



## Article

# Reliability Study of Equilibrium Moisture Content Methods for Sorption/Desorption Isotherms Determination of Autoclaved Aerated Concrete

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**Abstract:** Autoclaved aerated concrete (AAC) and its hygric parameters are a highly important issue in the field of building physics. There are several methods currently available to determine the equilibrium moisture content of building materials. Beside the conventional ones, new methods are constantly being introduced. This study explores the sorption/desorption properties of three types of commercially produced AACs with three different bulk densities and demonstrates the application of the relevant methods available to characterize these parameters. The reliable characterization of the studied material was done through the conventional static approach, using the desiccator and an environmental chamber, and a new automated method of dynamic vapor sorption is implemented. The goal is to compare and identify the reliability of all methods used with respect to the efficiency of the data measurement process. Sound consistency between the results of the conventional methods and the experimental data obtained indicates the dynamic vapor sorption technique is highly reliable when measuring the equilibrium moisture content—particularly exemplified during the AAC sample testing. Therefore, the methodology developed in this study is expected to provide the reference for measuring the sorption/desorption isotherms of building materials with both static and automated techniques.

**Keywords:** sorption; hygric properties; autoclaved aerated concrete; dynamic vapor sorption



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## 1. Introduction

The reliability of building structures is closely connected to various physical phenomena, among which moisture can be undoubtedly considered one of the highly relevant factors. Moisture affects the number of material properties, from thermal conductivity to the mechanical properties in terms of strength, etc. There are more parameters that describe the behavior of the material from a moisture perspective. The structure and material base [1] determine which phenomena will be applied during moisture transport and moisture binding. Capillary and porous materials are often used for building construction; however, they interact with moisture intensely. In building construction, autoclaved aerated concrete (AAC) is a frequently used material and can be considered a representative of a sustainable building material, considering its specific structural, mechanical and thermal insulation properties. This allows forming the building envelope structure with no additional thermal insulation layer and thermal bridges; its thermal conductivity ranges from 0.09 to 0.2 W·m<sup>-1</sup>·K<sup>-1</sup>), with bulk densities ranging from 450 kg/m<sup>3</sup> to 750 kg/m<sup>3</sup> [2]. On the other hand, the properties of this material are strongly dependent on moisture content, which also affects the strength characteristics and compressive strength [3,4]. In general, the moisture transport in concrete is determined by the moisture gradient

as well as the temperature gradient [5]. The effect of moisture content on the thermal conductivity of the AAC is significant, with a critical value of 15% moisture content [6]. Other studies, focused on lightweight concretes, expressed a linear dependency of the increase in thermal conductivity and moisture [7]. However, the hygrothermal behavior of AAC masonry walls is a subject absent in discussions of the scientific community [8]. Moisture transport by diffusion and capillary conduction is used in AAC. The ability to express moisture binding at a low saturation is expressed by the sorption curve. When dry porous material with an open-pore structure is exposed to a humid environment, moisture binds in its pores and even in some pore groups, getting completely filled. This state takes place gradually, characterized by a gradual and very slow weight gain, which in a certain time reaches the so-called equilibrium state. Measurements show that relative humidity varies instantaneously and linearly with the temperature at locations where the moisture content is supposed to be constant. These variations were found consistent with the temperature dependence of the sorption isotherm. [9]. Similarly, desorption, exposure of a moist material to a lower humidity environment, results in the gradual release of moisture from its porous structure, causing weight loss. Most materials are characterized by a very significant deviation between sorption and desorption, called hysteresis. The measurement of building material moisture, using well-established methods as well as the inclusion of new approaches [10,11], are one of the current relevant research challenges to establish more detailed studies of the basic physical processes fundamental to moisture transport. The results of measurements by standard steady-state procedures for moisture transfer in AAC building materials may show slightly underestimated data compared to the methodologies using dynamic effects [12]. In case of highly hygroscopic materials, such as AAC, the samples going through different preconditioning methods (especially in wet cup tests) may have a statistically different moisture content and permeability [13].

Therefore, adequate material parameters and their characterization are currently an important factor for identifying the reliability of the development and verification of theoretical models, such as the numerical prediction of heat, air and moisture transport (HAM) in porous building materials [14,15]. The effects of hysteresis on the sorption and moisture capacity—the key physical phenomena—need to be included in the mathematical model of transfer. Accordingly, the reliability of the measured material properties, which serve as the input for the HAM models, remains very uncertain. A round-robin test was performed within IEA/ECBCS 41 to determine the hygric properties of porous building materials in order to generate a data set for comparison of the numerical models [16]. As a result, recent studies experimented with quantification of the hygric properties from different aspects by applying different measurement techniques [17–19]. An important factor to consider is the specific technology and composition of the investigated porous material, which may differ significantly depending on the region as well as the manufacturer.

The energy performance of buildings has undergone major developments in recent years. In the framework of the current trends and global development, new technologies and structural solutions aim to increase the energy efficiency of buildings. AAC is a building material commonly used in Europe but also in other regions of the world [20,21]. Its preference stems from particularly advantageous thermal insulation properties and lightweight construction principles. Currently, its expansion is also extensive in regions of Asia and especially China [22], which operates several hundred production plants [23]. This proves that it is of interest all around the world. However, its use often confronted with other more natural forms of sustainable materials, due to the group of recent trends in the contemporary construction industry [24]. One of the key characteristics of AAC is its hygric properties [25]; they are highly dependent on the water content of the porous material [26]. As a result, high values of water content can significantly reduce the thermal performance of AAC, which may affect why its utilization is accompanied with various levels of application around the world. The composition of the material is a basic structure of Portland cement, lime, sand or fly ash and aluminum powder. Bulk densities of AAC range from  $400 \text{ kg}\cdot\text{m}^{-3}$  to  $800 \text{ kg}\cdot\text{m}^{-3}$  and predisposes its building application according

to the requirements and demands of incorporation into the structure in terms of purpose and function. Recently, these types of material have seen rapid progress in development, focusing mainly on their pore structure [27] and the impact of the filler by reusing industrial waste [28] or other recyclates [29,30]. Under similar conditions, ash-based AAC was found to exhibit a slightly higher moisture sorption compared to sand-based concrete. Improving the thermal insulation properties and the utilization of plastic waste can also be a promising solution for lightweight concrete [31]. There are also current efforts to improve AAC with the integration or incorporation of a high latent heat capacity [32,33].

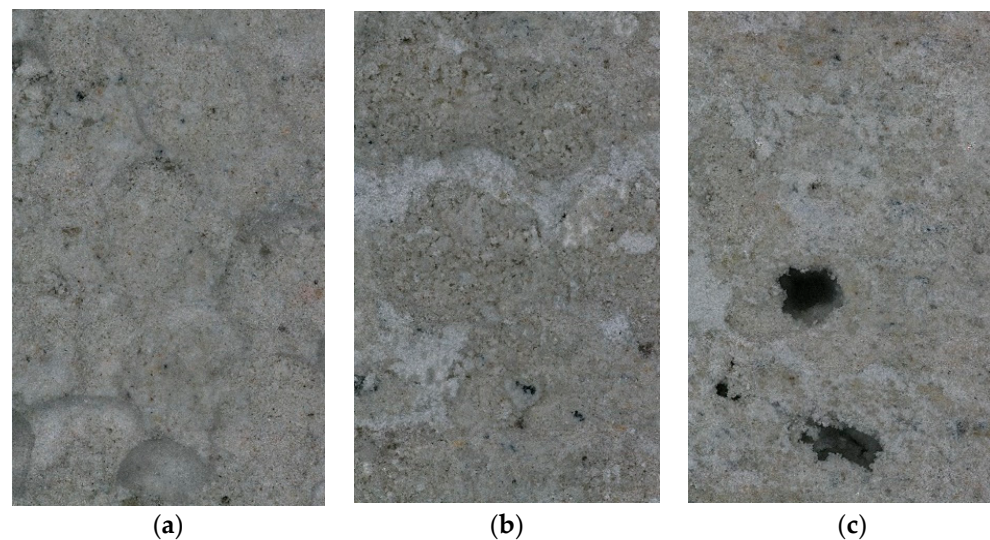
This paper analyses the equilibrium moisture content levels of three types of commercially produced AACs with different bulk density and compressive strengths, along with their basic physical properties by applying different measurement methods. Though there are some studies comparing conventional static methods and automated dynamic vapor sorption (DVS) methods [34,35], its application is still not widely used in relation to building materials. In addition, as the DVS method represents an effective method, it is considered suitable to be used for sorption isotherms determination of AAC up to about 80% relative humidity [35]. This represents an important limitation that needs further investigation. Thus, the data obtained by this research will make it possible to carefully reconsider this research finding, aiming to identify the suitability of the compared methods for measuring sorption/desorption properties. The methodology used is expected to provide the reference for measuring the sorption/desorption isotherms of AAC with both static and automated techniques. This will improve the quality of studies of the hygrothermal performance of building envelopes based on the commonly used AAC in building practice.

