

Technical Paper

## INFLUENCES OF SUPERPLASTICIZERS ON BASIC AND DRYING CREEP OF CONCRETE

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### Abstract

The influences of naphthalene-based plasticizers and polycarboxylate acid/salt superplasticisers on creep of concrete, including basic creep and drying creep, were investigated. Internal relative humidity and pore structure of concrete and the surface tension of pore solution were tested. The results show that polycarboxylate acid/salt superplasticizers refine capillary pores in concrete and reduce surface tension of pore solution. In addition, restrain internal moisture transmission and redistribution. As a result, creep of concrete is reduced. Compared with naphthalene-based plasticizer, polycarboxylate acid/salt superplasticizer causes a greater reduction of drying creep, but a smaller reduction of basic creep. This is because the moistures redistribution is quite feeble and quickly balanced in sealed condition. Concrete with polycarboxylate acid/salt superplasticizer has the lowest creep value because polycarboxylate acid/salt superplasticizer improves hydration degree and reduces porosity of macro pores.

Keywords: creep; internal humidity; superplasticizer; surface tension; pore structure

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## 1. Introduction

As a long-term deformation behavior, creep could greatly impact concrete structures, especially reinforced concrete structure and it has attracted much attention all over the world.

Polycarboxylate superplasticizers and naphthalene based plasticizers are two popular superplasticizers of concrete. Compared with naphthalene based plasticizers, polycarboxylate acid/salt superplasticizers have a better water reducing effect, but increase air content. The water reducing effect of naphthalene is caused mainly by electrostatic repulsion <sup>[1]</sup>, but water reduce mechanism of polycarboxylate superplasticizers is essentially generated by steric hindrance <sup>[2]</sup>. These different mechanisms may show different influences on basic creep and drying creep, but research in this area is rare <sup>[3]</sup>.

Creep is the phenomenon that the load induced deformation of material increases constantly as time increases under sustained load <sup>[4]</sup>. Basic creep is delayed deformation of specimens produced by constant load under equilibrium moisture conditions (equivalent to sealed conditions). Subtracted by the shrinkage of non-loaded specimens and basic creep of sealed concrete specimens, the remained part of creep under unsealed conditions is called drying creep <sup>[5]</sup>. Many studies suggest that basic creep is mainly caused by transmission of capillary pore water and gel pore water <sup>[6, 7]</sup> and slippage of gel layers <sup>[8, 9]</sup>. For drying creep, explanations based on micro cracking effect <sup>[10]</sup> and drying-induced theory <sup>[11]</sup> are involved with moisture diffusion to outside environment.

To investigate the effect of superplasticizers on basic creep and drying creep, we study the influence of superplasticizers on pore structure (i.e. pore size distribution) and properties of pore solution i.e. surface tension of pore solution.

## 2. Experimental

### 2.1 Raw materials and mix proportion

PO 52.5 cement, Grade I fly ash, S95 GBFS, medium sand ( $M_x = 2.7$ ) and graded gravel (5 to 25 mm) were used for preparing samples. Chemical compositions of PO 52.5 cement was shown in Table 1. Four different polycarboxylate salt/acid superplasticizers (hereafter referred as “PCA”) J1, J2, J3, J4, J5 and two kinds of naphthalene based plasticizers (hereafter referred as “NF”) N1, N2 from different companies were used in this study. Detailed information of superplasticizers was given in Table 2.

The designed compressive strength and elastic modulus of concrete were 50 MPa and 35.5 Gpa at 7d, meeting the requirements for pre-stressed concrete. The superplasticizers dosages were adjusted to make the initial slump of concretes, which are of the same proportion of raw materials, range from 200 mm to 220 mm. The specific mix proportion is shown in Table 3. Table 4 shows dosage of superplasticizers, initial slump and basic mechanical properties of concretes.

## 2.2 Method

### 2.2.1 Measurement of shrinkage, creep and internal relative humidity

Prism specimens with dimensions of 130 mm×130 mm×400 mm were casted and cured for 7d in a curing room where the relative humidity was 95% and the temperature  $20\pm 1^\circ\text{C}$ . After that the specimens were moved into a testing room where the relative humidity was  $60\pm 5\%$  and the temperature  $20\pm 1^\circ\text{C}$ . For measuring sealed shrinkage and basic creep, the specimens were sealed with epoxy immediately after moved into the testing room.

