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Measurements of Breakdown and Buildup of Thixotropy in Cement Pastes Containing Diatomaceous Earth

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Abstract: This paper proposes a comprehensive series of protocols that can make quantitative measurements on both breakdown and buildup aspects of thixotropic behaviors of cement pastes. While measuring breakdown and buildup separately, the correlations between these two aspects have also been investigated. Cement pastes with different water-to-binder ratios (w/b=0.4, 0.5 and 0.6) and different replacement levels of Diatomaceous Earth (DE) (0%, 2%, 6% and 10% by weight of cement) were studied. To simulate the real on-site conditions, parameters such as pressure and temperature were also considered during the measurement. The research found that increase in DE replacement dosage can reduce the energy required to breakdown thixotropic structure of cement pastes while no specific trend on buildup aspect was observed. Among the three used w/b ratios, the 0.5 w/b ratio has the most reduction effect in breakdown energy, where using 10% DE can lead to 65% of energy reduction in breakdown compared to the control sample. However, the measured yield stress for these specimens were very similar to each other. In addition, increase in temperature significantly increases the buildup ratio of cement pastes while increase in pressure as high as 20 MPa has a minimal impact on thixotropic properties of cement pastes.
Keywords: Thixotropy; Cement paste; Diatomaceous Earth; Structural breakdown; Structural buildup.

1. Introduction

Thixotropic materials, due to their microstructure, show complex rheological behavior. These materials are usually suspensions with high concentrated solid particles. The inter-particle forces between solid particles such as van der Waals attraction, electrostatic repulsion and steric hindrance result in formation of flocs which normally evolve into a space-filling particulate network [1]. The inter-particle bonds are, however, weak enough to be broken by the mechanical stresses that occur during flow. The result is that during flow the network breaks down in separate flocs, which decrease further in size when the shear rate is increased. However, reduce or stop in the shear rate can cause a growth of the flocs; i.e. the particulate network will rebuild at rest or lower shear rates.

Thixotropy is a common phenomenon for cementitious materials that includes both structural breakdown and buildup. The breakdown aspect of thixotropy can help to explain how the flow behavior of paste matrix can impact concrete pumping and the buildup aspect of thixotropy can help to estimate the lateral pressure on formwork and the concrete's resistance to segregation. Thus, a comprehensive understanding of thixotropic behaviors of cementitious materials requires a distinctive understanding of both aspects.

Hysteresis loop has been the most common method of measuring thixotropy of cementitious materials in last few decades [2-7]. In this method, the area between two subsequent flow curves, an up-curve followed by a down-curve, is measured and linked to thixotropic characterization of material. During the up-curve, shear rate is gradually increased from 0 to a maximum value which breaks down part of the already formed structure. Then, in down curve, the shear rate is gradually reduced from the maximum value back to 0, which let the sample to rebuild part of its breakdown structure. The area formed by the up and down curves is often used to quantify thixotropy of the material.

Although hysteresis loop is a quick quantitative method, it does not give an intrinsic value of any physical rheological parameters. Not only the shear history in the material is not considered, but also either in up-curve or down-curve, the sample does not reach to an equilibrium condition. Thus, test results from hysteresis loops are very dependent on experimental conditions such as the maximum shear rate and the rate of increase/decrease in shear rate. The measured area between hysteresis loop's flow curves do not reflect the breakdown and build-up rates separately. Therefore, to better understand the thixotropic behavior of cement paste, a better measuring protocol is needed.

Some modified methods based on hysteresis loop has been proposed in a try to address and minimize its deficiencies. For example, Ferron et. al [8] suggested a multiple hysteresis loop method. In this method, first, an equilibrium loop is conducted on the sample. Then, successive hysteresis loops at different resting times are conducted and the area between the up-curve of each hysteresis loop and the equilibrium line (specific rebuilding energy) is measured and plotted against resting time. The slope of this graph is considered as rate of rebuilding and is used to compare the rebuilding rate of different mixtures. Although the method minimizes some flaws of single hysteresis loop such as the influence of shear history from mixing, transporting, etc. by applying the equilibrium status, it fails to address the influence of shear history of each hysteresis loop on the subsequent loops. Also, the breakdown aspect of the thixotropy is not touched with this method. For cementitious materials, structural breakdown and buildup have different applications. As one represent material behaviors during construction and the other represents material properties after construction. A comprehensive understanding of thixotropic behavior of materials requires a distinctive understanding of both aspects. Therefore, more comprehensive methods to quantitatively measure both the breakdown and the build-up aspects of thixotropy are needed.

Diatomaceous earth (DE), on the other side, is a chalklike, soft, very fine grained, earthy, siliceous sedimentary material [9]. It is finely porous, less in density, essentially chemically inert with low thermal conductivity [9, 10]. DE consists of amorphous hydrous silica cell walls of dead diatoms (opal, $SiO_2 \bullet nH_2O$), which are microscopic single-cell aquatic plants (algae). There are wild ranges of applications for DE. In 2015 in US, 55% of diatomite was used in filter aids, 21% was used as cement additives, 14% was used as fillers, and 9% was used as absorbents. According to United States Geology Surveys (USGS), using DE as cement additives has increased significantly from 14% in 2014 to 21% in 2015 and now it is the second largest DE consumption in US [11, 12]. As a natural pozzolanic additive for concrete [13-15], it is believed that replacing Portland cement with DE to some levels can improve concrete properties, such as late age strength[16]. Because of its unique morphology, porous structure and high water absorption level, DE is excepted to influence properties of concrete especially its fresh properties, such as thixotropy.

