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## Influence of colloidal silica sol on fresh properties of cement paste as compared to nano-silica powder with agglomerates in micron-scale



Deyu Kong <sup>a, b, \*</sup>, David J. Corr <sup>b</sup>, Pengkun Hou <sup>b, c</sup>, Yang Yang <sup>a</sup>, Surendra P. Shah <sup>b</sup>

<sup>a</sup> College of Civil Engineering & Architecture, Zhejiang University of Technology, Hangzhou, 310014, China

<sup>b</sup> Department of Civil & Environmental Engineering, Northwestern University, Evanston, IL 60208, USA

<sup>c</sup> College of Materials Science & Engineering, Jinan University, Jinan, 250022, China

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#### ABSTRACT

Influence of colloidal silica sol (SS) with mono-dispersed nano-particles on fresh properties of cement paste was investigated as compared to nano-silica powder (NS) with agglomerates in micron-scale. The SS addition showed a much greater influence on sedimentation and rheological behavior of the paste than the NS incorporation, because the nano-particles in SS coagulate immediately once cement is mixed into water containing SS, forming loose floc and coating layer around cement particles. The loose floc cannot function as fillers to release free water, but possesses a more open microstructure, leading to a higher free water retention capacity than the agglomerates in NS. However, addition of SS presented an obviously better accelerating effect on cement hydration than that of NS, though the nano-particles in SS are nearly the same as those in NS in primary particle size and the flocs in the paste with SS addition are typically larger than the agglomerates in NS, implying that the acceleration may have nothing to do with the seeding effect. Through detecting calcium-absorbing properties of NS and SS, it is found that the accelerating effect is highly dependent on the rapid depletion of calcium ions in the paste. Finally, it was interestingly found that the CH crystals are even more prone to grow along (0001) plane with larger size in the paste with SS addition, because the coagulated gel network in the paste slows down the diffusion rate of the released ions and eliminate the convection in the system, thus the 3D nucleation and growth of the CH crystals were suppressed.

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### 1. Introduction

In modern concrete technology, reactive mineral admixtures, such as ground granulated blast furnace slag [1-4], fly ash [1,5,6], natural pozzolan [7,8], silica fume [4,8], *etc.* are widely used in concrete mixes to reduce the clinker consumption on the one hand and improve properties, especially durability of concrete structures on the other hand. The fine particles in these admixtures are thought to play three major effects. They act as fillers to physically fill in the void space among larger particles, releasing free water to improve fluidity and reducing porosity to enhance strength and durability [9-11]. They chemically react with calcium hydroxide, producing additional cementing materials (mainly pozzolanic C–S–H gels) to combine free water and reduce porosity [1-11]. The

E-mail address: kongdeyu@mail.hz.zj.cn (D. Kong).

pozzolanic C–S–H gels also function as seeds to provide nucleation sites for C–S–H gels hydrated from cement, causing pore as well as grain refinement of matrix and ITZ [12–15]. However, the early-age strength is often sacrificed if the admixture with low pozzolanic reactivity, for example, fly ash is used [1,5,6,16]. Though silica fume exhibits much greater pozzolanic activity than fly ash [17], it has been revealed that its pozzolanic reactivity at very early age is relatively low [17–20]. The XRD pattern of silica fume changed little up to 7 days while mixing into saturated CH solution and extensive C–S–H formation occurred only after 120 days [18].

In recent years, much attention has been paid to the applications of nano-silica in cement-based materials for further improvement of properties [21–33]. It is assumed that the nano-particles can act as fillers, pozzolan and seeds much more effectively due to its much finer particle size and much greater pozzolanic reactivity than silica fume [15,21–24]. However, the nano-particles in powder often exist in an agglomerated (loosely-held clusters) state with final grain size from submicron to as high as 100  $\mu$ m due to their very high specific surface area and energy [34,35]. Even in well-

<sup>\*</sup> Corresponding author. College of Civil Engineering & Architecture, Zhejiang University of Technology, Hangzhou, 310014, China.

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dispersed colloidal dispersion, the nano-particles still exist as aggregates (firmly-held clusters) with grain size in submicron [27].

With regard to the influence of nano-silica agglomeration on cement hydration and properties of the fresh and hardened cementitious materials, a previous study [36] revealed that the very large agglomerates in nano-silica powder cannot function as fillers to release free water to improve fluidity. They even absorb some free water originally contributing to fluidity. The rheological behavior of the fresh paste was thus greatly influenced while incorporating nano-silica with large agglomerates. Though the microstructure, hence the mechanical and durable properties of the hardened cement-based materials were enhanced, the improvement was limited, because the large agglomerates may become weak zones due to their low strength and elastic modulus and there even exists interfacial transition zone between the reacted agglomerates and the bulk cement pastes [37].

Colloidal silica sol denotes small particles consisting of an amorphous  $SiO_2$  core with a hydroxylated surface, which are insoluble and mono-dispersed in water, and with a particle size of 10–100 nm, which are small enough to remain suspended in a fluid medium without settling [29]. Many studies have tried to use colloidal silica sol to improve the filling and some other positive effects. However, recent studies [38,39] revealed that the nanoparticles in sol coagulate immediately when cement is mixed into water with silica sol addition. As a result, whether by using nanosilica powder, dispersion, or sol, it is the behavior of the final agglomerates or aggregates, rather than that of the individual nanoparticles, which controls the filling, the pozzolanic and the seeding effects.

