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Suitability of Static Yield Stress Evolution to Assess Thixotropy of Flowable Cementitious Materials

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Author's contribution

The sole author designed, analyzed and interpreted and prepared the manuscript.

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ABSTRACT

Behavior of self-consolidating concrete (SCC) after casting (such as stability, formwork pressure, and multi-layer interfaces) is directly affected by the flocculation aspect of thixotropy. The main objective of this paper is to evaluate the suitability of static yield stress (τ_0) evolution over time to assess the magnitude of thixotropy. Three series of highly flowable mortar mixtures were tested using the four-bladed vane method, and the results were compared with the cohesion (C) values obtained by direct shear. Test results have shown that τ_0 and C responses determined at given resting time are quite close to each other, indicating an adequate correlation between thixotropy determined using vane and direct shear methods. This reflects the suitability of considering τ_0 evolution over time to quantify the flocculation aspect of thixotropy, as well as its robustness as it is not affected by the testing method.

Keywords: Fresh concrete; thixotropy; four-bladed vane; direct shear test.

1. INTRODUCTION

The successful casting of highly flowable self-consolidating concrete (SCC) entails proper knowledge and monitoring of thixotropic properties. For instance, the cementitious matrix should be easily deflocculated during agitation with reduced apparent viscosity, thus facilitating

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placement by gravity with improved passing ability [1,2]. As soon as SCC placement is completed, the reversible phenomenon associated with the build-up of cementitious structure takes place over time. In vertical elements, a fast recovery is required as this improves stability and resistance towards aggregate segregation. Earlier studies showed that lack of stability can lead to surface defects, including bleeding and settlement that can weaken the quality of interface between aggregate and cement paste with direct effects on permeability, bond to steel, and mechanical properties [3,4]. Also, fast restructuring could be beneficial to reduce the SCC lateral stresses developed in vertical formworks [5].

In contrast, excessively high thixotropic SCC may not be appropriate when casting is made using injection or pumping techniques; i.e., if the material builds up its internal structure too fast and apparent yield stress exceeds a critical value, any stoppage (such as due to replenishment of buckets) may cause blockage of pipes and eventually abuse the equipment ultimate pressure in order to resume placement [1,2]. Also, high thixotropic SCC exhibiting fast structural recovery could not be appropriate during multi-layer casting in horizontal elements, as this creates cold joints and weak interfaces in the final structure. Some researchers reported mechanical and bond losses reaching 60% due to weak SCC interfaces [6,7].

Thixotropy of cementitious materials is often quantified by measuring the surface area during successive shear rate vs. stress measurements (Fig. 1). For example, because of the thixotropy transient and time-dependent nature, hysteresis loops are created when the plastic material is subjected to successive increasing/decreasing shear rates [1,2,5]. During the increasing ramp, de-flocculation occurs but not fast enough for a steady state stress to be reached. The measured stress is thus higher than what would be obtained if steady state was reached. During the decreasing ramp, flocculation occurs but again not fast enough for steady state to be reached, which creates the so-called hysteresis loops. Alternately, thixotropy can be quantified using the structural breakdown curves determined by subjecting the fresh material to given shear rate and recording stress variations over time [5]. The curves are typically characterized by peak yield stress that corresponds to the initial structural condition, and stress decay towards an equilibrium value (Fig. 1). Nevertheless, it is important to note that surface areas determined under dynamic conditions (i.e., hysteresis and structural breakdown curves) are highly dependent on the type of rheometer, testing protocol, applied shear rate, and flow history [2]. This prevents inter-laboratory comparison of test results and makes it difficult to assess the concrete properties using standardized testing protocols.

Under the concrete static condition after placement is completed, several authors studied the evolution of static yield stress (τ_0) over time [8,9,10], which would reflect the flocculation aspect of thixotropy and could be more relevant when assessing SCC behavior after casting especially the stability, formwork pressure, and multi-layer interfaces. The τ_0 is defined as the minimum stress required to initiate flow [11]; it reflects the physical restructuring of inter-particles links following a rest period coupled with attractive forces due to chemical reactions and formation of hydration compounds. The vane method is commonly used for measuring τ_0, because of its simplicity and the possibility of preventing slip during shearing [2,5,9]. Its principle consists of inserting a four-bladed vane of diameter (D) and height (H) in the plastic material and recording at sufficiently low shear rate the maximum torque (T_m) required to initiate flow. Considering the top edges of blades vane aligned with upper material surface (i.e., to eliminate over-head stress contribution on torque measurements) and yielding occurring at the cylindrical surface defined by the blade tips [11,12], T_m can be written as:

$$\mathbf{T_m} = \left(\frac{\pi D^3}{2}\right)\left(\frac{H}{D} + \frac{1}{6}\right)\boldsymbol{\tau_0} \quad (1)$$

When measurements are made at successive elapsed resting times after initial concrete mixing, τ_0 was found to increase linearly over time, which could be associated with the structuration rate of the cementitious matrix, as shown in Eq. 2:

$$\tau_0(t_{rest}) = \tau_0(t_0) + A_{Thix}\, t_{rest} \quad (2)$$

where t_{rest} is the resting time and A_{Thix} structuration rate (i.e., reflecting the magnitude of thixotropy) in Pa/s determined as the slope of tendency curve plotted between $\tau_0(t_{rest})$ and t_{rest}.

2. USE OF DIRECT SHEAR TO ASSESS THIXOTROPY

The direct shear test is widely used in soil mechanics to determine shear strength properties and analyze failure mechanisms occurring along interfaces. Its principle is quite simple, and consists of shearing two portions of a specimen by the action of steadily increasing force while constant load is applied normal to the plane of relative movement. Shear strength including cohesion (C) and angle of internal friction (ϕ) follow the Mohr-Coulomb law, given as:

$$\tau = C + \sigma' \tan \phi \qquad (3)$$

where τ and σ' refer to shear resistance and normal effective stress resulting from the solid grains, respectively.

