

DRYING SHRINKAGE AND MICROSTRUCTURE CHARACTERISTICS OF GROUND GRANULATED BLAST FURNACE SLAG-CEMENT MORTAR

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ABSTRACT

This paper presents an experimental study to investigate the effects of ground granulated blast furnace slag (GGBFS) fineness, cement type and curing age on the drying shrinkage and microstructure of GGBFS blended cement mortar. The test results revealed that the drying shrinkage evolution can be significantly affected by GGBFS powder fineness, cement type, specific surface area and 6~30nm porosity, but curing age. As a result of this study, the prediction equation of drying shrinkage is proposed based on ESW specific surface area, the volume of 6~30nm diameter pore and mass loss.

Keywords: drying shrinkage, ground granulated blast furnace slag, cement type, microstructure, mass loss, specific surface area, excess surface work.

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1. INTRODUCTION

Drying shrinkage, as one of the major reasons of cracks generated in the concrete structures [1], can be defined as the volumetric change owing to the drying of concrete. This change in volume of the concrete is related to the volume of water lost. The loss of free water, which occurs first, may induce shrinkage. As the drying process of the concrete continues, the adsorbed water held by hydrostatic tension in the small capillaries is reduced significantly [2]. The loss of the water (free water and adsorbed water) produces tensile stresses, which force concrete to shrink causing cracks that can adversely affect the structural performances, if not appropriately considered in the design stage.

Ground granulated blast furnace slag (GGBFS), as a kind of by-product of steel industry, has been widely used as a mineral admixture for blast furnace cement that is specified as a promoting green procurement for the global environment. It is well known that the large production of Portland cement consumes a huge amount of energy, and hence grows to be a main source of CO₂ and atmospheric pollution. Thus, the use of the GGBFS can be less expensive and eco-friendly solution for cement production [3].

There are many studies in the literature [1-5] that have dealt with hydration evolution, microstructure characteristics, and drying shrinkage of blast furnace cement systems. It was found that the hydration of GGBFS showed a different reaction process compared to cement minerals, and thus the hydration products and microstructure formation are different. The previous study [4] declared that the drying shrinkage of blast

furnace cement is associated with the amount of produced C-S-H gel, meaning that it may vary with cement hydration degree which could be affected by cement type and curing age. In spite of these efforts, the clarification of blast furnace cement characteristics and curing age effects on the drying shrinkage and microstructure formation has not been fully understood, which is quite important for drying shrinkage control in concrete with GGBFS.

The objective of this study is to clarify the influences of GGBFS powder fineness, cement type, and curing age on the drying shrinkage evolution of GGBFS blended cement mortar, and the correlation of drying shrinkage with microstructure formation as well. Based on this study, an equation that can predict reasonably the drying shrinkage in GGBFS-cement mortar is developed.

2. EXPERIMENTAL WORK

2.1 Materials and mix proportions of mortars

Ground granulated blast furnace slag (GGBFS) with fineness of 3000, 4000 and 8000 cm²/g, and three types of cement, which are ordinary Portland cement (N), moderate heat Portland cement (M), and low heat Portland cement (L), were used to produce mortar samples in this study. Their compositions are shown in Table 1. Table 2 shows the mix proportions by weight ratio of mortars which are not changed with different cement type and GGBFS fineness. The mortars were casted into 40 x 40 x 160 mm prisms. After 1 day, samples demoulded and cured at 20°C in water, as two sets, for 6 days and 27 days. After curing, samples were

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Table 1 Elemental composition of the materials used

Sample	Density (g/cm ³)	Blaine(cm ² /g)	Chemical composition (%)							
			SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	SO ₃
N	3.17	3390	21.06	5.51	2.69	65.47	1.66	0.40	0.24	1.91
M	3.22	3050	22.59	4.46	3.99	62.63	2.25	0.39	0.21	2.24
L	3.24	3540	25.36	3.34	3.43	62.33	0.80	-	-	2.75
GGBFS3000	2.90	3180	33.86	15.19	0.90	40.89	6.57	-	-	-
GGBFS4000	2.91	4030	34.12	16.07	0.38	42.06	6.21	-	-	-
GGBFS8000	2.91	7300	33.67	16.24	0.59	42.14	5.35	-	-	-

placed in a control chamber at 20°C and 60% relative humidity.

