Identification of Concrete Incompatibilities using Dynamic Shear Rheometer (DSR)

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Abstract

In concrete, the chemical interactions between chemical admixtures and portland cement is complicated and unpredictable. Occasionally these interactions may cause a serious set retardation or excessively fast stiffening. The objective of this study was to develop a reliable test method to predict potential concrete incompatibilities through the direct measurement of cement paste rheology using a dynamic shear rheometer (DSR).

Three cement paste mixtures with different dosage levels of a water reducing admixture were investigated with five different gaps of parallel plates in DSR. An effort was made to define which gap is the most sensitive and representative to identify incompatibilities between cement and chemical admixture at early ages. All the rheological tests were repeated three times to get the coefficient of variation. The results show that tests with 1mm plate gap is the most representative to distinguish three studied mixtures based on their rheological parameters with good reproducibility.

Introduction

The use of various chemical and mineral admixtures in portland cement concrete is a common practice, which sometimes deliberately or non-deliberately alter the hydration process and chemical interaction that leads to unexpected interactions between concrete ingredients. Complex chemical interaction between C_3A contents in cements, sulfate contents in pore solution, supplementary cementitious materials (SCM) and chemical admixtures along with temperature sometimes creates poor cement-admixture compatibility and can give rise to inadequate early workability or premature loss of workability along with setting time (early stiffening or excessive retardation) and heat evolution abnormalities (Taylor 2006).

For that reason, it is necessary to identify those concrete incompatibilities before the actual placement in order to avoid the problems in the placing and curing process. The measurement of rheological properties of cement pastes yields crucial information on the evolution of hydrating cementitious systems, micro-structural changes and particle interaction in cement paste (Zhang 2001), the relative performance of different chemical admixtures (e.g., water reducing admixture) - the optimum dosage and the consequences of excessive dosages (Ferraris and Obla 2001), and the compatibility of various cement, chemical and mineral admixtures combinations (Chen et al. 2006). Therefore, it is anticipated that cement paste (cement + supplementary cementitious materials + chemical admixtures + water) rheological measurements could be a good indicator to identify those incompatibilities (Banfill 2003). However, a successful application of the rheology approach needs a standard user friendly rheometer.

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Cement paste rheology studies using parallel plate fluid rheometer and rotational viscometers were conducted by several researchers in the past (Roussel 2006). It has been observed that mixtures with different admixture dosages and water to cementitious material ratio (w/cm) can clearly be distinguished based on paste rheological parameters such as viscosity and yield stress. Therefore, identifying cement-admixtures (chemical and mineral) incompatibilities through the measurement of paste rheological parameters instead of concrete rheological parameters is a sound concept (Ferraris 1992). Cement paste rheology measurement tests have the following advantages: (i) need much smaller amounts of testing materials, (ii) representative sample preparation by user-friendly preparation techniques, (iii) less testing time, (iv) more accurate results as it records true engineering properties of viscosity and yield stress, (v) less labor intensive than tests performed on concrete; existing concrete rheometers are with large size and heavy and not suitable for outdoor application. Moreover, aggregate effects in real concrete can be simulated during cement paste rheology measurements by some suitable means described in mixing procedure section (Yang and Jennings 1995). It is observed from the literatures that the parallel plate rheometer is more appropriate to impose these effects effectively than rotational concentric rheometers (Nehdi and Rahman 2004). The dynamic shear rheometer (DSR) had been adopted in superpave to characterize the viscous and elastic behavior of asphalt binders at high and intermediate service temperatures. The detailed DSR test procedure is given in AASHTO TP5. DSR is based on parallel plate technology and has the great potential to be considered as a user-friendly cement paste rheology measurement device after upgrading the device with necessary modifications. Most of the Department of Transportations (DoTs) have DSR as a standard test for measuring rheological properties of asphalt binder. Upgrading the existing DSR to fit into cement paste system is described in measurements section.

Research Significance

The objective of the present research is to investigate whether potential cement-mineral/chemical admixtures incompatibilities can clearly be identified through the direct measurement of cement paste rheology using the developed rheometer through laboratory testing program. This will ultimately help material suppliers, concrete producers, and other users to detect problematic combination of concrete ingredients before the concrete is placed and thereby, to avoid concrete cracking and other durability issues due to incompatibilities.

Measurements

Rheology measuring device. Two rheometers, DSR and AR 2000, were selected as laboratory rheology measuring devices in order to measure cement paste rheological parameters in a more accurate and effective way. DSR manufactured by Bohlin Instrument is commonly available in most of the DoTs whereas AR 2000 manufactured by TA Instrument is an advanced costly rheometer for research. The idea was to validate and establish DSR results through a comparative assessment between the results of the two rheometers. The rheometers with the adopted modifications are described below.

Two rheometers, DSR (Bohlin) and AR2000 rheometer, used in this study were upgraded at Texas Transportation Institute as shown in Figure 1. These two rheometers had been mainly used to characterize the viscous and elastic behavior of asphalt binders.

In the present study, both the rheometers were modified and optimized for measuring cement paste rheology by changing several features. Firstly, the Peltier heating/cooling system was installed to the DSR instead of a water-based temperature controller since the cement paste shouldn't comes in contact with water (in original DSR procedure, the tested asphalt specimen comes in contact with water while test is under progress to maintain the desired temperature). Secondly, a 240 grit size paper with adhesive back was installed in both upper and lower plates (25mm diameter for both the plates) in order to prevent slippage (Saak et al. 2001). The tested cement paste specimens are sandwiched between two parallel plates. An optimum gap was obtained by performing experiments with five different plate gaps (i.e., 0.2, 0.5, 1.0, 1.2, and 1.5 mm). The shear stress and strain rate of the tested specimens are recorded through a computer based data acquisition system. The test was carried out at a controlled temperature 23˚C ± 0.2˚C (73.4˚F ± 0.4˚F), which is the average cement paste temperature during the setting period. A computer program allows the user to customize test parameters, such as the number of readings, the gap of the parallel plate, the sampling interval between the readings, and specimen temperature. Both rheometers were programmed to sweep shear rate from $0.1 s^{-1}$ to $300\ s^{-1}$, and measure the corresponding shear stress. The yield stress and the plastic viscosity were determined by linear fit to the stress versus shear rate curve in accordance with Bingham model as defined in Equation 1.

$$\tau = \tau_0 + \mu\dot{\gamma} \quad \text{(Equation 1)}$$

where τ (in Pa) is the shear stress , τ_0 is the yield stress (calculated from the intercept of the best fit line), μ (Pa·s) is the plastic viscosity (determined from the slope of the best fit line), and $\dot{\gamma}$ (in s^{-1}) is the shear strain rate. The yield stress is a measure of the force necessary to start a movement of the paste, whereas the plastic viscosity is a measure of the resistance of paste against an increased speed of movement.