In the literature, a lot of work has been done to investigate the influence of nano-silica on cement hydration and properties of the fresh and hardened cement paste by using nano-silica powder [21–26], dispersion [27] or colloidal silica sol [28–33] as compared to silica fume. However, there is limited knowledge about the influence of nano-silica in different states on properties of the fresh and hardened cementitious materials. The present study mainly focused on the effect of colloidal silica sol with mono-dispersed nano-particles on fresh properties of the cement pastes as compared to nano-silica powder with agglomerates in micronscale. It is found that addition of colloidal silica sol showed an absolutely different influence on properties of the fresh cement paste, including the dispersion and sedimentation behavior of the cement particles in water, rheological behavior, heat evolution of the cement paste, and even crystallization of calcium hydroxide in fresh cement paste, as compared to that of nano-silica powder.

#### 2. Experimental programs

#### 2.1. Materials

Ordinary Portland cement (namely 52.5 grade) conforming to Chinese standard GB175-2007 was used and the physical and mechanical properties are shown in Table 1. A naphthalene-based superplasticizer (SP) with solids content of 30 wt% was used in the study to adjust the fluidity of the fresh cement pastes.

Commercially available nano-silica powder (NS) and ammonium stabilized colloidal silica sol (SS) were used in the study. The

Table 1						
Physical	and	mechanical	properties	of	Ρ·Ο	52.5

Consistency/%	Setting time/min		Strength/MPa				
			Flexural		Compressive		
	Initial	Final	3 d	28 d	3 d	28 d	
26	185	320	5.0	8.9	25.1	55.8	

specific surface area of NS measured through BET method was about 142.9 m<sup>2</sup>/g. The TEM photograph (Tecnai G2 F30 S-Twin) showed that the primary particles of NS are in nano-scale, but aggregate together, as illustrated in Fig. 1. The particle size distribution of the cement and the NS powder were analyzed by using a laser analyzer (Mastersizer 2000, Malvern), as given in Fig. 2. The results revealed that the measured sedigraph median particle diameter D<sub>50</sub> (the size at which the accumulative volume percent reaches 50%) of the NS and the cement were about 6.567  $\mu$ m and 18.857  $\mu$ m, and the specialized grain size D<sub>90</sub> reached 11.332  $\mu$ m and 50.529  $\mu$ m respectively. The chemical compositions and physical properties of SS are illustrated in Table 2.

In all tests, the raw materials used were stored in an airconditioned room with temperature of 22.0  $\pm$  2 °C for at least 24 h before preparing pastes.

#### 2.2. Methods

#### 2.2.1. Sedimentation behavior

A kind of sedimentation test was used to observe the dispersion and sedimentation behavior of cement particles in water with nano-silica addition. In the sedimentation test, the nano-silica was dispersed into 25 ml water in a colorimetric tube by shaking for 1 min and then 5.0 g cement was added into the water with nanosilica addition. The NS incorporated were 2.0% and the SS 2.0% and 18.75% (solid loading in mass of the cement) in the pastes. The dispersion behavior of cement particles in water was monitored by using a digital video camera. The tubes were shaken for another 1 min and then left settling without interference for 15 min. The sedimentation result of the paste was then observed by using the camera. For the paste with SS addition, it was found that the sedimentation volume of the paste was obviously higher than that of the other two pastes, as shown in Fig. 3. Thus about 10 ml upper paste was sucked out and observed by using TEM (Tecnai G2 F30 S-Twin). The obtained TEM photographs are also shown in Fig. 1. After the pastes were left settling without interference for 1 h, 4 h, 12 h, 24 h, 3d, 7d and 28d, the sediments were filtered out, washed twice with acetone and vacuum dried at 105  $\pm$  5 °C for 24 h. The dried samples were then scanned by using XRD differometer (X'Pert PRO, CuKa radiation) under the same condition. For the pastes cured for 7d, the sediments were vacuum dried at 105  $\pm$  5 °C for 24 h, then the top layer and bottom layer were observed respectively by using SEM (HITACHI, S-4700).

#### 2.2.2. Rheological behavior

Rheological behavior of the fresh cement pastes with water cement ratio of 0.40 and with 0.50wt% SP addition was measured at controlled temperatures using a rotational viscometer (Model NXS-11A, Chengdu Instrument Factory, China) equipped with cylindrical spindle. The as-prepared paste was filled into the water-jacketed stainless steel cylindrical vessel and the rotor depth was kept constant throughout the measurements. Temperature was kept at 20 °C by circulating water at the desired temperature around the reservoir containing the sample using the heat bath circulator. The torque was measured at different spindle speeds (5.6–360 rpm) with a 100-ml cylindrical vessel and spindle No.2, which corresponds to a shear rate from  $3.178 \text{ s}^{-1}$  to 204.3 s<sup>-1</sup>. Enough samples were used to cover the immersion groove on spindle shaft. The torque readings were taken after 20 s of rotation at each of the speeds for the same paste.

#### 2.2.3. Hydration heat

A semi-adiabatic calorimeter was used to evaluate the effect of nano-silica on cement hydration. Pastes with 40 ml water and 100 g cement were prepared and mixed manually in nearly the same Download English Version:

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