Cement and Concrete Research 39 (2009) 1007–1016

Contents lists available at ScienceDirect

Cement and Concrete Research

journal homepage: http://ees.elsevier.com/CEMCON/default.asp

# Estimating time and temperature dependent yield stress of cement paste using oscillatory rheology and genetic algorithms

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ARTICLE INFO

*Article history:*
Received 21 October 2008
Accepted 15 July 2009

*Keywords:*
Cement paste
Rheology
Modeling
Genetic algorithm
Temperature
Mixing time
Superplasticizer
Yield stress

ABSTRACT

A controlled shear stress–shear rate rheometer was used to determine the viscoelastic behavior of cement paste incorporating various superplasticizers and subjected to prolonged mixing at high temperature. At a low applied shear stress range, the oscillatory shear strain/stress curve of cement paste was characteristic of a linear elastic solid; while the higher stress range was characteristic of a viscous liquid exhibiting a linear strain increase with increasing applied shear stress. The transition from solid-like to liquid-like behavior occurred over a very narrow stress increment. This transition stress corresponded to the yield stress parameter estimated from conventional flow curves using the Bingham model. The yield stress from oscillatory shear stress tests was estimated using the intersection between the viscous part of the oscillatory shear strain/stress curve and the oscillatory shear stress axis. In this study, equations describing the variation of shear strain versus shear stress beyond the solid–fluid transition for cement pastes incorporating various superplasticizers at different ambient temperatures and mixing times were developed using genetic algorithms (GA). The yield stress of cement pastes was subsequently predicted using the developed equations by calculating the stress corresponding to zero strain. A sensitivity analysis was performed to evaluate the effects of the mixing time, ambient temperature, and superplasticizer dosage on the calculated yield stress. It is shown that the computed yield stress values compare well with corresponding experimental data measured using oscillatory rheology.



## 1. Introduction

Oscillatory shear tests are a rheological technique capable of providing information on the properties of viscoelastic materials. Fresh cement paste is considered as a viscoelastic material since it has both elastic and viscous properties. In recent years, oscillatory shear tests have been given special attention by several researchers who studied the rheology of cement paste [1–5]. Using this technique, the elastic and viscous behavior of cement paste can be characterized and it can be determined whether cement particles are dispersed or flocculated [6]. This technique was used for instance to investigate the continuously changing cement paste structure during hydration, which could not be investigated effectively using conventional flow tests, because such tests cause changes in the microstructure of cement paste [7].

Indeed, cement paste with flocculated cement particles is sensitive to the shear history, making the measurement of its yield stress a difficult task [8]. Conventional flow curves allow estimating the yield stress through extrapolation of the flow curve to determine the shear stress at zero strain [8]. One of the major drawbacks of this technique is the fact that yield stress is measured when cement paste is flowing (viscous state) after its structure has already been broken. Furthermore, the value of yield stress estimated using conventional flow curves depends on the rheological model used for its calculation since each model can fit the experimental shear stress–shear strain data differently [9]. Conversely, an oscillatory shear test estimates the yield stress of cement pastes by depicting the shift point from its elastic to viscous state. This is done through subjecting the cement paste to a sinusoidal shear stress and measuring the corresponding strain. The applied oscillatory stress can be expressed as follows:

$$\sigma = \sigma_o \cos \omega t \tag{1}$$

For an ideal elastic solid, the resulting oscillatory strain will be in phase with the stress and is expressed as follows:

$$\gamma = \gamma_o \cos \omega t^* \tag{2}$$

For a viscous liquid, the resulting oscillatory strain will be 90° out of phase with the stress, and is expressed in the following equation:

$$\gamma = \gamma_o \cos\left(\omega t^* - \frac{\pi}{2}\right) \tag{3}$$

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0008-8846/$ – see front matter © 2009 Published by Elsevier Ltd.
doi:10.1016/j.cemconres.2009.07.011

Thus, the resulting oscillatory strain for a viscoelastic material such as cement paste is given by:

$$\gamma = \gamma_o \cos(\omega t^* - \delta) \quad (4)$$

In the equations above $\sigma$ is the shear stress (Pa), $\sigma_o$ is the amplitude of shear stress (Pa), $\omega$ is the angular velocity (1/s), $t^*$ is time (s), $\gamma$ is the shear strain, $\gamma_o$ is the amplitude of shear strain, and $\delta$ is the phase shift. When a viscoelastic material is subjected to a sinusoidal stress, the resulting sinusoidal strain will have a lag of $\delta$ with that of stress, which varies in the range of 0 to $\pi/2$ [10,11].

At low shear stress, cement particles are typically in close contact with each other and cement paste behaves as a solid material, where its microstructure is not disturbed and can recover elastically [12]. When the applied stress becomes sufficiently high and reaches a certain value, the strain becomes significant and cement particles begin to separate from each other, thus cement paste starts to flow. The critical stress at which the cement paste transforms from elastic to viscous state corresponds to the yield stress parameter of the Bingham equation:

$$\tau = \tau_o + \mu_p \dot{\gamma} \quad (5)$$

Where $\tau$ is the shear stress (Pa), $\tau_o$ is the yield stress (Pa), $\mu_p$ is the plastic viscosity (Pa s), and $\dot{\gamma}$ is the shear rate ($s^{-1}$).

In the present study, the oscillatory shear stress technique was applied to cement pastes incorporating various superplasticizers and subjected to prolonged mixing at high temperature. The objective is to find a methodology based on the experimental oscillatory shear stress test results to predict the yield stress of superplasticized cement pastes under various ambient temperatures and mixing times.

## 2. Experimental procedure

The cement paste mixtures tested in this study are identical to those previously investigated by the authors using the conventional flow curve technique [13]. Ordinary Portland cement Type I was used along with liquid superplasticizers including a polycarboxylate-based superplasticizer (PC), melamine sulfonate-based superplasticizer (ML), and naphthalene sulfonate-based superplasticizer (NS) . The water to cement ratio (w/c) was 0.38 and the dosages of superplasticizers ranged from 0.2 to 0.4% for PC, 1.8 to 2.4% for ML, and 1.2 to 2.0% for NS by mass of cement.

