Determination of Moisture Properties for Materials Exposed to the Indoor Air

Kaisa Svennberg

Report TVBH-3042 Lund 2003
Department of Building Physics

Licentiate thesis

LUND INSTITUTE OF TECHNOLOGY
Lund University

To my father Renny, who would have asked all the questions,
and Henrik, who gave me the confidence to seek an answer.

Preface

This licentiate[1] thesis is a part of the project ”Relative humidity of the indoor air and on interior surfaces” that was initiated by dr. Lars-Erik Harderup and professor Arne Elmroth at the Department of Building Physics, Lund University. The project is affiliated with the Moisture Research Center at Lund University.

The financial funding by FORMAS – the Swedish Research Council for Environment, Agricultural Sciences and Spatial Planning, SBUF – the Development Fund of the Swedish Construction Industry and Byggrådet Malmö-Lund – the Council of Building Contractors in Malmö-Lund, is gratefully acknowledged.

This work has been done in close collaboration and with the help from a number of people. I want to express my gratitude to all whom in one way or another have contributed.

My special thanks go to:

My supervisors, dr. Lars-Erik Harderup and dr. Lars Wadsö, they have guided me patiently and always encouraging and generously shared both their knowledge and their time.

Professor Arne Elmroth, professor Jesper Arfvidsson and professor Johan Claesson at the Department of Building Physics, Lund University for good discussions and generous help.

All the colleagues at the department of Building Physics and elsewhere for they have always done work and life enjoyable. Agneta Ohlsson, Lilian Johansson and Birgitta Salmi for the practical help they given within the project. Dennis Johansson for his never-ending technical solutions and enthusiasm.

Dr. Lena Eliasson for widening my scientific world and always being a true friend. My mother Inger Gertsdotter and the rest of my worldwide family for their support and practical help. And the three most important people in my life – Henrik, Cecilia and Renny - their love and care made all the difference.

Lund   October, 2003

Kaisa Svennberg

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[1] The licentiate degree is a Swedish academic degree, midway between a M. Sc. and a Ph.D., equal of two years scientific work.

Abstract

Building materials as well as furniture exposed to variations in the surrounding climate will absorb moisture when the relative humidity (RH) increases and desorb when the RH decreases. Other researchers have shown that the correlation between simulated and measured moisture conditions of the indoor air is much better if the moisture buffering performance of surface materials is taken into account. Predictions of moisture related damage and optimization of energy and ventilation systems would improve if the moisture buffering performance was better described. More data on moisture properties for surface materials is needed in the continuing development of advanced hygrothermal calculations.

The interaction between surface materials and the humidity of the indoor air is the main objective of this project. Measurement methods to determine the material properties governing the moisture buffer performance of different surface materials are the scope of this report.

The report includes a proposed model to categorize materials based on their external and internal moisture properties, a literature survey considering methods to determine sorption isotherms and water vapor permeability and a discussion about important factors for the measurements: climate control, edge masking, surface mass transfer, area determination and equilibrium criteria.

We have succeeded in developing two inexpensive measurement methods, the vial-cup method for water vapor permeability measurements and the jar method for determining sorption isotherms. The vial-cup method is an inverted cup method, based on crimp-cap vials, for thin and highly permeable materials such as textiles and paper. The advantages with the vial-cup method are the lower interior resistance of the method together with a convenient way to fasten thin samples. The jar method is an improved method, based on glass jars with saturated salt solutions, to measure sorption isotherms. The sample is kept in the same ambient climate within the jar during the complete measurement period giving fewer disturbances. Another advantage is the setup that facilitates step-changes in RH. The jar method was validated on materials with known moisture properties (MCC and bentonite clay) and used in an investigation on plasterboard - paint system. The paint system showed unexpectedly to have a moisture capacity that could not be neglected.

Keywords: building materials, furniture, textiles, paper, indoor air, laboratory methods, moisture buffering performance, sorption isotherms, water vapor permeability.

Sammanfattning

Byggnadsmaterial och inredning som utsätts för fuktighetsvariationer i den omgivande luften kommer att ta upp fukt när den relativa fuktigheten (RF) stiger och på motsvarande sätt avge fukt när RF sjunker. Andra forskare har visat att överensstämmelsen mellan beräknade och uppmätta fukttillstånd i inomhusluften blir bättre om den fuktbuffrande förmågan hos ytmaterialen tas med beräkningarna.

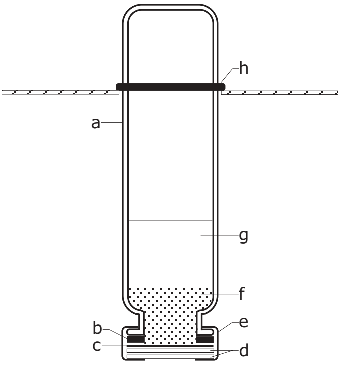
Mer kunskap om fuktbuffring skulle medföra att fuktrelaterade skador skulle kunna förutsägas med större noggrannhet och att uppvärmnings- och ventilationssystem skulle kunna optimeras bättre. För mer avancerade beräkningar av fukt och temperaturförhållande krävs mer värden på fuktegenskaper för ytmaterial.

Syftet med detta projekt är att undersöka samverkan mellan ytmaterial och luftfuktigheten inomhus. Omfattningen av denna rapport är begränsad till mätmetoder för att bestämma de fuktegenskaper som styr fuktbuffringen i ytmaterial.

Denna rapport innehåller ett förslag till modell för att dela in ytmaterial i olika grupper som baseras på deras ytegenskaper och inre fuktegenskaper, en litteraturundersökning av metoder för att bestämma sorptions isotermer och ångpermeabilitet och en diskussion om viktiga faktorer vid experimentella försök: klimatkontroll, kantförsegling av provkroppar, ytövergångsmotstånd, areabestämmning och jämviktskriterium.

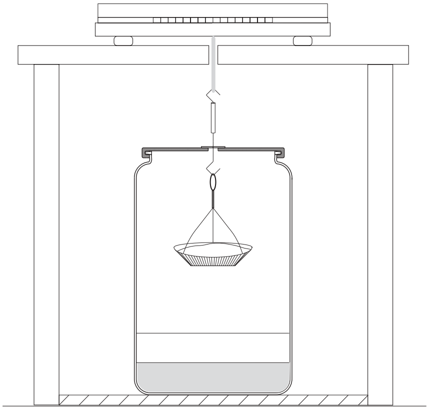
Inom projektet har vi lyckats utveckla två enkla och prisvärda metoder, vial-cup metoden för mätningar av ångpermeabilitets och burkmetoden för bestämning av sorptionsisotermer.

Vial-cup metoden är en omvänd koppmetod som är baserad på standardvialer med krymplock. Metoden är utvecklad för tunna och mycket permeabla material såsom papper och textilier. Fördelarna med metoden är att övergångsmotståndet inne i vialen är mycket lägre än i den traditionella upprättstående koppmetoden. Dessutom är fastsättningen av provmaterialet enklare.



Vial-cup metoden

Burkmetoden är en förbättrad metod för att bestämma sorptionsisotermer baserad på glasburkar och mättade saltlösningar. Provet är inneslutet i ett konstant klimat i burkarna under hela mätperioden, inklusive vägningarna, något som minskar störningarna. Metoden underlättar också stegändringar i RF då utformningen av lock och upphängningsanordning gör det enkelt att flytta provet från en burk till en annan. Metoden har validerats med mätningar på material med välkända fuktegenskaper (MCC och bentonit) och användes i en undersökning på ett system med gipsskiva och färg. Färgsystemet visade sig oväntat ha en fuktkapacitet som inte kan försummas.



Burkmetoden

Nyckelord: byggnadsmaterial, inredning, textilier, papper, inomhus luft, laboratoriemetoder, fuktbuffringsförmåga, sorptionsisoterm, ångpermeabilitet.

Contents

List of appended papers:

Paper I –

A modified cup-method for lightweight and highly permeable materials.
Svennberg, K and Wadsö, L (2003)
Published in *Research in Building Physics,* eds. Carmeliet, Hens & Vermeir , Lisse: Swets & Zeitlinger, ISBN 90 5809 565 7, pp.177-182. Printed with permission from Swets & Zeitlinger.

Paper II –

An experimentally simple method for measuring sorption isotherms.
Wadsö, L., Svennberg, K. and Dueck, A.
Submitted for publication to Drying Technology.

Paper III –

Time-dependent moisture properties for plasterboard with surface coating.
Svennberg, K. and Harderup, L-E. (2002).
Published in *Proceedings of the 9th International Conference on Indoor Air Quality and Climate –Indoor Air 2002,* Vol.IV, pp.66-72. Monterey, CA, USA: Indoor air 2002.

Nomenclature

A_c	Area of condensation	m^2
A_{sj}	Area of the surface material j	m^2
d	thickness	m
d_p	Penetration depth	m
g_{mbj}	Sorbed moisture flux for material j	$kg/(m^2s)$
G_{ms}	Moisture supply	kg/s
M	Mass	kg
m_f	Final mass	kg
m_i	Initial mass	kg
q	Ventilation rate	m^3/s
RH	Relative humidity	%
t	Time	s
t_p	Period time	s, h or days
u	Moisture ratio mass by mass	kg/kg
v	Vapor content	kg/m^3
v_e	Exterior vapor content	kg/m^3
v_i	Interior vapor content	kg/m^3
v_s	Saturation vapor content	kg/m^3
v_{ss}	Saturation vapor content at the surface	kg/m^3
V	Volume of the room	m^3
w	Moisture content mass by volume	kg/m^3
x_p	Active penetration depth	m
Z_v	Vapor resistance	s/m

β_v	Surface moisture transfer coefficient	m/s
δ_p	Water vapor permeability with vapor pressure as potential	m^2/s
δ_v	Water vapor permeability with vapor content as potential	m^2/s
φ	Relative humidity (RH)	$(kg/m^3)/(kg/m^3)$
φ_{und}	Undisturbed relative humidity (RH)	%
ξ	Moisture capacity	kg/m^3
Ω	Equilibrium criteria	s^{-1}

1 Introduction

Materials exposed to variations in the surrounding climate will absorb moisture when the relative humidity (RH) increases and desorb when the RH decreases. The response is to a large extent due to the material composition and structure and to its surface treatment. Already Leonardo da Vinci used this knowledge in a suggested hygrometer. The hygrometer consisted of a common beam balance where one of the scale pans was filled with wax and the other one with a hygroscopic material such as wool or cotton. As the RH increases, the pan with the hygroscopic material adds weight by absorption of moisture whereas the pan with wax does not add any weight since wax is a non-hygroscopic material. The balance will tilt over to the hygroscopic side. This is reversed when the RH decreases.

The objective of this project is to look at the interaction between surface materials in the indoor environment and the relative humidity of the indoor air. Both traditional materials of the building envelope and furniture and other furnishings have been considered. The scope of this report is the measurement methods used to determine the moisture properties for surface materials in the indoor environment.

A widely spread concern for the indoor air quality, which to some extent is determined by the level and fluctuations of the humidity of the indoor air, has been one of the reasons to initiate this project (Bornehag et al., 2001). The progress within hygrothermal and energy modeling another reason. A literature survey conducted earlier in this project shows that to be able to develop the hygrothermal calculation models further, there is a need for data concerning moisture properties for surface materials used in the indoor environment (Harderup, 1998). A more detailed description of the interaction between materials exposed to the indoor air and the moisture condition of the indoor air is necessary, as schematically described in the hygrothermal flow chart in Fig. 1.

Two new measurement methods for determining moisture properties for materials exposed to indoor air have been developed within this project and are presented in this report.

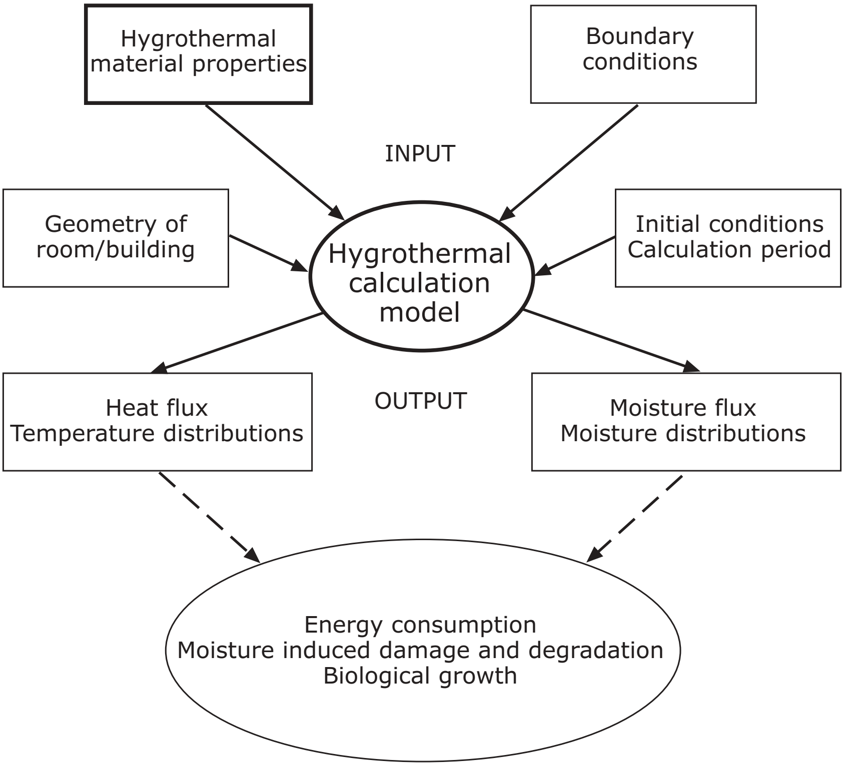


Figure 1. A hygrothermal flow chart, describing the input needed for hygrothermal simulations of a building and the expected use of the output. Adapted from Künzel and Kiessl (1997).