For each test (sealed shrinkage, drying shrinkage, basic creep and total creep), eight specimens were used. Shrinkage was measured by using dial indicators, as shown in Fig 1 (a). Creep was measured by using vibrating string extensometer, as shown in Fig 1 (b). For compressive creep test, specimens were loaded by using “gourd string” post-tension loading method (see Fig. 2) and stress caused by the loads on the specimens was 20 MPa. The specific values of basic creep and drying creep as a function of age were calculated with the collected data.

Internal relative humidity was measured by humidity sensors embedded in the center of the specimens (see Fig. 2) and the sensors were sealed by paraffin. The sensors only exchanged moisture with concrete, completely isolated from outside environment.

### 2.2.2 Mercury intrusion method

Mortar was sieved from the concrete casted for creep testing. Porosity and pore size distribution were tested by means of mercury intrusion method according to GB/T 21650.1-2008. Then we discussed the relations between information of pore structure and data of creep and internal moisture.

### 2.2.3 Measurement of surface tension of pore solution

In this study, the influence of superplasticizers on surface tensions of deionized water and pore solution were investigated. For the case of deionized water, the same proportion of superplasticizers and deionized water for preparing concrete was applied. For the case of pore solution, mortar obtained from concrete at the age of 7 days was ground until the particle size was less than 0.08 mm (checked with sieve). After that, the powder from mortar was mixed with deionized water with a proportion of 5:4. After incubated for 1 day, supernatant solution was extracted for tests.

The surface tension of solutions obtained by these two methods was measured by automatic surface tension meter.

## 3. Result and discussion

### 3.1 Influences of superplasticizers on shrinkage of concrete

Fig. 3 shows the total shrinkage of concrete while Fig. 4 shows the shrinkage due to self-desiccation. Compared with concrete with NFs, concretes with PCAs have smaller shrinkage. Among PCAs, concrete with PCA J1 has smallest total shrinkage and

self-desiccation shrinkage. For comparison, Fig. 5 shows relative value of drying shrinkage and self-desiccation shrinkage. Except for concrete with J2 and J3, self-desiccation shrinkage is less than drying shrinkage.

### 3.2 Influences of superplasticizers on basic creep and drying creep of concrete

Specific creep curves of concrete under unsealed conditions are shown in Fig. 6. Compared with concrete with NFs, concretes with PCAs have smaller creep. Among PCAs, PCA J1 shows more obviously effect.

Specific creep curves of concrete mixed with different superplasticizers under sealed conditions are shown in Fig. 7. The basic creep shows similar behaviors to total creep. But the reduction effect by PCAs on basic creep is less.

For better comparison, specific creeps, i.e., total creep, basic creep and drying creep, of concrete with N2 were normalized as 100%, respectively. Relative values of concretes with other superplasticizers are shown in Fig. 8. Drying creep represents additional creep of concrete under drying condition compared with creep under sealed condition.

J1 can significantly improve the strength and elastic modulus of concrete with same w/c ratio (see Table 4). Thus the deformation resistant ability was enhanced and creep was reduced significantly. Other PCAs' effects on strength and elastic modulus are not as obvious as J1. As can be seen in Fig.3, compared with N2, PCA J1,J2,J3,J4,J5 can reduce basic creep by 35%, 10%, 17%, 11%, 5%, and reduce drying creep by 38%, 37%, 41%, 29%, 28%, respectively. It can be inferred that, in addition to PCA J1, other PCAs have similar effect, greatly reduce drying creep, and have little effect on basic creep.

### 3.3 Internal relative humidity variation with time

The internal relative humidity of sealed and unsealed specimens with J1, J3, N2 were measured by using embedded humidity sensors. Internal relative humidity data is shown in Fig. 9.

It can be seen that internal relative humidity of specimens with different superplasticizers under sealed or unsealed conditions varies significantly. The influences of superplasticizers on internal relative humidity of specimens under sealed conditions are similar to those under unsealed conditions. Initially, the internal relative humidity of sealed and unsealed concrete shows no obvious difference. It indicates that the internal water loss is mainly caused by consumption of hydration reaction, which is called as self-desiccation. In the later age, the reduction rate of sealed specimens become much lower and finally close to zero, indicating that the hydration reaction becomes feeble. In comparison, internal relative humidity of unsealed specimens continues to decrease till the end of this experiment, caused by drying.