In this paper, a series of protocols are proposed to quantitatively measure thixotropic behavior of cementitious pastes. These protocols are intended to give a more comprehensive and reliable understanding of thixotropic behavior. For this purpose, four different thixotropic protocols are proposed. The first two protocols address the breakdown aspect of thixotropy and the other two protocols address the buildup aspect of thixotropy. The quantitative results from these protocols not only make it possible to analyze breakdown and buildup, separately; but also, provide the opportunity to study the correlation between these two aspects of thixotropy. Factors related to raw material properties including solid concentration and solid types were considered in this study. Cement pastes with water-binder (w/b) ratios of 0.4, 0.5, and 0.6 were used. In some pastes, diatomaceous earth (DE) was used to partially replace cement (2, 6, and 10% of weight of Portland cement). DE is a natural pozzolanic additive that has a chalklike, soft, very fine grained, earthy, siliceous sedimentary material [9]. Because of its unique morphology, porous structure and high water absorption level, DE significantly influences fresh properties of concrete [17], and hence has a great potential to influence the paste's thixotropic properties when used. To simulate the ambient conditions (i.e. on-site conditions), influences of both temperature and pressure on thixotropic behaviors were also investigated. It was found that the applied pressure did not have a significant influence on thixotropic behaviors.

The four protocols proposed by this paper allow researchers to study the both the structural breakdown and the buildup aspects of thixotropy. These two aspects have influences on different concrete properties, such as its pumpability, flowability, and resistance to segregation, etc. Developing such measuring protocols to quantify structural breakdown and buildup by considering the raw material characteristics will assist better design, casting, and curing of concrete.

2. Experimental procedure

2.1 Materials

Ordinary Type I Portland cement was used in all the mixtures. The mean size of the cement particles was 11.4 μm, determined through a laser particle size analyzer. The Blaine surface area was 400.8 m^2/kg. The chemical compositions of this type I cement were measured with XRF and they are listed in Table 1.

DE used in this study was a commercial pure diatomaceous earth usually powder available widely on the market. The mean size of DE particles was 19 μm based on a laser particle size analyzer. And the Blaine surface area was 593 m^2/kg. The chemical composition of DE is also listed in Table 1.

2.2 Mixing procedure and testing matrix

The details of mix proportions are listed in Table 2, where "OPC" indicates control samples with ordinary type I cement only, and "D" indicates the pastes with DE replacement. To study the effect of solid concentration, three w/b ratios of 0.4, 0.5, and 0.6 were used. For each w/b ratio, three replacement levels that are typically used in literatures of DE were studied.

When mixing the control pastes (OPC-0.4, OPC-0.5 and OPC-0.6), water was gradually added to the cement over the first minute of mixing and then continued to mix for 2 additional minutes at a low speed of 136 rpm. The sample was then allowed to rest for 2 minutes, which was followed by another 3 minutes of mixing at high speed of 195 rpm.

For cement pastes with DE replacement, cement powder and DE were firstly dry mixed for a minute with low speed of 136 rpm. Then, the same mixing procedure used for control pastes was followed. Mixing and all following tests were conducted at 25 °C unless otherwise specified.

Table 1. Chemical composition of type I portland cement and diatomaceous earth

Compound (%)	Type I cement	DE
SiO_2	19.70	93.5
Al_2O_3	4.84	1.6
Fe_2O_3	3.05	1.1
CaO	62.62	0.4
MgO	4.00	0.05
SO_3	3.23	0.12
Na_2O	0.15	2.51
K_2O	0.49	0.09
LOI	1.21	0.35

Table 2. Mix proportions

paste	w/b	DE (%)	Cement (g)	DE (g)	Water (g)
OPC-0.4	0.4	0	700	0	280
D2-0.4	0.4	2	686	14	280
D6-0.4	0.4	6	658	42	280
D10-0.4	0.4	10	630	70	280
OPC-0.5	0.5	0	700	0	350
D2-0.5	0.5	2	686	14	350
D6-0.5	0.5	6	658	42	350
D10-0.5	0.5	10	630	70	350
OPC-0.6	0.6	0	700	0	420
D2-0.6	0.6	2	686	14	420
D6-0.6	0.6	6	658	42	420
D10-0.6	0.6	10	630	70	420

2.3 Equipment and testing methods

An Anton Paar rheometer (MCR 502) was used to conduct rheological tests. This rheometer can control both shear stress and shear rate. Also, it is capable of controlling temperature from room temperature up to 400 °C. For temperatures below room temperature, a water bath can be connected to the rheometer to achieve desired temperature. For purpose of this study, concentric cylinders with conical end geometry (cup and bob) with a gap size of 1.13mm was selected.

During pumping, concrete is exposed to hydraulic pressures. With modern high-rise super high pressure pumps, concrete pressures as high as 400 bar is possible [18, 19]. Therefore, to simulate the material behavior during pumping, a pressure cell is also used to conduct rheological tests at high pressures. The pressure cell has almost the same geometry as regular cup and bob (as shown in Fig. 1). In pressure tests, after the sample was placed, the pressure cell was tightly capped using high strength bolt. Then, the desired pressure was achieved by pumping compressed Nitrogen gas into the cell. In the pressure cell, there is no physical contact between the inner cylinder (bob) and the rotor and a magnetic holder is used to transfer torque to the bob. Through this magnetic coupling, the desired shear is applied on the sample. For this paper, two pressure levels of 20MPa (200 bar) and 10MPa (100 bar) were selected.

2.4 Measurement Protocols for Thixotropy Quantification

2.4.1 Protocol 1

This protocol is developed to measure how easily cement pastes' structures can be broken down to a steady state (equilibrium condition) under a high shear rate. This steady state is considered as the most deflocculated state that cement paste can practically reach under a certain high shear rate. At this state, the rate of flocculation and de-flocculation are assumed to be in balance. First, all samples were pre-sheared at a high speed of 300 s^{-1} for 10 s, followed by 3 min rest to minimize the effects of shear history so that all the samples start from the same reference point. Then, a constant high shear rate of 300 s^{-1} was applied on the samples for 20 min. A schematic diagram of protocol 1 procedure is shown in Fig. 2a. During this 20 min, the alternation of shear stress was recorded and plotted against time. Therefore, minimum time to reach to equilibrium condition (ΔT), drop in shear stress (Δτ) and break-down energy (BDE) can be calculated as shown in Fig. 2b.