2 The moisture balance of a room

2.1 General description

The moisture balance in a room is determined by the outdoor climate, the moisture supply, the moisture buffering from surface materials of the room and furnishing, the ventilation rate, the possible condensation at surfaces and the variation of these parameters with time. The time scale for the moisture balance of a room can vary from a seasonal, taking the annual variations of the external climate into account, to a diurnal where the interesting time scale is hours to days. In this project, we focus on the short time scales that will be mostly effected by the behavior of the inhabitants (Rode, Grau and Mitamura, 2001).

Equation 1 shows an analytical expression for the change in vapor content, v_i, of the indoor air over time. The temperature dependency for the volume expansion of the air is neglected.

$$\frac{dv_i}{dt} = \frac{G_{ms} - \sum_{j=1}^{n} g_{mbj} A_{sj} - q(v_i - v_e) - \beta_v (v_i - v_{ss}) A_c}{V} \qquad [\mathrm{kg/(m^3\,s)}] \qquad \text{(Eq. 1)}$$

The moisture supply, G_{ms}, is assumed to be composed of two parts. A relatively constant basic supply from plants, inhabitants and pets and short moisture supply pulses that change almost momentarily. These short moisture supply pulses comprise, e.g., cooking, washing, showers and baths, activities that are carried out during a shorter period of the day. The moisture supply is assumed to be changed uniformly within the whole room volume regarded. Using the assumptions above, the variation with time for the moisture supply can be simplified to step variations. The variations in moisture supply and moisture production in Scandinavian dwellings have been studied earlier by for example Norlén and Andersson (1993). It has therefore not been the aim of this project to go deeper into this.

The ability of the interior surfaces to buffer moisture variations of the indoor air is described with, g_{mb}, the moisture absorption or desorption in the different surface materials at a given time. The sorption in materials will dampen the moisture variations of the indoor air. Both the surfaces of the inside of the building envelope such as ceilings, floors and walls as well as the furniture and other furnishing will give an impact on the moisture conditions in the room. For example Plathner and Woloszyn (2002) have shown that the correlation between simulated and measured moisture conditions of the indoor air is much better if the sorption of interior surface materials is taken into account. See Fig. 2.

The interaction between surface materials and the humidity of the indoor air is the main objective of this project. Measurement methods to determine the material properties governing the moisture buffer performance of different surface materials are the scope of this report.

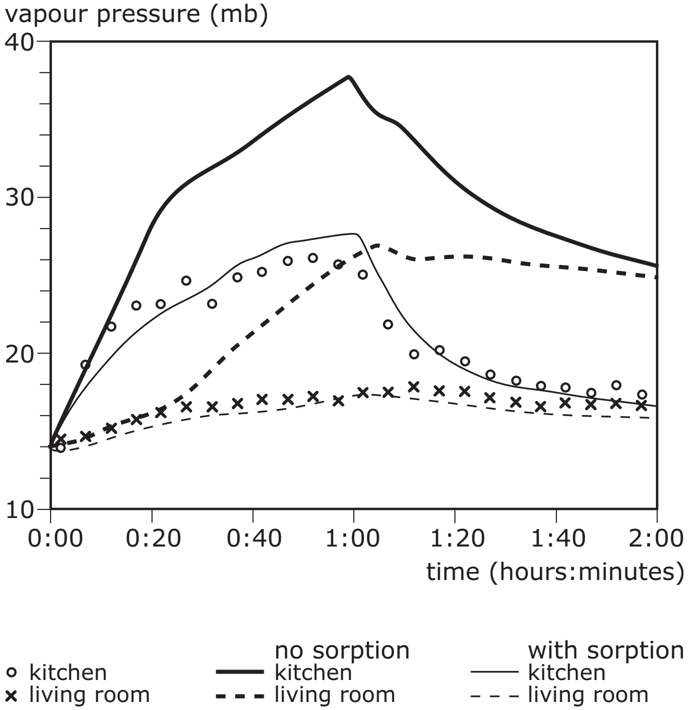


Figure 2. Comparison between measured and calculated moisture conditions of the indoor air. Calculations with the sorption of the surface materials taken into account show better correlation with measured values than the calculations where the sorption was not taken into account. The temperature was set to 24 °C for both calculations and field measurements. Adapted from Plathner & Woloszyn (2002).

The ventilation is crucial for the moisture condition of indoor air. The ventilation flow rate, *q*, is set to a minimum of 0.35 l/s per m^2 floor area in the Swedish building code, but for most application the advice is to have a larger air exchange, e.g., in practice offices will have ventilation flow rates of an approximately 20 l/s if the room has a floor area of approximately 10 m^2 and is occupied by one person. The main purpose of the ventilation is to supply the building and its inhabitants with the sufficient amount of fresh air, remove emissions from activities, people and materials and remove excess moisture, smells and hazardous substances (BBR, 2002). The impact of the ventilation depends on the ventilation rate, and the vapor content of the indoor and outdoor air. If the ventilation rate is very high it will control the RH of indoor air to a very high degree. If on the other hand the ventilation rate is low the impact of moisture buffering in surface materials is larger (Christoffersen, 1996).

The ventilation rate will govern the mean value of the RH variations in the indoor air and the moisture buffer performance will effect the amplitude of the RH variations. It should be noted that moisture buffering in materials could never replace ventilation since the ventilation is used to control more than the moisture condition of the indoor climate. The effect of ventilation has not been studied in this project.

At high RH, there is always the risk for condensation at surfaces. The risk for condensation is a function of the vapor content of the indoor air, v_i, and saturation vapor content locally at the surface v_{ss}. The saturation vapor content (or the saturation vapor pressure) is highly temperature dependent (Nevander, 1994; CRC Handbook of Chemistry and Physics, 74th ed., 1993). If the mean temperature in a room is + 20 °C there can still be places in the room where the surface temperature is much lower. This can be seen at windows, in corners and at thermal bridges. The amount of water that is withdrawn from the indoor air is dependent on the area and the temperature of the surface where the condensation takes place. The RH interval studied in this project is 30 - 85% RH. The risk for condensation has not been taken into account.

2.2 Moisture buffer performance

The moisture buffer performance of a material can be defined as the ability of the material to dampen moisture variations in the surrounding air. Today there is no single parameter used to express the moisture buffer performance, but a NORDTEST project to deal with this question has been initiated (Rode, 2003).

The moisture buffer performance is influenced by several material properties; e.g., moisture capacity, water vapor permeability and the period time of the variations.

The slope of the sorption isotherm ($dw/d\varphi$) defines the moisture capacity (ξ); see Fig. 3. For most materials the sorption isotherm is not linear and therefore it is important to use the moisture capacity for the actual interval of moisture variation. The sorption isotherm can either express the moisture uptake per volume of the material (w) or per mass of the material (u). If a material shows hysteresis in the sorption curve, this will have an impact on the effective moisture buffer capacity. It is therefore important to choose a moisture capacity (the slope of the sorption isotherm) that does not overestimate the moisture buffer performance.

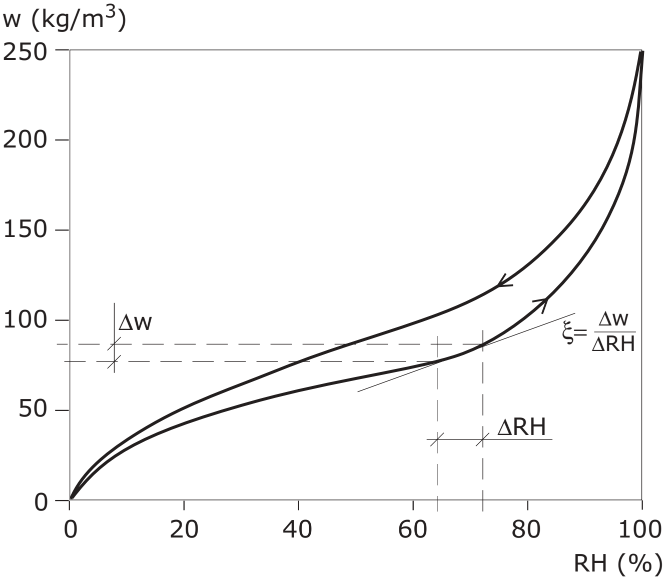


Figure 3. An example of a sorption isotherm for laminate flooring. Hysteresis is seen in the difference between the absorption curve (the lower curve) and the desorption curve (the upper curve). The slope of the curve defines the moisture capacity. Laminate flooring has a rather low moisture capacity in the RH interval in question. Data from Hedenblad (1996).

The water vapor permeability (δ_v or δ_p) or vapor diffusion coefficient (D_v or D_p) expresses the amount per area unit of water vapor that can move through the material per time unit under a given gradient. (v is the vapor content and p the vapor pressure). The water vapor permeability is a nonlinear function of the RH for many materials, see Fig. 4.

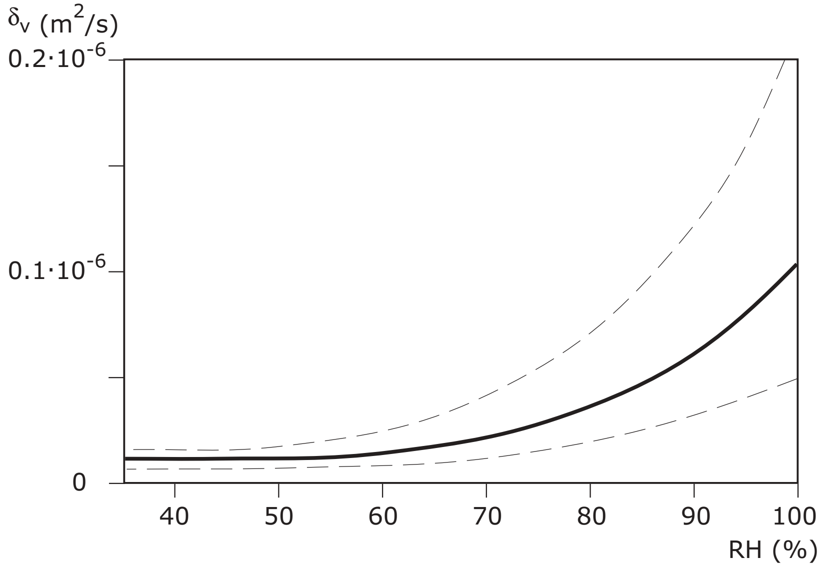


Figure 4. *The water vapor permeability for laminate flooring as a function of the relative humidity. The dashed lines show the uncertainty of the measurements. Data from Hedenblad (1996).*

The ability of a material to let water vapor through can also be expressed as a vapor resistance, Z. The relation between the vapor permeability δ_v and the vapor resistance, Z_v, is

$$Z_v = \frac{d}{\delta_v} \quad \text{[s/m]} \qquad \text{(Eq. 2)}$$

where d is the thickness of the material.

The time period for the variations can vary from a short time scale describing the moisture variations over a day or a week, mostly due to inhabitant activities or a longer time scale that describe the seasonal variations of the outdoor climate. In this project, our focus has been on the short time scales where the period time for the variations is minutes, hours or days.

Considering the basic properties mentioned above, a number of parameters to describe the moisture buffer performance have evolved. It is an advantage to base the parameter describing the moisture buffer performance on these basic properties to facilitate comparisons between different materials.

Penetration depth can be defined and calculated differently. One way is to define the penetration depth, d_p, as the distance from the surface where the amplitude of the periodic variation has decreased to 37%. For a step change the penetration depth can be described as the point where half of the possible step change has taken place at a given time (Hagentoft, 2001).

Arfvidsson (1999) defines the active penetration depth, x_p, as the depth where the variation in RH of the material is 1% and less for a periodic variation in boundary conditions with the period time t_p, see fig 5. At the penetration depth, x_p, an undisturbed relative humidity, φ_{und}, is defined. This value is not the same as the mean value of the variations in RH at the boundary due to the non-linearity of the moisture properties.

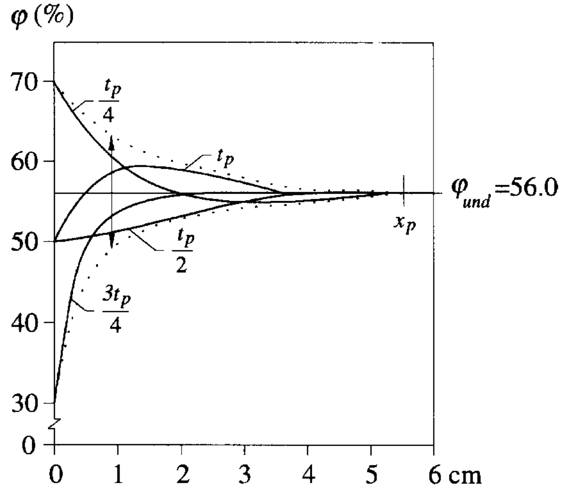


Figure 5. The moisture distribution at different times during the period time, t_p, due to a periodic variation in the relative humidity at the boundary. The amplitude of the variation decreases with the depth (dotted lines). At the penetration depth, x_p, where the relative humidity varies 1%, the undisturbed relative humidity is φ_{und}. From Arfvidsson (1999).

By analogy with the thermal effusivity the moisture effusivity or moisture accumulation ability is defined as a material property that represent the rate and amount of moisture intrusion after a given change in the boundary condition. The moisture effusivity can be calculated as a number that is proportional to the square root of the product of the vapor permeability and moisture capacity: $\sqrt{\delta \cdot \xi}$.

A term that is closely related is the available water that is proportional to the product of the penetration depth and the moisture capacity: $d_p \cdot \xi$ [kg/m^2] (Padfield, 1999).

