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POSTER PRESENTATIONS

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Method to Determine the Swelling Degree in Polymers

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Modular Ceramic-Polymeric Device for Analysis of Selected Elements in Liquid Using Microplasma

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Comparative Studies of Chemoresistive Gas Sensors Based on Multiple Nanowires and Arrays of Single Nanowires

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Development of a Device for Staged Determination of Water Activity and Moisture Content

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Development of a device for staged determination of water activity and moisture content

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Abstract: Moisture content and water activity are important parameters for quality characterization of products like bulk materials, powders, granules. Thus, an exact determination is necessarily required in a wide range of industrial applications. Moisture of materials is the content of non-chemically bound water in a solid or liquid. Water activity (*aw*) is a characteristic/parameter of the non-chemically bound ("free") water in materials and is measured as humidity over a solid/liquid surface at constant temperature (equilibrium moisture content). It is an important parameter to characterize the quality of e. g. pharmaceutical and food products. In our contribution, we present the developed MOISHUM device for staged determination of water activity and moisture content

Keywords: water activity, moisture, coulometric humidity sensor, MOISHUM

1. Introduction

The occurrence of water is essential for our daily life. It is omnipresent and has a great influence on nature and technology. Despite the indispensable necessity of water, it is also one of the most frequent contaminants and disturbances in technical and food areas. Among other things, it influences the quality and properties of materials. While a high-water content is desired in some goods, it can also lead to a reduction in product quality and an associated loss in value as well as function. The moisture in the material as well as the water activity have decisive influences on the product quality, shelf life of food or mould growth on building materials. This influence of moisture makes the quantification of moisture content and water activity as an important parameter for quality control, quality assurance and consumer protection.[1]

Moisture describes the presence of water in solid materials. This water can be adsorbed or desorbed by a material. In the literature moisture is described with various parameters. The moisture is defined as water mass m_w , which desorbs after drying. A common term for describing material moisture is the gravimetric water content W_g . This expresses the percentage of the water mass to the total mass of a material.

A reliable determination of humidity and moisture requires the integration of suitable measuring technology. In a ZIM project of the German Federal Ministry of Economic Affairs and Energy, an innovative measuring system named MOISHUM was developed, which allows the traceable, reproducible and reliable determination of moisture (MOIS) and water activity (HUMidity) of powdered materials in a staged process. Here are presented the results on powdered coffee.

2. Experimental setup

Figure 1 shows the designed MOISHUM device. The designed MOISHUM device includes two humidity sensors (a capacitive and a coulometric[2]) two Pt100 temperature sensors and two Peltier devices for precise temperature control of the chamber. An analytical balance AUW120D (Shimadzu) was used for mass determinations in comparison to the MOISHUM values.

The measurement for the a_w value determination started by moving the sample holder to position two Figure 1 (b). The temperature and in this case the equilibrium relative humidity ($U_{w,ERH}$) [1] which correspond to water activity are determined by an SHT-25 sensor (Sensirion).

$$a_{\rm w} = U_{\rm w,ERH} / 100 \%$$
 (1)

The water content of the investigated powdered coffee is determined by coulometric sensors.[2,3] These humidity measurements bases on FARADAY's law of electrolysis. Equation 2 describes the relationship, where m_w is the electrolytically determined water mass, Q is the charge amount, M_v the molar mass of water, F Faraday's constant F = 96485.33289 C·mol⁻¹ and z the number of exchanged electrons (z = 2).

$$m_{\rm w} = (Q \cdot M_{\rm v})/(F \cdot z)$$
 (2)

3. Results

3.1. Functionality test

The functionality of the measuring device was severally tested by putting amounts of distilled water into the aluminum sample holder. After closing the sample holder with a lid, it was inserted into the MOISHUM adapter. In the first step, it was pushed to the top position of the grid element and the lid screwed into the drilled thread. In the second step, the position of the sample holder was moved to the middle position of the raster element for a_w -value determination at set temperature. In this position, the sample is exposed to the capacitive humidity sensor (SHT25, Sensirion) that measures the humidity above sample at constant temperature. In this case, the a_w -value was determined without being influenced by the coulometric sensor. After approximately 30 min, the measured a_w -value raises to 0.99. The absolute deviation from the measured a_w -value compared to the theoretical of water $a_w = 1.00$ is $\Delta a_w = -0.01$. Thus, the SHT25 sensor has an uncertainty of 4 % at relative humidity above $U_w = 90$ %, this difference is negligible. In the third step, the water amount was electrolytically determined by increasing the sample holder temperature to (105 ± 0.2) °C. The relative deviation in comparison to the measured water amount on an analytical balance ranges between -2.2 and 2.2 %.

