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# Thixotropy and structural breakdown properties of self consolidating concrete containing various supplementary cementitious materials

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### 1. Introduction

Utilization of supplementary cementitious materials (SCM) in concrete has a tendency to increase by the future in order to provide greater sustainability in construction industry. SCM such as fly ash (FA), granulated blast-furnace slag (BFS) and silica fume (SF) are widely used in self-consolidating concrete (SCC) due to their several advantages. First of all, the high amount of paste requirement for better flow of SCC is easily achieved by SCM, which is superior to using only portland cement (PC). Also utilization of these SCM in SCC reduces the demand for PC, fine fillers and viscosity-enhancing chemical admixtures in SCC. Many studies [1-5] reported that use of SCM in SCC not only improves the mechanical, durability and long term properties of concrete, but also helps to adjust the rheological and thixotropic properties as well as stability of the fresh SCC for a given application. In other words, plastic viscosity or stability specifications can be tailored according to the desired performance in a variety of civil engineering applications by the utilization of SCM. Additionally, use of by-product SCM, like FA and BFS, can decrease the cost of SCC and the amount of the CO<sub>2</sub> production related to the use of PC in concrete. Therefore, use of SCM has become very important in SCC [6,7].

### ABSTRACT

In this study, thixotropy and structural breakdown of 57 self-consolidating concrete (SCC) mixtures containing various supplementary cementitious materials (SCM) were investigated by different approaches. The effects of SCM type and content on high range water reducer demand and plastic viscosity were also studied. For these purposes, various amounts of silica fume (SF), metakaolin (MK), Class F fly ash (FAF), Class C fly ash (FAC) and granulated blast-furnace slag (BFS) were utilized in binary, ternary, and quaternary cementitious blends in three water/binder (w/b) ratios. Results showed that except BFS, use of SCM in SCC mixtures increased thixotropy values in comparison with the mixtures containing only portland cement (PC). Good correlations were established between structural breakdown area and drop in apparent viscosity values for all w/b ratios. The different methods used to evaluate the thixotropy and structural breakdown got more consistent with each other as w/b decreased.

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Despite the above-mentioned advantages of SCM in SCC, they may present some deleterious effects on SCC properties compared to the plain SCC containing no SCM. For instance for a constant slump flow, MK may significantly increase plastic viscosity [8] and impair a sharp fall in the workability of fresh concrete although it can considerably reduce the permeability [3] of SCC. On the contrary, BFS may improve workability but it may decrease plastic viscosity of SCC [9,10]. Plastic viscosity of SCC mixtures containing SF could be similar or lower than of the control mixture without any SCM at constant slump flow values while stability and durability aspects are improved [4,5,8]. In addition, replacement of PC by FA in SCC mixtures can increases plastic viscosity [11] but may significantly decrease early strength values [12].

A high amount of plastic viscosity can reduce concrete flowability and workability, whereas a very low viscosity can accelerate the speed of segregation [13]. Moreover, a concrete with a high degree of thixotropy may show high segregation resistance [5] and decrease lateral pressure exerted on the formwork system [14] while high thixotropy may lead to an increase in entrapment of air in fresh concrete and formation of lift lines in multilayer casting [15]. Such opposite effects may be remedied by the combined use of the SCM. Therefore, combinations of SCM in ternary and quaternary cementitious blends have found significant importance in the presented study.

Although a number of studies about the effects of SCM on the fresh and hardened properties of SCC have been found in the







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literature, the effect of using SCM on the thixotropic properties were discussed only in limited number of studies [2,5,16]. Moreover, the potential benefit of using various amounts of SCM in ternary and quaternary combinations with PC on rheological and thixotropic properties of SCC is not well documented. Thus, the effects of different blends of SCM on rheological and thixotropic properties of SCC are presented in this study.

Thixotropy can be defined as a gradual decrease of the viscosity under shear stress followed by a gradual recovery of structure when the stress is removed [17]. The thixotropic behavior of cement paste is related to coagulation, dispersion and re-coagulation of the cement particles [18–20]. Coagulation is a result of the total potential energy interaction between them, which originates from the combined forces of van der Waals attraction, electrostatic repulsion and steric hindrance [19,21,22].

Tattersall and Banfill [21] explained the structural breakdown as follows: When cement particles and water come together, a hydrate membrane immediately covers and links the particles. If the cement paste is sheared, the linkages between them may be broken, separating the particles. The breaking of linkages was considered to be an irreversible process and thus non-thixotropic [21,23].

There are several ways of studying the thixotropy and structural breakdown. By gradually increasing and then decreasing the angular velocity of the viscometer vane, the corresponding torque values will form a hysteresis loop if the mixture is thixotropic. The area between the up and down curves can quantify the thixotropy [21,24,25]. A more widely used method to quantify the thixotropy and structural breakdown is to keep the shear rate constant and observe the change (decrease) in the corresponding torque values in time [21]. This approach can be repeated for several constant shear rates to study the behavior over a wider range of shear rates, enabling a more comprehensive evaluation [5,26]. The thixotropic behavior of the cementitious materials was also studied by evaluating the static yield strengths of the mixtures determined at very low shear rates [27]. Roussel defined a so-called "thixotropy index" as the ratio of the shear stress at rest to the characteristic time of flocculation [14]. The modeling of the time-dependent behavior of the cementitious materials have attracted the attention of many researchers [20-24]. In a recent research, Wallewik [23] showed, by both his model and experimental work, that the time-dependent behavior is governed by both thixotropy (combination of coagulation, dispersion and re-coagulation of the cement particles) and structural breakdown (breaking of chemically formed linkages between the particles). Very detailed reviews for thixotropy and structural breakdown can be found in [19,24,28,29].

In recent years, the study of thixotropy and structural breakdown of SCC has become an interesting area of research owing to the fact that it would help concrete technologists to predict some aspects such as the static stability, segregation resistance, formwork pressure and its decay after casting, air entrapment, surface quality, interlayer bond strength between consecutive concrete layers and pumpability of concrete [15,30–34].

SCC behaves thixotropically showing good segregation resistance and low lateral pressure on formwork whereas it probably can increase entrapped air in fresh concrete and lift lines in multilayer casting, reducing bond strength between layers [15,31,35]. Therefore, for a given application the mixture parameters of SCC should be adjusted to achieve a given profile of thixotropic properties that can take into account the various requirements. Many parameters such as w/b ratio [36], binder type and content [2,37], aggregate characteristics and content [38] can affect the rheological properties, thixotropic and structural breakdown behavior of SCC. Besides the type and dosage of high-range water-reducing admixture (HRWR) [36], type and concentration of viscosity-modifying admixture (VMA) [39] as well as use of set-modifying admixtures [30] are important parameters in this respect.