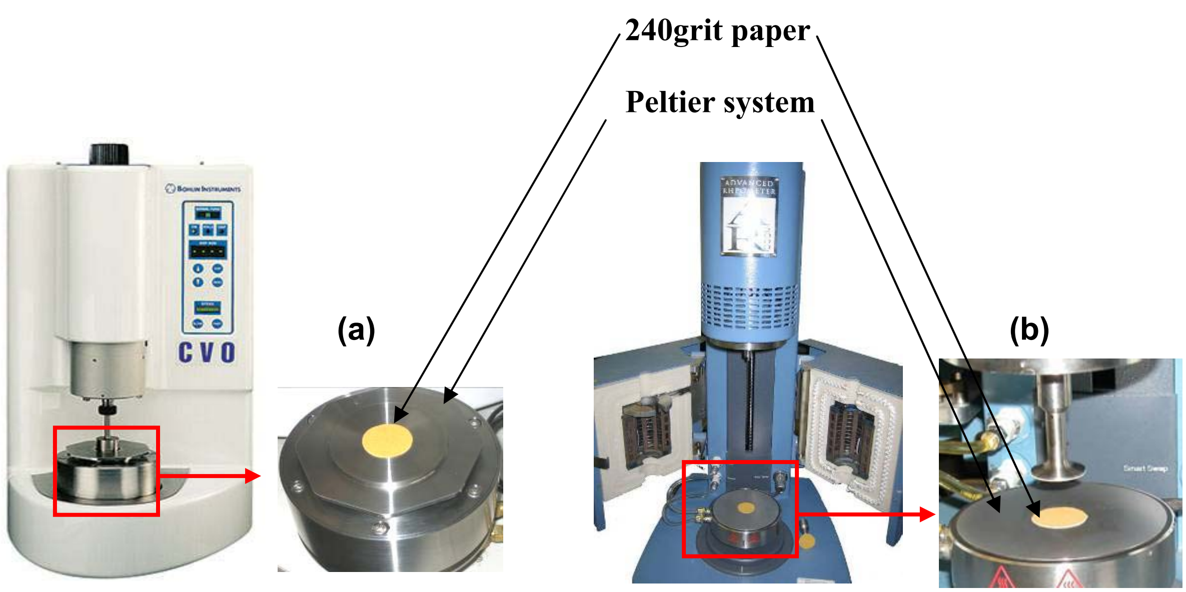

Figure 1 (a) Modified Bohlin rheometer (b) Modified AR2000 rheometer

Isothermal calorimeter and vicat needle apparatus were used to measure heat of hydration and setting time respectively as supporting tools in order to justify the rheology results. A short description of both the devices is given below

Isothermal calorimeter. An isothermal calorimeter (Model Super CRC) manufactured by Omnical Company was used to measure heat of hydration in fresh cement paste. Immediately after completing mixing procedure the cement paste was transferred into a glass cylinder, which was sealed at the top using a plastic layer cap and quickly placed to isothermal calorimeter. Heat evolution data were monitored and recorded during the time period of 50 hours with 12 seconds interval.

Vicat Apparatus. The setting time in this study was measured by the vicat apparatus according to ASTM C 191. A specimen of fresh cement paste was prepared with high shear mixer at 23°C (73°F) constant room temperature. Immediately after mixing the paste is placed in a frustum 40 mm (1.57 in.) in height. Initial set is considered as the time when the needle penetration is 25 mm ± 0.5 mm (1.53 in. ± 0.019 in.). The final set corresponds to less than 0.5 mm (0.019 in.) penetration.

Materials and Procedure

Materials. The materials that were used in this study are an ordinary ASTM Type I portland cement (OPC), a water reducing and retarding admixture (WRRA), and de-ionized water. The cement was characterized for its bulk chemical compositions by X-ray fluorescence listed in Table 1 and phase compositions by X-ray diffraction shown in Figure 2. The XRD data were collected in the range of 5° to 70° 2θ using the CuKα radiation. The step size was 0.02° and the scanning speed was 5° per minute. As expected, the four main minerals, i.e., tricalcium silicate (C_3S), dicalcium silicate (C_2S), tricalcium aluminate (C_3A), and tetracalcium aluminoferrite (C_4AF) along with gypsum and calcite (as limestone added to the cement) were identified in Figure 2(a) from the diffractogram. In order to ascertain the presence of any hemi-hydrate as some cements content hemi-hydrate due to excessive heat generation during clinker grinding, salicylic acid methanol extraction (SAM) method followed by quantitative X-ray diffraction (QXRD) were conducted as shown in Figure 2(b). SAM method dissolves the silicates (C_3S, C_2S) and increases the concentration of C_3A, C_4AF, and sulfates (gypsum/hemi-hydrate), which increases the possibility to detect and quantify hemihydrate (if any) by XRD. The results of mineral phase contents along with other physical properties are presented in the same Table 1.

The chemical admixtures used for this study was Daratard 17 provided by W.R. Grace. According to ASTM C 494, Daratard 17 is classified as Type B & D, i.e., water reducing and retarding admixture (WRRA). This is a common type of admixture that TxDOT has been using in the concrete pavement construction project. Daratard 17 is an aqueous solution of lignosulfonate and compound carbohydrates. The manufacturer's recommended range of dosage is 130-520mL/100kg of cement (2-8 fl oz/cwt) and typical dosage is 195mL/100kg (3fl oz/cwt).

