

# SOME QUESTIONS OF PRINCIPLES IN USING THE SORPTION ISOTHERMS

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(Received July 5, 1963)

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## The sorption isotherm

Technological processes in connection with hygroscopic materials have frequently significant drying-technical relations. To solve the drying-technical problems successfully the knowledge of the drying characteristics of materials is inevitable. Occasional difficulties occurring in practice in connection with drying may usually be attributed to unexplained drying characteristics.

The most important drying characteristics of the hygroscopic materials are those which determine the moisture equilibrium. The moisture equilibrium of a hygroscopic material may be interpreted in reference to ambient space. In most cases the ambient space is filled with air having certain conditions. In this case moisture equilibrium may occur between the given material and the ambient air at determined state characteristics and the moisture equilibrium may be characterized by these state characteristics. The question is more complicated when other hygroscopic materials are also to be found in the ambient space. In this case the sorption equilibrium must be interpreted in reference to the whole system. But the moisture equilibrium of the system comes into being, also in this case, by determined state characteristics of the ambient air independent of the fact that other hygroscopic materials also take part in forming the state of equilibrium.

In most cases in drying-technical practice the moisturing agent is water, and thus problems are connected with the altering of the water content of the materials by the adsorption and desorption of water-vapour. Those drying-technical problems which tend to influence the adsorbed quantity of other liquids (for instance solutions) and gases also raise similar questions, but they fall beyond the frame of this work.

The state characteristics of air — at a given pressure — which surrounds the hygroscopic material and thus influences the moisture equilibrium, are temperature and relative humidity (or relative vapour pressure). Every hygroscopic material with a given moisture content ( $W$ ) and temperature ( $T$ ) can in a state of moisture equilibrium ( $W = W_e$ ) be only at a definite humidity of air ( $\Phi_e$ ), generally called “equilibrium relative humidity” ( $ERH$ ). At a given material  $ERH$  may be written with the following function:

$$\Phi_e = \Phi_e(W, T) \quad (1)$$

In most of the drying-technical problems occurring for various reasons in practice — for instance, technological — the temperature is a fixed or limited value; in case of certain material structures resp. moisture-binding forms the influence of the temperature may be neglected at a certain moisture-content interval. Thus, the drying-technical practice is often content — partly because of rationalization, partly for measuring technics — with the knowledge of the function:

$$\Phi_e = \Phi_e(W)_T \quad (2)$$

This function and its graphic performance represents the *sorption isotherm* (*SI*) of the material which describes the connection between the space-tension and the moisture-content of the material.

### The sorption hysteresis

The hygroscopic material may approach the  $W_e$  state by desorption and adsorption, i.e. from two sides. The results of the afore-going researches show, that in most of the hygroscopic materials the desorption isotherm (*DSI*) diverges from the adsorption isotherm (*ASI*), i.e. sorption hysteresis develops.

In case of various moisture bonds there are different theories to explain the sorption irreversibility, i.e. in case of capillary-pory materials [1, 2, 3], it is partly explained by the presence of air and partly by the delay of the capillary condensation. In case of colloids the question arises [1], whether hysteresis appears as an error by approaching  $W_e$  from two sides, because of the limitedness of the measuring period. The results of investigations with materials of high molecular weight [4] — also supported by the meat-drying researches of the author — show that the rate of hysteresis depends greatly, in the course of desorption, on the alterations occurring in the material structure, resp. in the moisture bond [1, 2]. Therefore, the pretreatment (sorption history) of the material is of very great importance. In this respect the temperature applied in the course of pretreatment might have the greatest significance.

In case of certain drying-technical problems — for instance, the common storing of variously rated wet materials or storing without weight loss — the sorption hysteresis is not to be neglected and according to the character of the problem both *DSI* and *ASI* must be known and adequately adopted. Unfortunately in practice this question is frequently disregarded, although its economical effect might be significant concerning certain materials — especially from the viewpoint of spoilage and degree of preservation. In many cases the research-workers, having only the adequate production technology in mind, are content to determine *DSI* only. Undoubtedly some difficulties are met with because of knowing only few determining methods of *ASI*.

### Material-structure relations concerning sorption isotherms

From the *DSI* conclusions may be drawn concerning the sorption characteristics of the materials, the material-structures and the moisture bonds [1, 2, 5, 6, 7, 8]. Similarly from *ASI* significant conclusions may be drawn referring to pretreatment (sorption history) of the materials. Thus, for instance, in case of potatoes MAKOWER and DEHORITY [9] found that depending on the method of desorption significant differences between the sorption characteristics might have occurred. Meats, dried over 80° C, become quite rigid, in all probability because of extra cross-linkages occurring in the protein-chains.