2.2 Testing methods

The measurements of length change as a scale of the drying shrinkage of mortar prisms were conducted based on the JISA 1129-3 Dial Gauge Method. The initial gauge length of prisms was measured by a dial gauge and consecutive readings were obtained for 52 weeks. Each observed data point shown in the figures, such as Fig. 1, is the mean of measurement values of drying shrinkage for three mortar prisms with the same condition. After the specified curing time, mortar prisms were cut into 5mm cube samples, and then dried to stop the hydration reaction. The amount of capillary pore volume and gel pore volume were quantified based on Archimedes method [4, 5]. The capillary pore volume was calculated as the change of sample weight at 40°C, and the gel pore volume was estimated as the difference in sample weight between 40°C and 105°C.

Pore size distribution was determined with mercury intrusion porosimetry (MIP) method using Autopore Master33 porosimeter, in which a hydraulic pump generates the pressure and a contact sensor was applied to measure the mercury volume. The surface tension of the mercury was 0.480 N/m. The mercury density was 13.546 g/ml and the assumed contact angle was 140°.

Water vapor adsorption was measured using a hydrosorb1000. Surface area was calculated using BET theory [6] and ESW (Excess Surface Work) theory, which are successfully applied in research and industry patents [7].

3. RESULTS AND DISCUSSION

3.1 Drying shrinkage

Figs.1 and 2 show the strain developments versus time of drying shrinkage for different mortar mixes cured for 7 days and 28 days, respectively. Each of Fig. 1 (a) and Fig. 2 (a) presents the drying shrinkage evolution for mortar samples with the same condition except the type of cement for 7 days and 28 days curing, respectively. While Figs. 1 (b), (c) and (d) show the influence of GGBFS fineness as a mineral admixture on the relationship between shrinkage strain and drying time in mortars with Portland cement (N), moderate heat Portland cement (M), and low heat Portland cement (L) cases, respectively; same description can be given for Figs. 2 (b), (c) and (d),

Table 2 Mix proportions of mortars by weight ratio

Sample	C	GGBFS	W	S
Without GGBFS	100	0	50	300
With GGBFS	55	45	50	300

except the curing age which is 28 days.

It can be revealed from Fig.1 (a) and Fig.2 (a) that the shrinkage strain of cement mortars increases with the drying age, being hardly comparable at early ages and differed at later ages. Also, the increasing rate of shrinkage strain of mortars at early ages is considerably steep compared to the increase rate after 21 days, as shown in the figures.

By observing the drying shrinkage in each of Fig. 1 (a) and Fig. 2 (a), which exhibits the comparison for mortar samples without using GGBFS, it is obvious that mortar samples N and L showed greater shrinkage strain compared to M sample. While for the case of mortar samples incorporating GGBFS, as shown in Figs. 1 (b), (c), (d) and Figs. 2 (b), (c), (d), L mortar sample showed the greatest drying shrinkage strain among investigated mortar with different cement types. This observation can be evident by comparing L8-45-7 sample with M8-45-7 and N8-45-7, all these mortar samples with the same condition, which are 8000 cm²/g GGBFS fineness, 45% GGBFS replacement ratio and 7 days curing age, except the cement type, meaning that cement type can affect the drying shrinkage evolution.

The influence of GGBFS fineness, which was chosen in the study as 3000, 4000 and 8000 cm²/g, on drying shrinkage development was examined in this study. The results are shown in Figs. 1 (b), (c), (d) and Figs. 2 (b), (c), (d). It is clear from Fig.1 (b) and Fig. 2 (b), regardless of curing age, GGBFS fineness 3000 and 4000 cm²/g showed small effect on the shrinkage strain evolution in N cement mortar case, while 8000 cm²/g fineness produced larger strain values. Moreover, it can be revealed from Figs. 1 (c), (d) and Figs. 2 (c), (d) that the larger GGBFS fineness, the greater shrinkage strain can be obtained in M and L cement mortars. Therefore, it can be concluded that the drying shrinkage development in mortar is related to both GGBFS fineness and cement type.