The cement paste mixtures were subjected to prolonged mixing for up to 110 min at various ambient temperatures (22–45 °C). For the moderate temperature, the mixing took place in the lab environment with a controlled temperature of 22 °C, and for high temperatures (35 and 45 °C); the mixing was carried out in an environmental chamber with controlled temperature. The shear stress–shear strain and oscillatory shear tests were performed using an advanced rheometer (TA-Instruments AR-2000) with a coaxial concentric cylinders geometry. The geometry of the test accessory has a significant influence on the measured rheological properties of cement paste [14]. The coaxial concentric cylinder geometry was considered suitable for this study and was thus used throughout. This geometry consists of a smooth surface cylinder with a conical end that rotates inside a cylinder with a central fixed hollow. The gap between the rotating shaft and the hollow is 1 mm. It is necessary to use such a narrow gap so that the shear rate remains constant across the gap, which is important to minimize the effect of error in rheological measurements caused by wall slip [5]. The gap between the head of the conical end and the bottom of the hollow was set at 0.5 mm and kept constant for all experiments. It should be noted that no slippage or sedimentation of cement paste was observed during the experiments.

The first rheological measurement was taken at 20 min after the first contact between cement and water. Subsequently, a new cement paste sample from the same mixture subjected to continuous mixing was tested every half hour for up to 110 min.

The flow test procedure is described in detail elsewhere [13]. For the oscillatory shear test, the cement paste was subjected to a shear rate at 70 $s^{-1}$ for 2 min to create uniform conditions before testing by breaking down its structure [5]. The cement paste sample was then left to rest for 5 min in order to rebuild its broken structure. The pre-shear (70 $s^{-1}$) for 2 min was assured to be sufficient by pre-shearing the cement paste for 2, 3, or 4 min and conducting the oscillatory time sweep test immediately (which does not allow any time for the structure to form) after each pre-shearing time. It was found that the values of the initial shear modulus for all cases were comparable, indicating that this pre-shearing method is sufficient to break down the structure of the cement paste sample creating uniform conditions. It was also found that the increase in the shear modulus-time curve stopped after a time period less than 5 min and became constant over time, indicating that the 5 min period is enough for cement paste to fully rebuild its structure. Subsequently, a stress sweep was applied by stressing the cement paste sample from 0.008 to 500 Pa, and the resulting strain was recorded. The frequency of the oscillatory shear was maintained constant at 1 Hz throughout the rheological testing.

## 3. Experimental results

Fig. 1a shows a typical descending part of a conventional flow curve for cement paste. It is well established that cement paste reasonably follows a Bingham behavior [15], and therefore the flow curve in Fig. 1a was fit to the Bingham Eq. (5). The yield stress (the parameter of concern in this study) is determined by extrapolation of the shear stress–shear rate curve corresponding to a zero shear rate, and the plastic viscosity is the slope of this curve as shown in Fig. 1a.

Fig. 1b illustrates a typical oscillatory shear strain/stress curve displayed only over a narrow range of oscillatory shear stress (less than 10 Pa) to clearly visualize the transition of cement paste from a solid-like material to a liquid-like material. The relationship between oscillatory shear strain and oscillatory shear stress can be divided into three regions as shown in Fig. 1b. Region (1), the zone below the yield point, represents the elastic solid behavior of fresh cement paste. The

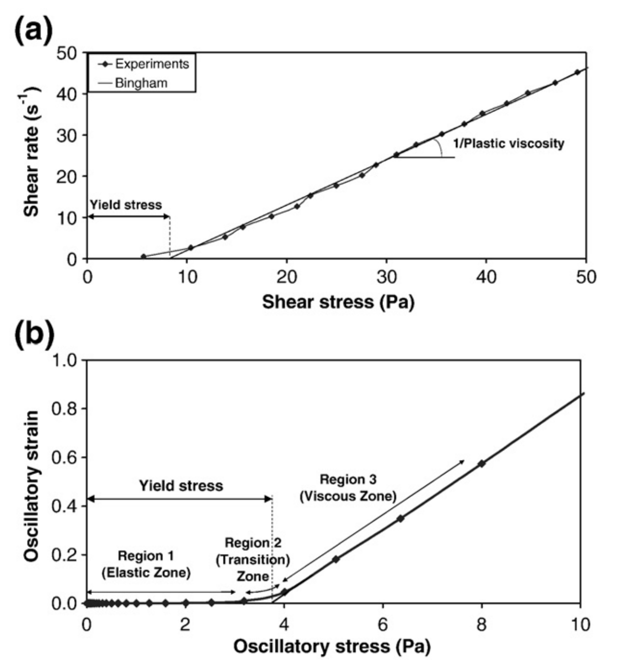


**Fig. 1.** Typical rheological curves: (a) shear rate-shear stress flow curve (only down curve shown), and (b) oscillatory shear strain/stress curve.

strain in this region is very small (about $10^{-6}$). The relationship between strain and stress is linear with a marginal slope (almost zero). At low stress, cement particles are flocculated due to mutual attraction forces exerted by a combination of van der Waals and electrostatic forces, and they stick together to form agglomerates. The elastic linear region of the oscillatory shear stress–shear strain curve indicates that at a small strain the structure of cement paste deforms without breakdown of its structure [6].

In region 2 (transition zone), the relationship between the oscillatory shear strain and oscillatory shear stress follows a quasi curve-linear form, which represents the beginning of structure breakdown of cement agglomerates. This zone identifies the shift of fresh cement paste from an elastic to a viscous state (the solid–liquid transition). The transition from solid-like to liquid-like behavior can be observed across a remarkably narrow stress increment (usually 1 Pa or less).

In zone 3, the cement paste deforms largely under the applied stress; the relationship between strain and stress is linear (Fig. 1b). The yield stress can be reasonably estimated as the intersection point between the linear viscous part of the strain/stress curve with the oscillatory stress axis as shown in Fig. 1b.

The yield stress for cement pastes at different ambient temperatures and mixing times and incorporating ML are estimated using both a conventional flow test (Bingham model) and an oscillatory shear test (Fig. 2). It can be observed that there are only 2 points below the equity line, but more than 30 points above it. Thus, the flow test generally gives a yield stress about 5 Pa higher than that from the oscillatory test. Struble and Schultz [1] reported a similar observation for cement pastes at different w/c and stated that the value of yield stress determined using the oscillatory technique estimated better the yield stress than that using the flow curve technique.

Fig. 3 shows the effect of the mixing time and ambient temperature on the yield stress of cement pastes incorporating various superplasticizers as estimated using the oscillatory shear test. It can be observed that the yield stress increased with the increase of mixing time, which is in agreement with findings of previous work [13] using conventional flow tests. The yield stress of cement pastes also increased with the increase of temperature (Fig. 3b). The rate of this increase became higher at higher temperature, which reflects the acceleration of hydration reactions of cement at higher temperature [16].