## 2. Materials and Methods

The material characterization included determination of the samples' bulk density and sorption characteristics, using various methods. The bulk density is dictated by a sample's dimensions and weight. Equilibrium humidity points were determined by conventional methods, using the desiccator method and an air-conditioning chamber, as well as an advanced new method of dynamic vapor sorption. The goal was to compare them and identify the reliability of each method in relation to their accuracy and efficiency for the quantification process. Measurement of bulk densities and sorption in desiccators with acid solutions was provided by the laboratory of the Department of Material Engineering and Physics, Faculty of Civil Engineering, Slovak University of Technology in Bratislava. The sorption of the materials in the desiccators with salt solutions, climatic chambers and the DVS system was corroborated by the laboratory of heat and moisture material properties of the Jossef Ressel Research Center in Útechov, which is part of the Faculty of Forestry and Wood Technology at Mendel University Brno.

### 2.1. Material Preparation and Description of the Used Samples

The AAC samples used for this study were provided by PORFIX—Probeton a.s. in Zemianské Kostol'any (Slovakia). Three sets of samples with different bulk densities were examined (see the microscale in Figure 1). The samples were marked AC-4, AC-5 and AC-6, where sample AC-4 had a bulk density of  $430 \text{ kg}\cdot\text{m}^{-3}$ , AC-5 was  $520 \text{ kg}\cdot\text{m}^{-3}$  and AC-6 was  $630 \text{ kg}/\text{m}^3$ . Each sample was cut into  $100 \text{ mm} \times 100 \text{ mm} \times 20 \text{ mm}$  cubes. As it is a standard aerated concrete, the composition segments of the samples are also commonly sold; thus, lime + cement (binder), silica sand (filler), water and aeration were provided with the aluminum powder. The individual compositions of each sample type are shown in Table 1.



**Figure 1.** A 200× magnified microstructure of the autoclaved aerated concrete (AAC) samples: (a) AC-4; (b) AC-5; (c) AC-6.

**Table 1.** The samples' composition.

Raw Material	Unit	AC-4	AC-5	AC-6
Water content in autoclave water	%	44	40	39
Burnt lime from Varín, CaO 88%, MgO 4%, loss on ignition	kg/m <sup>3</sup>	11.8	18.7	9.1
Cement 52.5 N from Rohožník	kg/m <sup>3</sup>	18.4	13.5	14.1
Fluid ash from Třinec 15 CaO, 35% CaO	kg/m <sup>3</sup>	2.4	1.7	1.8
ash Mondi silica from wood combustion SiO <sub>2</sub> 35%, CaO 30%	kg/m <sup>3</sup>	5.0	3.6	3.8
Cemperlite	kg/m <sup>3</sup>	4.2	3.1	3.3
Aluminum powder	kg/m <sup>3</sup>	0.150	0.083	0.065
Energogypsum—CaSO <sub>4</sub> , 2H <sub>2</sub> O—98%	kg/m <sup>3</sup>	5.3	4.2	6.9
Plasticizer	kg/m <sup>3</sup>	0.053	0.042	0.035
Sand Krnča, quartz up to 90% wt.	kg/m <sup>3</sup>	52.6	64.8	60.8

## 2.2. Fundamental Test Procedure for Sorption Isotherm Determination

The sorption isotherm was established on a sample dried to an unchanging weight, in an electric oven. The drying temperature varied from 40 to 105 °C, depending on the material type. In case of AAC, the temperature of drying was 105 °C. Subsequently, several points of relative humidity were defined to determine the related constant temperature curve. A minimum of four points were used to evenly cover the entire humidity interval. Dried material was exposed to the first point, representing the less humid environment. The weight of the sample was monitored continuously until it changed. The sample had reached the equilibrium when the mass variations in the last three measurements throughout a 24 h period did not exceed 0.1% of the total weight. At this point, the equilibrium point had occurred, and the sample was placed in another environment with a higher relative humidity, and the entire weighing procedure was repeated until all the intended equilibrium points had been determined. After measuring the point with the highest humidity, it was also possible to identify the desorption; this was measured similarly, except for the humidity in the sample's environment gradually decreasing at each individual equilibrium point. While the starting point for sorption was usually dry material, the desorption curve usually begins at a relative humidity of at least 95%. Sometimes desorption begins its measurement on samples with fully saturated pores that have been immersed in water for several days or weeks.

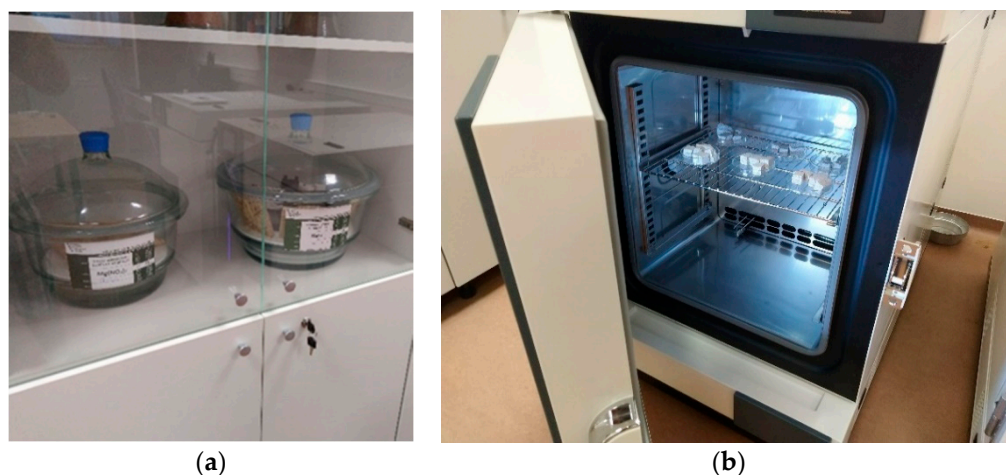
Weight accuracy is usually required to be within 0.01% of the weight of the sample, meaning a 100 g sample calls for a device with an accuracy of at least 0.01 g. Test specimens of common building materials should be at least 10 g. For materials with a bulk density of

less than  $300 \text{ kg m}^{-3}$ , a minimum sample size is required, which is a square with a side length of at least 100 mm. The number of samples should always be at least three pieces; due to the possible large variability, it is advisable to use more samples. Most often, these curves are determined for temperatures of  $23 \text{ }^\circ\text{C}$  in mild or  $27 \text{ }^\circ\text{C}$  in tropical areas.

### 2.3. Conventional Measurement Tests

Hygroscopic and porous materials bind moisture in their structure, depending on the relative humidity of the environment. There is an equilibrium moisture content for a certain state of ambient humidity in building materials. It represents the amount of moisture that had stabilized in the material under the given conditions. The determination of equilibrium moisture in building materials is commonly conducted using two conventional methods [36].

