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A NOVEL METHOD FOR WETTING AND DRYING ISOTHERMS OF MATERIALS BY THERMOGRAVIMETRY

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ABSTRACT

Moisture adsorption/desorption isotherms were constructed using a thermogravimetry-based technique. Isotherms were obtained by equilibrating samples at known relative humidities in salt chamber humidistats, and then measuring the moisture content using thermogravimetry. By pre-drying and pre-wetting the samples, both wetting and drying isotherms can be obtained.

INTRODUCTION

Recently, there has been great interest in adsorption/desorption phenomena and their quantitative measurement by thermal analysis [1–3]. In this study, a thermogravimetric (TG) method has been developed for the determination of the wetting and drying isotherms of various materials. The isotherms are essential for applications in moisture adsorption/desorption studies and passive and off-load cooling studies in energy research [4,5]. These isotherms allow the prediction of the properties of desiccants, construction materials and air-conditioning equipment under varying humidity conditions. TG provides a useful and reproducible way of constructing these curves, and of measuring approaches to saturation equilibria. Silica gel was chosen for the initial studies because its properties are well understood.

EXPERIMENTAL

Thermogravimetry

The TG tracings were obtained using a CAHN System 113 thermogravimetric analysis apparatus modified to allow the output data to be input directly into an IBM personal computer. Temperature and mass signals were

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TABLE 1

Composition of salt chamber humidistats

Relative humidity at 30.0 °C (%)	Salt
23	$KC_2H_3O_2 \cdot 1.5H_2O$
33	$MgCl_2 \cdot 6H_2O$
47	KSCN
64	$CoCl_2 \cdot 6H_2O$
79	$(NH_4)_2SO_4$

amplified by a pair of instrumentation amplifiers (AD624) to a full-scale range of 0–5 V DC. The amplified signals were digitized using a MetraByte DAS-08 interface card (MetraByte Corp., 440 Myles Standish Blvd, Taunton, MA 02780) mounted in an IBM-XT computer. The data was collected using LABTECH ACQUIRE (Laboratory Technologies Corp., 255 Ballardvale St, Wilmington, MA 01887), and plotted using GRAPHER (Golden Software Inc., 807 14th St., Golden, CO 80402). All TG runs were done at a heating rate of 5 °C min^{-1} from ambient temperature to 250 °C.

Sample drying, temperature and humidity control

Pre-drying of samples was carried out in an Equatherm Vacuum Oven Model 273-781 by heating for 3 h under vacuum at 200 °C. Pre-dried samples were immediately transferred to humidistats and allowed to equilibrate for 30 days. The humidistats were maintained in a Gallenkamp Plus Incubator using forced air circulation at 30.0 ± 0.2 °C. A set of five salt-chamber humidistats was used. The humidistats contain a 1 : 1 mixture of saturated salt solutions plus excess salt in sealed 1-l glass containers.

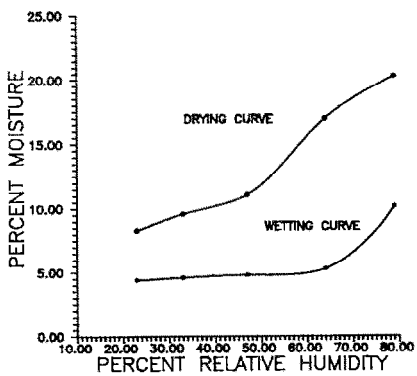

Fig. 1. Moisture isotherms for silica gel, type 59.

TABLE 2

Data for Fig. 1

Drying curve		Wetting curve	
RH [a] (%)	Moisture (%)	RH [a] (%)	Moisture (%)
23	8.3	23	4.4
33	9.6	33	4.6
47	11.1	47	4.9
64	17.0	64	6.3
79	20.3	79	10.2

[a] RH, relative humidity.

Samples for equilibration were held in position directly over the salt mixture. The composition of the solutions and the relative humidity maintained at 30.0 ° C are shown in Table 1. The silica gel used was type 59 (W.R. Grace & Co., Davison Chemical Division).

RESULTS AND DISCUSSION

The wetting and drying curves obtained for silica gel 59 are shown in Fig. 1 and the corresponding data is given in Table 2. The pre-dried and pre-wetted samples were prepared as described above. Points are shown for the normal range of humidities of interest in industrial and residential applications. The pre-dried samples were subdivided and allowed to equilibrate at various humidities in the incubator. After TG, each sample at each humidity produced one point on the wetting curve. The drying curve was obtained in the same manner, except that pre-wetted samples were allowed to equilibrate in the humidistats.

The sample exhibits the low percentage moisture characteristic of an intermediate-density silica gel in the relative humidity range studied [6]. Figure 1 shows data for which the wetting and drying curves are still well separated. With longer equilibration times the two curves gradually approach each other.

This technique for obtaining the wetting and drying curves is very well suited to investigations involving a variety of dissimilar materials. The equilibration process is readily tailored to individual applications. It is carried out under precise temperature control, and materials of various physical forms such as powders, granular mixes or composites, can be used. The equilibration time is easily adjusted and may be very long if desired. Because the moisture content is measured separately by TG, low-moisture materials requiring long equilibration times present no problems. Studies of materials for application in passive cooling systems are currently under way.

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