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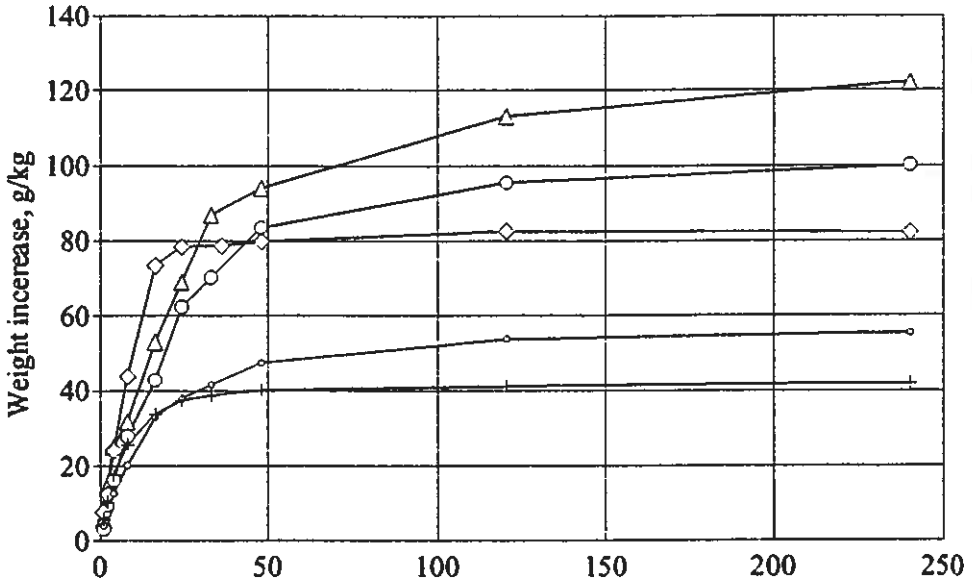

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A Standardised Approach for the Measurement of Hygroscopic Properties of Food Materials*

Beitrag zur Standardisierung der Messung hygroskopischer Eigenschaften von Lebensmitteln

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This paper presents a new practical approach for the standardised measurement of the hygroscopicity of food and related materials. The approach considers three aspects including the weight gain, the kinetics of the water uptake and the textural changes involved. The water vapour sorption measurements are performed using the standard technique developed within the COST 90 project (European Cooperation in the Field of Scientific and Technical Research). The test material is exposed to a relative humidity of 75% at 25°C and the weight gain determined gravimetrically at predetermined intervals. The water uptake at equilibrium (H_w) and an apparent diffusion coefficient (H_d) are calculated. A third textural parameter (H_t) which is of interest for the specific product must be defined and measured separately in the same humidity range. Experimental values of H_w and H_d are presented for milk powder, Na-caseinate, micro crystalline cellulose and starch.

Es wird ein Konzept zur Bestimmung der hygroskopischen Eigenschaften von Lebensmitteln und verwandten Materialien vorgestellt. Das Konzept berücksichtigt drei Aspekte der Hygroskopizität: die Menge und Kinetik der Wasseraufnahme unter standardisierten Bedingungen und die damit verbundenen Texturänderungen. Die Messung der Menge und Geschwindigkeit der Wasseraufnahme wird mit der im Rahmen der Aktion COST 90 entwickelten, einfachen Sorptionsmessung realisiert. (COST: Europäische Zusammenarbeit auf dem Gebiet der wissenschaftlichen und technischen Forschung.) Das zu prüfende Material wird bei 20°C einer relativen Luftfeuchtigkeit von 75% ausgesetzt und in vorbestimmten Intervallen gewogen. Aus den Gewichtszunahmen wird ein Gleichgewichtswassergehalt (H_w) und ein Diffusionsparameter (H_d) berechnet. Der dritte Parameter, der die strukturellen und funktionellen Änderungen reflektiert (H_t), muss für jede Produktegruppe speziell definiert und unter den gleichen standardisierten Bedingungen gemessen werden. Experimentell ermittelte H_w- und H_d-Werte für Milchpulver, Natriumcaseinat, mikrokristalliner Cellulose und Stärkepulver werden als Beispiele vorgestellt.

