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Determination of sorption isotherms of lemon balm using the dynamic vapour sorption

Agricultural and food materials absorb or release moisture during storage. This has a great influence on the stability of the dried product. The sorption isotherm is defined as the equilibrium relationship between the moisture content in the material and the relative humidity of the atmospheric surroundings at a given temperature. The dynamic vapour sorption (DVS) is a modern technique which can produce sorption data at different temperatures with high accuracy under controlled conditions in a short period of time. The adsorption isotherms of lemon balm leaves were established at temperatures commonly found in storage and the recommended values for their adequate preservation were computed by the Halsey model.

Keywords
Lemon balm, *Melissa officinalis* L., sorption isotherms, dynamic vapor sorption, drying, storage, shelf life

Abstract
Landtechnik 66 (2011), no. 2, pp. 88-91, 5 figures, 6 references

Lemon balm (*Melissa officinalis* L.) is a perennial herb of the family Lamiaceae, cultivated for the characteristic lemon-scented leaves. It is implemented for several purposes in the food, pharmaceutical and cosmetic industries due to its flavoring, medicinal and therapeutic properties. The most common method for processing and marketing of medicinal plants is by convective air drying in order to reduce the moisture content to a level for safe storage [1]. Knowledge of the equilibrium relationship between the moisture content in the material and the relative humidity of the atmospheric surroundings at a given temperature is of great importance. This relationship, known as sorption isotherm, predicts the physical, chemical and microbiological changes which can occur during drying and storage. Many methods to establish sorption isotherms usually rely on the principle described in the European COST study [2]. The static gravimetric method using thermally stabilized desiccators filled with saturated salt solutions provides accurate measurements, however, the time required to reach equilibrium can be extremely prolonged (>1 month), especially at higher target values of relative humidity. Recently, automated devices for the determination of sorption isotherms have been constructed to overcome some of the drawbacks associated with the standard gravimetric method. For instance, the dynamic vapor sorption (DVS) is a relatively new technique designed to measure the weight change caused by adsorption or desorption of the water vapor at any desired relative humidity in a short period of time. Limited studies on sorption isotherms have been reported in the fields of food and agricultural engineering using a dynamic, gravimetrical and fully controlled system [3; 4]. Consequently, the aim of the current work is to investigate the sorption behavior of *M. officinalis* leaves at three different temperatures by dynamic vapor sorption and to describe the experimental data using the appropriate moisture sorption model.

Material and methods

Plant material

Fresh plants of lemon balm (*M. officinalis*), cultivar Citronella were collected from an organic farm at Magstadt (Germany) in June and July before flowering (**figure 1**). Prior to experiments, the leaves were manually separated from the stalks.

The adsorption isotherms of *M. officinalis* leaves have been measured at temperatures commonly found in storage of medicinal plants by dynamic vapor sorption at the Reutlingen Research Institute, Reutlingen University using a gravimetric DVS-1000 analyzer (Surface Measurement Systems Ltd., London, U.K.) as it is depicted in **figure 2**. The instrument essentially consists of a Cahn microbalance with two sample crucibles made of quartz and the humidifier in a temperature controlled chamber. One of the crucibles is used for reference and the other contains the sample to be analyzed. A stream of dry and wet nitrogen gas flows along the crucibles. The relative humidity of the mixture is regulated by two electronic mass flow controllers. The apparatus is computer controlled, allowing the pre-programming of the set conditions and the continuous measurement of temperature, humidity and mass during the process.

Fig. 1

Fig. 1: Fresh lemon balm herb (M. officinalis)

Experimental procedure

Pre-dried sample with a mass varied from 10 to 15 mg was used for each experiment. The adsorption isotherms were determined at temperatures of 25, 35 and 45 °C by exposing the material to different values of relative humidity within the range of 0 and 95%. The sample was first dried by exposure to dry nitrogen until a constant weight of the sample was reached. Then, the relative humidity was subsequently increased stepwise and the sample weight was equilibrated at each step. Equilibrium was considered to have been reached when the change in mass became lower than 0.001 mg/min or the equilibration time had exceeded 360 min. To obtain the adsorption isotherms the moisture content of the sample in gram water per 100 gram dry solids (g/100 g d.b.) was directly provided by the equilibrium values of each relative humidity step. The measurements were performed in triplicate for each temperature at a total of fifteen target values of relative humidity.

Mathematical model

The Halsey equation was selected to fit the experimental data due to the fact that the model has already been tested for its effectiveness to describe the sorption isotherms of several medicinal plants [5; 6].

$$M_e = \left[\frac{-\exp(a + b \cdot T)}{\ln(a_w)}\right]^{1/c} \qquad \text{(eq. 1)}$$

$$a_w = \frac{RH_e}{100} \qquad \text{(eq. 2)}$$

where M_e is the equilibrium moisture content of *M. officinalis* leaves [g/100 g d.b.], T is the temperature [°C], a_w is the water activity [-], a, b and c are the model coefficients, RH_e is the equilibrium relative humidity [%].