## 4. Database

The genetic algorithm (GA) technique used in this study to model the yield stress of cement pastes must be trained using a relatively large and comprehensive number of representative data sets in order to become able to capture the relationships between the input and output parameters. In total, 108 cement paste mixtures were tested (36 mixtures for each superplasticizer used). For each superplasticizer, 26 mixtures were used during the training process, and the rest were used for testing the model. GA-based equations were optimized

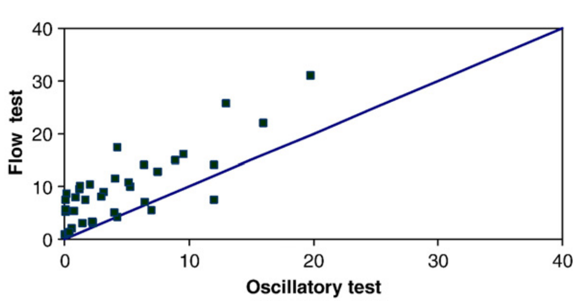


**Fig. 2.** Yield stress value (in Pa) estimated from shear flow tests versus corresponding oscillatory shear tests data for cement pastes at different temperatures, mixing times, and ML dosages.

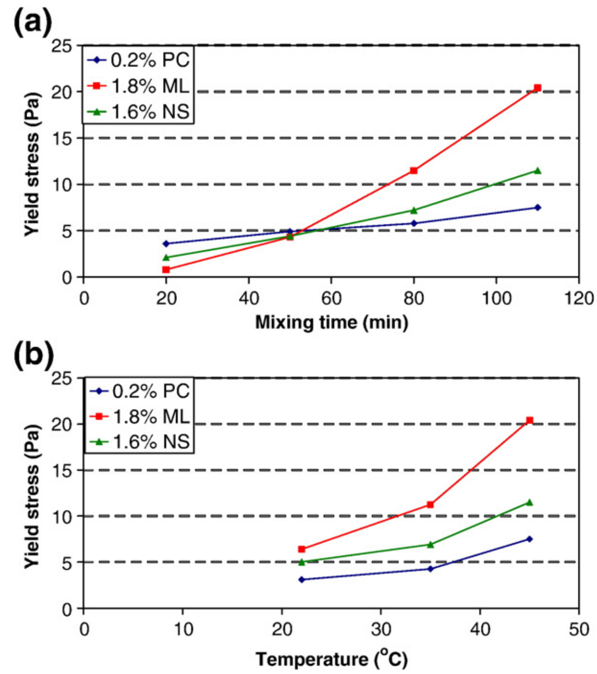


**Fig. 3.** Variation of yield stress of cement pastes versus: (a) mixing time at 45 °C, and (b) temperature after 110 min of mixing.

to predict the viscous part of the oscillatory shear strain/stress curve for each cement paste mixture. Each oscillatory shear strain/stress curve consists of 10 points. For each admixture, the highest value of shear stress at the end of the transition zone between the linear and viscous parts of the curve for all experiments performed on this admixture was depicted. Taking advantage of the fact that the viscous region is linear, the ten data points for each admixture were chosen from the range after the highest value of shear stress depicted (the viscous part). Hence, 260 oscillatory stress–strain data points (26 mixtures multiplied by 10 points each) for each superplasticizer were used for the training process, while 100 data points (10 mixtures multiplied by ten points each) were used for testing.

## 5. Modeling methodology

The rheology of cement paste is time, temperature, and shear history dependent; it is usually affected by high variability. Thus, modeling cement paste rheology considering the various factors is complicated and needs a powerful tool. The genetic algorithm approach was selected due to its advantages over the traditional regression models [17]:

- In GA modeling only the ranges of coefficients need to be determined, while in traditional regression analysis the estimates of the coefficients need to be determined and in some programs the derivatives of the equations need to be identified.
- GA modeling uses objective functions, not its derivatives. Hence, solving multivariable complex functions using GA, which considers several solutions at the same time in an evolutionary mode, can be considered advantageous when there is risk for the solution to be trapped in local optima or minima.

The genetic algorithm method is a global optimization technique for high dimensional, nonlinear, and noisy problems. It can be defined as a search technique based on the mechanism of natural selection and the natural genetics of biological evolution. The main characteristic of GA is its ability to provide optimum solutions to approximately formulated problems. GA starts the search of an optimum solution with an initial coded set of random solutions called *population*. The population includes a set of individuals called *chromosomes*; each chromosome represents a potential solution to the problem at hand. The process of GA optimization simulates the *Darwinian evolution* process to create populations from one generation to another. As such, GA selects the *fittest* individuals in the initial population and subsequently undergoes genetic operations including *selection*, *crossover*, and *mutation* to produce a new generation having a genetic structure *superior* to that of the *parents* [18].

The appropriate selection of some key model parameters (*selection method* and *pressure*, *recombination* type and rate, mutation rate and number of individuals) is critical to have a successful performance of GA. The selection method and pressure provide the driving force in a genetic algorithm towards promising solutions in the search space, and they are selected based on the complexity of the optimized problem. Selecting a high crossover rate reduces the chances of a false optimum because it allows exploration in the large solution space. However, a too high value of recombination rate might result in a waste of time due to a substantial computational effort in exploring unpromising regions of the solution space. If the mutation rate is too low, many useful chromosomes might not be tested, and if the mutation rate is too high, there will be much random perturbation and consequently the *offspring* will start losing resemblance to their *parents*, resulting in an algorithm which is unable to learn from the history of the search [19].

As mentioned earlier, the yield stress can be defined as the intersection between the viscous region of the oscillatory shear strain/stress curve and the oscillatory shear axis. This linear relationship between oscillatory strain and stress was found to have the following form:

$$\gamma = a \times \tau - b \tag{6}$$

where $a$, and $b$ are the experimental coefficients that are functions of the mixing time ($t$), ambient temperature ($T$), and superplasticizer dosage ($D$) (Figs. 4 and 5). These relationships can be presented as follows:

$$a(t) = -c_{at} \times t + e_{at} \quad \text{(for all superplasticizers)} \tag{7}$$

$$a(T) = -c_{aT} \times T + e_{aT} \quad \text{(for all superplasticizers)} \tag{8}$$

$$a(D) = +c_{aD} \times D + e_{aD} \quad \text{(for all superplasticizers)} \tag{9}$$

$$b(t) = -c_{bt} \times t + e_{bt} \quad \text{(for PC)} \tag{10}$$

$$b(t) = +c_{bt} \times t + e_{bt} \quad \text{(for ML and NS)} \tag{11}$$

$$b(T) = -c_{bT} \times T + e_{bT} \quad \text{(for all superplasticizers)} \tag{12}$$

$$b(D) = -c_{bD} \times D + e_{bD} \quad \text{(for all superplasticizers)} \tag{13}$$

where $c_{at}$, $e_{at}$, $c_{aT}$, $e_{aT}$, $c_{aD}$, $e_{aD}$, $c_{bt}$, $e_{bt}$, $c_{bT}$, $e_{bT}$, $c_{bD}$, and $e_{bD}$ are experimental coefficients.