Desiccator methods can be considered as the most well-known and widespread. The desiccator should be placed in a constant temperature environment, as some solutions significantly change the water vapor pressure above the saturated aqueous solution, even with small temperature changes. Figure 2a shows an example of a test with the desiccators placed in air-conditioned room. The second conventional method is based on the use of a climatic chamber that provides the environmental parameters to which the sample is exposed. The method is in principle identical to the desiccator method, where the sample is exposed to the intended environmental state. The difference is the samples are placed in a test chamber where they need to be weighed. The chamber should allow the handling of samples without opening it in order to avoid contamination from the environment; thus, to not affect the samples with the air of the laboratory. If this is not possible, lid-covered test dishes should also be used or the scale placed directly in the chamber and the samples handled via a hand-port. Both the mentioned conventional methods are standard according to EN 12 571 [37].



**Figure 2.** (a) Rack with desiccators in an airconditioned room; (b) samples in an environmental chamber.

Identifying the interaction of a material with air humidity is often not limited to one state—one point of moisture equilibrium. These points tend to be investigated at a constant temperature and various relative humidities to quantify the wider area. The line connecting a set of such equilibrium points at a constant temperature represents the sorption isotherm. For building materials, these curves most often range from 30 to 95%, where these properties are the most important for building applications. At a higher humidity, there may be problems with biological attack or degradation of the samples; also, the growth of fungi and molds on their surfaces may arise. Although many building materials can acquire a humidity higher than 95% or 97%, their measurement is difficult to accomplish. At such a high relative humidity, even small change in temperature can increase the risk of moisture condensation on the surface of the samples, which again may

affect the results of the experiment. The curves are basically detected in two modes. The sorption mode gradually saturates the material with moisture by increasing the relative humidity and determines the constant value of equilibrium humidity for each environment. On the other hand, the desorption regime is represented by a gradual reduction of the humidity in an environment. Both processes are very slow, and stabilization can take a very long time, especially for higher humidities. The desorption curve acquires greater values than the sorption curve. This provides the hysteresis and is attributed to several factors.

Dried samples with dimensions of 100 mm × 100 mm × 20 mm were first placed in the lowest humidity environment. The weight was determined at regular intervals, and after stabilization the samples were moved to a desiccator with a higher humidity. After measuring the highest humidity, the process was repeated, applying decreasing ambient humidity. The measurement of each point took approximately two weeks. The laboratory temperature during the experiment was around 22 °C. The same samples were used for the measurements and for the determination of the bulk density. From the point of view of the standard procedure, the samples were not enclosed in pans with lids due to their shape and size. Sulfuric acid solutions were prepared in desiccators according to Table 2.

**Table 2.** Mixing ratio for sulfuric acid and demineralized water.

Density of Solution (kg m <sup>-3</sup> )	Concentration of Sulfuric Acid in Solution	Relative Humidity Above Sulfuric Acid Solution
1350	42%	50%
1200	24%	80%
1100	12%	95%

Equilibrium moisture levels of the salt solutions in the desiccator were also measured in a similar manner to the acid solutions. Smaller samples, in the range of 20 to 35 g, were used for the measurements, which were created by breaking the original samples of 100 mm × 100 mm × 20 mm in size. The substances used for the saturated solutions are listed in Table 3. In contrast to the standard procedure, the test dishes were not covered at weighing. Dried samples were gradually exposed to environments with increasing relative humidity during consecutive measurements of their weight. After determining the sample mass at the highest relative humidity, the desorption branch was measured by gradually decreasing the relative humidity.

**Table 3.** Chemicals used for the preparation of the saturated solutions.

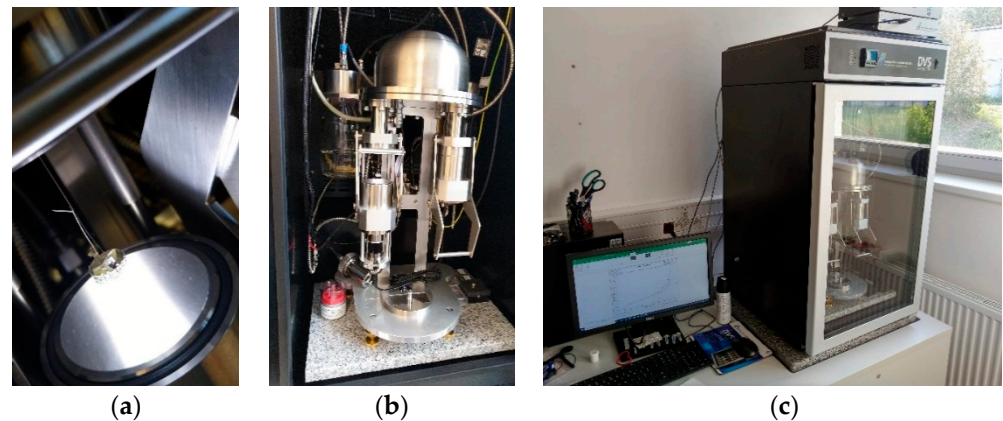
Relative Humidity at 23 °C	Name of Substance	Chemical Formula
53.5%	Magnesium nitrate	Mg(NO <sub>3</sub> ) <sub>2</sub>
75.4%	Sodium chloride	NaCl
84.7%	Potassium chloride	KCl
94.0%	Potassium nitrate	KNO <sub>3</sub>

The last conventional method condition for the aerated concrete samples was to place them in a JEIOTECH TH3-ME-100 air environmental chamber. Samples with a weight between 20 and 35 g, formed by breaking the larger 100 mm × 100 mm × 20 mm blocks, were conditioned at a relative humidity of 50, 75, 85 and 95%. The samples were placed in glass dishes and weighed at regular intervals with a laboratory scale with an accuracy of 0.01 g, until the weight stabilized. In contrast to the standard procedure, the testing dishes were not covered during weighing due to the shape of the samples.

#### 2.4. Dynamic Vapor Sorption Tests

The dynamic vapor sorption method (DVS) can be considered a novel technique and its application is still not commonly used in the building physics field. The device uses very sensitive scales with a high resolution, up to the microgram level. This allows to

reduce the sample size and speed up the whole measurement procedure. The sample is weighed continuously to track the real response of the material in real time and not just particular discrete points that are identified throughout several hours. The weighing dish of the sample has a diameter in the range of 10 to 30 mm and is placed on a hang down wire in the measuring chamber. The humidity of the environment is maintained by mixing dry and saturated humid air from a gas cylinder in the desired ratio. The air in the sample space has a large volume flow, which even allows the measurement of highly sorption active materials. Figure 3 shows details of the used DVS device.



**Figure 3.** (a) Sample pan with a sample in the chamber; (b) detail of dynamic vapor sorption (DVS) manifold; (c) DVS device with computer.

This study is specifically focused on the use of this system while determining the sorption characteristics of aerated concrete. For this purpose, it is necessary to capture the sample's weight response in multiple points with different relative humidities. The sample of the material is placed in the measuring chamber, and the relative humidity is precisely set for each point by mixing dry and saturated gas in the required ratios. The equilibrium humidity point is reached when the sample weight had stabilized. The measurement can take several days, and the DVS system is equipped with automated procedures that ensure continuity of the entire measurement without user intervention. The user selects individual equilibrium points of relative humidity by setting their parameters, and the system automatically performs the entire measurement.

The equilibrium point is in a steady state when the derivative of mass over time is zero. With regard to the super sensitive and accurate scale and its specifics, the exact weight is practically unattainable. The DVS system therefore determines the duration of the sorption time (TIME method) or it allows to set the value for the mass derivation over time in which it is considered to reach a steady state (DMDT method). Both methods find their application. The TIME method, with a precisely set time, is especially suitable when comparing the overall response of the sorption kinetics for several materials. When using the DMDT method, measuring the same materials may have a different total experiment time. Translating generated graphs then makes data post-processing more complex. The DMDT mode is more suitable to measure the moisture equilibrium points, where the monitored value represents the change in weight over time. In addition to the limit value, which is considered to be a steady state, the DMDT mode contains three other parameters. The first parameter is the length of stability, which represents the time required for the mass derivative to become equal or lower than the pre-set DMDT value. When the value of the derivative falls below the limit value, the timer is set off. If the further evolution of the mass does not exceed this limit value and reaches the total length of time required to achieve stability, the system assumes the equilibrium point has been reached and moves onto the next point. If the maximum value of the derivative is exceeded during this time, the device resets the timer and when the derivative falls below the limit, the counting of time starts again. The minimum duration indicates the time from which the derivative

is calculated. It is not always possible to realize the DMDT criterion, and the maximum duration of the step is then limited by the last parameter, indicating the maximum step duration. If the criterion is still unattained, the measurement moves to the next point after the maximum time is reached. Sometimes it is appropriate to use a combination of both fixed-duration and DMDT modes for the measurement.

