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Understanding Interfaces

*Understanding the **wetting properties of powders** is crucial to predict the behaviour when it comes into contact with a liquid phase. For a huge range of technical applications in the field of ceramics, clays, pharmaceutical or cosmetic preparations, the wettability of the involved powders is essential. The **Washburn method** [1] is used in this context to determine dynamic contact angles on the powders from which the **surface energy (SE)**, with its polar and dispersive parts can be calculated. In this application note the wettability of silica powder with various wetting liquids is assessed using a **tensiometer of the DCAT series** by DataPhysics Instruments and its corresponding software.*

Fig. 1: Silica powder

Keywords: DCAT ▪ Dynamic Contact Angle Measurement ▪ Washburn Method ▪ Powder ▪ Surface Energy

Technique and Method

The tensiometers of the DCAT series from DataPhysics Instruments are universal measuring systems for the force-based study of interfacial parameters and phenomena. With the software module DCATS 32 and suitable sample holders it can be used to measure dynamic contact angles on various solids, like implants, plates, films, powders, fibre bundles and even single fibres. The advancing contact angle of powders and fibre bundles can be determined by the Washburn method.

The Washburn method approximates the flow of a rising liquid through a packaged powder or fibres as a flow through a series of parallel tiny capillaries. Washburn [1] applied the Hagen-Poiseuille equation to calculate the flow rate in cylindrical capillaries:

$$h^2(t) = \frac{r\sigma\cos\Theta_{adv}}{2\eta}\cdot t \qquad \text{Eq. 1}$$

Where h is the distance travelled by the liquid front, σ the surface tension of the liquid, r the radius of the capillary,

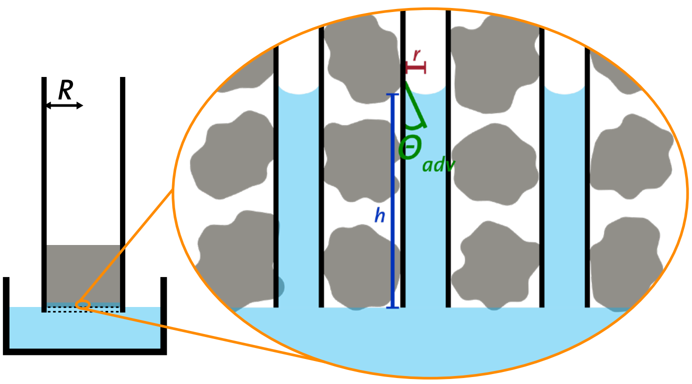

Fig. 2: Liquid transport through the porous solid is modelled by a series of parallel capillaries

Θ_{adv} the advancing contact angle, η the viscosity of the liquid and t the time of flow.

The filling height h is rather challenging to measure, hence a weight-based approach is preferred, which can be measured very precisely with a force tensiometer. Since the powder/fibres are measured within a test tube (see Fig. 2) the liquid height h and the mass gain by the absorbed liquid follow an easy relation. Assuming the liquid with density ρ forms a cylindrical column with radius R, height h, and with the sample porosity ϕ the mass gain m is given by:

$$m = \rho\cdot\phi\cdot V = \rho\cdot\phi\cdot\pi R^2 h \qquad \text{Eq. 2}$$

Combining Eq. 1 and Eq. 2 yields the **modified Washburn equation**:

$$m^2(t) = \underbrace{r\phi^2(\pi R^2)^2}_{C}\cdot\frac{\rho^2\sigma\cos\Theta_{adv}}{2\eta}\cdot t \qquad \text{Eq. 3}$$

in which C is the so-called geometric factor or capillary constant.

Notably, C depends on the dimensions of the sample particles and on their packing. It can be empirically determined in a referencing experiment with a liquid with very low surface tension, such as n-hexane (σ = 18.43 mN/m), for which one assumes complete wetting (Θ_{adv} = 0°) on almost any solid. Once the geometric factor is determined, it may be substituted into the modified Washburn equation and the advancing contact angle for other liquids can be calculated.

In the following the application of the method will be presented at the example of silica powder.

Experiment

In this application note the surface energy of silica powder (mesh 60) was determined via the Washburn method with a tensiometer of the DCAT series from DataPhysics Instruments.