Mitamura et al. (2001) suggest another way to express the moisture buffer capacity, the weight of the sample as a function of the RH variations. In this case the moisture buffer capacity is not based on the basic moisture properties and all materials need to be tested in the laboratory.

3 Materials exposed to the indoor air

The materials exposed to indoor air are a heterogeneous group of materials, from heavy concrete used in the building construction to lightweight textiles found in furniture and furnishings. Several of the materials used indoors are polymers like wood, cotton and paper. Besides, the materials exposed to the indoor air will often in one way or another have a surface coating.

Figure 6. A typical (?) indoor environment. Untreated wooden ceiling, painted interior walls, laminate flooring, painted wood furniture, cotton curtains and wool carpet. By courtesy of I. G. Börjeson

3.1 Theoretical description of the indoor materials

Materials can be divided into groups based on their moisture buffering performance. The same material can have different moisture buffering ability depending on the time scale for the same RH variation, it is therefore important to know which time scale the moisture buffering performance relate to. Martins and Verschoor (1986) divided materials into slow or fast materials. Plathner and Woloszyn (2002) describe the furnishing as a whole in the terms of soft and hard. Textiles and upholstery are examples of the soft furnishing and wooden furniture and hard flooring examples of the hard furnishing.

I suggest categorizing the surface materials according to the moisture properties governing the moisture transport in the material at the chosen time scale. The moisture properties are divided into: the external resistance, which comprises air boundary layer and surface coating if there is any, and the internal resistance depending on the diffusion coefficient of the material. Each group of materials is characterized by the properties that need to be taken into account when calculating the response to the variations in the boundary conditions, see Fig. 7.

		External resistance Air boundary layer and coating (if any)	
		Needs to be taken into account	Does not need to be taken into account
Internal resistance Diffusion coefficient	Needs to be taken into account	*Painted wood*	?
	Does not need to be taken into account	*Textile curtain*	*Stainless steel (inert)*

Figure 7. An example of how different surface materials can be divided into theoretical categories based on the moisture buffer performance. The time scale is diurnal.

For materials where both the external resistance and the internal properties have to be taken into account there is a solution for the step response due to a step change in the surrounding RH. It is based on Luikov’s (1968) analytical solution. The step-responses can be superimposed to handle periodic variations as well. Materials where only the internal properties have to be taken into account can be treated as a special case of the step response solution mentioned above. For materials where only the external resistance has to be taken into account, the step response can be described by an exponential function. For short time scales some material will act as practically inert (e.g., lacquered wooden flooring), whereas the same material for longer time scales can act as a moisture buffer.

3.2 Wood and wood based materials

Wood and wood based materials are used in building constructions as well as in furniture. The surface coating used on wood can vary from none for a wooden ceiling to a convertible coating on parquet flooring.

Wood has a heterogeneous structure based on its fibrous composition. The moisture uptake is to a high degree determined by the orientation of the fiber strands in relation to the moisture source. The moisture uptake as a function of the fiber orientation is approximately 1:2:20 for tangential, radial and parallel the fiber.

The moisture properties are also determined by the species of wood and the location it grew in. A simple division into soft woods and hard woods can be made due to the species. A hard wood (deciduous tree) has lower moisture permeability than a soft wood (coniferous tree).

Wood and wood based materials have a rather large moisture capacity and the moisture-induced deformation, such as swelling, is usually noticeable. Since wood is an organic material, the degradation process due to biological growth is moisture related involving, e.g., mold, mildew, rot, bacteria and various insects.

3.3 Textiles and upholstery

Textiles are used in furniture and furnishings such as curtains, carpets and bedding. Textiles have compared with most traditional building materials a more mesh-like structure making them lighter and more permeable. The yarn can be made from a number of fiber types, e.g., wool, cotton, flax, viscose and nylon. The moisture properties of the fiber in combination with weaving technique give each fabric its unique moisture properties. The upholstery of furniture can be done with a variety of materials, e.g., foam plastic, cotton, down, wool, straw and horsehair. The textile materials have usually a large moisture capacity but a low density. However, textiles in furniture and furnishing are highly represented in the indoor environment and as they to some extent “hide” traditional building materials they play an important role in regulating the moisture variations of indoor air especially for short time scales.

3.4 Paper

The paper found in the indoor environment can be of different types. Books, newspapers and magazines are one major part of the paper in the indoor environment exposed to the indoor air. Wallpaper made from paper another, even if they usually have an additional surface coating such as paint or vinyl film. The manners the paper products are stored or exposed to indoor air will influence the impact of the moisture buffering from them.

3.5 Plaster board and gypsum

Plasterboard is a gypsum-based board and the type used indoors in Scandinavia is usually covered with heavy paper on both sides. Plasterboard in the indoor environment is used for interior walls and ceilings. Usally a surface coating as wallpaper or paint is used.

Gypsum based plasters used indoors are common in the southern parts of Europe and play an important role in the moisture buffering performance of the room.

3.6 Cementious and ceramic materials

Concrete and aerated concrete is used for walls, floors and ceilings. The walls are usually surface coated in the same way as plasterboard and concrete ceilings are mostly painted. On concrete floors a layer of screed is usually applied and after that a flooring material for example PVC-flooring, laminate flooring or ceramic tiles. Most concrete is therefore not immediately exposed to the indoor air.

The composition of plasters and mortars can vary from lime mortar to cement mortar. The water vapor permeability decrease with the cement ratio of the mortar.

Ceramic tiles often have a glazing giving them an almost inert surface for the diurnal time scale. If the ceramic tiles are unglazed they have similar properties as brick. The mortar used to fill the joints between tiles is more porous and could buffer moisture. The area is usually small, and the moisture buffering of the joints can usually be neglected.

Brick without surface coating have a low moisture capacity in the RH interval of interest for normal indoor environments. Sometimes when brick is used in large volumes in, e.g., classrooms and churches, the moisture buffering of the brick cannot be neglected. Plasters are used with or without paint to surface coat brick. The moisture buffer performance will then to a high degree depend on the paint and plaster used.

3.7 Surface coatings and finish materials

Surface coatings are often based on polymers, e.g., latex paint, wax and plastic films. In the indoor environment, surface coatings vary from sparse oil treatments to thick and almost impermeable enamel paints. The surface resistance of a surface coating is dependent on the material used and the application. If there is a “heavy” surface coating on the top surfaces of furniture, e.g., a wooden table, usually the side underneath will be untreated. Therefore an estimation of the accessibility of indoor air to the potential moisture buffer areas has to be included in an estimate of the moisture buffer performance.

Oil and wax treatments are mostly used on wood flooring, furniture and furnishing. The treatment has to be repeated to maintain the surface coating desired. Therefore, the surface resistance of the coating may vary over time.

Paint is a broad and heterogeneous group of surface coatings that goes from whitewash to convertible coating. For surface coating of building components latex paint is most commonly used in the indoor environment. Furniture and furnishing can be varnished or lacquered using alkyd paint. Whitewash (or distemper) and temper paints are considered to be more “open paints” with a lover surface resistance.

Common wallpapers are coated either with paint or a vinyl film to make the surface cleanable. Adhesive and in some cases primer belongs to the wallpaper system. In Sweden glass fiber backing with different surface structure is used under the coats of paint. There are also wallpapers with a textile top surface.

Polymeric foils are mostly used for furniture, furnishing and floorings. Usually they are applied on board materials as chipboard or MDF boards.

3.8 Other materials

Stainless steel and other metals as well as glass will be inert to moisture buffering due moisture variations in the indoor air. Their thermal properties will in some locations give them a lower surface temperature than the room air and cause condensation.

There are a number of plastic materials (for example in floorings and furniture) in the indoor environment and their moisture properties are not homogeneous due to both the differences in composition and in the use of the material.

4 Methods to determine moisture properties

A literature survey considering methods to determine sorption isotherms and water vapor permeability has been conducted. The scope was on simple and inexpensive methods appropriate for materials in the indoor environment. A special focus has been on thin and permeable materials since this type of materials are not usually dealt with in building science. The literature survey presented here should only be seen as a taste of the buffet of methods and modifications available. A large literature survey covering a broader field of the interaction between surface materials and the indoor air was conducted earlier in the project (Harderup, 1998).

4.1 Sorption isotherm methods

Most methods for determining sorption isotherms - equilibrium water gain as a function of RH rely on equilibration over saturated salt solutions that keep a constant RH (Greenspan, 1974; Nyqvist, 1983) combined with gravimetric methods to determine the moisture content in the sample. (e.g.,Wolf et al, 1984; Chirife & Buera., 1995).

In these simple methods the disturbance on the sample if weighed outside the container with the salt solution is a large source of error, especially for less heavy sample with a low surface resistance. Gustafson & Hall (1974) and Zuritz et al. (1977) made attempts to solve this problem by using separate jars with lids for each sample. The jar method described in Paper II is a further development of these methods. In the jar method the sample can be kept in the desired environment all time including measurements without any large openings disturbing. This set up minimizes the disturbance in the sorption isotherm determination.

Saturated salt solutions are also used as humidity generator in small climate chambers where dynamic measurements can be done (e.g., Ojanen & Salonvaara, 2003; Time, 2002; Wadsö, 1993 a).

In many cases, the saturated salt solutions could be changed to unsaturated salt solutions (Chirife &. Resnik, 1984; Clarke et al., 1985) or sulphuric acid solutions of different molecular concentration (e.g., Speakman & Cooper, 1936).

McBain and Bakr (1926) developed another gravimetrical method with a spring balance made in quartz in a vacuum tube. The elongation of the spring is used to determine the vapor uptake. The method can be used not only for water vapor but also most other vapors, since the method use liquids as a vapor source.

Peralta (1995) used a combination of quartz spring balances, vacuum equipment and saturated salt solutions to determine sorption isotherms for wood samples.

The automated sorption balance (e.g IGA-sorp, DVS 1000) is a microbalance in a climatic chamber where the climate can be very carefully and automatically governed. The relative humidity desired is obtained by carefully mixing wet and dry air. The limitation with the method is the small sample size. (Arlabosse et al. 2003, Janz & Johannesson, 2001).

Leisen et al. (2002) used a sorption balance as a humidity generator and determined the moisture uptake of the sample using nuclear magnetic resonans (NMR) wich is a non-gravimetrical method.

Another non-gravimetrical method was developed by Wadsö and Markova (2002) using a double twin calorimeter to determine sorption isotherms. Others, for example Hansen et al. (1996) and Lehto & Laine (2000), have also used calorimetry for determination of sorption isotherms.

4.2 Water vapor permeability methods

The water vapor permeability, δ_v, or the diffusion coefficient, D_v, can be determined from steady state or unsteady state methods.

The most common steady state method is the cup method. In the cup method the sample is fasten as a lid over a container with a humidity generator (usually saturated salt solutions) or a desiccant. The modifications and alterations of the basic cup method is numerous, the different cup methods can be categorized as dry cups or wet cups depending on the content of the cup. (e.g., Newns, 1950, Turl & Kennedy, 1965; Joy & Wilson, 1965; McLean et al. 1990; Villadsen et al.,1993; Hedenblad, 1996; Takada et al., 2001)

Dry cup methods use desiccants inside the cup and a higher relative humidity outside. The vapor transport will go through the sample from the outside and in. In the wet cups usually saturated salt solutions are used as humidity generators,. the RH inside the cup is higher than outside.

The cup methods can also be divided into upright cups and inverted cups. In the upright cups there is a necessary air gap between the salt solution and the sample to avoid splashing the sample when doing measurements. This air gap will introduce an internal resistance of the cup. For highly permeable materials this resistance can be difficult to handle. (Newns, 1950)

In inverted cups the internal resistance of the method is less since there is no air gap. On the other hand a hydrophobic vapor permeable membrane is useful to ensure that the transport through the sample is a vapor transport (ISO/DIS 15496,2001; Paper I).

A modified cup method where the measurements are done in low pressure speeds up the measurements and gives additional information about the vapor and liquid transport if the measurements are done at several ambient pressures (Gailbraith et al., 2003).

The partition cell or diffusion cell where the sample material is placed as a wall between two chambers with different climate is another steady state method. (e.g., Nilsson et al., 1993)

The sorption method is a unsteady state method based on one step change in the RH surrounding the sample. There are many ways to accomplish this step change. For example climatic chambers as sorption balances and the jar method can be used. It is important that the step is made as perfect as possible. Thick air boundary layers or stagnant air will give large surface resistances making the step imperfect. (Wadsö,1994 a; Wadsö, 1994 b).

4.3 Dynamic methods

Padfield (1999) have developed a climate control system where a water basin is constantly weighed. Using a heat coil in half of the basin a humidification can be generated through vaporization of the water in the basin. The amount of vapor released in the chamber is monitored through the weight change of the basin. The other half of the basin is equipped with cooling facilities so the vapor of the chamber air can condensate in order to create a drying situation. The withdrawal of vapor from the air is also monitored through weighing the basin. This basic method has been employed in some climatic chambers for dynamic measurements with periodic variations (e.g., Padfield & Peuhkari, 2002, Peuhkari & Padfield, 2002, Mitamura et al., 2001).

Other ways of creating a varying climate for dynamic measurements is to use different salt solutions as mentioned earlier. The jar method (Paper II) is well suited for step changes in RH, since moving the sample from one climate to another is easily done.

4.4 Conclusion from the literature survey

The literature survey shows that aqueous salt solutions are a good way to create a desired RH. Both saturated and unsaturated salt solution can be used. There are few inexpensive sorption isotherm determination methods where the sample is undisturbed during the complete measurement period. For water vapor permeability measurements on thin and highly permeable materials the interior resistance in the method can influence the result to a high degree. The inverted cup provides a solution to this problem if the fastening of thin materials can be dealt with as well as the possible liquid transport in the inverted cup set-up.

