

Rice Hush Ash as smart material to mitigate autogenous shrinkage in high (ultra-high) performance concrete

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ABSTRACT

As an agriculture waste, rice husk ash (RHA) has been used a lot in construction industry to replace Portland cement. The main advantages of using RHA are the decrease of materials costs due to cement savings, environmental benefits related to the disposal of waste materials and the reduction of carbon dioxide emissions. In this paper, the application of RHA is extended to mitigate their early age autogenous shrinkage of high (ultra-high) performance concrete. The influence of particle size of RHA on the efficiency of mitigation of autogenous shrinkage and the water movement in the internal pore structure of RHA are studied experimentally. The autogenous shrinkage, relative humidity and water absorption of RHA are measured. Experimental results show that by adjusting the RHA particle size, the relative humidity inside UHPC increases and the autogenous shrinkage, caused by self-desiccation, were reduced effectively.

Keywords. rice husk ash, autogenous shrinkage, relative humidity, high performance concrete

INTRODUCTION

High performance concrete (HPC) and ultra high performance concrete (UHPC) shows excellent performances on durability and high compressive strength. However, HPC/UHPC usually experiences large autogenous shrinkage, which is caused by the very low water/binders ratio, and consequently takes high risk of early age cracking (Kovler et al., 2007). The early age cracking negates the numerous advantages of HPC/UHPC and thus considerably limits their prospective utilization in construction. Recent years, attempts are tried focusing on mitigating the autogenous shrinkage of HPC/UHPC. External curing and internal curing are two potential approaches for this problem. However, it have been reported that external curing is not effective enough to mitigate the autogenous shrinkage for HPC/UHPC because the microstructure of HPC/UHPC is so dense that the external water is difficult to penetrate into the concrete (Bentz et al., 2011). In comparison, internal curing is a more effective method to mitigate the autogenous shrinkage for HPC/UHPC. By now, water-saturated light weight aggregates (LWA) and superabsorbent polymers (SAP) are the most popular internal curing agents (Jensen et al., 2001, 2002). Unfortunately, because of the low strength, LWA as internal curing agents has negative effects on mechanical properties of HPC and even cannot be used in UHPC due to the strict requirements on the maximum size of aggregates for UHPC (Mechtcherine et al., 2008, Dudziak et al., 2008, Malhotra et al., 1987). With respects to SAP, after the water transport into concrete, it leaves voids even as large as 600 µm in concrete (Kovler et al., 2007, Bentz et al., 2011). This might also negatively influence the properties of HPC/UHPC.

Recent research in the Microlab, TU Delft was found that the rice husk ash (RHA) can be used as effective internal curing agents (Tuan, 2011) for HPE/UHPC. As an agriculture waste, rice husk ash (RHA) has been used a lot in construction industry in the role of supplementary cementitious materials to replace Portland cement. The main advantages of using RHA are the decrease of materials costs due to cement savings, environmental benefits related to the disposal of waste materials and the reduction of carbon dioxide emissions. Due to the internal nano/micro-porous structure (see Figure 1), the RHA suspects to “hold” the absorbed water in the internal pores and reduce slowly during the hydration process, thus reduce the autogenous shrinkage. Moreover RHA has pozzolanic properties, which can be treated as silica fume replacement with beneficial to environments (Tuan, 2011).

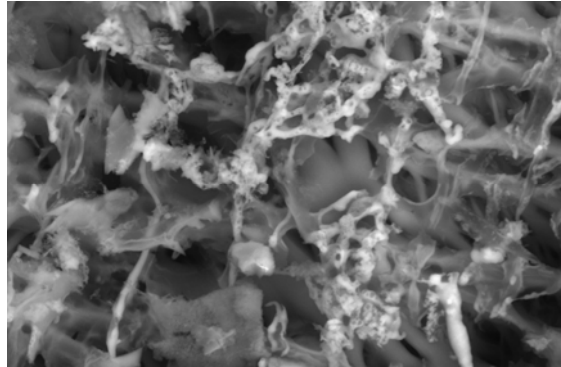


Figure 1. Internal nano/micro-porous structure of RHA

However the mechanism of mitigation of autogenous shrinkage by RHA is still not clear. For example how the particle size of RHA influence on the efficiency of mitigation of autogenous shrinkage and how much the water can be hold in the internal pore structure of RHA are the important issues to study the shrinkage phenomena. This paper aims at a fundamental study on the mitigation of autogenous shrinkage by RHA. In the experimental program, three particle sizes of RHA and replacement level 20% are studied. Autogenous shrinkage and relative humidity are measured. The experimental results are discussed.