In literature, the direct shear has often been employed as a reference test to develop and validate constitutive models characterizing the yield behavior of plastic materials. In fact, this test is standardized under ASTM D3080 [13] and available in most research centers; it is realized under quasi-static conditions whereby shearing takes place within the material along pre-defined interface represented by the horizontal surface area of shearing box. This physically overcomes the complications related to wall slip, secondary flow, and confinement conditions encountered in conventional rheometers [2,12,14]. Alfani and Guerrini [15] reported that direct shear is particularly suited for rheological characterization and interfacial flow behavior between extrudable cohesive pastes and equipment forming wall systems. Lu and Wang [16] considered the direct shear test to validate a constitutive model developed for predicting yield stress of cementitious materials. The C determined by direct shear was found to be closely related to the “true” τ_0 determined at low rotational speed using the four-bladed vane [10]. Recently, Assaad et al. [12] used the direct shear test to validate the effect of vane positioning on τ_0 responses of freshly mixed cement pastes and poly-vinyl acetate emulsions possessing different flowability levels. Over-estimation of τ_0 occurred when the vane was inserted inside the specimen, particularly for cohesive materials. Conversely, positioning the vane blades flush with the upper specimen surface eliminated the contribution of material self-weight on torque measurements and resulted in close C and τ_0 values [12].

This paper is part of a comprehensive research project undertaken to provide new insights on various approaches used to quantify thixotropy of cementitious materials. It does not aim at substituting the vane method by direct shear, especially knowing that the vane method is widely used, simple, and versatile. Rather, the main objective of this paper is to evaluate the suitability and robustness of considering the evolution of τ_0 over time determined by vane method in order to quantify the magnitude of thixotropy. Three series of highly flowable mortar mixtures were tested using the vane method, and results compared to those obtained by direct shear. Data presented in this paper can be of interest to researchers in various industries to facilitate inter-laboratory comparison and unify quantification of the flocculation aspect of thixotropy using standardized testing protocols.

3. EXPERIMENTAL PROGRAM

3.1 Materials

Portland cement and silica fume conforming to ASTM C150 Type I and C1240, respectively, are used. The surface areas of cement (Blaine) and silica fume (B.E.T.) were 340 and 20,120 m^2/kg, respectively; their specific gravities were 3.14 and 2.22, respectively. Continuously graded siliceous sand complying with ASTM C33 specification was employed; its nominal particle size, fineness modulus, and bulk specific gravity were 4.75 mm, 2.42, and 2.63, respectively.

A polycarboxylate-based high-range water reducer (HRWR) complying with ASTM C494 Type F was incorporated in all mixtures. It had a specific gravity, solid content, alkali content, and pH of 1.1, 42%, 0.34%, and 6.2, respectively.

Liquid viscosity-modifying admixture (VMA) and thixotropy-enhancing agent (TEA) were used. The VMA is based on hydroxyethyl cellulose (HEC) ether with a specific gravity and solid content of 1.04 and 18%, respectively. It is commonly used for SCC production, with recommended dosage rates varying from 0.15% to 1% of cement mass. This VMA is produced by substituting number of hydroxyl groups within the cellulose backbone by functional groups to improve water solubility through a decrease in the molecule crystallinity. Its average weight molecular mass and degree of substitution are equal to 310 kDa and 1.8, respectively.

The TEA is an organic cyclic propylene carbonate (PC) compound produced from propylene oxide and carbon dioxide with a zinc halide catalyst. Its specific gravity and pH are 1.03 and 6.5, respectively, and recommended dosage for cement-based materials varies from 0.2% to 1.2% of cement mass. As will be discussed later, the use of TEA was necessary to increase the magnitude of A_{Thix} beyond 1 Pa/s; in fact, increasing the HEC-based VMA concentration to achieve higher thixotropic level is accompanied with considerably increased HRWR molecules to maintain similar flowability, thus delaying cement hydration reactions and extending setting times beyond 24 hours [17,18]. Conversely, the delay in setting time was limited when the PC-based TEA was used in conjunction with HRWR.

3.2 Mixture Proportioning

Three mortar series proportioned with cement quantities of 375, 435, and 500 kg/m^3 and water-to-cement ratio (w/c) of 0.46, 0.41, and 0.34, respectively, were considered (Table 1). The mixtures were proportioned using the concrete-equivalent-mortar (CEM) approach; i.e., the cement content and w/c remained similar to those of corresponding concrete, except that all coarse aggregates are replaced by an equivalent quantity of sand in terms of specific surface area [19,20]. Aggregate-free CEM mixtures could better reflect the flocculation aspect of thixotropy, given that aggregates mostly affect internal friction that overshadows the build-up phenomenon of cementitious matrix [5,20].

A total of 12 CEM mixtures were tested. The silica fume, VMA, and TEA were added at relatively low to high dosage rates to achieve different A_{Thix} levels; i.e. silica fume at 5% or 10%, VMA at 0.35% or 0.8%, and TEA at 0.3% or 0.75% of cement mass (Table 1). In all mortars, the HRWR was adjusted to secure a flow of 220 $\pm$10 mm when determined as per ASTM C1437 (this flow corresponds to concrete slump flow of 650 $\pm$20 mm determined using ASTM C143 slump cone) [21].

3.3 Mixing and Stability Testing

The mortar mixing procedure consisted of homogenizing the sand with half of mixing water, then introducing the cementitious materials gradually over 30 seconds. The remaining part of water along with the VMA or TEA along with HRWR were then added and mixed for 1.5 minutes. After a rest period of 30 seconds, the mortar was remixed for 1.5 additional minutes. Testing and sampling were made at room temperature of 23 $\pm$2°C and 50% $\pm$5% relative humidity.

Right after mixing, the flow was measured by determining the material's average diameter after spreading on horizontal surface using a mini-slump cone having top diameter, bottom diameter, and height equal to 70, 100, and 50 mm, respectively [14]. The passing ability was evaluated using the Marsh cone having 12.7-mm outlet diameter; a volume of 500-mL was filled in the cone and allowed to rest for 5 seconds prior to flow time measurement. The bleeding was determined as per ASTM C232, and consists of measuring the relative quantity of mixing water that has bled from the fresh material placed in 75-mm diameter and 150-mm height container. For measurements, the container was slightly tilted and free water collected using a pipet from the specimen surface. The percentage of bleed water was obtained by dividing the collected water by the total mixing water in specimen.