5 Important factors for the measurements

Moisture property measurements, regardless of the method used, involve a number of error sources that have to be controlled as far as possible. The factors discussed in this report are closely associated to gravimetrical methods with aqueous salt solutions as humidity generator.

5.1 Climate control

Saturated salt solutions have temperature dependencies that vary. NaCl has a temperature coefficient of 0.03%(RH)/K, whereas $Mg(NO_3)_2$ has 0.30%(RH)/K, respectively (Greenspan, 1977). The temperature should consequently be held constant and only small and slow temperature changes be allowed to avoid a large error or in the worst-case condensation on samples.

The relative humidity of the climate room is of course also temperature dependent. For cup methods where the water vapor permeability is measured even a small change in RH will give a large error in the moisture flux that are used to evaluate water vapor resistance or water vapor permeability. For example an increase of 1,5% RH will generate a moisture flow that is 9% higher for the vial cup method in the interval of 33 – 54% RH. (Paper I).

5.2 Edge masking and area determination

For non-particular samples as samples from boards and sheets, the edge is usually edge masked to obtain a one-dimensional moisture flow. This can be done in a number of ways. The compatibility with the sample material is one of the major considerations. Using solvent based or fluid compounds for edge masking there is a risk of making a larger area hydrophobic to consider.

Claesson et al. (1994) have developed a calculation model to predict the maximum error that the edge masking will give for a given set of material properties and geometrical shape. This model is developed for cup method measurement where only the edge is masked.

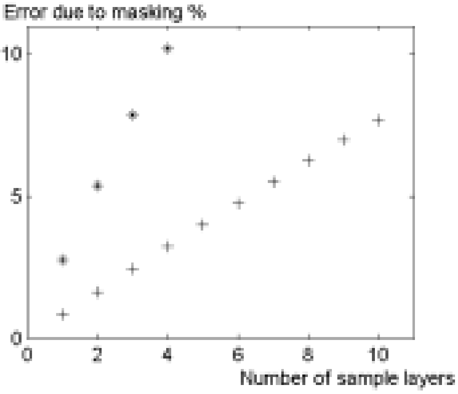


Figure 8 The error due to edge masking in the vial cup for paper (+) and cotton textile (∗). The thickness of the paper is 0.13 mm and the cotton textile 0.45 mm. (Paper I)

Area determination is important since the weight changes used both for the sorption method and the cup method must be related to the volume or the area of the sample.

In paper III a simple method to determine the surface area based on digital pictures is proposed. With a digital camera a photograph was taken of the open side of each sample exposed to the step change in humidity. By fitting a circle on top of the digital picture in high resolution an equivalent exposed area was determined. See Fig. 9. Especially for samples with a very ragged edge this will not always be easily done and to some extent subjective. The exposed areas are not truly circular so fitting a circle will not always be correct and therefore introduce errors. These errors are present also in the earlier used method with the slide caliper rule and in the same magnitude as estimating the diameter with a slide caliper rule.

One source of error is the distortion of the length and width relationship using a photo. If there are imperfections in the lens it could cause a systematic error especially at the edges. It is therefore important to restrict from too close up pictures.

Figure 9 A plasterboard sample with wax sealing and a circle describing the estimated area. (Paper III)

One advantage of using the digital photo method is that the sample and the sealing are not subjected to sharp tools, minimizing the risk to harm the sealing. Counting pixels in an automated fashion could be an option that would save time and increase the accuracy of the area determinations (Paper III; Svennberg, 2002).

5.3 Surface mass transfer coefficient

The resistance of air gaps and boundary layers will result in uncertain surface mass transfer coefficients since the air movements within jars and cups are usually unknown. A larger surface mass transfer coefficient will in methods where the step-response of a material is to be measured result in a more imperfect RH step. (Wadsö, 1994 a; Wadsö, 1994 b). In cup measurements the RH interval is effected by the surface mass transfer and therefore the calculated diffusion coefficient will not be correct (Hansen, 1993). To avoid these problems the airflow should as far as possible be controlled, and the air gaps with stagnate air should be minimized.

5.4 Equilibrium criteria

For several materials such a wood, organic textiles and other polymers the Non-Fickian behavior or anomalous sorption with a slow but creeping moisture regain will make it almost impossible to find true equilibrium (e.g., Wadsö, 1993; van der Wel & Adan, 1999; Håkansson, 1995; Peuhkari, 2003). It is therefore necessary to state an equilibrium criteria. The measurements and the material properties will therefore be dependent on the equilibrium criteria used (Chirife & Buera, 1995).

The equilibrium criteria can be stated as a maximum weight change per time unit and is usually connected with the accuracy of the balance used. This type of criteria is for example used in automated sorption balances

The weight change per time unit can also be combined with only a time criteria to avoid prolonged measurements. The time criteria alone is also used (e.g., Jowitt& Wagstaffe, 1989), but has the disadvantages that it does not take into account the balance accuracy or the RH interval for the actual measurements. It has therefore a limited use.

In paper II an attempt to use an equilibrium criteria, that takes into account the sample size, the moisture capacity of the sample and the RH interval where the measurements are performed is presented. In the end of the measurements the equilibrium criteria can be expressed as

$$\Omega = \frac{dm}{dt} \cdot \frac{(\varphi_f - \varphi_i)}{(m(t) - m_i)} \quad [\mathrm{s}^{-1}] \qquad \text{(Eq. 3)}$$

A rather arbitrary value $\Omega < 5 \cdot 10^{-9}$ s^{-1} is suggested in paper II, based on the accuracy of the balance (0.0001 g).

6 Summary of appended papers

6.1 A modified cup-method for lightweight and highly permeable materials – Paper I

The paper presents a modified inverted cup-method specially designed for thin, light and highly permeable materials such as textiles and paper. The method is based on standard laboratory crimp cap vials, Fig.11. In the paper the background to the novel method is presented — the larger uncertain interior resistance of the traditional cup method together with problems to fasten the samples in the traditional cups are discussed.

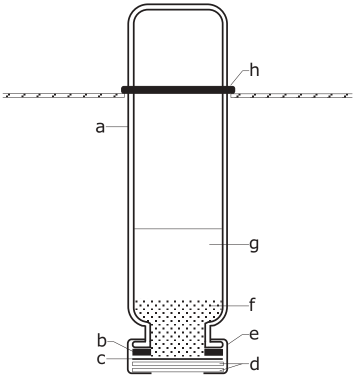


Figure 11. The vial (a) is charged with a saturated salt solution (g) with an excess of salt (f) to ensure that solution is saturated during the experiment. A seal (b) is placed with the coating towards the glass rim. A vapor permeable membrane (c) ensures that only water vapor is transported through the test material (d). The crimp cap (e) holds the seal, membrane and test material in place and provides an edge masking of the test material. A gasket ring (h) holds the complete configuration in place in an airflow rack.

The method for evaluation of water vapor resistance of the sample material adapted from Huldén and Hansen (1985) is presented together with a small series of test measurements performed on cotton textile and filter paper. The advantages as well as the special considerations of the method are presented together with suggestions for future work including the optimization of the method.

6.2 An experimentally simple method for measuring sorption isotherms – Paper II

The paper presents a method for determination of sorption isotherms. It is a development of the methods proposed by Gustafson & Hall (1974) and Zuritz et al. (1977). The method is a gravimetrical method based on conventional food preserve jars using aqueous salt solutions as humidity generator within the jar. The set up is shown in Fig. 12.

When a sorption isotherm is determined, the equilibrium criteria used is one important aspect to consider. For many materials the moisture uptake or release close to equilibrium is very slow, making it difficult to determine the true equilibrium. In this paper an attempt to define an equilibrium criteria taking into account the moisture capacity and the size of the sample is made.

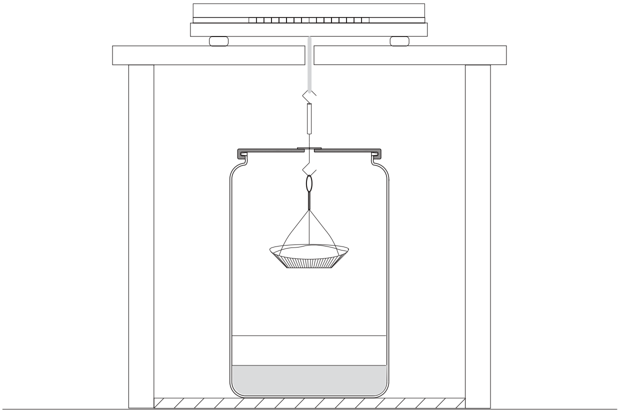


Figure 12. The measurement set-up for the jar method with the sample in weighing position. The sample hangs in the wire that goes through the hole in the lid. The washer floating on top of the lid makes this hole quite tight and the rubber tubing on the wire tightens the hole in the washer when weighing is not being made.

The validation of the method was done on microcrystalline cellulose (MCC) and bentonite clay and compared with literature results. For the bentonite clay a comparison with a sorption isotherm determined with a sorption balance was made. The deviations between these measurements are discussed in the terms of the slow moisture regain for bentonite clay and the influence of the time scale on the measurements. The leakage of the method was investigated both experimentally and through a simulation and was found to be within acceptable limits.

6.3 Time-dependent moisture properties for plasterboard with surface coating – Paper III

This paper presents a study made on the step-response of plasterboard painted with an acrylic latex paint system subjected to a step-change in RH. The jar-method (Paper II) was used even if the method had not been validated for transient measurements. The step-responses were monitored by gravimetrical measurements. Figure 13 shows the sample in weighing position.

The paint system comprise oil based primer, adhesive, glass fiber backing and two coats of acrylic latex paint and was applied on a standard plasterboard with paper on both sides. The samples were taken from each of the stages of the plasterboard - paint system.

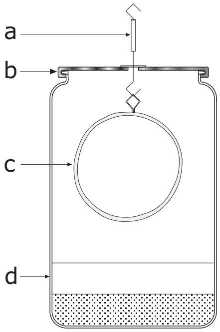


Figure 13. The jar method. A ordinary glass jar for foods with screw lid (b)with saturated salt solutions (d) was used. The hole in the lid was covered with a thin plastic washer with a minute hole in it that only allows the wire to pass through. A small rubber tube was placed over the wire (a) to seal the hole in the washer when measurements were not being done and to keep the sample in a vertical position. Determination of the moisture absorption-desorption was made with below balance weighing by connecting the wire (a) to the balance.

Unexpectedly the results show that the moisture capacity of the paint system cannot be neglected. A comparison with the step response model for a material with a surface coating based on Luikov's analytical solution (1968) showed satisfactory results.

The paper also presents a simple method determining the exposed area based on digital photo.

7 Concluding remarks

The two new laboratory methods that have been developed within this project and are presented in this thesis are inexpensive methods based on standard glassware. It has been one of the aims to use simple and inexpensive solutions instead of highly complicated methods. One reason is the possibility that other researchers and laboratories will use the same method, which is facilitated when the method is simple and inexpensive. Another reason is that inexpensive methods do not restrict the number of samples used as more expensive methods do. Both methods are also based on gravimetrical measurements, a base that is easily understood and the physical relations well defined.

The vial-cup method for determining water vapor permeability could be used not only for materials in the indoor environment. Both the paper and pulp industry and the textile industry are interested in measuring transport properties for their products and the vial cup method could be used in these areas too.

The jar method as a method to measure sorption isotherms gives the method a wide range of applications since sorption isotherms is of high interest not only for building science but also food science, pharmaceutical science, agricultural science, soil science etc. One of the major uses for the results from measurements performed on building and furnishing materials are as input to hygrothermal simulation models, see Fig. 1. If the method will be validated for transient measurements, see section 8 - Future work, the method will give even more information since the step response of a material subjected to a step change in RH can be closely monitored.

An increased knowledge of the moisture buffer performance of surface layer materials will provide a base for a better prediction of indoor air moisture condition and better optimizing of HVAC system particularly when air conditioning with chilling is used. Also the risk of emissions from materials, mold growth and dust mites could be more carefully correlated to the moisture conditions of both the indoor air and the surface layer materials.

8 Future work

In this project we have succeeded in developing two new methods for determining moisture properties – the jar method for sorption measurements and the vial-cup method for water vapor permeability measurements. The methods also have the potential to be useful for other scientific areas.The continuation of this project will concentrate on:

- An optimization and validation of the vial-cup method and using the method for determination of water vapor permeability for textiles, paper and even paint films.

- Validation of the jar method for transient measurements, including a modified sample pan and an experimental set up with a fan to minimize the resistance of the air gap.

- The determination of sorption isotherms and monitoring step-responses for materials common in the indoor environment with and without surface coating. The material data will be gathered in a database and used in the categorizing of different materials into different theoretical material groups according to their moisture buffer performance.

- In collaboration with dr. Carsten Rode at DTU, Denmark, room-scale climate chamber measurements are performed. Comparison between data from the jar method and the vial-cup method and the climate chamber measurements will be done to see the correlation between small-scale experiments and more realistic climate chamber experiments.

- Develop a model, on material level, for the interaction between the RH variations of the surrounding air and the surface materials.

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Paper I

A modified cup-method for lightweight and highly permeable materials.

Svennberg, K and Wadsö, L (2003)

Published in *Research in Building Physics,* eds. Carmeliet, Hens & Vermeir, Lisse: Swets & Zeitlinger, ISBN 90 5809 565 7, pp.177-182. Printed with permission from Swets & Zeitlinger.

A modified cup-method for lightweight and highly permeable materials.

K. Svennberg
Building Physics, Lund University, Sweden

L. Wadsö
Building Materials, Lund University, Sweden

ABSTRACT: With an increasing interest for dynamic hygrothermal calculation models for short time periods, there is a need for uncomplicated and reliable methods to determine the moisture properties for materials used in the indoor environment.
The traditional cup-method is commonly used for building materials to determine the water vapor permeability. When used on lightweight and highly permeable materials, the unidentified inner surface resistance of the cup-method will be problematic and fastening the sample to the cup can be troublesome. To address these problems, we have developed a modified cup-method based on standard laboratory crimp cap vials to determine the water vapor resistance for lightweight and highly permeable materials, such as textiles and paper**.** This modified cup-method together with a validation is presented in the paper.