Results

The experimental procedure performed to construct the adsorption isotherms of *M. officinalis* leaves by the dynamic vapor sorption method at 25 °C is shown in **figure 3**. The dry reference mass was established in approximately 16 hours. The effect of relative humidity on adsorption can be observed as an increase in mass. The time required for all samples to reach equilibrium was 6 hours except those ones equilibrated at values of relative humidity higher than 90 % which exhibited longer periods. The data from the sorption profile were further used to estimate the equilibrium moisture contents at the different target values of relative humidity. **Figure 4** shows the experimental adsorption isotherms of *M. officinalis* leaves at 25 °C obtained from a triplicate experiment. The curves closely resemble the characteristic sigmoid shape of the type II pattern isotherm indicating relatively small amounts of water at low values of relative humidity and exhibiting an asymptotic trend as relative humidity approaches 95 %. Furthermore, the experimental sorption data fitted by the modified Halsey equation are shown in **figure 5**. As expected, the equilibrium moisture content of leaves increased with relative humidity at constant temperature. An increase of temperature resulted in a slight decrease of the equilibrium moisture content of leaves at constant relative humidity. The equilibrium moisture content of the *M. officinalis* leaves at the hygienically safe water activity of 0.6 is 11.9 ± 0.78 gram water per 100 gram dry solids. In other words, to avoid microbial growth or spore germination at 25 °C, lemon balm is recommended to be stored at a maximum moisture content of 10% wet basis.

Conclusion

The dynamic vapor sorption method has been successfully employed for the determination of the adsorption isotherms of *M. officinalis* leaves. The DVS method produced equilibrium moisture content data in the entire working range of relative humidity under controlled conditions with high accuracy. Moreover, the relatively low sample mass used significantly reduced the time for the material to reach equilibrium. The modified Halsey equation adequately described the sorption characteristics of *M. officinalis*. The recommended values of moisture content for the adequate storage of lemon balm leaves at any temperature between 25 and 45 °C can now be computed by the model.