The Surface Measurement Systems DVS Resolution was used to measure the sorption parameters. Each bulk density was measured several times. The weight of the samples ranged from 36 to 55 mg. Initially, the samples were placed in the desiccator with a potassium sulphate solution with a relative humidity at 97% for two weeks to saturate the samples naturally. Subsequently, the cycle of sequential desorption and sorption was measured twice for each sample. Detailed steps of the method used are shown in Table 4, in the column of the first and second measurement. After measuring the first two cycles, the samples were stored in the laboratory for about a month. They were then placed in the desiccator again, with a potassium sulphate solution, for one week. After conditioning, one cycle was repeated, including desorption and sorption, with a four-hour drying period in between. This procedure is captured in Table 4.

**Table 4.** DVS method settings.

First and Second Measurement (Two Cycles)								Third Measurement (One Cycle)			
Stage	Criterion	Criterion Value (min; %/min)	Relative Humidity (%)	Stage	Criterion	Criterion Value (min; %/min)	Relative Humidity (%)	Stage	Criterion	Criterion Value (min; %/min)	Relative Humidity (%)
0	dm/dt	0.002	97	19	dm/dt	0.0002	95	0	dm/dt	0.002	97
1	Time	120	95	20	Time	120	90	1	Time	120	95
2	dm/dt	0.0002	95	21	dm/dt	0.0002	90	2	dm/dt	0.0001	95
3	Time	120	90	22	dm/dt	0.0002	85	3	Time	120	90
4	dm/dt	0.0002	90	23	dm/dt	0.001	80	4	dm/dt	0.0002	90
5	dm/dt	0.0005	85	24	dm/dt	0.001	70	5	dm/dt	0.0002	85
6	dm/dt	0.001	80	25	dm/dt	0.001	60	6	dm/dt	0.0002	80
7	dm/dt	0.001	70	26	dm/dt	0.001	40	7	dm/dt	0.0005	70
8	dm/dt	0.0005	60	27	dm/dt	0.001	20	8	dm/dt	0.0005	60
9	dm/dt	0.0005	40	28	dm/dt	0.001	0	9	dm/dt	0.0005	40
10	dm/dt	0.0005	20	29	dm/dt	0.001	20	10	dm/dt	0.0005	20
11	dm/dt	0.0005	0	30	dm/dt	0.001	40	11	dm/dt	0.0002	0
12	dm/dt	0.0005	20	31	dm/dt	0.001	60	12 <sup>1</sup>	Time	240	0
13	dm/dt	0.0005	40	32	dm/dt	0.001	70	13	dm/dt	0.0002	0
14	dm/dt	0.0005	60	33	dm/dt	0.001	80	14	dm/dt	0.0005	20
15	dm/dt	0.0005	70	34	dm/dt	0.0002	85	15	dm/dt	0.0005	40
16	dm/dt	0.0005	80	35	dm/dt	0.0002	90	16	dm/dt	0.0005	60
17	dm/dt	0.0005	85	36	dm/dt	0.0002	95	17	dm/dt	0.0005	70
18	dm/dt	0.0002	90					18	dm/dt	0.0002	80
								19	dm/dt	0.0002	85
								20	dm/dt	0.0002	90
								21	dm/dt	0.0001	95

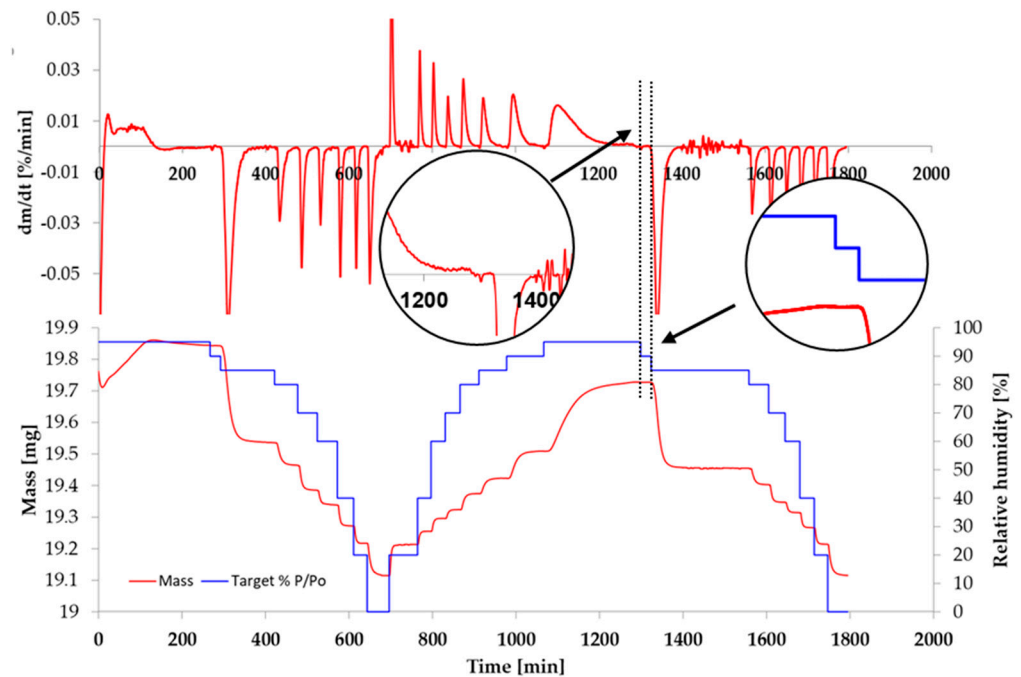
<sup>1</sup> Drying period at 105 °C.

The desorption curve included eight relative humidity points of 95, 90, 85, 80, 60, 40, 20 and 0%. The sorption curve contains the same values in reversed order. Prior to the measurement itself, the sample in the DVS system was conditioned in a 97% relative humidity environment to eliminate moisture loss during the sample transfer from the desiccator to the DVS.

The individual points on the curve used the DMDT criterion. For relative humidity points up to 80%, the DMDT derivation value was set to 0.0005%/min as a criterion for reaching the steady state. The criterium value for humidity from an 80% relative humidity was lower (0.0002%/min) to achieve a higher accuracy. The DMDT criterion has the advantage of saving a measurement cycle time at a low relative humidity, where rapid stabilization occurs. The DMDT criterion monitors the change in the weight of the sample over time. However, there is a risk of a slower sorption response of the material, which



would distort the result at a higher humidity region. Only a small change in relative humidity will start to show after more than a 20 min interval time at high levels. Thus, with short stability intervals, the incorrect weight may be considered stable and the method would proceed to the next point. Such an example can be seen in Figure 4. It may happen that after reducing the relative humidity from 95% to 90%, the weight of the sample still does not decrease significantly enough for some time and the DMDT value practically does not change. Therefore, this state is considered to be steady and thus the equilibrium point at a 90% relative humidity can acquire the same or even higher equilibrium humidity value than the sorption point at a 95% relative humidity; but, this is not practically possible.



**Figure 4.** Sorption kinetics of the AAC and an example of a delayed response error.

To avoid this error, it is advisable to insert a fixed time interval, which requires the necessary exposure of the sample in a lower concentration environment until layers of the sample weight begin to show. Only after the fixed time interval is the DMDT criterion switched on, which will then be terminated by reaching the equilibrium point. The error preceding the configuration point used for this material is shown in Table 4.

The measurements were performed in nitrogen atmosphere at a gas volume flow of 200 standard cubic cm per min (SCCM). The incubator temperature was set at 25 °C, which provided a sorption temperature of approximately 24.5 °C. The preheater was also used in the third measurement cycle drying the sample at 105 °C for 4 h between the desorption and sorption cycles.

