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Influence of bleeding on properties and microstructure of fresh and hydrated Portland cement paste

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HIGHLIGHTS

- Gypsum particles are rise to upper part of fresh cement paste during bleeding.
- Ettringite crystal is formed in the upper part of cement paste after hydration.
- Segregation of gypsum and clinker particle due to the size and density difference.
- Bleeding impairs strength and anti-penetration ability of hydrated cement paste.
- Segregate results in color variation due to hydration product color difference.

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ABSTRACT

The influence of bleeding on slump flow and solid phase distribution of fresh Portland cement paste, as well as the hydration product distribution, splitting behavior, and porosity of the hydrated cement paste were investigated. It is manifested that bleeding results in heterogeneous distribution of solid phases in fresh cement paste, clinker particle is descended due to gravity and the suspension made of liquid and gypsum particle is impelled to ascend. This results in the abundance of gypsum particle and ettringite crystals in the upper part of fresh and hydrated cement paste respectively. Meanwhile, bleeding can impair mechanical property and anti-penetration ability of the hydrated cement paste.

1. Introduction

Fresh Portland cement paste is a heterogeneous system, consisting of a liquid phase, made of water and soluble chemical admixtures (dispersants, air bubble stabilizers, set retarder, set accelerator, etc.), and various solid phases, such as particles of clinker, calcium sulfate (gypsum, anhydrite or hemihydrate gypsum), and SCM (fly ash, slag, silica fume, etc.). The solid phases are usually of different apparent density and different particle size distribution. In cement paste, organic molecules can be absorbed on the surface of the solid particles, changing the particle interaction and particle-water interface characteristic. Under the effects of gravity, Brownian motion, particle interaction and liquid-solid surface tension, the liquid phase and solid phases tend to move to their balance position, resulting in the phenomena of bleeding and segregation.

Bleeding is defined as the movement (relative to solid particle) of free water to the upper direction of a fresh mixture. Segregation is defined as the separation of constitutes of a heterogeneous mixture. For cement paste, bleeding and segregation can result in non-uniform distribution of particles and hydration product in fresh and hydrated cement paste.

In practice, during the bleeding process of a fresh cement paste, not only water, but also fine particles can be floated up to the upper part or surface of the cement paste, resulting in the simultaneous behavior of bleeding and segregation. The float up of fine particle in fresh cement paste is generally resulted from the descending of big particles (bigger than the sustaining ability of non-Newtonian fluid), impelling the suspension made of liquid and fine particles to rise up. Moderate bleeding (bleeding rate equals to evaporating rate) is beneficial for mitigating the risk of plastic shrinkage cracking [1]. However, over bleeding (bleeding

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Table 1
Chemical components of Portland cement.

Oxide (wt%)									LOI (wt%)
CaO	SiO_2	SO_3	Fe_2O_3	Al_2O_3	MgO	K_2O	Na_2O	TiO_2	
60.20	21.18	4.40	4.04	3.97	2.49	1.03	0.15	0.28	1.95

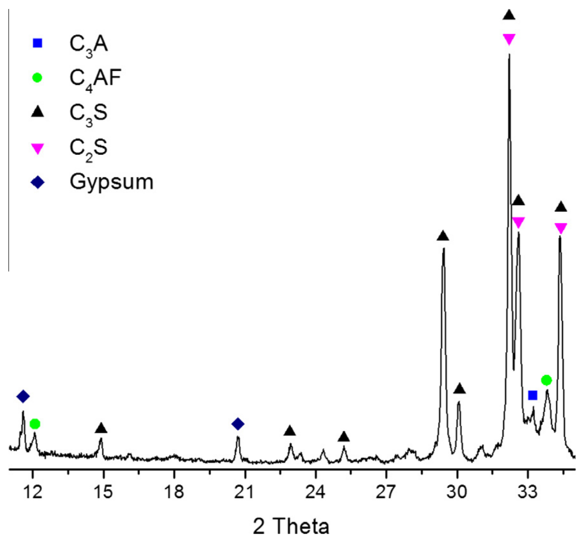

Fig. 1. XRD pattern of Portland cement.

rate significantly overwhelms evaporating rate) can impair mechanical property, anti-penetration ability of cement paste and bonding ability of paste with rebar or aggregate [2].

