- Technical Paper -

INTERNAL RELATIVE HUMIDITY MEASUREMENT ON MOISTURE DISTRIBUTION OF MORTAR CONSIDERING SELF-DESSICATION AT EARLY AGES

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ABSTRACT

Using Blast Furnace Slag (BFS) with low water-binder ratio in concrete mixture to improve the performance of concrete can cause early-age cracks due to autogeneous shrinkage. In this study, the internal relative humidity (IRH) of mortar under ambient condition was measured by using new proposed method to study the effect of water-binder ratio and BFS in mixture on moisture loss. Decrement of IRH in mortar specimens at low water-binder ratio depends on moisture diffusion and self desiccation. Using BFS in mortar mixture leads lower IRH compare to pure Portland cement. Keywords: autogeneous shrinkage, internal relative humidity, water-binder ratio, blast furnace slag

1. INTRODUCTION

Physical and chemical properties of concrete always change with time depending on moisture, curing, environment and etc. These material properties such as concrete strength, elastic modulus, creep, and shrinkage are significantly influenced by heat of hydration and moisture content in concrete at early ages. Consequently, for that reason it is necessary to know the behavior of moisture distribution in concrete structures under arbitrary ambient condition. Internal relative humidity (IRH) is an effective parameter to assess the moisture condition in concrete. It also relates to moisture content to shrinkage strain

When there is no moisture movement allowed with the environment, shrinkage in concrete at early ages will occur due to cement hydration. Water consumption of un-hydrated cement results relative humidity in the concrete pores (IRH) reduced. This phenomenon is known as self-desiccation. It causes volume change called autogeneous shrinkage [1]. This deformation may cause micro cracking due to restrained auotogeneous shrinkage. It may form macro-cracks after connecting into continuous crack pattern [2]. This is a serious matter regarding durability, aesthetics and strength as well. Problems such as autogeneous shrinkage and change of IRH have been a great concern on using high performance concrete with low water-binder ratio.

In practice, measuring IRH at early ages is a tough work because of its accuracy. From saturated condition to dry condition, measurable reduction in IRH will only occur after pores with radii of 50 to 100 nm have been emptied [3]. IRH measurement also is necessary to indicate the performance of curing method

when weight loss is impossible to be measured in practical. Due to some disadvantages and toughness from existing method in measuring IRH, a new method of measurement is proposed.

In addition, experimenting durability of concrete structure in long span (10-100 years) is not practical. Consequently, simulation program will be useful to evaluate durability performances over the whole service life since cement comes into contact with water. A thermodynamics oriented model [8] so-called DuCOM has been developed in the author's research group According to this computational system, microphysical properties of cementitious materials can be precisely predicted. In this research, DuCOM can compare experiment results to analytical results.

2. IRH MEASUREMENT

There are many types of hygrometers for relative humidity measurement. Chilled mirror, resistive and capacitive sensors seem to be most popular in concrete research. In this research capacitive sensors were adopted. The capacitive sensor used for IRH needs proposed by Z.C. Grasley [3]. It has accuracy $\pm 1.8\%$ for RH and $\pm 0.3^{\circ}$ C for temperature. It is not simple to measure IRH, because to get a reliable reading value, it is necessary to wait until the probe and surrounding concrete pore vapor are stable. The sensor includes a capacitive polymer-sensing element for relative humidity and a band-gap temperature sensor is used in this research (Figure 1). This

In trial experiment based on existing method proposed by Grasley [4], the sensor and connector were wrapped using waterproof/breathable fabric made from polytetrafluoroethylene (PTFE) and sealed with

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silicone. This fabric pouch includes adhesive material leading permanently seal. It is necessary to wait for stabilization of relative humidity and temperature reading. Therefore the sensor covered with fabric pouch was soaked in water to obtain 100% of relative humidity before embedding in fresh mortar. After that, the sensor was set in the center of cylindrical specimen and fixed by a small wire as shown in Figure 2. Finally, an adhesive-backed aluminum foil was used as sealing material at the top surface of mold in order to prevent any evaporation of moisture to the surrounding.