A comparison of drying shrinkage for mortar samples for 7 days and 28 days curing age is given in Fig. 3. Increasing the curing age from 7 to 28 days resulted in slightly increasing the shrinkage strain of M and L cement mortars, while it showed significant effect in N cement mortar case. Meanwhile, the curing

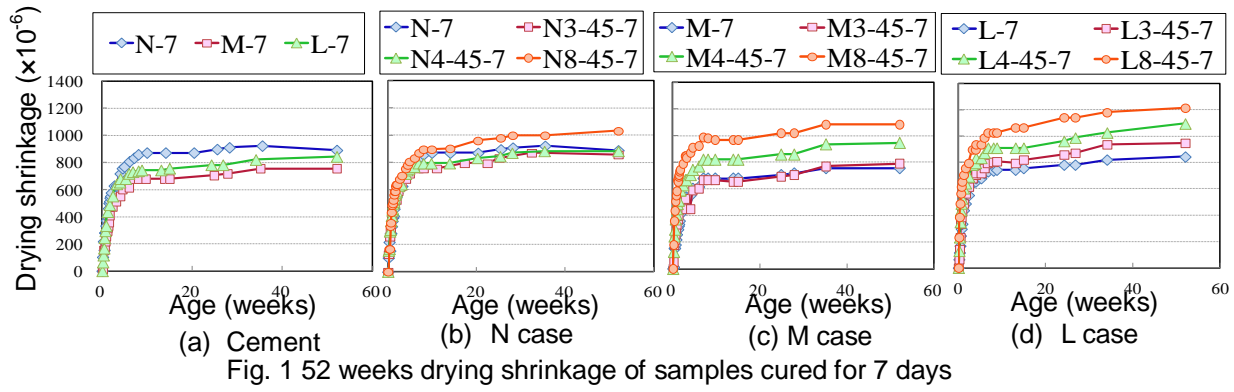


Fig. 1 52 weeks drying shrinkage of samples cured for 7 days

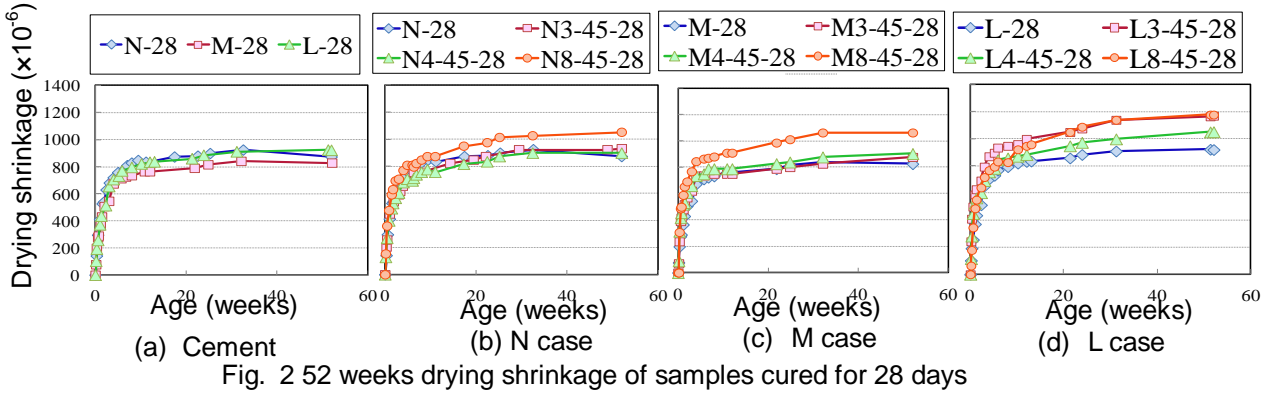


Fig. 2 52 weeks drying shrinkage of samples cured for 28 days

- [Symbol]
1. Cement type and GGBFS
 2. Replacement ratio of GGBFS is 45%
 3. Curing age 7 and 28 days

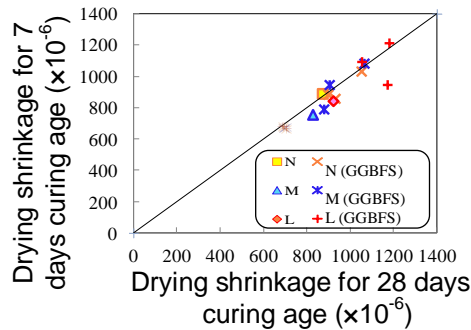


Fig. 3 Effect of curing age (7 and 28 days) on drying shrinkage at 52 weeks

age result has the same tendency with Yoon et al. [9]. Although, the curing age before drying is different, the drying shrinkage shows almost similar tendency. Hence, it is considered that the curing age has inconsiderable effect on the drying shrinkage. Therefore, the drying shrinkage evolution for 7 days curing age case was used in the discussion of the following sections.

