Construction and Building Materials 190 (2018) 255–264

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Effect of a nanoscale viscosity modifier on rheological properties of cement pastes and mechanical properties of mortars

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HIGHLIGHTS

- Nanoclay can significantly improve the plastic viscosity and yield stress of paste.
- The thixotropy of paste increases obviously with the increase of nanoclay content.
- Small amount of nanoclay addition can improve the mechanical properties of mortar.
- Excessive nanoclay content has adverse effect on the mechanical properties of mortar.
- The reticulate structure formed by nanoclay modifies the rheological properties.

ARTICLE INFO

Article history:
Received 26 July 2018
Received in revised form 16 September 2018
Accepted 18 September 2018

Keywords:
Nanoclay
Viscosity modifier
Rheology
Mechanical properties
Thixotropy

ABSTRACT

The effect of a nanoscale viscosity modifier (called NP) on rheological properties of cement pastes and mechanical properties of mortars was experimentally studied. The morphology of NP agglomerates and its dispersed status was investigated by SEM. With different NP contents, the yield stress, plastic viscosity and thixotropy of pastes and the flexural strength and compressive strength of mortars were investigated and compared with plain cement paste and mortar at different water-to-cement ratios. The results illustrate that the incorporation of NP can increase plastic viscosity and yield stress of cement paste significantly. Its thixotropy varies simultaneously as well. The mechanical properties of mortars can be improved when the NP content is less than 1.0%. The reticulate structure formed by nanoscale rod-like NP particles with huge surface area in paste flocculates some water to modify the rheological properties of cement paste.

1. Introduction

At present, the highly flowable cementitious materials such as self-compacting concrete (SCC) and self-levelling floor mortar have been widely used due to their technical and economical advantages [1]. However, the high flowability and high segregation resistance which are main properties of cementitious materials may not be obtained simultaneously. It is often difficult to meet the requirements of cohesiveness and segregation resistance for normal strength SCC on account of its large water-to-binder ratio and small viscosity. Furthermore, the composition fluctuation of cementitious materials and poor compatibility of chemical additive and binder may bring about segregation or bleeding and subsequently, degeneration of strength and durability of concrete in the actual application process. Therefore, the viscosity of fresh concrete should be modified suitably to enhance the cohesion and hence, the stability of cementitious materials [2].

Many viscosity modifying admixtures (VMAs) including organic and inorganic-based materials have been utilized in cementitious materials in order to improve their viscosity and avoid the solid-liquid separation. Some researches [3–6] demonstrated that the organic materials such as natural, semi synthetic and synthetic polymers had a significant influence on the rheological properties of cement paste or mortar which were closely related to the cohesion. Among the admixtures, microbial polysaccharides inclusive of welan gum, diutan gum, guar gum, xanthan gum and other derivatives which are widely used as thicker can improve the plastic and apparent viscosity, the yield stress and the stability of paste [7–9]. By contrast, another widespread organic VAM called cellulose ethers (especially hydroxyethyl methyl cellulose and hydroxypropyl methyl cellulose) has also been a popular thicker because of its good adhesive and water retention properties [8,10]. Nevertheless, the adverse effect on the mechanical properties of cemen-

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https://doi.org/10.1016/j.conbuildmat.2018.09.110
0950-0618/

titious materials may limit the application of some organic VAMs such as hydroxy-propyl-methyl cellulose, the addition of which can result in decrease of compressive strength of mortars [8]. As for the inorganic-based materials, plenty of researches have been conducted to improve viscosity and rheological properties of cementitious materials by using various fine mineral admixtures such as silica fume, ground blast furnace slag and ultrafine fly ash [11,12]. For the past few years, nanomaterials have been introduced as an admixture in cementitious materials to improve various aspects of their properties, especially rheological properties [13–16]. In fact, the combination of emerging nanotechnology and traditional cementitious materials can modify the microstructure of composites at the nanoscale level and improve their performance at the macro level, providing a new approach to solve the shortcomings of cementitious composites [17–19]. The evolution of rheological behavior of cementitious materials containing nanoscale admixtures such as carbon nanofiber, nano-silica and nano-TiO_2 has been found as well which shows their viscosity and cohesiveness can be fortified [20–23]. Furthermore, the mechanical properties of cementitious materials incorporating different nanoscale admixtures were experimentally studied [24–25].

Recently, some reports showed that small addition of attapulgite nanoclay can significantly influence the rheological behavior of cement paste, mortar or concrete and improve the shape-stability [26–28]. Nanoclay particle is negatively charged along its axis and positively charged at its ends, thus tending to bond with each other [29]. Previous research demonstrated that the incorporation of nanoclay in cementitious materials increased their thixotropy and had effects on their structural rebuilding kinetics [28,30,31]. Due to the improvement on thixotropy, nanoclay is considered an effective admixture to reduce formwork pressure of SCC [32]. The influence of nanoclay on adhesive properties of cement pastes (0.2% and 0.5% addition by mass of cement) and hardened properties of mortars (0.3% addition by mass of cement) have also been investigated [27,33]. However, the research on the improvement of paste viscosity by nanoclay is not impeccable, especially considering the influence of nanoclay on mechanical properties of cementitious materials. It makes sense that the change in viscosity and strength of cementitious materials with various nanoclay dosages and the action mechanism should be investigated in detail.