1 INTRODUCTION

With an increasing interest for dynamic hygrothermal calculation models for short time periods, there is a need for uncomplicated and reliable methods to determine the moisture properties for materials used in the indoor environment.

The traditional cup-method is commonly used to determine the water vapor permeability of building materials e.g. wood, flooring and board materials (e.g. ASTM 96-00, 2000, Hedenblad, G., 1993, McLean, R. et al. 1990, Joy, F.A. & Wilson, A.G., 1965). However, when used on lightweight and highly permeable materials, such as textiles and paper, the normally used cup-designs are difficult to use because thin materials cannot be fastened reliably in the cup and the high interior surface resistance is difficult to handle (Newns, A.C. 1950, Mackay, J.F.G., 1971).

Several attempts have been made previously to develop a method that deals with these problems. The draft to an international standard method ISO/DIS 15496 (2001) primarily to be used for quality control is an inverted cup-method for textiles where the cup is immersed into water. Nilsson, L. et al. (1993) used a diffusion cell where the test material was placed as a partition between two chambers with different relative humidity. Takada, S. et al. (2001) describe a modification of the wet-cup-method specialized for textiles.

The objective of this paper is to present a simple method for determining the water vapor permeability for lightweight and highly permeable materials using basic laboratory equipment.

2 METHOD

The modified cup-method to determine the water vapor permeability for lightweight and highly permeable materials is based on standard laboratory crimp cap vials and tear-away crimp caps. Saturated salt solutions inside the vials in combination with a well-controlled ambient climate create a gradient in vapor pressure.

The material to be tested is placed on a highly vapor permeable, hydrophobic and micro-porous membrane as a lid over the vial opening and is held in place by the crimp cap. Figure 1, shows the details of the experimental configuration of the vial cup. The vial is placed upside-down in an airflow rack exposed to a controlled airflow and a constant climate, Figure 2.

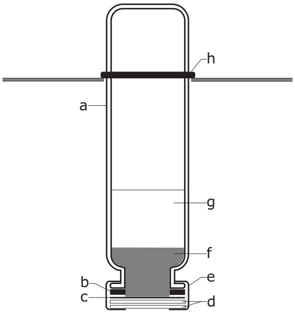

Figure 1. The vial (a) with a diameter of 20 mm and a height of 75 mm is charged with a saturated salt solution (g) with an excess of salt (f) to insure that solution is saturated during the experiment. A Teflon coated silicone seal (b) is placed with the coating towards the glass rim. A vapor permeable membrane (c) (e.g. Durapore GVHP, Millipore, Bedford, USA) ensures that only water vapor is transported through the test material (d). The crimp cap (e) holds the seal, membrane and test material in place and provides an edge masking of the test material. The holes in the seal and in the crimp cap are 14 mm in diameter. A gasket ring (h) holds the complete configuration in place in the airflow rack.

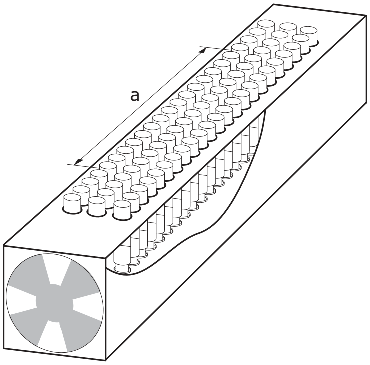

Figure 2. The airflow rack with the vials mounted. The fan provides a controlled airflow. The section in the middle (a) is used for measurements.

The weight change of a vial over time is used to determine the moisture flow. By measuring the moisture flow for different number of sample layers under identical ambient conditions, we can calculate the water vapor resistance, Z_m, for one layer of the sample material, as the surface vapor resistance is the same in all vials. The evaluation is done as described by Huldén, M. & Hansen, C.M. (1985). Figure 3, illustrates the used nomenclature.

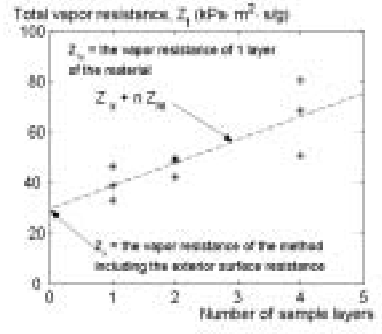

Figure 3. The nomenclature used in the evaluation of the method. Z_t the total vapor resistance, Z_x the vapor resistance of the method including the inner and exterior surface resistance found in the intercept with the y-axis, Z_m the vapor resistance for one layer of the sample material and n the number of sample layers. Measurement for a textile in an experiment using $MgCl_2$ as an absorbent (∗).

3 TESTS OF THE METHOD

We have made a series of test measurements to evaluate the modified cup-method.

3.1 *Materials*

The crimp cap used is a tear-away type leaving a circular area exposed (14 mm in diameter).

In the test measurements two types of Teflon coated seals were used, silicone and butyl rubber. The silicone seal is 1 mm thick and the butyl rubber 2.8 mm. The seals were made by using a hollow punch to make a center hole (14 mm in diameter) in a regular 20 mm vial septa. The measurements show a significant difference in leakage between the two types of seals; the butyl rubber seals had a leakage of salt solution in 67% of the vials compared with 13% for the silicone.

Two types of vapor permeable membrane were tested – Durapore GVHP (polyvinylidene fluoride) and Flouropore (PTFE on a LDPE backing) both with a pore size of 0.22 µm and manufactured by Millipore, Bedford, USA.

Using a hollow punch the test material was cut into circular pieces 20 mm in diameter. For each measurement, we used two or three sets of different layer set-ups (e.g. 1, 2 and 4 layers). Table 1 presents a description of the test materials used in the test series.

Table 1**.** Test materials used in test series.

	Textile	**Paper**
Type	Cotton/linen (58/42%) “Josef” Almedahl AB, Dalsjöfors, Sweden	Quantitative Filter paper 0OH Ash content 0.007% Munktell Filter AB, Grycksbo, Sweden
Surface mass (g/m^2)	273	88
Thickness (mm)	0.451 ± 0.001	0.132 ± 0.005

3.2 *Climate conditions*

We used two climates (+20± 0.5 °C, RH 35 ± 1%) and (+20 ± 0.7 °C, RH 58 ± 1.5%) in the climate room where the airflow rack was placed.

Saturated salt solutions provide controlled relative humidity inside the vials. During the test measurements, we used $MgCl_2$ (RH 33% at +20 °C), $Mg(NO_3)_2$ (RH 54.4% at + 20 °C), NaCl (RH 75.5% at + 20 °C) and distilled water (RH 100. In some of the experiments, the RH was higher in the vial than in the climate room, causing the vials to loose weight, in other experiments the RH was lower in the vials so that the vial increased in weight. A correction of the relative humidity inside the vial in relation to the actual temperature was done according to Greenspan (1977).

An airflow rack holding the vials gave a controlled airflow over the exposed area of the test materials, an elevated airflow also decreases the exterior surface vapor resistance. (Newns, A.C., 1950)

3.3 *Measurements*

The moisture flow was determined from the weight changes of the vials. Typical intervals for the weighing were every 6–8 hours during the first 2 days, thereafter once a day. During the weighing, the vial is placed right side-up on a balance. The weighing of each vial takes approximately 30 s.

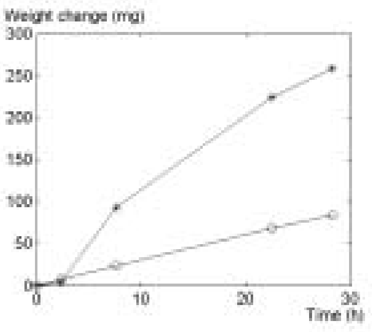

Figure 4. The weight changes of the vials as a function of time. (∗) 1 layer of cotton textile, (o) 8 layers of paper. The linear part of the measurements is used for calculating the vapor resistance. Thereby the initial disturbances are not taken into account.

In the test experiments, we have studied the following factors: seal materials, different membrane types, two types of test materials, the effect of air penetrating the test material, the air velocity and the use of different saturated salt solutions.

3.4 *Results*

Since the measurements were done under constant conditions, the weight change plotted as function of time should be linear. Small deviations from this is seen in the beginning of the measurements and referred to as initial disturbances. (Fig.4)

From the gravimetric measurements, the total vapor resistance, Z_t, of the vial set up can be calculated, see Fig 5. Using the evaluation method described by Huldén, M. & Hansen, C.M. (1985) the vapor resistance, Z_m, for each layer can be determined. The magnitude of vapor resistance of the method, Z_x (including exterior surface resistance etc.) was estimated to 1-2 times the vapor resistance Z_m for each layer for the present measurements.

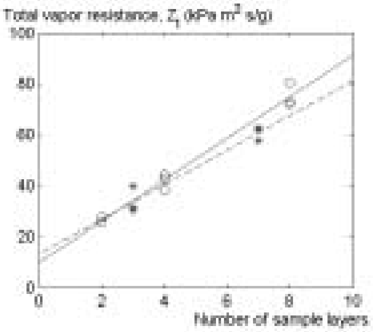

Figure 5. The total vapor resistance, Z_t, plotted as a function of numbers of paper layers. Data from measurements on paper letting a salt solution dry through the sample and the vapor permeable membrane:
(∗) 4.5 g NaCl(s) + 4.5 ml NaCl(aq, sat),
(o) 9 ml NaCl(aq, sat) together with a small amount of NaCl(s). The value Z_t is calculated from the linear part of the measurements. An outlier is removed and not used in the figure or the regression.

The effect of the increased airflow over the surface was studied in with paper as test material. One group of the samples was prepared in a normal manner, see Fig 1, one group was made with a double layer of membrane in the normal position and one group had one membrane in the normal position plus one additional membrane covering the exposed area (in that way protecting the sample material from penetrating airflow).

The evaluation shows no significant difference between the three groups, Fig. 6.

Figure 6. Comparison between samples with membrane covering the exposed area (o), one membrane in normal position (∗) and two membranes in normal position (□).

The masking of the edges of the sample will introduce an error in the determination of the water vapor resistance of the material, Z_m. With an increasing thickness of the sample e.g. increasing number of layers, this error will also increase (Claesson et al., 1994). We have made calculations to estimate the magnitude of the error due to masking, see Figure 7.

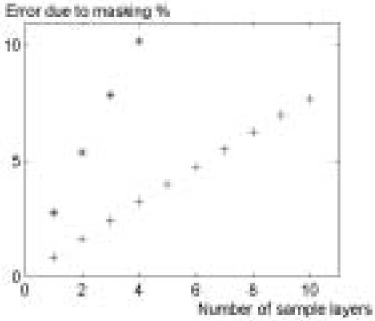

Figure 7. The error due to the effect of the masked edges, for textile (∗) and paper (+), calculated according to Claesson et al. (1994).

The maximum error was found to be approximately 6% for the paper (8 layers with a thickness 0.132 mm) and 10% for the textile (4 layers with a thickness 0.451 mm). The error calculations were made for an isotropic material.

The air speed was measured with an anemometer with a probe that was placed in a free vial place of the airflow rack while the other vials remained in their places. The air speed varied over the cross section as seen in Figure 8, similar distributions were seen for the complete test measurements. The moisture variations of the samples show a small variation over the cross section, Figure 9.

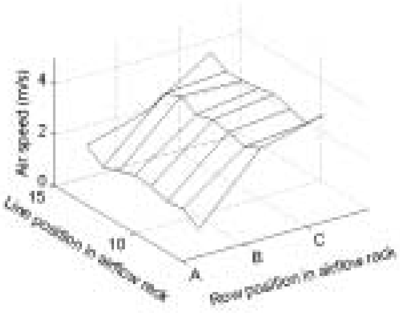

Figure 8. Air speed distribution for the middle section of the airflow rack during measurements on paper using $MgCl_2$ as an absorbent.

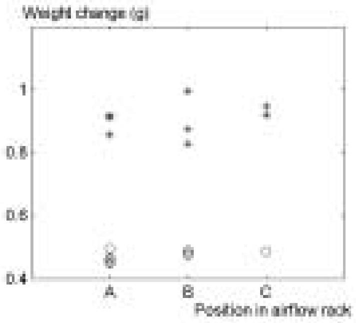

Figure 9. The moisture variation of the sample as a function of the position in the airflow rack, 3 (∗) and 7 (o) layers of paper using $MgCl_2$ as an absorbent. The measurements were made for the time period of one week.

4 DISCUSSION

Advantages with the method:

- The method avoids the problems caused by uncertain interior surface resistances.

- The use of crimp caps makes it easier to fasten lightweight material and provide an edge masking.

- Standard laboratory equipment makes the method simple and inexpensive.

- It is a rapid method. Optimized measurements should probably not take more than two days.

Sources of error and special considerations for the method:

- Variations in the ambient climate. The relative humidity of the climate room varied by up to 1.5% over a measurement period. Such a change in RH results in up to 9% changes in the flow rates and is a factor that should be taken into account when this type of measurements are evaluated (this has not been done in the present measurements).

- Initial disturbances giving non-linear plot over the weight changes as a function of time. This is most probable caused by starting the measurements without conditioning the vial set-up properly. For experiments where the salt solution absorbs water and it could be beneficial to use a saturated salt solution with a high or even very high degree of undissolved salt this question needs careful considerations. This should be taken into account when optimizing the method of measurement.

- Disturbances caused by the measurement procedure were the vial during weighing is turn from upside-down to right side-up and back again. This is can be minimized by using a stand on the balance allowing the vial to weighed in an upside-down position.

