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DETERMINATION OF EQUILIBRIUM MOISTURE CONTENTS OF BUILDING MATERIALS: SOME PRACTICAL DIFFICULTIES

ABSTRACT: Though the definition of equilibrium moisture content of porous materials is straight forward from the point of view of equilibrium thermodynamics, experimental determination of this quantity may not always be easy. Many factors play their roles from an experimentalist's point of view. Hysteresis, the reference dry state, the initial moisture content, the history of the test specimen, the size of the test specimen, the drying technique and drying temperature - all influence the value that is arrived at by an experimentalist. This paper reports results from several series of measurements on gypsum board, eastern white pine, aerated concrete, and calcium silicate brick using the ASTM Standard C 1498 in the hygroscopic ranges and pressure plate techniques above the hygroscopic range of equilibrium moisture contents. These results show the ambiguities that may surround the experimental definition of equilibrium moisture content. The results presented in the paper demand a few refinements in the existing ASTM Standard C 1498 as well as in the procedure that is used in the pressure plate method. The paper proposes a few refinements for both. The need for the development of an ASTM standard for the pressure plate technique is identified.

KEYWORDS: adsorption, desorption, equilibrium moisture content, hysteresis

Introduction

All porous building materials, when in contact with moist air, adsorb or desorb water molecules to attain an equilibrium moisture content in them [1]. This equilibrium moisture content depends very strongly on the partial pressure of the water vapor in the surrounding air and rather weakly on the air temperature [2-6] that are commonly

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experienced in buildings. The International Energy Agency Annex 24 Report on material properties [6] gives three definitions for "moisture content" in building materials. These are:

i)   mass of moisture per unit volume of the dry material

ii)   mass of moisture per unit mass of the dry material and

iii)   volume of condensed moisture per unit volume of the dry material.

In this paper the second definition is invariably used.

If the moisture content of the material does not vary with time for a given combination of water vapor pressure and air temperature, it is then said that it has reached the equilibrium moisture content (EMC) of the material at that water vapor pressure and temperature. When left undisturbed, the natural end of any adsorption or desorption process is the attainment of EMC. This, though a natural process, is often very slow. Building materials may take several days or weeks to reach equilibrium.

When the water vapor pressure of the air approaches the saturation water vapor pressure at the temperature of the air, the EMC of building materials rapidly increases. At these stages, the process undergone by the building material is not only adsorption. Water vapor begins to condense within the pore structures of the building materials. Theoretically, if the building material is in contact with air that is 100 % saturated for a very long period, all pores of the material should be filled with the condensed moisture. The EMC that corresponds to that hypothetical state is called the saturation moisture content of the material. But in practice the rate of this process becomes infinitesimally small at an EMC that is known as the capillary saturation moisture content and is often substantially less than the saturation moisture content referred to above. In practice, in order to reach the saturation moisture content, a fully evacuated porous building material is kept immersed in de-aerated water for several days. For this reason, this EMC is often referred to as the vacuum saturation moisture content.

The EMC of a building material thus spans from an absolute dry state of zero moisture content to its vacuum saturation moisture content, as the partial pressure of water vapor varies from zero to full saturation value for a given temperature. But the path that connects these two extremes is often not unique. This is due to a phenomenon called hysteresis. For example, in an investigation at the Institute two specimens of identical