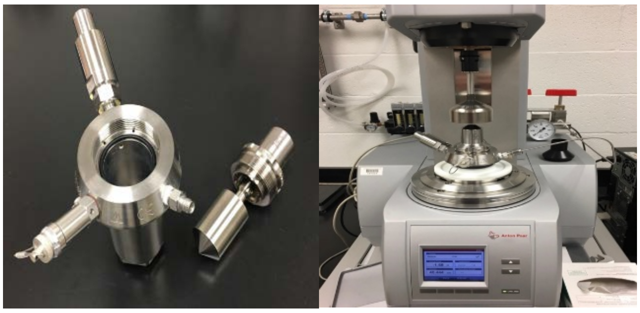


Fig. 1. Pressure cell and magnetic coupling

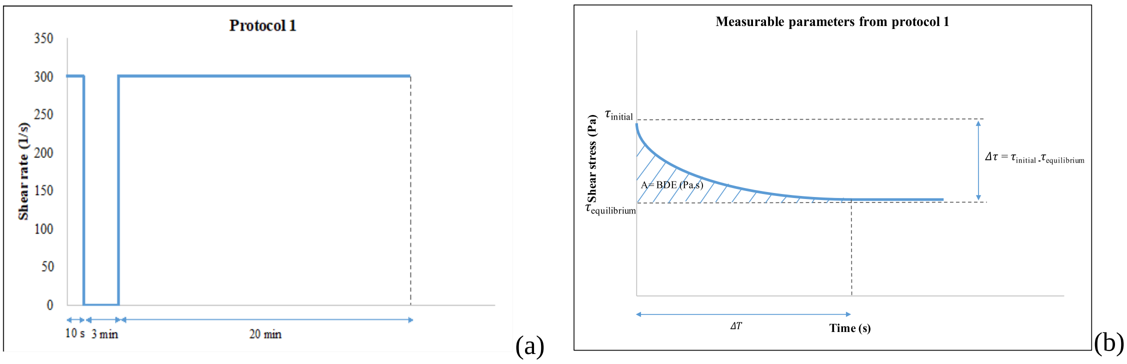
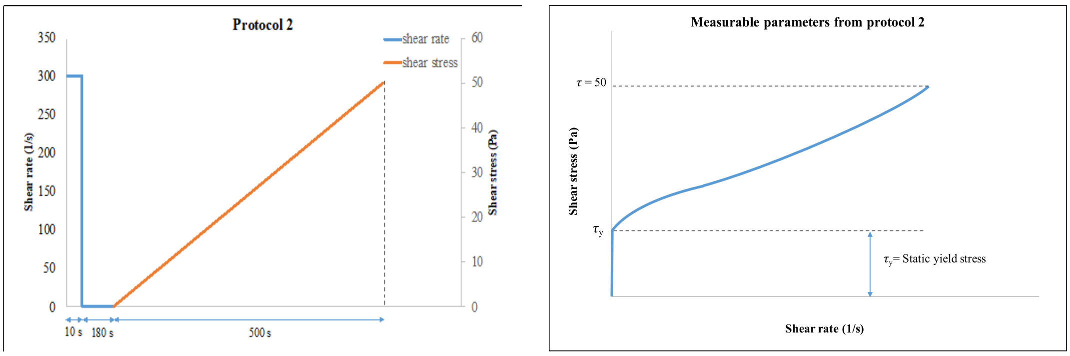


Fig. 2. Protocol 1 (a) Testing procedure (b) Measureable parameters

2.4.2 Protocol 2

This protocol is intended to measure the easiness to initiate flow in a cementitious paste. For this purpose, after usual 10 s of pre-shear at 300 s^{-1}, and 3 min rest, a simple shear stress ramp from 0 to 50 Pa (assuming 50 Pa is big enough to surpass the static yield stress of the measured paste) in rate of 0.1 pa/s is applied on samples. During this time, the alternation of shear rate is recorded and plotted against shear stress. Thus, the shear stress at which samples start to yield, static yield stress, are measured. A schematic diagram of protocol 2 procedures and an example of obtained data are shown in Fig. 3.



(a) (b)

Fig. 3. Protocol 2 (a) Testing procedure (b) Measureable parameters

2.4.3 Protocol 3

Protocol 3 is intended to calculate the buildup ratio of sample before and after its structural breakdown. In this protocol, after pre-shearing, a very small shear rate of 0.0001 s^{-1} is applied on the sample for 3 min and the buildup of shear stress is recorded. The shear rate is purposely chosen to be within its linear viscoelastic region (LVER) to prevent damaging the micro-structure of the cement paste. Then, a constant high shear rate of 300 s^{-1} is applied for 8 min to break the structure down to a steady state. After structural breakdown, the low shear rate of 0.0001 s^{-1} is applied again to monitor the rebuilding of structure. The measurement continues till sample reaches its initial state just before the breakdown step (see Fig. 4). By plotting the measured shear stress against time, buildup ratio and time to rebuild initial state are calculated as shown in Fig. 4b.