Cementitious materials characteristics such as concentration, packing density, fineness and incorporation of SCM such as FA, SF and BFS are among the factors that affect the rheology and thixotropy of concrete [14,21,40]. Roussel et al. [41] reported that high thixotropy value in the mixtures containing SCM, like fine silica or limestone particles, is related to nucleation effect of these materials in PC mixtures. In fact, the increase in thixotropy without any workability loss can be obtained if the mixing power is sufficient to break the additional C–S–H bonds created by these products. It was reported that utilization of SCM in concrete increases the internal friction and hence attractive forces among solid particles, which in turn, increase the degree of physical and chemical bonds during cement hydration [14,21,40].

Assaad [2,5] studied the rheological and thixotropic properties of SCC containing SF, BFS and FA. It was reported that mixtures containing a binary cement (PC + SF), a ternary cement (PC + SF + FA) and a quaternary cement (PC + SF + FA + BFS) showed lower plastic viscosity and higher thixotropy values than corresponding plain SCC mixtures in the time interval between 0 and 30 min. However, reduction in the amount of thixotropy was observed in quaternary mixtures up to 150 min. It was also reported that mixtures with higher amount of cement content and lower amount of aggregate showed lower thixotropy values.

In a more recent study, Rahman et al. [16] reported that the use of SF in SCC in the range of 2.5–7.5% by weight of cement did not influence the thixotropy significantly from that of the control mixture. However use of FA in the range of 5–10% by weight of cement was found to increase the thixotropy of the mixture considerably.

In this paper, the rheology, thixotropy and structural breakdown of SCC made with various amounts of SF, FA, MK and BFS as a partial replacement of PC were studied. The aim of the first part of this study is to evaluate the effect of using these SCM in binary, ternary, and quaternary cementitious blends on HRWR demand and rheology. In the second part, the effect of these SCM on the thixotropy and structural breakdown of SCC mixtures was evaluated by different approaches. The effects of the SCM were also compared to those of a VMA. A total of 57 SCC mixtures were designed to have three w/b ratios with various binder contents. The measurements were made with a coaxial cylinder concrete rheometer.

#### 2. Research significance

Rheology, thixotropy and structural breakdown of SCC have been recognized as important tools to be tailored to achieve a multifold set of engineering properties required for successful accomplishment and performance of the intended application. For a given application, the mixture's properties should be adjusted to achieve a given profile of static stability, segregation resistance, formwork pressure, air entrapment, surface quality and interlayer bond strength between consecutive concrete layers. Utilization of SCM in concrete can not only improve the mechanical and durability properties of the mixtures, but also improve rheological and thixotropic properties as well as stability of the fresh concretes [1–3,5]. Although a number of studies about the effects of using FA, SF and BFS on the fresh and hardened properties of SCC have been found in the literature, the effect of using these SCM on the rheological and thixotropic properties were discussed only in limited number of studies [2,5,16]. Moreover, the potential benefit of using various amounts of SCM in ternary and quaternary blends on thixotropic properties of SCC is not well documented. The study presented herein aims at filling these gaps in the literature.

## 3. Experimental methods

## 3.1. Materials

The SCC mixtures studied in this study were prepared with an ordinary PC (CEM I 42.5 R), compliant to ASTM C 150 Type I [42]. SF, FAC, FAF, MK, and BFS were five SCM used in binary, ternary, and quaternary cementitious blends. The physical and chemical properties and particle-size distribution of PC, SF, FAF, MK, and BFS are presented in Table 1 and Fig. 1. Moreover, the particles of these SCM were inspected by a scanning electron microscope (SEM) as shown in Fig. 2. As seen in Fig. 1, SF is obviously the finest of all SCM. The next finer material was MK which was considerably different from the other SCM. In addition, the particle size distribution of PC, BFS, FAC and FAF were similar to each other. Crushed limestone aggregate with maximum particle size of 15 mm (0.59 in) and 4 mm (0.157 in), respectively for coarse and fine aggregate, were employed. The bulk specific gravity of the coarse and fine aggregates was 2.64 and 2.61, and their absorption capacities were 0.21% and 0.67%, respectively. A polycarboxylate ether-based HRWR conforming to ASTM C494 Type F [43] with specific gravity of 1.06 and solid content of 28% was employed. A liquid polysaccharide based VMA with 39% solid content was utilized in some SCC mixtures.

#### 3.2. Mixtures proportions

A total of 57 SCC mixtures having three w/b ratios (0.44, 0.50, and 0.56) with various binder contents (454.5 kg/m<sup>3</sup> (766.3 lb/  $y d^{3}$ , 400 kg/m<sup>3</sup> (674 lb/y d<sup>3</sup>) and 357 kg/m<sup>3</sup> (602 lb/y d<sup>3</sup>)) and constant water content were designed. The mix proportions are summarized in Tables 2-4. For all SCC mixtures the fine aggregate-to-total aggregate ratio, by mass, was set at 0.53. The HRWR dosages used in the mixtures were adjusted to secure an initial slump flow of  $650 \pm 10$  mm ( $25.59 \pm 0.39$  in). Three control mixtures were made with only PC as binder, whereas the other mixtures were made with binary (PC + SF, PC + FAC, PC + FAF, PC + MK and PC + BFS), ternary (PC + SF + BFS, PC + FAC + BFS, PC + FAF + BFS and PC + MK + BFS) and quaternary (PC + SF + FAC + BFS) cementitious blends by replacing a part of the PC with the SCM. The substitution levels were on mass basis by 4%. 8% and 12% for SF. 4%. 8%. 18% and 36% for MK. 18% and 36% for FA and only 18% for BFS. Furthermore, for each w/b ratio, additional mixtures were prepared without any SCM but with a constant amount of VMA (Tables 2-4). The dosage of VMA was selected according to the recommended dosage range given by

Table 1	
Physical and chemical	properties of PC and SCM.