MATERIALS AND METHODS

Materials and mixture proportion

The materials used in this study were Portland cement (CEM I 52.5N), RHA, silica sand with a particle size ranging 100 to 300 μm , and a polycarboxylate-based superplasticizer (Glenium ACE30) with 30% solid content by weight. The RHA was produced by a drum incinerator developed by PCSIR (Cook, 1996) and modified by Bui (Bui, 2001). The ash was grinded to different particle size, i.e., the mean particle sizes of 3.6, 5.6 and 9.0 μm . The properties of these ashes were shown in Table 1. The chemical composition of cement and RHA are shown in Table 2.

Standard test (soluble silica) was used to evaluate amorphous silica in the rice husk ashes according to NEN-EN 192(2). The percentage of reactive silica contained in the RHA used in this research was more than 95%.

Four types of UHPC mixtures were made. The mixture proportion is listed in Table 3. The dosage of superplasticizer was 3.5% solid by weight of binder. All mixtures were prepared in a 20-litre Hobart mixer, and followed the mixing procedure in (Tuan, 2011).

Table 1 Properties of different types of RHA

	RHA-3.6	RHA-5.6	RHA-9.0
Mean particle sizes, μm	3.6	5.6	9.0
Specific surface area (BET N_2 absorption), m^2/g	15.0	20.6	18.3
Average pore width (BJH absorption), μm	18.12	20.94	23.73
Total pore volume (BJH absorption), cm^3/g	0.0610	0.0863	0,0876

Table 2 Chemical composition of cement and RHA used in this study

Components (Chemical properties, % by weight)	Cement*	RHA**
CaO	64.00	1.14
SiO ₂	24.00	87.96
Al ₂ O ₃	5.00	0.30
Fe ₂ O ₃	3.00	0.52
SO ₃	2.40	0.47
Na ₂ O	0.30	-
K ₂ O	-	3.29
Loss on ignition (LOI)	1.30	3.81

*) Data provided from the company

**) Chemical composition determined by X-Ray Fluorescence Spectrometry method

Table 3 Mix compositions of UHPC used to study autogenous shrinkage

Mixture	w/b ratio (by weight)	Sand/binder ratio (by weight)	RHA (%, by weight)	The mean particle size of RHA (μm)
REF	0.18	1	0	
RHA20-3.6	0.18	1	20	3.6
RHA20-5.6	0.18	1	20	5.6
RHA20-9.0	0.18	1	20	9.0

Test method

In the experimental program the autogenous deformation, autogenous relative humidity changes and sorption isotherm were carried out.

Autogenous deformation measurement: The autogenous deformation of UHPC specimens was measured by dilation bench indicated in the ASTM C1698 standard developed by Jensen and Hansen (Jensen et al., 1995), in which three sealed corrugated moulds of 440 mm \times ϕ 28.5 mm were tested for each mixture [see Figure 2]. After preparing and mixing, the fresh UHPC paste was carefully filled into three sealed corrugated tubes. All samples and test instrument were kept in a thermostatically controlled room during the whole test. The temperature was maintained at 20 centigrade degree.



Figure 2. Dilatometer bench with accessories used to determine autogenous shrinkage

Relative humidity (RH) measurement system: The autogenous RH measurement system consists of two main parts (see Figure 3). The first part is the environmental chamber: due to high temperature sensitivity of the RH measurement, the whole test system has to be well temperature controlled. The second part is the moisture meter. Two Rotronic hygroscopic DT station equipped with HC2-AW measuring cells was used to measure the autogenous RH inside the UHPC. The whole instrument was put in the environmental chamber which was kept at target temperature with ± 0.1 °C accuracy during the test. The development of the RH in the samples and the temperature were continuously measured for a period of about 1 week after mixing. The RH sensors were calibrated by three saturated salt solutions in the range of 65-95% RH before and after every experiment. According to the calibration, the maximum measurement error of the RH sensors was $\pm 0.5\%$. In practice, autogenous RH of the mixtures with RHA always reached 100% within 24 h after mixing, even the temperature deviation of test environment was less than 0.2 °C. In order to avoid this problem, the RH measurement for the samples made with RHA addition started one day later. After mixing, the specimens were sealed for 24 hours and crushed quickly in a plastic box with 95% RH avoiding the loss of moisture, then transferred to RH measurement system.