To study the influence of different superplasticizers on internal humidity of sealed specimens, we draw curves of internal relative humidity loss (relative humidity decreased due to self-desiccation) as a function of loading time (see Fig. 10).

From the curves, we can see that relative humidity loss due to self-desiccation of J3 and N2 are similar, but much lower than J1. It can be inferred that specimens with J3 and N2 have similar amount of hydration products while the amount of hydration products in specimen J1 is relatively higher. It is consist with the phenomenon that concrete with J1 has higher elastic modulus and strength than with J2 and N2. Also, the elastic modulus and strength of concrete with J3 and N2 are close to each other.

We subtracted internal humidity loss of sealed specimens mixed with same superplasticizer from values for unsealed ones. Then we got the relative humidity loss due to drying, as shown in Fig. 11.

The internal relative humidity loss of J1 and J3 caused by drying are distinctly lower than N2. At 360d, the loss of relative humidity of J1 and J3 only account for 59% and 57% of the value for N2, respectively. Therefore, PCAs could significantly restrict water transport towards outside environment. Also, when concrete specimens are sealed, the transport and redistribution of water in the concrete are also influenced by PCAs, thus reducing the basic creep.

### 3.4 Test and analysis of pore structure

Superplasticizers can improve the workability of concrete and disperse cement particles (see Table 4), so they may influence pore structure. The dispersion mechanisms of PCAs and NFs are not exactly the same. Thus, their influence on pore structure may show some difference. Fig. 12 shows the pore distribution curves of mortar sample mixed with superplasticizers J1, J3 and N2 at 28d.

For the same mix proportion, PCAs can reduce porosity at all scales. J1's reduction of the pores larger than 100nm is much more obvious. Therefore, concrete with J1 is more compact. The other two superplasticizers have similar effect on porosity of pores smaller than 100nm. Therefore, it can be seen that PCA can refine pore structures and reduce pore connectivity, which is in good agreement with some other studies<sup>[13]</sup>. As a result, the water transport in concrete is restricted.

### 3.5 Surface tension of pore solution

Table 5 shows the surface tension value of solutions with different superplasticizers measured by two methods.

All PCAs have reduction effect on surface tension of pore solution, but NFs show no effect. The effect of J1 is the most significant, while other PCAs show relatively weaker effect. This is similar to drying creep and internal relative humidity loss due to drying effect.

### 3.6 Influence mechanism of PCA on concrete creep

From previous studies on creep mechanism, we can see that, both basic creep and drying creep are caused by interaction of cement paste, pore and pore solution under load and environmental effects. Superplasticizer, as a system dispersing agent, is bound to impact pore structure and pore solution. From these aspects, we can explain the influence of PCA on concrete creep.

According previous experiment and discussion, we analyzed the influence mechanism of PCA on concrete creep. Compared with NF, concrete with normal PCA has less hydration products, but the porosity of the paste is also low. Thus the deformation resistance is essentially the same. PCA can lower porosity of micro pores and connectivity of pores. PCA can also reduce surface tension of pore solution, improve water conservation ability, and, therefore, restrain micro-diffusion<sup>[14]</sup> of adsorbed water in capillary pores and gel pores. Therefore, they reduce additional pressure<sup>[15, 16]</sup> on capillary walls, inhibit glide deformation of C-S-H gel and damage degree of gel combined due to moisture transmission<sup>[17]</sup>. As a result, creep is reduced. In addition to these effects, early strength PCA can also improve hydration

degree and reduce porosity of macro pores, thus the elastic modulus of concrete is higher. The creep deformation of early strength PCA is lower than normal PCA. In the dry state, humidity gradient internal concrete is formed induced by external low humidity, thus the moisture transport is enhanced. PCAs can reduce the diffusion and greatly lower the drying creep. In the sealed state, distribution of internal moisture become equilibrium faster, the influence of transport and redistribution of pore water become feeble, so the effect of PCA on basic creep is much lower.

#### 4. Conclusions

- (1) Compared with NF, PCA can significantly reduce creep of concrete. For the same mix proportion and fluidity of concrete mixture, PCAs selected in this study can reduce total creep by 38%, 17%, 24%, 16%, 11%, respectively; reduce basic creep by 33%, 9%, 17%, 11%, 4%, respectively; and reduce drying creep by 53%, 38%, 44%, 31%, 28%, respectively. Among them, the reduction effect of early strength PCA is most significant.
- (2) Compared with NF, concrete with normal PCA has lower porosity and connectivity of micro pores, and reduce surface tension of pore solution, so PCA can restrain micro-diffusion of adsorbed water in capillary pores and gel pores; reduce additional pressure on capillary walls, inhibit glide deformation of C-S-H gel and damage degree of gel combine due to moisture transmission. As a result, creep is reduced. In addition to these effects, early strength PCA can also improve hydration degree and reduce porosity of macro pores, enhance elastic modulus of concrete, thus creep of early strength PCA is lower than normal PCA.