Introduction

Dried materials are commonly defined as hygroscopic or non-hygroscopic depending on their ability to take up moisture from the surrounding atmosphere. The water uptake effects, in most cases, the functional or sensory properties, processing characteristics and shelf of the materials. Hygroscopicity is therefore of major industrial and practical importance and there is a need to develop methods to quantify this physical property. The difficulty in quantifying hygroscopicity lies in the lack of suitable practical definitions and satisfactory standardised measuring procedures.

A combined, semi-empirical approach is proposed in the present paper, where hygroscopicity is characterised by three relevant parameters: two sorption-related parameters, the total water uptake after equilibration with the surrounding atmosphere and the apparent water diffusion coefficient and a third textural parameter which characterises the changes in mechanical or rheological properties.

Concept

Equilibrium approach

The equilibrium approach is based on the adsorption isotherm and focuses on the weight gain of the product tested when exposed to a humid atmosphere. The difference in water vapour pressure (Karel, 1974; Markowitz and Boryta, 1961; Pawlikowski et al., 1959) or the slope of the sorption isotherm can be considered as a measure of the driving forces. «Hygroscopic points» or «moisture pickup onset» have been defined as the relative humidity (r.H.) at which the material takes up a certain amount of water (Modrzejewsky and Poskora-Bartyzel, 1966, Sinclair, 1978). These approaches, however, do not take into account the kinetic aspect and the structural situation (Bondareva et al., 1978) nor the textural changes which are in many cases relevant in practice. A standard method based on the equilibrium water content after exposure to water vapour has been proposed for milk powder (Haugard Sorensen et al., 1978). The water uptake in a equilibrium situation after exposure of the product to a relative humidity of 75%, will be defined in this paper as H_w (g/kg).

Kinetic approach

The rates of water vapour sorption should also be considered when quantifying the hygroscopic property of a food material. The rank orders based on the weight gain after predetermined time intervals has been used as hygroscopicity scale for inorganic materials (Sinclair, 1978). These rank orders, however, are not satisfactory indicators of hygroscopicity. If the moisture transfer in food materials is controlled by diffusion, the process can be described by Fick's law and the diffusion coefficient can be determined experimentally. A simplified solution of Fick's law for the conditions of the sorption experiments described below is shown in Eqn. [1] (Crank, 1976).

$$\frac{M_t}{M_\infty} = \frac{2}{h}\sqrt{\frac{D \cdot t}{\pi}} \qquad \text{Eqn. [1]}$$

where:

M_t = mass of adsorbed water at time t
M_∞ = mass of absorbed water at time ∞ (equilibrium)
D = diffusion coefficient
h = sample thickness.

If the weight gain is plotted vs. the square-root of time, an approximate diffusion coefficient can be calculated from the slope of the regression line according to this equation. In practice, acceptable linearity is usually found in a limited segment between about $M_t/M_\infty = 0.2$ and 0.8. The parameter proposed for this approach, as a measure of the diffusion rate, is H_d (m^2/s) and corresponds to the slope of the water uptake vs. $\sqrt{t}$-regression lines in the range mentioned above.

Textural approach

Hygroscopic materials change their textural properties and fine structure to various extents upon absorption of water. Lumpiness, stickiness, swelling, change in flow properties, crispiness etc. often accompany the water uptake and are sometimes the determining criteria for the use or acceptability of the product. Therefore, a textural parameter H_t is proposed as a measure of the change in this specific physical or functional property. This parameter

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must be defined specifically for the product and its use. Rheological, mechanical or sensory test can be used to measure and quantify the desired property. Because of the great variety of criteria and different nature of products this paper does not include the description of techniques for the measurement of H_t.

Combined approach

The concept proposed in this paper consists of the combined approach, where the two sorption related parameters (H_w, weight gain at equilibrium and H_d, initial apparent diffusion coefficient) and the textural parameter (H_t) are determined by exposing the material to a relative humidity of 75% (saturated NaCl solution) at 25°C under standardised conditions, using the technique developed within the COST 90 project (Spiess et al., 1983).

