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Cement and Concrete Research 35 (2005) 1539–1545

Cement and Concrete Research

Autogenous relative humidity change and autogenous shrinkage of high-performance cement pastes

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Received 18 December 2003; accepted 14 June 2004

Abstract

In this paper, the effects of water to cementitious material ratio (w/cm), silica fume (SF) and ground blast-furnace slag (GBFS) on autogenous relative humidity (RH) change and autogenous shrinkage (AS) of high-performance cement pastes were studied. The mechanism of self-desiccation caused by mineral admixture and reduction of w/cm were studied by the parameters of mineral admixture self-desiccation-effect coefficient k and efficient w/cm r_e proposed. Furthermore, the relationship between autogenous RH and AS of high-performance paste was established. The results indicate that w/cm is a chief factor that affects autogenous RH change and AS of cement pastes. The lower the w/cm of paste is, the more reduction the autogenous RH and the increment of AS are. SF increases autogenous RH reduction and AS increment of cement paste at early ages, and GBFS increases autogenous RH reduction and AS increment at later ages. The effect of mineral admixtures on autogenous RH change of paste resulting from self-desiccation can be reflected effectively by the nonlinear equation with the parameters of k and r_e. There exists a good linear correlation between autogenous RH change and AS of cement pastes.

Keywords: Autogenous shrinkage; Cement paste; Mineral admixture; Relative humidity; Self-desiccation

1. Introduction

Self-desiccation is one common phenomenon of high-performance cementitious materials characterized by low water to cementitious material ratio (w/cm), incorporated with reactive mineral admixtures and superplasticizers [1–4]. Self-desiccation of paste in concrete normally leads to a decline in its autogenous relative humidity (RH) and therefore results in autogenous shrinkage (AS) of concrete, which increases the risk of cracking of concrete structures, especially at early ages [5,6]. Studies on change laws of autogenous RH and AS of concrete with age caused by self-desiccation and the relationship between autogenous RH and AS play an important role in controlling its shrinkage as well as cracking of structure resulting from AS.

It is the cement paste in high-performance concrete that is prone to result in AS of concrete in which many literatures in this field have reported [4–12]. Experimental study on autogenous RH change and AS of cement paste/concrete with or without mineral admixtures was conducted [9,12], and thermodynamic limitation and mechanism of self-desiccation was discussed [10,11]. However, due to differences and limitation of experimental methods, a systematic research has not been performed on change laws of autogenous RH and AS of cement paste or concrete, and on their relationship yet. Therefore, the main research effort on autogenous RH and AS of high-performance cement paste in concrete was carried out in this experimental program, and effects of w/cm, mineral admixtures, such as silica fume (SF), ground blast-furnace slag (GBFS), etc., on autogenous RH change and AS of cement pastes were studied. Based on this, their relationship was discussed and established. The parameters of coefficient of mineral self-desiccation effect, k, and efficient ratio of water to cementitious materials, r_e, were proposed, and experimental

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doi:10.1016/j.cemconres.2004.06.028

results were fitted nonlinearly and analyzed using these proposed parameters, k and r_e. In addition, the mechanism in which mineral admixture and w/cm affect self-desiccation effect of high-performance paste system under different conditions was discussed in detail.

2. Theoretical analysis of self-desiccation effect in high-performance paste

The decline of autogenous RH of high-performance cement paste is not only a significant consequence of self-desiccation effect, but also a direct cause of its AS. Self-desiccation effect means that, under the condition of constant temperature and isolated humidity, unhydrated cementitious particles in paste continues to hydrate to cause reduction of water in capillary pores, and as a result, autogenous RH of cement paste decreases and AS results [4,13–16].

Obviously, there are many different factors, including w/cm, activity and content of mineral admixtures and age, which influence autogenous RH of cement paste directly. Research over the last few years [4,6,12] indicates that different activities of mineral admixtures, such as SF, GBFS and fly ash, in high-performance cementitious materials affects its autogenous RH change differently. Under the same w/cm and constant content of cementitious material condition, autogenous RH decline of concrete or paste with mineral admixtures is different from that of concrete or paste without mineral admixtures. Thus, for cement paste with mineral admixtures, the parameter of w/cm could not reflect real effects of mineral admixtures on autogenous RH change. On the basis of activity of mineral admixtures, the parameters of mineral admixture self-desiccation-effect coefficient k and efficient w/cm r_e have been proposed in this paper to attempt to better reflect the effect of mineral admixtures on self-desiccation of high-performance cement paste at different ages.