(3) In the dry state, humidity gradient is formed inside concrete and water diffusion from concrete to the outside environment is improved. PCA can reduce the diffusion and greatly lower the drying creep. In the sealed state, distribution of internal moisture become equilibrium faster and the influence of transmission and redistribution of pore water become feeble. Therefore, the effect of PCA on basic creep is much lower.

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Tab. 1: Chemical compositions of PO 52.5 cement

Compositions	CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	L.O.I	Total
Percentage (%)	64.90	20.80	6.23	4.88	0.98	2.21	100

Tab. 2: Properties of different types of superplasticizers

Series	Types	pH	Surface tension (mN/m)			
			Mn	Mw	Mz	
J1	Acrylic acid-based	5.54	50.8	5770	30000	74700
J2	Maleate-based	3.53	66.4	2800	12100	34700
J3	Acrylic acid-based	6.18	64.8	5390	21600	57200
J4	Maleate-based	7.31	68.4	4910	14600	34000
J5	Maleate-based	7.27	67.5	5900	27200	69400
N	Polynaphthalene	7.57	74.5	--	--	--

\*Mn refers to Number-average molecular Weight; Mw refers to Weight-average molecular Weight and Mz refers to Z-average molecular weight.

Tab.3 Mix proportion for concrete / (kg/m<sup>3</sup>)

Cement	Fly ash	Mineral powder	Sand	Gravel	water
336	96	48	708	1062	142

Tab. 4 Admixture, initial slump, compressive strength and elastic modulus of concretes

Admixture Classes	Superplasticizers (Kg/m <sup>3</sup> )	Initial slump (mm)	Compressive Strength at Loading Age	Elastic modulus at Loading Age
J1	2.17	205mm	57.8Mpa	38.9Gpa
J2	2.41	200mm	52.8Mpa	37.1Gpa
J3	2.30	210mm	53.1Mpa	36.7Gpa
J4	2.20	205mm	50.5Mpa	37.8Gpa
J5	2.43	215mm	50.9Mpa	36.3Gpa
N1	4.07	200mm	52.9Mpa	36.4Gpa
N2	4.55	210mm	51.7Mpa	35.9Gpa

Tab. 5 Surface tension of pore water measured by two methods

Class	J1	J2	J3	J4	J5	N1	N2	Blank	
Surface tension	I	50.8	66.4	64.8	68.4	67.5	73.4	74.5	72
(mN/m)	II	61.4	69.4	68.5	69.8	69.6	72.8	73.8	72

## Figures



(a)



(b)

