Construction and Building Materials 93 (2015) 278–288

Contents lists available at ScienceDirect

Construction and Building Materials

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

Effect of mixing method on microstructure and rheology of cement paste

Dongyeop Han [a,1], Raissa Douglas Ferron [b,*]

[a] Engineering Research Institute, Seoul National University, 151-744, Seoul, South Korea
[b] Department of Civil, Architectural, and Environmental Engineering, The University of Texas at Austin, 78712 Austin, TX, United States

HIGHLIGHTS

- Influence of mixing intensity on rheological behaviors of cement paste was assessed.
- High mixing intensity can result in increasing the rheological properties.
- Plasticized pastes were more sensitive to effect of mixing intensity than non-plasticized pastes.
- Cement pastes prepared using high mixing intensity can be more agglomerated than pastes prepared using low mixing intensity.
- Effects of the mixing intensity also affected hydration.

ARTICLE INFO

Article history:
Received 2 February 2015
Received in revised form 1 May 2015
Accepted 29 May 2015
Available online 8 June 2015

Keywords:
Mixing
Microstructure
High shear mixer
Rheology
Flocculation

ABSTRACT

Mixing is one of the most important processes for producing concrete. The influence of different mixing methods on the rheological properties and fresh state microstructure of cement pastes were evaluated. In this work, the mixing process of cement paste was based on two different sample preparation methods: ASTM C305 and ASTM C1738. ASTM C1738 uses a high shear mixer, whereas ASTM C305 uses a planetary mixer to homogenize cement paste. A considerable increase in the rheological properties was seen in pastes prepared using the ASTM C1738 protocol versus those prepared according to ASTM C305, especially when a superplasticizer was incorporated. Not only were the rheological properties affected, but differences in hydration kinetics and fresh state microstructure were also observed, with mixtures prepared with ASTM C1738 generally displaying more flocculated microstructural features and accelerated hydration kinetics than mixtures prepared with ASTM C305.

1. Introduction

The mixing process is one of the most significant factors governing the properties of concrete [1]. Mixing not only homogenizes the composition of concrete; it also plays a significant role on the rheological properties of fresh concrete [2]. Numerous studies have been published regarding the influence of mixing intensity on the rheological properties of cement-based mixes [2–5]; generally as the mixing intensity increases, the viscosity and yield stress of concrete decreases. In addition, the shear forces generated during mixing also influence the agglomeration strength amongst cement particles [6] and the morphology of the hydration products on the surface of cement particles [7]. Mixing intensity is affected by many factors, including, but not limited to the type of mixer, mixing speed, and mixing time. In the lab, concrete is often mixed for approximately 8 min [8], however the mixing time of concrete in the field differs drastically. For example, in a central-mixed batch plant, concrete may be mixed in a stationary mixer for as little as 60 s prior to being discharged into a concrete truck agitator [9,10]. Thus, the concrete undergoes additional mixing after it is mixed in the plant due to the mixing that occurs in the concrete truck as it is being transported from the plant to the site. The length of mixing and degree of agitation that the concrete is subjected to during the delivery process can vary, while 90 min is a common limit for the maximum mixing time; allowable mixing times can range from 30 to 120 min and can be dependent on temperature [11]. The length of the mixing is an important factor to consider as this can influence microstructure and rheological properties of cement paste in concrete [12–14]. Prasittisopin and Trejo [11] observed an increased 28-day porosity and saw changes in the hardened state microstructure of mortars that were subjected to increased mixing time and increased mixer revolutions. Furthermore, with the increasing incorporation of various

* Corresponding author. Tel.: +1 512 232 2691.
E-mail addresses: donald.dyhan@gmail.com (D. Han), rferron@mail.utexas.edu (R.D. Ferron).
[1] Tel.: +82 10 5685 2009.

http://dx.doi.org/10.1016/j.conbuildmat.2015.05.124
0950-0618/

admixtures into concrete, additional mixing time is often needed to aid with admixture dispersion. For example, self-consolidating concrete may require an additional 30–90 s more of mixing versus conventional concrete [15].

All concrete, whether it is for the field or the lab, must be mixed; yet the mixing process is probably one of the least areas studied with respect to the life cycle of concrete. Ideally the sample preparation technique used in the experimental test should simulate the processing conditions that would occur in the field. ASTM C305 [16] is a popular standard used to prepare cement paste samples in the lab. This preparation technique uses a planetary mixer to prepare the samples, but studies have shown that the shear rates generated in this mixer are not representative of the shear rates that the cement paste matrix experiences during the mixing of concrete [2,17,18]. Cement paste in concrete is subjected to higher shear rates than that imparted by the ASTM C305 procedure because of the ball-milling action of the aggregates in concrete [19]. Williams et al. evaluated the effects of hand mixing, mixing with a Hobart planetary mixer, and mixing with a Ross high shear mixer on the rheology of cement paste, and determined that the shear rate experienced by cement paste in concrete mixture is most similar to the shear rate experienced by cement paste mixed with the Ross high shear mixer [3]. Recently, another standard, ASTM C1738 [20], has been introduced for preparation of cement pastes. Different from ASTM C305, this standard uses a high shear mixer for preparation of cement pastes. Although ASTM C1738 was introduced as a method to prepare pastes for rheological evaluation, since it is a sample preparation technique, in theory it can be used for preparing samples for other evaluations (e.g. setting time, calorimetry, etc.) in which simulating the conditions of the paste in concrete is important [2,18,20].

