which inevitably reduces the compressive strength of mortar.

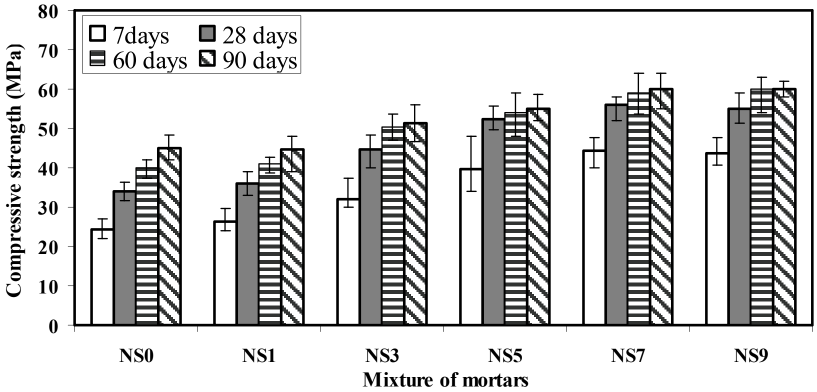


Fig. 1. Compressive strength of mortars at different contents of nano-SiO_2

Table 5. Standard deviation of compressive test results(MPa)

Mix number	Standard deviation (Mpa)			
	7 days	28 days	60 days	90 days
NS0	1.94	1.94	1.64	2.65
NS1	2.02	2.20	1.60	3.14
NS3	2.61	3.23	2.15	3.17
NS5	5.0	2.35	3.86	2.75
NS7	3.3	2.28	4.24	3.42
NS9	2.35	3.36	3.56	1.72

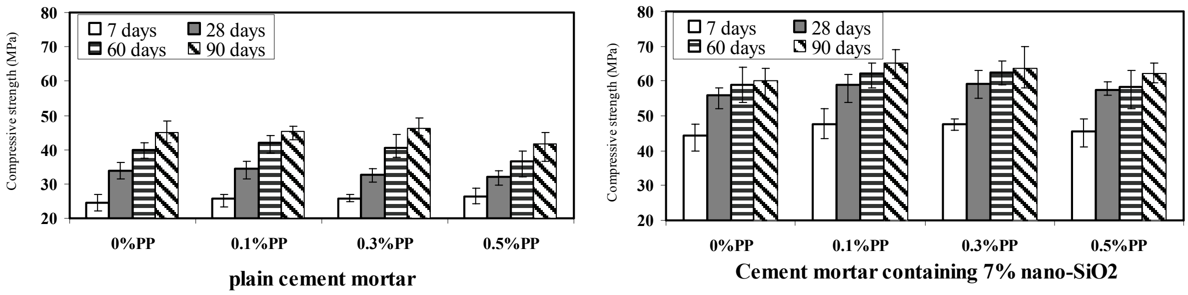


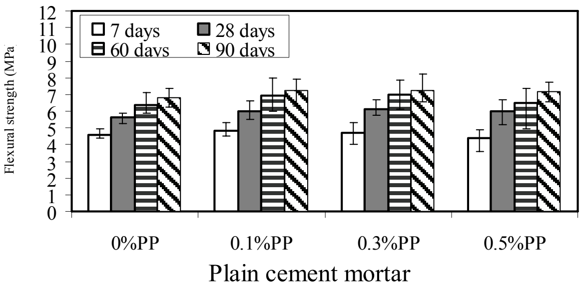
Fig. 2. Compressive strength of different mortar mixtures according to the PP content

Table 6. Standard deviation of compressive test results (MPa)

Mortar type	Ordinary cement mortar			
	7 days	28 days	60 days	90 days
0% fiber	1.949359	1.943983	1.640325	2.653614
0.1% fiber	1.194013	2.021633	2.087502	1.40961
0.3% fiber	0.837655	1.594287	2.773265	1.907529
0.5% fiber	1.67023	1.518771	2.646066	2.838779
	Cement mortar containing 7% nano-SiO_2			
0% fiber	3.335716	2.280351	4.243426	3.425639
0.1% fiber	3.132252	2.828427	3.013083	3.046747
0.3% fiber	1.199444	3.249615	2.378235	3.967745
0.5% fiber	2.887213	1.952178	4.469191	2.17501