Fig. 1 (a) Measurement of shrinkage and (b) Measurement of creep

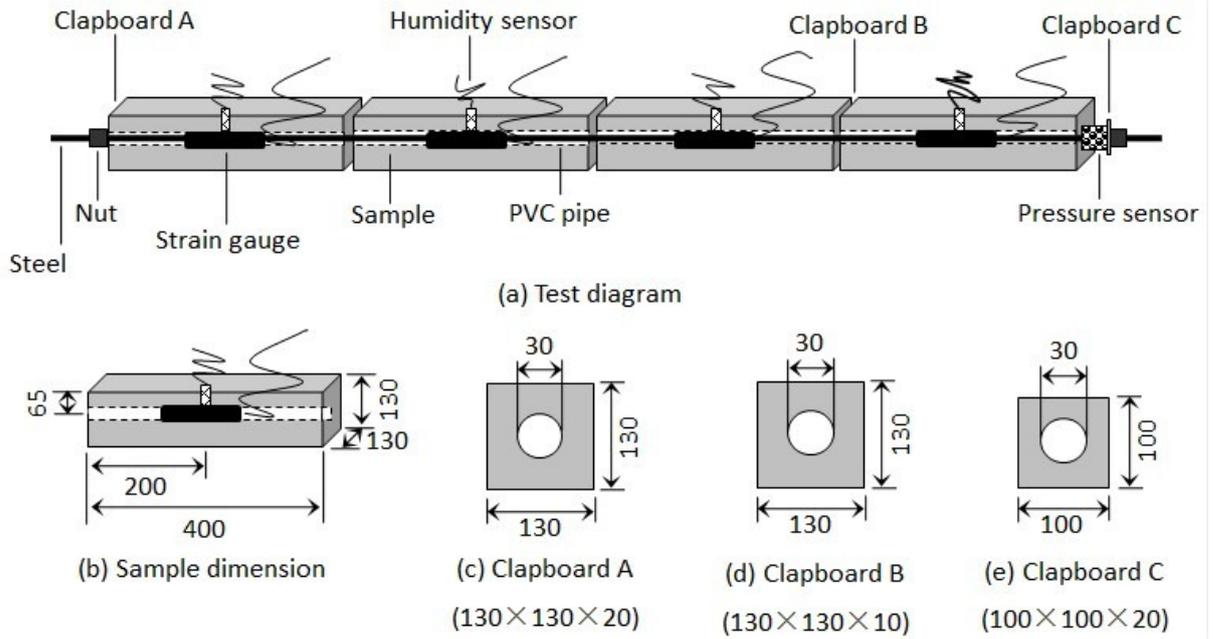


Fig. 2 Schematic of “gourd string” post-tension loading method

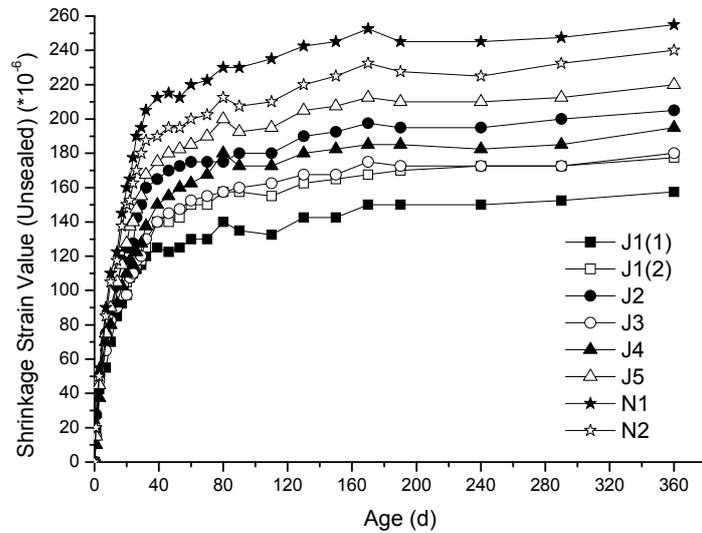


Fig. 3 Total shrinkage of concrete with different superplasticizers

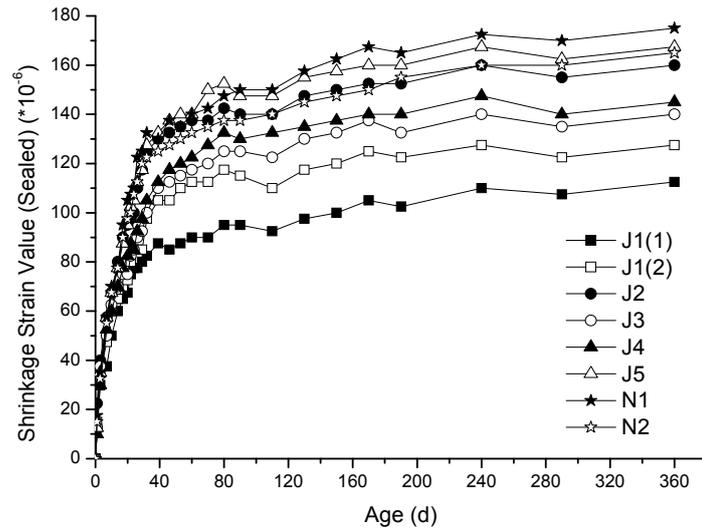


Fig. 4 Self-desiccation shrinkage of concrete with different superplasticizers



Fig. 5 Comparison between drying shrinkage and self-desiccation shrinkage of concrete with different superplasticizers