dimensions of eastern white pine were prepared. Both were dried to a reference dry state at 105°C. One of them was brought to capillary saturation while the other was kept in the dry state. Then both were exposed to air at 21°C and 67% RH for several days. The specimen that underwent adsorption attained an EMC = 0.082 kg kg^{-1} while the specimen that underwent desorption attained an EMC = 0.120 kg kg^{-1}. This is entirely due to hysteresis. Thus for adsorption and desorption there are at least two separate sets of moisture contents for a building material that exhibits hysteresis. This generally results in an EMC diagram similar to the one shown in Figure 1, for many common building materials. The two curves in such an EMC diagram are referred to as sorption-desorption isotherms.
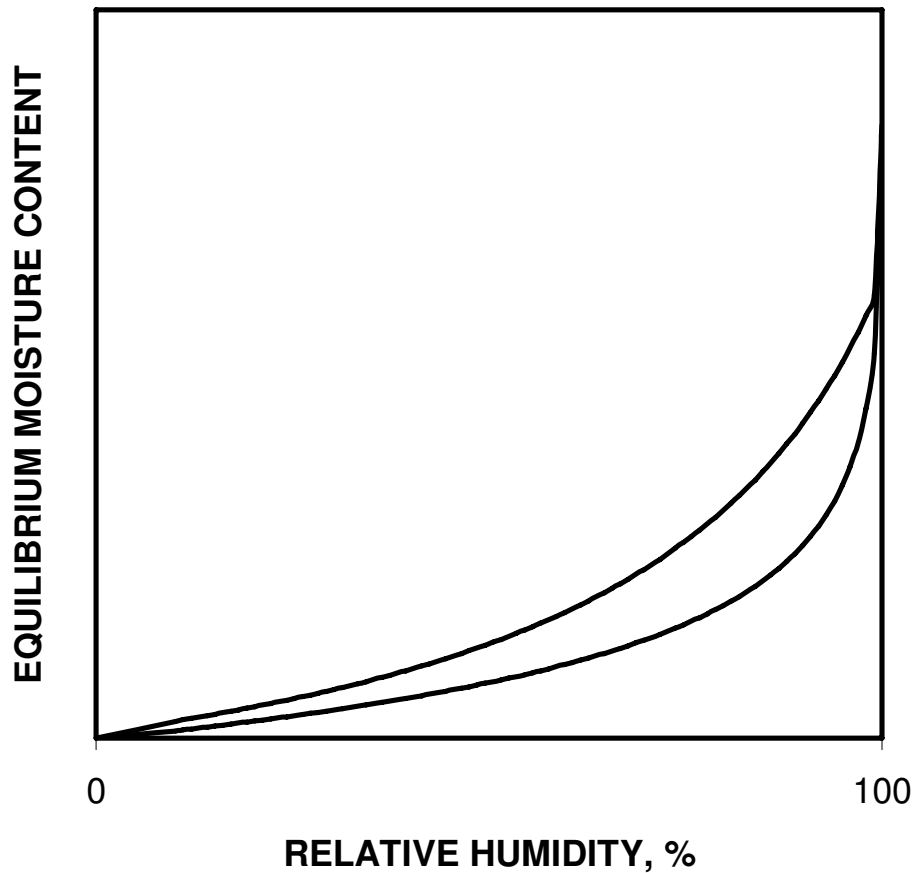

Figure 1. Sorption-desorption isotherm for porous building materials; the upper curve represents desorption and the lower sorption.

The definition of EMC appears to be rather straight forward. But the experimental determination of this quantity may not always be easy. Many factors play major roles in a reliable determination of EMC. Hysteresis obviously is a factor. In addition, the reference dry state, the initial moisture content, the history of the test specimen, the size of the test specimen, the drying technique and drying temperature - all influence the EMC value that is arrived at by an experimentalist.

Internationally accepted procedures are available for the determination of sorption-desorption isotherms. ASTM Standard Test Method for Hygroscopic Sorption Isotherms of Building Materials (C 1498) and the Pressure Plate Method [7] are generally used for this purpose. The principles of these two methods are given below.

ASTM Standard C1498:

For sorption measurements, the test specimen is dried at an appropriate drying temperature to constant weight. While maintaining a constant temperature, the dried specimen is placed consecutively in a series of test environments, with relative humidity increasing in stages, until equilibrium is reached in each environment. Equilibrium in each environment is confirmed by periodically weighing the specimen until constant weight is reached. From the measured weight changes, the EMC at each test condition can be calculated and the adsorption isotherm drawn.

The ideal starting point for the desorption measurements is from an equilibrium condition very near 100% RH. While maintaining a constant temperature, the specimen is placed consecutively in a series of test environments, with relative humidity decreasing in stages, until equilibrium is reached in each environment. Equilibrium in each environment is confirmed by periodically weighing the specimen until constant weight is reached. Finally, the specimen is dried at the appropriate temperature to constant weight. From the measured weight changes, the equilibrium moisture content at each test condition can be calculated and the desorption isotherm drawn. ASTM Standard Test Method for Hygroscopic Sorption Isotherms of Building Materials (C1498) gives further details of the procedure.