Silica powder was chosen as our example because it shows good absorption characteristics and has a wide-ranging relevance in many analytical and industrial applications.

To determine the surface energy of a powder with its polar and dispersive parts, the contact angle is measured with several test liquids and evaluated according to the OWRK-model (Owens, Wendt, Rabel and Kaelble).

Crucial to the reliability of this method is a consistent packing of the powder in a series of tubes used for this evaluation. The powder must be packed reproducibly for each measurement within clean and dry tubes. The bottom of each tube is closed with a porous ground glass filter or frit. A small circular filter paper is placed on top of the frit before filling the tube to avoid clogging the pores of the glass frit. A precisely weighed quantity of the powder is placed into the tube. In order to achieve a reproducible packing of the powder the tube is manually tapped on top of the laboratory table until the powder has found its level. A good general rule for powder packing is that a higher degree of reproducibility between tubes will be obtained the tighter the packing is. Please note that compressing the powder with e.g., a plunger is not advised.

When determining the capillary constant *C* for a particular powder, a liquid with a low surface tension is chosen and the contact angle against the powder is assumed to be 0°. *n*-hexane is generally used for this purpose.

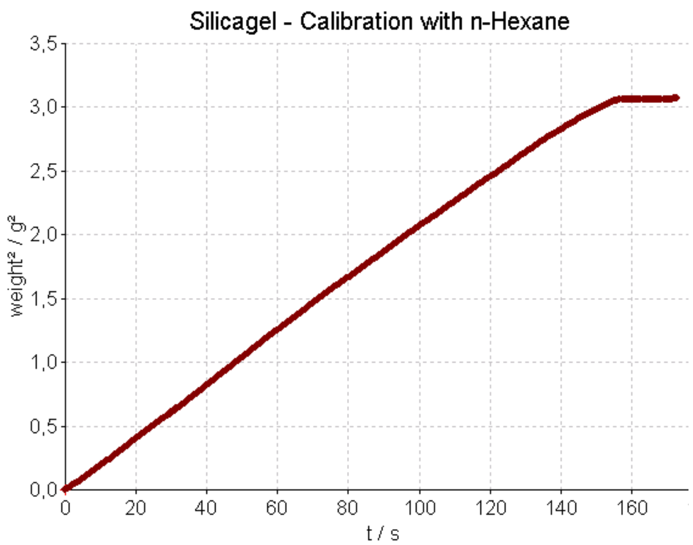

Fig. 3: For the Washburn method the increasing squared weight of the rising liquid is measured over time

Having determined the capillary constant C, with *n*-hexane, the determination of surface energy of silica powder with different liquids of different polarities can be carried out.

Generally, water, ethylene glycol, diiodomethane and dimethyl sulfoxide (DMSO) are selected (for their range of polarities) as test liquids from which the wetting contact angles can be determined and then subsequently the surface energy for the powder can be calculated.

The experimental procedure described can be transferred to any material, in powder form, for which the wetting properties are to be determined.

Results

The contact angles on silica powder (mesh 60) were determined with water and diiodomethane. Table 1 details the measured contact angles.

Table 1: Contact angle on silica powder with different liquids

	Diiodomethane	Water
contact angle [°]	50.92 ± 2.90	47,17 ± 2.58

Evaluating the surface energy according to the OWRK-model based on these contact angles yields the values given in table 2. The results show a high polarity of silica powders.

Table 2: Surface energies and their dispersive and polar parts

	SE (total) [mN/m]	Dispersive part [mN/m]	Polar part [mN/m]
Silica powder mesh 60	52.06	21.37	30.69

Summary

The tensiometers of the DCAT series with their dedicated software from DataPhysics Instruments make the **study of wetting properties of powders easily accessible**. Knowing the wettability of powders plays a crucial role in the development and production of clays, ceramics, pharmaceutical & cosmetic preparations, and a large range of other technical applications. Using the Washburn method contact angles can be measured and the **surface energy of powders can be calculated reliably and reproducibly**.

Literature

[1] Edward W. Washburn, *The dynamics of capillary flow*, Phys. Rev. **17** (1921), 273–283

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