Table 1 shows the experimental ranges of these coefficients for each superplasticizer; these ranges were used by the GA model in its search for optimum solutions. Using the method of separation of variables, the oscillatory shear strain/stress relationship (Eq. (6)) can be expressed as follows:

For PC:

$$\begin{aligned}\gamma = {} & [(-c_{at}t + e_{at})(-c_{aT}T + e_{aT})(c_{aD}D + e_{aD})] \\ & \times \gamma - [(-c_{bt}t + e_{bt})(-c_{bT}T + e_{bT})(-c_{bD}D + e_{bD})]\end{aligned} \tag{14}$$

For ML and NS:

$$\begin{aligned}\gamma = {} & [(-c_{at}t + e_{at})(-c_{aT}T + e_{aT})(c_{aD}D + e_{aD})] \\ & \times \gamma - [(c_{bt}t + e_{bt})(-c_{bT}T + e_{bT})(-c_{bD}D + e_{bD})]\end{aligned} \tag{15}$$

Where $t$ is time (min), $T$ is temperature (°C), and $D$ is the superplasticizer dosage (% by cement mass).

The genetic algorithms technique (e.g. see Michalewicz [18] and Goldberg [19]) was used to optimize the coefficients of Eqs. (14) and (15) so that the viscous part of the experimental oscillatory shear strain/stress curves for various cement paste mixtures can be predicted with acceptable accuracy within the ranges of test parameters, i.e. mixing time, ambient temperature, and superplasticizer dosage. Separate equations were developed for different superplasticizers, because it was found in previous work by the authors [2] that these superplasticizers had different effects on the rheology of cement pastes.

The GA search process was terminated when a set of coefficients was found that minimizes the objective function constructed to measure how well the model-predicted output agrees with the corresponding experimentally measured data. Table 2 presents the parameters used in the genetic algorithm setting. The three optimized equations are as follows:

For cement pastes incorporating PC:

$$\begin{aligned}\gamma_{PC} = {} & [(-0.001t + 0.8) \times (-0.01T + 0.66) \times (200D + 0.41)] \\ & \times \tau - [(0.001t + 0.21) \times (-0.0001T + 2) \times (-400D + 2)]\end{aligned} \tag{16}$$

For cement pastes incorporating ML:

$$\begin{aligned}\gamma_{ML} = {} & [(-0.005t + 2.4) \times (-0.0065T + 0.4) \times (126D - 2)] \\ & \times \tau - [(0.021t - 0.41) \times (-0.00001T - 0.21) \times (-453D + 6)]\end{aligned} \tag{17}$$

For cement paste incorporating NS:

$$\begin{aligned}\gamma_{NS} = {} & [(-0.0027t + 0.861) \times (-0.018T + 1.5) \times (71.4D - 0.68)] \\ & \times \tau - [(0.009t + 0.0011) \times (-0.0226T + 0.001) \times (-188.6D + 0.65)]\end{aligned} \tag{18}$$

## 6. Results and discussion

### 6.1. Validation of GA-based oscillatory shear strain/stress equations

The performance of the GA-based oscillatory shear strain/stress equations was assessed by examining their ability to predict experimental data not previously used during the training process, and thus unfamiliar to the model. A comparison between the experimental and GA model-predicted oscillatory shear strain of cement paste in the viscous range is presented in Fig. 6, which shows both training and testing data points. The results for cement paste incorporating PC at 22, 35, and 45 °C are illustrated in Fig. 6a, b, and c, respectively. It is shown that the data points predicted by the GA model are located on or within a small range around the equity lines. Similar to PC, the predicted data points for ML are located close to the equity lines (Figs. 6d, e, f). The data points corresponding to NS are depicted in in Figs. 6g, h, and i. Again the GA model proved to be able to predict the viscous part of the oscillatory shear strain/stress curve for cement paste made with NS. Fig. 6 shows that strain values are lower at higher temperature. This is due to the fact that cement paste gets stiffer (deforms less) with increasing temperature.

To further validate the ability of the GA-based oscillatory shear strain/stress equations developed in this study to accurately predict the strain values of cement pastes with variation of the mixing time and ambient temperature, the average absolute error (*AAE*) (Eq. (19)) along with the ratio of measured to predicted shear strain ($\gamma_m/\gamma_p$) were calculated.

$$AAE = \frac{|\gamma_m - \gamma_p|}{\gamma_m} \times 100 \tag{19}$$

Table 3 presents the values of the standard deviation (*SD*) and coefficient of variation (*COV*) of the measured-to-predicted oscillatory shear strain ratio and average absolute error (*AAE*) for all testing data

