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APPLICATIONS OF CI MICROBALANCES

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Abstract

Electronic microbalances, manufactured by CI Electronics Ltd., are used by numerous researchers to study processes and phenomena which can be followed by changes in the sample masses. Less sensitive electronic millibalances are incorporated into integrated systems for the quality control of small and valuable items such as pharmaceutical products and diamonds. A further application of microbalances is the automatic water sorption analysis of samples by the gravimetric method with the CISorp. This integrated system records kinetic and isotherm data of the changes in the relative humidity, sample masses, and temperature and allows the study of physical and chemical properties of sample surfaces.

Keywords: checkweigher, electronic microbalance, isotherm, sorter, water sorption analyser

General microbalance uses

Some physical and chemical phenomena and processes can be studied by the accompanying changes in the sample masses. The changes are usually very small, and the ability to weigh to microgram accuracy is therefore a requirement.

For such studies, microbalances and their controllers are usually incorporated into rigs which are purpose-built by academic and industrial researchers. Phenomena studied include the thermal decomposition of samples, high temperature oxidation, hydrogen absorption by metals, surface and interfacial tension, thin film deposition, magnetic susceptibility and vapour sorption.

The microbalances have a capacity of approximately 5 g and a weighing range of ±500 mg.

Accurate weighing to milligram accuracy is important for the sorting and checkweighing of small and valuable loads such as pharmaceutical products and diamonds. This has led to the development of integrated equipment for the purpose of sorting and checkweighing of pharmaceuticals or diamonds.

The automatic water sorption analyser

Electronic microbalances have also been incorporated into the CISorp. This is a fully integrated system for the automatic study of water sorption phenomena. The equip-

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ment houses two microbalances which are exposed to the same conditions of humidity and temperature. It can therefore be used for the simultaneous study of two samples or for their direct comparison. The relative humidity can be varied between near 0% and 95%. The experimental temperature used ranged from 10 to 50°C.

To run an experiment, individual steps in terms of the relative humidity, temperature and equilibrium conditions are pre-programmed by the user. The experiment then runs automatically without any supervision. Kinetic and isotherm data are recorded automatically. These allow the graphical and mathematical evaluation of the results.

Water sorption tests

The CISorp has been used to perform a number of water sorption tests which demonstrate the range of its applications. Thus the difference has been shown in the water sorption behaviour between hydrophilic and hydrophobic samples, i.e. those with high and low affinity for water. In everyday life such a difference can be seen in the comparison of the wettability of paper with that of glass.

In water sorption tests, such a difference can be deduced most easily from the shape of the isotherms, as previously reported [1, 2]. Then it was shown that an isotherm with large water uptake at low humidity is indicative of a polar surface, and vice versa.

The presence and absence of surface pores, as well as their size distribution, can also be determined from the isotherms. This information is contained either in the shape of the isotherm or in the detailed structure of the hysteresis. Hence a Type I isotherm indicates a microporous sample, and hysteresis in a Type IV or V iotherm points to a mesoporous sample [3]. This has also been reported in an earlier publication [2].

Other information that can be gained from water sorption profiles is the effect of compressing a powder to a tablet. This is seen most clearly in the comparative kinetic profiles of such samples, as shown in Fig. 1. The results confirm that compression does not affect the microscopic surface area of the particles which make up the tablet. It is in-

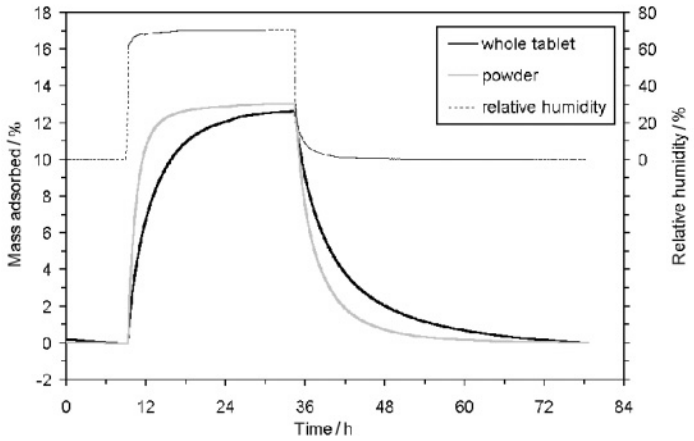

Fig. 1 Comparative water sorption kinetic profile of powder and tablet

dicated by the fact that the amount of water taken up at equilibrium is almost indistinguishable for the two forms of the substance. The rate of water sorption, by contrast, is greatly reduced after compression, indicating that diffusion of the water molecules across the surface is inhibited.