	PC	SF	FAC	FAF	MK	BFS
CaO (%)	64.06	0.25	36.56	3.24	0.3	35.2
SiO <sub>2</sub> (%)	17.74	87.92	31.94	59.5	51.1	40.3
Al <sub>2</sub> O <sub>3</sub> (%)	4.76	0.4	13.5	18.5	39.1	10.2
Fe <sub>2</sub> O <sub>3</sub> (%)	3.17	0.35	4.09	6.96	2.15	0.67
MgO (%)	1.28	3.97	1.42	2.03	0.7	6.9
SO <sub>3</sub> (%)	2.94	0.21	3.86	0.47	0.08	1.4
K <sub>2</sub> O (%)	0.8	0.81	0.94	1.93	1.78	0.97
Na <sub>2</sub> O (%)	0.45	1.79	1.1	1.27	0.11	1.12
Free lime (%)	2.21	-	2.69	0.42	-	-
Other minor oxides (%)	0.64	1.43	0.91	1.26	0.88	1.34
Loss on ignition (%)	1.95	2.87	2.99	4.32	3.8	1.9
Specific gravity	3.13	2.29	2.73	2.38	2.54	2.97
Blaine fineness (cm <sup>2</sup> /g)	3310	-	3470	3220	-	3650
Surface area B.E.T. (cm <sup>2</sup> /g)	-	245,100	-	-	154,100	-
Residue 45 µm (%)	4.2	-	17.4	19.5	0.4	1.3

Notes:  $1 \text{ cm}^2/\text{g} = 0.488 \text{ ft}^2/1\text{b}$ .

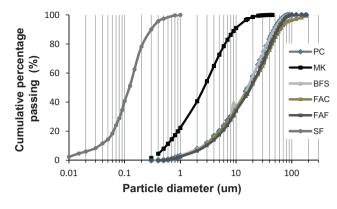


Fig. 1. Particle size distributions of PC and SCM.

the manufacturer. Rheology of SCC during its production depends on mixing efficiency, mixer type, mixing sequence, ambient temperature, etc. [44,45]. Therefore; these factors were kept constant throughout the study. The mixing procedure for concrete mixtures consisted of homogenizing the aggregate with 35% of the mixing water for 1 min in a batch of 20 L using a rotary planetary mixer. Cementitious materials with 40% of water were added following a rest period of 1 min to allow the saturation of the aggregates. Mixing continued 2 min and then the HRWR diluted with the remaining water was introduced gradually over 2 min. Following 2 min of rest, the concrete was mixed for 3 additional min. The ambient temperature during mixing and testing was maintained at approximately 20 ± 2 °C (68 °F). The mixtures were designated according to the type and the amount of cementitious materials included. For example, 8SF18FAC18BFS shows the guaternary mixture containing 8% SF, 18% FAC and 18% BFS.

## 3.3. Testing procedures

The measurement of fresh SCC properties was started as soon as mixing of all materials was finished (9 min after the initial contact of water with cement). The slump flow values were represented by the mean diameter (measured from two perpendicular directions) of the concrete spread after lifting the standard slump cone. After a slump flow of 650 mm (25.59 in) was ensured, the rheological parameters were evaluated using a coaxial cylinder concrete rheometer (ConTec 4SCC). A four-bladed vane rotating coaxially was used for the impeller. The container of the rheometer had an internal diameter of 240 mm (9.44 in) and a depth of 240 mm (9.44 in), allowing a concrete sample volume of approximately 8L (0.28 ft<sup>3</sup>). Following the rheology tests, thixotropy and structural breakdown tests were performed.

### 3.3.1. Evaluating torque plastic viscosity

The testing procedure consisted of increasing the impeller speed, *N*, gradually to 0.7 rps during 36 s subsequently reducing the speed stepwise to carry out the measurements of the torque, *T*, data. Each rotational velocity (0.70, 0.55, 0.40, 0.25, and 0.10 rps) was maintained for 6 s to ensure breakdown of the structure. The torque-rotational velocity data in the descending curve were linear corresponding to a Bingham fluid, as follows: T = g + hN, where *g* (in N mm) and *h* (in N mm s) are coefficients for apparent yield stress and torque plastic viscosity, respectively. The linear fit always resulted in coefficient of correlation ( $R^2$ ) values greater than 0.92.

## 3.3.2. Evaluating thixotropy and structural breakdown

Thixotropy and structural breakdown of the mixtures were evaluated by following three different ways. First method was the one recommended by Lapasin et al. [26]. In this method,

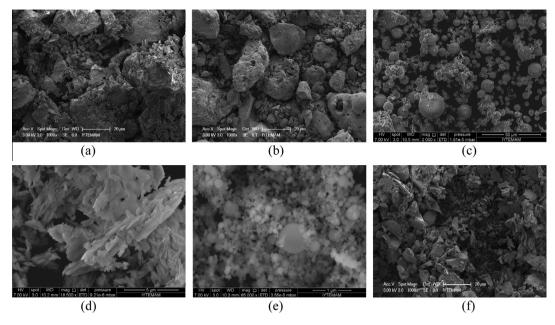


Fig. 2. SEM images of (a) PC, (b) FAC, (c) FAF, (d) MK, (e) SF and (f) BFS particles.

Table 2
Mix proportions of SCC mixtures having $w/b = 0.44$ (kg/m <sup>3</sup> ).

Mixture ID Water PC Si	Water	PC	SF	FAC	FAF	MK	BFS	VMA	HRWR	Aggregates (SSD)		$V_{\text{paste}}$ (L/m <sup>3</sup> )
								Fine	Coarse			
Control	200	454.5	-	-	-	-	-	-	5.75	883	783	365.6
4SF	200	436.5	18	-	-	-	-	-	6.7	880	778	368.6
8SF	200	418.5	36	-	-	-	-	-	7.5	875	774	371.5
12SF	200	400	54.5	-	-	-	-	-	8.0	870	771	374.1
8SF18BFS	200	337	36	-	-	-	81.5	-	6.7	874	774	372.1
18FAC	200	373	-	81.5	-	-	-	-	5.82	878	778	369.5
36FAC	200	291	-	163.5	-	-	-	-	6.35	872	773	373.8
36FAC18BFS	200	209.5	-	163.5	-	-	81.5	-	5.5	871	772	374.4
18FAF	200	373	-	-	81.5	-	-	-	4.67	872	772	372.8
36FAF	200	291	-	-	163.5	-	-	-	4.36	859	762	380.7
36FAF18BFS	200	209.5	-	-	163.5	-	81.5	-	4.31	858	761	382.0
4MK	200	436.5	-	-	-	18	-	-	6.18	881	781	367.3
8MK	200	418.5	-	-	-	36	-	-	6.5	878	779	368.9
18MK	200	373	-	-	-	81.5	-	-	7.5	873	773	373.2
36MK	200	291	-	-	-	163.5	-	-	10	861	762	381.7
36MK18BFS	200	209.5	-	-	-	163.5	81.5	-	9.35	860	761	382.5
18BFS	200	373	-	-	-	-	81.5	-	5.35	881	781	366.6
8SF18FAC18BFS	200	255.5	36	81.5	-	-	81.5	-	7.0	868	769	376.2
VMA	200	454.5	-	-	-	-	-	4.6	8.0	883	783	367.7