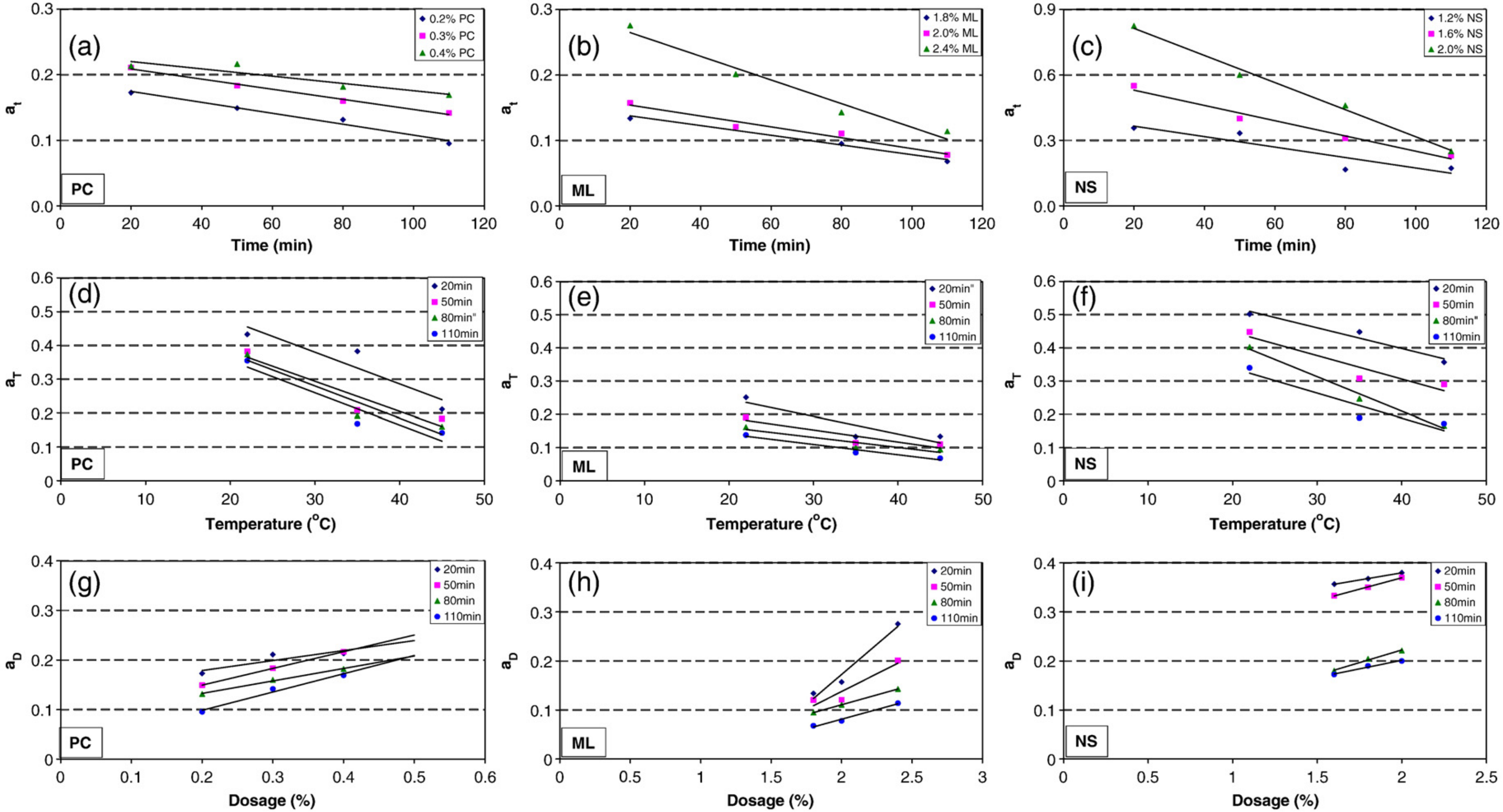


**Fig. 4.** Experimental coefficient (a) of oscillatory shear strain/stress equations as a function of test parameters (mixing time, temperature, and superplasticizer dosage).

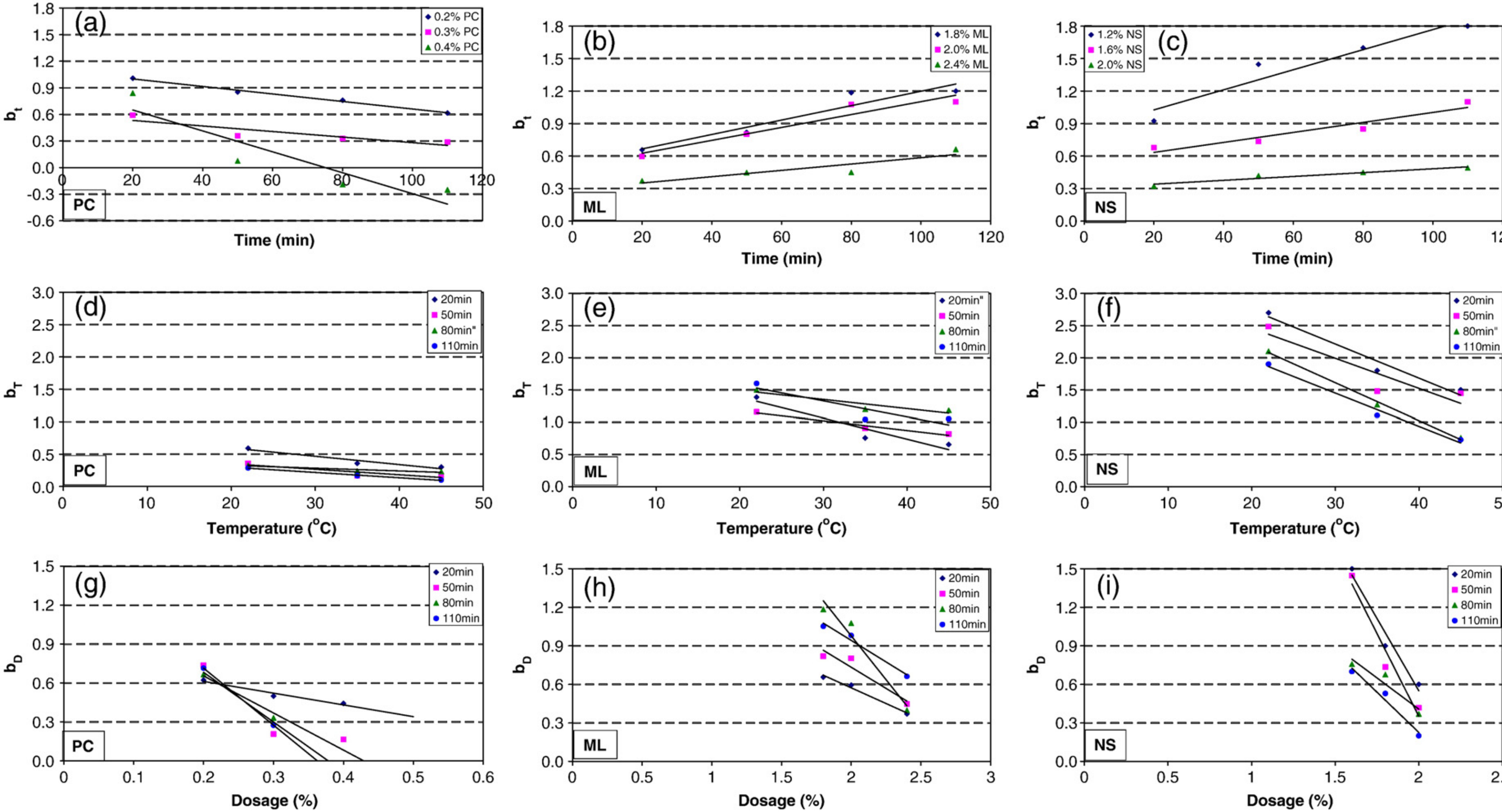


**Fig. 5.** Experimental coefficient (b) of strain/stress equations as a function of test parameters (mixing time, temperature, and superplasticizer dosage).