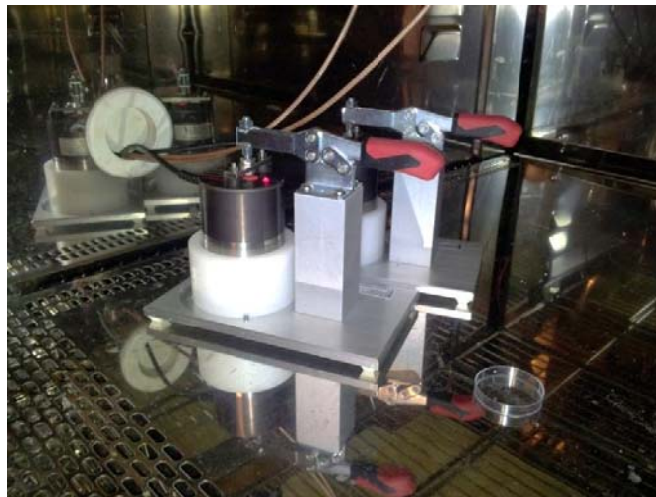


Figure 3. Internal RH measure system

Sorption isotherm: Sorption isotherm of RHAs was carried by TA Instrument, near 20 mg sample was tested for each RHA. During the test RH change ranged from 50% to 98%, temperature was 20 °C.

RESULTS AND DISCUSSION

Autogenous shrinkage of UHPC mixtures containing RHA

The test result of autogenous shrinkage of UHPC made with three different fineness RHA is depicted in Figure 4. Comparing to reference sample it is clear that the addition of 20% of RHA in UHPC reduce autogenous shrinkage effectively when the mean size of RHA particles ranges between 5.6 μm and 9.0 μm . The sample containing the RHA with a smaller particle size, *i.e.* 3.6 μm , shows a higher autogenous shrinkage contrast to 5.6 μm and 9.0 μm samples.

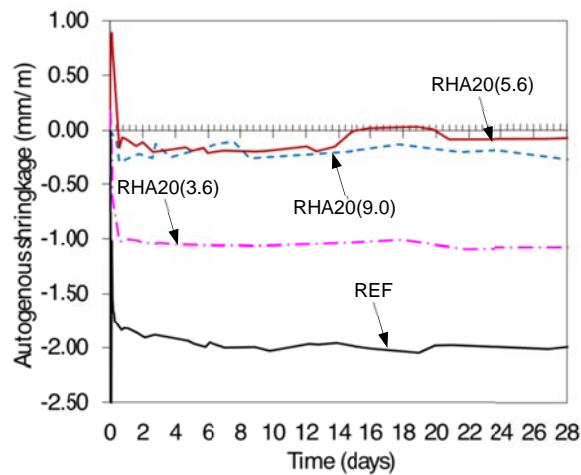


Figure 4. Autogenous shrinkage of UHPC mixtures containing RHA with different mean particle sizes measured from the final setting time, w/b ratio = 0.18 by weight

This result can be explained by the change of pore structure of RHA particles at some certain grinding degrees. In this study, the specific surface area of RHA increases in the size range from 5.6 μm to 9.0 μm but the total pore volume and the average width of pore decreases (Table 1). This means that the grinding process may only separate the particles through the big pores, which increases the surface outside of the RHA particles and does not significantly collapse the porous structure of RHA particles as illustrated in Figure 5.

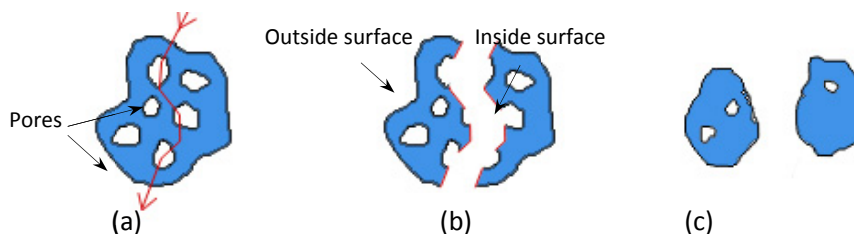


Figure 5. Different stages of grinding for the RHA particles: (a) original, (b) ground without collapsing the porous structure, and (c) ground with collapsing the porous structure