Three cement pastes (P1, P2, and P3) with varying proportions of Daratard 17 are considered for the present laboratory investigation and their mixture proportions are presented in Table 2. P1 and P2 mixtures were formulated using a typical and a maximum recommended dosage of the studied WRRA respectively whereas P3 was formulated with excessively high dosage of the WRRA (i.e., double of the maximum recommended dosage). P3 mix was designed to create an artificial incompatible mix in the laboratory.

Table 1 Chemical and physical characteristics of cement

Component/properties	Percentage of Mass
Chemical composition (%)	
SiO_2 (%)	19.830
Al_2O_3 (%)	5.121
Fe_2O_3 (%)	1.853
CaO (%)	63.912
MgO (%)	1.208
SO_3 (%)	3.303
Na_2O (%)	0.115
K_2O (%)	0.474
CO_2 (%)	0.52
Equivalent total alkalis (%)	0.42
Insoluble Residue (%)	0.08
Limestone (%)	1.22
$CaCO_3$ in limestone (%)	96
Loss of ignition (%)	2.44
Chemical composition based Bougue's phase composition (%)	
C_3S (%)	63.00
C_2S (%)	9.326
C_3A (%)	10.44
C_4AF (%)	5.640
Sulfate minerals (%) from SAM extraction / QXRD	
Gypsum (%)	5
Hemihydrate (%)	1.4
Anhydrate (%)	0
Gypsum-to- hemihydrate Ratio	3.57
Physical properties	
Blaine Fineness (cm^2/g)	3920

Table 2 Mix design of cement paste

Mixture	Water L/m^3 ($gal./yd^3$)	Cement kg/m^3 (lb/yd^3)	w/c	Chemical Admixture Type	Chemical Admixture Dosage (%)	Range of Dosage
P1	550 (111)	1375 (2308)	0.4	WRRA	0.2% of cement weight	Typical recommended dosage
P2	550 (111)	1375 (2308)	0.4	WRRA	0.5% of cement weight	Maximum recommended dosage
P3	550 (111)	1375 (2308)	0.4	WRRA	1% of cement weight	Double of the maximum recommended dosage

Note: WRRA= water reducing and retarding admixture

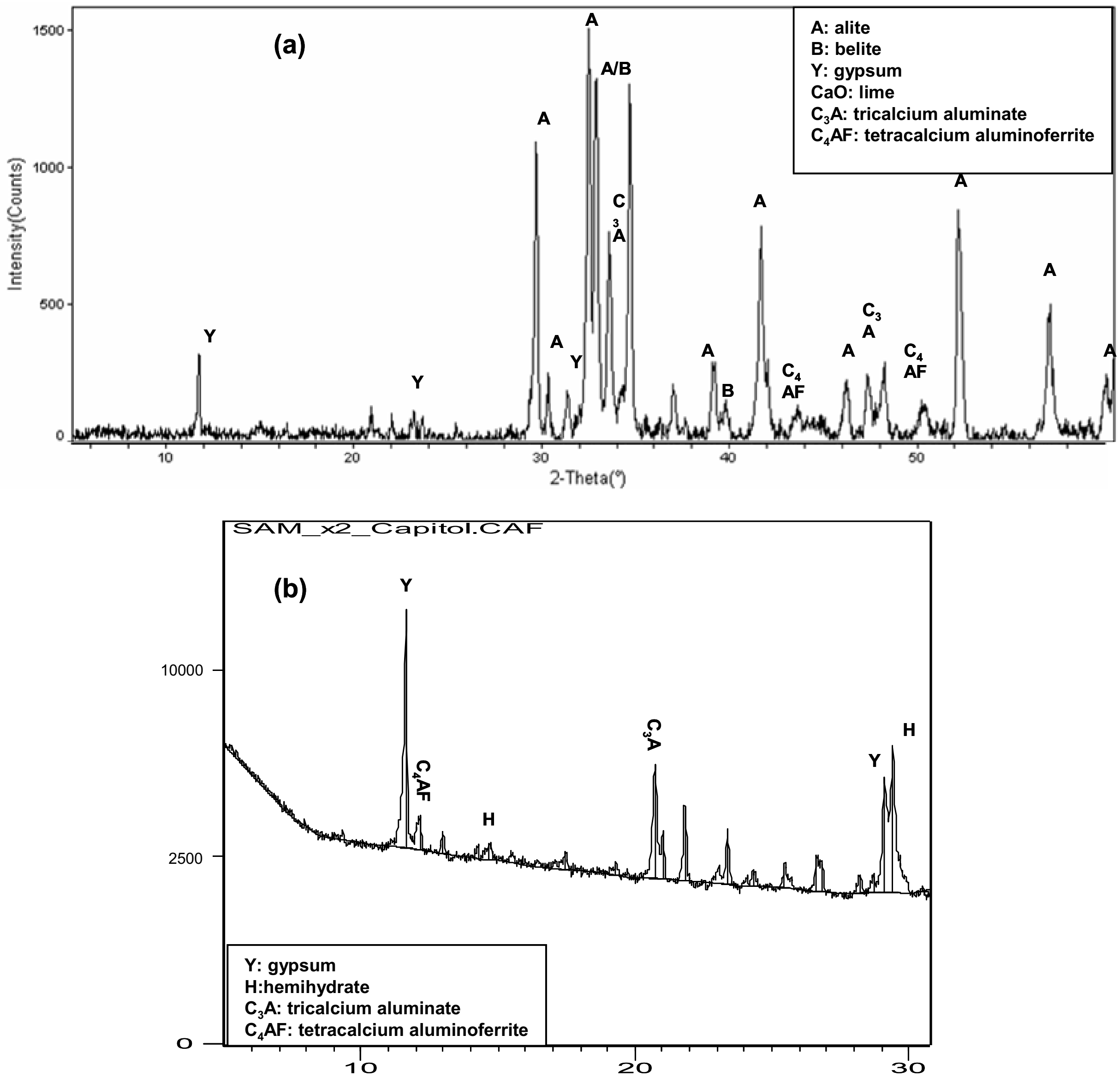

Figure 2 (a) XRD pattern of the bulk cement (b) XRD pattern of the cement after Salicylic Acid/Methanol Extraction (SAM)

Mixing procedure. The mixing procedure for the cement paste sample preparation was developed by Texas Transportation Institute with the basic steps adopted from the procedure developed by PCA and later on NIST (Ferraris 1996). A combination of two mixers, i.e., a planetary mixer (Model KSM-150PSMC, KitchenAid Company, 325 Watts, and up to 400 rpm) and a high shear mixer (Model ME-100LC, Charles Ross and Sons Company, Hauppauge, NY, USA), was used to develop the mixing procedure as follows:

- Pouring the measured water in the mixing container of the planetary mixer followed by switching on the mixer with 300 rpm speed.
- Adding the measured amount of cement powder slowly within 1 min to the container while the mixer impeller is in rotation with 300 rpm speed.
- Scrapping the sides of the mixing bowl with a rubber paddle with 1 min pause.