### 3. Experimental Measurements

#### 3.1. Bulk Density Measurement

The bulk density of the samples was determined by measuring the dimensions of the samples and their mass. The dimensions of the samples were measured with a caliper to the nearest 0.1 mm. Weight was determined with a laboratory scale with an accuracy of 0.1 g. Samples in the shape of the square with dimensions of 100 mm × 100 mm × 20 mm, with an average weight between 85 and 128 g, were dried in an electric oven at 105 °C for 48 h before performing the measurement of the mass. The determined bulk density of the samples is shown in Table 5.

**Table 5.** The samples' density measurements.

	Width (mm)	Length (mm)	Thickness (mm)	Mass (g)	Density (kg m <sup>-3</sup> )
AC-4_1	100.5	100.4	19.8	86.16	431.6
AC-4_2	100.8	100.2	19.7	85.98	432.5
AC-4_3	100.9	100.8	19.7	86.25	429.6
AC-4_4	100.9	100.8	19.7	86.08	428.8
AC-4_5	100.7	100.7	19.7	85.99	430.3
AC-4_6	101.1	100.4	19.6	86.65	435
average				86.19	431.3
deviation				0.228	2.05
AC-5_1	100.5	101.3	20.3	105.14	509.1
AC-5_2	100.8	101.2	20.1	105.59	514.5
AC-5_3	100.5	101.5	19.9	105.93	521.3
AC-5_4	100.6	100.6	19.4	105.29	535.4
AC-5_5	100.6	101.1	19.9	103.64	513.4
AC-5_6	101.6	101.1	20	104.57	509.3
average				105.03	517.2
deviation				0.747	9.12
AC-6_1	100.8	100.7	19.9	126.59	628.5
AC-6_2	100	100.8	19.6	126.38	638.2
AC-6_3	99.9	100.8	19.7	126.17	635.8
AC-6_4	100	100.6	19.9	126.15	632
AC-6_5	99.9	100.6	19.8	127.43	642.1
AC-6_6	100.5	100.8	19.7	127.48	637.9
average				126.7	635.7
deviation				0.554	4.43

### 3.2. Equilibrium Points Obtained by Conventional Methods

The data measured using the three conventional methods are presented in this section, both expressed in the form of values and charts. The results measured in the desiccators with acid solutions are shown in Table 6. Each of the points was measured for at least two weeks until the sample reached a steady state.

**Table 6.** Results of the sorption in sulfuric acid solutions.

	Desorption				
	Sorption			80	50
	50	80	95		
AC-4_1	1.11	2.16	3.92	2.61	1.66
AC-4_2	1.04	1.69	3.33	2.08	1.19
AC-4_3	1.15	1.89	3.57	2.27	1.4
AC-4_4	1.17	1.91	3.59	2.34	1.45
AC-4_5	1.16	2.08	3.74	2.52	1.63
AC-4_6	1.04	2.1	3.83	2.57	1.71
average	1.112	1.972	3.663	2.398	1.507
deviation	0.059	0.175	0.212	0.205	0.197
AC-5_1	1.17	2	3.54	2.39	1.46
AC-5_2	1.36	2.18	3.67	2.52	1.56
AC-5_3	1.21	1.97	3.48	2.3	1.32
AC-5_4	1.21	1.94	3.4	2.28	1.35
AC-5_5	1.26	2.1	3.58	2.5	1.56
AC-5_6	1.26	2.05	3.57	2.47	1.57
average	1.245	2.040	3.540	2.410	1.470
deviation	0.066	0.089	0.092	0.103	0.112
AC-6_1	1.27	1.96	3.51	2.38	1.45
AC-6_2	1.37	2.09	3.51	2.44	1.51
AC-6_3	1.46	2.12	3.59	2.51	1.6
AC-6_4	1.4	2.07	3.54	2.43	1.55
AC-6_5	1.37	2.14	3.61	2.57	1.69
AC-6_6	1.32	2.14	3.57	2.53	1.66
average	1.365	2.087	3.555	2.477	1.577
deviation	0.065	0.068	0.042	0.071	0.091

Table 7 shows the results from the desiccators with saturated salt solutions. The time of the individual points is presented in the table. The total measurement time was approximately 77 days, which represents 11 weeks. Figure 5 shows the course of the measured values. While the lower relative humidity levels stabilized rapidly, the higher humidity regions needed a longer time period.

Table 7. Results of the salt solution tests.

Density	Samples Set	Mass	Desiccators						
			Sorption				Desorption		
			53.5	75.4	84.7	94	84.7	75.4	53.5
430	1	20.589	1.282	1.681	1.982	3.002	2.53	2.312	2.03
	2	21.524	1.375	1.77	2.035	2.978	2.527	2.281	2.016
	a <sup>1</sup>		1.329	1.725	2.008	2.99	2.529	2.297	2.023
	d <sup>2</sup>		0.046	0.045	0.027	0.012	0.002	0.015	0.007
520	1	20.406	1.073	1.387	1.71	2.612	2.215	2.004	1.725
	2	28.594	1.182	1.542	1.826	2.71	2.336	2.137	1.882
	a <sup>1</sup>		1.128	1.465	1.768	2.661	2.276	2.071	1.803
	d <sup>2</sup>		0.054	0.078	0.058	0.049	0.061	0.066	0.078
630	1	28.903	1.384	1.751	2.027	2.906	2.398	2.083	1.775
	2	29.535	1.364	1.737	2.011	2.854	2.36	2.116	1.815
	a <sup>1</sup>		1.374	1.744	2.019	2.88	2.379	2.099	1.795
	d <sup>2</sup>		0.01	0.007	0.008	0.026	0.019	0.017	0.02
duration (days)			12	10	11	24	7	7	6

<sup>1</sup> Average; <sup>2</sup> standard deviation.

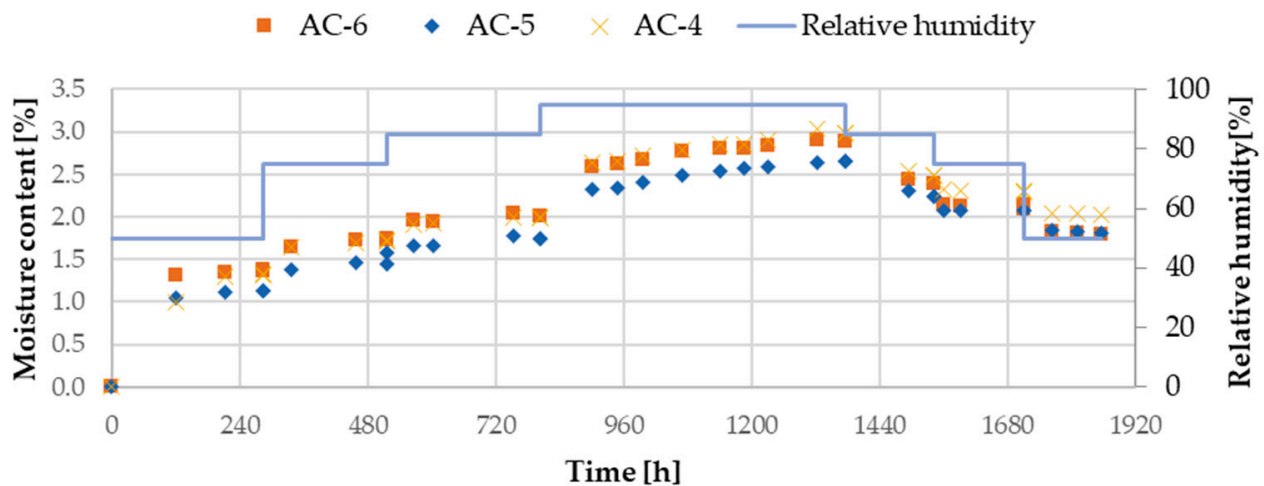


Figure 5. Moisture content of the samples in the desiccators with saturated salt solutions.