Notes:  $1 \text{ kg/m}^3 = 1.6842 \text{ 1b/y } \text{d}^3$ 

after five minutes of rest in the bowl of the rheometer, the mixture was subjected to a constant rotational speed of 0.2 rps for 10 s. Then, after another five-minute rest period, the mixture was subjected to a constant rotational speed of 0.4 rps for 10 s. The same procedure was repeated for speeds of 0.6 and 0.8 rps. For each speed, the time-dependent change in torque was recorded, and then the initial torque ( $T_i$ ) and equilibrium torque ( $T_e$ ) values were determined (Fig. 3a). Then, initial torque values and equilibrium torque values were plotted on torque-rotational speed graphs (Fig. 3b). Second order polynomial functions were fitted to the data (Fig. 3b). The area between the initial torque curve and equilibrium curve, calculated by integration, was used to quantify the thixotropy and structural breakdown. This area is called "breakdown area" (Fig. 3b).

Second method was similar to the one recommended by Legrand [46]. In this calculation method, only a part of the data

obtained for the breakdown area calculations were used. The difference between the initial torque  $(T_i)$  and equilibrium torque  $(T_e)$  values determined for 0.4 rps was divided by the rotational speed N = 0.4 rps. In other words, drop in apparent viscosity  $(\Delta_{app}$  was determined by  $\Delta_{app}(T_i - T_e/N)$ .

In the third method, after a rest period of 5 min in the rheometer bowl, a constant and very slow rotational speed of 0.03 rps was applied to the vane immersed in fresh concrete mixtures, and the resulting torque was measured as a function of time [27]. Torque-time graph shows a linear elastic region up to the yield torque value. Beyond this value, the bond is broken and the applied shear allows the flow of the material, therefore the torque value decreases. This yield (maximum) torque value ( $\tau_{r0}$ ), is called as "yield value at rest". A typical torque-time graph (Fig. 4) for the determination of yield value at rest is illustrated for 36FAF mixture with w/b ratio of 0.44.

#### Table 3

Mix proportions of SCC mixtures having  $w/b = 0.50 (kg/m^3)$ .

Mixture ID Water PC	Water	PC	SF	FAC	FAF	MK	BFS	VMA	A HRWR	Aggregates (SSD)		$V_{\text{paste}}$ (L/m <sup>3</sup> )
								Fine	Coarse			
Control	200	400	-	-	-	-	-	-	4.5	908	805	347
4SF	200	384	16	-	-	-	-	-	5.0	905	802	349.4
8SF	200	368	32	-	-	-	-	-	5.33	901	799	351.5
12SF	200	352	48	-	-	-	-	-	6.33	897	795	354.4
8SF18BFS	200	296	32	-	-	-	72	-	4.8	900	798	352.3
18FAC	200	328	-	72	-	-	-	-	4.6	905	801	350.5
36FAC	200	256	-	144	-	-	-	-	5.05	900	797	354.3
36FAC18BFS	200	184	-	144	-	-	72	-	4.47	898	796	355
18FAF	200	328	-	-	72	-	-	-	4.0	899	796	353.8
36FAF	200	256	-	-	144	-	-	-	3.67	890	787	360.7
36FAF18BFS	200	184	-	-	144	-	72	-	3.65	889	786	361.9
4MK	200	384	-	-	-	16	-	-	4.85	906	803	348.5
8MK	200	368	-	-	-	32	-	-	5.1	904	801	350
18MK	200	328	-	-	-	72	-	-	6.0	899	797	353.8
36MK	200	256	-	-	-	144	-	-	8.0	889	788	361
36MK18BFS	200	184	-	-	-	144	72	-	7.45	888	787	361.7
18BFS	200	328	-	-	-	-	72	-	4.30	907	803	348
8SF18FAC18BFS	200	224	32	72	-	-	72	-	5.6	895	793	356.4
VMA	200	400	-	-	-	-	-	4.6	6.0	908	805	348.4

Notes: 1 kg/m<sup>3</sup> = 1.6842 1b/y d<sup>3</sup>.

## Table 4

Mix proportions of SCC mixtures having  $w/b = 0.56 (kg/m^3)$ .

Mixture ID Water PC	ID Water F		SF	FAC	FAF	MK	BFS	VMA	HRWR	Aggregates (SSD)		V <sub>paste</sub> (L/m <sup>3</sup>
								Fine	Coarse			
Control	200	357	-	-	-	-	-	-	3.9	929	822	332.7
4SF	200	342.7	14.2	-	-	-	-	-	4.2	925	820	334.6
8SF	200	328.5	28.5	-	-	-	-	-	4.53	922	818	336.6
12SF	200	314.5	42.5	-	-	-	-	-	5.66	918	815	339.3
8SF18BFS	200	264.5	28.5	-	-	-	64	-	4.5	921	816	337.7
18FAC	200	293	-	64	-	-	-	-	4.0	925	818	335.8
36FAC	200	228.5	-	128.5	-	-	-	-	4.15	919	815	339.0
36FAC18BFS	200	164.5	-	128.5	-	-	64	-	4.0	918	814	340.0
18FAF	200	293	-	-	64	-	-	-	3.47	921	814	338.7
36FAF	200	228.5	-	-	128.5	-	-	-	3.23	911	807	345.0
36FAF18BFS	200	164.5	-	-	128.5	-	64	-	3.2	910	806	346.1
4MK	200	342.7	-	-	-	14.25	-	-	4.1	927	822	333.9
8MK	200	328.5	-	-	-	28.5	-	-	4.2	925	820	335.1
18MK	200	293	-	-	-	64	-	-	5.26	920	816	338.8
36MK	200	228.5	-	-	-	128.5	-	-	7.2	911	807	345.4
36MK18BFS	200	164.5	-	-	-	128.5	64	-	6.8	909	805	346.1
18BFS	200	293	-	-	-	-	64	-	3.65	928	820	333.6
8SF18FAC18BFS	200	200.5	28.5	64	-	-	64	-	4.87	917	812	341.0
VMA	200	357	-	-	-	-	-	4.6	5.48	929	822	334.2

Notes:  $1 \text{ kg/m}^3 = 1.6842 \text{ 1b/y } \text{d}^3$ .