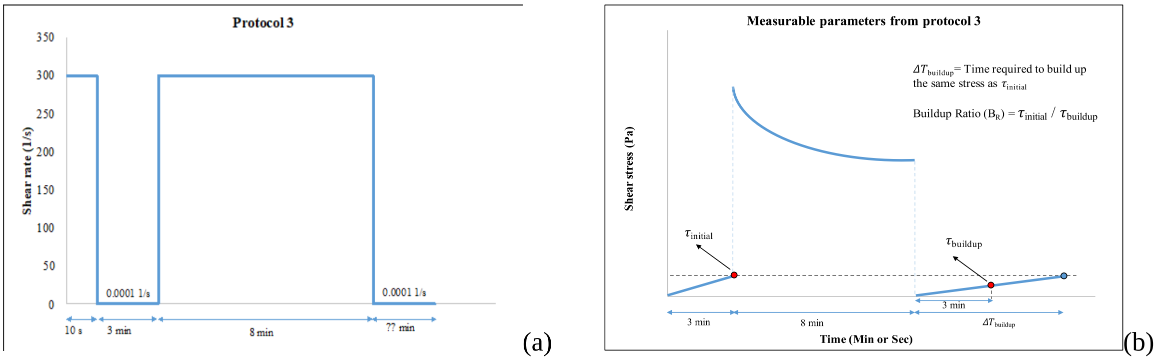


Fig. 4. Protocol 3 (a) Testing procedure (b) Measureable parameters

2.4.4 Protocol 4

This protocol is designed, based on protocol 2 and 3, to measure the rebuilding ratio of samples before and after the breaking down of their structure. In protocol 4, instead of monitoring shear stress buildup, the static yield stress before and after structural breakdown are monitored. As shown in Fig. 5, after pre-shearing and 3 min rest, a shear stress ramp with the rate of 0.1 Pa/s for 4 minutes is applied. This is followed by a constant high shear rate of 300 s^{-1} shear rate for 8 min like protocol 3 to achieve structural breakdown and then another shear stress ramp with the same rate of 0.1 Pa/s and 4 min is applied again. Thus, by plotting shear stress against shear rate two static yield stresses, one before structure breakdown and one after structure breakdown, are obtained. They have been used to calculate buildup ratio (see Fig. 5b).

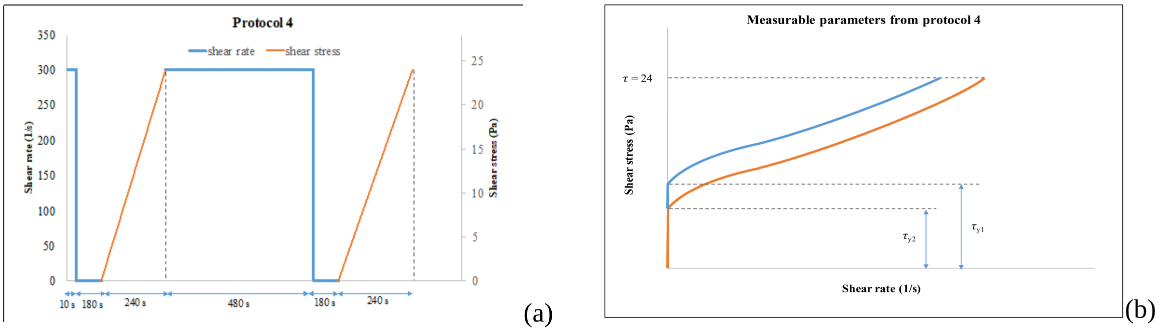


Fig. 5. Protocol 4 (a) Testing procedure (b) Measureable parameters

3. Experimental results and discussion

3.1 Structural breakdown

Fig. 6 shows the calculated breakdown energy of cementitious pastes (see shaded area in Fig. 2b) for each w/b ratio against their DE replacement level. It can be seen that by increasing DE content, breakdown energy of cement pastes with almost all w/b ratios was decreased. In other words, under a constant high shear rate, increasing DE content can help to reduce the needed energy to reach the steady state of the flow. So, it can be concluded that cement pastes with higher DE content are easier to break down. Also, from Fig. 6, by increase in w/b ratio at any DE replacement level, breakdown energy was decreased. This is reasonable because the sample with a higher w/b ratio has a loose microstructure with less solid particles to breakdown. Fig. 7 plots the drop of shear stress ($\Delta\tau$ in Fig. 2b) to reach the steady state against DE content. It can be seen that for each w/b ratio, pastes with higher DE content in general show slightly higher stress drop than those with lower DE contents. Also, cement pasts with higher w/b ratio have lower drop in shear stress. Again, this corresponds to the solid concentration in cement pastes. Fig. 8 shows the time to reach to steady state (ΔT in Fig. 2b) against DE content and w/b ratio. It can be seen that for a given w/b ratio, ΔT decreases with the increase in DE content. This indicates that a paste with a higher DE content is probably easier to breakdown. This can be attribute to the negative charges [16]. From Fig. 8, it can also be seen that almost for all DE replacement levels, increase in water content leads to longer time to reach to steady state. However, for 6% replacement level higher water content leads to slightly shorter ΔT. This indicate that 6% of DE content is probably a threshold that convert the breakdown aspect of thixotropic behaviors of the paste.