- Fluctuations and disturbances in the air flow in the airflow rack.

- Air penetration of the sample material because of the elevated airflow in the airflow rack. Our test indicates that this is a minor source of error.

- The area determination needs special attention and especially the risk of large spread due to the variations from the opening of the tear-away crimp cap.

- A thick and stiffer seal, as the butyl rubber, seems to increase the risk for leakage and limits the number of layers of test materials that to be used.

- Salt crystals growing through the membrane. This is known to be a risk for measurements where salt solutions are being dried through a vapor permeable membranes. Special care should be taken to avoid this, mainly by performing the measurement over a short time (1-2 days).

- As an indicator for leakage due to capillary transport, a color marker of the salt solution (e.g. ordinary food color) can be used.

- Due to the small area exposed the more heterogeneous a material is the larger spread can be expected. Using an increased number of identical sample set-ups will help to obtain a more correct value for the water vapor resistance.

Future work:

- To optimize the method of measurement so that it will be rapid and reliable. Improve the evaluation method so that e.g. leaking vials are more easily detected. There is also a need to develop some aid to minimize the differences between different persons performing the preparations and measurements.

- Continue the validation of the method with an increased number of test materials.

- Investigate the possible problem caused by pressure changes in the vials.

5 CONCLUSIONS

We have developed a modified cup-method for lightweight and highly permeable materials that has the potential to be reliable, simple and practical to use. The method deals with the disturbances and errors caused by the inner surface resistance in the air gap of the traditional cup method and provide an easy fastening of thin and lightweight sample. The improvement and validation of the method will continue.

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Paper II

An experimentally simple method for measuring sorption isotherms.

Wadsö, L., Svennberg, K. and Dueck, A.

Submitted for publication to *Drying Technology*

AN EXPERIMENTALLY SIMPLE METHOD FOR MEASURING SORPTION ISOTHERMS

Lars Wadsö(1), Kaisa Svennberg(2) and Ann Dueck(3)

1. Building Materials, Lund University, Box 118, 221 00 Lund (corresponding author, lars.wadso@byggtek.lth.se)

2. Building Physics, Lund University, Box 118, 221 00 Lund, Sweden

3. Clay Technology AB, IDEON Research Center, 223 70 Lund, Sweden

Keywords

sorption isotherm, measurement method, saturated salt solutions, glass jar

ABSTRACT

We describe a simple, yet practical and precise, way of measuring sorption isotherms with each sample in its own glass jar with a saturated salt solution. The measurements are done with below-balance weighing and with the sample kept inside the closed jar during the whole measurement period, providing constant relative humidity (RH) conditions. The technique has been tested on microcrystalline cellulose and bentonite clay. The agreement with literature values was good; the differences seen for bentonite at high RH are discussed in terms of the slow attainment of equilibrium for this material.

INTRODUCTION

Measurement of sorption isotherms – equilibrium water gain as a function of relative humidity (RH) – is a common practice in many fields of science and engineering, e.g. food science, building material science, soil science and pharmaceutical science. Many methods are based on the equilibration over saturated salt solutions, e.g. the method tested in an European COST study [1]. Recently the use of automated sorption balances have increased, see for example references [2-4]. More advanced methods like NMR [5] and sorption microcalorimetry [6] are also used.

Simple methods to determine sorption isotherms usually rely on conditioning above saturated salt solutions that keep constant relative humidities (RH) and gravimetric methods to determine the moisture content in the sample. In many such methods the RH around the sample is disturbed during each weighing because the sample has to be taken out of the controlled RH for each measurement. A solution to this is to keep both the balance and the sample at the same RH, but this is impractical, as many different RH are needed to determine a complete sorption isotherm.

This paper deals with a further development of the methods of Gustafson and Hall [7] and Zuritz et al. [8] in which each sample is kept in its own glass jar with saturated salt solutions and the weighing of the sample is made while the sample is still in the jar.

METHOD

Saturated solutions of different salts give different constant relative humidities (RH) over their surfaces [9] (unsaturated solutions may also be used if one keeps control of the concentration of the solution). When a sorption isotherm is to be measured with saturated salt solutions each sample is kept over a salt solution and weighed regularly. When the mass of the sample does not change anymore according to the equilibrium criterion being used, that mass is noted as the equilibrium mass of the sample at that RH. Thereafter the sample may be taken to another RH where the procedure is repeated. The moisture content of a sample is usually based on the dry mass of the sample and the sample is therefore dried before or after the measurement.

Figure 1 shows the set-up used in the present method. An ordinary 750 ml glass jar for food preserves with a lacquered metal screw lid is used. The sample holder hangs in a 0.70 mm stainless steel wire through a hole (5.0 mm) in the metal lid. The hole is covered with a polycarbonate washer (diameter 11 mm, thickness 1.6 mm) with a 0.80 mm hole in it that allows the wire to freely pass through. The washer is free to move on the upper surface of the lid. A small silicone rubber tube (inner diameter 0.64 mm) is placed on the wire to keep the sample pan in position above the salt solution and to seal the hole in the washer when measurements are not being done. The silicone tubing is cut perpendicular to its length to minimize leakage through the hole in the washer.

The top of end of the wire is made into a hook that can be attached to the below-balance hook at the bottom side of a balance for weighing. When taking a measurement the wire is attached to the balance and the jar is adjusted so that the wire is swinging freely, moving the washer slightly back and forth. After about 10 s when the swinging have stopped, the wire will go centrally through the hole in the washer without touching the washer. Then a reading is made on the balance and the wire is unhooked from the balance. The jars are placed on a glass plate during the weighing. Because of the low glass-to-glass friction this makes it easier to adjust the position of the jar to get stable readings.

In order to minimize the leakage and to obtain stable measurements the holes in the steel lids should be made in such a way that the top surfaces are perfectly flat. Otherwise, there will be leakages under the washers or the washers will not move freely on top of the lid making it difficult to get stable measurements. A too small hole in the washer will also give stability problems.

A Sartorius balance BP 221S with a below-weighing and a last digit of 0.1 mg was used. The balance was placed on a stand to enable below-balance weighing, see Fig.1. The measurements were performed in a 55% RH climate room with a temperature of 20±0.5 °C.

An important part of a method to measure sorption isotherms is the equilibrium criterion used. As different materials can take quite different times to reach equilibrium because of internal diffusion limitations and non-Fickian phenomena [10] it is important that the change in the sample mass with time is followed until it is certain that the sample has come close enough to equilibrium for the chosen purposes. It is not efficient to set a certain time when it is thought that equilibrium is attained and then only make one measurement.

In the present study the following criterion was used to decide when a measurement could be stopped:

$$\Omega = \frac{dm/m_i}{dt} \cdot \frac{\varphi_f - \varphi_i}{(m_f - m_i)/m_i} \qquad \text{(Eq. 1)}$$

The equilibrium criterion Ω takes into account the mass (*m*) of the sample and moisture capacity of the sample in the relative humidity (φ) interval used in the measurement (*i* and *f* refers to the initial and final states of a sorption step). The first factor is the fractional mass change rate. The second factor is the inverse of the moisture capacity expressed in terms of *m* and φ. When the major part of a sorption step has taken place $m(t) \approx m_f$ and we can write:

$$\Omega = \frac{dm}{dt} \cdot \frac{(\varphi_f - \varphi_i)}{(m(t) - m_i)} \qquad \text{(Eq. 2)}$$

In the present paper we have considered a sorption step to be finished when Ω is less than the rather arbitrarily chosen value of $5 \cdot 10^{-9}\ \mathrm{s}^{-1}$.

TEST MEASUREMENTS

We have used the method with glass jars to measure absorption isotherms for two materials at 20 °C: microcrystalline cellulose (MCC, Avicel PH-101, SERVA Electrophoresis, Heidelberg, Germany) and bentonite clay (commercial bentonite clay with the quality symbol MX-80 from American Colloid Co., which is a blend of natural Wyoming bentonite horizonts [11]). The MCC measurements were done at six different RH (33, 43, 54, 75, 85 and 95%) using saturated solutions of six salts ($MgCl_2$, K_2CO_3, $Mg(NO_3)_2$, NaCl, KCl, KNO_3). Samples of 3.5 g MCC were used and placed in aluminum sample pans that were hung about 2 cm above the salt solution surfaces. Three samples in individual jars were used at each RH. To increase the sample mass a stainless steel weight of about 20 g was attached to each sample pan. This was done to minimize leakage, cf. Discussion below. The samples were kept over the salt solutions for about two weeks when equilibrium was reached according to the equilibrium criterion. The dry mass of the MCC samples was determined by conditioning over drying agent (0.4 nm molecular sieves) in the method jars for ten days until equilibrium was reached.

The bentonite clay measurements were done at 6 different RH (11, 33, 59, 75, 85 and 93%) using saturated salt solutions of five salts (LiCl, $MgCl_2$, NaBr, NaCl, KCl) plus one aqueous unsaturated NaCl solution for 93% RH. Only one sample was used at each RH. The unsaturated aqueous solution was a two molal solution of NaCl. The vapor pressure p over this solution is 2179 Pa [12] and the saturation vapor pressure p_{sat} over pure water is 2339 Pa at 20 °C.

Since the relative humidity, RH, is defined as RH = p/p_{sat} the 2 molal NaCl solution at 20 °C gives RH = 93%. The jar with the unsaturated aqueous solution was exchanged once a week to minimize the effect of evaporation and leakage. Each solution was weighed before and after being used and the mass loss was found to be less than 2% of the total weight of the solution.

Samples of 10 g, with an initial moisture content of 0.4%, were placed a couple of cm above the saturated or unsaturated aqueous solutions in the same type of sample pans as used for the MCC. The initial state of the bentonite was a result of conditioning over drying agents for 28 days. Stainless steel weights, as mentioned above, were also used here. After 82 days the tests were finished (Eq. 2 gave a Ω less than $5 \cdot 10^{-9}$ s^{-1}). The samples were dried at 105 °C for 24 hours and then placed over molecular sieves (0.4 nm) in the glass jars and weighed. The weight after oven drying was regarded as the dry mass.

The sorption isotherm for bentonite clay MX-80 was also determined with a sorption balance (DVS 1000, Surface Measurement Systems, London, UK [13]). The initial weight of the material was 15 mg and the sample was initially dried in the instrument in dry nitrogen. The temperature during this test was

25 °C. Both absorption and desorption were determined in steps of 9.5%. Each sorption step took 5-20 h. The sorption balance measurements were not done with the equilibrium criterion being used for the jar measurements.

RESULTS

Figure 2 shows the absorption isotherm measured for MCC and Fig. 3 the absorption isotherm for bentonite clay. For MCC the highest standard deviation was 0.14% moisture content (at 85% RH).

DISCUSSION

There is a good agreement regarding MCC between the present method and the isotherm obtained with another method by the COST collaborative study [1]. The COST method used weighing bottles in which the samples were placed. The samples were first dried over phosphorus pentoxide and thereafter conditioned over saturated salt solutions in sorbostats. The measurements were done outside the conditioning environment and the equilibrium was determined on day 7 after the samples have been placed in the sorbostats. These measurements were done at 25 °C. The dry weight was determined after the samples had been dried in 105 °C for 4 h.

Figure 3 shows a comparison with results regarding bentonite from Kahr, Kraehenbuehl and co-workers [14, 15] determined with a gravimetric apparatus of the McBain type [16]. The initial weight was approximately 0.8 g and before the water uptake the material was dried for 24 hours at 100 °C and at a pressure of $10^{-4} – 10^{-5}$ mbar. The test was made at 20 °C.

The bentonite clay is used in this investigation as an example of a slowly sorbing natural material for which the jar method works quite well as it is possible to conduct long term studies without excessive costs. The results from the jar method agree well with both the sorption balance measurements and the results from Kahr, Kraehenbuehl and co-workers [14, 15] in the lower region of RH but show higher moisture contents in the high range of RH. To study this phenomenon further the increase in moisture content with time in two RH ranges is shown in Fig. 4. It is seen that for both methods the water content is still increasing when the measurements were terminated, but the jar measurements are closer to equilibrium as they have been continued for longer time. Our sorption balance measurements were not made with the equilibrium criterion used for the jar measurements. This time dependence in the water uptake is a possible reason for the difference in water content shown at higher RH in Fig 3. The very slow changes in moisture content at the end of the experiments with the present method are probably caused by slow re-organisation of the sorbed water, i.e. a type of non-Fickian behavior.

There are a number of advantages with the present method compared to most other commonly used simple experimental methods to measure sorption isotherms:

- There is no significant RH-disturbance to the sample during a measurement as there is only a minute hole between the washer and the wire. This is the main difference between the present method and the methods used by Gustafson & Hall [7] and Zuritz et al. [8] in which there was a rather large opening in the lid during each measurement.

- A sample is easily transferred from one RH-condition to another simply by moving the lid together with the sample from one jar to another. If this is done quickly in a suitable RH, the disturbance will be small (e.g. the sample will not change limb on the hysteresis loop).
- If the jars and the tubing are resistant to 105°C, the set-up can be used for drying samples at this standard temperature. This is further discussed below.

Errors from the measurements and uncertainties in the ambient conditions will cause an error in the determined points of moisture content as a function of RH on the sorption isotherm. This error is made up from mainly three parts: errors in the weighing, errors in the generated RH, and errors caused by variations in temperature.