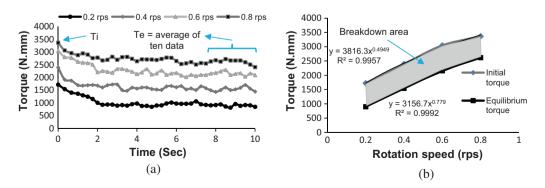


Fig. 3. Breakdown area method for the 36FAF mixture with w/b = 0.44 (a) breakdown curves and (b) area calculation.

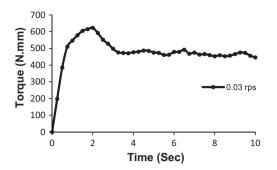


Fig. 4. Typical torque-time profile for the 36FAF mixture with w/b = 0.44.

### 4. Results and discussion

## 4.1. HRWR demand

The HRWR concentrations to produce a 650 mm (25.59 in) slump-flow diameter for each SCC mixture are illustrated in Fig. 5a-c. As can be seen in the left-hand side of the control mixtures, FAF and BFS decreased the HRWR requirement relative to the control mixture. In addition, replacement of PC by SF, MK and FAC increased the HRWR demand compared to that of the mixtures made with only PC. Incorporation of greater amounts of SF, MK and FAC present in binary, ternary, and quaternary cements necessitated greater HRWR demand, while the HRWR demand decreased as the FAF content increased.

Among the SCM having extremely high fineness (SF and MK), HRWR demand in the mixtures containing SF was more pronounced in comparison with MK. For example, HRWR demand was increased by approximately 40% when the percentage of SF was increased from 0% to 12% but the increase in HRWR demand was approximately 30% when the percentage of MK was changed from 0% to 18% for w/b ratio of 0.44. The greater HRWR demand of SF than that of MK was also noted in other investigations [8,47].

The high HRWR demand of SF is probably due to extremely fine nature and high surface area of grain content (Fig. 2e) and in the case of MK it maybe owing to their regular structure, elongated shape and high surface area of MK particles (Fig. 2d).

All mixtures containing FAC required additional HRWR to secure the target slump flow value, as compared to the control mixtures. On the other hand, in all w/b ratios, FAF exhibited the lowest HRWR demand values in binary and ternary mixtures. This can be attributed to the irregular shape, cellular surface and high porosity of the FAC particles (Fig. 2b) and to the spherical morphology, smooth surface texture of the grains and low porosity of the FAF grains (Fig. 2c).

In all of the ternary mixtures that contain BFS, HRWR demand was lower when compared to the mixtures without BFS. This effect of BFS can be attributed to the morphology of glassy slag particles as well as their smooth surface, resulting in low adsorption potential (Fig. 2f).

Mixtures containing VMA required more HRWR when compared to the control mixtures. The above results are in agreement with other studies [5,8,9,47].

The effect of w/b ratio on the HRWR demand is shown in Fig. 5d. As expected, HRWR contents increased as the w/b decreased. It is also possible to conclude that the trend of HRWR demand was similar for all w/b. In other words, the variation of HRWR demand with the binder system followed the same behavior regardless of the w/b ratio.

#### 4.2. Torque plastic viscosity

The torque plastic viscosity (h) results of the mixtures are given in Fig. 6a–c. As demonstrated in these figures for all w/b ratios, the partial replacement of PC by FAC, FAF and MK increased the h values of the mixtures (see the mixtures at the right-hand side of the

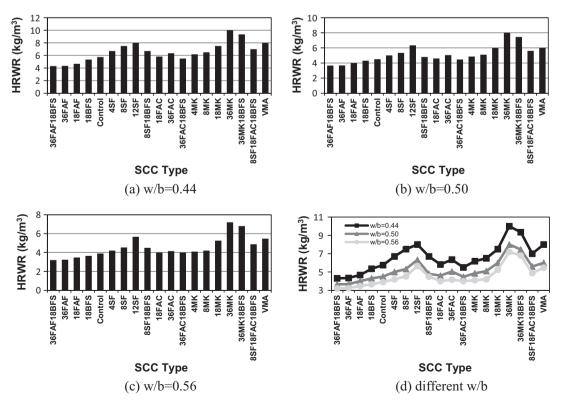


Fig. 5. HRWR demand of the mixtures with (a) w/b = 0.44, (b) w/b = 0.50, (c) w/b = 0.56 and (d) different w/b.

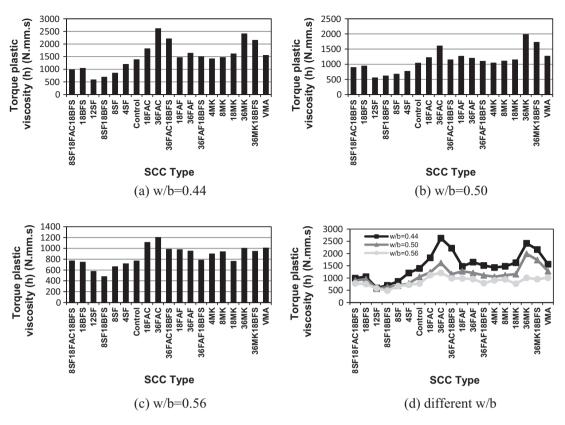


Fig. 6. Torque plastic viscosity of the mixtures with (a) w/b = 0.44, (b) w/b = 0.50, (c) w/b = 0.56 and (d) different w/b.

control mixtures in these figures). 36MK mixture with w/b ratio of 0.50, whose plastic viscosity was approximately 90% greater than that of the control mixture, showed the highest increase in plastic viscosity.

On the other hand, incorporation of SF and BFS reduced the h values in all w/b ratios (Fig. 6a–c). 12% replacement with SF in the mixture with 0.44 w/b ratio resulted in the greatest reduction (approximately 2.3 times less) in h. The amount of reduction was less as the percentage of SF was decreased. This behavior was contrary to the behaviors of MK and FAC: as replacement level of MK and FAC was increased, h values also increased. Meanwhile, except for the mixture with w/b ratio of 0.44, negligible reduction in h was observed when amount of FAF was increased. Similar results were reported by the other investigations for MK bearing mixtures [8]. However, the data related with the effect SF on the plastic viscosity of SCC is contradictory. Some of the researchers found that SF reduces the plastic viscosity (similar to the finding of this study) but the others reported that SF has not a significant effect on the viscosity of SCC [5,8].

