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Bleeding and sedimentation of cement paste measured by hydrostatic pressure and Turbiscan

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Sedimentation and bleeding of cement pastes with lignosulfonate were studied by visual observation, HYdroStatic Pressure Test (HYSPT) and Turbiscan measurements showing two bleeding stages: a fast initial phase followed by a phase with diminishing sedimentation rate. A turbid bleeding zone establishes during the fast bleeding phase and the top layer gradually becomes transparent in the diminishing phase within minutes or hours depending on admixture and solid fraction. The bleeding rates measured visually and by HYSPT in the first 2 h are higher than the ones calculated by Kozeny-Carman Equation, whereas turbiscan shows lower rates. Both HYSPT and Turbiscan monitor the particle and fluid fluxes and thus describe bleeding from turbid to clear zone respectively by observing the density variation of bulk paste or the change in optical density of the surface region. Lignosulfonate reduces bleeding by improving particle dispersion to various degrees depending on types and dosages.

1. Introduction

Instability phenomena of cement paste and concrete occur due to density differences between the basic constituents spanning from water with density 1000 kg/m^3 to solids with density 2700–3150 kg/m^3. Generally, bleeding of cementitious materials is the sedimentation consequence of temporarily suspended particles. In 1939 Powers [1] concluded that the major factors controlling bleeding of cement paste and concrete are the water content and the surface area of the solids. He also explained that bleeding occurs in two stages: a period of fast constant rate followed by one with diminishing rate. The bleeding rate during the constant period can be assumed to follow general laws derived from Darcy [2] and Poiseuille's law [3] of capillary flow [4] in a bed of fines and solid particles, leading to the Kozeny-Carman (K-C) Equation [5], as shown in Eq. (1). There, Q is the bleeding rate (m/s), ε is the paste porosity (m^3/m^3), k_c is the Carman constant ($k_c = 4.1$ according to Powers [5]), μ is the viscosity of the fluid phase (Pa·s), g is the gravity acceleration (m/s^2), σ is the specific surface of the particles (m^2/m^3), ρ_s and ρ_f are the densities (kg/m^3) of the solid particles and fluid.

$$Q = \frac{\varepsilon^3}{1-\varepsilon}\cdot\frac{\left(\rho_s-\rho_f\right)g}{k_c\mu\sigma^2} \qquad (1)$$

All of Powers research was based on visual observation on the movement of the interface between bleed water and segregated paste or concrete. However, novel research [6,7] shows that the bleeding front is not always sharp in modern concrete mixes due to slow sedimentation of fine particles and a preference for higher contents of fine particles combined with use of chemical admixtures such as superplasticizers (SP) and Viscosity Modifying Agents. Daczko [8] also proposed that a mixture can be unstable even without clear bleeding. More recent research [9] on cement pastes with initial solid volume fractions 0.3–0.5 (corresponding to w/c = 0.4–0.7) and varying powder content or admixtures addition show laminated bleeding zones with both clear bleeding layer and a soft turbid layer consisting of well dispersed fines. In-situ measurements of the w/c ratio by pipette [7] demonstrated substantial variation in the top surface zone. Therefore, the traditional term "bleeding" is about to lose its meaning for modern cement paste like in Self Compacting Concrete (SCC). This implicates that the properties of the hardened concrete at the surface can vary considerably from the bulk properties which may have large influence on both the durability and the surface quality.

For cement paste with high initial solid fraction, simple analysis of a single particle sinking in the fluid according to Stokes' Law [10]

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is no longer valid. Instead, multi-particle interactions must be taken into account. The presence of a large number of fine particles has four profound effects on the properties of cement paste. First, the motion of a single particle will be limited by the surrounding particles and thus the effective particle settling rate decreases. Secondly, an increasing volume fraction raises the viscosity and yield stress towards infinity. Thirdly, emergence of yield stress and associated percolated network also decreases the particle settling rate. Finally, the sum of inter-particle forces or flocculation state influences the sedimentation process [7,9]. In addition to the effect of solid fraction, the composition of solid phase and presence of admixtures will affect the settling. Furthermore, the reabsorption of bleeding water may also affect particle sedimentation and bleeding rate [10–12]. In Ref. [9] it was found that the Richardson-Zaki Equation (R-Z) [13] to some extent is applicable to describe the sedimentation in cement paste or filler modified matrix. It describes the empirical relation between Stokes terminal sinking velocity for a single particle and the effect of surrounding particles at arbitrary volume fractions (Φ). As multiple particles sink, various zones form [14]. Kynch [15] first described the movement of particles between different zones. Fitch [16] later included compression of the sediment cake and suggested a relationship between the settling rate U_p, the flocculation tendency and solid concentration Φ. In this research, zone formation in cement pastes by sedimentation are defined as follows: bleeding water collected on the top followed by a homogeneous concentration zone, a variable concentrated zone and finally a sediment cake at the bottom, as illustrated in Fig. 1.