3.2 Relationship between drying shrinkage and water diffusion

The drying shrinkage in the case of 7 days curing age is plotted versus the water loss in Fig. 4. When the GGBFS is not used, as shown in Fig. 4 (a), the amount of the water diffusion follows the order $N < M < L$. Furthermore, it can be revealed from Fig. 4 (b), (c) and (d) that greater GGBFS powder fineness resulted in less amount of water diffusion for the same shrinkage strain.

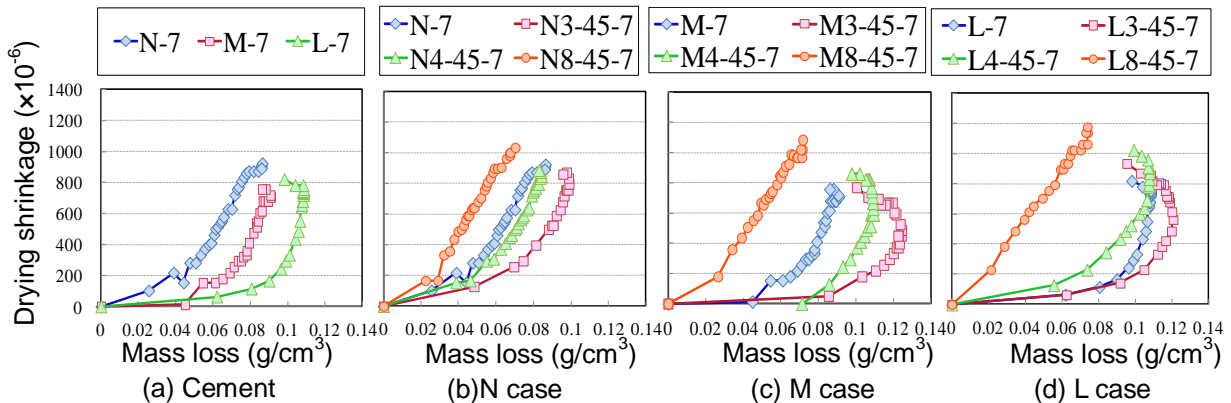


Fig. 4 The relationship between mass loss and drying shrinkage of samples cured for 7 days

It is clear that the drying shrinkage became greater with the increase of water diffusion; however, drying shrinkage mechanism is not only related to the mass loss. Moreover, under a certain drying age, it was found that during the process of mass increase, the amount of water diffusion has a decreasing tendency, as shown in the figure, which may be resulted from carbonation.

3.3 Relationship among the capillary pore volume, gel pore volume and drying shrinkage

Fig. 5 discusses the relationship between the drying shrinkage and the amount of both capillary pore volume and gel pore volume measured by Archimedes method. Generally, as shown in the figure, with the increase of the volume of capillary pore, the drying shrinkage strain tends to increase. It is interesting to notice that a scatter was obtained in the result between the drying shrinkage and the amount of gel pore volume. The relationship of porosity structure and drying shrinkage of cement hardened paste is that: the formation of the liquid-vapor in the pores can ultimately lead to shrinkage. The Young-Laplace equation (Eq. 1) can be used to describe the size of the menisci to the capillary pressure.

$$\Delta P = 2\gamma / r_s \quad (1)$$

Where ΔP (Pa) is capillary pressure, γ (N/m) is the surface tension of pore fluid, r_s (m) is meniscus or porosity radius. From one side, it can be argued that the smaller pore radius corresponds to a higher capillary pressure. In other words, the stress becomes larger with