- Adding chemical admixture to the cement and water mixture in the container and mixing again with 400 rpm setting for another 30 sec.
- Removing the mixing bowl from the planetary mixer and placing it to the controlled temperature bath (bottom part of the bowl immersed in the bath) attached in the high shear mixer.
- Mixing in the high shear mixer with an initial speed of 2000 rpm for 1 min.
- Scrapping the mixer walls with a rubber paddle with 1 min pause.
- Mixing again in the same high shear mixer with a higher speed (i.e., 5000 rpm) for another 30 seconds.
- Placing back the container in the planetary mixer and continue mixing with slow rpm till the first measurement starts.

In the high shear mixer, two uniaxial impellers drive the paste through narrow apertures located in a small ring around the base of the support column as shown in Figure 3. The speed of the mixer was electronically controlled at a specified rpm. The above mixing procedure is a close representation of the shearing effects that cement paste experienced due to coarse aggregates during concrete mixing. The application of a temperature controlled 18°C (65 °F) water bath while mixing in the high shear mixer allowed maintaining the paste temperature to be approximately 24 to 25°C (75 to 77°F) at the end of the mixing cycle. The slightly cooled base of the mixer container is allowed to simulate the heat sink effects by the aggregates in concrete. Without the temperature controller, the temperature of the cement paste would have been much higher due to heat generation from friction of high shear mixing in addition to the heat of hydration and this would have been affected the hydration of the paste as well as its rheological behavior (Williams et al. 1999).

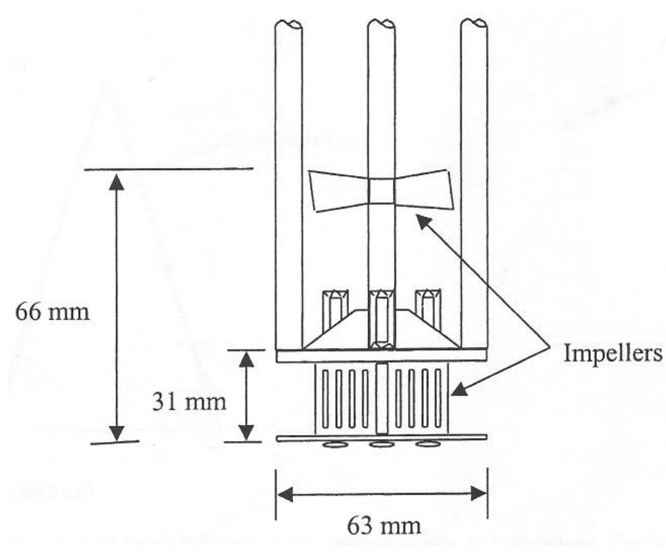

Figure 3 Ross High Shear Mixer (Model ME-100LC)

Rheometer test procedure. Each mixture, according to mixture proportion in Table 2, was prepared by using the mixing procedure described in the previous section. Each mix was tested for a total of 5 plate (25 mm diameter) gaps (i.e., 0.2, 0.5, 1.0, 1.2, and 1.5 mm) for both the rheometers where a fresh mix was prepared for each test corresponding to each gap. Therefore, a total of 45 runs for all three mixtures having 15 runs (3 runs for each plate gap) were conducted for each rheometer. A typical data of shear rate versus shear stress is presented in Figure 4(a). The plastic viscosity and yield stress were determined from Bingham model described in Equation 1 using the slope of the linear region of the down curve of the hysteresis loop illustrated in Figure 4(b). Average viscosity and yield stress and their respective coefficient of variation (CV%) based on 3 runs were calculated corresponding to each test and repeated the same calculation for all 90 runs (45 runs for each rheometer) and are presented in Figures 5 for DSR and 6 for AR 2000.