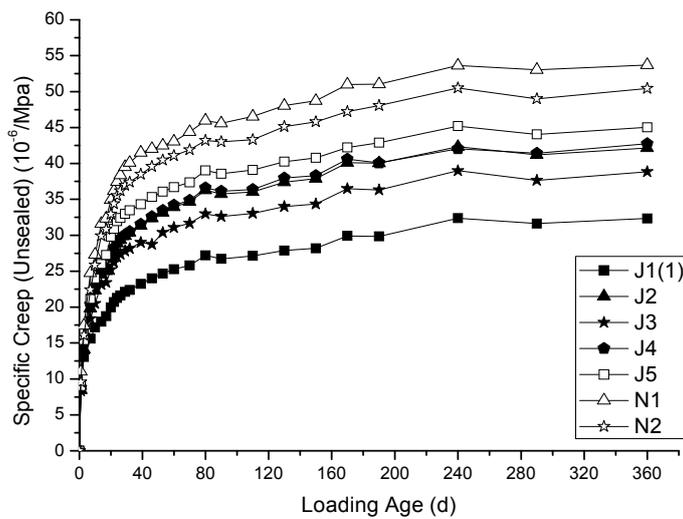


Fig. 6 Specific creep of concrete mixed with different superplasticizers under unsealed condition (Total creep)

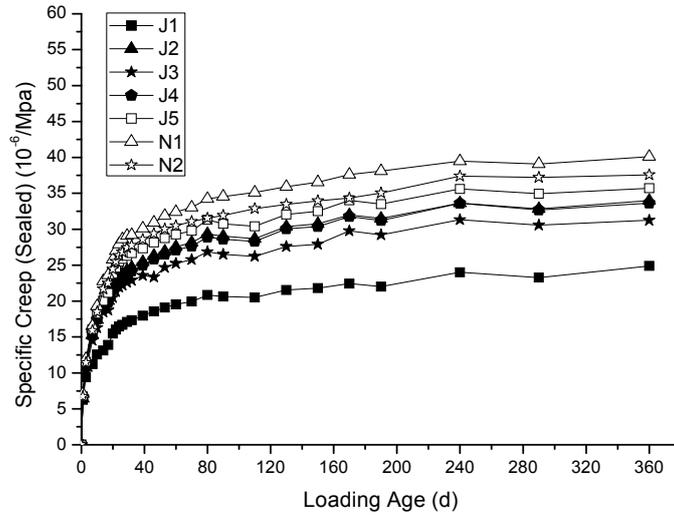


Fig. 7 Specific creep of concrete mixed with different superplasticizers under sealed condition (Basic creep)

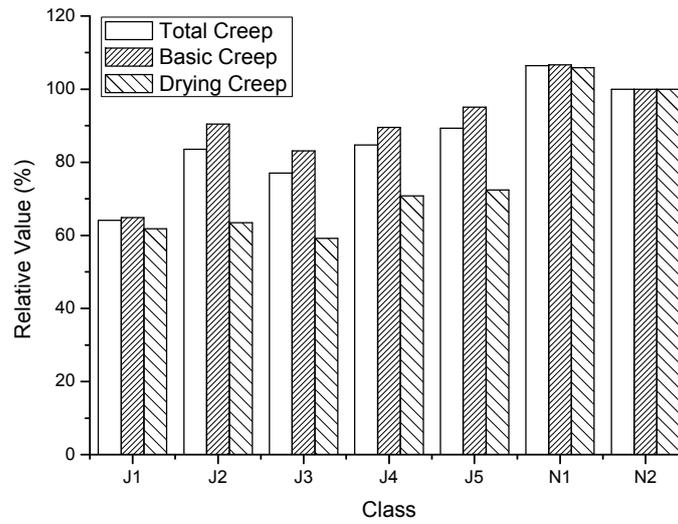


Fig. 8 Relative value of creep of concrete mixed with different superplasticizers