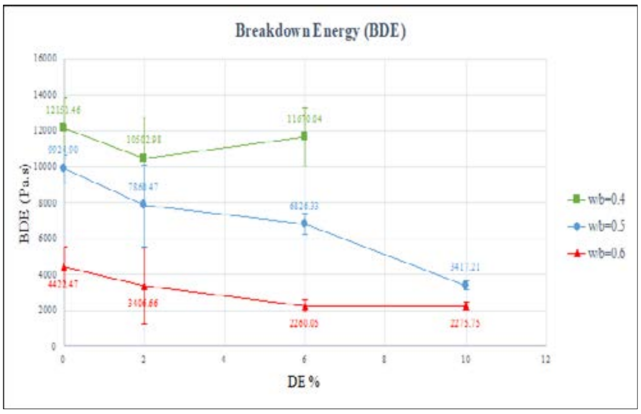


Fig. 6. Breakdown energy against DE replacement level from protocol 1

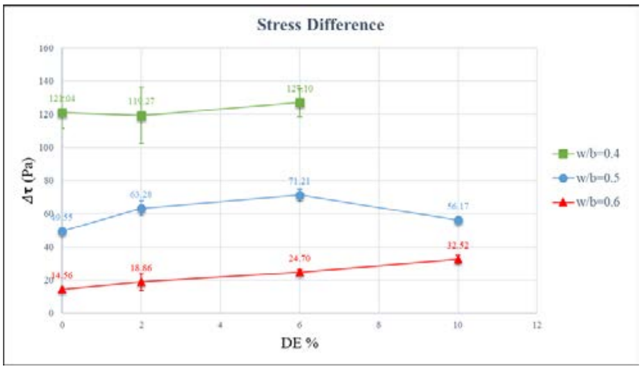


Fig. 7. Drop in shear stress against DE replacement level for protocol 1

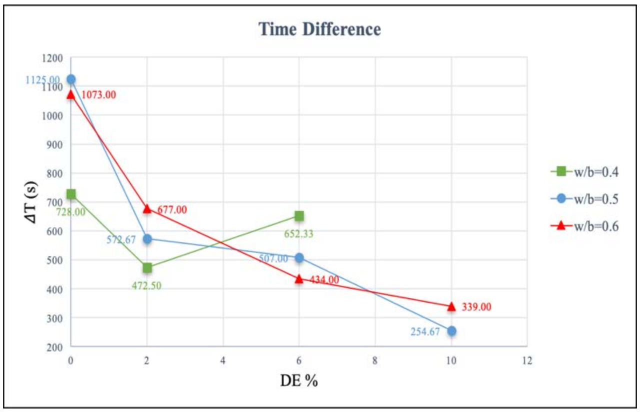


Fig. 8. Time to reach to steady state against DE replacement level for protocol 1

It should be noted that at higher DE levels, solid volume fraction increases due to larger surface area, lower specific gravity and higher water absorption of DE particles. This increase in solid volume fraction results in closer gaps between solid particles. These close gaps, especially between cement particles, leads to formation of a network of interconnected agglomerates at rest. When the high shear rate is applied, the resulted induced shear stress breaks this network and cause a quick and significant drop in shear stress. On the other hand, again due to high solid volume fraction, the inter-particle interaction between solid particles is high and thus, after a relatively short time, de-flocculation and flocculation rate reach to an equilibrium condition, i.e. cement paste reaches to its steady state. However, in lower DE levels, the gap between solid particles are larger and formation of such a fully interconnected network of agglomerates is more difficult. Therefore, de-flocculation happens in a lower rate and drop in shear stress is not as steep as pates with higher DE content. Also, due to lower inter-particle interaction between solid particles, it takes longer for these cementitious pastes to reach to their equilibrium condition. Therefore, overall, the breakdown energy required for cement pastes with higher DE content to reach to their steady state is lower than those with lower DE content.

Fig. 9 shows the static yield stress of all mix proportions against DE level and w/b ration from protocol 2. For 0.6 w/b ratio, static yield stress has not changed significantly for different DE contents. For 0.5 w/b ratio, a slight increase for 6% and 10% replacement levels is observed, but it's not significant. For 0.4 w/b ratio, not much a difference is seen till 2% replacement, however, from 2 to 6% replacement level, static yield stress increases drastically. This is to some extent in agreement with breakdown energy from protocol 1 (Fig. 6) which shows a slight increase from 2% to 6% at 0.4 w/b ratio. It can be due to very low water content of this cement paste caused by high DE level. The figure also shows that for all given DE replacement levels, static yield stress has decreased with increase in w/b ratio, as expected.

Also, it should be mentioned that although the concept of protocol 1 and 2 are both related to breakdown aspect of thixotropy of cement pastes, they measure two different parameters. What measured in protocol 2 is the critical amount of shear that is needed to initiate the flow (i.e. the starting point of breakdown) [20]. While, protocol 1 measures the easiness to achieve the steady state (i.e. the required input energy for breakdown). By comparing protocol 1 and 2, it can be concluded that increase in DE content of cement pastes can decrease the amount of total energy required to breakdown cement pastes' structures to their most practically possible deflocculated state (steady state), however, the minimum shear stress needed to initiate the flow (structure breakdown) may not change significantly with variable DE replacement.

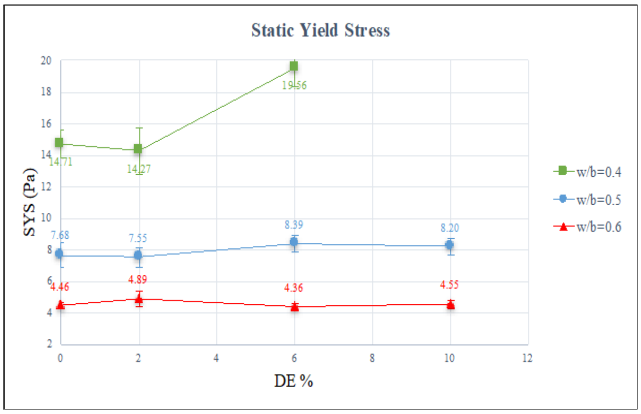


Fig. 9. Static yield stress against DE content from protocol 2

3.2 Structural buildup

Fig. 10 shows build-up ratio (BR) against DE replacement level from protocol 3. Pastes with different w/b ratio show different trends with increase in DE content. For 0.4 and 0.5 w/b ratios, an overall decrease in build-up ratio is observed when DE content is increased. While, for 0.6 w/b ratio, a significant increase in build-up ratio is observed from 0 to 6%, then decreased when 10% of cement was replaced by DE. The observed phenomenon may be explained by solid volume fraction. When DE content is increased, it absorbs larger amount of free water, leaving less space in between particles that contributes to a higher flocculation and build-up ratio. When a threshold DE replacement level is surpassed (e.g. 6% for w/b=0.4), the repulsive forces from DE particles become dominant due to the shortened gap between solid particles, which reduce the build-up ratio.