3.4 Assessment of τ_0 Using the Four-bladed Vane

Right after mixing, the mortars were placed in 5 separate cylindrical recipients having each 120-mm height and 100-mm diameter for τ_0 measurements at 5 different t_{rest} intervals (i.e., at 0, 20, 40, 60, and 80 min). Anton Paar rheometer connected to four-bladed vane having 24-mm height and 12-mm diameter was used. For each measurement realized at given t_{rest}, the vane was gently introduced in the mortar in a way to position the top vane edges aligned with the upper material's surface. This was found particularly important when testing was realized at longer t_{rest} intervals of relatively moderate to high thixotropic mortars, as this avoided the material disturbance during the vane insertion process. It is to be noted that, prior to vane insertion, care was taken to tilt the recipient gently in order to remove using a pipette the eventual bleed water that occurred during the rest period (all mortars filled in recipients were covered by wet burlap during the rest period). The testing protocol consisted on subjecting the mortar to very low rotational speed of 0.3 rpm and recording the changes in torque as a function of time (mortars tested at t_0 were allowed to rest for 1 min prior to testing).

3.5 Assessment of C by Direct Shear

An ELE Direct Shear apparatus complying with ASTM D3080 [13] was used for measuring C values of tested CEM (Fig. 2). The metal shear box measuring 100 mm diameter and 58 mm height is divided into two halves horizontally;

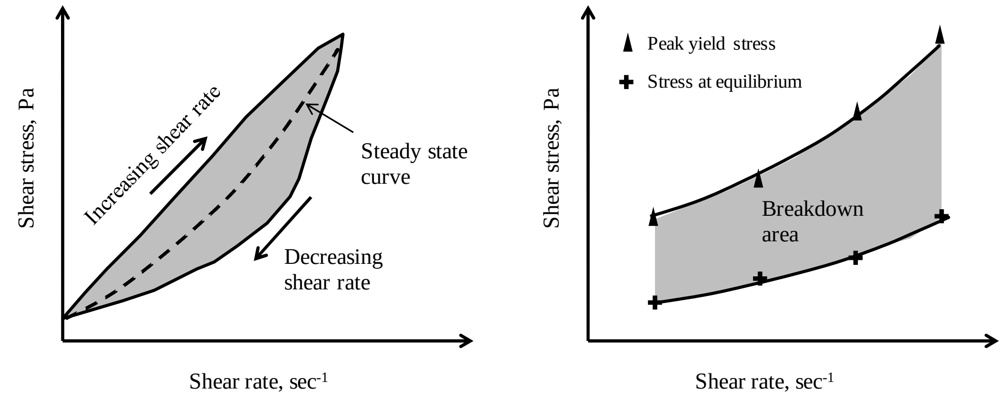

Fig. 1. Typical hysteresis loops and structural breakdown area for assessing the magnitude of thixotropy

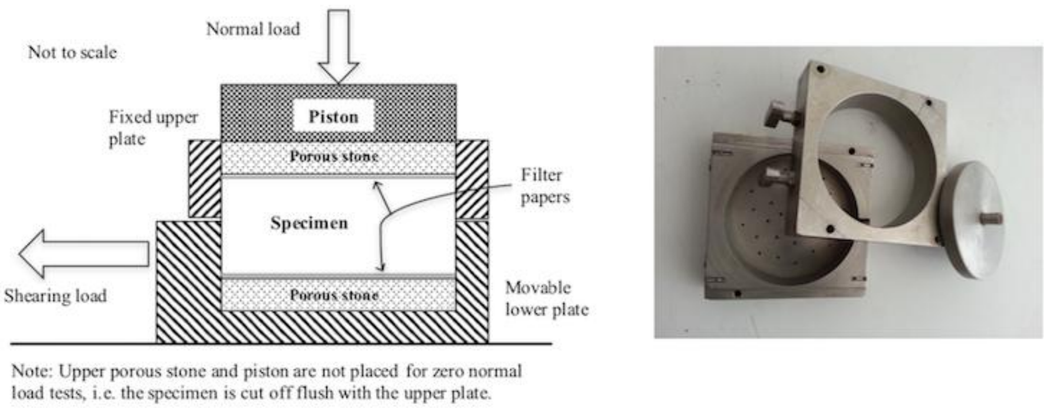

Fig. 2. Photo for direct shear test

Table 1. Typical SCC classes and corresponding CEM composition

	Typical classes of SCC mixtures		
Cement, kg/m^3	375	435	500
w/c	0.46	0.41	0.34
Sand (0-4.75 mm), kg/m^3	970	935	920
Aggregates (1.18-9.5 mm), kg/m^3	825	795	780
Targeted slump flow, mm	650 ±20	650 ±20	650 ±20
	Tested mortars using the CEM approach		
Cement, kg/m^3	375	435	500
w/c	0.46	0.41	0.34
Sand (0-4.75 mm), kg/m^3	1065	1045	1010
Cement paste / sand, by volume	0.718	0.795	0.853
Silica fume, % of cement mass	0%, 5% and 10%		
VMA, % of cement mass	0%, 0.35%, and 0.8%		
TEA, % of cement mass	0%, 0.3%, and 0.75%		
HRWR, % of cement mass	Varies depending on CEM composition to achieve similar initial flow of 220 ±10 mm		

the lower section can move forward at different constant velocities varying from 0.001 to 9 mm/min, while the upper section remains stationary. In order to eliminate friction between the two sections during movement and allow C measurements in the order of few Pa, four perfectly aligned 10-mm long channels were laser-grooved in the bottom part of the shear box [12]. A steel ball having 2.5-mm diameter was then placed in each channel, thus allowing the lower plate of the shear box to behave like a roller with respect to the upper plate. The gap between both plates was 10 $\pm$1 μm, and filled with grease to avoid material's leakage. The shear stresses were calculated by dividing the horizontal load by the specimen's cross-sectional area, i.e. 7850 mm^2. The complete description of direct shear test used can be seen in [12].