Equilibrium Moisture Content from Pressure Plate (Desorption) measurements

The test specimens are saturated with water under vacuum. Those are then introduced in a pressure plate apparatus that can maintain pressures up to 100 bar for several days. The plates in perfect hygric contact with the specimens extract water out of the pore structure until an equilibrium state is established. The equilibrium values for moisture contents in the specimens and the corresponding pressures (measured as the excess over atmospheric pressure; the negative of this value is referred to as the pore pressure while the absolute value is the suction) are recorded. The equilibrium pressure, p_h, can be converted to a relative humidity, φ, using the following equation:

$$\ln\varphi = -\frac{M}{\rho RT} p_h \qquad (1)$$

Where,

M = the molar mass of water

R = the ideal gas constant

T = the thermodynamic temperature and

ρ = the density of water

The Nordtest Technical Report [7] briefly describes a procedure for pressure plate measurements for building materials and reports the results from an interlaboratory comparison. The 'nordtest' method, NT BUILD 481[8] also describes the procedure. ASTM International Standards D 2325, Standard Test Method for Capillary-Moisture Relationships for Coarse- and Medium Textured Soils by Porous-Plate Apparatus and D 3152, Standard Test Method for Capillary-Moisture Relationships for Fine-Textured Soils by Pressure-Membrane Apparatus present test procedures to determine equilibrium moisture contents for soils. No standard is yet developed for building materials.

This paper reports many sets of adsorption, desorption and suction measurements on test specimens of aerated concrete (density 460 ± 15 kg m^{-3}), eastern white pine (density 460 ± 60 kg m^{-3}), interior gypsum board (density either 625 ± 7 kg m^{-3} referred to as A or 720 ± 10 kg m^{-3} referred to as B) and calcium silicate brick (density 2000 ± 80 kg m^{-3}) and examines various practical difficulties that one may encounter during the implementation of the two test methods that are mentioned above.

THE DRY "REFERENCE" WEIGHT

As mentioned above, the EMC at any given condition is calculated in terms of a weight change with reference to a dry state. But what is meant by a dry state? Theoretically, the dry state corresponds to an equilibrium state that is attained by a test specimen when exposed to absolutely dry air or 0 % RH. How does one practically attain a dry state? The most common method is to dry the test specimen to a constant weight in an oven that is maintained at a temperature that is much higher than the standard laboratory temperature. Here is the practical difficulty. The drying ovens that are used for this purpose are integral parts of laboratories. Then, the air inside the oven is supplied from the laboratory air that is not dry. Even if the temperature inside the oven is 100°C, if it is in contact with the laboratory air that is at 20°C at 50 % RH, the air inside the oven is just above 1 % RH. At lower oven temperature the effect is much higher; say at 50°C the air inside the oven will be near 10 % RH! For various reasons, such as softening of the test specimen in the case of expanded polystyrene insulation specimen, or loss of water of crystallization as in the case of interior gypsum board test specimens cannot be dried at much above 50°C. Obviously, such test specimens never reach the theoretical dry state if dried in an oven that is in contact with the laboratory air. An alternative procedure is to dry the test specimens using desiccants. But this also needs much attention, as is illustrated below.

A set of test specimens of interior gypsum board B were dried at 50°C, then equilibrated in air at 23°C and 33 % RH and once again dried, this time using anhydrous calcium chloride as a desiccant. The results are listed in Table 1.

Table 1. Weights of test specimens of interior gypsum board B as dried using two different procedures.

Specimen No.	Weight after drying at 50°C. g	Weight after exposure to 23°C and 33 % RH. g	Weight after drying with the desiccant. g
1	32.315	32.415	32.295
2	32.948	33.053	32.927
3	32.063	32.165	32.045
4	32.396	32.500	32.391
5	32.747	32.851	32.731
6	32.261	32.364	32.240
7	32.742	32.846	32.731
8	33.195	33.304	33.180
9	32.370	32.478	32.352

It can be seen that for all the nine specimens that are listed in Table 1, the dry mass obtained by oven drying is slightly higher than that obtained using the desiccant. The former yields an EMC equal to $(3.20 \times 10^{-3} \pm 7 \times 10^{-5})$ kg kg^{-1} and the latter $(3.7 \times 10^{-3} \pm 2 \times 10^{-4})$ kg kg^{-1}. Does it then mean that the procedure that uses the desiccant gives the right dry weight? There is no straight forward answer to this question either, as illustrated below.

In a second series of measurements, test specimens of interior gypsum board B were first oven dried at 50°C, equilibrated at 23°C and 80 % RH, and then dried using the desiccant. The results are listed in Table 2.

Table 2. Weights of test specimens of interior gypsum board B as dried using two different procedures.