b) Flexural strength

The flexural strength of the mortar specimens are presented in Fig. 3 (the standard deviation of measurements is given in Table 7). Assessment of the flexural strength of nonfibrous specimens revealed that nano-SiO_2 effectively increased the flexural strength of mortar. Results of fiber reinforced specimens showed that the flexural strength in fiber reinforced mortars was slightly higher than in mortars without fibers. The values of the flexural strength of mortars increased with increasing the fiber content until reaching an optimal amount of 0.3%, then dropping to some lower value at 0.5%. However, for mortar containing nano-SiO_2 a slight increase of flexural strength was observed beyond 0.3%. It should be noted that the presence of nano-SiO_2 in the cement matrix improved the effectiveness of the fibers in strengthening the mortar. The microstructure of cement paste at the interface between the fiber and matrix is the most important region influencing the fibers effectiveness. Adding nano-SiO_2 strengthens this weak region through reduction of the internal porosity, especially in the transition layer by consumption of porous portlandite crystals which array in the interfacial between fiber and matrix. Therefore, fiber/matrix contact area increases and higher friction can be formed between the two.



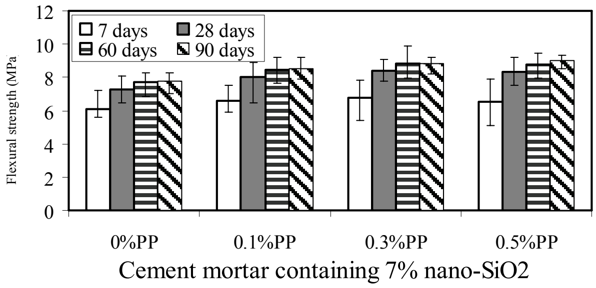


Fig. 3. Flexural strength of different mortar mixtures according to the PP content

Table 7. Standard deviation of flexural test results (MPa)

Mortar type	Ordinary cement mortar			
	7 days	28 days	60 days	90 days
0% fiber	0.216333	0.268384	0.411906	0.44186
0.1% fiber	0.298976	0.410386	0.750424	0.583179
0.3% fiber	0.43652	0.365605	0.701471	0.663315
0.5% fiber	0.469201	0.546763	0.84301	0.496226
	Cement mortar containing 7% nano-SiO_2			
0% fiber	0.572145	0.515429	0.493292	0.408461
0.1% fiber	0.692897	0.899815	0.585335	0.49245
0.3% fiber	0.617792	0.483598	0.797839	0.338684
0.5% fiber	0.825631	0.621375	0.485912	0.311551

Typical flexural load deflection response of different mixtures containing 0%, 0.1%, 0.3% and 0.5% PP fibers at 90 days are represented in Fig. 4. The test was controlled automatically by computer with a constant cross head movement of 1mm/min. From the figure it can be seen that unreinforced mortar demonstrated brittle behavior. The samples fully fractured with the increase of mid span deflection after peak load, while fiber reinforced mortar exhibited ductile behavior. Study of the load-deflection graphs showed that mortar containing nano-SiO_2 was obviously more brittle than that of plain mortar, however, integrating PP fibers somewhat compensated this shortage. A small effect was noted upon fiber volume fraction of 0.1% and a relatively bigger increase was observed when increasing the fiber content to

0.5%.When cracks occur and propagate fibers are able to bridge across the surface of the cracks, preventing the crack-face separation in the tension half of the reinforced beam, the fibers sustain the load until they pullout from the matrix. This mechanism provides an additional energy-absorbing which leads to a stable fracture process and higher fracture energy. The presence of nano-SiO_2 enhanced the efficiency of the transforming load from matrix to fiber by increasing the friction coefficient between the fiber and the matrix. Hence the effect of PP fibers on post-peak resistance was more obvious for mortars containing nano-SiO_2.

