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Refereed Proceedings

Summer 6-24-2014

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Recommended Citation

Qier Wu, Thomas Rougelot, Nicolas Burlion, and Xavier Bourbon, "Experimental study of water retention properties of a high performance concrete with small sliced samples" in "5th International Conference on Porous Media and Their Applications in Science, Engineering and Industry", Prof. Kambiz Vafai, University of California, Riverside; Prof. Adrian Bejan, Duke University; Prof. Akira Nakayama, Shizuoka University; Prof. Oronzio Manca, Seconda Università degli Studi Napoli Eds, ECI Symposium Series, (2014). http://dc.engconfintl.org/porous_media_V/57

Proceedings of the 5th International Conference on Porous Media and its Applications in Science and Engineering
ICPM5
June 22-27, 2014, Kona, Hawaii

EXPERIMENTAL STUDY OF WATER RETENTION PROPERTIES OF A HIGH PERFORMANCE CONCRETE WITH SMALL SLICED SAMPLES

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ABSTRACT

The aim of present study is to develop a practicable accelerated experimental method to obtain desorption isotherm of concrete more quickly and more reliably. A large number of sliced samples with thickness of 1, 2 and 3 mm have been de-saturated from fully water-saturated state to dry state. Time needed to obtain a mass stabilization varies from 14 to 30 days for concrete CEM I at a given relative humidity. Time saving is remarkable especially at lower relative humidity. Thinner samples lose their masses more quickly in the first week, but the thickness has hardly any influence on the total relative mass loss once the mass stabilization is reached. The analysis of images obtained by X-ray microtomography shows that water content and relative mass loss between two successive relative humidity steps have a nearly linear relationship with aggregate volume fraction except for very high relative humidity, which may be attributed to the difference between volume fraction of interfacial transition zone in different slices.

INTRODUCTION

French Radioactive Waste Management Agency (Andra) is responsible for the design and assessment of the feasibility and the safety of the long-term nuclear waste repository in France. Some concretes have been designed for containers and tunnels which are built for the disposal of radioactive wastes. In order to protect the environment, the durability of these structures should be firstly taken into account to choose the concretes. However, the durability is mainly determined by combined effects of various physical and chemical processes which are dependent on the moisture. Therefore, the assessment of moisture properties of concrete is inevitably required to predict and improve the durability of concrete structures.

Various methods can be used to conduct water vapor desorption experiments. Some researchers carried out water vapor desorption and experiments by means of the saturated salt solution method [1-3]. In order to limit the effect of significant variability among different samples, a better method to obtain desorption isotherm of concrete consists in submitting the same set of initially fully water-saturated samples to decreasing relative humidity steps and waiting for the mass stabilization for each relative humidity till the reference dry state. However, this method is highly time-consuming since the time needed to reach equilibrium is very long when the experiment is performed with large size samples. Poyet [1] performed water desorption experiment for a concrete based on CEM I cement with cylindrical samples (diameter 40 mm, height 40 mm) spanned over 1200 days at 30°C. The results show that time needed to reach 90% of mass loss is more than 1 year. In the same laboratory, a water desorption experiment [4] with cylindrical samples (diameter 37.5 mm, height 50 mm) in the climatic chamber took more than five years till the relative humidity of 30%. In order to reduce the experiment duration, most of the researchers chose to follow different set of samples for each relative humidity. In the meanwhile, small samples are used in some experiments, such as powder samples [5], sliced samples [6], sawn or crushed samples [7, 8].

The present study focuses on developing an accelerated experimental method to obtain desorption isotherm of concrete more quickly and more reliably by following the same samples along the whole de-saturation process. A large number of sliced samples with thickness of 1, 2 and 3 mm of a concrete based on CEM I cement have been de-saturated from fully saturated state till the reference dry state to ensure that experimental results can be statistically considered representative for the concrete. The results are compared with former studies on the same concrete from different batches to validate the proposed accelerated experimental method. An analysis of images obtained by X-ray microtomography has been carried out to study the influence of aggregate volume fraction on relative mass loss and water content.