In this section, the mineral admixture self-desiccation-effect coefficient k and the efficient w/cm on r_e are defined and discussed. k means the ratio of the effect of mineral admixtures on autogenous RH change of cement paste to the effect of cement on autogenous RH change under same condition. k depends on type (mainly its activity) and content of mineral admixtures, curing age and w/cm of paste, etc. For a certain mineral admixture, k is a function of age t and varies with hydration characteristics of mineral admixtures. Based on hydration characteristics of mineral admixture and previous research [4,6], k can be expressed as follows:

$$k_i(t) = a + bt \tag{1}$$

Here, k_i is the self-desiccation-effect coefficient of mineral admixture i and is a nondimensional variable, a and b are both constant and depend on types of mineral admixture and its hydration characteristics, and t is age (day).

As SF and GBFS used in this experimental program are concerned, k_1 for SF and k_2 for GBFS are considered to be the function of age t and can be described as Eqs. (2) and (3), respectively:

$$k_1 = 2.4 - 0.0016t \tag{2}$$

$$k_2 = 0.9 + 0.0018t \tag{3}$$

Herein, t is age of cement paste (day), 0 day $\leq t \leq$ 300 days.

Furthermore, according to the definition of mineral admixture self-desiccation-effect coefficient k, the conception of efficient w/cm on r_e is proposed and the parameter r_e is described as follows:

$$r_e = \frac{W}{C + k_1\mathrm{SF} + k_2\mathrm{GBFS} + k_3\mathrm{MB}} \tag{4}$$

Here, C, W, SF, GBFS and MB are unit mass of C, W, SF, GBFS and other mineral admixtures (MB) in cement paste, respectively; k_1, k_2 and k_3 are the self-desiccation-effect coefficients of SF, GBFS and other mineral admixtures, respectively.

When unit mass of cementitious material in paste keeps constant, k can effectively reflect the effect of different mineral admixtures on self-desiccation of cement paste, compared to that of a control cement paste. The coefficient r_e can reflect the real magnitude of self-desiccation effect of cement paste with mineral admixtures at any age. According to the definition of r_e, for control cement paste without mineral admixtures, its efficient ratio of water to cementitious materials r_e is equivalent to w/cm, namely, water to cement ratio.

3. Experiment

3.1. Raw materials and mix proportion

In this experiment, 52.5 ordinary Portland cement according to Chinese standards with compressive strength of 63.7 MPa at 28 days was used. The mineral admixtures of SF and GBFS are also used. Table 1 gives chemical components and physical properties of cement, SF and GBFS. To make paste with expected fluidity, a naphthalene–formaldehyde condensate-type high-range water-reducing admixture (HRWA) was also employed. Mix proportions of high-performance cement pastes with different w/cm and with mineral admixtures of SF and/or GBFS and the compressive strength of specimens are given in Table 2.

Table 1
Chemical components of cement and mineral admixtures

Raw materials	SiO_2	Al_2O_3	CaO	MgO	Fe_2O_3	SO_3	TiO_2	K_2O	Na_2O	Specific area (m^2/kg)	Density (g/cm^3)
Cement	21.4	5.6	63.3	1.9	2.7	3.4	0.4	0.7	0.2	351	3.10
SF	96.1	0.5	0.3	0.6	0.5	0.5	0.0	0.8	0.0	19600	1.91
GBFS	37.4	16. 7	28.6	6.2	4.0	0.6	0.8	0.9	0.2	642	2.78

3.2. Experimental method

3.2.1. Testing method of autogenous RH

Autogenous RH of all cement pastes at different ages was measured using a YIDA NSWB150 probe and digital indicator made in China. The probe is composed of a humidity-sensitive capacitive sensor, a temperature-sensitive capacitive sensor and porous sleeve. RH and temperature can be measured at the same time. The dimensions of specimens for measuring autogenous RH are 100×100×100 mm. While specimens were cast, holes for probe of transducer in the middle of specimen top surface were formed. After removal of the molds at the age of 1 day, all six surfaces of specimens were sealed with the epoxy resin sealant with the thickness of 2 mm to avoid moisture loss, which guarantees that autogenous RH change in specimens merely results from self-desiccation. Then, specimens were moved to cure in a container with curing temperature of 20 °C and RH of 60±5%. The detail of testing set is shown in Fig. 1. Autogenous RH of specimens at prescribed ages was measured. The probe of sensors were inserted and sealed in specimens with a rubber ring at certain ages. When RH equilibrium of probe was reached, the initial RH and temperature displayed on the indicator was recorded.