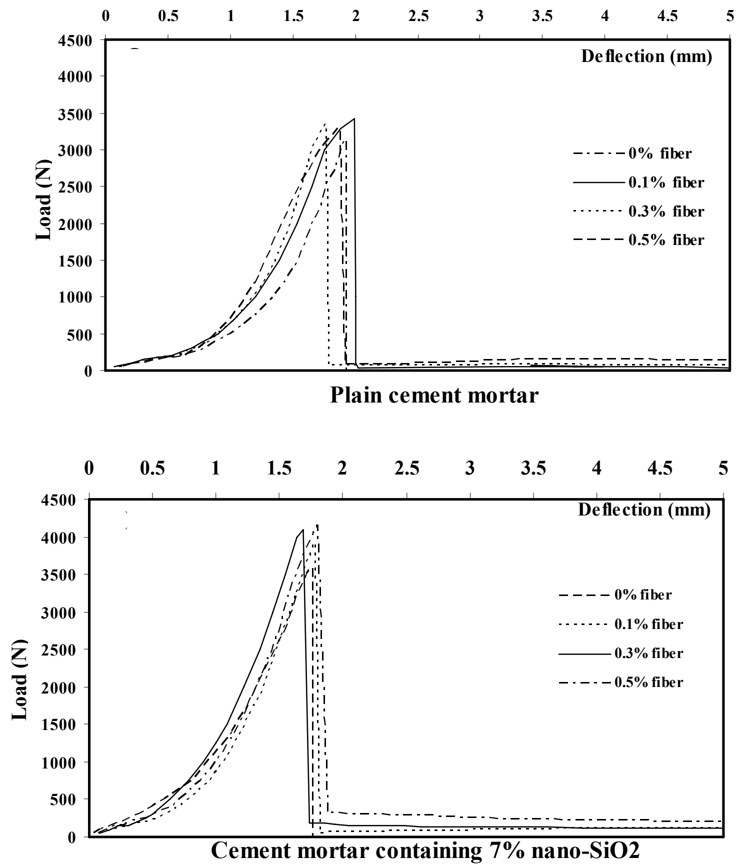


Fig. 4. Flexural behavior (load-deflection) of different mixtures

c) Water absorption

The water absorption of the specimens is shown in Table 8. A study of the water absorption values of the unreinforced specimens revealed that incorporating nano-SiO_2 into cement mortar improved the water absorption properties of the products. The reason for this observation is that the fine particles of pozzolan block the channels connecting the capillary pores in cement paste and generate a more homogenous distribution of CSH gel, resulting in less pore structure and permeable voids [21]. Adding PP fibers changed the water absorption properties. The water absorption values of the mixtures decreased at 0.1% fiber content. It was observed that increasing the fiber percentage increased the water absorption of cement mortars. The reason behind this observation could be the poor dispersion of PP fibers in mortar, consequently increasing the pore volume of the cement matrix. For plain cement mortar, water absorption started to rise up at 0.3% fiber content, while in mortars containing nano-SiO_2 at 0.5%. This means that the presence of nano-SiO_2 in cement matrix provided better fiber dispersion. The reason may be due to the increase in the cohesiveness of the cementitious matrix by nano-SiO_2, which is beneficial for better dispersion of PP fibers [22].

Table 8. Water absorption and standard deviation of different mortars (%)

Batch No	Absorption (%)	Standard deviation (%)	Batch No	Absorption (%)	Standard deviation (%)
1	6.12	0.096	5	6.45	0.108
2	4.23	0.059	6	4.19	0.078
3	6.04	0.119	7	7.09	0.634
4	4.20	0.149	8	4.21	0.325

d) Shrinkage behavior

The shrinkage behavior of mortars is presented in Figs. 5 and 6 (the standard deviation of the measurements is given in Table 9). From the results it can be concluded that the presence of nano-SiO_2 in mortar increased the drying shrinkage. This may be due to self desiccation caused by the pore size refinement of nano-SiO_2 [23]. Moreover, from the data presented by previous researchers it is seen that nano-SiO_2 particles act as an activator to accelerate cement hydration [24]. Therefore, the autogenous shrinkage related to chemical shrinkage can be increased.