Fig 2 Sensor in fabric pouch embedded in mortar.

However, using of this existing method in the trial experiment showed unreasonable result when it is used at curing temperature of 60° C or if cement is partially replaced with BFS. The observed IRH was over 100% up to 120% during measurement especially when curing specimens at high temperature even if cement only is used as shown in Figure 3a, 3b and 3c. It means no physical meaning for the reading or malfunction of the sensors. Table 1 show the summary of results from existing method. Error readings are shown by IRH measurement over 100% and marked by 'failed'.



(a) Mixture using BFS at 20°C



Temperature 60°C 140 120 > 100% 100 Internal RH (%) 80 Sealed 60 W/C 25%, BFS 40%, RH 60% 10 EXP: WC25 Cement 20 EXP: WC25 CBES 10 15 25 30 Age (Day)

(c) Cement and BFS mortar at 60°C Fig 3 Result of internal relative humidity from trial experiment.

| Tahle | 1. | Summary | ⁄ ∩f | results | hv | evisting | method |
|-------|----|---------|------|---------|-----|----------|----------|
| Iavic | | Summan | 0 | ICSUIIS | DV. | CAISUIN | IIICUIUU |

| Temperature (°C) | Pure cement mortar | BFS-cement mortar |
|------------------|-----------------------|----------------------|
| 20 | OK | Failed |
| 40 | OK | Failed |
| 60 | Failed | Failed |

From this reason, a cause of problem was then investigated considering usability of the sensor and housing for sensor. This issue is avoided by putting the sensor into concrete after it set. Because concrete is quite dry, no need to protect the sensor from water in setting stage.

The author proposed a method to insert relative humidity sensor directly into the measuring point after the concrete is sufficiently hardened by providing inserting path (pre-embedded pipe) before casting. This proposed method will be discussed later in 3.1.

3. EXPERIMENT WORK

3.1 Proposed Method in Measuring IRH

(1) Pipe and Molds Preparation

Cylindrical molds with diameter 10 cm and 20 cm in height are used for IRH measurement. Acrylic pipe with inner diameter of 11 mm, is fixed by two small wires at the center of the mold. Rubber is inserted through the pipe until an end to prevent concrete flow into the pipe during casting as shown in Figure 4. (2) Inserting Sensor

The sensor is sealed surrounding also by rubber to prevent drying of the measuring point as shown in







Fig 5 Rubber surrounding the sensor

After 2 to 6 hours, depends on setting time of specimens, the rubber is replaced by relative humidity sensor immediately to not disturb moisture equilibrium inside the pipe. After that, silicone is filled in the empty space to make sure that vapor from concrete cannot escape during measurement. Then, measurement can be monitored as shown in Figure 6.



Fig 6 IRH measurement setting

Finally, reasonable result of all cases in both using of blast furnace slag and at high curing temperature can be obtained. The summary of results from the proposed method is shown in Table 2.

| Table 2: | Summar | y of | proposed | method. |
|----------|--------|------|----------|---------|
| | | | | |

| Temperature (°C) | Pure cement | BFS-cement | |
|------------------|-------------|------------|--|
| (c) | mortar | mortar | |
| 20 | OK | OK | |
| 40 | OK | OK | |
| 60 | OK | OK | |

(2) Material and mix proportion

Mortar specimens with water-binder ratio of 25% and 50% were prepared as shown in Table 3. BFS is used about 40% replacements by cement weight. In order to improve its workability, a water-Reducing Agents Rheobuild SP8SBs was employed especially in the mix with low water-binder ratio. River sand was used as fine aggregate with 50% by volume. Table 4 shows an accurate chemical composition of the special cement for research. Physical properties of the cement and BFS are given in Table 5.