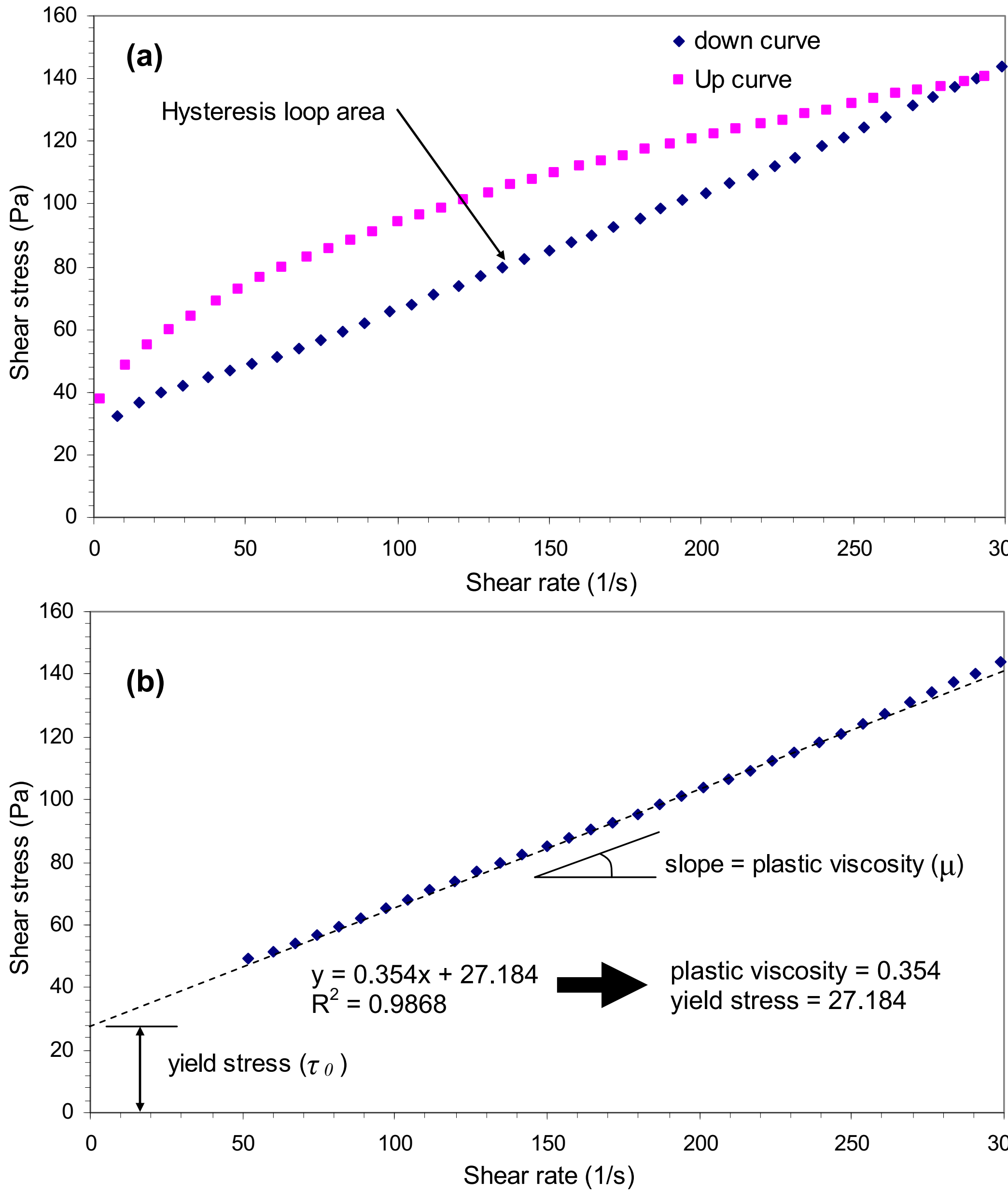

Figure 4 (a) Typical shear stress vs. shear rate (b) calculation of rheological parameters using Bingham model for the mixture containing 0.2% of WRRA with 1.2 mm plate gap

Results and Discussion

Rheometer tests. The plastic viscosity and yield stress as a function of gap between two parallel plates and dosage of WRRA for all 15 combinations with DSR (Bohlin) are graphically presented in Figure 5(a) and 5(b) respectively. CV% of viscosity and yield stress corresponding to the same 15 combinations are compared in Figure 5(c). A perusal of Figure 5 showed the following observations:

- Both plastic viscosity and yield stress corresponding to each cement paste mixture (P1, P2, and P3) decreased obviously with increasing dosage of WRRA for all the 5 plate gaps – similar trend of paste viscosity and yield stress using high-range water reducing admixture are reported in the literature (*4*) and this supports the present observation
- The greater rate of decrease of both viscosity in Figure 5(a) and yield stress in Figure 5(b) are the characteristics with plate gaps of 0.2, 0.5 and 1.0 mm whereas smaller rate of decrease of both the parameters are noticed for plate gaps of 1.2 and 1.5 mm.
- The rheological parameters with large gaps (1.2, and 1.5 mm) showed similar values in accordance with smaller rate of decrease, which didn't allow to distinguish three mixtures based on viscosity and yield stress, although reproducibility still remained good.
- CV% of both viscosity and yield stress with 1mm plate gap was found to be approximately below 10% indicating good reproducibility whereas those for plate gap smaller than 1 mm (i.e., 0.2 and 0.5mm) was found to be more than 10% indicating poor reproducibility as shown in Figure 5c.

Similarly, the viscosity, yield stress, and CV% as a function of plate gap and WRRA dosage for the same 15 combinations with AR 2000 rheometer are graphically represented in Figure 6(a), (b), and (c) respectively. Following are some important observations based on the AR 2000 test results. The same decreasing trend of viscosity and yield stress as a function of WRRA dosage was observed in AR 2000 for all the studied mixtures and plate gaps. The same higher CV% of viscosity and yield stress with smaller plate gaps (i.e., 0.2, 0.5 mm) and lower sensitivity (especially with yield stress) with the higher plate gaps (i.e., 1.2, 1.5 mm) were also evident in AR 2000 data.

The following key observations were made based on a comparative assessment of the test results of both the rheometer.

- Both the rheometers with 1mm plate gap can clearly distinguish three mixtures (P1, P2, and P3) with the lowest CV% which implied its better sensitivity and reproducibility.
- Below 1mm plate gap, the sensitivity still remained good, however, reproducibility became poor as manifested by CV% > 10. Permissible reproducibility of low viscous materials (P3) can still be maintained with lower plate gap (e.g., 0.2, 0.5mm) whereas reproducibility for high viscous materials (P1) with lower plate gap can't be maintained.
- Above 1mm plate gap, reproducibility remained good as manifested by CV% < 10, however, sensitivity became poor since no such considerable difference between rheology of three mixtures was noticed.