Fig. 11 shows the build-up times of all mix proportions from protocol 3 against DE replacement levels. From the figure, for w/b=0.5 pastes, it takes longer to build up stresses when DE content is increased from 0 to 6%, but once this threshold is passed, the buildup time decreases. Comparing the results from Fig. 10 and Fig. 11, a longer build-up time corresponds to a lower build-up ratio. This indicates roughly a linear trend between the build-up ratio and time.

Fig. 12 shows build-up ratios from protocol 4 against DE replacement levels for each w/b ratios. Again, for 0.6 w/b ratio, the build-up increases with DE content till 6% and then decreases. For 0.4 and 0.5 w/b ratios, an opposite trend is observed. Apart from the fact that OPC-0.5 and D2-0.5 show unexpectedly higher build-up ratios, the overall trends are in very good agreement with the results obtained from protocol 3.

3.3 Breakdown and build up correlation

By comparing the results from protocol 1 and 2 (breakdown) with the results from protocol 3 and 4 (build-up), no clear correlation between breakdown and build-up aspects was observed. Both 0.5 and 0.6 w/b ratios show a drop in breakdown energy by increase in DE content from protocol 1 and no significant change in static yield stress from protocol 2. But when it comes to buildup ratios from protocol 3 and 4, at least till 6% replacement level, 0.5 w/b ratio shows a continuous drop in build-up ratio, while 0.6 w/b ratio shows a continuous increase. Therefore, it can be concluded that to fully understand thixotropic behavior of cementitious pastes under different conditions, both breakdown and build-up aspects of their thixotropic behavior should be investigated. Any assumption based on only one of these two aspects can lead to misunderstanding of cementitious pastes characteristics.

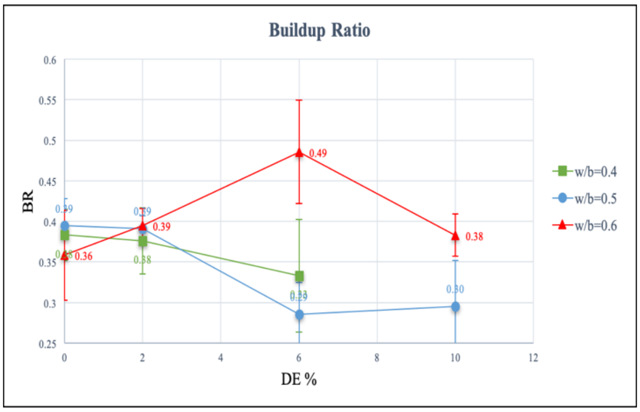


Fig. 10. Build-up ratios from protocol 3 against DE replacement levels

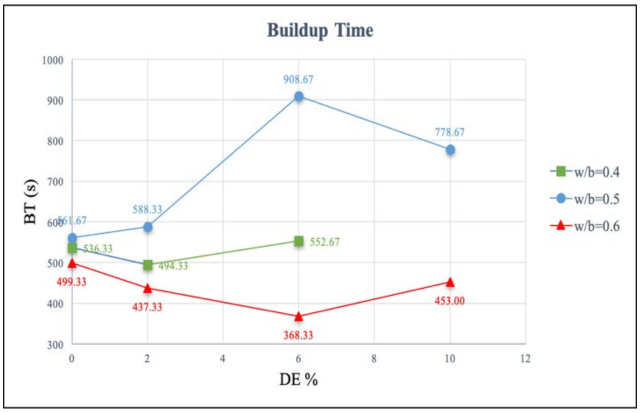


Fig. 11. Build-up time from protocol 3 against DE replacement levels

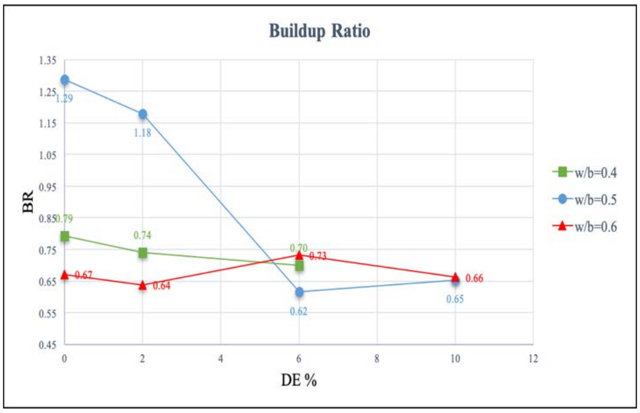


Fig. 12. Build-up ratios from protocol 4 against DE replacement levels

3.4 Temperature tests

To further investigate the temperature effect on thixotropy, protocol 2 and 4 were applied to pastes with w/b ratios of 0.5 and 0.6 with curing temperature changing from 10˚C to 40˚C. Fig. 13, shows the obtained results from protocol 2 for both w/b of 0.5 and 0.6. From both figures, it is observed that for all cementitious pastes, independent of w/b ratio and/or DE replacement level, static yield stress increases with increase in temperature. In other words, increase in temperature makes it more difficult to initiate the flow of cementitious pastes. This can simply be due to increase in hydration rate caused by the temperature. For both w/b ratios, 0.5 and 0.6, the change in static yield stress shows almost similar trend for all three temperatures. This hints that the thixotropic behavior of the paste is more sensitive to the temperature rather than the mix ingredient.