Traditional experimental methods to measure bleeding [17–21] rely on the measurement of visual bleeding depth or total bleeding and therefore cannot be used without a clear bleeding front. Microscopy or spectroscopy may be used to study the variation of concentration and the consequent bleeding, but the upper limit in solids fraction is only around 0.1. Therefore, the development of new test methods is important to investigate the bleeding of cement paste with normal solid fraction. The applicability and repeatability of HYSPT for fresh cement paste was explained in Ref. [7]. There is a reasonable correspondence between bleeding rate measured with pipette and calculated from hydrostatic pressure gradient. In the calculation of the bleeding rate from dP/dt, the pressure gradient at a specific depth, bleeding could be assumed to be a sharp front and a homogeneous zone beneath it [7]. HYSPT also indicates the paste flocculation state which relies on that at a given initial Φ_0 better dispersed particles sink slower than flocculated ones. The bleeding depth was calculated according to Eq. (2), where P (h,t) is the detected pressure (Pa) at the height to paste surface h (m) and time t (s), h_b is the bleeding depth (m), Φ_0 is the initial solid fraction (in homogeneous zone Φ is assumed to be equal to Φ_0), ρ_p and ρ_f are respectively the mass densities of the particle and fluid (kg/m^3).

$$P(h,t) = \rho_f g h_b + \left[\rho_p \Phi_0 + \rho_f (1 - \Phi_0)\right] \cdot g \cdot (h - h_b) \tag{2}$$

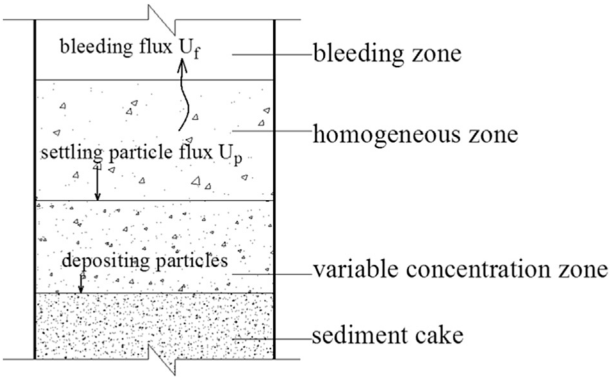

Fig. 1. Different zones in suspension during sedimentation.

Time dependent turbidity measurement on the bleeding layer is the other method employed in this research to study sedimentation and formation of a bleeding layer. A Turbiscanner was used to measure the turbidity as a function of time for sedimenting cement paste. This apparatus has been used to detect sedimentation of dispersed or flocculated particles and the consequent bleeding of emulsions and suspensions, but rarely applied for cement paste. Meunier [22] first described it as a new concept to evaluate stability in concentrated colloidal dispersions. Later Meunier et al. [23,24] and more recently Lemarchand et al. [25] applied it to analyze stability of emulsions or suspensions. The effect of polycarboxylate (PCE) superplasticizer on supernatant of suspensions was observed with Turbiscan in the research by Autier et al. [26], but the solid fractions of the suspensions are much lower than normal cement paste. Here we for the first time attempt to use the intensity variation of the transmitted light to investigate bleeding of cement paste with practical w/c between 0.4 and 0.7.

As mentioned, modern cement pastes often have a high content of fine particles and various admixtures which reduce the particle sedimentation rate and cause turbid bleeding above the denser bulk paste. Turbid bleeding is hard to observe by traditional bleeding test methods and often not addressed in research about bleeding phenomena. The scope of this work is to investigate the mechanism of bleeding, the development of a turbid bleeding zone and the effect of lignosulfonate (LS) representing a traditional plasticizer, on this process. For this purpose, sedimentation and bleeding of neat cement pastes with or without LS were measured by observing the variation in hydrostatic pressure of fresh paste as well as by optical scanning of the turbid bleeding zone by Turbiscan. Both methods follow the evolution from fresh homogeneous suspension to segregated paste with a combined clear-turbid bleeding zone and a dense sediment cake. In addition, the bleeding rates measured with the two new methods are compared with those from traditional visual bleeding tests and the K-C Equation. Hopefully the research on the turbid bleeding and the application of new measurement methods can improve the understanding of how bleeding occurs in modern cement paste.

2. Materials and measurement methods

2.1. Materials

The recipes of the samples for all measurements are shown in Table 1. Four types of LS, described by a DP-number are applied with main characteristics shown in Table 2, where Mn is the number average molecular weight, Mw is the average molecular weight. All the LS dosages are solid dosage by cement weight (% sbwc). The cement used in this research is Norwegian standard Portland cement STD-CEM I with main chemical and physical characteristics shown in Table 3. Paste samples for Turbiscan experiments were prepared by weighing cement, water and LS into a beaker. The paste was stirred by an overhead mixer with a U-shaped impellor for three min at 200 rpm, kept at rest for five min followed by mixing at 200 rpm for two more min. Around 20 ml paste was filled into the Turbiscan vial with 2.5 cm diameter (filling height: 4.5–5 cm) and placed into the instrument. The Turbiscan measurements normally commenced around 15 min after first contact between cement and water. Around 3 L of cement paste was mixed separately in a 5 L Hobart mixer for HYSPT and visual bleeding tests with the following mixing procedure: the dry