Limited studies have been conducted to examine how mixing variables affect early-age characteristics of cement-based mixtures as summarized in Table 1. The objective of the research presented in this manuscript was to compare how ASTM C305 and ASTM C1738 procedures influence the microstructure, hydration and rheological properties of cement paste. In addition, the influence of additional mixing time on the rheological properties was also taken into consideration. The evaluation of the rheological properties and the microstructure of cement pastes were conducted using a rheometer and a focused beam reflectance measurement (FBRM) probe, respectively. The FBRM probe uses backscatter light principles to measure the chord length of particles in a suspension [6,21,22]. The FBRM technique enables the user to conduct the test without diluting the cement paste suspension [6] and provides a way to indirectly observe the dynamic microstructure of cement paste during mixing.

2. Experimental plan

2.1. Materials and mix proportions

A commercially available Type I/II cement, conforming to ASTM C150 [23], was used in all of the mixtures, and according to the manufacturer of the cement, the cement had a Blaine fineness of 401 m^2/kg, an initial setting time of 100 min, and a specific gravity of 3.15. The oxide composition of the cement is given in Table 2. A commercially available Type F polycarboxylate superplasticizer (SP), conforming to ASTM C494 [24], was used in most of the mixtures. The solid concentration of the admixture was reported by the manufacturer to range between 29% and 36%. All of the mixtures were prepared using deionized water.

The mixture proportions were based off of a neat paste that was proportioned to have a solid volume content (vol.% solid) of 0.45, which corresponds to a water-to-cement ratio (w/c) of 0.39. This paste was considered the “control” mixture. Three additional mixtures with different vol.% solid and containing SP were also prepared (see Table 3). For the mixtures incorporating SP, the SP dosage was set to 0.20% by mass of cement, and the water content of these mixtures were corrected to take into account the water added due to using the SP.

Table 2
Oxide composition of the cement.

Items	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O
Amount (%)	20.70	4.50	3.30	65.10	1.40	2.60	0.18	0.48

Table 3
Mixing proportions.

Sample name	vol.% solid	w/c	SP included	Quantity for 1 L paste		
				Cement (g)	Water (g)	SP (g)
045	0.45	0.39	No	1417.50	550.00	–
045_SP	0.45	0.39	Yes	1417.50	547.34	2.84
047_SP	0.47	0.36	Yes	1480.50	527.22	2.96
050_SP	0.50	0.32	Yes	1575.00	497.04	3.15

Table 1
Overview of research pertaining to the effect of mixing methods on rheology and fresh state microstructure.

Researchers	Mixing method	Rheological properties investigated	Fresh state microstructure	Refs.
Yang et al.	Hand-mixed Paddle mixer (300 rpm) High shear blender (3000 rpm)	Yes, shear stress	Yes, ESEM*	[2]
Williams et al.	Hand-mixed Paddle mixer (140–285 rpm) High shear blender (2500 rpm)	Yes, viscosity; thixotropy	No	[3]
Roy et al.	Hand-mixed Spindle (100 rpm) High shear blender (9000–17,000 rpm)	Yes, viscosity; yield stress	No	[4]
Dils et al.	Concrete mixer (1.4–1.6 m/s)	Yes, Flow curve behavior	No	[5]
Ferron et al.	Hand blender (low speed)	No	Yes, FBRM	[6]
Rößler et al.	Planetary mixer (140–285 rpm)	Yes, torque	Yes, ESEM	[7]
Prasittisopin et al.	Planetary mixer (140–285 rpm)	No	No	[11]
Vandanjo et al.	Concrete mixer (23.1 rpm)	No	No	[14]
Ferraris et al.	High shear blender (4000–10,000 rpm)	Yes, viscosity; yield stress	No	[17]
Juilland et al.	Hand-mixed High shear blender (200–2000 rpm)	No	Yes, ESEM	[40]
Vlachou et al.	Propeller mixer (4000–12,000 rpm)	No	Yes, ESEM (frozen sample)	[43]
Vlachou et al.	Propeller mixer (4000–12,000 rpm)	Yes, viscosity	Yes, ESEM (frozen sample)	[44]
Lei et al.	Hand-mixed	Yes, yield stress	Yes, SEM**	[45]

* ESEM: Environmental scanning electron microscopy.
** SEM: Scanning electron microscopy.