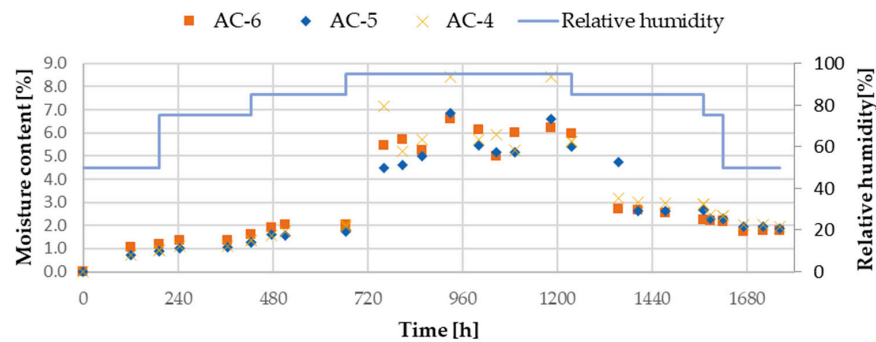
Table 8 shows the results measured in the environmental chambers. The time of individual points is again expressed in the table. The entire measurement lasted 74 days, which is 11 weeks. Figure 6 displays the time course of the measurement. There was a problem with the climate chamber at a high relative humidity. Condensation occurs on the inner walls of the chamber and the dripping condensate caused contamination of the samples. Samples were later protected by foil, but the measured weights still showed large oscillation and deviation. Although the climate chamber is designed for operation at temperatures between  $-20$  to  $150$  °C, and the relative humidity lever between 20 to 95%, some areas show considerable instability and a limited duration of operation. These limitations need to be followed during usage according to the working diagram of the

chamber. The short operation time of the required conditions were compensated for by rotating the samples between the two chambers.

**Table 8.** Results of environmental chamber tests.

Density	Samples Set	Mass	Environmental Chamber						
			Sorption				Desorption		
			50	75	85	95	85	75	50
430	1	22.939	0.998	1.447	2.093	5.728	2.838	2.485	1.944
	2	21.242	0.829	1.257	1.93	5.513	2.665	2.368	1.94
	a <sup>1</sup>		0.913	1.352	2.011	5.62	2.751	2.426	1.942
	d <sup>2</sup>		0.085	0.095	0.081	0.108	0.087	0.058	0.002
520	1	26.687	0.794	1.173	1.641	5.268	2.496	2.256	1.885
	2	26.082	0.978	1.346	1.791	5.582	2.557	2.285	1.883
	a <sup>1</sup>		0.886	1.259	1.716	5.425	2.526	2.27	1.884
	d <sup>2</sup>		0.092	0.086	0.075	0.157	0.031	0.015	0.001
630	1	33.247	1.194	1.627	1.97	6.013	2.496	2.184	1.739
	2	34.249	1.183	1.609	1.942	5.927	2.435	2.134	1.79
	a <sup>1</sup>		1.188	1.618	1.956	5.97	2.466	2.159	1.764
	d <sup>2</sup>		0.006	0.009	0.014	0.043	0.031	0.025	0.026
duration (days)			8	10	10	24	14	2	6

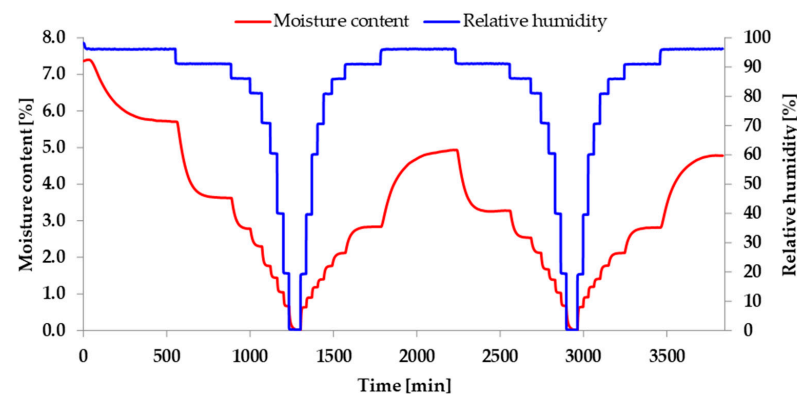
<sup>1</sup> Average; <sup>2</sup> standard deviation.



**Figure 6.** Moisture content of the samples in the environmental chambers.

### 3.3. Dynamic Vapour Sorption Measurements

The result obtained by the DVS device is shown in Table 9, including the total duration of the individual measurements for the sorption and desorption cycle. An example of the time course of the sorption measurement using the DVS device is shown in Figure 7.



**Figure 7.** Sorption kinetic of the AC-4 sample at the first and second cycle.

Table 9. Results of the dynamic vapor sorption.

Density	Relative Humidity	Measurement 1 and 2				Measurement 3		Min	Average		Max	Standard Deviation	
		S	D	S	D	S	D		S	D		S	D
	mass	44.2999 mg				43.2445 mg							
AC-4	0	0.000	0.000	0.000	0.000	0.000	0.570	0.000	0.000	0.190	0.570	0.000	0.269
	20	0.636	0.665	0.644	0.672	0.660	1.096	0.636	0.647	0.811	1.096	0.010	0.202
	40	0.901	1.043	0.910	1.029	0.958	1.479	0.901	0.923	1.184	1.479	0.025	0.209
	60	1.185	1.438	1.196	1.383	1.252	1.896	1.185	1.211	1.572	1.896	0.029	0.230
	70	1.400	1.761	1.412	1.669	1.461	2.198	1.400	1.424	1.876	2.198	0.026	0.231
	80	1.765	2.297	1.775	2.120	1.866	2.635	1.765	1.802	2.351	2.635	0.045	0.214
	85	2.120	2.781	2.118	2.536	2.185	3.103	2.118	2.141	2.807	3.103	0.031	0.232
	90	2.836	3.620	2.814	3.274	2.699	3.970	2.699	2.783	3.621	3.970	0.060	0.284
	95	4.930	5.705	4.780	4.930	3.671	5.510	3.671	4.460	5.382	5.705	0.561	0.329
	duration (hours)	64				64							
	mass	36.6564 mg				36.3126 mg							
AC-5	0	0.000	0.000	0.003	0.003	0.000	0.468	0.000	0.000	0.001	0.157	0.002	0.220
	20	0.541	0.568	0.550	0.573	0.473	0.986	0.473	0.473	0.521	0.709	0.034	0.196
	40	0.776	0.907	0.784	0.881	0.699	1.315	0.699	0.699	0.753	1.034	0.038	0.199
	60	1.012	1.241	1.018	1.145	0.929	1.638	0.929	0.929	0.986	1.341	0.041	0.214
	70	1.183	1.502	1.188	1.342	1.092	1.925	1.092	1.092	1.154	1.589	0.044	0.246
	80	1.477	1.985	1.477	1.658	1.415	2.326	1.415	1.415	1.457	1.990	0.029	0.273
	85	1.740	2.383	1.735	1.936	1.688	2.610	1.688	1.688	1.721	2.310	0.023	0.280
	90	2.209	3.057	2.178	2.405	2.085	3.139	2.085	2.085	2.157	2.867	0.053	0.328
	95	3.303	4.437	3.240	3.303	2.998	3.961	2.998	2.998	3.181	3.901	0.132	0.465
	duration (hours)	65				54							
	mass	54.0236 mg				53.1790 mg							
AC-6	0	0.000	0.000	0.004	0.004	0.000	0.234	0.000	0.001	0.080	0.234	0.002	0.109
	20	0.612	0.640	0.613	0.637	0.597	0.924	0.597	0.607	0.733	0.924	0.007	0.135
	40	0.855	0.962	0.856	0.934	0.848	1.215	0.848	0.853	1.037	1.215	0.003	0.126
	60	1.093	1.337	1.087	1.204	1.083	1.516	1.083	1.087	1.352	1.516	0.004	0.128
	70	1.266	1.648	1.254	1.410	1.243	1.770	1.243	1.254	1.609	1.770	0.009	0.150
	80	1.564	2.167	1.547	1.735	1.574	2.171	1.547	1.562	2.024	2.171	0.011	0.205
	85	1.827	2.612	1.810	2.024	1.856	2.509	1.810	1.831	2.382	2.612	0.019	0.256
	90	2.291	3.341	2.274	2.525	2.341	3.110	2.274	2.302	2.992	3.341	0.028	0.343
	95	3.431	4.818	3.391	3.431	3.413	4.148	3.391	3.412	4.132	4.818	0.016	0.567
	duration (hours)	55				67							