A simple error analysis for the weighing may be made as follows. Consider a sample of dry mass m_0 and a mass m_1 at a moisture content u on a dry basis. Then:

$$u = \frac{m_1 - m_0}{m_0} \qquad \text{(Eq. 3)}$$

Assume that both mass determinations have absolute errors of Δm and that the errors have different signs (worst case). Then:

$$u + \Delta u = \frac{m_1 + \Delta m - (m_0 - \Delta m)}{m_0 - \Delta m} \qquad \text{(Eq. 4)}$$

Here, Δu is the error in the moisture content. If we solve Δu from Eqs. 3 and 4 and simplify the expression by noting that Δm is much smaller than m_0, and making the approximation valid for low moisture contents that $m_0+m_1 \approx 2m_0$ we get

$$\Delta u \approx \frac{2\Delta m}{m_0} \qquad \text{(Eq. 5)}$$

For the present set-up with glass jars we have found that repeated weighing gives us a standard deviation of about 0.2 mg, i.e., more than 99% (three standard deviations) of all results are within about ±0.6 mg. If Δm = 0.6 mg and m_0 = 10 g we get $\Delta u \approx 0.0001 = 0.01\%$. We therefore suggest the use of a balance with a last digit of 0.1 mg for samples of 10 g.

The relative humidity (RH) of the used saturated salt solutions may be found in Greenspan [9], who also gives uncertainty limits for each salt. The uncertainties estimated by Greenspan are 0.14-0.33% RH for all our salts except for KNO_3 that has a higher uncertainty of 0.66% RH. Except for KNO_3, these uncertainties are rather low.

Another factor that is often overlooked is the effect of hysteresis. Normally one measures an isotherm either in absorption (by initially drying the sample) or in desorption (by initially humidifying the sample). This works well as long as the RH is constant. However, if the RH fluctuates the samples will tend to absorb and desorb cyclically, resulting in scanning curves between the limiting absorption and desorption isotherms. The most common and important source of RH fluctuations in a sorption jar is probably fluctuations in temperature. If the temperature changes rapidly the air in the jar will not have time to stay in equilibrium with the salt solution. A temperature increase from 20 to 21 °C will cause the RH of a saturated NaCl solution to drop from 75 to 71% RH. A second effect is the temperature dependence of the RH of the saturated salt solutions. For some salts (NaCl) this effect is very small, but for others (e.g. $Mg(NO_3)_2$ and KNO_3) it is more important. NaCl has a temperature coefficient of 0.03%(RH)/K, whereas $Mg(NO_3)_2$ and KNO_3 have 0.30 and 0.18%(RH)/K, respectively [9]. Therefore the temperature should be kept constant or at least only allowed to change slowly.

Regarding the unsaturated aqueous solution another source of RH variations is the change in concentration. For a 2 molal NaCl solution a 2% increase in concentration will give a decrease in RH from 93.2% to 93.0% when interpolating between vapor pressures given as function of concentration by Clarke and Glew [12]. Regarding the sensitivity in RH for changes in temperature a slow increase from 20 to 25 °C will only give a decrease from 93.2 to 93.1% in RH determined from partial vapor pressures and saturated vapor pressures for each temperature given by Clarke and Glew [12].

The seal in the lid of sorption jar is not perfect. We have tested the sorption jars by charging them with a drying agent (0.4 nm molecular sieves) in the sample pans only and keeping them in a room with 30% RH. The measured leakage was about 10 and 1 mg per 24 hours for a light sample (8 g) and a heavy sample (28 g), respectively. A heavier sample will press the rubber tubing against the washer and the washer against the lid, and will thus reduce the leakage. When a sample of low mass is used it is therefore advantageous to add some extra mass, e.g. a stainless steel weight to reduce the leakage, as was done in the test measurements.

A leakage of 1 mg/day may seem to be quite high compared to the mass changes of the samples, but the error caused is negligible because of the very much higher capacity for absorption/desorption by the saturated salt solution than by the sample. The saturated salt solutions have sorption isotherms that are vertical, e.g. the RH does not change when the water content changes. The same is true for good drying agent. Normal samples have sorption isotherms with a limited slope. Consider a sample at equilibrium with the saturated salt solution. If water vapor that leaked into the sorption jar would be absorbed by the sample, the RH of the sample would increase. Then the sample would have a higher RH than the saturated salt solution and this would lead to a flow from the sample to the salt solution and a restoration of the original equilibrium.

We have further studied this problem by making finite element computer simulations with FEMLAB (Comsol, Stockholm, Sweden). For the case with a RH difference of 50% between the jar and the room a leakage of 1 mg per 24 hours through the seal changes the RH of the sample (at worst case stationary conditions with perfectly stagnant air in the jar) by less than 0.04%. This is less than the total uncertainty of the RH in the sorption jars. However, if the leakage is much higher than 1 mg per 24 h it will be difficult to accurately know the RH in the jars.

The method with glass jars have been used to measure equilibrium sorption isotherms. In principle the method could also be used to follow transient mass changes, e.g. to determine mass transport coefficients, but the rather large mass transfer resistance between the sample and the vapor surface/sink may influence such results considerably.

One advantage with the present technique is that one can easily move a lid with a sample from a jar with one salt solution to another jar with another salt solution. It is also possible to dry the samples in the jars to get the dry mass. Drying could either be with a drying agent or at 105°C. In the latter case the lid with the sample has of course to be opened, but at the end of the drying the lid can be closed and the dry mass determined. If the jar is insulated it is quite possible to make this weighing while the sample is still warm. As long as hot air from the warm jar does not disturb the balance we have found that we can make a measurement on a warm sample directly after ending the drying. A correction for the buoyancy of the sample may be needed.

CONCLUSIONS

We have developed an experimentally simple yet good device to measure sorption isotherms in which a sample is kept inside a glass jar during the whole measurement period. The following details should be kept in mind for the method to be accurate:

- The leakage will be minimized if the lid of the jar and the washer are perfectly flat.
- The tubing should be cut perpendicular to its length direction.

- The mass of the sample should be increased with a dummy mass if the sample itself is light.

ACKNOWLEDGEMENTS

We thank Bengt Nilsson and Natalia Markova for their help with previous versions of this method. This research was funded by FORMAS (LW, KS) and the Swedish Nuclear Fuel and Waste Management Company (AD).

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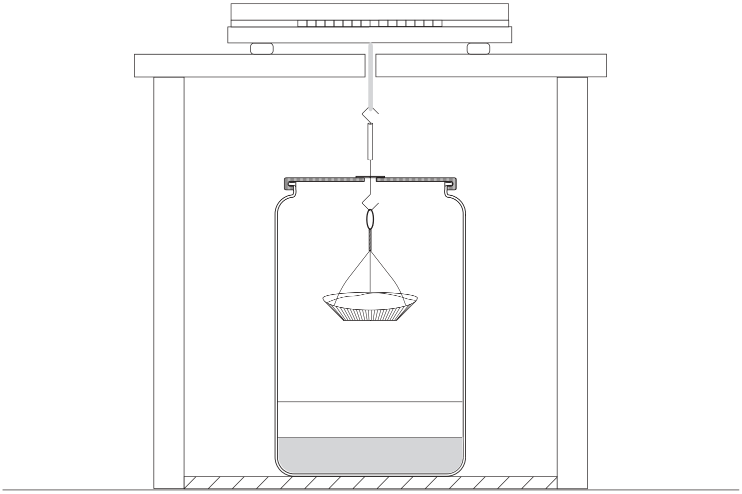


Figure 1. The measurement set-up with the sample in weighing position. The sample hangs in the wire that goes through the hole in the lid. This hole is made quite tight by the washer floating on top of the lid (and the rubber tubing on the wire that tightens the hole when weighing is not being made). Note that the glass jar stands on a glass plate so it is easy to place it in the correct position for a weighing.

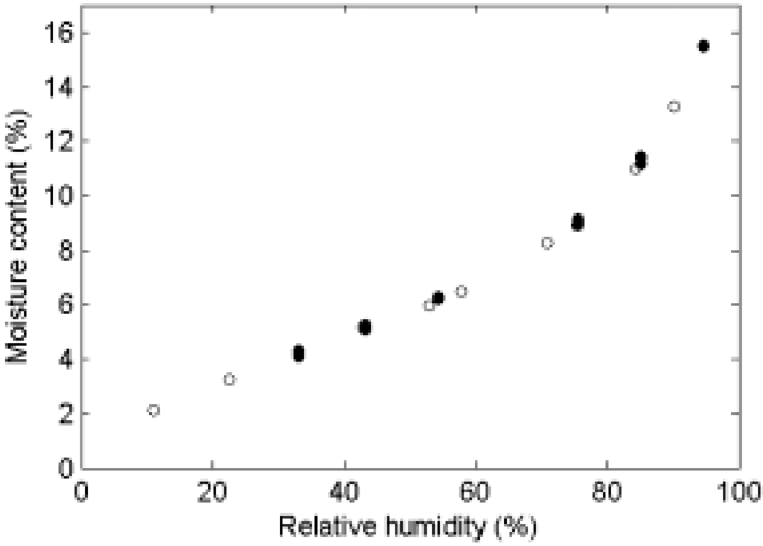


Figure 2. Measurements of the sorption isotherm for microcrystalline cellulose (MCC) made with the present method at 20 °C (filled circles) compared to literature values from Wolf et al. [1] measured at 25 °C (open circles). Moisture contents are calculated on a dry basis.

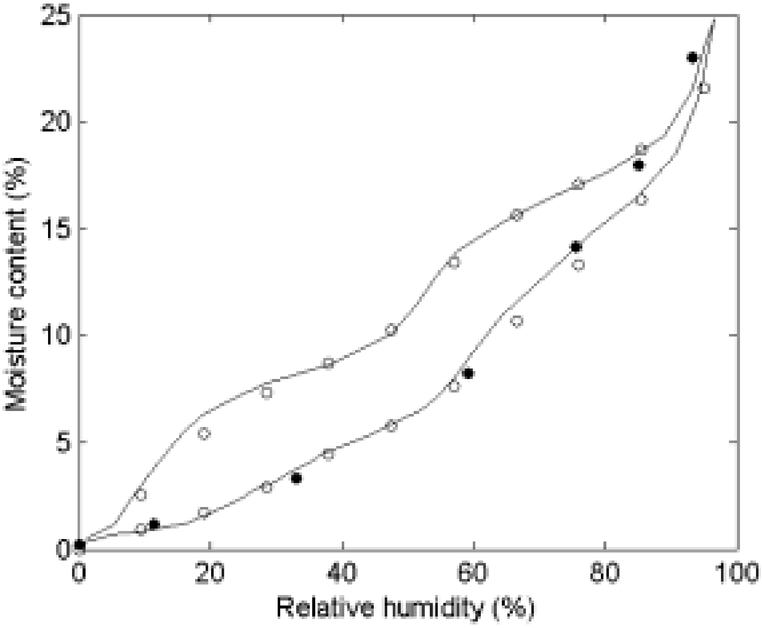


Figure 3. Measurements of the sorption isotherm for bentonite clay made with the present method at 20 °C (filled circles) compared to sorption balance measurements at 25 °C (open circles) and literature values from Kraehenbuehl et al. [12, 13] at 20 °C (solid line). Moisture contents are calculated on a dry basis.

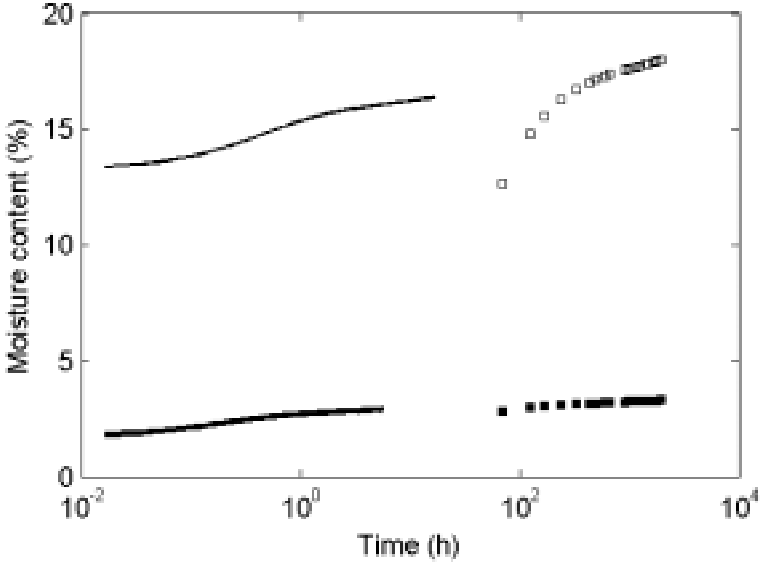


Figure 4. Increase in water content as a function of the logarithm of time for sorption steps made with a sorption balance and the present jar method, ending at approximately the same RH-values. Legend: sorption balance, final RH 28.5% (thick line); sorption balance, final RH 85.5% (thin line); jar method, final RH 33% (filled squares); jar method, final RH 85 % (open squares)

Paper III

Time-dependent moisture properties for plasterboard with surface coating

Svennberg, K. and Harderup, L-E. (2002).

Published in *Proceedings of the 9th International Conference on Indoor Air Quality and Climate –Indoor Air 2002,* Vol. IV, pp. 66-72. Monterey, CA, USA: Indoor air 2002.

TIME-DEPENDENT MOISTURE PROPERTIES FOR PLASTERBOARD WITH SURFACE COATING

K Svennberg*, L-E Harderup

Dept. of Building Physics, Lund University, P.O. Box 118, SE-221 00 LUND, Sweden

ABSTRACT

The level and fluctuations of indoor humidity plays an important role for the indoor air quality. The buffering effect of the internal surfaces in the building has been more emphasized lately. This study focus on the buffering effects for short time periods (hours, days) since the surface materials at this time scale may play a significant role. The objective is to determine the time-dependent moisture properties of plasterboard for indoor use and the different layers of a dispersion paint surface coating system. The effect of moistening and drying is studied in the interval RH 33–54% (+20°C). The surfaces step-response was determined through a gravimetrical method while the sample was in a well-defined relative humidity (RH). Calculated values for the moisture capacity, ξ, together with an evaluation of the experimental data are presented. The experimental results have a satisfying agreement compared with the calculations.

