

New sorption and solvation measuring methods: Forced flow through liquids and solid state fluidised bed sorbents in high pressure gravimetry

Tobias Fieback^{a,*}, Frieder Dreisbach^b, Marcus Petermann^a, Roland Span^a, Eckard Weidner^a

^a Ruhr-Universität Bochum, 44801 Bochum, Germany

^b Rubotherm GmbH, Universitätsstraße 142, 44799 Bochum, Germany

ARTICLE INFO

Article history:

Received 29 June 2010

Received in revised form

27 November 2010

Accepted 30 November 2010

Available online 15 December 2010

Keywords:

Absorption

Carbon dioxide

Fluidised bed

Forced flow through

Gravimetry

Magnetic suspension balance

Solvation

Sorption measuring

ABSTRACT

A novel measuring method is presented here that allows sorption and solvation measurements near to different process conditions. Especially for the sorption kinetics it is important that the measuring conditions are similar to later applications. Particularly for high pressure sorption measurements this claim could not be fulfilled for most technical applications.

The advantage of the measuring methods presented here is that solid bed samples in high pressure gravimetry can be measured under flow-through conditions in both directions and, for the first time in high pressure gravimetry, liquid samples can be measured under flow-through conditions. Especially for ionic liquids, of which a large variety of new substances with unknown sorption potentials have been synthesised in the last few years, this results in much shorter measuring times.

The first measurements presented here show that the new system allows for measuring times around 26 times shorter for an ionic liquid.

© 2010 Elsevier B.V. All rights reserved.

1. Introduction

Since decades sorption processes have been used for cleaning or purification of gases. Lately, with rising interest in renewable energy supplies, a large amount of new gas cleaning and storage processes applying ionic liquids as solvent have been developed, particularly for CO₂ – and hydrogen containing gases [1–10]. Design and optimisation of these processes require precise instruments for sorption measurements, because the accurate knowledge of sorption isotherms is essential for any process layout. Moreover the sorption kinetics must be taken into account to develop commercially successful processes; therefore the measuring conditions should be as similar as possible to the real process conditions. Additional requirements for future measuring instruments evolve from the application of ionic liquids as alternative solvents [11,12]. The large number of synthesised substances in this class, most with unknown sorption potential, raises a demand for faster sorption measuring methods usable for liquid samples [13].

Most sorption isotherms are measured with a volumetric, a gravimetric or a combined volumetric/gravimetric instrument. In

only few cases are other methods used [14]. For measurements at conditions similar to process conditions the volumetric method has some limitations, because only static atmospheres can be measured and the accuracy of the obtained data declines with rising pressure. Here the gravimetric measurement, in particular using a magnetic suspension balance, has inherent advantages. Sorption measurements can be performed in static and flowing atmospheres at pressures up to 200 MPa with nearly constant measuring accuracy [15].

2. Conventional gravimetric measurements

The main difficulty when using conventional gravimetric instruments is the direct contact between the measuring cell (the sample atmosphere) and the weighing instrument [16,17]. The balance can be damaged or disturbed by the sample gas and the measuring atmosphere can be adversely affected by flushing gases needed as purge for the balance. To avoid these effects, magnetic suspension balances are used. With such a device it is possible to weigh samples contactlessly in nearly all environments. The investigated sample is not attached directly to the balance but is linked to a so-called suspension magnet which consists of a permanent magnet, a sensor core and a device for decoupling the sample (Fig. 1). An electromagnet, which is attached to the underfloor weighing hook of a

* Corresponding author. Tel.: +49 234 3226392; fax: +49 234 3214119.

E-mail addresses: t.fieback@thermo.rub.de, tobias@fieback.com (T. Fieback).