The test results from protocol 4 for various temperatures are shown in Fig. 14. From the figure, it can be seen that for both 0.5 and 0.6 w/b, buildup ratio is increased drastically for 40 °C compare to 10 and 25 °C. This can be explained by increase in hydration rate due to increase in temperature. For 0.5 w/b ratio (Fig. 14a), from 10 to 25 °C, buildup ratio shows no significant change; while for 0.6 w/b ratio (Fig. 14b), slight decrease in buildup ratio is observed. One possible explanation for this outcome may be the unique characteristics of water. At temperatures, close to 4 °C, the specific volume of water is at its minimum [21]. Although at these temperatures hydration rate is expected to decrease but the closer gap between solid particles, due to lower specific volume of water, may compensate for that or even lead to higher buildup ratios. This impact can be seen clearer at 0.6 w/b ratio due to its higher water content.

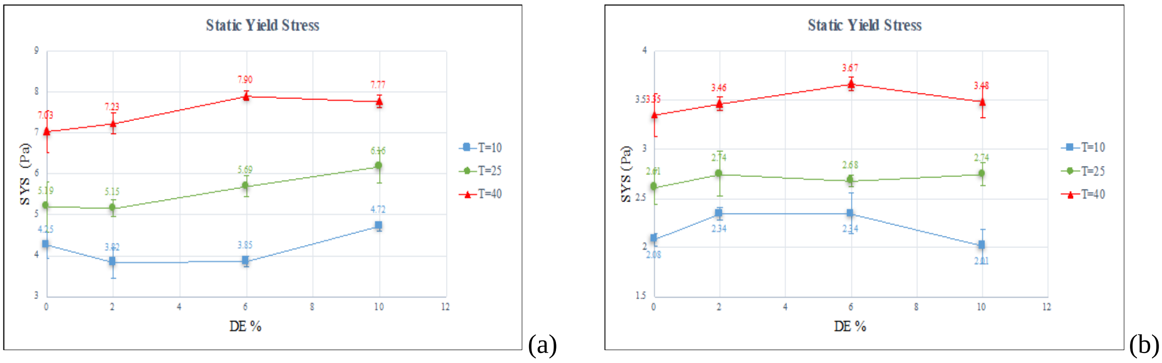


Fig. 13. Static yield stress from protocol 2 against DE replacement level (a) w/b=0.5 (b) w/b=0.6

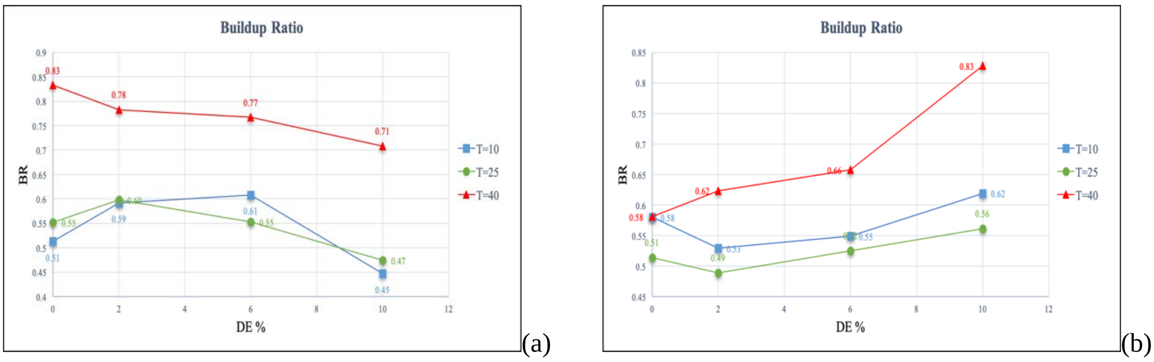


Fig. 14. Build-up ration from protocol 4 against DE replacement level (a) w/b=0.5 (b) w/b=0.6

3.5 Pressure tests

To mimic the influence of pumping speed or casting rate on the thixotropic behavior, protocol 2 and 4 were used to test the pastes of OPC-0.6 and D10-0.6. During pumping, concrete can be exposed to pressures as low as 60 bar to as high as 400 bar. Therefore, two pressure conditions of 20MPa (200 bar) and 10MPa (100 bar) were selected as the applied pressures in the tests. Fig. 15 shows the static yield stress and buildup ratio of OPC-0.6 and D10-0.6 mix proportions under different pressures. It is observed that an increase in pressure from 0 to 20 MPa has minor impact on yield stress or buildup ratio of these cement pastes. The minimal decrease in yield stress or build-up ratio can be due to slight de-flocculation caused by high pressure. This hints that the impact of high pressure up to 20 MPa on thixotropic behavior of cement pastes with 0.6 w/b ratio is minimal and can be neglected. This conclusion is in good agreement with findings of Kim et al. [22].

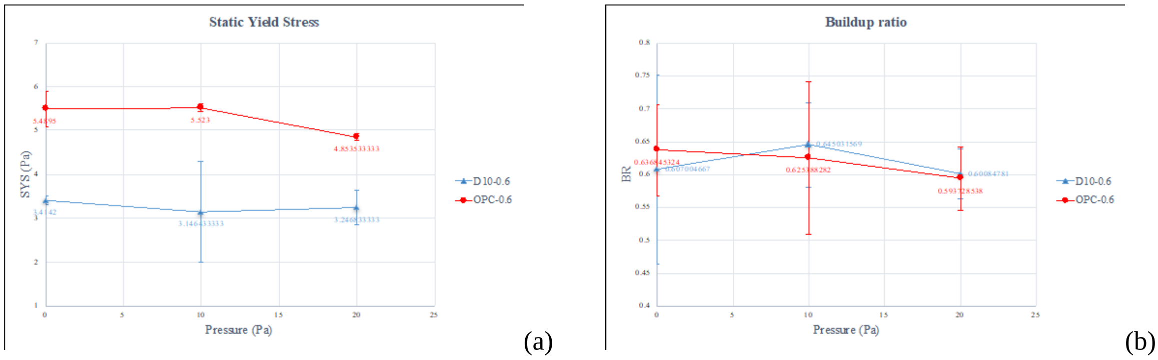


Fig. 15. Static yield stress from protocol 2 at different pressures for (a) OPC-0.6 (b) D10-0.6

4. Conclusions

It can be concluded that the proposed thixotropic protocols are fully capable of quantitatively measuring both breakdown and build-up aspects of thixotropic properties of cement pastes. Based on the results of this experimental investigation, the following conclusions are drawn:

1) No clear correlation between breakdown and build-up aspects of different cement pastes was observed. Hence, for each cement paste with specific mix proportion both aspects should be investigated.