In other materials — for instance, in dried eggs — MAKOWER [10] found unchanged sorption characteristics independent of the drying method. This experience was also supported by the researches of GANE [11], although in the course of drying the denaturation of the protein undoubtedly occurred.

Unfortunately data concerning the pretreatment effect are still very incomplete. However it can be stated that pretreatment may significantly change the character of the basic material. These changes may be deduced by comparing *DSI* and *ASI* presuming that the sample used for the determination of *ASI* was the same as used in *DSI*. To determine absolute hysteresis and to complete examinations, it is advised after the determination of *ASI* to re-measure *DSI*, because in consequence of material-structure changes in the first desorption it may occur that the *DSI* on first determination is not reproducible, and thus may not be compared with the *ASI* measured later. Further it is advisable to produce *ASI* with samples pretreated at very low temperatures. Measurements of this character, although very complicated, may to a large extent clear the sorption phenomena of the materials.

### ERH values of mixtures and materials in casings

In industrial technologies there often occur such drying-technical problems, where the material is inhomogeneous, i.e. it is a mixture of different materials and eventually besides this has a casing, too. Such are the dehydrated precooked foods, meat-products filled in casing and materials with natural casing, such as fruits. Experiences concerning the *ERH* values of mixtures are not quite univocal and they seem to support the above-mentioned assumptions.

Practice in many cases showed that proceeding from the interaction of the components the weighed means of the *ERH* values, resulting from the isotherms of the components, give the *ERH* values of the multicomponent system [2]. For instance, GANE [11] also found that the sorption isotherm of the whole egg powder — in conformity with the afore-said — agrees well with the isotherms calculated from the separately measured isotherms of egg

white and yolk. Good conformity could also be proved by comparing the isotherm of dehydrated soup and the isotherm calculated from the isotherms of the components. In this case, however, no conformity was to be detected if the fresh ingredients were mixed before dehydrating [12.] From this circumstance the conclusion may be drawn that the components interact during desorption.

Examining the effect of the non-hygroscopic components in case of dried milk with various fat content it was found [13, 14] that its isotherms are in good accordance with that calculated on basis of fat free material. Thus, the fat — as a component — does not count. Author experimented with meat-pastes of various bacon-content and found that the effect of bacon was unnoticeable. Spices however, and above all salt, admixed to the meat-pastes, make it impossible to calculate from the components [21].

Thus, on examining the *ERH*, resp. *SI* values of inhomogeneous materials it was essential to examine the pure components, but as the interaction of the components does not often give reproducible results, it also became important to measure the isotherm of the mixture. On determining the *ERH* values of the mixtures, special care must be taken to examine the sample-composition, for determining the possible minimum sample-quantity and for the procedure of sampling.

A further essential viewpoint is that no conclusions can be drawn from the sorption isotherm in respect to time during which — in a certain environment — the equilibrium came into being, for under the same conditions — also depending on other drying characteristics of the components, and so the results of measurements which do not regard carefully the time-factor may also differ from the calculated ones. As the examinations of author showed [15, 16, 17], the *SI* of the materials in a hygroscopic casing is first of all determined by the *SI* of the casing. Thus, the filled materials cannot theoretically be looked upon as a multicomponent system, although their internal substance is usually inhomogeneous (for instance salami, sausage, etc.). In course of drying of materials in casing the outwards tending moisture-flow comes into being because of the moisture gradient in the internal substance. Thus, the surface moisture (moisture content of the casing) may change, not only under the influence of environmental-state-characteristics, but also under the influence of the internal — compensating — moisture-flow. Thus, for materials in casing we can at best speak of momentary *ERH* value which belongs to the momentary moisture-distribution inside the materials. So the determination of the *ERH* values — the determination of the sorption isotherms — in measuring-technics is problematical. The separate examination of the casing and the inner substance makes the determination of the momentary *ERH* in case of whatever moisture distribution theoretically possible, but often there is no time for it. For instance, when producing salami, the pre-

scription of the drying parameters of the *ERH* value is immediately needed, therefore, such a method must be adopted which makes possible the neglect of the compensating effect of moisture-flow and produces a result in a short time, and so makes the examination also an entity taken from the technological process possible [18].