Therefore the positive effect of using RHA on mitigating autogenous shrinkage of UHPC is not decreased at this grinding degree. However, increasing the grinding to a certain degree will partly collapse the porous structure of RHA (see Figure 5c). This results in a reduction of specific surface area and total pore volume of RHA (see Table 1). This leads to a reduction of the volume of water containing in pores, or the positive effect of using RHA on internal curing is decreased and the higher autogenous shrinkage of UHPC using RHA with the mean particle size of 3.6 μm can be observed. This also can be observed from sorption isotherm. Figure 6 shows sorption isotherm of three types of RHA particle sizes. From the Figure 6, the RHA in different particle size showed nearly same trend on absorbing and desorbing water, this indicates that the grinding procedure doesn't break the major porous structure of RHAs. It is assumed that RHA absorbs water completely during mixing. When the RH decreased from 98% to 50%, the water inside RHA3.6 desorbed at 14% of mass of RHA, the water inside RHA5.6 and RHA9.0 desorbed at 15% of mass of RHA. The RHA which desorbed more water can keep the autogenous RH at higher value, thereby decrease the autogenous shrinkage. The RHA5.6 and RHA9.0 desorbed more water than RHA3.6, which showed the same relative trend in autogenous RH and autogenous shrinkage test results, which will be discussed in more detail in the following section.

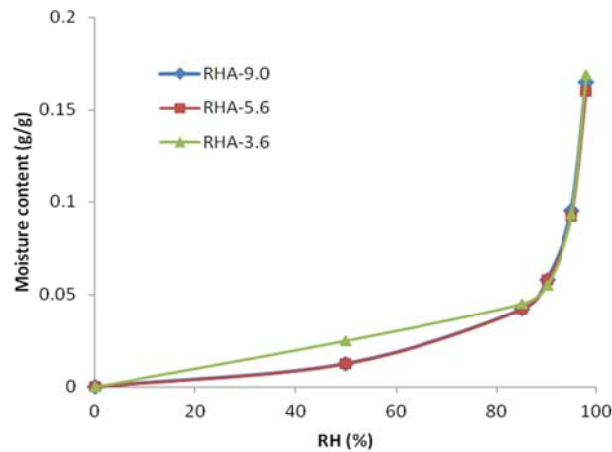


Figure 6. Sorption isotherm of different particle size of RHA

Internal relative humidity of UHPC incorporating RHA

Figure 7 shows the experimental results of internal relative humidity of UHPC samples incorporating RHA in comparison with that of reference sample in the test period of one week. For the reference sample, the RH measurement started from 12 hours after mixing, whereas for RHA samples, the RH measurement starts from 24 hours after mixing. Because in the very beginning the RH value are higher than 99% in the sample, this value is beyond the range of Rotronic hygrosopic DT station. From Figure 7, it can be seen that the development of internal RH of the samples incorporating RHA with mean particle size of 9.0 μm and 5.6 μm is similar. When the mean particle size of RHA is smaller than 5.6 μm , i.e. 3.6 μm , the internal RH values is lower. However the internal RH values of the RHA samples are much higher than those of reference sample.

The above results show significant high of RH value in the samples incorporating RHA. These results could explain the positive effect of using RHA on mitigating autogenous shrinkage of UHPC.