#### 4. Result Analysis and Discussion

The analysis and comparison of the individual methods are based on the data measured by each method. Figures 8–10 show the averaged values of the equilibrium points forming the sorption and desorption isotherms. The measurement results reached applying the DVS method were projected onto the graph in the form of envelope curves. They are formed by lines connecting the minimum and maximum values of all the performed measurements. The area between the curves is highlighted blue and it determines the possible equilibrium states of the material. Data obtained using the conventional methods were projected onto the same graph in contrast to the results of the dynamic sorption method. The results obtained applying individual methods are approximately identical and with same tendencies. They differ significantly in the endpoints, representing the maximum value. In the legend, the sorption curves are denoted by the prefix S, and the desorption curves are denoted by the prefix D. The results obtained from the desiccators with salt solutions are denoted by the suffix DE. The suffix EC indicates the results obtained from the environmental chamber. The DVS system uses the DVS suffix for its results and the desiccators with sulfuric acid solutions use the SA suffix.

Figure 8 shows the results of the AC-4 sample measurements. The values obtained by the individual methods have small differences between each other. The conventional methods closely follow the curves of the DVS method, within the range of 50 to 85% relative humidity. The biggest deviation occurs in the area of high relative humidity. While the

upper curve values of the desiccator methods end at the value determined by the DVS method, the samples from the climate chamber contaminated with condensation reach values of the level of the desorption envelope curve.

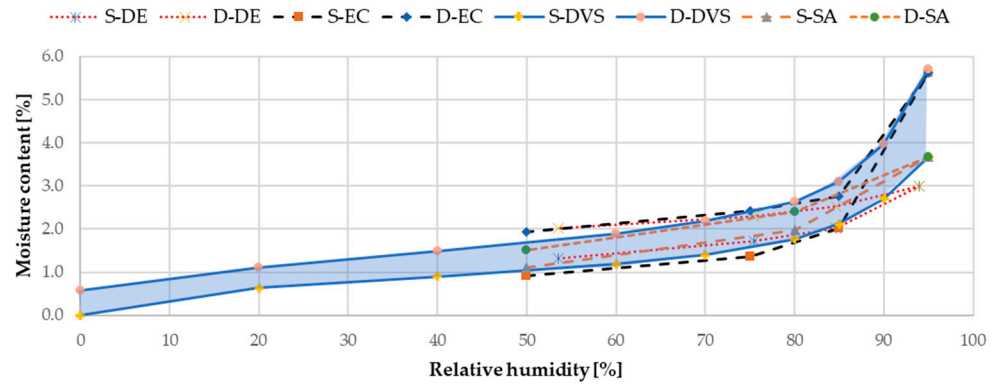


Figure 8. Sorption and desorption equilibrium points for the AC-4 sample.

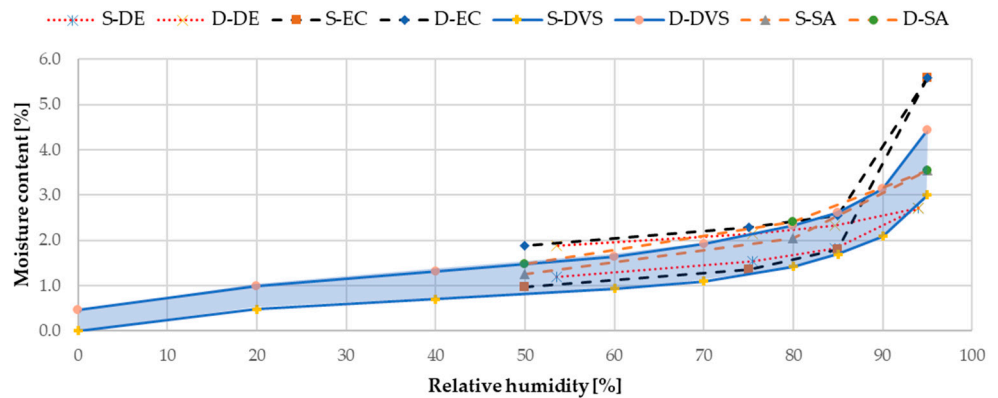


Figure 9. Sorption and desorption equilibrium points for the AC-5 sample.

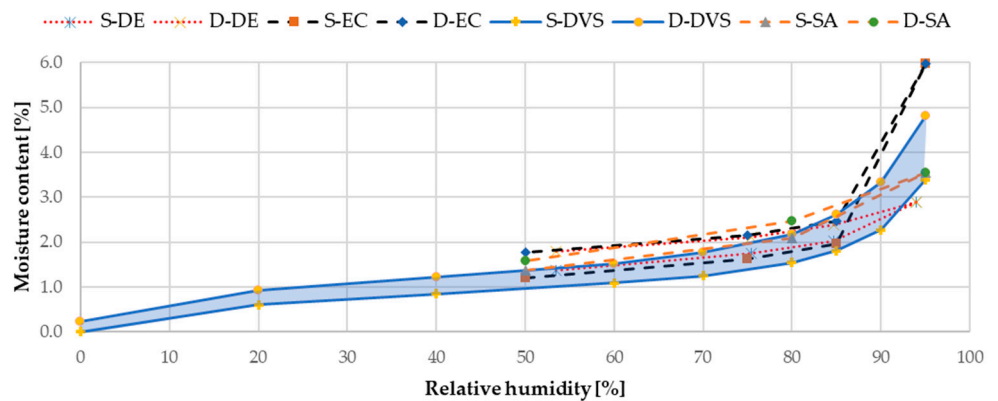


Figure 10. Sorption and desorption equilibrium points for the AC-6 sample.

Figure 9 shows the results for the AC-5 sample measurements. Most of the results show identical tendencies, and the envelope of sorption and desorption in the region of 50 to 85% relative humidity approximately copies the tendencies observed performing with conventional methods. The most significant deviations are similar to the AC-4 samples in the area of high relative humidity. The values of the samples from the environmental chamber are distorted at the last point by the influence of the moisture condensation accumulated in the chamber.

Measurement results of the AC-6 samples are shown in Figure 10. There is a slight shift in isotherms. The sorption and desorption envelope curves lie below the values obtained from the conventional methods. The last point set using the environmental chamber is again distorted.

Based on the data obtained, it can be seen that, for all three bulk densities, the tendencies are mostly identical in the whole range and only small shifts in the results are observed in the measured area. It should be noted that the shift may be caused by the high variability of the aerated concrete material in the samples. The sample volume used for the DVS system is only about one thousandth that of the sample volumes used for the climate chamber or saline desiccators, and about one three thousandth of the sample used for the acid desiccators. The representative volume for DVS can thus be skewed with local anomalies in the sample. For these reasons, it is therefore advisable to test more samples taken from different locations of the large sample. The deviations in the individual methods at the highest humidity point are caused by several factors. Distortion of samples in the environmental chambers is caused by condensate accumulated on the walls of the environmental chamber, which dripped uncontrollably and could have wetted the surface of the samples. The desorption curve in the DVS measurement acquires higher values at the highest point, as is presented. The desorption curve starts at 97% relative humidity, so the peak of the sorption and desorption curve does not meet in one point as the other methods do. Due to these two reasons, the highest points are not comparable.

Another important aspect of the sorption measurement is the time duration. The total time of measurements for each method is given in Table 10. The results were obtained fastest using the DVS method. These were obtained comprehensively in two to three days for the whole spectrum throughout the range of 17 points. For other methods, where there were only seven points, the successive measurements lasted approximately 80 days.

**Table 10.** Total time duration of the measurements.

Sample	Method			
	Sulfuric acid Solution	Salt Solution	Environmental Chamber	Dynamic Vapor Sorption *
AC-4				27–64
AC-5	1700	1854	1762	25–65
AC-6				24–67

\* More measurements were obtained; the length depends on the number of cycles and accuracy. Approximate ranges for one measurement cycle are given.