The decrease in torque plastic viscosity due to the presence of SF in the SCC mixture was attributed to the following facts: The sphericity of SF particles provides better packing than PC particles resulting in lower shear stress and lower plastic viscosity values [21,48]. Another reason for reduction of h with increasing percentage of SF can be related to the difference in the volume of the binders. Owing to the difference between the specific gravity of PC and SF, mixtures containing SF had greater binder volumes causing reduction in h.

MK particles, owing to their high porosity, can absorb water and HRWR, leading to a reduction in inter-particle distance between solid particles and an increase in the level of attractive forces within solid particles that can be significant sake for obtaining high h values. Moreover, MK particles have elongated shape and

irregular surface texture (Fig. 2d), and contrary to the lubrication effect of spherical shape and smooth surface (as was the case in SF), they can reduce workability of SCC mixtures thereby increase the h values. Cassagnabere et al. [49] reported that in addition to high open porosity at the surface, elongated shape, irregular surface texture, least rounded and most rugged MK particles reduce workability and flowability of the mixtures.

In all w/b ratios, the mixtures containing FAC showed higher torque plastic viscosity in comparison with FAF ones. FAF particles, owing to their spherical shapes (Fig. 2c) which typically pack better than irregularly-shaped FAC particles, can lead to a higher ballbearing effect to reduce interparticle friction, thereby enhancing workability and reduction in h.

As shown in Fig. 6a–c lower *h* values were obtained in the quaternary system (8SF18FAC18BFS) in comparison with control mixtures and 18FAC mixture in all w/b ratios. In addition, utilization of 18% BFS with other SCM in ternary systems resulted in a considerable decrease in h values compared to those of binary systems without BFS. The same result was also obtained by Boukendakdji et al. [9]. They noted that the SCC mixtures incorporating BFS exhibited lower plastic viscosity than the reference mixture containing only PC. Moreover, the decrease in h was more pronounced when BFS content was increased.

The reason of this kind of treatment can be related to the degree of hydration of these mixtures. In quaternary mixtures, SCM replaced approximately 44% of PC by mass. Solid volume concentration obtained from hydration of PC and magnitude of restructuring and strength of inter-particle links which have significant effect in plastic viscosity values will be less in quaternary systems owing to the lower reactivity of SCM than PC. Moreover, in the quaternary system, PC is replaced with three types of finer SCM (SF, FAC and BFS). The fine particles of these SCM can fill the spaces between PC particles. Similarly, SF can take place between FAC and BFS particles, resulting in higher ball-bearing effect. This can help reducing the friction between cementitious materials and decrease the plastic viscosity of quaternary systems [50]. Another reason for the lower viscosity of quaternary mixtures is their higher paste volumes [21] (For example, paste volumes of quaternary and control mixtures with w/b = 0.44 are 376.2 and 365.5 L/m<sup>3</sup>, respectively, as can be seen form Table 3).

The torque plastic viscosity of VMA mixtures was higher than that of control mixtures, as expected. In this study, it was aimed to see whether VMA can be an alternative material to SCM in adjusting the plastic viscosity, which is necessary for stability of SCC. The results showed that VMA resulted in comparable *h* values and HRWR demand with SCM.

In all SCC mixtures, amount of water was kept constant and different w/b values were achieved by varying the binder content (Tables 2–4). Accordingly, the relative content of aggregate increased with the reduction in binder content as w/b ratio was increased. For example, when the control mixture is considered, aggregate volume (1000 L minus P.V. in Tables 2-4) was 634.4 L, 653 L and 667.3 L for w/b ratios 0.44, 0.50 and 0.56, respectively. In other words, aggregate volume increased by 5.19% when the w/b ratio was increased to 0.56 from 0.44. Binder content, aggregate content and w/b are among the most important factors affecting plastic viscosity of SCC mixtures. Higher degree of internal friction and resistance to flow thereby greater plastic viscosity is an expected phenomenon in SCC mixtures with high concentration of aggregate for a constant w/b ratio. On the other hand, higher degree of cohesiveness and progressive tendency to formation of a gel structure therefore greater amount of plastic viscosity in rich mixtures is reported by several authors [5,37,38,51]. As summarized in Fig. 6d, in spite of the high aggregate concentration in high w/b ratios mixtures, h values decreased as w/b ratio was increased. This kind of behavior proposes the importance of w/b ratio in SCC mixtures. Increase in the paste fluidity due to higher w/b ratio resulted in a decrease in plastic viscosity of SCC.

#### 4.3. Thixotropy and structural breakdown

#### 4.3.1. Breakdown area values

The breakdown area values for binary, ternary and quaternary mixtures containing SCM or VMA are given in Fig. 7a–c. As illustrated in these figures, greater values were obtained for SCC mixtures made with SF, FAC, FAF and MK when compared to that of the control mixtures. The increase was more significant as percentage of FAC, FAF and MK replacements were increased. The highest value, which was approximately 2.2 times greater than that of the control mixture, was observed in 36MK mixture with w/b ratio of 0.44.

Breakdown area calculations strongly depend on the factors that affect initial shear stress and equilibrium shear stress values determined during the tests [26]. As cited by Assaad et al. [5,40], Struble [52] suggested that particle packing and interparticle links responsible for flocculation are important factors that affect thixotropy in cement based system. It was reported that incorporation of SCM with higher surface area increases the number of inter-particle links and the level of attractive forces within solid particles, leading to higher initial shear stress values (upward shift of initial torque, *T<sub>i</sub>* curve in Fig. 3b). As long as the concrete is left at rest, the structural build-up becomes more significant, thus requiring higher initial shear stress  $(T_i)$  to break down the structure coming to the equilibrium shear stress. Tattersall et al. [21] concluded that in cement based systems, SCM with high packing density can reduce the equilibrium shear stress values by filling the voids and providing additional lubricant around aggregate particles (downward shift of equilibrium torque,  $T_e$ , values in Fig. 3b). Meanwhile, substitution of PC with SCM in cement based system

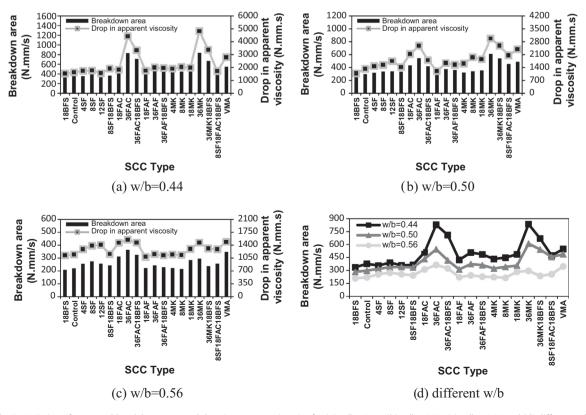


Fig. 7. Variation of structural breakdown area and drop in apparent viscosity for (a) w/b = 0.44, (b) w/b = 0.50, (c) w/b = 0.56 and (d) different w/b.

increases binder volume owing to the difference between the specific gravity of these materials, and therefore some decrease in equilibrium shear stress values can be expected [5]. When a concrete mixture which was left at rest for a sufficient time is sheared the particle spatial distribution and alignment become parallel to the flow direction, and the number of entanglements or associations decreases to a minimum, resulting in similar values of shear stress at equilibrium ( $T_e$ ) during shearing [5,53].