Specimen No.	Weight after drying at 50°C.	Weight after exposure to 23°C and 80 % RH.	Weight after drying with the desiccant.
	g	g	g
1	31.751	32.038	31.778
2	32.174	32.462	32.205
3	32.651	32.931	32.671
4	32.271	32. 566	32.289
5	32.962	33.266	32.983
6	32.085	32.494	32.106
7	32.237	32.537	32.258
8	32.789	33.093	32.812
9	32.218	32.515	32.240

In this case the weight obtained after oven drying is consistently lower than that obtained using the desiccant. The former yields an EMC, $(9.6 \times 10^{-3} \pm 1.2 \times 10^{-3})$ kg kg^{-1} and the latter $(8.8 \times 10^{-3} \pm 1.2 \times 10^{-3})$ kg kg^{-1}. It means that drying with the desiccant in this case resulted in unacceptable dry weight. The reason for this discrepancy is not clear.

This problem may be very specific to the interior gypsum board. Nevertheless, when the ASTM Standard C 1498 is applied to this material, very specific instructions are to be given to assign the dry weight for the material. By consensus it can be the mass that corresponds to oven drying at 50°C. It could equally be from a specified procedure that uses the desiccant as the drying material. Then, indeed, the starting point of the drying process also need to be specified.

Specification of the drying temperature and procedure is important for each type of building material, as is illustrated with a series of measurements on test specimens of eastern white pine. The results from this series are listed in Table 3.

Table 3. Weights of test specimens of eastern white pine as dried progressively at five different conditions.

Specimen No.	Weight of the specimen, g				
	4 days at 50°C	Constant weight at 50°C	Constant weight at 70°C	Constant weight at 95°C	At 105 °C for 2 h
1	4.519	4.485	4.439	4.408	4.404
2	4.679	4.643	4.600	4.566	4.562
3	4.628	4.598	4.554	4.520	4.516
4	4.515	4.491	4.445	4.414	4.408
5	4.475	4.454	4.409	4.379	4.372
6	4.593	4.578	4.530	4.498	4.493

It can be seen that there are appreciable and consistent differences in the dry weights of all six test specimens for the five different progressive drying conditions used. If the EMC for eastern white pine is calculated with reference to the dry weight obtained at 50°C after a four-day-drying period, from the measurements that followed at 95 % RH and 22.3°C, the result would have been (0.20 ± 0.01) kg kg^{-1} instead of (0.215 ± 0.005) kg kg^{-1}. Here the error is not very large, but at a lower RH the error can be substantial. For example, for a set of measurements at 70 % RH and 22.7°C for the dry weights obtained at 50°C the EMC is (0.076 ± 0.002) kg kg^{-1} instead of (0.101 ± 0.001) kg kg^{-1}, the EMC with reference to the final dry weight at 105°C. For wood, 105°C appears to be the appropriate drying temperature. However, to avoid charring of the specimens, it is advisable to dry them first at 95°C to constant mass and then keep at 105°C for a short period of 2 h. Like wise, for each class of building materials an appropriate drying temperature and procedure shall be specified in the Standard. For practical purposes, the intent of any such specification need not be the attainment of an absolute dry state as the reference, but a reproducible one, such that results from different sources on the same material allow inter-comparisons. For building applications one does not have to know

the absolute moisture content of a material. The increment from a practically reproducible reference state will be sufficient for any building design considerations.

THE SIZE OF THE TEST SPECIMEN

The time that a test specimen takes to attain equilibrium as prescribed by C1498 depends on the nature of the building material as well as the size of the specimen. It may vary between several days to several weeks. A 5 cm X 5 cm test specimen of interior gypsum board (1/2" nominal thickness) attains equilibrium at 75 % RH near standard laboratory temperature within a week. A similar eastern white pine specimen may take several weeks to reach equilibrium in a similar environment. For a stucco specimen it can be even longer. Therefore, in order to reduce the duration of the exposure at each test condition, authors in the past have used powdered specimens, shavings, very thin specimens, etc. But how does such sizing down of test specimen affect the EMC? In an attempt to address this a series of measurements was done with pine, aerated concrete and calcium silicate brick. The results from that series are presented below.

Several test specimens of the three building materials were prepared in three categories as follows.

Pine: Twelve specimens, 5 mm X 5 mm and 20 mm thick, twelve specimens, 5 mm X 5 mm and 10 mm thick, and six specimens of saw dust (approximately 90 g). All 30 specimens were dried to constant weight in a ventilated oven that was maintained at 100°C.

Aerated Concrete: Twelve specimens, 5 mm X 5 mm and 25 mm thick, twelve specimens, 5 mm X 5 mm and 13 mm thick, and six specimens of fine dust after crushing (approximately 50 g). All 30 specimens were dried to constant weight in a ventilated oven that was maintained at 80°C.

