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Rheological properties of cementitious composites with nano/fiber fillers

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HIGHLIGHTS

- Rheology of cement paste with different types of nano- and micro-fillers are compared.
- Rheological behaviors of composites are described by modified Bingham model.
- Rheological properties of composites are strongly correlated with fillers, w/c ratio, superplasticizer content.
- Ultrasonic time and mix speeds have strong effect on rheological properties of composites.

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ABSTRACT

In this paper, rheology of cement pastes with various types of nano- and micro-fillers are tested by rheometer and the rheological behaviors are described by modified Bingham model. The fillers include nano-SiO_2, nano-TiO_2, carbon nanotubes (CNTs), carbon nanofibers and carbon fibers. Yield stress and plastic viscosity of cement pastes are calculated and compared. Experimental results indicate that the rheological properties are strongly correlated with the types and content of fillers, the w/c ratio, the superplasticizer (SP) content, the ultrasonic time and mix rates. High specific surface area of fillers is the main reason affecting the rheology of cement paste. The yield stress of cement paste with fiber fillers is much higher than that with particle fillers due to the intertwining effect of fiber fillers. The rheological properties can be effectively regulated by w/c ratio with extremely small change, especially for cement paste with CNTs. The SP dosage exists a saturation point that above this dosage the fluidity of paste rises slowly. Ultrasonic treatment can overcome the problem of filler aggregation and significantly decrease the yield stress of cement paste. Both yield stress and the plastic viscosity of cement paste decrease with the increasing mixing rate in a certain range. However, the effect of the mixing rate is inferior to ultrasonication.

1. Introduction

Viscoplastic fluids are an important class of non-Newtonian materials that exhibit no deformation up to a certain level of stress, known as the yield stress. Such a material behaves as a solid when the shear stress bellows the yield stress. To make them flow, sufficiently strong shearing forces are necessary to break the bonds between grains which is initial yield value [1]. As a kind of viscoplastic fluid, cementitious composite has been extensively studied about its rheology in the last decades. Moreover, the Bingham equation was determined in steady state for the rheological properties of traditional cementitious composites [2]. With the introduction of superplasticizer and the decline of w/c ratio, the flow of cementitious composites presents a nonlinear stress–shear rate relationship [3].

Nano- and micro-fillers can change the microstructure and mass transfer in microscale to improve the mechanical strength and service life of cementitious composites [6–11]. Various types of nano- and micro- fillers have been used to modify cement-

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based systems. The representatives are nano-SiO_2 (NS), nano-TiO_2 (NT), carbon nanotubes (CNTs), carbon nanofibers (CNFs) and carbon fibers (CFs) [12–16]. However, due to the extreme small size and large specific area of these fillers, the workability of cementitious composites, especially cement paste, can be affected significantly to be poor. Since concrete may be regarded as a dispersion of coarse aggregates in a matrix of fine particles, its rheological properties greatly depend upon the quantity and characteristics of the fine components. For this reason, a full understanding of the rheological behaviors of fresh cement pastes is needed [4]. So the rheology of cement paste is studied to provide more detailed information.

In recent years, many studies have been done on the rheological properties of cementitious composites containing nano-fillers. For example, Banfill et al. indicated that CF reinforced cement conformed to the Bingham model [24]. Wang et al. proved that suitable fluidity and viscosity are helpful for dispersion of CNFs in cement paste. A good CNFs dispersion resulted in an improved piezoresistive performance [17]. Nadiv et al. revealed that dispersant reduced the viscosity of the cementitious composite containing CNTs due to its ability to diffuse and stabilize the cement particles [18,19]. Quercia et al. studied the influence of NS on the rheology of self-compacting concretes (SCC) and oil-well cement slurries [20–22]. Jalal et al. compared the effects of F fly ash, NS and silica fume on the properties of high performance SCC [23].

The rheological properties are critical to cementitious composites such as SCC and oil-well cement. Not only the homogeneity and workability of fresh mixture of cementitious materials are determined by the rheological properties, but also the hardened properties of cementitious materials have relevant with it. It is also used as a secondary method to evaluate the service performance of cement materials. However, little work has been done on the rheology of cementitious composites containing nano-fillers with rheological model by a rheometer. The comparison of the rheology of cementitious composites with diverse fillers is much rarer. In this study, cement pastes containing nano- and micro- fillers with diverse filler content, w/c ratio, SP content, ultrasonic time and mix speeds were fabricated and tested for rheological properties. Modified Bingham model was applied to describe the shear stress–shear rate relationship of cement pastes and two rheological parameters are obtained. The comparison of the rheology of various samples was performed by contrasting their yield stress and plastic viscosity.

