Construction and Building Materials 160 (2018) 117–126

Contents lists available at ScienceDirect

Construction and Building Materials

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

Effect of mineral admixtures on the structural build-up of cement paste

Qiang Yuan [a,*], Dajun Zhou [a], Baiyun Li [a], Hai Huang [a], Caijun Shi [b]

[a] School of Civil Engineering, National Engineering Laboratory for High Speed Railway Construction, Central South University, Changsha 410075, China
[b] College of Civil Engineering, Hunan University, Changsha 410082, China

HIGHLIGHTS

- Structural build-up of cement pastes was characterized by thixotropic area and growth of static yield stress.
- Structural build-up of cement pastes with various mineral admixtures was tested.
- Structural build-up of cement pastes was related with their calorimetric curve and zeta potential.

ARTICLE INFO

Article history:
Received 4 July 2017
Received in revised form 6 November 2017
Accepted 11 November 2017
Available online 20 November 2017

Keywords:
Structural build-up
Thixotropy
Mineral admixtures
Cement
Static yield stress

ABSTRACT

Structural build-up is of great importance in many concrete applications, such as formwork pressure, multi-layers casting, slip form paving, stability, and 3D printing. In this paper, the growth of static yield stress (τ_s) and thixotropic area were used to characterize the structural build-up of cement paste. The effects of different mineral admixtures, i.e. silica fume (SF), ground slag (GS), fly ash (FA), attapulgite (AG), nano calcium carbonate (NC), and nano silica (NS) on the structural build-up of cement pastes were studied. Calorimetric curves, zeta potential and conductivity of the pastes were also measured. Results showed that thixotropic behavior characterized by the growth of τ_s and thixotropic area gave different ranking on cement pastes with various mineral admixtures. NS, AG, NC and SF increased the growth of τ_s over 120 min, and NS was the most effective one. FA and GS decreased the growth of τ_s over 120 min. Hydration of cement played a dominant role in the structural build-up of cement paste. However, high hydration rate didn't always lead to high structural build-up rate.

1. Introduction

Structural build-up refers to the phenomenon that the "strength" of fresh paste/concrete increases with time due to chemical reaction and/or physical actions. It is of great importance to concrete technology [1]. Fresh concrete at rest tends to flow and deform under the action of gravity, and it tends to segregate due to the density difference of the constituents of concrete. High structural build-up rate benefits to the stability and shape-holding capacity of concrete. When fresh concrete is at rest, the interactions between particles develop because of colloidal flocculation and cement hydration, and thus the structure of cement builds up. As a consequence, cement paste can hold the aggregate against gravity and prevent segregation from happening, and the shape-holding capacity of concrete increases. Therefore, high structural build-up rate is always desired in many applications, such as reduction of formwork pressure, slip form paving, stability, and 3D printing concrete [2–7]. However, high structural build-up rate may also be detrimental in some cases. For example, in the application of multi-layers casting, high structural build-up rate reduces the bonding strength between layers [8].

In concrete science, the terminology of thixotropy, instead of structural build-up, is often used to describe the increase of the "strength" of fresh concrete. Thixotropy is generally defined as the continuous decrease of viscosity with time when a shear rate is applied to a sample that has been previously at rest, and the subsequent recovery of viscosity in time when the shear stops [9,10]. It has three characteristics: 1) the determination of thixotropy is often based on the decrease of viscosity; 2) thixotropy is a time dependent property; 3) thixotropy is a reversible process, and related with the break-down and build-up of flocculates of suspension particles. It seems that the terminology of thixotropy is not applicable to cement-based materials in most cases. On one hand, evolution of static yield stress (τ_s), instead of viscosity, is more often used in cement-based materials. On the other hand, once cement is in contact with water, both reversible and irreversible reactions happen simultaneously, and thixotropy is mainly dealing

* Corresponding author.
E-mail address: 209176@csu.edu.cn (Q. Yuan).

https://doi.org/10.1016/j.conbuildmat.2017.11.050
0950-0618/

with a reversible process. Strictly speaking, thixotropy is not applicable to cement-based materials. As stated above, structural build-up at rest, which covers reversible and irreversible effects, is of more interests in many applications. Thus, structural build-up at rest is more applicable to cement-based materials than thixotropy. However, reversible effects stemming from colloidal flocculation may dominate for the first hour, and cement hydration, which is irreversible effects, might be ignored [11,12]. In certain sense, the terminology of thixotropy and structural build-up are often interchangeable in the early age of fresh cement-based materials. It seems that most researchers preferred to use thixotropy, and a few researchers used structural build-up. It is worth to note that there are differences in the terms of thixotropy and structural build-up. For the purpose of comparing with literature, these two terms only refer to the process of structural build-up of cement paste in this study, and thus they are interchangeable.