After mixing, the mortar was filled in the shear box and allowed to rest for the specified time interval (a new mortar was batched for each test). To alleviate the experimental program, 3 tests at different t_{rest} were realized for each mortar, expect the 0.46-5%SF and 0.41-5%SF mortars where 4 tests are conducted. The displacement rates were fixed at 0.5 mm/min, a value found experimentally enough to overcome the restoring forces due to reorientation of particles and structural development due to cement hydration [12,22]. It is to be noted that the ϕ parameter was not determined in this study, given that testing was realized without normal load applied on top of specimen during the shearing process.

4. TEST RESULTS AND DISCUSSION

4.1 HRWR Demand, Setting Times, and Stability Testing

Table 2 summarizes the HRWR dosage needed to achieve flow of 220 $\pm$10 mm along with the resulting unit weight, setting time, and stability indexes used to characterize CEM behavior. Briefly, the HRWR demand increased when mortars contained higher silica fume or VMA concentration. The increase in HRWR/VMA lengthened the setting time due to higher molecules adsorption onto cement particles that partly blocks the hydration reactions [17]. For example, the setting was delayed from 9:30 to 10:15 and 14:15 hr:min for 0.46-5%SF, 0.46-10%SF, and 0.46-0.8%VMA, respectively. Mixtures containing TEA exhibited remarkably reduced setting times, as compared to equivalent mortars made with VMA.

As summarized in Table 2, the unit weights varied from 1920 $\pm$15 to 2050 $\pm$30 and 2140 $\pm$20 kg/m^3 for mixtures made with 0.46, 0.41, and 0.34 w/c, respectively. Generally speaking, the flow time increased with the reduction of w/c, particularly with the addition of silica fume or VMA, given the increased inter-particle friction and cohesiveness [2,3]; values varied from 38 to 90 sec. Mortars incorporating TEA exhibited relatively moderate flow times of 73 sec, given that the mixture was not allowed to rest and build its structure prior to testing [17,18].

Typical variations in cumulative bleeding over time for selected mortars are given in Fig. 3. Depending on CEM composition, the bleed water increased at different rates during the initial 20-min after placement, and tended to stabilize thereafter. For example, the bleeding rate decreased from 0.353 to 0.125 and 0.065 %/min for the 0.46-5%SF, 0.41-10%SF, and 0.34-0.35%VMA, respectively; the corresponding maximum bleed water determined after stabilization was 10.3%, 3.3%, and 1.5%, respectively. The 0.34-0.75%TEA mortar exhibited the lowest bleed rate and stabilized value, given its fast restructuring. Table 2 summarizes the bleeding rates determined over the initial 20-min and maximum bleed values obtained after stabilization.

4.2 The τ_0 and C Responses – Repeatability of Testing

Typical shear stress vs. horizontal displacement curves obtained by direct shear for selected mortars at different t_{rest} intervals are given in Fig. 4. As can be seen, the shear stress profiles showed linear elastic region until reaching the maximum peak value (taken as C). The presence of maximum value is an index of flocculation aspect of thixotropy that can be explained by the concept of structural build-up of bonds in the flocculated system [9,11,12]. Further horizontal displacement causes the stresses to decrease towards a steady state region. At maximum shear value, the horizontal displacement of bottom shear box varied from 1 to 3 mm, depending mostly on t_{rest} interval. It is to be noted that the direct shear profiles are very similar to those typically obtained using the four-bladed vane [11,12,16], which reflects the similarity of both testing methods. The τ_0 and C values determined at various resting intervals are summarized in Table 3.

In order to evaluate repeatability of testing, three selected mortars possessing low to high thixotropic levels were tested 3 times using the vane and direct shear methods (a new batch was considered for each test). The coefficients of variation (COV) calculated as the ratio between standard deviation of responses and their mean values, multiplied by 100, are shown in Fig. 5. Generally speaking, the moderately thixotropic mixtures (i.e., 0.41-0.8%VMA) exhibited adequate repeatability, regardless of t_{rest} interval. Hence, the COV of various responses determined by the vane varied from 5% to 7.5% and from 4.6% to 8.4% when using direct shear. The COV that resulted from direct shear increased up to 11.7% and 15.4% at t_0 for low and high thixotropic mixtures (i.e., 0.46-10%SF and 0.34-0.75%TEA, respectively). For the former category of mixtures, the increased COV can be related to reduced stability including bleeding and sedimentation, which affect variability of C responses. In contrast, the increased COV resulting from high thixotropic mixtures can be attributed to faster flocculation rates that make measurements quite sensitive to accuracy of testing procedures.

Table 2. Effect of mortar composition on HRWR demand and stability indices

	HRWR, % of cement	Initial flow, mm	Final set time, hr:min	Unit weight, kg/m^3	Flow time, sec	Bleeding	
						Bleed rate, %/min	Max. bleed, %
0.46-5%SF	0.62	225	9:30	1910	38.25	0.353	10.3
0.46-10%SF	0.65	220	10:15	1930	40.45	0.235	8.5
0.46-0.35%VMA	0.65	225	11:45	1915	48	0.267	8.3
0.46-0.8%VMA	0.77	225	14:15	1930	63.5	0.14	5
0.41-5%SF	0.8	220	11:45	2080	49	0.165	4.2
0.41-10%SF	0.86	225	13:00	2050	52.25	0.125	3.3
0.41-0.35%VMA	0.85	220	15:00	2040	59.5	0.13	3.1
0.41-0.8%VMA	0.95	230	16:45	2065	80.25	0.085	2.1
0.34-5%SF	1.12	225	14:45	2145	86.25	0.095	2.3
0.34-0.35%VMA	1.1	230	15:30	2160	90	0.065	1.5
0.34-0.3%TEA	1.05	225	12:15	2130	72.5	0.047	0.9
0.34-0.75%TEA	1.05	230	12:45	2135	74	0.03	0.5

Mixture codification refers to: w/c - Percent and type of additive used (i.e., silica fume, VMA, or TEA)