Calcium Silicate Brick: Twelve specimens, 5 mm X 5 mm and 20 mm thick, twelve specimens, 5 mm X 5 mm and 8 mm thick, and six specimens of fine dust after crushing (sizes varied between 35 g and 75 g). All 30 specimens were dried to constant weight in a ventilated oven that was maintained at 60°C.

The test specimens in each category for each material were grouped into three batches and then one batch each of all three materials was equilibrated at 22.5°C and

50.1 % RH, the second batch at 22.5°C and 70.9 % RH, and the third at 22.5°C and 88.7 % RH. The results are summarised in Table 4.

Table 4. EMC as determined according to C1498 for three building materials, with three variations in the dimensions of the test specimens; the numbers in parenthesis give deviations from the reported mean value.

Building Material	RH, %	Equilibrium Moisture Content, kg kg^{-1}		
		Thicker Specimen	Thinner Specimen	Fine Particles
Eastern White Pine	50.1	0.070(0.001)	0.072(0.001)	0.0653(0.0002)
	70.9	0.099(0.001)	0.100(0.001)	0.0915(0.004)
	88.7	0.164(0.001)	0.166(0.001)	0.162(0.001)
Aerated Concrete	50.1	0.024(0.003)	0.021(0.001)	0.018(0.003)
	70.9	0.034(0.005)	0.031(0.003)	0.023(0.003)
	88.7	0.066(0.004)	0.060 (0.003)	0.058(0.001)
Calcium Silicate Brick	50.1	0.0075(0.0006)	0.0069(0.0008)	0.0072(0.0001)
	70.9	0.0108(0.0010)	0.0104(0.0005)	0.0109(0.0002)
	88.7	0.0246(0.0005)	0.0255(0.0009)	0.0254(0.0003)

From the data given in Table 4 it is not possible to generalize the influence of specimen size on the EMC obtained. If one looks at the data for pine alone it appears that the saw dust gave consistently lower values for the EMC. The reason for this can be only speculated at this stage as an increase in the reference dry weight due to some unknown reasons. The EMCs for the 20 mm thick and 10 mm thick specimens are the same within the limits of the experimental uncertainties. It is noted that the time taken by the saw dust to equilibrate was not significantly different from the other two sets of specimens, for all three conditions. Therefore it is advisable not to use saw dust for the determination of the EMC for wood based materials. When the experimental uncertainties are taken into consideration, for the aerated concrete and calcium silicate brick specimens, the dimensional differences seem to have little effect on the EMCs that are listed above. Here also, the dusts did not attain final equilibrium significantly faster than the block specimens. For each building materials, it is advisable to recommend a representative

thickness. For interior gypsum board, this can be the full nominal thickness. For wood, 10 mm thick slabs appear to be representative. Similarly 10 mm thick slabs of aerated concrete and calcium silicate brick appear to be a practical thickness for the test specimens.

STARTING MOISTURE CONTENT FOR DESORPTION MEASUREMENTS

For some materials the initial moisture content has a measurable influence on the EMC attainted after a desorption process. Measurements that are listed below, on interior gypsum board B specimens, illustrate this behavior.

Three batches of six test specimens each, 60 mm X 60 mm and ½" nominal thickness, were oven dried to constant weight at 50°C. Specimens in one batch was equilibrated at 94 % RH and 23°C. These specimens were left to desorb and equilibrate at 79.5% RH and 23°C, then at 53% RH and 23.2°C, and finally at 33% RH and 23°C. A second batch was allowed to equilibrate initially at 79.5% RH and 23°C and were left to desorb and equilibrate at 53% RH and 23.2°C, and finally at 33% RH and 23°C. The third batch was equilibrated at 53% RH and 23.2°C and allowed to desorb and equilibrate at 33% RH and 23°C. The results from these measurements are listed in Table 5.

The results from Table 5 are plotted in Figure 2. For reference, a sorption curve for the material is included in the figure. It can be seen that the total picture is not as simple as depicted in Figure 1. The desorption curve for the interior gypsum board is not unique. Depending on where the desorption starts, the EMC attains different values. Higher the starting moisture content (or RH), higher is the EMC attained, as is seen in the last column of Table 5. Currently, no detailed information like this has been generated on other building materials. There may be other materials that show similar behavior. Then, in order to determine an upper limit of the desorption curve one has to start the desorption process from the highest possible moisture content that is practically reproducible. The capillary saturation state may be used as that starting point. Then again, this may not be possible for all materials. In such situations the standard should prescribe the starting point explicitly so that data from different sources allow inter-comparison. Also, in practice it may be important to know the intermediate desorption curves, as shown in Figure 2, of some building products. The Standard should facilitate the generation of such information wherever applicable.