NOMENCLATURE

RH = Relative humidity
T = Temperature
$m(RH)$ = Stabilized mass at a given RH
m_{dry} = Dry sample mass
m_{sat} = Fully water-saturated sample mass
$w(RH)$ = Water content at a given RH
$S_w(RH)$ = Water saturation degree at a given RH
d^i = Smallest drained pore diameter
$\gamma_w(T)$ = Surface tension of water/air at T
M_w = Water molar mass
R = Perfect gas constant
$\rho_w(T)$ = Water specific mass at T
r = Pore repartition coefficient

1 Experimental approach

1.1 Materials

The concrete studied here is considered as ‘a reference concrete’ (denoted as ‘CEM I’) in the framework of Andra’s radioactive waste management. It is based on CEM I cement and the water-to-cement ratio (*W/C*) is equal to 0.43. The formulation is presented in Table 1.

Table 1: CEM I concrete composition

Component	Nature	Quantity (kg/m3)
Cement	CEM I 52.5 R	400
Sand	Limestone [0-4mm]	858
Gravel	Limestone [5-12mm]	945
Super-plasticizer	Glenium 27	10
Water	-	171
W/C	0.43	

1.2 Sample preparation

The concrete is poured into beams with a section of 14×14 cm^2 and a length of 56 cm after mixing. Each beam is made from the same batch. Plastic sheets are used to cover the upper beam surface in order to avoid early desiccation. After unmolding at 5 days, the concrete beams are cured in lime-saturated water at 20°C for 4 months. Concrete cylinders (diameter 37.5 mm) are cored from a single beam and then are cured again in lime-saturated water at 20°C. After two months of maturation, 2 cylinders numbered BI 1 and BI 2 are sawed into slices with a thickness of 3 mm (20 slices for each cylinder), 2 cylinders numbered BI 3 and BI 5 are sawed into 2 mm slices (30 slices for each cylinder) and 3 cylinders BI 6, BI 7 and BI 8 are sawed into 1 mm slices (40 slices for each cylinder). All the sliced samples are numbered and marked by the cylinder number and the position number. For example, ‘I1 5’ means that the slice is the 5-th sawed piece from cylinder BI 1. All these 220 slices are cured again in lime-saturated water at 20°C for 4 months till the start of experiment to ensure a fully water-saturated state.

1.3 Water desorption experiment

Water desorption experiment is carried out at a constant temperature of 20°C. A range of relative humidity is imposed successively with values *RH*= [95%; 90%; 80%; 65%; 50%; 30%; 12%]. Both temperature and relative humidity are controlled by a climatic chamber.

Nylon boxes with 10 compartments are manufactured to keep the sliced samples. Every time a box of 10 slices is taken out for mass monitoring. Each slice will not be exposed to the surrounding air for more than 1.5 minute during the mass measurement, which can limit the carbonation of slices caused by atmospheric CO_2 [9] and additional mass variation. A continuous injection of nitrogen into the climatic chamber is maintained during the experiment, and additional nitrogen is added into the climatic chamber after each series of measurement to prevent the carbonation.

Sample mass monitoring is carried out under controlled conditions using a balance with a resolution of 0.1 mg. When a new *RH* is imposed, the sample mass is recorded after 1 day, 4 days, 7 days then every 7 days till mass stabilization. Mass stabilization is considered to be reached once the sample mass does not vary by more than 0.02% between three consecutive measurements. Water content *w* (%) of a given *RH* is defined as:

$$w(RH) = \frac{m(RH) - m_{dry}}{m_{dry}} \tag{1}$$

The dry state is obtained by an oven-drying at 105°C until the sample masses remain constant. The water saturation degree at a given *RH* is determined as:

$$S_w(RH) = \frac{m(RH) - m_{dry}}{m_{sat} - m_{dry}} \tag{2}$$

The fully water-saturated sample mass is measured after surface drying with clean tissues at 20°C.

1.4 Image collection and reconstruction

Some sliced samples are scanned by an Ultratom X-ray microtomography at 80 kV and 300 µA. The size of each voxel of the reconstructed volume is 24×24×24 µm. After image reconstruction with X-act, the images are processed with ImageJ to estimate the aggregate volume fraction of sliced samples and to compare with mass loss and water content. The volumes of aggregates and cement matrix are measured by counting the number of voxels associated with each phase. The volume fraction of interfacial transition zone is estimated by the total perimeter of aggregates.

2 Experimental results

2.1 Mass variation kinetics

Figure 1 presents the average relative mass variation of concrete CEM I for three different slice thicknesses.

Generally, time needed to reach the mass stabilization at each relative humidity varies from 14 to 30 days. Since the former experimental results of concrete CEM I show that it takes more time to reach the mass stabilization at lower relative humidity, the samples had been kept at RH=50% and 30% for three months in spite of the seemingly obvious presence of mass stabilization after 30 days. No significant mass loss can be observed during the two other additional months.