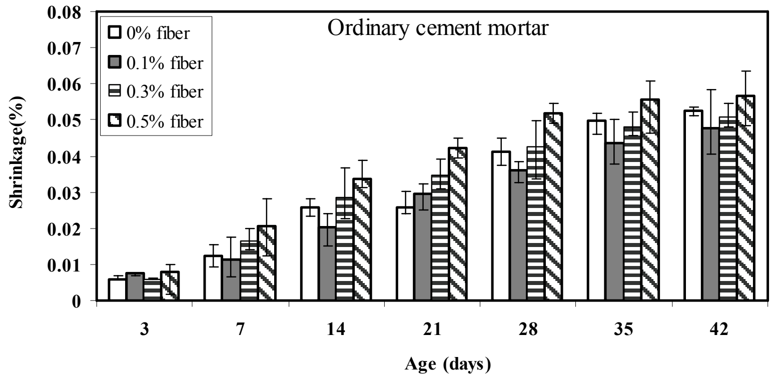


Fig. 5. Effect of PP fibers on shrinkage of plain cement mortar

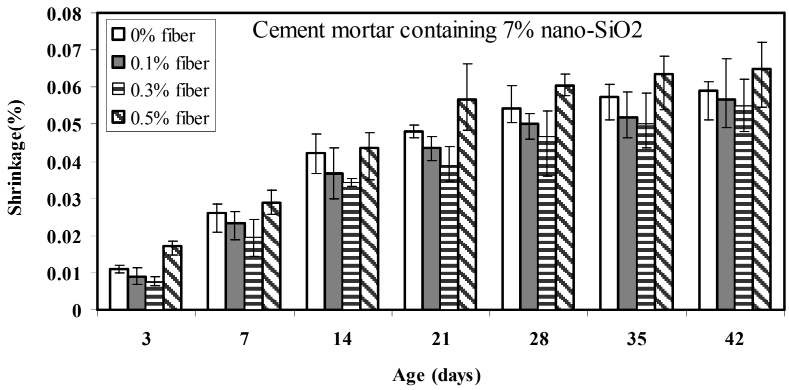


Fig. 6. Effect of PP fibers on shrinkage of cement mortar containing 7% nano-SiO_2

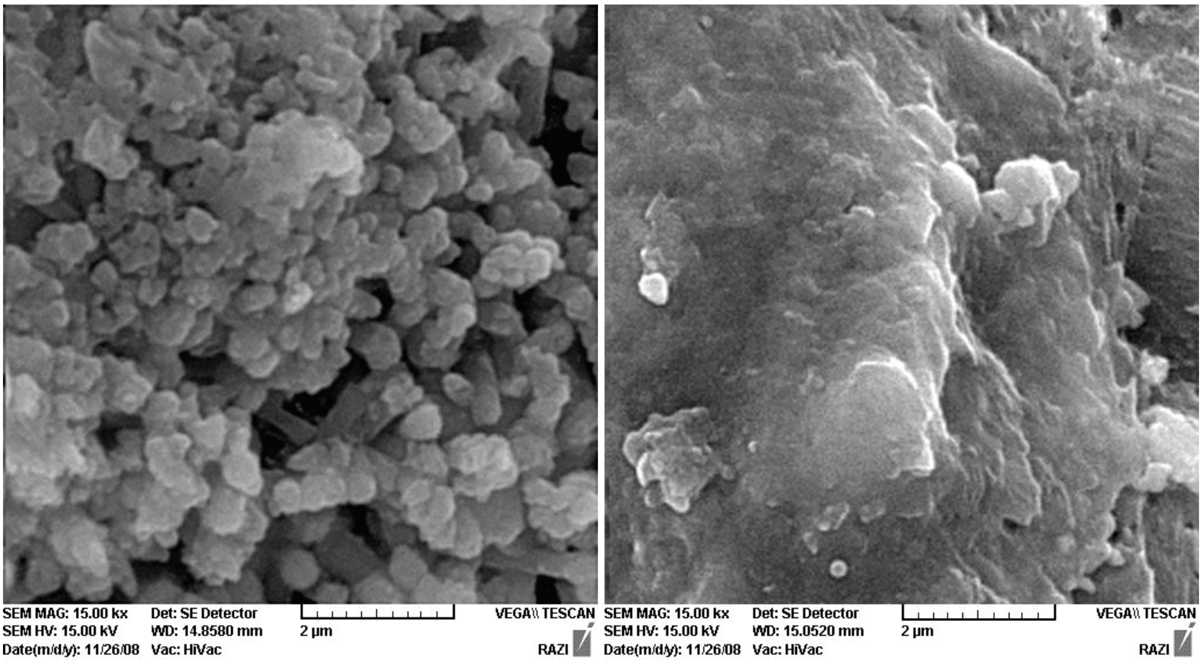
Results of fiber reinforced specimens demonstrated that small amounts of fiber could contribute positively to moderate the length change caused by drying shrinkage. All the mortars reinforced with 0.1% PP fiber provided better improvement for shrinkage. Using a higher content of PP fibers (beyond 0.3%) obviously did not work for moderating the shrinkage strain. At 0.5% PP content, drying shrinkage of all specimens increased even more than the reference mortars. More investigations are needed to explain this effect.