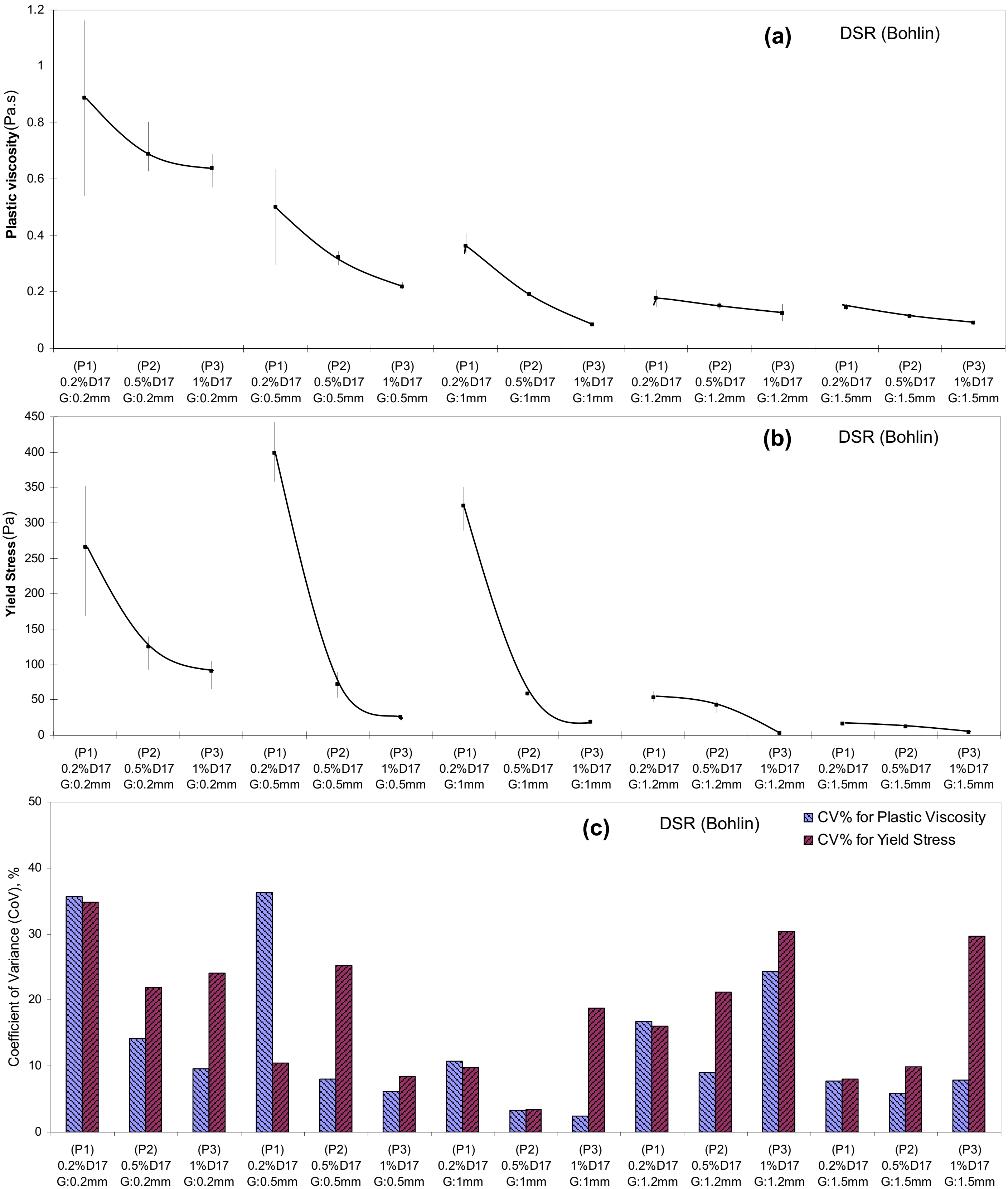
(a)
DSR (Bohlin)
Plastic viscosity(Pa.s)
(b)
DSR (Bohlin)
Yield Stress(Pa)
(c)
DSR (Bohlin)
CV% for Plastic Viscosity
CV% for Yield Stress
Coefficient of Variance (CoV), %
(P1) 0.2%D17 G:0.2mm
(P2) 0.5%D17 G:0.2mm
(P3) 1%D17 G:0.2mm
(P1) 0.2%D17 G:0.5mm
(P2) 0.5%D17 G:0.5mm
(P3) 1%D17 G:0.5mm
(P1) 0.2%D17 G:1mm
(P2) 0.5%D17 G:1mm
(P3) 1%D17 G:1mm
(P1) 0.2%D17 G:1.2mm
(P2) 0.5%D17 G:1.2mm
(P3) 1%D17 G:1.2mm
(P1) 0.2%D17 G:1.5mm
(P2) 0.5%D17 G:1.5mm
(P3) 1%D17 G:1.5mm

Figure 5 (a) Plastic viscosity (b) yield stress, and (c) CV% data from DSR (Bohlin)