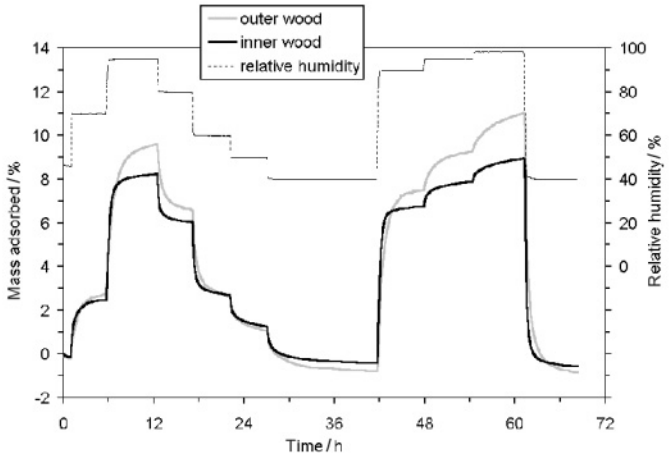

Fig. 2 Comparative water sorption kinetic profile of the two types of wood from a pinecone

These findings are in contrast to the water sorption of the two types of wood inside a pinecone. Here the amounts of water taken up by the two samples differ, while the rates of water sorption are similar. However, the rates of water sorption are similar (Fig. 2). The interpretation is that the two types of wood are different in their physical surface properties, either in terms of their absolute surface area or the specific surface area with an affinity for water. These two tests demonstrate well the advantage of being able to investigate samples simultaneously under the same conditions.

Some unusual water sorption behaviour of samples such as that of freeze-dried samples or crystalline compounds has already been reported. There rigorously dried samples were found to reabsorb water irreversibly, and crystal structures were created or interconverted as a result of water sorption [1, 2].

Unusual results are also obtained in the study of an unknown crystalline compound at different experimental temperatures (Fig. 3). In line with many samples, this shows reduced water adsorption at increased temperature. Normally such behaviour is a sign of purely physical adsorption whereby more energetic (‘warmer’) water molecules are less likely to adhere to the surfaces. In such cases, the general shape of the isotherm remains unchanged with temperature.

In the present sample, water adsorption is, indeed, reduced at raised temperature. However, the shape of the isotherms and that of the hysteresis are also changed. From the corresponding kinetic profiles (Fig. 4) it is clear that equilibration was far from complete, and plotting the isotherms was not strictly valid. A possible interpretation of the available data is that the process of water sorption of this sample causes temperature-dependent crystal transformations.

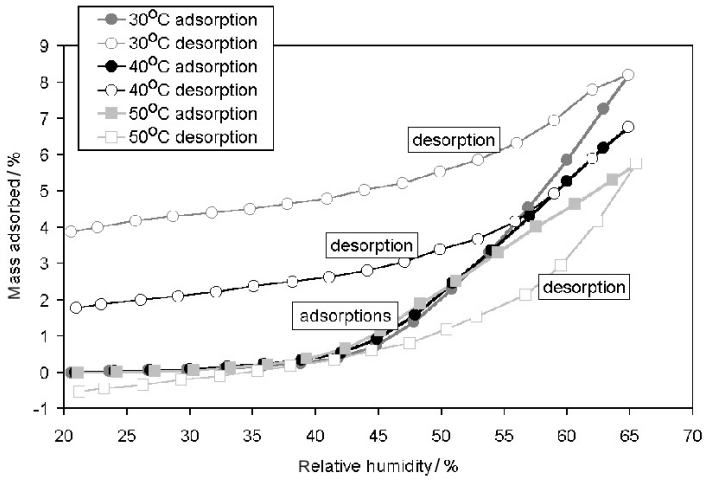

Fig. 3 Water sorption isotherms of an unknown crystalline compound at different temperatures

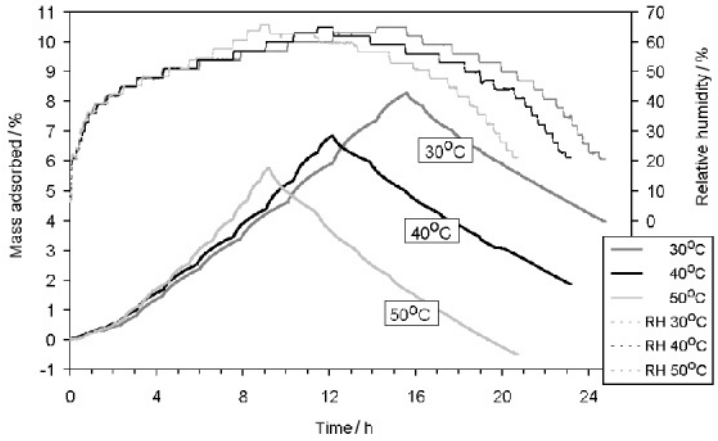

Fig. 4 Water sorption kinetics of an unknown crystalline compound at different temperatures

Similarly complex behaviour was already observed in commercial milk powder where there was an interaction between the different components during changes in humidity [2].

Conclusions

The monitoring of mass changes of compounds is useful as a technique for the investigation of a number of processes and phenomena. Such techniques are used by numerous researchers with their own purpose-built equipment, or with dedicated equipment for the study of water sorption.

The monitoring of absolute masses for the sorting and checkweighing of compounds is a further widespread use of gravimetric equipment.

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