| Table 3: Mix | proportions | of mortar | specimens. |
|--------------|-------------|-----------|------------|
| | | | |

| W/C | Water * | С * | Sand * | SP * | | |
|---------------|---------|-------|--------|------|--|--|
| 25 | 191.5 | 804.7 | 1315 | 9.7 | | |
| 50 | 278.9 | 557.7 | 1315 | - | | |
| * Unit: kg/m3 | | | | | | |

| W/B | Water * | С * | BFS * | Sand * | SP * | |
|---------------|---------|-------|-------|--------|------|--|
| 25 | 187.2 | 471.9 | 314.9 | 1315 | 9.4 | |
| 50 | 274.4 | 329.3 | 219.6 | 1315 | - | |
| * Unit: ka/m2 | | | | | | |

* Unit: kg/m3

Table 4: Chemical composition of cement

| Oxide | SiO ₂ | Al ₂ O ₃ | Fe ₂ O ₃ | CaO | MgO | SO ₃ |
|------------|------------------|--------------------------------|--------------------------------|-------|-------|-----------------|
| Cement for | 21.0% | 5.41% | 2.77% | 64.7% | 1.74% | 2.09% |
| research | | | | | | |

Table 5: Physical properties of cement and BFS

| | 7 | | | - |
|------------|-----------------|--------|---------|------|
| | Density (g/cm3) | Blaine | surface | area |
| | (cm2/g) | | | |
| Cement for | 3.18 | 3 | 340 | |
| research | | | | |
| BFS | 2.88 | 4 | 100 | |

(3) Experiment method

For measuring IRH, both sealed mortar and drying specimens were adopted. To measure the variation of IRH due to only self-desiccation, the specimens were sealed in the mold since just after casting. An adhesive tape aluminum foil was used to seal the top surface of specimens. One day after casting, the specimens for drying experiment were de-molded and let evaporation from all surfaces occur. Both specimens for sealed and drying condition were controlled at various temperatures at constant humidity of 60%. The detail of experiment variables is shown in Figure 7 and 8. For each condition, two identical specimens were tested.



Fig 7 Test variables at normal temperature



Fig 8 Test variables at high temperature

3.2. Double Measurement

The issue to find a reliable measurement on IRH makes clear, why it is difficult to obtain extremely accurate relative humidity measurements. Here, a modification is proposed to get a higher accuracy measurement by inserting two sensors in the center of mold. An average value will be obtained from two readings. By using this method, step 1 and 2 in Figure 5 can be disregarded. Sensors are soaked in the water to reach initial saturated condition at least one day before inserting. Then, after setting time, sensors are slid inside acrylic pipes, which are covered by fabric cap. It will allow vapor from mortar but prevent the hydration products reaching the sensors during hydration process. This proposed method will contribute more accurate information and avoid failed measurement if only one sensor is used.



Fig 9 IRH measurement using double sensors

4. EXPERIMENT RESULTS AND DISCUSSION

4.1 Internal Relative Humidity (IRH)

(1) The effect of water-binder ratio and curing

IRH measurement result in specimen due to self-desiccation is shown in Figure 12. In higher water-cement ratio, the pores were fully saturated at the start of the experiment. This trend is similar to experiment results of previous research by Kim and Lee [5]. As seen from Figure 12, IRH of sealed cement mortar with water-binder ratio of 25% decreases to 72% at 60 days (Figure 12a). However, at higher water-cement ratio, it only decreases down to 95% (Figure 12b). It indicates that more IRH decreased by self-desiccation as decrease of water-binder ratio, especially at early age. The lower water-binder ratio is, the higher the self-desiccation results to impact decreasing of IRH of cementitious material. In addition, the difference in IRH with drying time due only to moisture diffusion was small in mortar with low water-to-binder ratio. This is due to the fact that the dense pore structure of low water-to-binder mortar decreases the rate of moisture diffusion, but increases self-desiccation.



(a) Water-binder ratio = 25%



(b) Water-binder ratio = 50% Fig 12 IRH of sealed and dry cement mortar.