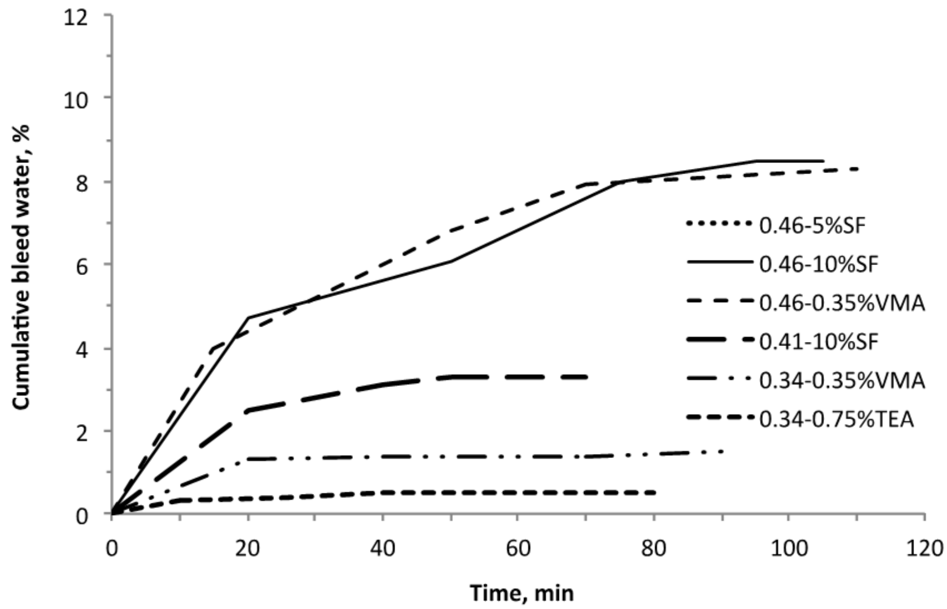

Fig. 3. Typical cumulative bleeding curves vs. time for selected mortars

Table 3. Determination of A_{Thix} by four-bladed vane and direct shear

	Four-bladed vane method		Direct shear method	
	t_{rest} (min) and corresponding τ_0 (Pa)	$A_{Thix}(\tau_0)$, Pa/s	t_{rest} (min) and corresponding C (Pa)	A_{Thix}(C), Pa/s
0.46-5%SF	t_0 = 39.4; t_{20} = 85.7; t_{40} = 146.2; t_{60} = 271; and t_{80} = 355.8	0.0682	t_0 = 45.1; t_{20} = 146; t_{40} = 363.5; and t_{60} = 612.7	0.16
0.46-10%SF	t_0 = 42.6; t_{20} = 79; t_{40} = 200.8; t_{60} = 284; and t_{80} = 418	0.0796	t_0 = 41.8; t_{20} = 162; and t_{40} = 382	0.142
0.46-0.35%VMA	t_0 = 36.7; t_{20} = 101.4; t_{40} = 273; and t_{80} = 522.6	0.105	t_0 = 38; t_{20} = 186; and t_{30} = 360.4	0.171
0.46-0.8%VMA	t_0 = 45; t_{20} = 273; t_{60} = 881; and t_{80} = 1264	0.254	t_0 = 39.8; t_{10} = 171; and t_{30} = 559	0.293
0.41-5%SF	t_0 = 53.2; t_{20} = 183.5; t_{40} = 383; and t_{80} = 725.6	0.143	t_0 = 49; t_{20} = 174.5; t_{40} = 386; and t_{60} = 556	0.144
0.41-10%SF	t_0 = 48.3; t_{20} = 206; t_{40} = 415; and t_{60} = 691.2	0.178	t_0 = 55; t_{20} = 188.6; and t_{40} = 426	0.154
0.41-0.35%VMA	t_0 = 57.6; t_{20} = 422.1; t_{40} = 682; and t_{60} = 1274	0.326	t_0 = 57.1; t_{10} = 359; and t_{30} = 732	0.366
0.41-0.8%VMA	t_0 = 61; t_{20} = 428; t_{40} = 1806; t_{60} = 2844; and t_{80} = 4206	0.892	t_0 = 57.2; t_{10} = 429; and t_{30} = 1517	0.825
0.34-5%SF	t_0 = 63.3; t_{40} = 1308; t_{60} = 1802; and t_{80} = 2947	0.575	t_0 = 64.7; t_{40} = 1022; and t_{60} = 1905	0.495
0.34-0.35%VMA	t_0 = 67.3; t_{20} = 493; t_{40} = 1482; t_{60} = 2734; and t_{80} = 4033	0.848	t_0 = 70.4; t_{20} = 620; and t_{50} = 2408	0.796
0.34-0.3%TEA	t_0 = 51.2; t_{20} = 769; t_{40} = 2275; and t_{60} = 4238	1.172	t_0 = 58.3; t_{10} = 566; and t_{30} = 1894	1.032
0.34-0.75%TEA	t_0 = 50.3; t_{20} = 1106; t_{40} = 3275; and t_{60} = 5266	1.484	t_0 = 49.7; t_{20} = 985; and t_{40} = 2995	1.227

4.3 Effect of Mortar Composition on τ_0 and C

The τ_0 measurements determined after mixing and 20 min later for tested mortars are plotted in Fig. 6. As expected, mortars prepared with combinations of increased cement content and reduced w/c led to higher τ_0 values, given the increased inter-particle links and reduced free mixing water. For example, such increase at t_0 was from 39.4 to 53.2 and 63.3 Pa for the 0.46-5%SF, 0.41%-5%SF, and 0.34-5%SF, respectively. Also, for given w/c, τ_0 increased with the addition of silica fume (due to increased packing density of matrix) or VMA (due to polymer entanglement and hydrogen bonds) [2,3,7]. At longer elapsed resting times (i.e., at t_{20}), all mortars exhibited increased τ_0 responses, depending on the flocculation rate associated with cement hydration reactions that occurred during the rest period.

It is interesting to note that relatively low τ_0 values were determined right after mixing (i.e., at t_0) for mortars containing TEA, but then significantly increased over time. For example, τ_0 of 0.34-0.75%TEA was 50.3 Pa at t_0, but reached the highest value of 1106 Pa at t_{20}. This clearly reflects the thixotropic mode of action of this agent through which the physico-chemical interactions of propylene carbonate with cement particles lead to significant structural build-up at rest with increased τ_0 responses [17,18].