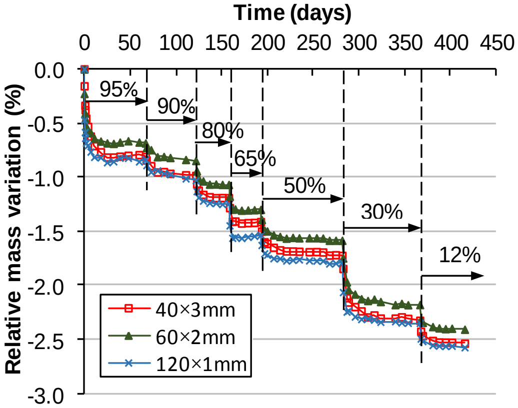

Figure 1: Drying kinetics for three different thicknesses

It is noted that the mass loss kinetics is more important for the thinner samples in the first days. Surface effect may be responsible for the strong mass loss of sliced samples in the first days. The thinner the thickness is, the more important the surface effect plays a role. However, the difference in relative mass loss rates reduces quickly with time and does not exist any longer after a few days. The thickness has hardly any influence on the total relative mass loss for a given relative humidity once the mass stabilization is reached, confirming by the fact that the 3 mm sliced samples have a larger relative mass variation than 2 mm sliced samples while have a smaller relative mass variation than 1 mm sliced samples. A strong mass loss can be observed at high relative humidity 95%, which could be attributed to a percolating pore network which drains very quickly.

2.2 Comparison with mass variation kinetics obtained by Brue et al. [4]

The aim of present study is to develop an accelerated experimental method to obtain desorption isotherm more quickly and more reliably by following the same samples along its whole de-saturation process. Since Brue et al. [4] determined the desorption isotherms of concrete CEM I with the same formulation by following the same samples along its whole de-saturation process in the climatic chamber, similar to the present study, the mass variation with relative humidity is compared to their results.

Figure 2 presents the comparison of relative mass variations after 21 days, 35 days and 63 days for each relative humidity step. The relative mass loss amplitude is greater in present study. No obvious mass loss can be observed between 21 days result and 35 days result in the present study. The mass almost does not change any more after 35 days for lower relative humidity 30% and 50% in present study, which is proved by the approximate overlap of the curves between 35 days and 63 days.

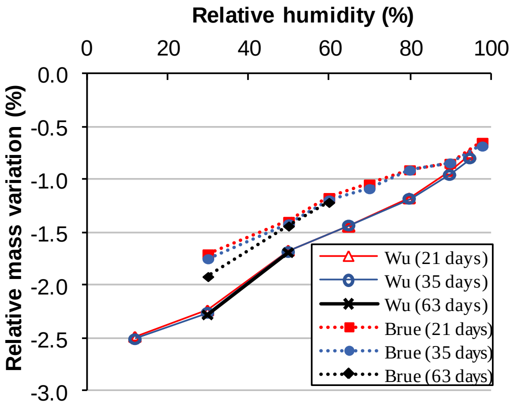

Figure 2: Comparison of relative mass variation with Brue et al. [4]

As to the mass variation in [4], difference between 21 days and 35 days results is noticeable for lower relative humidity. The additional mass loss of 63 days at *RH*=30% nearly doubles that of 35 days. In fact, more than 1 year is spent on obtaining the mass stabilization at *RH*=30% in their experiment.

It can be concluded that, as expected, time saving is remarkable by using the present experimental method, especially for lower relative humidity.

3 Analysis and discussion

3.1 First desorption isotherm

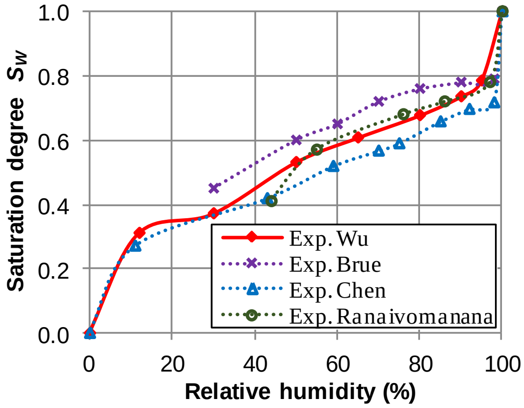

Figure 3: Comparison of first desorption isotherms of CEM I concrete with other different batches [2-4]

The first desorption isotherm obtained in the present study is compared to those of Ranavaivomanana et al. [3], Brue et al. [4] and Chen et al. [2] in Figure 3. Their desorption experiments were down to the relative humidity of 44%, 30% and 12%, respectively. The samples used in their studies are sliced samples (70 mm×35 mm×5 mm), cylindrical samples (diameter 37.5 mm, height 50 mm) and cylindrical samples (diameter 37.5 mm, height 30 mm), respectively.