Table 5. Desorption EMCs of interior gypsum board specimens with different initial moisture contents; the numbers in parenthesis indicate standard deviations.

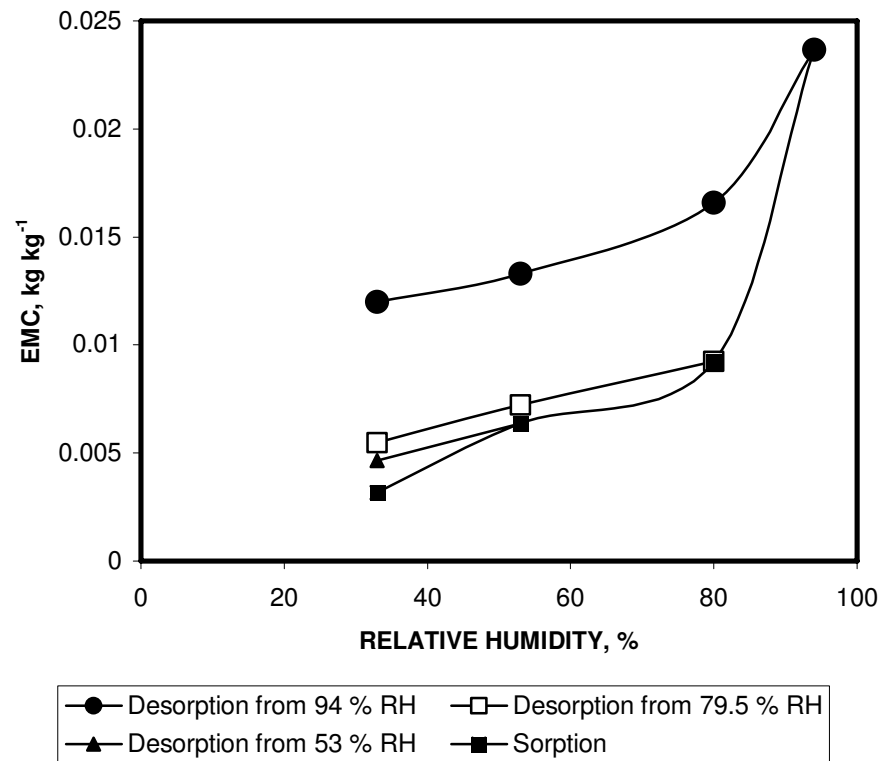| Initial Conditions	Equilibrium Moisture Content, kg kg^{-1}		
	At 79.5% RH and 23°C	At 53% RH and 23.2°C	At 33% RH and 23°C
94% RH and 23°C	0.0166 (0.0001)	0.0133(0.0001)	0.0120(0.0001)
79.5% RH and 23°C		0.00924(0.00006)	0.00548(0.00003)
53% RH and 23.2°C			0.00465(0.00008)

Figure 2. Sorption-desorption curves for interior gypsum board B.