4.3.1 Comparison with C values

With the exception of 3 mortars made with 0.46-w/c possessing unstable nature (i.e., 0.46-5%SF, 0.46-10%SF, and 0.46-0.35%VMA), the order of C magnitude determined by direct shear at given t_{rest} was pretty close to that of corresponding τ_0 (Table 3); the measurements remained within the repeatability of testing. In the case of unstable mortars, the C values determined after certain t_{rest} were higher by around 1.5 to 2.5 times than corresponding τ_0. For instance, the C of 0.46-5%SF mortar registered after 20, 40, and 60 min rest was 146, 363.5, and 612.7 Pa, respectively; while corresponding τ_0 was 85.7, 146.2, and 271 Pa, respectively. This could be related to reduced stability, including bleeding and sedimentation that increase concentration of solid particles towards the lower half of the shearing box where interfacial failure plane is expected to occur, thus leading to increased shear stresses. The difference in material concentration was felt when trying to move a spatula manually from the top surface to interfacial region in the shearing box [12].

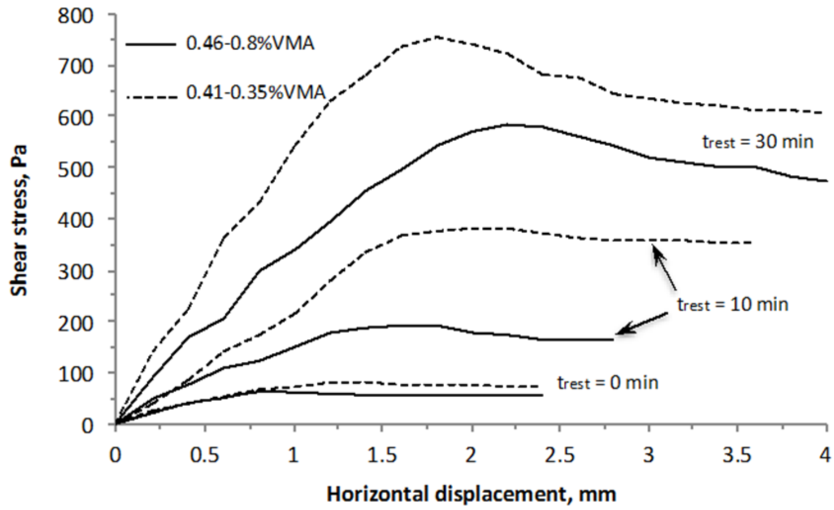

Fig. 4. Typical shear stress vs. horizontal displacement plots determined at different t_{rest} by direct shear