A strong fall of isotherm at high relative humidity can be observed for all the presented isotherm curves, which means that it is independent of the concrete batch and the sample size. It could be attributed to a percolating pore network that drains very quickly when relative humidity decreases. The slope of isotherm in the relative humidity range [12%, 30%] is very slight in the present study, which indicates strong water retention ability associated with C-S-H porosity in this relative humidity range. Till RH=55%, the isotherm of fully saturated sliced samples is very close to that of Ranaivomanana et al. [3] which is also obtained by sliced samples with a thickness of 5 mm and cured after manufacturing. However, their isotherm shows a much bigger slope between RH=55% and 44%. Since their study was performed with different samples for each relative humidity and no further desorption experiment for other relative humidity between RH=50% and 30% has been investigated in the present study, the difference may be attributed to the variability of samples in their study so far. The water saturation degree is always higher than the others for the isotherm of Brue et al. [4]. However, the samples used in their study could be considered partially water-saturated, compared to the present study. The isotherm of Chen et al. [2] is always located under the others and no obvious decrease in slope could be observed for lower relative humidity range [12%, 30%], which may also be attributed to the variability of concrete samples since they also followed different samples for each relative humidity.

3.2 Pore structure investigation

CEM I concrete porosity obtained after a 105°C oven-drying in the present study is 9.3%, which is smaller than 10.1% obtained by Brue et al. [4], 12.3% by Ranaivomanana et al. [3] and 12.3% by Poyet [1] . Although all the porosity identifications are based on the concrete with the same formulation, differences in sample manufacturing and curing may lead to significant divergence in pore microstructure of mature concretes.

Pore microstructure of concrete CEM I was identified by MIP (mercury intrusion porosimetry) in the study of Brue et al. [4]. Three distinct peaks in pore size distribution can be observed: the first one is located between 1 and 2 µm, which can be attributed to large capillary pores, entrapped air bubbles and/or to micro-cracks due to oven-drying; the second one is centered around 200 nm, which is smaller and corresponds to capillary pores; the greatest, the third peak is centered around 30 nm. In fact, the lowest accessible diameter of MIP is 6 nm in their experiment, so interlayer C-S-H pores (2-5 nm) are not observed. The bi-modal pore distribution obtained by MIP after desorption at 30°C of Poyet [1] corresponds to C-S-H nanoporosity (around 3 nm) and capillary porosity (around 70 nm).

The capillary theory [10] is based on the analysis of water chemical potential. Kelvin-Laplace's equation applied to water allows calculate the smallest drained pore diameter d^i at a given temperature and humidity conditions, as:

$$d^i = -\frac{4\gamma_w(T)M_w}{RT\rho_w(T)\ln(RH)} \tag{3}$$

A simple pore network model [11] is proposed to describe the pore repartition. The pore repartition coefficient r is calculated as follow,

$$r(d^{i+1}) = \frac{S_w(d^i) - S_w(d^{i+1})}{\log(d^i) - \log(d^{i+1})} \tag{4}$$

Pore repartition coefficient r can be interpreted as the mass loss between RH^i and RH^{i+1} ($RH^{i+1} < RH^i$), related to a decrease of smallest drained pore diameter form d^i to d^{i+1}.