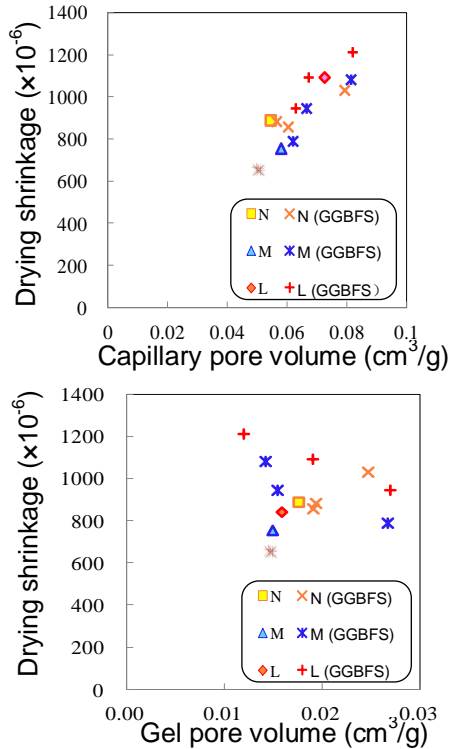


Fig. 5 The relationship between capillary pore volume, gel pore volume and drying shrinkage

the increase of pore volume whose radius is smaller than the parameter r_s . On the other side, during the process of hydration, pore size distribution becomes finer and water diffusion becomes slow. The actual drying shrinkage can be related to the balance of the influences caused by both sides. However, other factors, besides capillary and gel pore volume, on drying shrinkage should be considered for the quantitative shrinkage assessment of GGBFS-cement mortar, such as specific surface area and pore volume.

3.4 The effect of specific surface area and pore volume on the drying shrinkage

Fig. 6 shows the relationship between the drying shrinkage strain and specific surface area, which is calculated by BET theory, for examined mortar samples. BET calculation is based on the multilayer adsorption theory, which is extension of Langmuir monolayer adsorption theory, over a relative pressure range of 0.05~0.35 for the isotherm [6]. It can be observed from Fig. 6 that the larger is the specific surface area, the greater is the amount of shrinkage with increase tendency based on the cement type. For ordinary Portland cement (N) cement case, uncorrelated variation can be seen. While in the M and L cement case, it can be observed that the drying shrinkage relates well the specific surface area with a correlation coefficient (R) around 0.9. The small effect of GGBFS fineness on the shrinkage strain in N cement mortar case (section 3.1) is the reason for this behavior, but the measurement value of specific surface area is distinguished. From the relationship between drying shrinkage and specific surface area, it is considered that the matrix of both hardened cement and cement blended with GGBFS is not the same.

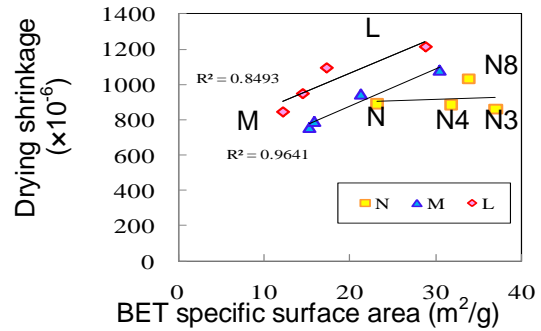


Fig. 6 Specific surface area by BET method and drying shrinkage relationship

The water vapor isotherm was also analyzed by ESW (Excess Surface Work) theory, which is related to exhaustive adsorption thermodynamics for the whole relative pressure, which is different from BET theory. Fig. 7 shows the comparison result of specific surface area of studied mortars by BET and ESW methods. It can be inferred from the figure that ESW specific surface area due to water vapor adsorption isotherm are in a good agreement with BET specific surface area with a correlation factor of 1.2.

Based on the previous research findings [10], generally, at 55~60% relative humidity, the diameter of the capillary pore, which is involved in the drying shrinkage, is assumed to be 30nm or less. Besides, in the laboratory, the mercury intrusion porosimetry cannot be applied to measure the amount of 6nm diameter pores or less. Therefore, the relationship between the amount of drying shrinkage and pore volume of 6~30nm diameter is plotted in Fig. 8. The figure shows that, for all cement types, with the increase of 6~30nm diameter pores volume, the shrinkage strain tends to be higher. Among them, the highest correlation was obtained in L cement case. This result is consistent with Bentur et al. study which demonstrated that the drying shrinkage is directly related to the drying shrinkage value of pore volume below 30nm. Together with the previous discussion on the specific surface area, it can be said that shrinkage increases proportionally with the smaller pore volume.