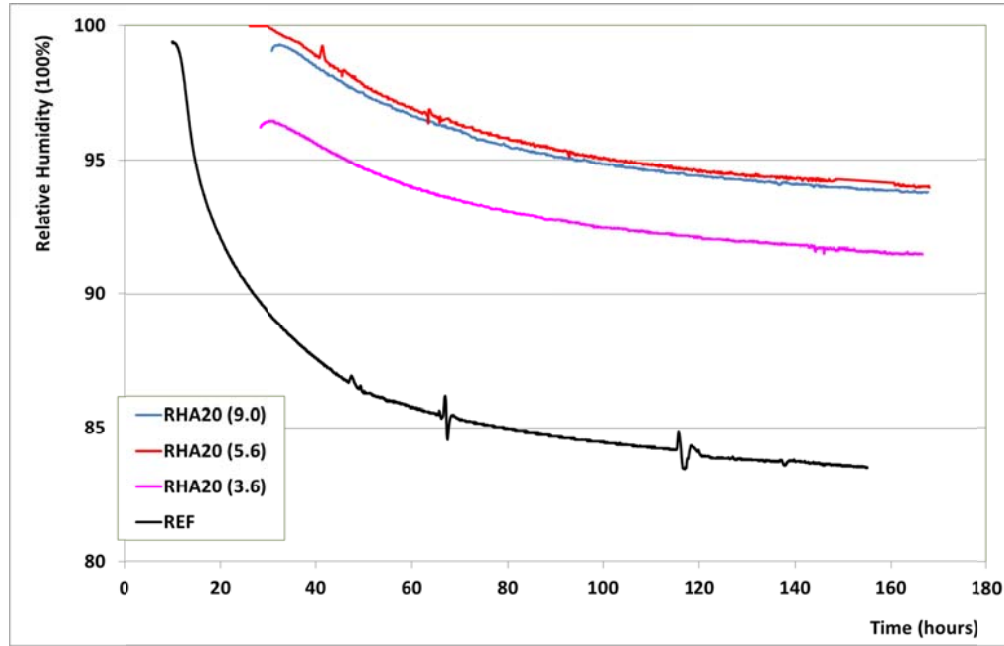


Figure 7. Internal relative humidity of cement paste incorporating RHA with d_{meanRHA} from 3.6 μm to 9.0 μm , w/b ratio = 0.18 by weight

In one hand, the specific pore volume of ground RHA with a mean particle size of 5.6 μm is approximately 0.086 cm^3/g . It is assumed that the water will fill all these pores. Theoretically because 20% RHA was added corresponding to about 220 $\text{kg RHA}/\text{m}^3$ concrete, the total absorbed water by RHA is about 18.9 l/m^3 UHPC. The extra water for internal curing of UHPC by super-absorbent polymers (SAP) ranges from 33 - 64 l/m^3 UHPC (Mechtcherine et al., 2008). Thus, the absorbed water in RHA in UHPC is also significant. In addition, the distribution of water reservoirs also plays an important role for internal curing because it relates to the effective distance for transporting water to the vicinity of these water reservoirs (Kovler et al., 2007). With a lower w/b ratio in particular for UHPC, the microstructure of cement paste is very dense which constrains the water from reservoirs to migrate to the surroundings. This means that the water reservoirs should be separated into smaller ones to enhance the effectiveness of using internal curing agents in the system. In this respect, RHA is much better than SAPs.

In other hand, the effectiveness of water released from inside of the pores of RHA particles to the surrounding cement matrix depends on the suction force, which in turn depends on the porosity and RH in cement paste (Kovler et al., 2007). From the development of RH in UHPC samples as shown in Figure 7, according to Kelvin's equation (Lura et al., 2003), the corresponding pore size (r , in nm) from which the water can be hold in the pores can be deduced:

$$r = -\frac{2\gamma V_m}{\ln(\text{RH}\%) \cdot RT}$$

Where, γ is surface tension, 0.055 N/m for pore fluid of cementitious materials, V_m is the molar volume of the liquid, for cementitious materials, $1.8 \times 10^{-5} \text{ m}^3/\text{mol}$, R is the universal gas constant, 8.3144 $\text{J}/\text{mol} \cdot \text{K}$, T is the temperature, 293.15 K.

The pore radius from which the pore water can be hold in the pores of four samples as function of curing age is shown in Figure 8.