Table 9. Standard deviation of shrinkage test results (%)

Mortar type	Ordinary cement mortar						
	3 days	7 days	14 days	21 days	28 days	35 days	42 days
0% fiber	0.000671	0.006731	0.001904	0.002299	0.003273	0.002093	0.000866
0.1% fiber	0.032707	0.003851	0.003595	0.002894	0.002219	0.004207	0.008265
0.3% fiber	0.000112	0.002038	0.004906	0.003185	0.008804	0.002253	0.002452
0.5% fiber	0.003186	0.005341	0.002867	0.002279	0.001848	0.005563	0.007721
	Cement mortar containing 7% nano-SiO_2						
0% fiber	0.000775	0.002745	0.003678	0.001257	0.003609	0.003372	0.003816
0.1% fiber	0.001789	0.00361	0.005068	0.003101	0.002562	0.004475	0.008535
0.3% fiber	0.000867	0.003903	0.000872	0.003815	0.008835	0.005223	0.005441
0.5% fiber	0.001446	0.002196	0.004939	0.006194	0.001847	0.005563	0.008592

4. MICROSTRUCTURE

Application of scanning electron microscopy (SEM) facilitates the characterization of the cement and concrete microstructure. It has been found that nano structure features, in particular nano porosity, play a notable role in cement paste characteristics [25]. Accordingly, in this section the effect of nano-SiO_2 on the microstructure of cement paste was evaluated. To this end, cement pastes with 0% and 7% nano-SiO_2 with the same water to cement ratio were prepared. Samples were cured under standard condition for 28 days. For enhancement of the sample surface conductivity, samples were sputtered with gold preparatory to imaging. The microstructure of the samples with the same magnifications (15000x) is presented in Fig. 7. It can be seen that the plain cement paste has a porous microstructure, but when 7% nano-SiO_2 by weight of cement was added the microstructure significantly changed. Nano-SiO_2 particles formed a very dense and compact texture of the hydrated products through both filler and pozzolanic effects. These results are in agreement with the better performance of the fiber reinforcement in mortar containing nano-SiO_2.



(a) (b)

Fig. 7. SEM micrograph of cement paste containing nano-SiO_2: (a) 0% nano-SiO_2, (b) 7% nano-SiO_2

5. CONCLUSION

Results of the experimental study on the properties of fiber reinforced cement composite mortar containing nano-SiO_2 particles were presented in this paper. From the investigation it appeared that utilizing PP fibers in cement matrix caused a slight enhancement in compressive and flexural strength. The contribution of further increase of the fiber content to mechanical strength was not positive. A possible reason for this observation could be the poor dispersion of PP fibers in mortar that increases pore volume and creates more micro defects in cement matrix. The fiber reinforced mortar demonstrated a higher post-peak flexural strength compared with reference mortars. This effect was more obvious at larger content of fibers. The effectiveness of the fiber reinforcement in mechanical strength somewhat improved with the incorporation of nano-SiO_2 particles. This can be due to reduction of internal porosity, especially in the fiber/matrix transition zone that provides a higher contact surface and hence friction between the two. Water absorption of mortars deceased by incorporating nano-SiO_2. Adding small amounts of PP fibers resulted in an improvement in water absorption characteristics, however higher amounts of fiber, especially in ordinary cement mortar, had no positive effect. The presence of nano-SiO_2 in cement matrix increased the dying shrinkage of mortars. The inclusion of fiber reinforcement within composite cement mortar could moderate this effect. However utilizing high content of fiber (beyond 0.3%) had no positive impact on shrinkage strain. SEM observation showed that nano-SiO_2 apparently decreased the porosity of hardened cement paste and improved the microstructure of cement paste in dense and compact form.

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