2) Increase in DE content of cement pastes can decrease the amount of energy required to breakdown cement pastes structures to their most practically possible deflocculated state (steady state) under certain shear rate but does not significantly change minimum shear stress needed to initiate their flow (structure breakdown).

3) For short resting times cement pastes with higher DE content show higher de-flocculation rate because of their higher state of flocculation caused by colloidal forces. However, at longer resting times, higher colloidal forces and higher solid volume fraction compare to cement pastes with less DE content results in stronger hydration nucleation bonds and consequently lower de-flocculation rate.

4) Increase in temperature results in acceleration in hydration and flocculation rate of cement pastes, which consequently leads to higher static yield stress and build-up ratio.

5) Impact of high pressure up to 20 MPa on thixotropic behavior of cement pastes with 0.6 w/b ratio is minimal and can be neglected.

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6. References

[1] Wallevik, J.E. Rheological properties of cement paste: thixotropic behavior and structural breakdown. Cement and Concrete Research. 2009; 39(1): 14-29.

[2] Roussel, N. Steady and transient flow behaviour of fresh cement pastes. Cement and concrete research. 2005; 35(9): 1656-1664.

[3] Petkova, V. and V. Samichkov. Some influences on the thixotropy of composite slag Portland cement suspensions with secondary industrial waste. Construction and Building Materials. 2007; 21(7): 1520-1527.

[4] Quanji, Z., G.R. Lomboy, and K. Wang. Influence of nano-sized highly purified magnesium alumino silicate clay on thixotropic behavior of fresh cement pastes. Construction and Building Materials. 2014; 69:295-300.

[5] Kolawole, J.T., R. Combrinck, and W.P. Boshoff. Measuring the thixotropy of conventional concrete: The influence of viscosity modifying agent, superplasticiser and water. Construction and Building Materials. 2019; 225: 853-867.

[6] Assaad, J., K.H. Khayat, and H. Mesbah. Variation of formwork pressure with thixotropy of self-consolidating concrete. Materials Journal. 2003; 100(1): 29-37.

[7] Assaad, J., K.H. Khayat, and H. Mesbah. Assessment of thixotropy of flowable and self-consolidating concrete. Materials Journal. 2003;100(2): 99-107.

[8] Ferron, R.P., et al. Rheological method to evaluate structural buildup in self-consolidating concrete cement pastes. ACI materials journal. 2007; 104(3): 242.

[9] Robert D. Crangle, J. DIATOMITE, Mineral Yearbook 2013, in annual. 2013, U.S. Geological Survey: Mineral Yearbook. p. 22.1-22.5.
[10] Bakr, H. Diatomite: Its characterization, modifications and applications. Asian journal of materials science. 2010; 2(3): 121-136.
[11] Sally Jewell and S.M. Kimball. MINERAL COMMODITY SUMMARIES 2016, in annual. 2016, U.S. Geological Survey: USGS.
[12] Sally Jewell and S.M. Kimball. MINERAL COMMODITY SUMMARIES 2015, in annual. 2015, U.S. Geological Survey.
[13] Miller, S.A., et al.. Diatomaceous Earth as a Pozzolan in the Fabrication of an Alkali‐Activated Fine‐Aggregate Limestone Concrete. Journal of the American Ceramic Society. 2010; 93(9): 2828-2836.
[14] Jud Sierra, E., et al.. Pozzolanic activity of diatomaceous earth. Journal of the American Ceramic Society. 2010; 93(10): 3406-3410.
[15] Kipsanai, J.J.W., Paul M.; Namango,Saul S., Amziane, Sofiane, A Review on the Incorporation of Diatomaceous Earth as a Geopolymer-Based Concrete Building Resource. Materials. 2022; 15(20): 7130.
[16] Hasan, M.J., Muhammad; Saidi, Taufiq. Mechanical properties and durability of ultra-high-performance concrete with calcined diatomaceous earth as cement replacement. Journal of Mechanical Behavior of Materails. 2023; 32(1):20220272.
[17] Hasanzadeh, B. and Z. Sun. Impacts of Diatomaceous Earth on the Properties of Cement Pastes. Journal of Building Materials and Structures. 2018; 5(2): 197-211.
[18] Aldred, J. Burj Khalifa–a new high for high-performance concrete. in Proceedings of the Institution of Civil Engineers-Civil Engineering. 2010. Thomas Telford Ltd.
[19] Riding, K.A., et al.. Best practices for concrete pumping: technical summary. 2016, Kansas. Dept. of Transportation.
[20] Qian, Y. and S. Kawashima. Distinguishing dynamic and static yield stress of fresh cement mortars through thixotropy. Cement and Concrete Composites. 2018; 86: 288-296.
[21] Fierro Jr, P. and E.K. Nyer. The water encyclopedia: Hydrologic data and internet resources. 2007: CRC press.
[22] Kim, J.H., et al.. Rheology of cement paste under high pressure. Cement and Concrete Composites, 2017; 77: 60-67.