4.3.1.1. MK. Higher breakdown area values of the mixtures containing MK can be related to difference in the volume of the binders. Owing to the difference between the specific gravity of cement and MK, mixtures containing MK had greater binder volumes. For example, for w/b ratio of 0.44, paste volume of the control mixture was 365.6 L while that of the MK mixtures ranged between 367.3 L and 381.7 L. corresponding to an increase of 0.46% and 4.4% respectively. The increases in the paste volume were almost the same also for other w/b ratios. Higher paste volumes caused reductions in equilibrium torque values. Another reason for increase in thixotropy and structural breakdown values with higher percentage of MK can be attributed to the higher surface area and particle shape of MK particles: MK particles have higher surface area (Table 1), increasing the number of inter-particle links and the level of attractive forces within solid particles, and they have elongated shape (Fig. 2d), increasing the internal friction. Therefore, they can increase initial torque values.

4.3.1.2. FAC. Greater breakdown area values obtained for FAC mixtures in comparison with the control mixture containing only PC can be related to porous nature of the former binder. FAC particles, owing to their high porosity (Fig. 2b), can absorb water and HRWR. Hence, internal friction between solid particles can increase. As a result, coagulation of the system is increased and this can increase a higher initial torque values.

4.3.1.3. *SF and FAF.* In the case of mixtures containing SF, except some irregularity in mixtures having w/b ratio of 0.44, breakdown area values were also increased. Since spherical particles can be assumed to pack better than angular ones, like those of PC [48], higher packing density of the mixtures containing FAF and SF, which have spherical particles (Fig. 2c and e), can reduce the equilibrium torque values. In addition, higher binder volume of the mixtures with SF and FAF and high surface area of SF particles can reduce the equilibrium torque values and increase initial torque values.

4.3.1.4. BFS. As plotted in Fig. 7a–c, for all w/b ratios, breakdown area values were negligibly reduced when BFS were incorporated in the binary system. 18% replacement of BFS in the mixture with 0.44 w/b ratio caused in the greatest reduction (approximately 10%) in the results. Besides, in all ternary systems, use of 18% BFS with other SCM resulted in a considerable decrease in the breakdown area values compared to those of binary systems without BFS. The BFS particles have a high specific surface area (Table 1) and smooth surface (Fig. 2f). Moreover, they have lower hydration reaction ability than PC. Also, BFS particles can fill into the spaces between larger particles of PC and lead to a decrease in frictional forces in BFS blended mixtures [47,54]. Due to these factors, replacement of PC with BFS resulted in a decrease initial torque values.

4.3.1.5. Quaternary blend. As shown in Fig. 7a–c, combination of several SCM types in the quaternary system (8% SF, 18% FAC, 18% BFS) led to an increase in breakdown area values relative to the control mixture in all w/b ratios. The results of the quaternary system showed that the effect of SF (increase in initial torques and

decrease in equilibrium torques) and FAC (increase in initial torques) on increasing the breakdown area values was dominated to the effect of BFS (decrease in initial torques).

4.3.1.6. VMA. As seen in Fig. 7a–c, the breakdown area values of VMA mixtures were higher than that of control mixtures. Assad [5] reported that tending of water to be physically adsorbed by hydrogen and ionic bonding to the long chain polymers of the VMA is an important factor that affects the degree of thixotropy in fresh concrete. Besides, these molecules may develop attractive forces among each other, to form a gel and inter-particle links causing higher cohesiveness, thereby, greater degree of thixotropy. Depending on the VMA dosage, its effect on SCC rheology can be altered. In this study, only a single dosage of VMA which lies between the ranges recommended by the manufacturer was used. For the dosage value used in this study VMA resulted in comparable thixotropy values with other SCM.

4.3.1.7. *W/b.* As mentioned earlier, in all SCC mixtures, amount of water was kept constant and different w/b values were achieved by varying the binder content. Hence, the relative content of aggregate increased with the reduction in binder content as w/b ratio was increased (As can be seen in Table 2, the binder content and aggregate content of the control mixture with w/b = 0.44 was  $365.6 \text{ L/m}^3$  and  $634.4 \text{ L/m}^3$  respectively. However, when the w/b was increased to 0.56 (Table 4), the binder content decreased to  $332.7 \text{ L/m}^3$  and the aggregate content increased to  $667.3 \text{ L/m}^3$ ).

It is well known that the binder and aggregate contents and w/b ratio are among the important factors affecting the degree of thixotropy and structural breakdown of SCC mixtures. The increase in aggregate concentration can lead to higher degree of internal friction and produce greater collision and resistance to flow, thereby resulting in greater initial shear stress values. On the other hand, for constant water contents, after a given period of rest, individual binder particles begin to flocculate and develop internal network in mixtures with higher binder contents. This leads to higher degree of cohesiveness, progressive tendency to formation of a gel structure and inter-particle links and developing internal networks. Therefore, greater initial shear stress values are expected in rich mixtures [5,51]. As shown in Fig. 7d, breakdown area values decreased as w/b ratio was increased. From these results it can be emphasized that among the two opposite effects (high aggregate content and low binder content) on the degree of thixotropy and structural breakdown, the effect of reduction in binder content (or increase in w/b ratio) dominated the effect of increase in aggregate content.

Finally, it should be emphasized that the trend of change of breakdown area values with binder system was similar irrespective of w/b ratio of the mixtures (Fig. 7d).

#### 4.3.2. Drop in apparent viscosity values

As noted earlier, drop in apparent viscosity is an approach to assess the amplitude of the time-dependent phenomenon. The drop in apparent viscosity for 57 SCC mixtures with three different w/b ratios are presented in Fig. 7a–c. It is possible to conclude that for a given w/b, the change of drop in apparent viscosity values with binder system is similar to the change of breakdown area values as can be seen from any of Fig. 7a–c. Therefore, similar discussions made for the breakdown area values can also be made for drop in apparent viscosity values.