**Table 1**
Range of model coefficients in Eqs. (14) and (15).

| Coefficient | PC | | ML | | NS | |
|---|---|---|---|---|---|---|
| | *a* | *b* | *a* | *b* | *a* | *b* |
| $c_t$ | 0.0001–0.001 | −0.001–0.05 | 0.0001–0.005 | −0.03–0.05 | 0.0007–0.009 | 0.0007–0.03 |
| $e_t$ | 0.2–1 | 0.21 | 1–5 | −10–2 | 0.1–0.9 | 0.001–2 |
| $c_T$ | −0.001–0.01 | 0.0001–0.05 | −0.001–1 | −0.0001–0.1 | 0.0007–0.04 | 0.0001–10 |
| $e_T$ | 0.2–1 | 0.1–2 | 0.1–1 | −8–3 | 0.1–1.5 | 0.001–10 |
| $c_D$ | 20–200 | 50–400 | 100–1000 | 50–1000 | 10–100 | 2–300 |
| $e_D$ | 0.01–0.5 | 0.5–2 | 0.2–2 | 1–20 | 0.1–1 | 0.1–10 |

points. The results indicate that the GA model reasonably captured the relationship between the test parameters and oscillatory shear strain of cement pastes.

### 6.2. Performance of GA-based oscillatory shear strain equations for predicting yield stress of cement paste

As explained earlier, yield stress values were calculated using the developed equations by determining the stress corresponding to zero strain (the intersection of the strain/stress curve in the viscous range with the oscillatory shear stress axis). The predicted yield stress values versus the corresponding experimentally measured values are presented in Fig. 7a, b, and c for cement pastes incorporating PC, ML, and NS, respectively. It is shown that the predicted and measured yield stress data points are located close to the equity lines.

The *AAE* along with the average, standard deviation, and covariance of the ratio between the predicted and experimental yield stress results for the testing data are shown in Table 4. It can be observed that the *AAE* values were less than 20%, indicating that the equations predicted the yield stress with reasonable accuracy. It should be noted that cement paste rheology is time, temperature, and shear history dependent, and is hence affected by high variability. For instance, typical *AAE* for predicting yield stress in the literature include 28% for mortar [20] and 46% for concrete [21]. So, the predictions of the present GA model with an error lower than 20% are indeed reasonably accurate.

### 6.3. Sensitivity analysis

The GA-based oscillatory shear strain/stress equations displayed satisfactory performance in predicting the yield stress of cement pastes incorporating the superplasticizers investigated within the practical training range of test parameters. The ability of these equations to capture the effects of each input parameter on the yield stress of cement pastes is explored below. To examine the sensitivity of the developed equations to variation of the mixing time, one mixture for each superplasticizer was randomly selected from the experimental data, and the yield stress was calculated with changing only the mixing time and maintaining the rest of the experimental parameters constant. To examine the sensitivity of the developed equations to the variation of temperature, the mixing time was kept constant and the ambient temperature was varied. Concerning the effect of the superplasticizer dosage, both the mixing time and ambient temperature were maintained constant and the yield stress values were calculated while only varying the superplasticizer dosage. It should be noted that some values of the parameters were not tested experimentally but were considered in the model calculations.

The cement paste mixtures investigated were made with 0.3% PC, 1.8% ML, and 1.6% NS. The responses of Eqs. (16)–(18) in predicting the yield stress with variation of the experimental parameters is illustrated in Fig. 8 along with the corresponding experimental results. It can be observed that the predicted yield stress increased with the mixing time, which is in agreement with the trend of the experimental data (Fig. 8a). Furthermore, the predicted yield stress values were comparable to the experimentally measured values. This indicates that the GA-based oscillatory shear strain Eqs. (16)–(18) have successfully captured the sensitivity of the yield stress to variation of the mixing time.

To investigate the sensitivity of the predicted yield stress to variations of the ambient temperature, the mixing time was fixed at 50 min and yield stress values were calculated while only varying the temperature. As shown in Fig. 8b, the predicted yield stress values increased with the increase of temperature, which is in conformity with the experimental trend. The results of the experimental yield stress values were also located close to the corresponding ones predicted by the GA-based equations, indicating that the yield stress predicted by the GA model was sensitive to the change in the ambient temperature (Fig. 8b).

The influence of the superplasticizer dosage on the predicted yield stress is illustrated in Fig. 8c. The yield stress was predicted while setting the mixing time to 50 min and the temperature to 45 °C and varying the superplasticizer dosage. It can be observed that the developed equations have also captured the effect of the superplasticizer dosage on yield stress; the yield stress decreased as the dosage increased and the experimental data points are located close to the corresponding predicted values. As shown in Fig. 8c, beyond a certain superplasticizer dosage, the yield stress versus superplasticizer dosage curve became nearly a plateau where the decrease in the predicted yield stress with a higher dosage became marginal. The shape of the predicted yield stress-superplasticizer dosage relationship is in good conformity with the experimental relationship between yield stress and superplasticizers dosage reported by others [22]. The intersection of the two tangent lines to the ends of each curve was found to give an approximate estimate of the experimentally measured superplasticizer saturation dosage for cement pastes made with ML and NS. For pastes incorporating PC, the predicted saturation dosage can be found at the intersection point between the tangent line of the top end of the curve with the *x*-axis (Fig. 8c). The saturation dosages were predicted at 0.37% for PC, 2.45% for ML, and 1.6% for NS, which are reasonably close to those experimentally determined values, which are 0.4%, 2.8%, and 1.8% for PC, ML, and NS, respectively [13].