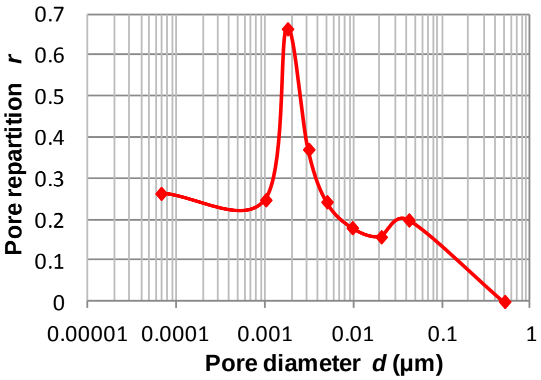

Figure 4: Pore repartition coefficient vs. pore diameter

Pore diameter at RH=100% is arbitrarily chosen as 500 nm since Kelvin-Laplace's equation is not defined at RH=100%. According to the MIP result of Brue et al.[4], pore diameter 500 nm corresponds to negligible intruded pore volume. The pore repartition issued from desorption isotherm is plotted in Figure 4. Two distinct peaks can be observed. The smaller peak is centered about 40 nm, which is mainly attributed to capillary phenomena. The larger one is centered about 2 nm, which is mainly attributed to C-S-H porosity. However, the centers of peaks are very sensible to the relative humidity imposed during the water desorption experiment. If the first relative humidity in the experiment is chosen to be greater than 95%, the first peak will move to the right, corresponding to a bigger pore diameter as shown in the pore size distribution obtained by MIP [1, 4]. Additional desorption step at relative humidity between 30% and 50% (corresponding to pore diameter of 3.1 nm) may shift the larger peak to the right.

3.3 Relative mass loss distribution

Since all sliced samples are numbered and marked by the number of cylinder from which they were sawed and by the order indicating their position in the cylinder, it is possible to analyze the distribution of additional relative mass loss between two successive relative humidity steps related to slice positions. Taking the cylinder BI 6 as an example, Figure 5 presents its relative mass loss distribution according to slice positions in the cylinder. The differences between different slices are very important when the fully water-saturated sliced samples are submitted to the relative humidity 95%. However, the differences in relative mass loss become much weaker for the other relative humidity decreases.

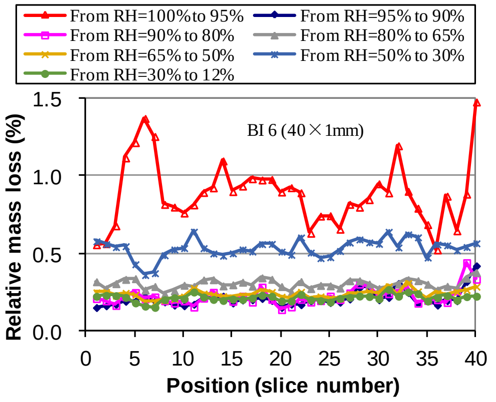

Figure 5: Relative mass loss distribution according to slice position for cylinder BI 6

3.4 Water content distribution

The water content distribution according to slice position for cylinder BI 6 is presented in Figure 6. Considerable divergence can be observed, which can be attributed to the difference in aggregate volume fraction in each slice. Noticeable differences may exist in some neighbor slices, but if several consecutive slices are chosen together to calculate the mean water content, the difference may be removed in view of non-existence of concentration zone presenting much higher or lower water content. Therefore, it is possible to reduce the time of experiment by sawing several consecutive slices from the same concrete cylinder.

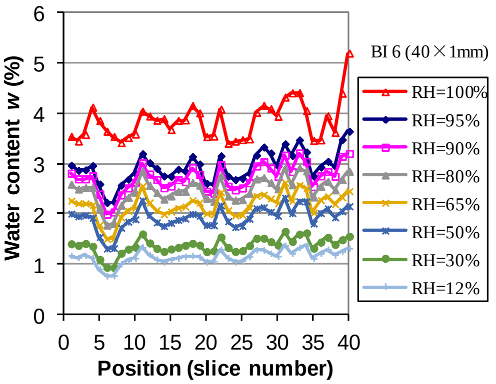

Figure 6: Water content distribution according to slice position for cylinder BI 6

3.5 Image Analysis

Six sliced samples from the cylinder BI 6 (‘I6 4’, ‘I6 5’, ‘I6 6’, ‘I6 38’, ‘I6 39’ and ‘I6 40’) are scanned by X-ray microtomography. Figure 7 presents the image of the sliced sample ‘I6 4’ obtained by X-act reconstruction and ImageJ processing. The particles analysis allows showing the outline of aggregates and exporting the data of aggregate surface and perimeter. Only the aggregates with a surface bigger than 40 voxels are taken into account. The aggregate volume fraction and the volume fraction of interfacial transition zone can be estimated with the data.