The relationships between the structural breakdown area and  $\Delta_{app}$  values determined at 0.4 rps for all SCC mixtures having different w/b ratios are plotted in Fig. 8a–c. From these figures, it can be concluded that good correlations with moderately high coefficient of correlation ( $R^2$ ) values ranging between 0.83 and 0.91 were observed. This shows that both the structural

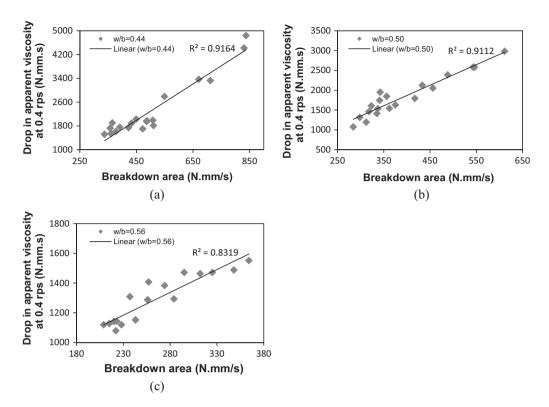


Fig. 8. Relationship between the structural breakdown area and  $\Delta_{app}$  values determined at 0.4 rps for (a) w/b = 0.54, (b) w/b = 0.50, and (c) w/b = 0.56.

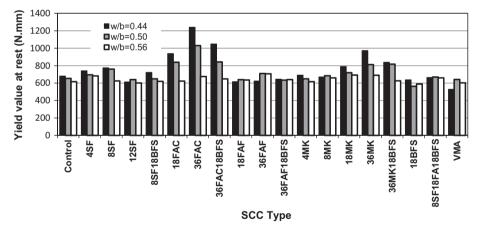


Fig. 9. Variation of at rest yield values for different w/b.

breakdown area method determined using four rotational speeds or the drop in apparent viscosity at a given rotational speed can be used to determine thixotropy and structural breakdown of SCC mixtures. However it should be noted that the breakdown area can be more exact in evaluating thixotropy as it employs four measurements, reducing the error in determining thixotropy and structural breakdown [2].

## 4.3.3. Yield value at rest

Yield value at rest indicates the coagulation state of the material that enables it to re-gain its shear strength when left at rest [5]. In this test, the interparticle links and attractive forces formed during the rest period are broken under very low shear rates. After elastic deformations, the bonds are broken upon reaching a critical stress (static yield stress), which is analogous to yielding behavior of steel

under tension. The bond strength (yield value at rest) depends on the initial structuring in the fresh concrete and the discussions made above on the increase in the initial torque values also hold true for yield values at rest: Increase in the number of interparticle links and level of attractive forces affect the yield values at rest. The properties of SCM that affect these factors include the water and HRWR absorption characteristics, fineness and shape of the particles.

The yield values at rest of the SCC mixtures containing different binder systems are given in Fig. 9. However, as can be seen from this figure, the results did not vary significantly even for different w/b ratios. Therefore, the differences between the mixtures were not clearly observable. Nevertheless, the mixtures with FAC had obviously higher values than the other mixtures especially at lower w/b ratios. This result is consistent with those determined by the breakdown area and drop in apparent viscosity methods. The correlation between the breakdown area and yield value at rest results was also investigated. The coefficient of correlation ( $R^2$ ) was 0.58, 0.46 and 0.04 for mixtures having w/b ratios of 0.44, 0.50 and 0.56, respectively. The obtained results showed that there are not significant relations between the breakdown area values and yield values. Similar to the observations made in Fig. 8,  $R^2$  increased as w/b ratio decreased. In other words, the thixotropy and structural breakdown data obtained by different methods get more consistent for lower w/b ratios. It can also be noted that the drop in viscosity and breakdown area correlations are better than yield value at rest and breakdown area correlations.

## 5. Conclusions

The main conclusions derived from this study can be summarized as follows:

- 1. For the same slump flow values, FAF and BFS decreased the HRWR requirement relative to the control mixture. In addition, replacement of PC by SF, MK and FAC increased the HRWR demand compared to the mixtures made with only PC. Incorporation of greater amounts of SF, MK and FAC necessitated greater HRWR than control mixtures.
- 2. The partial replacement of PC by FAC, FAF and MK increased the plastic viscosity of the mixtures, regardless of the w/b ratio. The highest increase (90%) in plastic viscosity was observed in 36% MK mixture with w/b ratio of 0.50. As the percentage of MK and FAC was increased, plastic viscosity was also increased.
- 3. Incorporation of SF and BFS reduced the plastic viscosity values in all w/b ratios. 12% replacement with SF in the mixture with 0.44 w/b ratio resulted in the greatest reduction (approximately 2.3 times less) in plastic viscosity.
- 4. Breakdown area of the mixtures containing SF, FAC, FAF, MK and VMA was higher than that of the control mixtures, regardless of the w/b ratio. The highest increase (2.2 times greater) was observed in 36% MK mixture with w/b ratio of 0.44. The amount of enhancement was stronger as percentage of substitution of FAC, FAF and MK were increased.
- 5. Breakdown area values were negligibly reduced when BFS were incorporated in the binary system. 18% replacement of BFS in the mixture having w/b ratio of 0.44 reduced the result approximately by 10%.
- 6. The quaternary system containing 8% SF, 18% FAC and 18% BFS led to an increase in breakdown area relative to the mixture containing only PC. Also in all ternary systems, use of 18% BFS with other SCM resulted in a considerable decrease in breakdown area compared to those of binary systems without BFS.
- 7. Thixotropy and structural breakdown was reduced as w/b ratio was increased. Since the water content of the mixtures was kept constant, any increase in w/b ratio caused reduction in binder content and consequent increase in aggregate content. Among these two factors having opposite effects on the thixotropy and structural breakdown, the former one dominated over the latter. The trend of change of structural breakdown area with binder system was similar for different w/b ratios.
- 8. Good correlations were established between structural breakdown area and drop in apparent viscosity values for each w/b. Hence, both the structural breakdown area method and the drop in apparent viscosity method can be used to determine thixotropy and structural breakdown values of SCC mixtures.
- 9. Comparison of the breakdown area and at rest yield values showed that there are not statistically clear relations between these approaches. Nevertheless, the different methods to evaluate the thixotropy and structural breakdown get more consistent when w/b ratio of the mixtures gets smaller.

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