2. Experimental programs

2.1. Materials

The raw materials used to fabricate cementitious composites include cement, water, water reducer and fillers. The chemical composition of P.O 42.5R cement is shown in Table 1. The water reducer is 3310E polycarboxylate superplasticizer whose solid content is 45% and it can reduce water to an extent of 30%. In order to compare the enhancing effect of diverse fillers on cementitious materials, five types of representational fillers are employed in this paper. The fillers include nano particles i.e. NS and NT, nano carbon fibers i.e. CNTs and CNFs as well as micro carbon fibers i.e. CFs in lengths of 3 mm and 6 mm.

The CNTs are multi-walled while the CNFs are PR-24-XT-HHT type. The physical properties of NT, NS, CNTs and CNFs are shown in Tables 2 and 3. The performance parameters of 3 mm and 6 mm PAN-based CFs are listed in Table 4.

2.2. Preparation

Three groups of specimens were fabricated for various research purposes to study the effects of filler dosages, w/c ratio and super-plasticizer dosages on the rheological properties of cement paste.

(1) To study the effects of filler dosages, nano/fiber fillers were added to cement pastes at dosages of 0.1%, 0.5%, 1.0% and 1.5 wt% of cement. The water to cement ratio was fixed at 0.2 and the super-plasticizer dosage was 0.75 wt% of cement. The mix proportion of cement pastes with different fillers is presented in Table 5.

(2) To study the effects of w/c ratio on the rheological properties, the w/c ratio was controlled at 0.18, 0.2, 0.22 and 0.24 by adjusting the consumption of water. The dosage of NS, CNTs and 3 mm CFs were fixed at 0.5%, 0.5% and 1.0 wt%, respectively. The dosage of SP was fixed at 0.75%. The mix proportion design of cement pastes with different w/c ratio is provided in Table 6.

(3) To study the effects of super-plasticizer dosages on rheological properties, specimens with various SP dosages at 0.5%, 0.75%, 1.0% and 1.25 wt% were fabricated. The dosages of fillers were fixed the same as (2) and w/c ratio was 0.2. The mix proportions of cement pastes with different SP dosages is enumerated in Table 7.

(4) To study the effect of ultrasonic time on rheological parameters, cement pastes with 0.5% NS, 0.5% CNTs and 1% 3 mm CFs were fabricated with 0, 2, 5 and 10 min ultrasonic time, respectively. The mix proportions are the same as NS05, CNT05 and 3CF10.

(5) To study the effect of mixing rate on rheological parameters, cement pastes adding with 0.5% NS, 0.5% CNTs and 1% 3 mm CFs were fabricated with mixing rate by 500, 1000, 2000 and 3000 r/min, respectively. The mix proportions are the same as NS05, CNT05 and 3CF10.

The process of fabricating cement pastes with different types of fillers is presented in Fig. 1. The details are as following: (1) Water, fillers and SP were mixed by a cement paste mixer for 30 s in low rate to disperse the filler into the solution. Otherwise, a probe sonicator was used to disperse the CNTs and CNFs instead of mixer for 5 min (or 0, 2, 10 min) because it is difficult to disperse them well with mixer. The suspension was sonicated by a sonicator with a cylindrical tip and a temperature controller. The ultrasonic power is 400 W and the ultrasonic frequency is 20–25 kHz. (2) Cement was put into the mixture with a stir at 100 r/min, and then the mixture was stirred for 3 min at 500 r/min (or 1000, 1500 and 2000 r/min). (3) The mixture was transferred to a container for rheological test.

2.3. Measurement

After all raw materials were well mixed, fresh cement pastes were immediately conducted to rheological test by a rotational rheometer, as graphed in Fig. 1. The shear processes of the rheolog-

Table 1
Chemical composition of cement.

Chemical composition	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	Na_2O
Wt%	61.13	21.45	5.24	2.89	2.08	2.05	0.77