**Table 2**
Parameters used in genetic algorithm setting.

| | |
|---|---|
| Number of individuals | 70 |
| Variable format | Real values |
| Maximum generations | 10000 |
| Selection method | Roulette wheel selection |
| Selection pressure | 1.7 |
| Recombination name | Extended line |
| Recombination rate | 0.74 |
| Mutation rate | 0.01 |

## 7. Conclusions

The oscillatory shear behavior of cement pastes incorporating various superplasticizers and subjected to different mixing times and ambient temperatures was characterized using a controlled shear stress–strain rheometer. The behavior of cement paste depended on the applied shear stress; at low shear stress levels, cement paste behaved as an elastic solid, while at higher stress range, it behaved as a linear

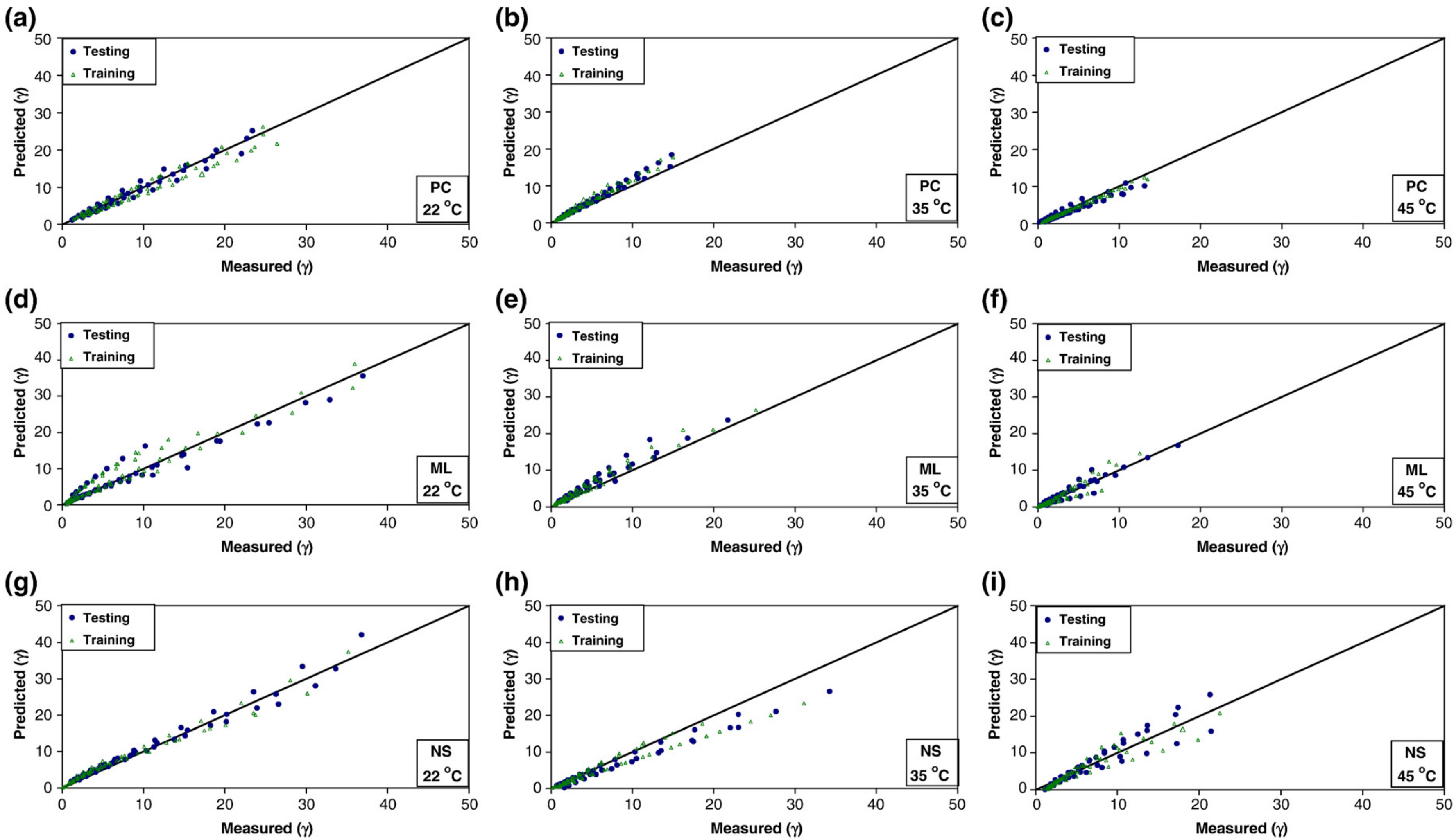


**Fig. 6.** Measured versus predicted oscillatory strain for cement pastes incorporating different superplasticizers at different temperatures and times.

**Table 3**
Performance of GA-based oscillatory shear strain equations of cement paste mixtures.

| Temperature (°C) | Superplasticizer | *AAE* (%) | $\gamma_{(measured)}/\gamma_{(calculated)}$ (all test points) | | |
|---|---|---|---|---|---|
| | | | Average | *SD* | *COV* |
| 22 | PC | 12 | 1.01 | 0.13 | 13 |
| | ML | 19 | 0.98 | 0.23 | 23 |
| | NS | 17 | 0.90 | 0.13 | 15 |
| 35 | PC | 15 | 0.90 | 0.07 | 8 |
| | ML | 25 | 0.80 | 0.13 | 16 |
| | NS | 19 | 1.10 | 0.16 | 15 |
| 45 | PC | 16 | 1.14 | 0.19 | 16 |
| | ML | 23 | 1.08 | 0.37 | 35 |
| | NS | 22 | 1.07 | 0.29 | 27 |

viscous liquid. Cement paste shifted from a solid-like to a liquid-like behavior in a narrow range of stress of usually less than 1 Pa. The yield stress of cement paste was estimated as the intersection of the linear curve with the shear stress axis in the viscous range. The oscillatory yield stress values were about 5 Pa less than the corresponding values obtained using conventional flow curves. The yield stress estimated using the oscillatory shear technique varied with the mixing time, ambient temperature, and superplasticizer dosage. The present study attempted to find a methodology to predict the yield stress of superplasticized cement paste subjected to various ambient temperatures and mixing times using both the oscillatory shear test and the genetic algorithms technique. As such, genetic algorithm-based equations for predicting the oscillatory shear strain/stress curves for the experimentally investigated cement paste mixtures (in the viscous region) have been developed considering the various test parameters. The oscillatory shear strain calculated using the proposed equations agreed well with the corresponding experimental results and allowed to predict the oscillatory yield stress. Hence, the main objective of this paper has been successfully achieved. The following conclusions can be drawn from this work:

**Table 4**
Performance of GA-based oscillatory shear strain equations in predicting yield stress of cement paste mixtures.

| Superplasticizer | *AAE* (%) | $\tau_{o(measured)}/\tau_{o(calculated)}$ (all test points) | | |
|---|---|---|---|---|
| | | Average | *SD* | *COV* (%) |
| PC | 16 | 1.09 | 0.22 | 21 |
| ML | 20 | 0.92 | 0.19 | 0.2 |
| NS | 19 | 1.05 | 0.21 | 20 |

- The GA-based oscillatory shear strain equations allowed predicting the yield stress of cement paste incorporating PC, ML, or NS with reasonable accuracy.
- The GA-based oscillatory shear strain equations developed in this study can depict the effect of the elapsed mixing time on the yield stress of cement pastes incorporating various superplasticizers and exposed to various temperatures.
- The GA-based oscillatory shear strain equations were sensitive to the increase in temperature and captured the increase of yield stress with increasing temperature.