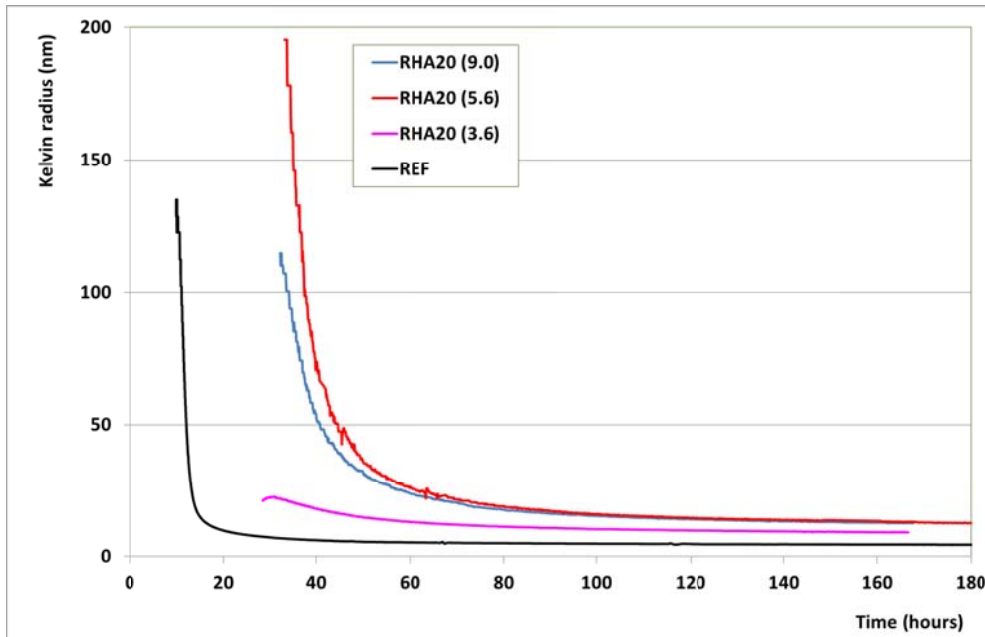


Figure 8. Pore radius from which the pore water can be hold in the pores of four samples

From Figure 7 and Figure 8, at 168 hours, the RH in the UHPC pastes with average particle size 9.0 μm , 5.6 μm and 3.6 μm are 93.99%, 94.00% and 91.51%, respectively, corresponding to the Kelvin pore radius of 13.14 nm, 13.12 nm and 9.19 nm. It means only water in the pores above those value of RHA particles can be released in the first week of hydration. Previous measurement of pore size distribution of RHA, for example, the pore size distribution of RHA with mean particle size of 5.6 μm is shown in Figure 9 (Tuan, 2011). It is clear that the pore size inside RHA is almost smaller than 100 nm. According to Figure 8, the water inside the RHA will be hold in all pores in the first 40 hours of hydration. After about 1 week hydration, the pore with radius of 13.12 nm still hold the pore water inside the RHA. According to Figure 9, a certain amount of pores is smaller than 13.12 nm, where the water remains in the smaller pores and can be gradually released at later ages, even after 28 days.

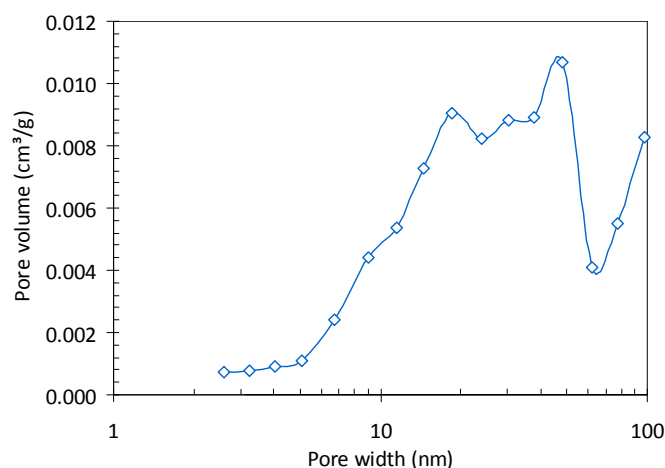


Figure 9. Pore size distribution of the RHA powder, $d_{\text{RHAmean}} = 5.6 \mu\text{m}$ (Tuan, 2011)

CONCLUSION

This paper studied the influence of particle size of RHA on the efficiency of mitigation of autogenous shrinkage and the water movement in the internal pore structure of RHA. Following conclusion are drawn:

1. The addition of 20% of RHA in UHPC reduce autogenous shrinkage effectively when the mean size of RHA particles ranges between 5.6 μm and 9.0 μm . The sample containing the RHA with a smaller particle size, *i.e.* 3.6 μm , shows a higher autogenous shrinkage contrast to 5.6 μm and 9.0 μm samples.
2. When the mean particle size of RHA is smaller than 5.6 μm , *i.e.* 3.6 μm , the internal RHA values decrease. However the values of internal RH values is much higher than those of reference sample.
3. The specific surface area, internal pore size of RHA are main reason to hold the water in the small pore at early age hydration. 20% of RHA is enough to absorb enough water to mitigate the autogenous shrinkage caused by self-desiccation.

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