Bleeding of cement-based materials has received quite intensive studies, mainly focused on bleeding rate measurement or modeling. Powers assumed the bleeding process of cement paste as the sedimentation of suspended solid particle, and proposed that there were two bleeding stages: a period of constant bleeding rate, followed by a period of diminishing bleeding rate, controlled by cement particle specific surface area, water content and immobile water layer on cement particle [3]. Wainwright et al. researched the bleeding behavior of cement pastes made of different cementitious materials, and revealed the influence of cement content and particle size distribution on bleeding rate; the research showed that increasing cement content and narrowing cement particle size distribution were beneficial for lowering bleeding rate of concrete [4]. Based on a self-weight consolidation model, Josserand et al. considered the bleeding of concrete as an aging consolidation process, revealed that bleeding of concrete depends mainly on cement content and temperature during hydration, and the increase of cement content and temperature can benefit the aging process and diminish the bleeding rate [5]. Morris et al. has proposed a linear finite and small-strain analytical solution to determine the bleeding rate of cement paste, mortar and concrete, considering the effect of hydration and setting time [6]. Jae Hong Kim et al. researched the external and internal behavior, the amount and rate of bleeding of cement paste, showing the effect of water to binder ratio, SCM, oedometric modulus and diffusivity of cement paste on bleeding [7,8]. By considering the effect of particle interaction, Brownian motion and gravity, Perrot et al. characterized the bleeding tendency of cement paste, and reported that the volume and particle size of cement as well as the absorption of dispersants on the cement particle surfaces can influence the bleeding tendency of cement paste [9]. Peng et al. investigated the influence of water to cement ratio, admixtures and filler on sedimentation and bleeding of cement paste, and noticed the floating particle on bleeding water and the soft transit layer underneath the bleeding water in cement paste with high dosage of super plasticizer (Fig. 11 of the paper), without providing further explanation and identification of these phases [10].

In this paper, the segregation of solid phase in fresh cement paste due to bleeding and the influence of bleeding on hydration products, porosity, and mechanical property of hydrated cement paste are investigated.

2. Materials and sample preparation

Portland cement was used, its chemical components are given in Table 1 and mineral components are shown in Fig. 1. It can be seen that gypsum is used as setting regulator in the Portland cement.

A Polycarboxylate water reducing admixture (WRA), with solid content of 20% (by weight), was used. Deionized water, with resistivity larger than 10.0 MΩ cm was used.

Two cement pastes, with and without WRA, were studied. As show in Table 2, both of the cement pastes had a water to cement ratio (W/B) of 0.385. The purpose of introducing WRA to PCP-2 is to make the cement paste status to be bleeding and segregated. The pastes were prepared using a blade mixer (rotating speed of 780 rpm). First, cement paste without WRA (PCP-1) was prepared by mixing for 3 min and the slump flow was measured. Then, cement paste with 0.769% of WRA (PCP-2) was prepared by mixing for 3 min, after mixing, it was visually observed that PCP-2 had significant bleeding and segregated, slump flow was measured and specimens for solid phase distribution, hydration product distribution, mechanical property and porosity measurement were prepared.

Right after mixing, slump flow of PCP-1 and PCP-2 were evaluated using a mini-slump cone (top diameter = 35 mm, base diameter = 60 mm, height = 60 mm).

After the slump flow test, PCP-2 was re-mixed for 1 min. A part of the cement paste was taken out and cast in 3 plastic cuvettes (Fig. 2) right after the mixing. The rest of the cement paste was kept in the container for the next procedure.

The 3 plastic cuvettes cast with cement paste of PCP-2 were placed in a plastic bag, then vacuumed and sealed. After that, the plastic cuvettes were kept in upright position and held still, so that the bleeding and hydration processes can proceed without disturbance.

The cement paste of PCP-2 left in the container was undisturbed for 10 min to carry out bleeding process. After that, the container was tilted at 45° for another 2 min. At this moment, light-brown colored fluid and the dark-gray colored sediment were observed in the container (Fig. 4). Then, about 20 g of paste from the fluid part and the sediment part were taken out for vacuum oven dry, to investigate the segregation of solid phase caused by bleeding. The pastes were spread in a corundum plate and vacuum oven dried (T = 60 °C, P = −0.098 MPa) for 48 h. After that, the specimens were grounded and sieved through an 80 μm sieve for XRD test.

After 28 days of hydration, hydrated cement paste specimens in the 3 plastic cuvettes were taken out by carefully cutting off the plastic cuvettes along axis; the size of the 3 hydrated cement paste cylinders was 13.2 mm in diameter and about 65 mm in length. One of the cylinders was used for investigation of the hydration product distribution along the axis (bleeding direction); one of the cylinders was used for investigation of color variation along the axis; and one of the cylinders was used for investigation of mechanical property and porosity along the axis.

For investigation of hydration product distribution, two 5 mm-thick discs were cut from the top and bottom ends of the hydrated cement paste cylinder, microstructure was examined by scanning electron microscope (SEM), and hydration product type was examined by X-ray Diffraction (XRD). Specimens prepared for SEM observation were oven dried at 60 °C for 6 h and sputtered with carbon.

Table 2
Cement pastes mix proportion.

No.	Cement (kg)	Water (kg)	W/B	WRA (kg)	Dosage of WRA (wt%)	Cement paste status (visual inspection)
PCP-1	5.20	2.00	0.385	0	0	Homogenous
PCP-2	5.20	1.97	0.385	0.0400	0.769	Bleeding and segregated