3.2 Water activity, moisture determinations and sorption isotherm of a powdered coffee

The MOISHUM measuring cell was applied for the determination of water activity, water content and sorption isotherm at 25 ± 0.2 °C. The determined a_w value by MOISHUM measuring cell results in a relative humidity of 22.5% after 16 minutes which corresponds to an a_w -value of 0.225. The control measurement with the a_w measuring instrument (HygroPalm, Rotronic) determines a a_w value of 0.27 and the literature defines an a_w value from 0.1 to 0.3.[4] Thus, both values are within the limit for coffee. In general, water activity can be characterized as a) dry up to $a_w < 0.6$, b) semi-humid $a_w = 0.6$ to 0.85 and c) humid $a_w = 0.85$ to 1.00.

The relation between water content and the water activity can be given by sorption isotherms.[1,5] Figure 3 shows the determined sorption isotherm at 25 °C by MOISHUM measuring cell and in comparison, to a reference. The applied model function is the Guggenheim,

Anderson, and de Boer (GAB) model.[6]

$$Wg = \frac{X_{m}ZK_{a_{w}}}{(1-Ka_{w})(1-Ka_{w}+CKa_{w})}$$

where W_g is the water content, a_w is the water activity value and the parameters Z, K, Xm.

The course of the sorption isotherm is in accordance with the values from the reference.[7] However, there are deviations in the higher a_w value range.

3.2. Figures and Tables

Table 1. Overview of electrolytically determined amounts of water by MOISHUM measuring cell.

Initial water mass mwo / mg	Q/C	Determined mw/mg	Relative deviation $\Delta m_w / \%$
10.1	105.831	9.88	-2.2
1.93	20.667	1.93	0.0
2.10	22.059	2.06	-1.9
2.04	21.952	2.05	0.5
1.80	19.275	1.80	-2.2
1.84	19.703	1.84	2.2

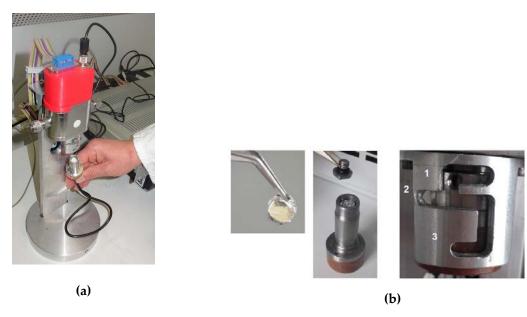


Figure 1. View on the MOISHUM measuring cell **(a)**, aluminum sample cap for the sample under test | sample holder and sample cap | process steps element **(b)**.

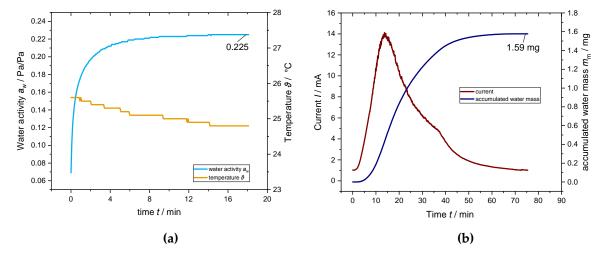


Figure 2. Stepped process results on the determination of a_w -value (a) and water amount m_w (b) on a powdered coffee sample by the MOISHUM measuring cell (DC voltage 18 V, Shunt resistance 100 Ω).

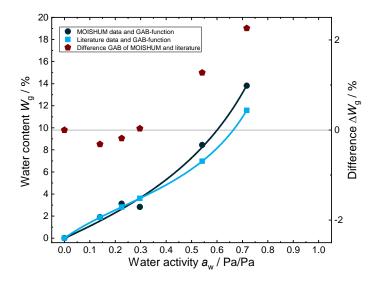


Figure 3. Sorption isotherm at 25 °C of powdered coffee with MOISHUM data and GAB-function in black and the difference in brown pentagons - MOISHUM GAB-parameters: Xm = 8.230, C = 1.556 and K = 0.822 in comparison to Cepeda et al.'s parameter: Xm = 4.203, C = 4.186 and K = 0.941.[6]

4. Conclusion

The MOISHUM measuring cell was developed for staged a_w value and water content determination. MOISHUM's applicability was tested and proven on powdered coffee in comparison to data from literature. The measuring times for a_w values are in the range of 20 min to 90 min (5 % to 95 % relative Humidity) and for water content determinations in the range 45 min to 180 min.

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Conflicts of Interest: The authors declare no conflict of interest.

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