On treating materials in casings, one must also consider the changes occurring in the casing during drying. Such a change might be caused by salt which enters the casing by diffusion or moisture-flow. The examination of author was able to show with salami-casings [17] that the influence of this effect must be taken into consideration during the whole drying process because, compared with the pure casing isotherm, it may cause a not negligible difference depending on the concentration of the salt solution and the structure of the casing.

#### Nominal *ERH* values of accumulated goods

In case of big drying rooms, where a great quantity of piece of goods are dried, the moisture-content differences — occurring in consequence of the measure-differences of the single pieces, and other causes (for instance the unequal distribution of the drying air) may have an essential influence. As a consequence the *ERH* values of the single pieces differ from each other. Therefore, the drying must so be directed that in spite of the measure differences a constant and adequate grade compensating effect should be achieved which can be realized by considering the average or nominal momentary *ERH* values [18]. The nominal *ERH* values might be determined by knowing the measure dispersions and by calculating the weighed mean values, satisfying the requirements of practice, with sufficient accuracy.

#### Common storing of various materials

If different sorts of materials are to be stored in a common air-space suitably and without loss one can ascertain this only on the basis of sorption isotherms. By knowing the *SI*-s and using them as characteristic curves it is possible to determine the changes occurring as a consequence of the interactions. Similar problems arise in the common-packing of food-stuffs [19.] As was already mentioned on examining the isotherms already at the beginning, it is advisable to choose which — *DSI* or *ASI* — must be taken into consideration for the different materials.

In the course of storing the various hygroscopic materials in a common air-space moisture exchange occurs and air is the transferring medium. The

grade of moisture-exchange depends on the water content, the quantity, the *SI* and other drying characteristics of the materials.

The grade of the possible moisture-exchange must be known in advance so as to be able to avoid the inadmissible grade of changes in the moisture-content of the single materials.

By moisture-exchange the multicomponent system tends to reach a state of equilibrium. Equilibrium comes into being at such  $W$  values of the single materials, to which in every component the same *ERH* value belongs (spontaneous equilibrium).

When storing in ventilated, conditioned store-rooms the applied optimum air-conditioning parameters could be determined on basis of the *SI*-s. These might be the values which answer to the spontaneous state of equilibrium of the system. In this case ventilating and air-conditioning decreases when approaching the state of equilibrium and eliminates meteorological effects. But certain cases might occur, when the forming of spontaneous equilibrium-state must be prevented and another, more convenient, controlled state of equilibrium must be created.

### Spontaneous equilibrium-state of multicomponent systems

For the sake of simplicity we shall take a two-component system and from the sorption isotherms conclude the grade of moisture-exchange. The *SI* of material *A* and *B* are to be seen, where the initial water contents are denoted in Fig. 1 by 1 indices. From the initial conditions it might immediately be verified on basis of  $\Phi_{B1} < \Phi_e < \Phi_{A1}$ , that the spontaneous equilibrium-state comes into being by the drying of material *A* and moisture-adsorption of material *B*, thus, we must use *DSI* in material *A* and *ASI* in material *B*. Assuming a closed system, where the absolute moisture-content-change of the air is negligible, we may state that the  $\Delta V$  changes, occurring in the water content of the materials when forming the spontaneous equilibrium-state ( $\Phi_e$ ), are of the same grade:

$$\Delta V_A = \Delta V_B \quad (3)$$

$$\text{i. e. } G_{SA}(W_{A1} - W_{A2}) = G_{SB}(W_{B2} - W_{B1}) \quad (4)$$

where  $G_S$  — is the dry weight of the materials, and

$W$  — the water content referring to the dry basis.

From equation (4) it is to be seen that the moisture change  $\Delta W = W_1 - W_2$  — and thus also  $\Phi_e$  — is dependent (as on initial conditions) also on the weight of the stored materials. Considering that in single materials — from viewpoint of the spoilage-grade and quality-changes — the permissible maxi-

imum  $\Delta W$  value is known, the storable quantities in regard of both materials may be determined, i.e. the value of  $\Phi_e$  may be directed by influencing the  $G_S$  values. (For the example in Fig. 1  $G_{SA} = 0.5 G_{SB}$ , i.e.  $\Phi_e$  was established at  $\Delta W_A = 2 \Delta W_B$ .)