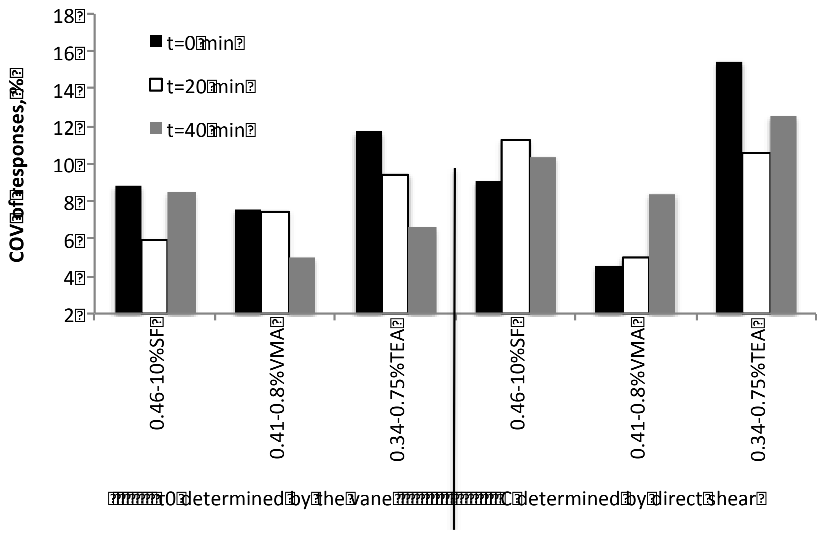

Fig. 5. COV of τ_0 and C responses determined at different t_{rest}

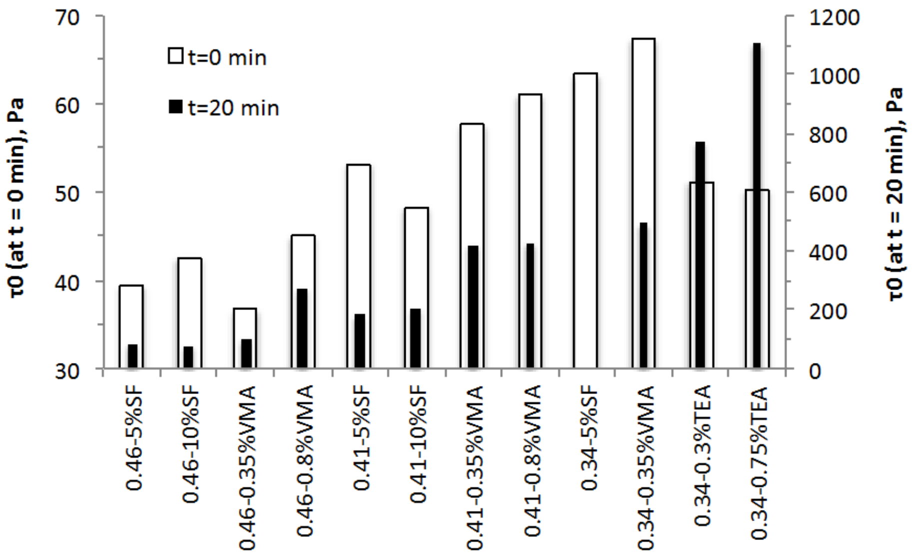

Fig. 6. Effect of mortar composition on τ_0 values determined at t_0 and t_{20}

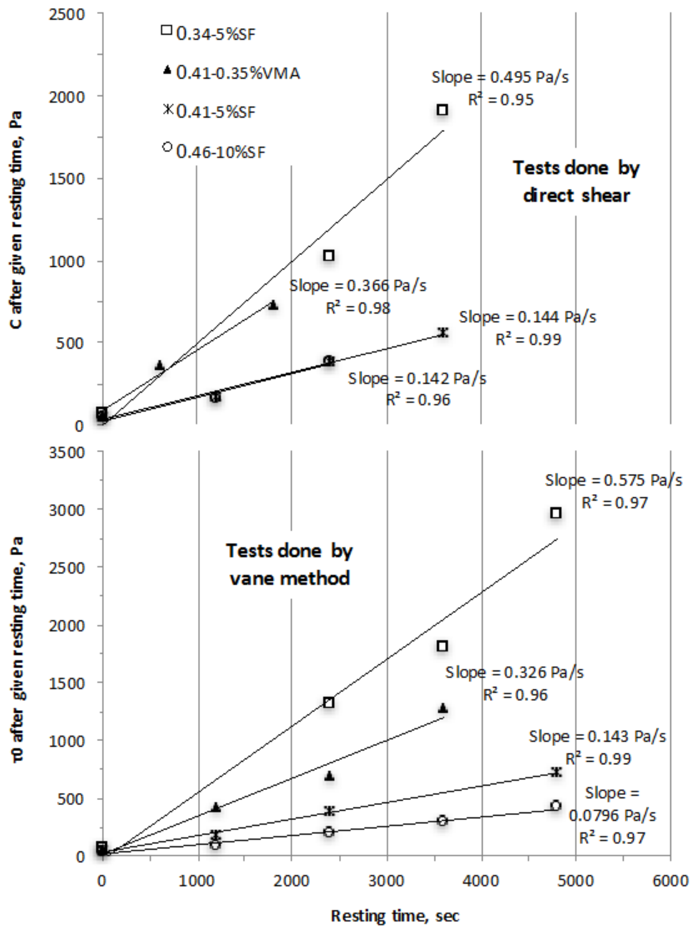

Fig. 7. Determination of $A_{Thix}(\tau_0)$ and $A_{Thix}(C)$ for selected mortars

The relationships between τ_0 and C responses for all tested mortars measured at various t_{rest} along with their correlation coefficients (R^2) are given below (the relationships were forced to intercept the origin of axis, thus having the form y = A x).

At t_0: $C = 1.104\ \tau_0 \quad R^2 = 0.82$ (4)

At t_{20}: $C = 0.964\ \tau_0 \quad R^2 = 0.92$ (5)

At t_{40}: $C = 0.905\ \tau_0 \quad R^2 = 0.97$ (6)

4.4 The A_{Thix} Values Determined by Different Methods

Typical example showing the determination of A_{Thix} by considering the slope of tendency curves of τ_0 or (C value) determined at various t_{rest} using the vane or direct shear methods is given in Fig. 7; the results obtained are summarized in Table 3. Clearly, the τ_0 and C values followed increasing trends with resting time, depending on mortar constituents and ability to restructure skeleton at rest. The R^2 of all tendency curves were higher than 0.95, reflecting that both methods can appropriately be used to assess A_{Thix} of cementitious materials.

The effect of CEM composition on $A_{Thix}(\tau_0)$ magnitudes is shown in Fig. 8. Following the same phenomena described earlier, $A_{Thix}(\tau_0)$ increased for mortars made with combinations of increased cement content and reduced w/c. For example, such increase was from 0.0682 to 0.143 and 0.575 Pa/s for the 0.46-5%SF, 0.41%-5%SF, and 0.34-5%SF, respectively. Also, for given w/c, $A_{Thix}(\tau_0)$ increased with the addition of silica fume or VMA; at 0.41 w/c, this reached 0.178 and 0.892 Pa/s for 0.41-10%SF and 0.41-0.8%VMA, respectively. The highest $A_{Thix}(\tau_0)$ of 1.172 and 1.484 Pa/s corresponded to 0.34-w/c mortars made with 0.3% or 0.75% TEA, respectively, mostly related to the thixotropic nature of this agent.

4.4.1 <u>Comparison with A_{Thix}(C) values</u>

As can be seen in Fig. 9, the ratio of $A_{Thix}(C)/A_{Thix}(\tau_0)$ varied from 1.5 to 2.5 for the unstable 0.46-5%SF, 0.46-10%SF, and 0.46-0.35%VMA mortars. As previously explained, this can be related to reduced stability that over-estimated the shear stresses and resulted in higher $A_{Thix}(C)$. Subsequently, the $A_{Thix}(C)/A_{Thix}(\tau_0)$ ratio hovered around 1.0 for all other CEM, implying that the magnitude of A_{Thix} becomes almost similar for relatively stable mixtures, regardless of testing method. This reflects the accuracy of considering the slope of τ_0 determined at various t_{rest} to quantify the flocculation aspect of thixotropy, as well as its robustness as it is not affected by the testing method. The relationship between both indices for all tested mortars is given as:

$$A_{Thix}(C) = 0.884\ A_{Thix}(\tau_0) \qquad R^2 = 0.97 \qquad (7)$$

The relationships between τ_0 or C responses determined after mixing (i.e., at t_0) and corresponding magnitude of A_{Thix} are plotted in Fig. 10. If excluding mortars prepared with TEA, it is interesting to note that τ_0 determined by the vane method can well be used to predict $A_{Thix}(\tau_0)$ with acceptable R^2 of 0.82. Mixtures containing TEA exhibited moderate τ_0 values at t_0, albeit their rates of increase were significantly accentuated. A relatively moderate R^2 of 0.54 resulted from C determined by direct shear right after mixing and corresponding $A_{Thix}(C)$ data.