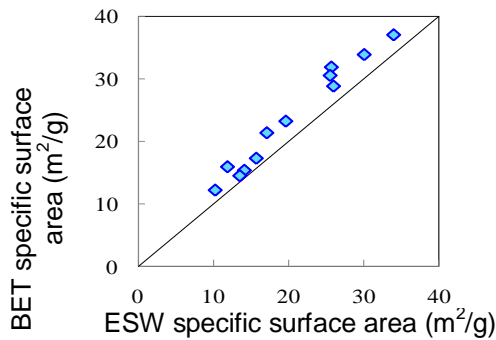


Fig. 7 Comparison of specific surface area calculated by BET method and ESW method

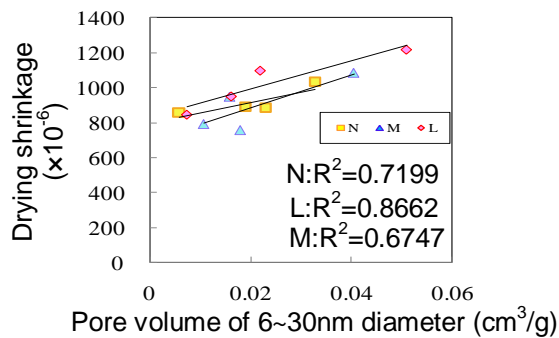


Fig. 8 The relationship between pore volume of 6~30nm diameter and drying shrinkage

3.5 Multiple linear regression analysis

As discussed above, the pore volume, mass loss and specific surface area are closely related to the drying shrinkage. Drying shrinkage strain (y), as the target variable, can be calculated by a multiple regression analysis using three variables ESW specific surface area (x_1) in m^2/g , the pore volume of 6~30nm (x_2) in cm^3/g and mass loss (x_3) in g/cm^3 as shown in Eq. 2.

$$y = 5.36x_1 + 14165.1x_2 + 10468x_3 - 400.11 \quad (2)$$

Fig. 9 shows that the predicted values of drying shrinkage by Eq. 2 has an acceptable correlation with the actual test values, with coefficient of determination (R^2) of 0.85, which explain the good accuracy of drying shrinkage prediction formula. The prediction equation of the drying shrinkage was proposed and could express the reliability of drying shrinkage on these factors through the experiments conducted. In the future, further investigation will be done to clarify this point for concrete structures.

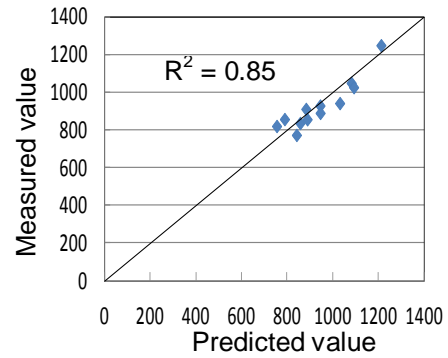


Fig. 9 The relationship between predicted and measured drying shrinkage values

4. CONCLUSIONS

Based on the experimental results, the main findings of this study, which are useful information for drying shrinkage control, can be drawn as follows:

- (1) It was found that for mortar samples without GGBFS, N and L mortar samples showed greater shrinkage strain evolution compared to M sample. While for the case of mortar samples incorporating GGBFS, L mortar sample showed the greatest drying shrinkage strain among investigated mortar mixes.
- (2) GGBFS showed a small effect on the drying shrinkage when it was added to N mortar case. For M and L Portland cement mortar, the drying shrinkage increased significantly. This tendency was also remarkable for the increase of GGBFS fineness in all tested cement mortars, implying that the cement type and GGBFS fineness are main factors on drying shrinkage. On the other hand, the test results revealed that curing age has insignificant effect on the drying shrinkage.
- (3) The drying shrinkage became larger with the increase of capillary pore volume. However, a scatter was obtained in relationship between the gel pore volume and the drying shrinkage.
- (4) According to the microstructure results, the drying shrinkage has significant correlation with specific surface area and pore volume. Smaller specific surface area and less pore volume of 6~30nm would decrease effectively the drying shrinkage.
- (5) A prediction equation for drying shrinkage was developed based on ESW specific surface area,

the volume of 6~30nm diameter pores and mass loss, which showed a good accuracy to the test results.

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