**Controlled equilibrium of multicomponent systems**

In the case, when the quantity of materials to be stored in a common space is not to be influenced systematically, nevertheless we wish to ensure the adequate conditions for spontaneous equilibrium, then we must discontinue the

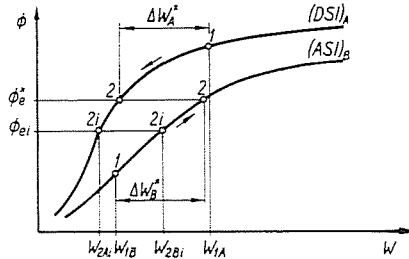
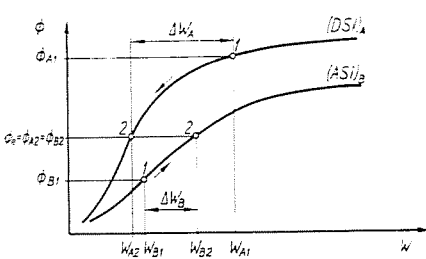


Fig. 1. Spontaneous equilibrium of a two-component system ( $G_{SA} = 0.5 G_{SB}$ )

Fig. 2. Controlled equilibrium of a two-component system ( $G_{SA} = G_{SB}$ )

closedness of the system and the “superfluous” or “required” moisture must be taken out or brought in into the system by conditioned air.

Starting out from the example in Fig. 1 let us assume that equal quantities of materials *A* and *B* ( $G_{SA} = G_{SB} = G_S$ ) are stored and the initial water content conditions are the same  $W_{A1} = W_{B1}$ , then from equation (4) we obtain  $\Phi_e^x$  according to conditions  $\Delta W_A^x = \Delta W_B^x$ . As can be seen from Fig. 2  $\Delta W_B^x$  and thus  $\Phi_e^x$  is much greater than in Fig. 1 so this is inadmissible.

Using conditioned air we may control the establishing of equilibrium by blowing air, with the advantageous condition  $\Phi_{ei}$ , in. Of course, in this case the system is not closed and the conditioned air takes

$$\Delta V = G_S[(W_{1A} + W_{1B}) - (W_{2A} + W_{2B})] \tag{5}$$

water out of the storing room.

The period, needed to establish the controlled equilibrium depends on the quantity, the drying characteristics of the materials and the applied quantity of air.

### The possibility of intermediate conditions

The conclusions concerning the equilibrium end-state of the multicomponent system are independent of time, thus, to approach this end-state, the intermediate conditions depend on the sorption time-constants of the single materials.

The value of the sorption time-constant (for which at given initial conditions the drying characteristics of the material are decisive) may be significantly different for the single materials. Consequently in case of the respective components the achievement of the given end-state  $\Phi_e$ , adsorption or desorp-

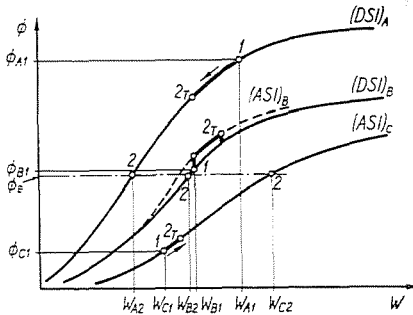


Fig. 3. Forming of intermediate states

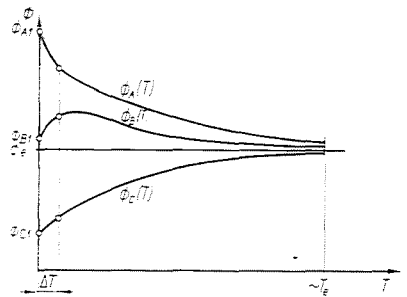


Fig. 4. Forming of spontaneous equilibrium of a three-component closed system

tion, starts with various rates: therefore, it may occur that at a certain intermediate state the desorbed and adsorbed water quantities do not correspond and the difference is accumulated in or extrahated from the air of the store-room until they are conform.

Considering that the air — as a transferring medium which is also hygroscopic — adsorbes moisture from the material with the greatest  $\Phi_1$  till it reaches the *ERH* value, independently of the rate of adsorption of other materials. Thus, it may be stated that in the intermediate conditions practically  $\Phi_e$  can never be looked at as the mean value. Therefore it may occur that the respective kinds of materials get temporarily into the inadmissible moisture condition.