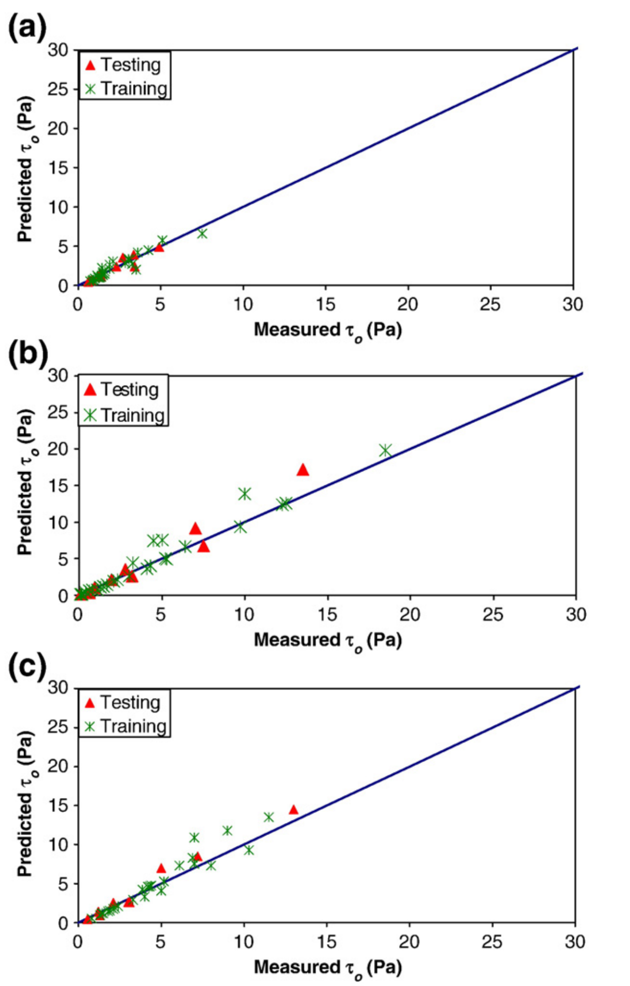


**Fig. 7.** Measured versus predicted yield stress for cement paste mixtures at different temperatures and mixing times and incorporating: (a) PC, (b) ML, and (c) NS.

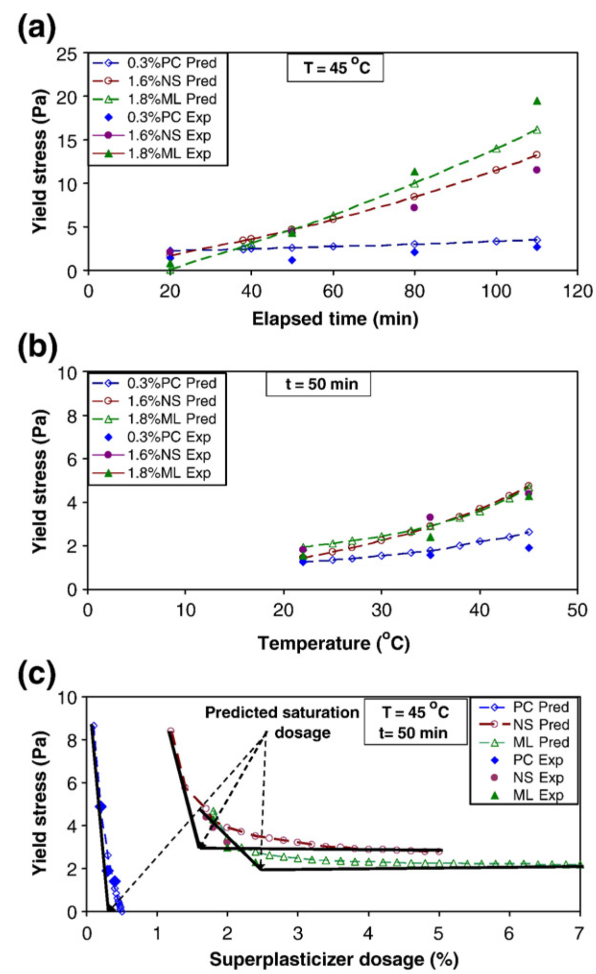


**Fig. 8.** Variation in yield stress of superplasticized concretes versus: (a) elapsed time at 45 °C, (b) ambient temperature at 50 min, and (c) superplasticizer dosage at 45 °C and 50 min.

- The GA-based oscillatory shear strain equations were also sensitive to the increase in the superplasticizer dosage; yield stress decreased in a curvilinear mode when the superplasticizer dosage increased.
- The prediction of the superplasticizer saturation dosage using the approach suggested in this study gave satisfactory results when compared to corresponding experimentally measured values.
- It should be emphasized that the GA-based oscillatory shear strain equations thus developed are valid for the type of materials tested and within the range of parameters investigated in this study. Therefore, further experimental research is needed to validate these equations using different materials and ranges of test parameters.

*List of Symbols*

| | |
|---|---|
| $\sigma$ | Oscillatory shear stress (Pa) |
| $\sigma_o$ | Amplitude of shear stress (Pa) |
| $\omega$ | Angular velocity (1/s) |
| $t^*$ | Time (s) |
| $\gamma$ | Oscillatory shear strain |
| $\gamma_o$ | Amplitude of shear strain |
| $\delta$ | Phase shift |
| $\tau$ | Shear stress (Pa) |
| $\tau_o$ | Bingham yield stress (Pa) |
| $\mu_p$ | Plastic viscosity (Pa s) |
| $\dot{\gamma}$ | Shear rate ($s^{-1}$) |
| $a$, $b$ | Experimental coefficients, |
| $t$ | Mixing time (min) |
| $T$ | Ambient temperature (°C) |
| $D$ | Superplasticizer dosage (% by cement mass) |
| $c_{at}$, $e_{at}$, $c_{aT}$, $e_{aT}$, $c_{aD}$, $e_{aD}$, $c_{bt}$, $e_{bt}$, $c_{bT}$, $e_{bT}$, $c_{bD,}$ $e_{bD}$ | Model constants |

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