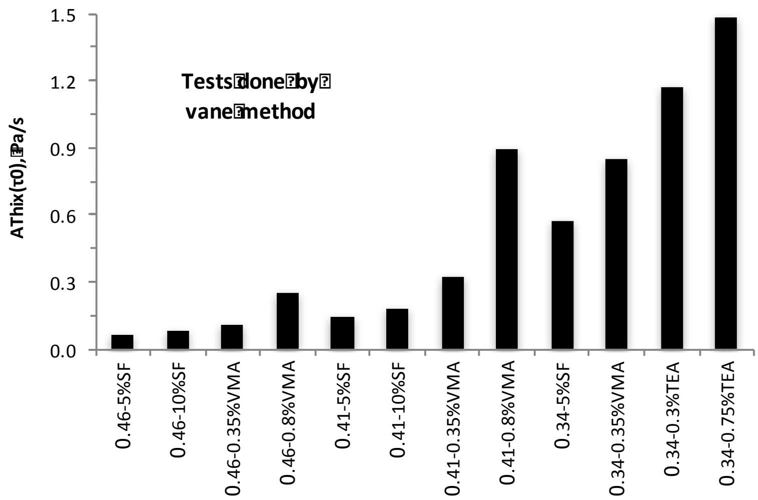

Fig. 8. Effect of mortar composition on $A_{Thix}(\tau_0)$ measurements

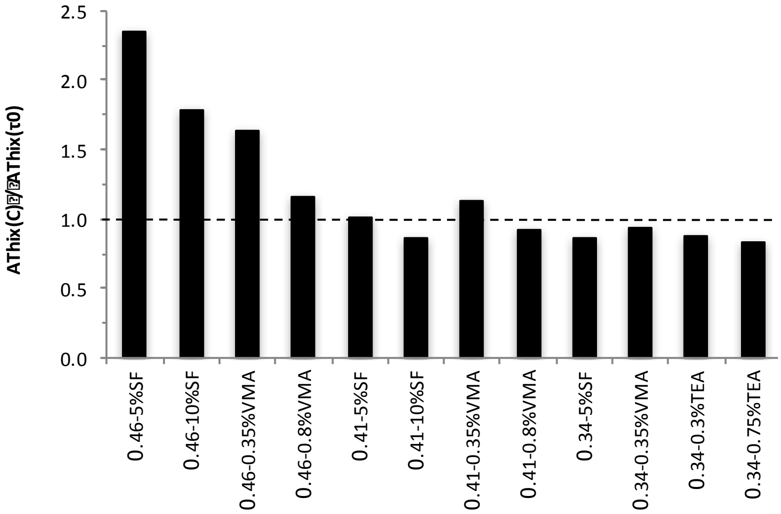

Fig. 9. Ratio between A_{Thix}(C) and $A_{Thix}(\tau_0)$ measurements for tested mortars

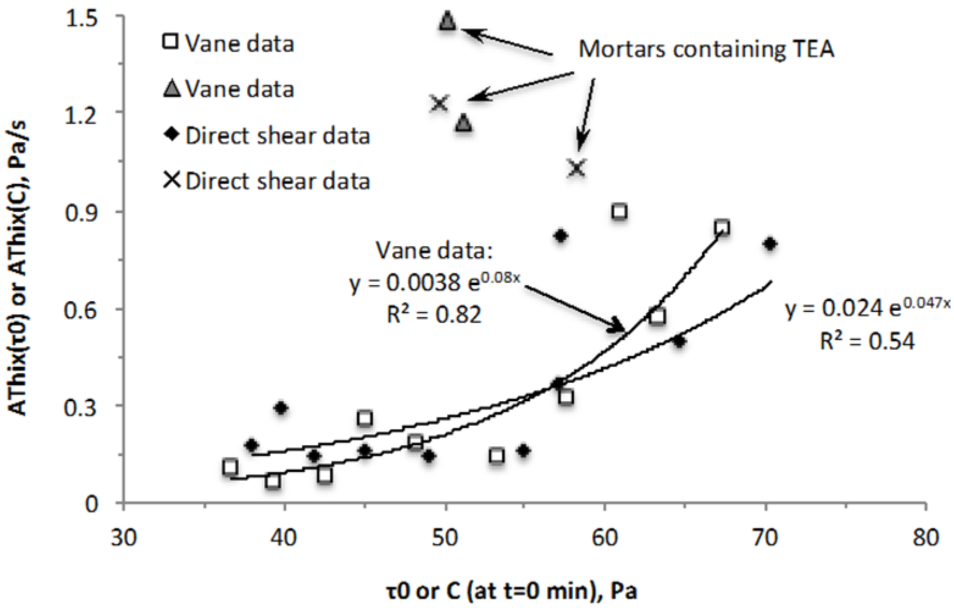

Fig. 10. Prediction of $A_{Thix}(\tau_0)$ from τ_0 at t_0 (and A_{Thix}(C) from C at t_0)

5. CONCLUSIONS

Monitoring the flocculation aspect of thixotropy is essential to predict SCC properties after casting such as stability, formwork pressure, and multi-layer interfaces. The main objective of this paper is to evaluate the suitability of τ_0 evolution over time determined by vane method in order to assess the magnitude of thixotropy. Three series of highly flowable mortars were tested using a four-bladed vane, and results compared to C values obtained by direct shear. Standardized under ASTM D3080 and available in most research centers, the direct shear can be considered as a reference test to unify quantification and validate constitutive models intended for yield behavior of cementitious materials.

Based on the foregoing, test results from this study showed that τ_0 and C values increased when mixtures are prepared with reduced w/c and/or addition of silica fume or VMA. The TEA led to remarkably high τ_0 and C values, given the fast build-up of cementitious matrix. The $A_{Thix}(C)/A_{Thix}(\tau_0)$ ratio varied from 1.5 to 2.5 for low thixotropic and unstable mortars, which was attributed to bleeding and sedimentation that alter concentration of solid particles where interfacial failure is expected to occur. In contrast, $A_{Thix}(C)/A_{Thix}(\tau_0)$ ratio hovered around 1.0 for stable mixtures, reflecting similar magnitudes of thixotropy. Adequate correlation exists between thixotropy determined by four-bladed vane and direct shear methods. This reflects the suitability of considering the slope of τ_0 determined at various rest intervals to quantify thixotropy, as well as its robustness as it is not affected by the testing method.

COMPETING INTERESTS

The author declares that there is no conflict of interest regarding the publication of this paper.

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