INDEX TERMS

Indoor humidity, plasterboard, Surface coating, Moisture capacity, Step-response

INTRODUCTION

The indoor air quality is to a great extent determined by the level and fluctuations of the humidity of the indoor air. The indoor humidity depends on a combination of factors such as moisture sources, ventilation rates, the occupants and their day-to-day activities such as cooking, bathing, washing and drying laundry. One factor that has been more emphasized recently is the buffering effect of the internal surfaces in the building and how this influence the indoor climate in the timescale hours to days. For this timescale the surface's capacity, to act as moisture buffers, may play an important role for the humidity levels and fluctuations, (Rode, Grau and Mitamura, 2001). These fluctuations of the indoor humidity can be simulated in a variety of ways (Harderup, 1998) either with cyclic or step variations. Also the thermal properties of the building envelope and the external climate influence the indoor humidity.

The overall objective for our research project is to develop a numerical hygrothermal calculation model based on an analytical model (Luikov, 1968) that can take into account moisture buffering as well as a varying humidity supply. The calculation model assumes step changes in the ambient climate and these step changes can be superposed. By superposing different step changes the fluctuations in the humidity can be handled properly. For the special case when the surface layer can be treated as semi-infinite, the solution can be represented by the complementary error function (*erfc*) (Harderup, 1983). To be able to develop the calculation models further, data concerning time-dependent moisture properties for surface materials used in the indoor environment is needed (Harderup, 1998). This study focuses on the step-responses and moisture properties for short time periods (hours, days) for plasterboard with different stages of surface coating and was studied in the relative humidity (RH) interval 33-

*Contact author email: kaisa.svennberg@byggtek.lth.se

54 % (+ 20° C). The ambient climate correspond to common Scandinavian indoor conditions during heating season (Norlén and Andersson, 1993).

METHODS

Materials

Plasterboard for indoor use (density 720 kg/m^3, thickness 12,5 mm) lined with paper on both sides was used. The surface coating system was of dispersion paint type according to the Swedish General Material and Implementation Descriptions (Hus-AMA98, 1998). The system consisted of: an oil emulsion primer, a water-soluble adhesive, one layer of glass fiber fabric (thickness 0,38 mm) and two coats of acrylate dispersion paint. The first coat of paint had a matt sheen and the topcoat a semi-matt sheen.

Sample preparation

10 samples were prepared from each of the six stages in the surface coating system resulting in a total of 60 samples. See Table 1.

Table 1. Description of the six sample groups.

Sample group **Description of stage**	**A**	**B**	**C**	**D**	**E**	**F**
Plasterboard	X	¤	¤	¤	¤	¤
Primer		X	¤	¤	¤	¤
Adhesive			X	¤	¤	¤
Glass Fiber Fabric				X	¤	¤
Paint - matt sheen					X	¤
Paint – semi-matt sheen						X

X- stage of surface coating system applied and exposed

¤- stage of surface coating system applied

The samples were sawed out of the plasterboard with a 63 mm diameter hole saw. The raw paper edges were trimmed and a thin stainless steel wire used for hanging was applied. A 50/50 mix of bees wax/paraffin wax as recommended in the Swedish standard for the cup-method (SS 02 15 82,1980) was used for sealing. A thin plastic sheet was cut and centered on the open side to mask it from the wax. Thereafter, the backside and the edges were sealed with the wax. The seal extended approximately 1 mm from the outside edge over the open side. After the samples were sealed they were conditioned at RH 33% respectively 54% for at least 6 weeks (Svennberg, 2002). The conditioning took place in the same type of equipment as used for the measurements see Fig. 1 below.

Five samples from each sample group were subjected to a step change (RH 33-54 %) resulting in a moistening (absorption) and the other five to a drying step change (desorption) in the humidity interval. The temperature was held constant (+ 20°C) throughout the whole experiment.

Measurement set-up

To provide a constant ambient climate for the samples and at the same time facilitate the step change in humidity a method developed by Wadsö (1995) was used. Stable humidity within each jar was obtained by saturated salt solutions (Fig.1d) [$MgCl_2$, $6H_2O$ (RH 33%) or $Mg(NO_3)_2$,$6H_2O$ (RH 54%), at + 20 °C] (Greenspan, 1977).

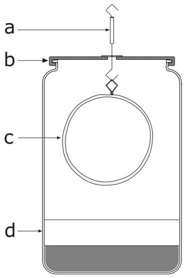


Figure 1. Measurement set-up in weighing position.

A 750 ml ordinary glass jar for foods with screw lid (Fig.1b) was used. The sample (Fig.1c) hanged on a stainless steel wire through a hole in the lid. The hole in the lid was covered with a thin plastic washer with a minute hole in it that only allows the wire to pass through. A small rubber tube was placed over the wire (Fig.1a) to seal the hole in the washer when measurements were not being done and to keep the sample in a vertical position.

Determination of the moisture absorption/desorption was made with below balance weighing by connecting the wire (Fig. 1a.) underneath the balance. This was done while the sample was in the jar and without any significant disturbances in the ambient conditions.

The accumulated moisture flow for a given time, |*G*|, was determined by repeating the measurement method above.

Determination of the exposed area

A digital camera was used to photograph the open side of each sample exposed to the step change in humidity. By fitting a circle on top of the digital picture in high resolution an equivalent exposed area was determined. The advantage of using this method is that adjusting for irregularities is easily done. The error is in the same magnitude as estimating the diameter with a slide caliper rule.

RESULTS

The measured weight differences were used for calculating the moisture absorbed and desorbed respectively. The absolute values of these moisture variations were plotted as shown in Fig. 2 (Svennberg, 2002).

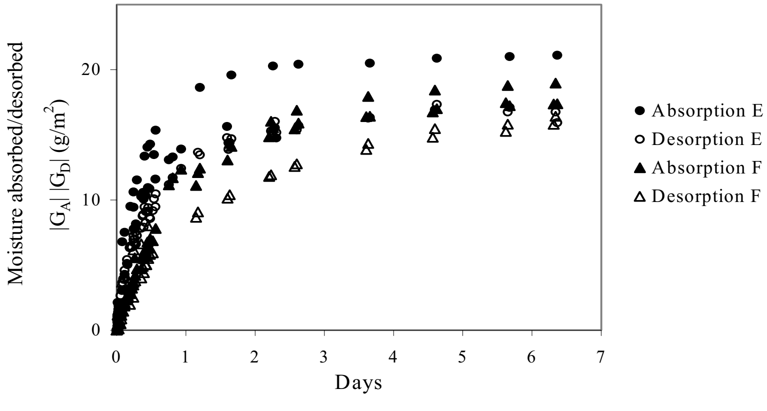


Figure 2. Absolute values of the moisture absorbed / desorbed for samples with 1 layer of paint (E) respectively 2 layers of paint (F). Only reliable data is shown in the figure.

A few measurements were found to be strongly deviating most likely due to mistakes during the experimental procedures. These measurements were discarded. Statistical evaluation of the experimental data was done using the unpaired Student's t-test. (P being the probability that series A = series B.)

Table 2. The moisture absorbed respectively desorbed for each sample group at 8 h, 2 and 6 days. The confidence interval is ± 2 %.

Moisture Absorbed / Desorbed (g/m^2),	**A**	**B**	**C**	**D**	**E**	**F**
Absorption, G_A t = 8 h	12,46	10,49	3,62	3,35	10,42	5,12
Absorption, G_A t =2 days	13,98	13,78	13,80	16,48	16,26	13,91
Absorption ,G_A t = 6 days	14,47	15,17	15,55	17,35	21,06	17,87
Desorption, G_D t = 8 h	-7,94	-8,38	-7,81	-9,85	-8,14	-5,32
Desorption, G_D t = 2 days	-13,43	-12,09	-14,19	-16,04	-15,03	-11,86
Desorption, G_D t = 6 days	-14,31	-13,36	*	*	-16,53	-15,83

*No reliable data available

At 8 h the moisture absorption for the samples with one and two coats of paint differs significantly (P = 0,007), see Fig.2. The order of precedence for the moisture absorption at 8 h agree with study made by Mauritz, Solar and Pfitzner (1990) on cyclic variations with 8 h period. At 2 days the glass fiber fabric (D) differs significantly from the underlying layers (A-C) (P = 0,021). The highest moisture absorption can be seen for the samples with 1 layer of paint (E) at 6 days, see Table 2. There is a significant difference in absorbed moisture at 6 days between sample groups (A-C) and (E-F) (P <0,01).

When the samples have reached equilibrium the moisture capacity, ξ (–), can be calculated.

$$\xi = \frac{G}{L \cdot \left(v_1 - v_0\right) \cdot G'} \quad (\text{-}) \quad G' \to 1 \text{ when } t \to \infty \qquad (1)$$

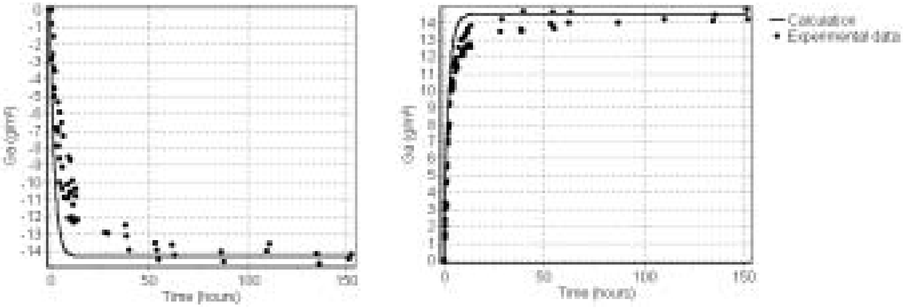
where G (g/m²) is the moisture uptake at equilibrium, L (m) is the thickness of the surface layer, v_0 and v_1 are the vapor concentrations (g/m³) of the air before and after the step-change, respectively. G' is a dimensionless function that describes the moisture absorption or desorption in the material at different times.

Table 3. Moisture capacities, ξ, mean values for each sample group (A – F).

Moisture capacity, ξ	**A**	**B**	**C**	**D**	**E**	**F**
Absorption, ξ_A	310,4 ±0,2	312,5 ±1,2	319,7 ±0,5	358,7 ±0,5	354,6 ±1,2	378,7 ±0,5
Desorption, ξ_D	306,5 ±1,2	304,1 ±0,6	345,7 ±1,3	362,3 ±1,7	379,8 ±0,9	417,9 ±1,0

It is worth noting that the moisture capacity, ξ, increases with an increasing number of layers in the surface coating system. The magnitude of the increase was not anticipated.

The calculated values for moisture capacity, ξ, (Tab. 3) was used as input data in a numerical calculation based on the model in (Harderup, 1983) and (Svennberg, 2002). The vapor permeability, δ_v, was set to 3,0 · 10^{-6} (m^2/s) (Hedenblad, 1996). The internal surface resistance, Z_i, is assumed to be 400 (s/m). In Fig. 3 the comparison between calculation and experimental data is shown. The agreement is relatively good.



a b

Figure 3. Comparison between experimental data and model calculations for plain plasterboard a) desorption $\xi_D = 307$, b) absorption $\xi_A = 310$.

Fig. 3 also shows that the step-response to a humidity step change can be divided into one initial rapid phase followed by a slower concluding phase. Preliminary analysis indicates that the first phase can be approximated with a polynomial of the 3rd to 5th order [2]depending on the surface coating of the sample (Svennberg, 2002). The concluding phase appears to be more linear. More analysis is needed for statistical justification.

DISCUSSION

Moisture absorption and desorption

A difference in absolute amount of moisture desorbed compared with the amount absorbed (Tab. 2) and (Fig. 2 & 3) is noticed as expected. This hysteresis phenomenon is expected to increase for higher levels of relative humidity.

There is also a difference between the absorption and desorption curves in what we call the "transition area" connecting the rapid initial phase with the slower concluding phase. The curves of desorption tend to be more blurred in this area compared to the absorption curves (Fig. 2). The "transition area" is also where the largest error between calculations and experimental data is seen, (Fig. 3). When the step-response is attempted to be divided into two approximations, one covering the initial rapid phase and one covering the more linear and slower concluding phase, special attention should be paid to the "transition area" to minimize the error.

Moisture capacity, ξ, and the surface coating system

The most conspicuous result from this study is that the moisture capacity, ξ, increases with the increasing number of stages in the surface coating system (Tab. 3). The assumption was that the surface coating had a negligible moisture capacity. Our study shows that for the investigated system with a glass fiber fabric backing the dispersion paint there is probably a significant moisture capacity. A probable cause is the asperity and porosity of this type of surface coating. Verification of the results is needed.

Correlating the data in Tab. 3 with the absolute values of moisture absorption (Tab. 2) indicates that asperity of the surface due to the surface coating will have an important role for the moisture processes. This area needs more penetration both theoretically and experimentally.

CONCLUSIONS AND IMPLICATIONS

The experiment will continue with a number of step changes up to RH 85 %. Measurements from several step changes facilitate to consider the variation in ξ at different intervals of relative humidity. The moisture properties of surface coating systems, such as vapor permeability, $\delta_{v,}$ and moisture capacity, ξ, should be more carefully studied.

The numerical solution will speed up if the surface step-response division into two approximations is realizable without introducing large errors. Further evaluation of the experimental data in comparison with the analytical model is needed.

The calculation model will also be validated with periodical climate chamber experiments in collaboration with dr. C. Rode at DTU, Denmark and field measurements.

ACKNOWLEDGEMENTS

The financial support by the Swedish Research Council for Environment, Agricultural Sciences and Spatial Planning and the Development Fund of the Swedish Construction Industry is gratefully acknowledged.

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ISRN LUTVDG/TVBH--03/3042--SE(80)
ISSN 0349-4950
ISBN 91-88722-27-9