Experimental

Materials

The following products were used as model systems for the determination of H_w and H_d: skim milk powder (MSK-AGGL, 35A, 1.002/AS, KORECO CO., CH-3510 Konolfingen), starch powder (MERCK, no. 11685, lot K11331085), soluble starch (MERCK, no. 1252, lot F497352, Na-caseinate (SIGMA, no. C-8654, lot 109F0022) and micro crystalline cellulose (MCC, MERCK, no. 2330, lot 4180309). Since reliable sorption data are available for MCC it can be used as reference material for testing the equipment (Wolf et al., 1984). The initial water content of the products was determined by Karl Fischer titration (Rüegg and Moor, 1987). The temperature was risen to 50°C during the titration. The water contents (means and standard deviations of three replicates) were for skim milk powder 57.4 (0.5), starch 112.7 (1.2), soluble starch 78.0 (1.3), Na-caseinate 84.1 (0.5) and, MCC 48.8 (1.0) g/kg dry matter.

Sorption measurements

Sorption measurements were made using the gravimetric technique described in detail by Spiess et. al. (1983). The samples were inserted into weighing bottles to a height of 5 mm and the exact weight determined. 50 samples of each product were divided into 10 recipients containing a solution of saturated sodium chloride. The measurements were performed in a thermostated room at 25 ±1°C. Always one recipient was opened after 1, 2, 4, 8, 16, 24, 33, 48, 120 and finally 240 h, and the weight gain (M_t) determined. For practical reasons the sorption devices were prepared in three series. The first series for the weighings after 1, 2, 4 and 8 h exposure had to be prepared in the morning. The second series for the data points at 16, 24 and 33 h were prepared at the end of the same day. The third series for the data po-

Material	H_w, g/kg		H_d, 10^{-10} m²/s		$t_{1/2}$, h	
	$\bar{x}$	s_x	$\bar{x}$	s_x	$\bar{x}$	s_x
Soluble starch	122.9	2.4	9.7	1.9	14.5	2.6
Sodium caseinate	98.2	5.5	12.4	1.7	11.2	1.5
Skim milk powder	83.0	2.1	44.6	2.3	3.1	0.2
Starch	54.8	0.6	17.9	1.1	7.7	0.5
Micro crystalline cellulose	40.9	0.9	30.5	5.9	4.6	0.8

H_w: total water uptake at 75% r.H. and 25°C (g/kg of initial weight)
H_d: apparent diffusion coefficient, estimated from the linear segment of the water uptake vs. √t-regression lines
$t_{1/2}$: time required to gain 50% of the equilibrium water content

Table 1: Hygroscopicity data of some food and related materials at 75% r.H. and 25°C