This paper paid attention to the influences of the nanoclay as a viscosity modifier on rheological properties and fluidity of cement paste and the mechanical properties of cement mortars containing NP. Microstructure of NP and mortar was investigated by SEM to explain the action mechanism of NP.

2. Experimental program

2.1. Raw materials

P.I 42.5 Portland cement conforming to Chinese National Standard GB 175-2007 (equivalent to European CEM I 42.5, EN197-1:2011 [34]) was used. The viscosity modifier, NP, is a pure, uniformly sized, rod-shaped mineral particle but spheroidal agglomerates when the particles are dry. The chemical compositions of NP and cement are presented in Table 1. A polycarboxylic superplasticizer (PCE) with 50% solid content was incorporated to make the cement paste reach a required fluidity. Manufactured according to the European Standard EN196-1:2005 [35], ISO standard sand, the maximum particle size of which is less than 2 mm, was utilized as aggregate to prepare cement mortar.

Table 1
Chemical compositions of NP and cement *w*/%

Composition	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	Na_2O	K_2O	LOI
NP	5.86	52.76	10.28	3.40	7.86	0.167	0.449	0.598	16.7
Cement	63.27	22.59	4.42	3.44	2.43	2.41	0.125	0.388	1.21

Note: *w*-mass fraction.

2.2. Mixture design

The mix proportions of cement pastes for rheological test are shown in Table 2. Two series of pastes were prepared with different water-to-cement (W/C) ratios of 0.25 and 0.40. 0%, 0.5%, 1.0%, 1.5% and 2.0% of NP was added into the cement paste. The dosage of PCE was slightly adjusted to ensure that the PCE dosage of each cement paste was saturation dosage. Mortars with aggregate/binder (A/B) ratio of 3:1 and W/C ratios of 0.25 and 0.40 were prepared to evaluate the effect of NP on their mechanical properties. The mix proportions of mortars are listed in Table 3.

2.3. Testing procedures

The particle morphology of NP and its agglomerates was observed with a Hitashi SU8220 scanning electron microscope (SEM). The original agglomerates of NP coated with carbon film were observed by SEM at an accelerated voltage of 15 kV. The morphology of NP particles was observed by SEM after the ultrasonic dispersion of agglomerates in ethanol for 3 min or soak of agglomerates in water. The morphology of NP particles in hardened mortar was also investigated.

The fluidity of cement pastes with different dosages of NP and PCE was tested with flow table test. The saturation dosages of PCE for each series of pastes were determined when the fluidity of paste did not increase with the increase of PCE dosage.

The rheological properties of cement paste were tested using a Brookfield RST-SST rheometer with a 4-bladed vane of the height (h) of 40 mm and radius (R1) of 10 mm. The outer cylinder, the radius (R2) of which is 30 mm, was stationary during the testing procedure. Described in Fig. 1a, the scheme for measuring rheological parameters has a total testing time of 7.5 min and the rotational speed of rheometer reaches its maximum of 60 rpm ranged from 0 to 2 min. The rotation of vane is kept at the speed of 60 rpm for 20 s to make the fresh cement paste be in a steady flow state. The rotational speed of vane then declines from 60 rpm to 0 rpm in descending order for 11 steps. Effective values of stabilized torque and

Table 2
Mix proportions of pastes.

Samples	Cement (*w*/%)	NP (*w*/%)	W/C ratio
P0.25-0	100.0	0	0.25
P0.25-0.5	99.5	0.5	0.25
P0.25-1.0	99.0	1.0	0.25
P0.25-1.5	98.5	1.5	0.25
P0.25-2.0	98.0	2.0	0.25
P0.40-0	100.0	0	0.40
P0.40-0.5	99.5	0.5	0.40
P0.40-1.0	99.0	1.0	0.40
P0.40-1.5	98.5	1.5	0.40
P0.40-2.0	98.0	2.0	0.40

Table 3
Mix proportions of mortars.

Samples	Cement (*w*/%)	NP (*w*/%)	W/C ratio	A/B ratio
M0.25-0	100.0	0	0.25	3:1
M0.25-0.5	99.5	0.5	0.25	3:1
M0.25-1.0	99.0	1.0	0.25	3:1
M0.25-1.5	98.5	1.5	0.25	3:1
M0.25-2.0	98.0	2.0	0.25	3:1
M0.40-0	100.0	0	0.40	3:1
M0.40-0.5	99.5	0.5	0.40	3:1
M0.40-1.0	99.0	1.0	0.40	3:1
M0.40-1.5	98.5	1.5	0.40	3:1
M0.40-2.0	98.0	2.0	0.40	3:1