3.2.2. Calibration and accuracy of measured RH using probe

RH using the probes is affected by several factors, such as drift, temperature, response time, etc. The drift of the probes are very low and they are about 1% RH per year. The probes are salt calibrated by the manufacturer using the dew point meters each half of year. The temperature of the holes in cement paste also affects RH value greatly. Generally, 1 °C between the sensor and cement paste can yield an error of approximately 6% RH [17]. In fact, a little difference between the temperature of the air inside the measurement holes and the temperature of 20 °C in ambient conditions is inevitable. To calibrate the error from temperature, measured RH is amended according to the equation:

$$\Phi = \Phi_{\mathrm{m}} + 6(20 - T) \tag{5}$$

Here, Φ is the amended RH, Φ_{m} is measured RH and T is the temperature in holes measured as RH. Slow response of capacitive probes occurs at high RH level in holes, especially in high-performance cement paste. The appropriate response time is required for RH equilibrium in holes. Because several calibration methods were used, no systematic error seemed to exist.

3.2.3. Testing method of AS

AS of cement paste was measured using scale micrometer method. The dimension of specimens for AS is 40×40×160 mm. Immediately after casting, a layer of soft impermeable polypropylene film with the thickness of 0.5 mm was covered on the surface of specimen and placed in fog room with the temperature of 20 °C to cure. After demolding at the age of 1 day, specimens were first sealed with several layers of soft impermeable polypropylene film with the thickness of 0.2 mm and then covered with a layer of paraffin on six surfaces. These measures may guarantee that specimens shrink unlimitedly and avoid moisture loss. To test impermeability of sealed specimens, a series of specimens was weighed at 1 and 28 days, the average weight loss of the specimens at 28 days was about 0.05% of the weight at 1 day. It is proved that the sealed coat prevents moisture diffusion in specimens effectively. After initial length measurement, specimens were cured in the curing room maintained at 20 °C and

Table 2
Mix proportions of cement pastes with different w/cm and mineral admixtures of SF and GBFS

Sample	w/cm	HRWA (%)	Mix proportion (%)			Compressive strength (MPa)
			Cement	SF	GBFS	28 days
H1	0.2	1.0	100	0	0	119.0
H2	0.3	0.5	100	0	0	81.0
H3	0.4	0	100	0	0	69.3
H4	0.5	0	100	0	0	48.3
H5	0.3	0.5	90	10	0	88.0
H6	0.3	0.5	70	10	20	89.7

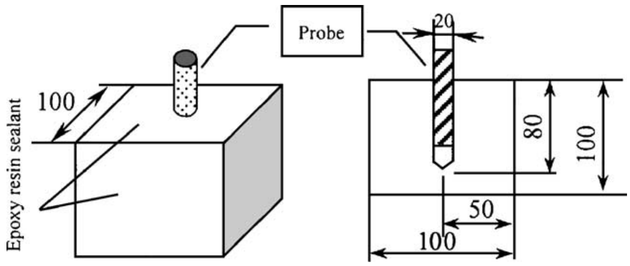

Fig. 1. Dimensions and probe position of specimens for self-desiccation (unit: mm).

60±5% RH. At prescribed ages, AS of specimens was measured.

4. Results and discussion

4.1. Autogenous RH change with age under different conditions

Autogenous RH changes of pastes with different w/cm (equivalent to w/c, herein) with age are shown in Fig. 2. It can be seen clearly that w/cm plays an important role in autogenous RH change of cement pastes. Autogenous RH of cement paste with w/cm of 0.5 at 300 days decreased little to 94% only. However, autogenous RH of cement paste with w/cm of 0.2 at 300 days decreased dramatically down to 79%. Autogenous RH reduction rate of cement pastes increases with the decrease of w/cm, especially at early ages. Mainly, with the decrease of w/cm, free water for cement hydration in paste reduces, and consequently, their autogenous RH reduction rate caused by self-desiccation increases.