STARTING MOISTURE CONTENT FOR PRESSURE PLATE (SUCTION) MEASUREMENTS

Pressure plate measurements are always desorption measurements. One invariably starts the process from a saturation moisture content. Just as in the case of desorption measurements discussed above, the starting moisture content affects the EMC attained by the specimens at each applied over pressure. This is illustrated with three series of measurements on aerated concrete in Figure 3.
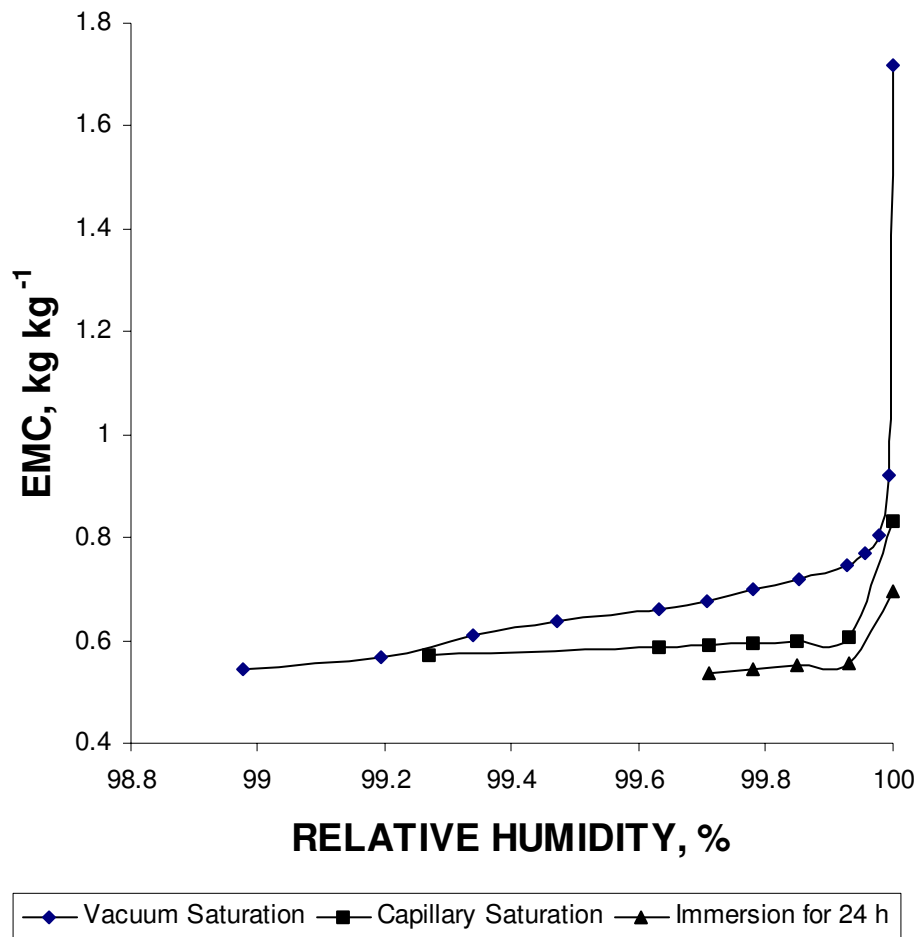
Figure 3. EMC of aerated concrete as measured using the pressure plate method, starting from three different initial moisture contents.

In one series of measurements shown in Figure 3, eight 40 mm X 40 mm X 6 mm specimens were vacuum saturated with de-aerated water to attain an average initial

moisture content of 1.72 kg kg^{-1}. Measurements on these specimens with the pressure plate apparatus resulted in the uppermost curve in Figure 3. In the next series similar set of six specimens were immersed in water and allowed to attain capillary saturation. The average initial moisture content was 0.83 kg kg^{-1}. These specimens gave the middle EMC curve shown in Figure 3. In a third series, the pressure plate measurement were started after 24 h of immersion in water with an average initial moisture content of 0.69 kg kg^{-1}. (Admittedly this was a case of incomplete saturation.) The result was as shown in the lowermost curve in Figure 3. The need to specify the saturation procedure in the pressure plate method is quite obvious from these results. For practical reasons, it is advisable to start with capillary saturation and a prescription for attaining the capillary saturation for each building material. The vacuum saturation is a hypothetical case that is rarely reached in real buildings. The air at high pressure that is trapped in porous building materials during capillary saturation will resist the attainment of this hypothetical saturation point.

MATERIALS THAT DISINTEGRATE ON SATURATION

Some inorganic building materials disintegrate on saturation, when done in the normal way using de-aerated pure water. Interior gypsum board is an example. Often this is due to the loss of ionic compounds that dissolve in water. For these materials, it is advisable to use water that is already saturated with the ionic compounds for the initial specimen saturation. A piece of the material can be ground and stirred into the water that subsequently will be used for saturation[4]. As illustrated in Figure 4, at the Institute this technique has successfully yielded at least an approximate suction curve for the interior gypsum board A, which was otherwise impossible to obtain.

Eight test specimens, each 40 mm X 40 mm at ½" nominal thickness, were used in these measurements. The reference dry weight was obtained after oven drying at 50°C. The starting point was vacuum saturation. All data are plotted in Figure 4 to highlight another inherent practical problem in the measurement of EMC. EMC measurements on building products often results in large scatter of the experimental data. In such cases it is imperative to provide information on the large mean deviation.