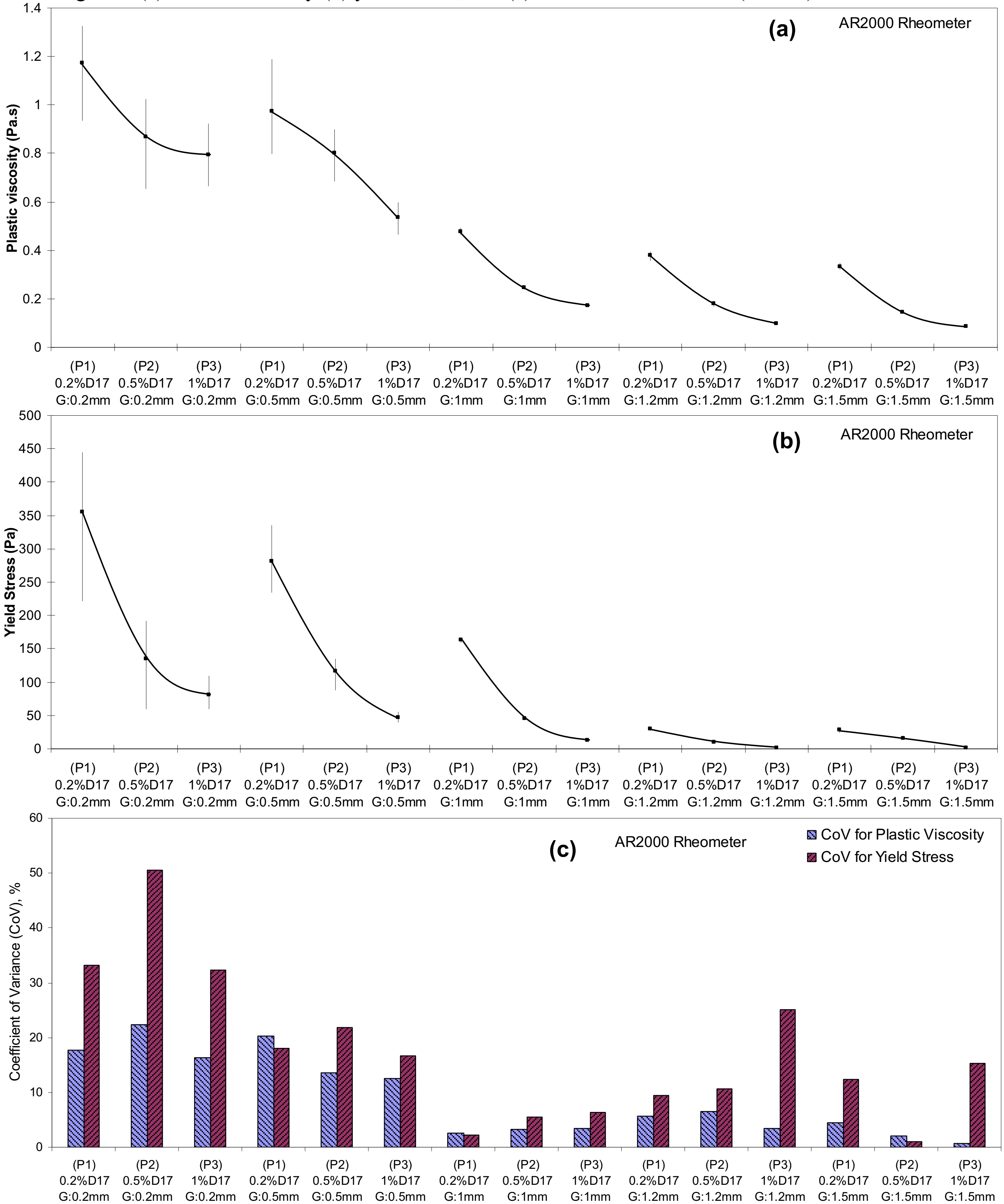

Figure 6 (a) Plastic viscosity (b) yield stress, and (c) CV% data from AR2000 rheometer

Heat of hydration test. The isothermal calorimeter tests were performed for each mixture (P1, P2, and P3) as a supporting tool to verify whether these three mixtures can clearly be distinguished (in accordance with the rheology based distinction) based on their heat evolution characteristics at early ages. All the mixtures were tested three times to verify the reproducibility in the same manner of the rheology tests. Figure 7(a) shows that the occurrence of the second peak of heat evolution is a function of WRRA dosage and induction period of cement hydration increases as the dosage of WRRA increases. As for example, in P3 mixture containing high dosage of WRRA the induction period is around 16 hours showing the evidence of incompatibility whereas in P2 mixture it is around 4-5 hours. P1 mixture behaves as a normal mixture. A good reproducibility of the heat of hydration results for all the three mixtures is manifested by closer superimposition of the three graphs generated from three consecutive tests. Figure 7(b) shows that the integrated heat evolution drastically decreases as the dosage of WRRA increases, i.e., three mixtures having three clearly distinct integrated heat evolution curves. Therefore, the three mixtures have clearly been distinguished based on their heat evolution characteristics, which support the rheology-based observation in the previous section.

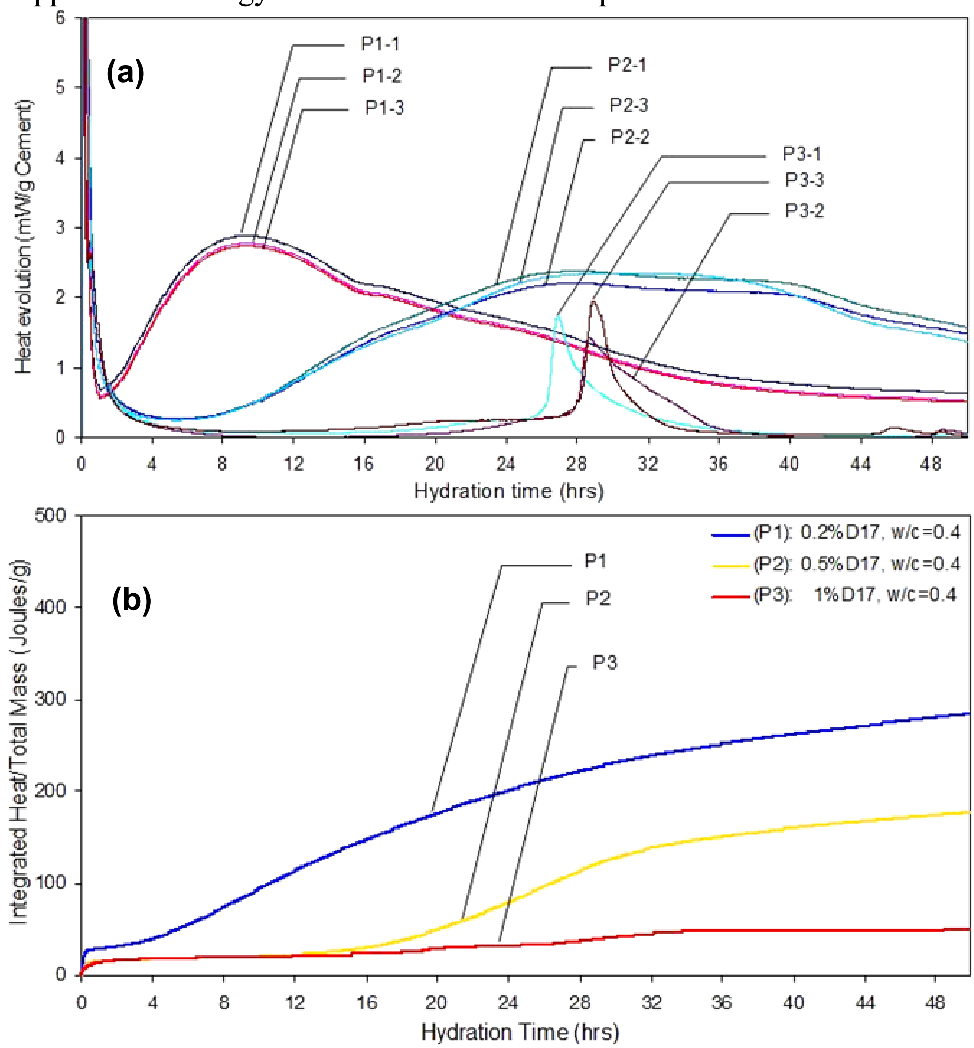

Figure 7 (a) Heat evolution (b) integrated heat evolution data as a function of time

Setting time test. Three different mixtures (P1, P2, and P3) listed in Table 2 were tested three times repeatedly by vicat needle apparatus tests. Coefficient of Variation (CV%) based on three measurements of initial and final setting time was calculated for each mixture to verify the reproducibility of the test results. The results of initial and final setting time, and their CV% from vicat test for all the three mixtures are presented in Figure 8. Figure 8(a) shows that the initial and final set is drastically retarded with P3 mixture containing high dosage of WRRA. Incompatibilities in P3 mixture is clearly manifested by setting time abnormality and this is in accordance with both heat of hydration and rheological behavior as discussed previously. A good reproducibility in setting time results for all the three mixtures is evident in Figure 8(b).