Autogenous RH change curves of cement paste incorporated with 10% SF and/or 20% GBFS mineral admixtures at the same w/cm of 0.3 are given in Fig. 3. As seen from it, autogenous RH reduction rate and decrement of cement paste with 10% SF is higher than that of control paste. Autogenous RH reduction rate and decrement of cement paste with 10% SF and 20% GBFS is also higher than that of control paste and even higher than that of cement paste with only 10% SF after 14 days. Pozzolanic effect and filling effect of mineral admixtures

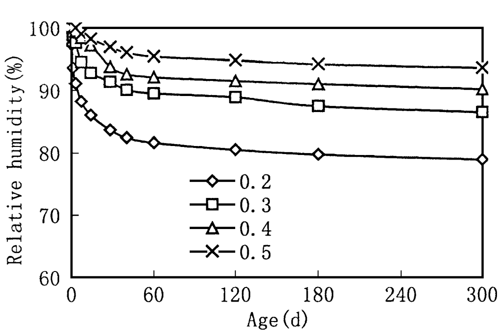

Fig. 2. Effect of w/cm on autogenous RH change of cement pastes.

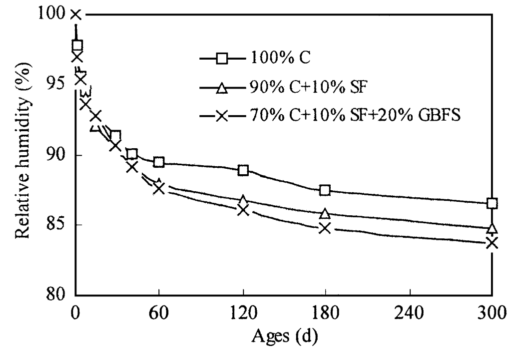

Fig. 3. Effect of mineral admixtures on autogenous RH change of cement pastes with the same w/cm of 0.3.

are mainly responsible for it. SF accelerates cement hydration for its active super-fine components taking part in hydration reaction rapidly at early ages and GBFS accelerates second hydration reaction at later ages. Thus, addition of mineral admixtures into paste alters hydration rate and microstructure characteristics, such as pores size distribution and amount of cement paste, which consequently results in high self-desiccation effect.

4.2. Autogenous RH change laws and interpretation

Based on the above-proposed parameters and experimental results, it is obvious that autogenous RH denoted as h_s of cement pastes is the function of efficient w/cm r_e and age t. Based on previous research [4,6], h_s can be expressed as Eq. (6) as follows:

$$h_s(r_e, t) = 100 + dr_e + f\ln(1+t) \tag{6}$$

where h_s is RH in paste, d and f are the coefficients of r_e and t, respectively. Both d and f depend on many factors, such as w/cm, type and content of mineral admixtures, mix proportion, etc.

According to the above-proposed equations, the effect rules of mineral admixture on RH of paste with different w/cm are further characterized and interpreted in detail in the following section.

4.2.1. Autogenous RH fitting of cement paste with different w/cm and without mineral admixtures

The experiment results of RH of pastes with different w/cm in Fig. 2 were fitted according to Eq. (6) whose principle is the Levenberg–Markuardt iteration method using least squares algorithm [18]. The value of coefficient obtained by nonlinear fitting and the fitting equations are listed in Table 3. It can be seen from the fitting results that the correlation coefficient R^2 decreases with the increase of w/cm and all the correlation coefficient values are above 0.98. It can be concluded that in high confidence level, Eq. (6) is adaptable to attribute autogenous RH change of cement paste with different w/cm resulting from self-desiccation.

It also can be seen from the values of d and f that the value of d increases with the decrease of w/cm from

Table 3
Autogenous RH fitting equations of concrete with w/cm and without mineral admixtures

No.	w/cm	r_e	Nonlinear fitting equations	R^2
H1	0.2	0.2	h_s=100−22.070r_e−3.215ln(1+t)	.969
H2	0.3	0.3	h_s=100+0.218r_e−2.462ln(1+t)	.984
H3	0.4	0.4	h_s=100+2.296r_e−1.978ln(1+t)	.962
H4	0.5	0.5	h_s=100+2.426r_e−1.249ln(1+t)	.954
H5	0.3	3/(11.4−0.0016t)	h_s=100−2.233r_e−2.607ln(1+t)	.994
H6	0.3	3/(11.2+0.002t)	h_s=100−1.513r_e−2.798ln(1+t)	.993

negative to positive. Based on the principle of its mathematical model, the physical meaning of coefficient d reflects the effect of r_e on autogenous RH of cement paste at early age. It indicates that the lower the w/cm, the higher effect of r_e on autogenous RH of cement paste is. The absolute value of coefficient f, which reflects the effect of age on autogenous RH of cement paste, increases with the decrease of w/cm. Hence, it shows that the lower the w/cm is, the higher the effect of age on autogenous RH change of paste is.