Fig 13 IRH of experiment result compared to analysis result by DuCOM

The analytical results of relative humidity distribution of mortar specimen obtained from experimental results were then compared by analytical result by using DuCOM. It is an integrated computational system designed based on physical and thermodynamics theories to evaluate durability performances since cement comes into contact with water. DuCOM is versatile for not only the commonly used concrete but also other types of concrete such as fly ash, blast furnace slag concretes, and etc. Experiments verified that this computational system can accurately predict hydration processes and relative humidity for different mix proportions under various temperatures [10-13]. The input data used in the analytical program such as the geometric conditions, mix proportions, and the atmospheric condition are the same as those used in the experiment. According to the comparison shown in Figure 13, the experiment result agrees with the analytical result.

(2) The effect of Blast Furnace Slag (BFS)

In Figure 14, at the same water-binder ratio given, IRH decrement of cement mortar incorporated with 40% BFS is higher than that of pure cement at the same ages. The reason that higher chemical shrinkage of the BFS cement might lead to greater self desiccation since there is no water supply under sealed curing. If the water supply is restricted, the pores empty and air-water menisci form [6]. Here, moisture consumption is faster than that in pure cement mortar.

In addition, pozzolanic effect of blast furnace slag mineral admixture accelerates slag hydration at early ages, so moisture in capillary pores of the paste was consumed greatly. The early consumption of Calcium Hydroxide during hydration reaction may induce higher reactivity of slag [7].

Fineness of BFS is also considered as mechanical accelerator [8, 9]. As BFS has finer particles, which has larger surface area, it increases both the pozzolanic reaction and the water consumption. It reduces pore sizes in cementitious matrix. This finer structure which is the product of the dissolution of the Calcium Hydroxide crystals and the precipitation of CSH produced by the pozzolanic reaction may cause the pores filled up. Those finer pores lead IRH decrease very fast.









(3) The effect of temperature

Here, IRH at high temperatures (40 and 60 degree Celsius) is considered (Figure 15). The behavior of IRH is significantly different from that at normal temperature (20°C). It can be clearly seen that IRH of specimens with sealed condition at the higher temperatures (40°C and 60°C) decrease due to hydration reaction in an early age, since it promotes as curing temperature rises. In fact, at the highest curing temperature of 60°C, at particular time, IRH gradually increases by time.

The increment of IRH at later age can be explained by inkbottle effect. Water trapped in the inkbottle pores is gradually released under sealed conditions at high temperature. The vapor can be redistributed to other pores as condensed water. From this reasons, internal relative humidity rises again at elevated temperature [10-12].





Fig 15 Experiment result: sealed specimen under various temperatures



(a) Pure cement mortar



(b) BFS mortar Fig 16 Analysis result by DuCOM: sealed specimen under various temperatures.

Ishida et al. [10] has proposed a new moisture model in which the rates of transport and dispersion of water trapped by inkbottle effect are strongly dependent on temperature. The interdependence among hydration process under sealed condition, temperatures and internal relative humidity of pores, and the moisture state inside the material were focused on.

Figure 16 shows analytical result by simulation model by DuCOM. This interesting phenomenon is agreed with author's experiments. The model-simulated changes of internal relative humidity reasonably well despite there are small differences between experiment and simulation results in the periods of increase and decrease.

5. CONCLUSIONS

- (1) Accurately measuring the internal relative humidity plays an important role to device more information about moisture distribution in cementitious material. It is not an easy work but advances in sensor technology are rapidly improving the available techniques. Due to many disadvantages and some toughness occurred from the existing methods, a new method for internal relative humidity measurement could show good results.
- (2) Internal relative humidity (IRH) mainly decreased with time due to self-desiccation and moisture diffusion. IRH in mortar specimens with high water-binder ratio was mainly affected by moisture diffusion. In contrast, for mortar with low water-binder ratio, its internal relative humidity decrement depends not only on moisture diffusion but also on self-desiccation. This experiment result also shows a good agreement with analytical result by DuCOM.
- (3) When partially replaced cement by blast furnace slag, the results showed lower internal relative humidity than that in Portland cement mortar.
- (4) The IRH of mortar with sealed condition at high temperature decreased due to hydration reaction in an early age, and then gradually increased due to the release of inkbottle water.

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