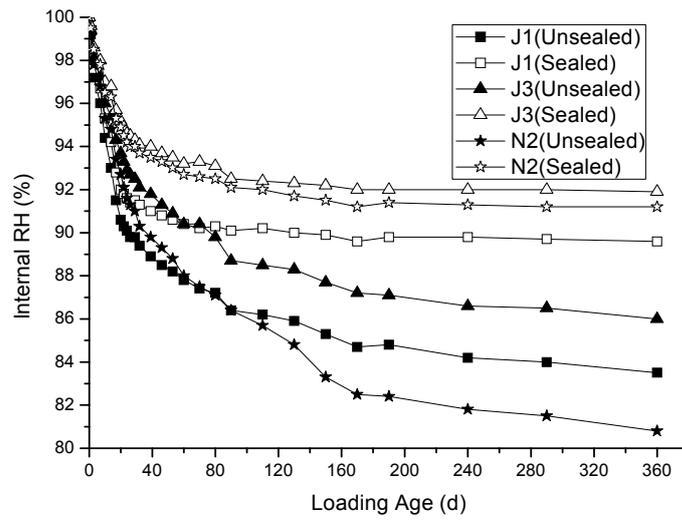


Fig. 9 Internal humidity of loaded concrete under sealed and unsealed condition

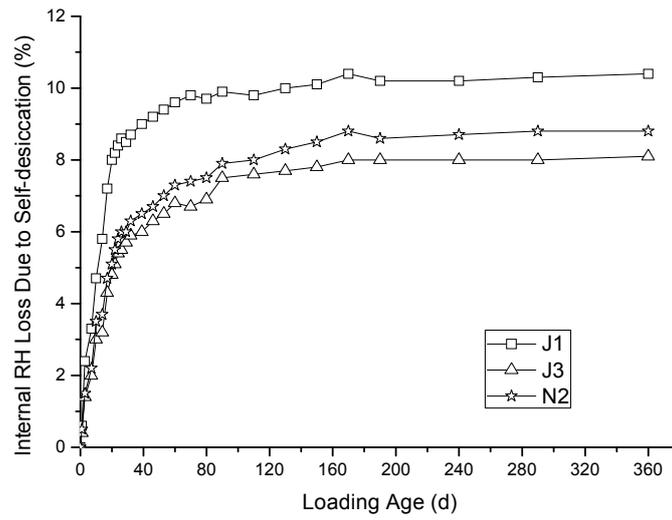


Fig. 10 Internal Relative humidity loss due to self- desiccation

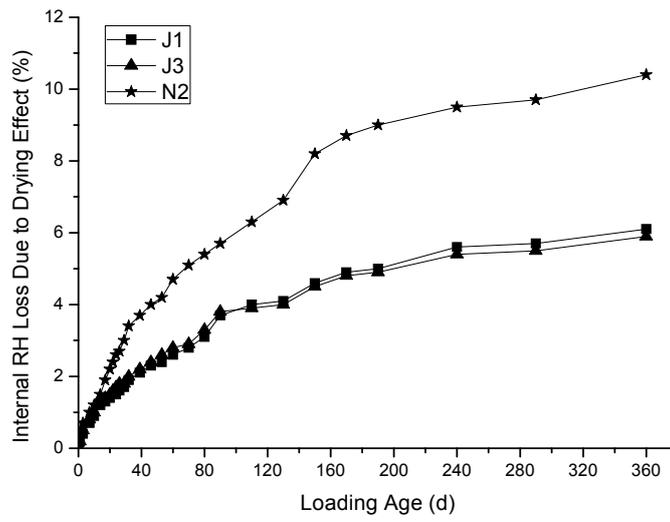


Fig. 11 Internal humidity loss due to drying effect

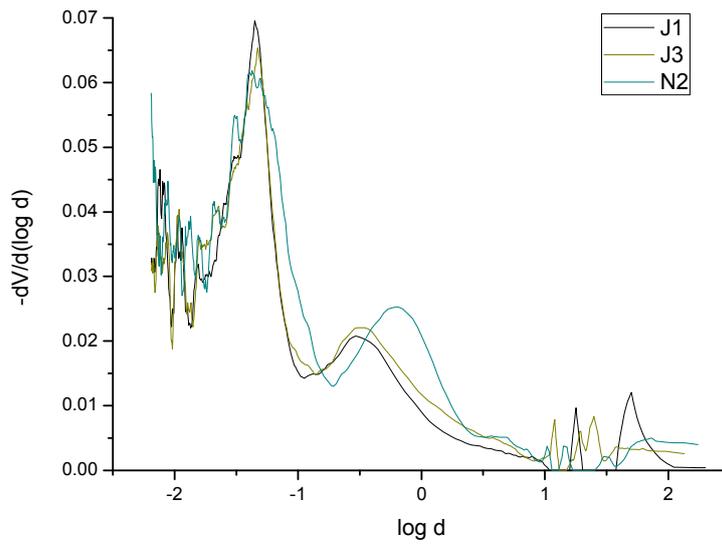


Fig. 12 Pore size distribution of concrete mortar