4.2.2. Autogenous RH fitting of cement pastes incorporated with mineral admixtures

For cement pastes incorporated with mineral admixtures, r_e can be calculated by Eqs. (2)–(4) based on the mix proportion of cement paste (Table 1). The value of r_e of paste with mineral admixtures is not constant but decreases with an increase of age t (in Table 3) and is smaller than w/cm of the corresponding cement paste. It indicates that the decline of r_e can characterize the effect of mineral admixtures on paste hydration and autogenous RH change at different ages. The experimental results of cement pastes incorporated with 10% SF and/or 20% GBFS in Fig. 3 were fitted by nonlinear Eq. (6). The nonlinear fitting equations and the values of coefficients are shown in Table 3. It can be seen that their correlation coefficient is over 0.99 and its goodness of fit is perfect in high confidence level. The value of coefficient d of paste with 10% SF is higher than that of control paste and paste with 10% SF and 20% GBFS, which indicates that SF affects significantly autogenous RH at early ages. The value of coefficient f of paste with 10% SF and 20% GBFS is higher than that of control paste and paste with 10% SF, which indicates that GBFS increases autogenous RH change of cement paste at later ages.

The results of nonlinear fitting further show that r_e can reflect effectively the effect of mineral admixtures on autogenous RH change of cement paste resulting from self-desiccation at different ages. r_e of mineral admixtures can be calculated based on their hydration characteristics, and Eq. (6) can reflect change laws of autogenous RH of cement paste with mineral admixtures.

4.3. AS of cement pastes

AS curves of cement paste with different w/cm with age resulting from self-desiccation are given in Fig. 4. It can be seen clearly that AS of cement pastes increases quickly at early ages and slowly at later ages. AS of cement pastes increases with a decrease of w/cm, especially at early ages. Fig. 5 shows AS curves of cement paste incorporated with 10% SF and 20% GBFS mineral admixtures at the same w/cm of 0.3. AS of cement paste with 10% SF is far higher than that of control paste. AS of cement paste with 10% SF and 20% GBFS is also higher than that of control paste and cement paste with 10% SF after 14 days.

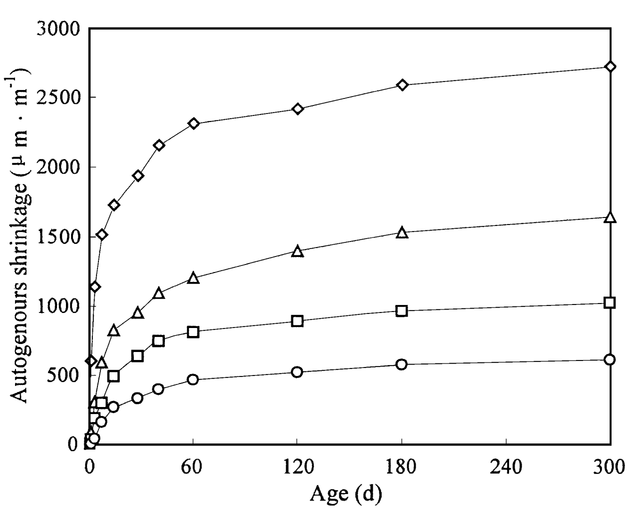

Fig. 4. AS curves of cement pastes with different w/cm: ◇ 0.2, △ 0.3, □ 0.4, ○ 0.5.