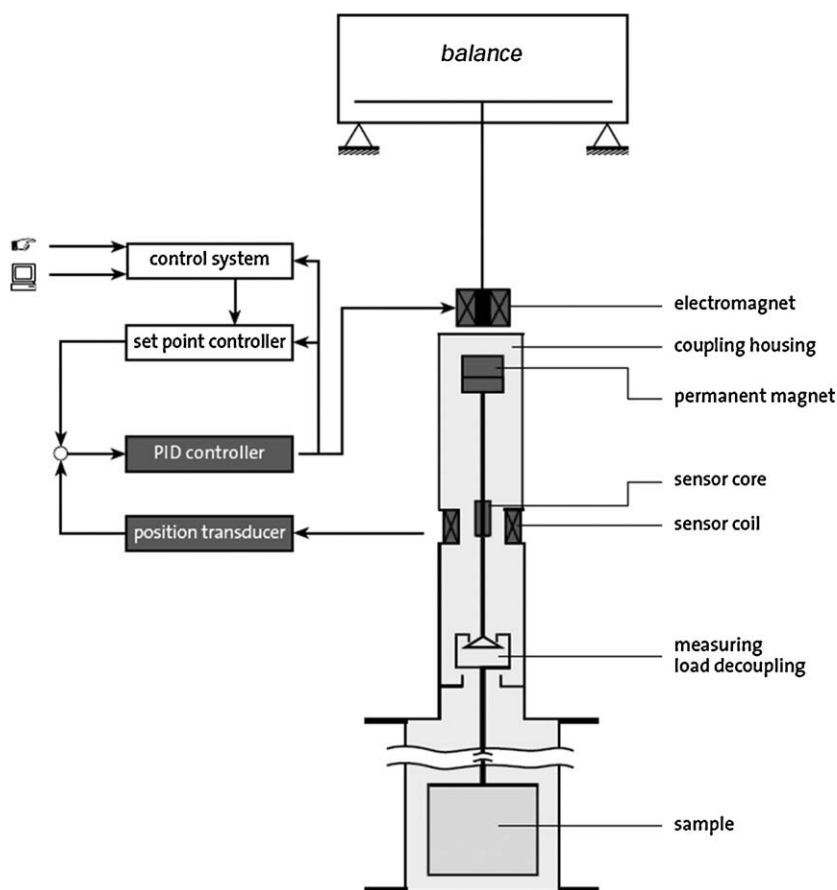


Fig. 1. Principle of the magnetic suspension balance [18].

balance, maintains a freely suspended state of the suspension magnet via an electronic control unit. Using this magnetic suspension coupling, the measuring force is transmitted contactlessly from the measuring chamber to the microbalance, which is located outside the chamber under ambient atmospheric conditions [15].

The magnetic suspension balance (MSB) offers the possibility of lowering the suspension magnet in a controlled way to a second stationary position a few millimetres below the measuring position. Then, a small carrier to which the sample is connected is set down on a support. Now the sample is decoupled from the balance. The suspension magnet alone is in a freely suspended state and only its weight is transmitted to the balance. This so-called zero point position, which corresponds to an empty balance pan in a normal weighing procedure, allows a taring and calibration of the balance at all times.

A further important development is related to the ability to decouple the measuring load in a way that allows measuring the mass change of two samples with only one magnetic suspension balance [19]. In addition to the first measuring load decoupling, a second one is arranged in the magnetic suspension coupling. Three different vertical positions of the suspension magnet, which can be realized in a controlled way, correspond to three different measuring positions (Fig. 2):

1. Zero point: The permanent magnet alone is in a freely suspended state, allowing the balance to be tared and calibrated.
2. Measuring point 1: The first sample is lifted up and its mass is weighed.
3. Measuring point 2: The second sample is raised together with the first and both masses are weighed together. By subtracting

the first measuring point value from the second the mass of the second sample is measured.

The second sample measured in measuring point 2 is a so-called sinker, an inert sample with well known volume and vacuum mass. The measurement of the apparent weight change of the sinker is necessary for a buoyancy correction of the gravimetric measurement. Any sample weighed at a pressure (m_s) opposed to vacuum ($m_{s,vac.}$) shows an apparent mass defect because of buoyancy effects due to sample volume (V_s) and fluid density (ρ^f):

$$m_s = m_{s,vac.} - V_s \rho^f \quad (1)$$

To correct the buoyancy effect the weight change of an inert sample (sinker) with measured actual mass (m_{si}) and well known vacuum mass ($m_{si,vac.}$) and volume (V_{si}) can be measured. By rearranging Eq. (1) for the sinker the fluid density can be calculated, even if no equation of state is available:

$$\rho^f = \frac{m_{si,vac.} - m_{si}}{V_{si}} \quad (2)$$

With known sample volume combining Eqs. (1) and (2) yields the buoyancy correction for the sample weight.

$$m_{s,vac.} = m_s + V_s \frac{m_{si,vac.} - m_{si}}{V_{si}} \quad (3)$$

For performing the measurements in fixed solid beds, where the measuring gas streams through the sample, special sample containers are available. To implement process-near conditions, these containers can be set on a sealed support in the zero position (Fig. 3).

Download English Version:

<https://daneshyari.com/en/article/202723>

Download Persian Version:

<https://daneshyari.com/article/202723>

[Daneshyari.com](https://daneshyari.com)