Fig. 2

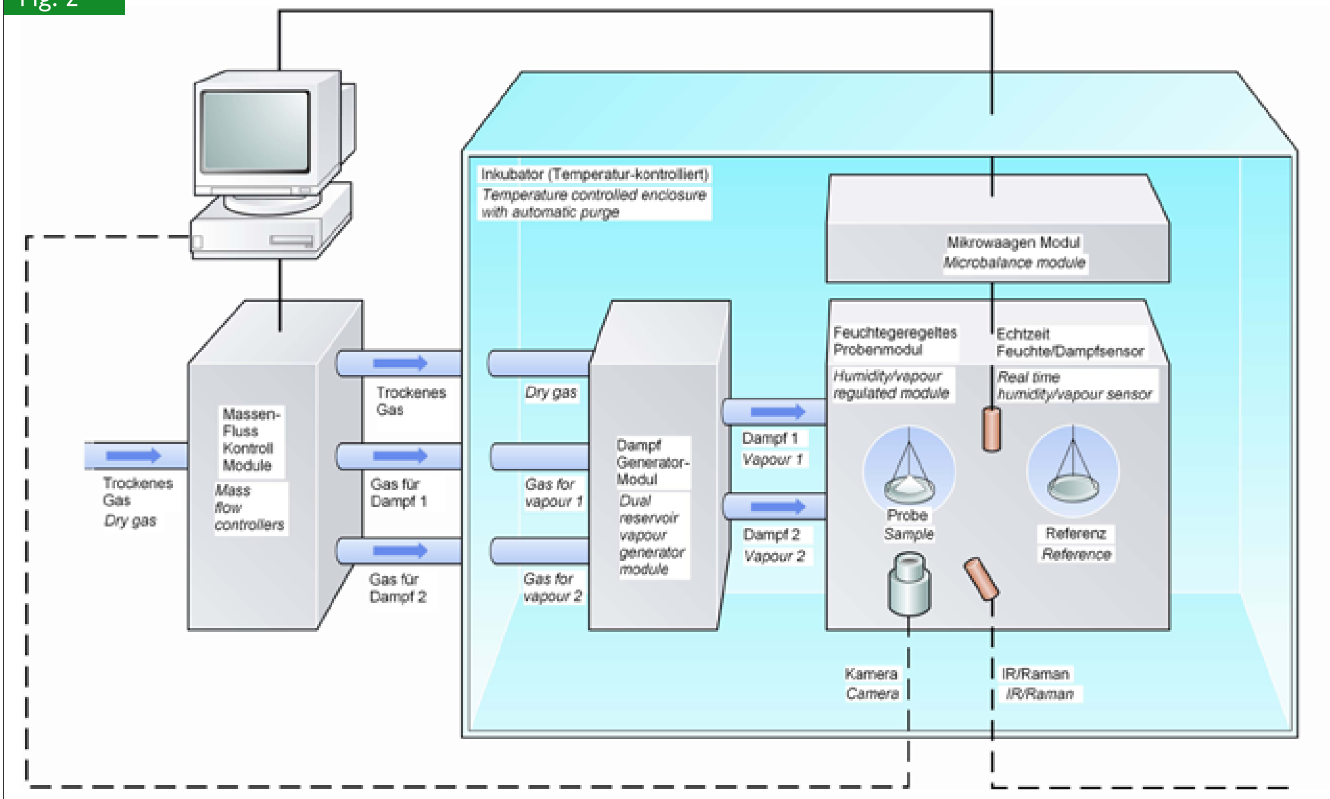

Fig. 2: Schematic diagram and principle of the dynamic water vapour sorption apparatus

Fig. 3

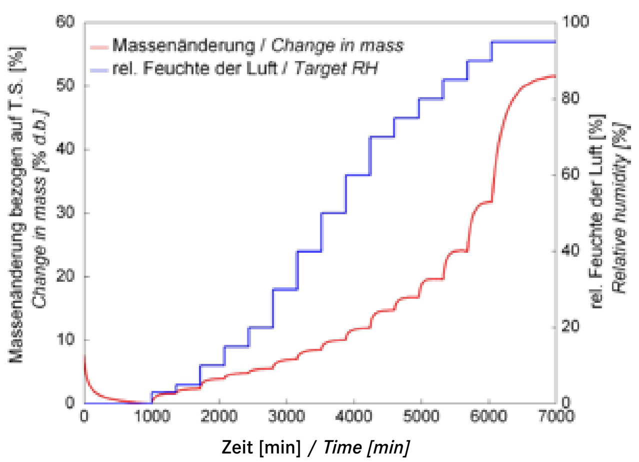

Fig. 3: Drying curve and equilibrium moisture adsorption profile of M. officinalis exposed to different values of relative humidity at 25 °C

Fig. 4

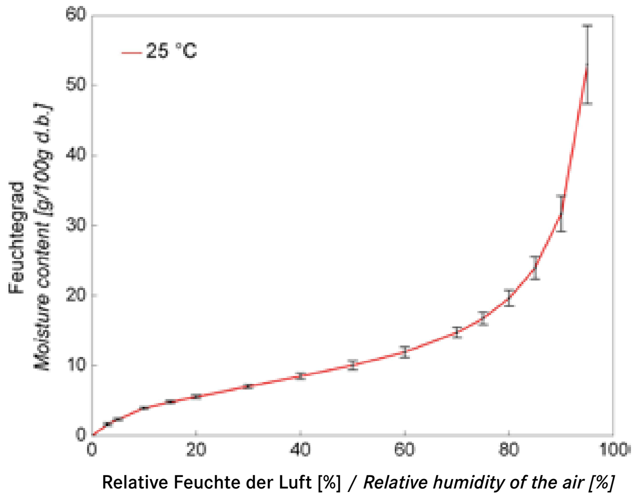

Fig. 4: Adsorption isotherm of M. officinalis at 25 °C

Fig. 5

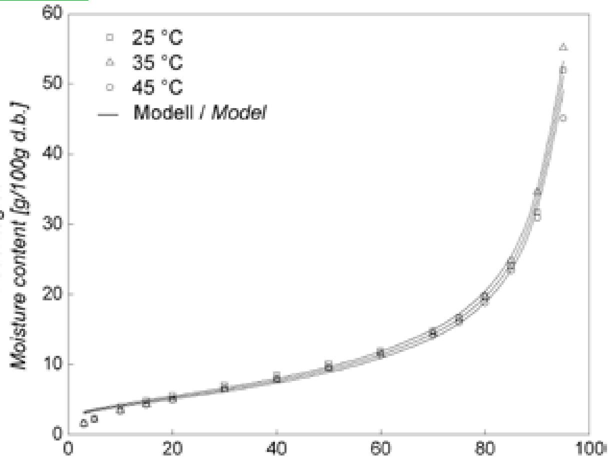

Fig. 5: Adsorption isotherms of M. officinalis fitted by Halsey-model

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Acknowledgment

The authors express their gratitude to the Bundesministerium für Ernährung, Landwirtschaft und Verbraucherschutz (BMELV) for the promotion of the project (support code: 22012509) by financial support through the Fachagentur Nachwachsende Rohstoffe e.V. (FNR) as project executing organisation of the BMELV for the promotional program renewable primary products.