The previous sections describe the measurement results and compare their determined sorption/desorption isotherms. The results show sound compatibility between the sorption/desorption isotherms obtained using two conventional methods and the DVS method throughout the entire relative air humidity range. The absolute differences in the moisture content values of the investigated range are acceptably small. Additionally, the following tendencies were observed: at the 430 bulk density level, the conventional method leads to practically the same moisture content values for a RH > 50%, and at the 520 level for a RH > 70%; however, at the 630 level, for the whole RH range investigated, the conventional method gives slightly higher values than the DVS method. It should be noted that in the area of higher relative humidity the results do not differ significantly, as was found here and strongly pointed out in previous research studies (e.g., in [34,35]). At the highest humidity level measured (RH = 97%), the climatic chamber method provides a slightly higher (of about 1%) and the desiccator a slightly lower (up to 1%) equilibrium moisture content for all bulk densities in comparison to the DVS method. This represents a very low and highly acceptable deviation between the results measured.

Regarding the test methods used, the results obtained provide several points for discussion:

- Desiccators: Represent a simple and undemanding method that can be implemented in every laboratory without any special investment. Basically, it is possible to use

sulfuric acid or solutions of salts, which are currently more preferred because of safety. If the laboratory can maintain a stable room temperature, the costs of these measurements are minimal. The desiccator methods show less accuracy and greater variation in the results. It is due to the influence of the ambient temperature in the room where the desiccators are located and its changes. To eliminate these phenomena, test dishes are used that can be covered while the desiccator is open for the sample to be removed. For stability, it is possible to place the desiccators in an environmental chamber, or to use the climatic chamber directly for conditioning the samples, but this approach increases the measurements cost.

- Environmental chamber: May improve the thermal stability of the environment, yet it brings other problems in terms of the accuracy of the humidity control and its stability. As indicated in the provided tests, the environmental chambers had to be regularly defrosted during the measurement process. This procedure complicates the measurement, and it requires alternating between two chambers after a few days of conditioning. The samples always needed to be transferred to the next chamber during the weighing, where conditioning continued until the first chamber was defrosted and vice versa. At low humidity levels, this approach may be suitable, but at higher relative humidity levels, it could lead to oscillations of mass because each chamber has its own deviation of humidity and temperature sensors. Environmental chambers set to a high relative humidity are prone to condensation of water vapor on the chamber walls and samples as well. Condensate can drip onto the samples and distort the effect of the sorption. We have observed these errors at the highest point of relative humidity of 95% in our chambers. Condensate droplets lead to oscillations of samples mass. Later, a small piece of PE foil was used to prevent direct dripping of condensate from the chamber ceiling onto the samples.
- Operating temperature: There should be an absolutely constant temperature during the whole measurement cycle in the laboratory. In an actual, real operation, even an air-conditioned room with a set temperature of 23 °C will still slightly change its temperature during the year and the air conditioners also oscillate in cycle on and off. Any presence of temperature changes leads to changes in humidity followed by a change in mass. Temperature changes in the room could have influenced the scale as well. Heat-compensated scales are commonly used for weighing in laboratories. For long-term measurements, this fact may still introduce some inaccuracies in the final results. This effect can be suppressed with the use of a larger sample weight where the errors caused by the temperature compensations should be very small. However, larger samples prolong the measurement time and make it difficult to use the samples in enclosed dishes.
- Time management: Both conventional methods require a long time and samples weighing in regular intervals. The measurements are very time consuming, as it can take several weeks to reach each level of moisture equilibrium. A larger series of samples are used to make the measurements more effective, where the samples are simultaneously exposed to different environments. Alternatively, if the character of the material allows and the result is not distorted, it is possible to divide the sample into smaller parts, which increases the area of the sample and intensifies its saturation more quickly. The area and especially the thickness of the samples often have a non-negligible dimension, therefore the time for the moisture to enter the sample to redistribute and reach equilibrium state is significantly prolonged.
- Samples contamination: Another phenomenon affecting the accuracy and reproducibility of results is the opening of the chamber or desiccators. The time until the dish is closed takes a few seconds, or due to the size of the sample, it is not always possible to use closable dishes, as in the case of our experiments. The degree of contamination bias together with the accuracy of the weighing depends on the experience of the operator, the measurement technique and the type of measured samples.



- **Measurement stability and suitability:** Stabilizing the weight of the sample while applying these methods takes considerable amount of time, and constant collection and weighing of samples can damage them if handled carelessly. This risk rises with low-density samples, which are more fragile and prone to disintegrate. The possibility of simultaneous exposure of several samples and parallel measurement brings a slight improvement to the measurement stability. However, even here it is necessary to mind the optimal capacity of the desiccator or chambers, so the samples do not interfere with the moisture. In general, conventional methods work well at a lower relative humidity for larger and higher bulk density samples. At a higher humidity, the variance of the measured values increases, and the results are prone to errors due to limitations in the equipment and methodology.
- **DVS method:** The results for the whole curve can be obtained within a short period, which can significantly save measurement time, even when it is necessary to test a large number of samples. Continuous mass measurements and a shorter measuring time together with a high accuracy scale enables a higher accuracy of the results. The sample is not contaminated during measurement and there is no risk of damage during weighing. The limitation of this method stems from the significantly higher purchase price of the equipment compared to the air-conditioning chambers or desiccators, as well as the consumption of energy and measuring gas.
- **Accuracy and repeatability:** The DVS system is characterized by a higher accuracy and repeatability of results compared to conventional methods. The robustness of the system is also ensured by the automation of the entire process. Just at a very high relative humidity do changes in the moisture content of the material take place very slowly. These changes, expressed as the change in mass moisture concentration per minute, take place at a very high humidity, and in order of the resolution of the system itself and its error. Therefore, at a very high relative humidity, the DVS system also shows higher variance, even when increasing the measurement accuracy of the DMDT criterion. This will not affect the measurement accuracy, but its reliability slightly decreases. Despite this fact, dynamic vapor sorption appears to be a very progressive and robust method that surpasses conventional methods in many ways. Continuous weighing and shorter measuring times together with the high accuracy of the scale result in a higher accuracy. The sample is not contaminated during measurement and there is no risk of damage during weighing.

## 5. Conclusions

The presented research aims to compare the application reliability of suitable measuring methods used for sorption and desorption isotherm measurements of building materials, particularly exemplified on AAC samples. The paper describes the reasoning behind the usefulness of all the applied methods, through an analysis of their specifics and a comparative assessment of their application.

The sorption/desorption isotherms of AAC produced with two conventional methods and the dynamic vapor sorption test method were determined. These parameters are expected to provide the required inputs of the hygric properties, both for research and practical issues. The proper material characteristics are a highly important issue for reliable hygrothermal calculations, which still has crucial importance in the building physics and building engineering fields. This may lead to improve the quality of studies of the hygrothermal performance of building envelopes based on the commonly used AAC in building practice.

As result, the compatibility between all the results obtained is found to be in good agreement. This also means that, compared with previously published data (e.g., in [34,35]), no significant differences could be found at the different bulk densities that were analyzed. As result, the consistency of the procedures in measurement methods and test principles should be carefully considered, specifically at a high relative humidity.

Laboratory testing justified the statement that the DVS method represents a proper new, powerful and reliable technique. One of the key attributes is the factor of no risk of contamination of the environment between the points and measurements, as well as a fully automated control of the operation conditions and their stability at the maximal required level. The use of a small sample size in combination with a very sensitive scale and large volume flow means the results can be acquired in a significantly shorter period of time compared to the conventional and commonly used standard test methods.

When conventional test methods are employed, there are a few uncertainties regarding the data obtained at a higher humidity, which have been discussed in detail due to the results achieved by previous studies; these have not been proved by this study and it appears that the key aspect of disproportions is caused by the limits of each conventional method employed and the particular approach. Therefore, the reasoning behind the methodology developed in this paper is expected to provide the reference for measuring the sorption/desorption isotherms of building materials. That is why, for future studies, this should be carefully considered in the laboratory.

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