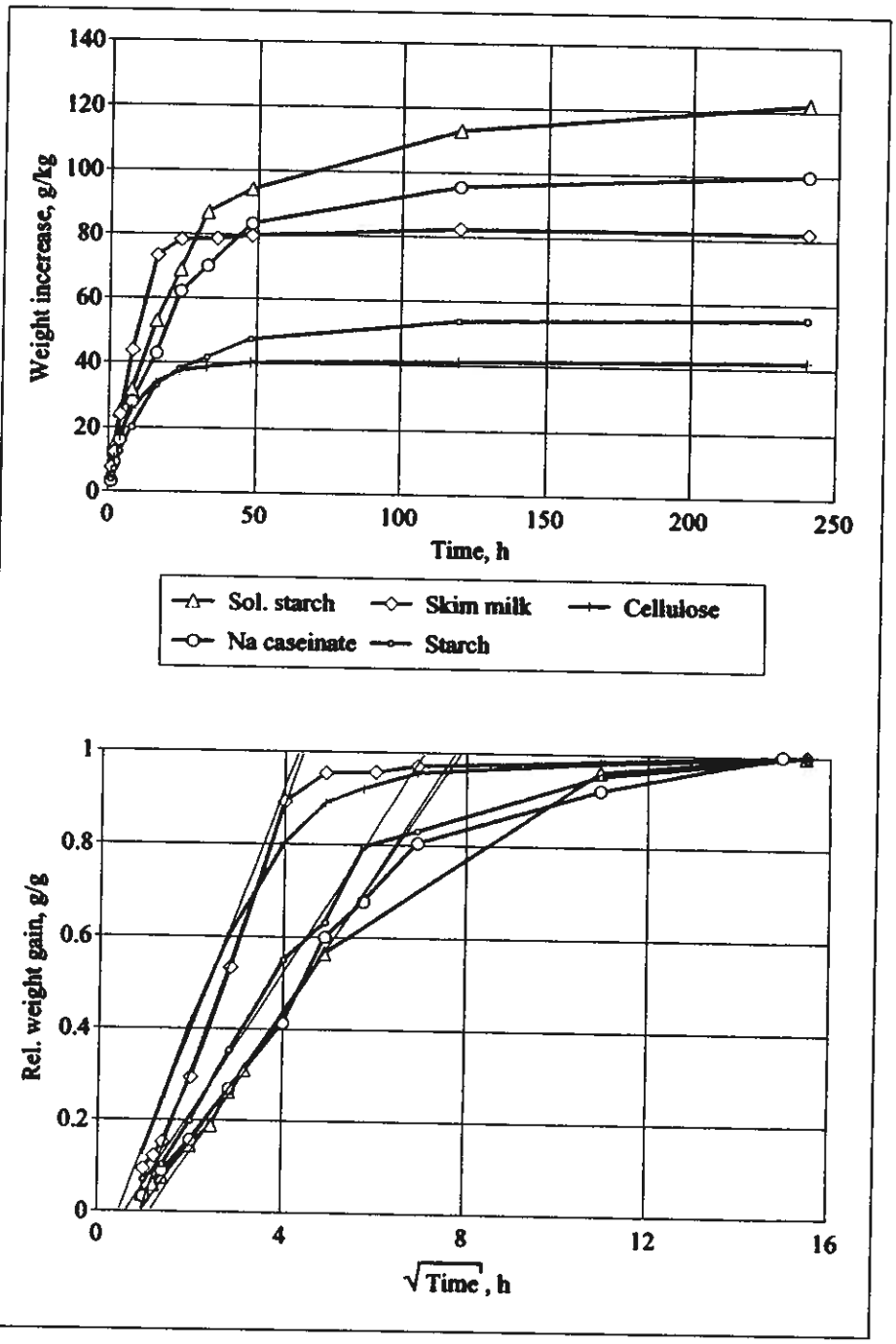

Fig. 1.

ints after 48, 120 and 240 h were performed separately. The total water uptake (M_∞) was calculated from the weight obtained after 240 h. The apparent diffusion coefficient was estimated from the slope of the regression line fitted to the data points within the range of relative weights of about 0.2 to 0.8 using equation [1] (linear segment of curves of M_t/M_∞. vs. $\sqrt{t}$).

Results

Fig. 1 shows the weight increase of the products tested as a function of time as well as the relative weight gain as a function of $\sqrt{t}$. The hygroscopicity data calculated from these curves are listed in **Table 1.** The means and standard deviations were calculted from 3 independent sorption experiments containing 5 replicates each. The products are listed in the order of increasing H_w-values.

For comparison, the time required to gain 50% of the equilibrium water content ($t_{1/2}$) was added in **Table 1.** For the standard experimental set-up, the $t_{1/2}$-values could alternatively be used as a measure of the rate of water uptake.

Conclusions

The concept proposed allows the characterisation of the most relevant aspects of hygroscopicity of food products. The experimental determination does not require complicated equipment and is based on a established technique developed within an international COST project (Spiess et al., 1983). Reference materials, such as microcrystalline cellulose, are available for the validation of the method (W. Wolf et al., 1984). In some cases the two parameters related to the sorption isotherm, the equilibrium water content H_w and the apparent diffusion coefficient H_d, are sufficient for the characterisation of the hygroscopic properties of the product. The textural parameter, which is not discussed in detail in this paper, must be defined specifically for each product.

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