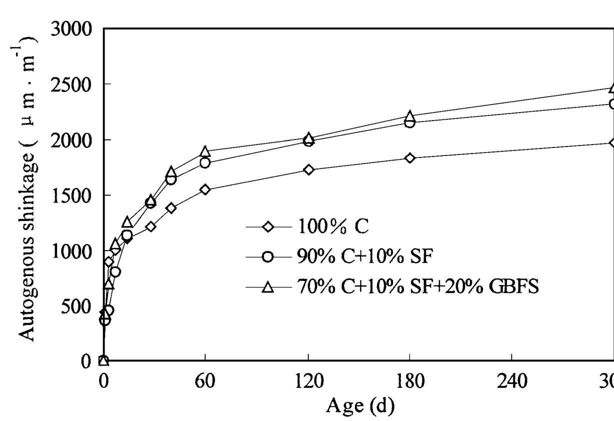

Fig. 5. AS curves of cement pastes incorporated with SF and GBFS in the same w/cm of 0.3.

4.4. Relationship between of autogenous RH and AS of cement pastes

In terms of mechanism of self-desiccation, it is believed that self-desiccation results in autogenous RH reduction of cement paste, which causes AS of cement paste directly. The experimental data of autogenous RH and AS of cement pastes in Figs. 2–5 at different ages were analyzed and regressed. The results shown in Fig. 6 and 7 indicate that AS and autogenous RH has great significant linear relevance for cement paste with different w/cm and with mineral admixtures. And the linear relationship between AS and autogenous RH could be described as Eq. (7):

$$\varepsilon_s(h_s) = mh_s + n \tag{7}$$

Here, ε_s is AS of cement paste; h_s is autogenous RH of cement paste resulting from self-desiccation; m and n are constant, which depend on w/cm, types and content of mineral admixtures, etc.

From the knowledge of mathematic model, m is the slope of Eq. (7), which reflects change degree of AS with a decrease of autogenous RH. n is the intercept, which means the final theoretical AS of cement paste when autogenous RH of cement paste decreases down to zero. In fact, autogenous RH of cement paste is impossible to decrease to zero. It could be seen from Fig. 6, for cement pastes with different w/cm, the lower the w/cm is, the higher the absolute value of m is. It also indicates AS increases manifestly with a decrease of autogenous RH, which is in accordance with the experimental results. The parameter n decreases gradually with an increase of w/cm. It indicates that the lower the w/cm is, the higher the final theoretical AS is.

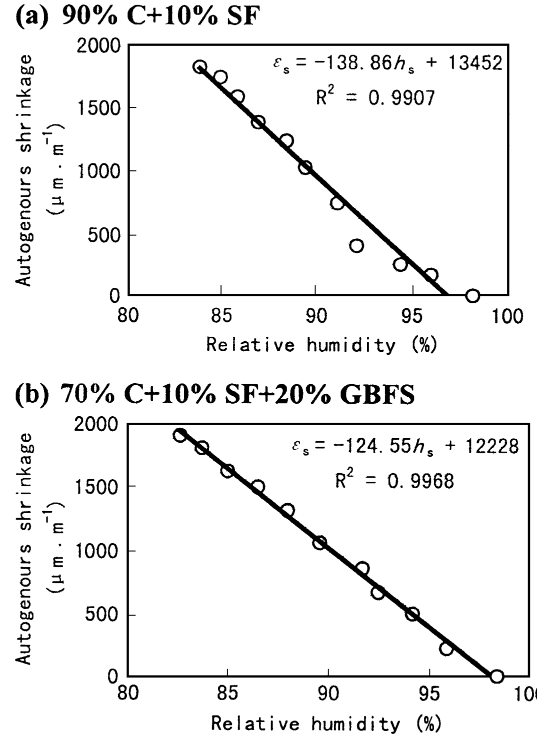

Fig. 7. Linear regress results of autogenous RH and AS of cement pastes incorporated with 10% SF and/or 20% GBFS.