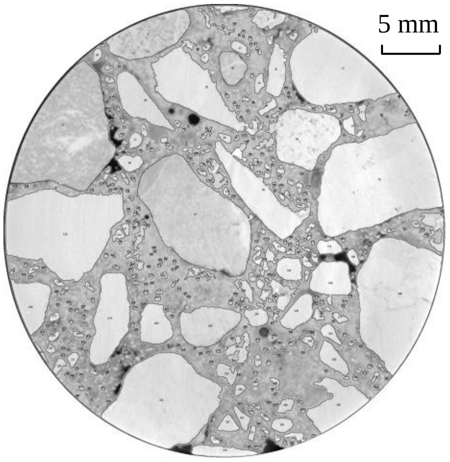

Figure 7: Two-dimensional image showing aggregates and cement matrix for the slice ‘I6 4’

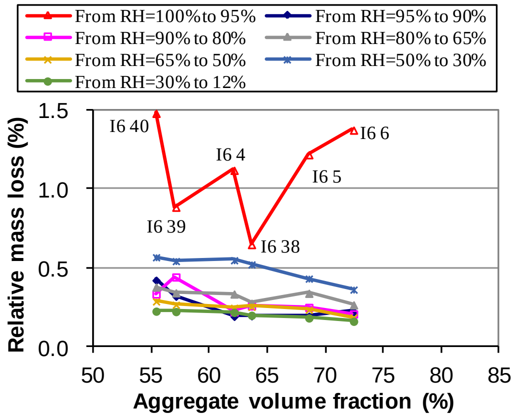

Figure 8: Mass loss vs. aggregate volume fraction

Figure 8 presents the relationship between relative mass loss and estimated aggregate volume fraction for different relative humidity decreases, showing a nearly linear tendency except for the relative mass loss between RH=100% and 95%. The aggregate volume fraction of slice ‘I6 40’ (0.553) is a bit smaller than slice ‘I6 39’ (0.570), but the volume fraction of interfacial transition zone is much bigger for slice ‘I6 40’ according to the image analysis (a total perimeter of aggregates of 1325 mm for ‘ I6 40’ and 1137 mm for ‘I6 39’). Higher porosity and more large pores could be expected in the interfacial transition zone [12], which may lead to an increase in mass loss of slices at high relative humidity 95% due to the water loss in the large pores.

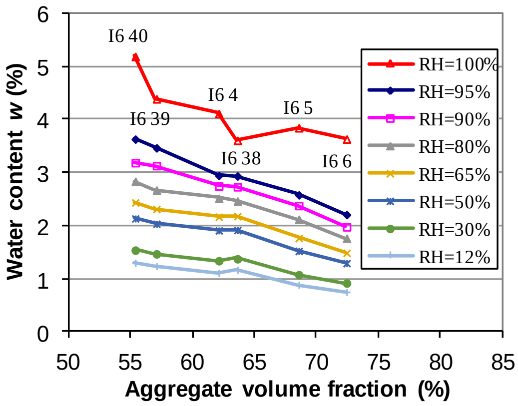

Figure 9: Water content vs. aggregate volume fraction

Figure 9 presents the relationship between water content and estimated aggregate volume fraction for different relative humidity. The higher the aggregate volume fraction is, the lower the water content can be expected. A linear relationship can be observed except for the fully saturated water content (RH=100%), which may also be attributed to the difference between volume fraction of interfacial transition zone.

CONCLUSIONS

An experimental campaign was undertaken to develop an accelerated experimental method to obtain desorption isotherm of concrete more quickly and more reliably by following the same samples. A large number of sliced samples with thicknesses of 1, 2 and 3 mm of a concrete based on CEM I cement have been de-saturated from fully saturated state till the reference dry state.

The first desorption isotherm at T=20°C obtained by present experimental method compares well with the results of former studies.

The results show that time needed to obtain a mass stabilization varies from 14 to 30 days for concrete CEM I at a given relative humidity. Time saving is remarkable considering that several months are needed for the mass stabilization with larger cylindrical samples especially at lower relative humidity.

The analysis of size effect in the drying kinetics shows that thinner samples lose their masses more quickly in the first week, but the thickness has hardly any influence on the total relative mass loss for a given relative humidity once the mass stabilization is reached.

The analysis of images of sliced samples obtained by X-ray microtomography shows that the water content and relative mass loss between two successive relative humidity steps have a nearly linear relationship with aggregate volume fraction except for very high relative humidity, which may be attributed to the difference between volume fraction of interfacial transition zone in different slices.

ACKNOWLEDGEMENT

The authors wish to gratefully acknowledge Andra (French Radioactive Waste Management Agency) for its scientific and financial support to the project that led to the development of the present accelerated experimental method.

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