Numerous studies have been carried out on the thixotropy of concrete. Generally, four methods have been used to measure the thixotropy of materials, i.e. hysteresis, stepwise changes in shear rate or shear stress, start-up or creep tests, and dynamic moduli. Quanji et al. [13] used hysteresis area between up-curve and down-curve to represent the thixotropy of cement-based materials. Assaad [14] sheared concrete at different shear rate, and used the area between the initial shear stresses and the equilibrium stresses representing the thixotropy. Yuan et al. [15] used the storage modulus obtained from small amplitude oscillatory test to evaluate the structural build-up of cement paste. It was pointed out that dynamic moduli method is the most suitable method to follow the structural build-up [15]. However, it needs delicate and expensive rheometer which is not available in most concrete laboratories. Roussel et al. [1] proposed a thixotropic model for concrete, and the increasing rate of shear stress at rest over time was called thixotropic index (A_{thix}). Roussel et al. [1] classified self-compacting concrete (SCC) into three types based on A_{thix}: non-thixotropic SCC ($A_{thix} < 0.1$ Pa/s) , thixotropic SCC (0.1 Pa/s $\leq A_{thix} \leq 0.5$ Pa/s) and highly thixotropic SCC ($A_{thix} > 0.5$ Pa/s). The start-up test, also referred to as the static yield test or shear growth test, is the most often used approach for cement-based materials. Amziane et al. [16] developed a novel method to follow the structural build-up of cement-based materials, where a rough plate was placed in a fresh paste and the weight of the plate was continuously monitored. The weight of the plate decreased with time due to the structural build-up of fresh paste. Omran et al. [17] used portable vane method to evaluate the structural build-up at rest of SCC. Khayat et al. [18] evaluated five field-oriented test methods for the structural build-up at rest, including inclined plane (IP), portable vane (PV), undisturbed slump spread (USS), cone penetration (CP), and K-slump test methods.

Assad et al. [2] studied the effect of viscosity enhancing admixture (VEA) on the thixotropy of SCC, and found that the type, combination, and addition level of VEA and high-range water-reducing agent (HRWRA) had a significant effect on the thixotropy, and relatively low addition level of VEA could result in high thixotropy. Khayat et al. [3] reported the addition of HRWRA decreased the structural build-up rate of SCC. Khayat et al. [19] used a propylene carbonate-based thixotropy-enhancing admixture (TEA) to increase the thixotropy of SCC. Assad et al. [4] reported that the thixotropy of SCC with binary or ternary binder was much lower than that of SCC without supplementary cementing materials, and aggregate content decrease the structural build-up rate. Perrot et al. [20] studied the structural build-up of rigid fiber reinforced cement-based materials, and found that the structural build-up rate was increased by the incorporation of fibers and aggregates. Kawashima et al. [21] reported that purified attapulgite clays significantly increased the structural build-up rate of pastes, especially at early ages. Quanji et al. [13] found that nano-sized highly purified magnesium alumino silicate clay can significantly increase the structural build-up rate of pastes. Ahari et al. [22] examined the effect of supplementary cementitious materials (SCM) on structural build-up of SCC, and found that except slag, the use of fly ash, silica fume and metakaolin in SCC mixtures increased thixotropy values in comparison with SCC without SCM. Rahman et al. [23] found that the addition of silica fume (2.5% replacement), limestone powder (15% replacement) and fly ash (10% replacement) increased the thixotropic rate significantly. Roussel et al. [11] ascribed high structural build-up to the CSH bridging effect between cement particles. Thus, they believed that the addition of particles known for their nucleation properties, such as silica fume and limestone, will increase the nucleation rate of CSH, and benefit structural build-up. Although there are a few studies dealing with the factors affecting the structural build-up of cement-based materials, the studies were based on different testing methods, and the results may not be comparable. Thus, it is necessary to comprehensively evaluate the effect of different mineral admixtures on the structural build-up of cement paste by using an appropriate method.

Although Jarny et al. [12] pointed out that the hydration of cement in the first hour can be ignored by using MRI velocimetry, it is well known that there is an initial heat liberation peak in the first hour of cement hydration [24]. According to Taylor [24], the initial heat liberation peak is ascribed to the exothermic wetting and early-age hydration, and a gelatinous coating on cement particles and rods of AFt phase are formed. Thus, it would be very interesting to see the relationship between hydration heat liberation and structural build-up at very early age, which is seldom reported in literature. In addition, a large amount of ions (Ca^{2+}, K^{+}, Na^{+}, SO_4^{2-}, OH^{-}) are dissolved into the pore solution in the first hour. The electrical double layer, which is very important for the interaction and flocculation of particles, is instantaneously developed on the particle surfaces. Consequently, the zeta potential may have an influence on the structural build-up of cement paste. The use of mineral admixtures makes the electrical double layer more complicated. Thus, it is also interesting to investigate the evolution of zeta potential of pastes containing various mineral admixtures with time, and its relationship with structural build-up.

In this paper, the effects of different mineral admixtures (silica fume, slag, fly ash, attapulgite, nano-calcium carbonate and nano-silica) on the structural build-up rate of cement paste were studied by static yield stress growth test and thixotropic area method. In order to provide more insightful evidence of structural build-up, calorimetric test was conducted to measure the hydration rate of cement paste, and zeta potential and conductivity of cement paste were also measured.

2. Materials and experimental programs

2.1. Materials and sample preparation

P II 52.5 portland cement which meets Chinese Standard GB175-2007 and a polycarboxylate-based high-range water-reducing agent (HRWRA) was used in this study. The mineral admixtures, including silica fume (SF), ground slag (GS), fly ash (FA), attapulgite (AG), nano calcium carbonate (NC) and nano silica (NS), were used to partially replace cement. The chemical compositions and physical properties of the powders are given in Table 1. The particle size distributions of cement, GS, FA and AG are determined by laser particle size distribution analyzer, as shown in Fig. 1. It can be seen that GS, FA, and AG are finer than cement, and cement is the coarsest powder. Since SF, NC, NS are very fine powders, the particle sizes cannot be characterized by laser particle size distribution analyzer.