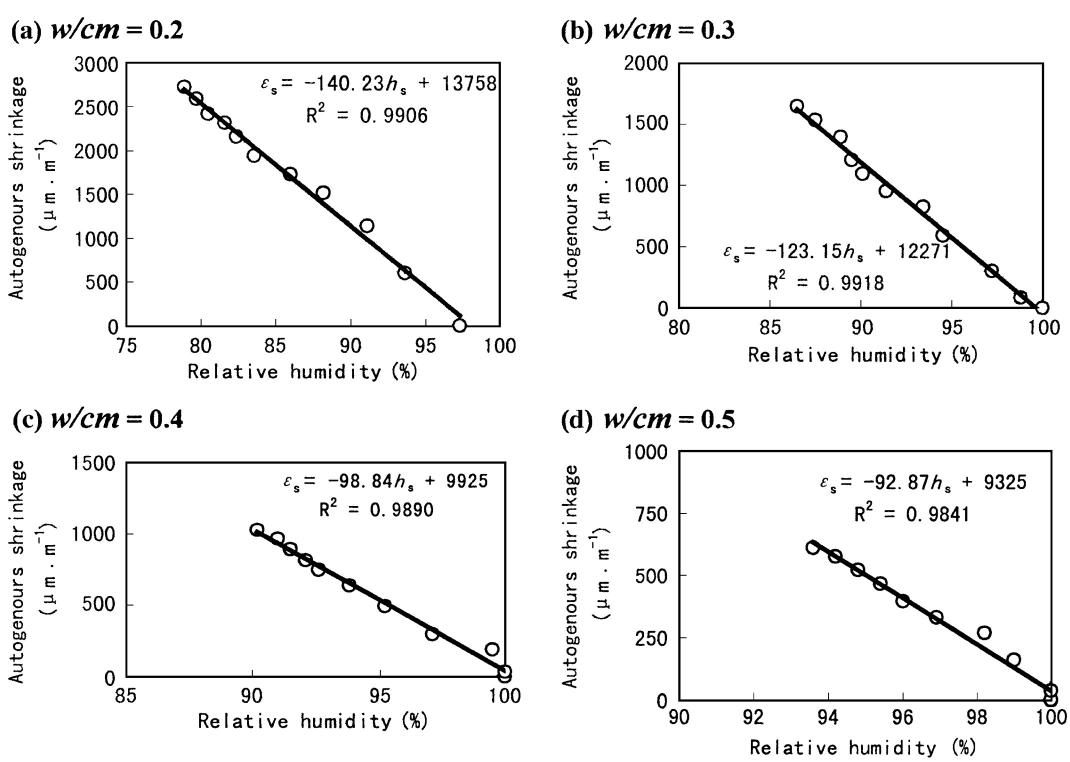

Fig. 6. Linear regress results of autogenous RH and AS of cement pastes with different w/cm.

As seen from Fig. 7, the relationship of autogenous RH and AS of cement paste with mineral admixtures of 10% SF and/or 20% GBFS still has great significant linear relevance. The absolute value of m of cement paste with 10% SF and with 10% SF and 20% GBFS is higher than that of control cement paste. It shows that the addition of SF and GBFS into paste increases autogenous RH reduction of cement paste and consequently increases AS. However, the absolute value of m of cement pastes with 10% SF and 20% GBFS is lower than that of paste with 10% SF, which indicates AS rise rate of paste with 10% SF and 20% GBFS is lower than that of paste with 10% SF. The parameter n of cement paste with 10% SF and with 10% SF and 20% GBFS is higher than that of control cement paste.

It indicates that the addition of SF and GBFS increases the final theoretical AS of cement paste.

AS and autogenous RH have remarkable linear relevance for cement pastes with different w/cm and mineral admixtures at different ages. Based on this relevance, AS of cement pastes could be predicted and controlled through testing and controlling autogenous RH in cement pastes.

Consequently, crack risk of high-performance concrete at early ages will be reduced to the minimal degree.

5. Conclusions

(1) Autogenous RH reduction rate and decrement of cement pastes increase with a decrease of w/cm, especially at early ages. SF increases autogenous RH reduction and AS increment of cement paste at early ages, and GBFS increases autogenous RH reduction and AS increment at later ages.

(2) Both mineral admixture self-desiccation-effect coefficient k and efficient ratio of water to cementitious materials r_e can reflect effectively the effects of mineral admixtures on autogenous RH change of cement paste at different ages. The value of r_e of cement paste with mineral admixtures is not constant but decreases with an increase of age and is smaller than the value of w/cm of the corresponding cement paste. The change laws of autogenous RH of cement pastes with or without mineral admixtures and with different w/cm can be characterized in a nonlinear equation.

(3) AS and autogenous RH have great significant linear relevance for cement pastes with different w/cm and mineral admixtures at different ages, and the relationship could be described with the equation of $\varepsilon_s(h_s)=mh_s+n$, ε_s is AS of cement paste; h_s is autogenous RH of cement paste resulting from self-desiccation, and m and n are constant.

Acknowledgement

The authors gratefully acknowledge the financial support provided by National Basic Research Project of China (2001CB610704) and the Engineering Development Fund of Tongji University.

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