Let us take a three-component system (*A*, *B*, *C*), where in the initial state the stored dry weights are conform ( $G_{SA} = G_{SB} = G_{SC}$ ). From the drying characteristics of the components it is known that the ratio of the sorption time-constants is approximately  $T_A : T_B : T_C = 1 : 2 : 3$ . The  $\Delta T$  time after starting the storing the water content changes proportionally (see Fig. 3, spot  $2_T$ ), thus for the further period of storing new initial conditions are given. In our example — in consequence of the small adsorption-activity of material *C* — material *B* adsorbed temporarily a greater quantity of water



than its end-value belonging to  $\Phi_c$ . Thus, material *B* temporarily adsorbed water (Fig. 3 *ASI* phase) and arrives to its end-state by desorption (Fig. 3 *ASI* phase). Under disadvantageous conditions one of the components — here *B* — may take up moisture to a not admissible measure (Fig. 4), and this circumstance cannot be reached on the basis of the isotherms only.

The above-mentioned facts draw attention to the importance of other drying characteristics of the materials.

### The role of the controlled distribution of the conditioned air

In some kinds of materials it may occur that the admissible maximum  $\Delta W$  values cannot be co-ordinated. Let us assume that by the materials of Fig. 1  $\Delta W_A > \Delta W_{A \text{ admis.}}$  and  $\Delta W_B > \Delta W_{B \text{ admis.}}$ . In this case even by adequately choosing the quantity of the stored materials it is impossible to obtain satisfactory results.

However, a solution may be found if the store room is conditioned, namely, the conditioned air goes in or out of the store-room-space with the help of a regulable, branched distribution air-pipe network and the various kinds of goods are stored in predetermined places. Thus, each of the goods receives air periodically by alternately operating the pipe-branches. The time-periods adopted to the respective materials and the adequate choosing of air-conditions makes it possible to store the materials in a definite moisture-zone.

Let us assume in case of the goods introduced in Fig. 3 and 4 that the values  $\Delta W_{\text{admis.}} = W_1 - W_2$  (and from the isotherms  $\Phi_{\text{admis.}} = \Phi_1 - \Phi_2$ ) are known and we also know that the conditions  $W_A \leq W_{A1}$ ,  $W_C \geq W_{C1}$  and  $W_B \leq W_{B1}$  must be fulfilled. In the first period of the process (for  $T_1$  time) material *A* gets air nearly in the state  $\Phi = \Phi_{A1}$ . During time  $T_1$  — as perfect separation is not possible —  $\Phi_{B1}$  increases to  $\Phi_{B2}^*$  and  $\Phi_{C1}$  to  $\Phi_{C2}^*$  (Fig. 5 *ASI-s*) (1, 2). At the end of  $T_1$  period we switch in the pipe-system of material *B* and during time  $T_2$  (second period)  $\Phi < \Phi_{B2}$  air state is applied. During time  $T_2$   $\Phi_A$  decreases to  $\Phi_{A2}$  (Fig. 5 [*DSI*]<sub>A</sub>),  $\Phi_{B2}$  tends to  $\Phi_{B1}$  (*DSI*)<sub>B</sub> and  $\Phi_C$  increases to  $\Phi_{C3}^*$  (*ASI*)<sub>C</sub>. At the end of period  $T_2$  the pipe-system of material *C* opens and remains opened during  $T_3$  time (third period) using  $\Phi < \Phi_{C1}$  air state. In the third period  $\Phi_A$  further decreases to  $\Phi_{A3}$  (*DSI*)<sub>A</sub>,  $\Phi_B$  decreases to  $\Phi_{B3}$  (*DSI*)<sub>B</sub> and  $\Phi_C$  approximately to  $\Phi_{C1}$  (*DSI*)<sub>C</sub>. After this only for the material *A* is  $\Phi \geq \Phi_{A1}$  air used, that it should approximately return to the initial state (*ASI-s*).

The problem might thus be solved with one and the same air-conditioning equipment. After the co-ordinating of the air-condition characteristics and period times, to the drying characteristics of the materials and controlled experimentally, automatic program control may be applied. A program might be worked out for various storing weight relations.

In so far as there are not very strict limitations concerning the storing temperature-region, then it is possible to produce a basic-air appropriate to dew-point of the minimum  $\Phi$  value which might be adjusted to the optimum  $\Phi$  values of the respective materials with the aid of afterheaters built into the fore-parts of the respective pipe-branch. In this case the periodical operation is not necessary and automatization may be solved more simply. The possibility of the simplification can be decided by knowing the  $SI$ -s, the  $\Delta W$  values and the admissible temperatures.