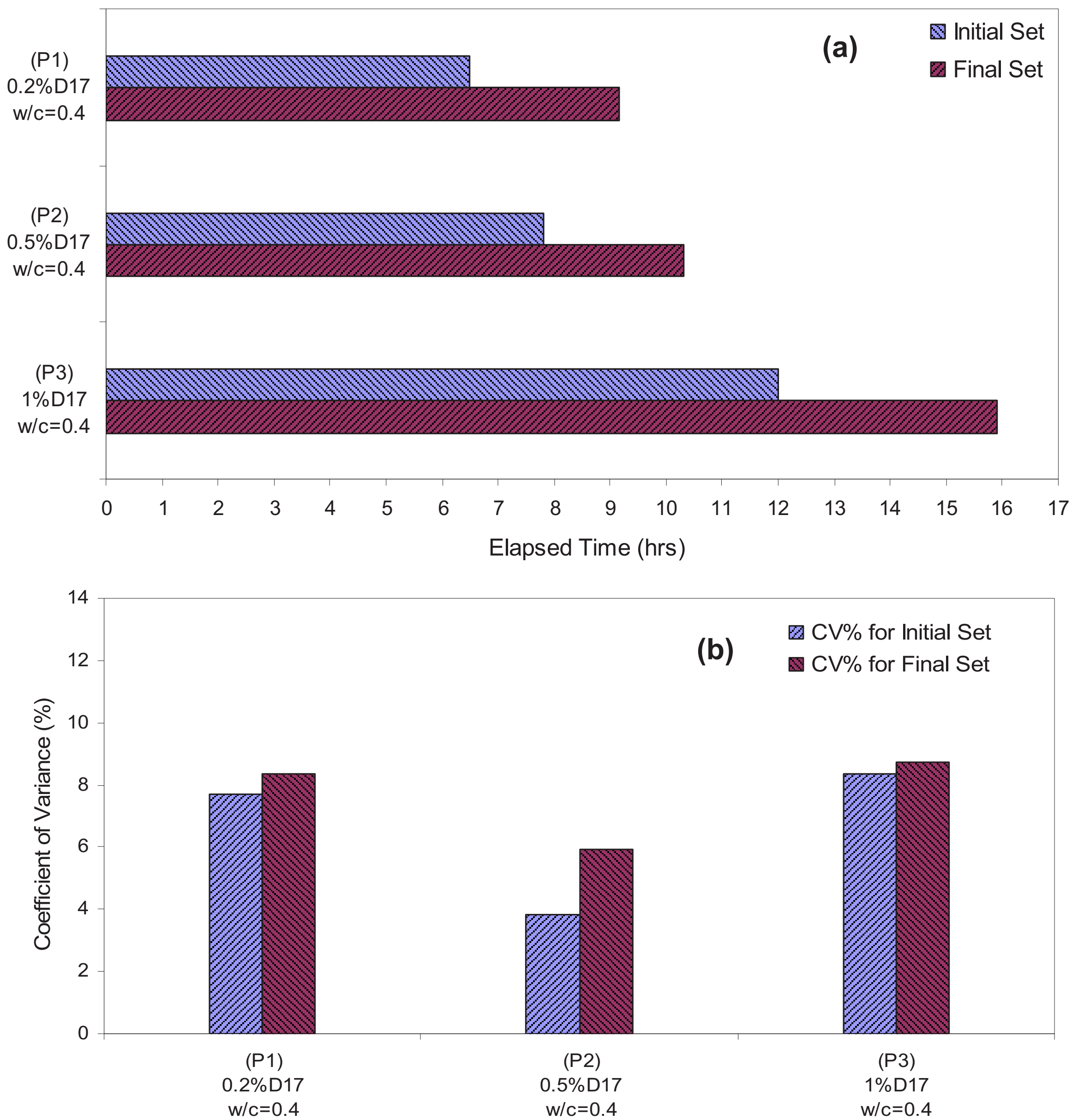

Figure 8 (a) Initial and final set (b) CV% for initial and final set from vicat needle test

Conclusions

The rheological parameters as a function of chemical admixture dosages and plate gaps were investigated using two rheometers, DSR (Bohlin) and AR2000 rheometer. Isothermal calorimeter tests for heat of hydration and vicat apparatus tests for setting time were performed and analyzed as supporting evidences. Based on the results presented in this paper, the following conclusions can be drawn:

1. Cement paste rheology technique using the modified dynamic shear rheometer (DSR) has a great potential to identify cement-chemical admixture incompatibilities.
2. Both the rheometers in modified form are capable to measure cement paste rheology with permissible reproducibility and sensitivity.
3. For both rheometers, a 1mm plate gap is the most representative to distinguish three studied mixtures based on their rheological parameters with the best reproducibility and sensitivity
4. The decreasing trend of viscosity and yield stress as a function of WRRA dosage remain almost the same in both rheometers. The sensitivity and reproducibility of both plastic viscosity and yield stress as a function of plate gaps (e.g., 0.2, 0.5, 1.0, 1.2, and 1.5mm) remained almost the same in both the rheometers. This was considered as a reason of validation for DSR (readily available in DOTs) to be considered as a standard rheometer for cement paste rheology measurement. The purpose of using AR 2000 in this research was to validate the DSR test results.
5. The heat of hydration data from isothermal calorimeter test and setting time behaviors from vicat needle test have strongly supported the rheology based observations. In other words, incompatible mixtures can also be detected based on heat of hydration and setting time characteristics, however, these test methods are time consuming and labor intensive compared with the rheometer test.

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