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[4] Admittedly, this may affect the equilibrium point due to the difference in the chemical potential of water in the the saturated solution in relation to that of pure water, but the procedure allows an estimate of the desorption curve of materials that disintegrate on saturation.
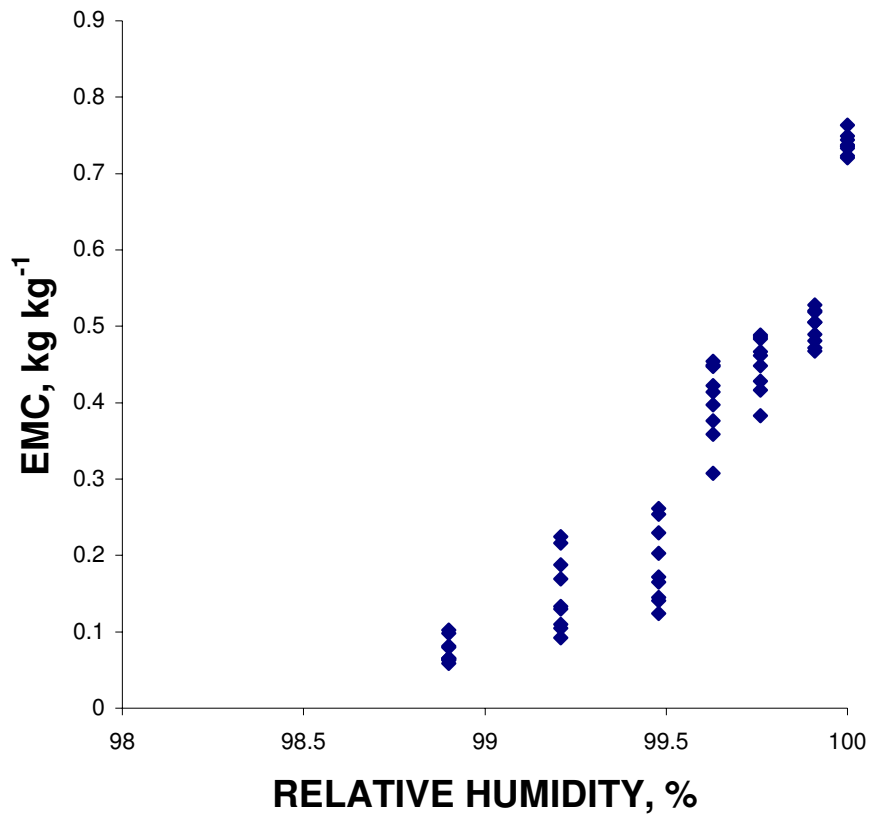
Figure 4. EMC of eight test specimens of interior gypsum board A from pressure plate measurements.

CONCLUDING REMARKS

In spite of the simple definition of equilibrium moisture content, various results that are presented in the preceding sections show that the experimental determination of this quantity with good precision is a challenging task. Perhaps the greatest challenge is to accommodate the non-homogeneity, as is evident from Figure 4, of porous building materials. This alone can introduce an uncertainty that is more than 20 %! So it should always be the recommended practice to include the uncertainty in each set of measurements. To assess this uncertainty, as many as eight to ten randomly selected specimens from a material need to be equilibrated at each exposure condition.

For each building material, or at least for each class of building materials, a drying temperature as well as a drying procedure should be explicitly defined, to determine the reference dry weight. Otherwise results from different laboratories will

have different meanings. This may require well-planned round robin series of measurements that involve several laboratories.

It is better to avoid fine particles of building materials for the measurement of EMC, because in certain cases it may affect the pore size distribution of the building material. Use of fine particles is not substantially improving the time that is taken for equilibration. Furthermore, at least in the case of wood, a systematic error is introduced in the EMC data when sawdust is used as the test specimen. It appears that for most materials 40 mm X 40 mm X 10 mm specimens are quite practical dimensions.

It is always advisable to start with the highest possible moisture content to construct upper limits of the desorption isotherms. Practically, capillary saturation is a reasonably reproducible high-moisture content starting point for many materials. This is true for the suction measurements in the pressure plate apparatus too. It is advisable not to start from the vacuum saturation state, for this rarely happens in building practice. Special procedures need to be developed to saturate those building materials that disintegrate on saturation.

For practical reasons, if the information on the intermediate desorption curves of building materials, as shown in Figure 2, is needed, the Standard should facilitate the generation of such information. In this regard further experimental data on other building materials need to be generated.

The ASTM Standard C 1498 should be further refined to accommodate the findings that are reported here. There is a need to develop an ASTM Standard for the use of the pressure plate apparatus to determine the EMC of building materials at high humidity (above 98 % RH) conditions. ASTM C16 Committee should take the initiative in this regard. Efficient inter-laboratory collaborations will be necessary to arrive at consensuses and address these recommendations.

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