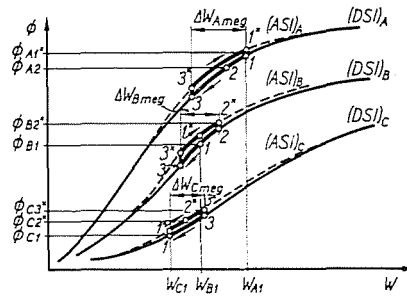


Fig. 5. Assuring limited moisture-content-change when storing by conditioning and directed air-distribution

### The resultant isotherm and its application

In multicomponent systems — as was shown above — it is essential to determine the  $\Phi_e$  value indicating the spontaneous sorption equilibrium of the system.  $\Phi_e$  may be determined from the  $SI$ , the  $G_S$  weight and the initial water content of the components.

The problem may also be formulated in such a way that the utmost water content of the components must be searched for ( $W_{A2}$ ,  $W_{B2}$ , etc.) besides which the  $\Phi_{A2} = \Phi_{B2} = \dots = \Phi_e$  condition is fulfilled.

SALVIN and SLAVSON [19] further TÖRÖK and SZALAY [20] report an approaching method for solving this problem. In case of a closed system, where the water content of the airspace may be neglected and assuming that the isotherms in the phase between the initial- and end-state are represented by a straight line, their slope is:

$$m = \frac{\Phi_e - \Phi_1}{W_1 - W_2} \tag{6}$$

Thus using equation (4) we may write for the two-component system:

$$\frac{1}{m_A} G_{SA}(\Phi_e - \Phi_{1A}) = \frac{1}{m_B} G_{SB}(\Phi_e - \Phi_{1B}) \tag{7}$$



Applying the calculating method after drawing tangents in the initial point  $\frac{1}{m_{A1}} = A$ ;  $\frac{1}{m_{B1}} = 2.5$  in the first approximation from equation (7)  $\Phi_{e1}^x = 84.7\%$  is derived. In the second approximation on basis of values  $W_2^x$  the corrected values are:

$$\frac{1}{m_{A2}} = \frac{W_{A1} - W_{A2}^x}{\Phi_{e1}^x - \Phi_{A1}} = 1.075$$

and

$$\frac{1}{m_{B2}} = \frac{W_{B1} - W_{B2}^x}{\Phi_{e1}^x - \Phi_{B1}} = 1.182.$$

From these based on equation (7) the result is  $\Phi_e = 78.8\%$  which corresponds very well with that obtained by the determination on basis of the resultant isotherm.

In case of more than two components the resultant isotherm may be deduced from the partial resultants. Experiences show that this method is quick and accurate.

### Conclusions

It is essential to know the *SI*-s to solve the drying-technical problems successfully.

In case of certain materials the grade of the sorption hysteresis is not to be neglected, therefore, the use of both *DSI*-s and *ASI*-s may be necessary. The comparing of *DSI* and *ASI* may concern the structure of the material and it may render possible to draw conclusions in connection with the sorption history of the material.

The *ERH* values of mixtures — multicomponent systems — cannot always be determined from the *ERH* values of the pure components, therefore it is indispensable to determine the isotherms of the mixture, too. The *SI*-s of the materials with casing, is determined first of all by the isotherm of the casing.

In case of common-storing of various materials the spontaneous equilibrium may be determined from the *SI*-s, as characteristic curves. The air-content of the space is the moisture transferring medium. Controlled equilibrium may be produced by air-conditioning.

It is impossible to draw conclusions in reference to time as to how the phenomena proceed but from the sorption isotherms, and it is essential to know the other drying characteristics of the materials, too. With the knowledge of the drying characteristics the intermediate states must also be examined.

In applying conditioning it is possible to supply periodically with air the various kinds of materials with an adequate air-canal system. With certain storing temperature differences for the various materials air with changing relative vapour-content from basic air may be produced.

### Summary

The significant drying characteristics of the hygroscopic materials are the desorption and adsorption isotherms which determine the sorption equilibrium. In case of materials with casing the sorption equilibrium is first of all determined by the casing. On evaluating the phenomena occurring during the course of common-storing of the various materials, the knowledge of the *SI*-s is essential. The conditions of store-house-climatization may also be determined on basis of the *SI*-s. The spontaneous equilibrium characteristics of a multicomponent system may be determined on basis of the resultant isotherm method.

To determine the state characteristics of the spontaneous equilibrium the method of the resultant isotherm is correctly applicable.

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