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Application Note 10

Determination of surface free energy of powders with DataPhysics DCAT 11

Determination of wetting properties of powders by the Washburn method.

Problem

An understanding of the wetting properties of powders, together with the mechanisms involved and the consequences there of, is crucial within a number of application fields. Whether the area of interest be; ceramics, clays, pharmaceutical or cosmetic preparations or a huge range of 'technical' applications, the wettability of the powders involved is essential. In order to assess this important material property evaluation of the surface free energy, as well as its polar and dispersive parts, is most useful.

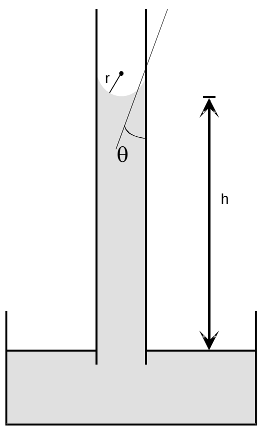
This application note introduces the Washburn method [1], applied to silica powder, by which surface free energy will be calculated from contact angle assessment with a range of wetting liquids by use of the DataPhysics DCAT 11 and the software module, SCAT11. Silica powder was chosen as our example because it shows good absorption characteristics and has wide ranging relevance in many analytical and industrial applications.

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

[1] E. W. Washburn, Phys. Rev. Ser. 2, 17, 1921, p. 273

Method

The Washburn method, by which contact angle is measured, relies on the rise of a liquid into a capillary, filled with a porous medium. In so doing the solid-air interface within the capillary is replaced by a solid-liquid interface.



The Washburn equation defines the flow of a liquid into the capillary:

$$h^2 = \frac{t \cdot r \cdot \gamma_L \cdot \cos\Theta}{2\eta}$$

Where: h is the distance travelled by the liquid front, γ_L the surface tension of the liquid, r the radius of capillary, Θ the advancing contact angle, η the viscosity of liquid and t the time of flow.

A powder which is packed in a column can be described as a bundle of capillaries with a mean capillary radius $\bar{r}$. This modifies the Washburn equation as follows:

$$h^2 = \frac{(c \cdot \bar{r}) \cdot t \cdot \gamma_L \cdot \cos\Theta}{2\eta}$$

where $(c \cdot \bar{r})$ is a constant to approximate the tortuous path a liquid must take through the described bundle of capillaries. In fact, this term is an empirical constant which depends on particle size and degree of packing.

When employing the DataPhysics DCAT 11 (balance based tensiometer), for contact angle measurements, the rise of the liquid is replaced with mass gain. Modifying the Washburn equation further:

$$\frac{m^2}{t} = \frac{\left[(c \cdot \bar{r})\varepsilon^2(\pi R^2)\right]\rho^2 \gamma_L \cos\Theta}{2\eta}$$

where m is the weight of the penetrating liquid, ρ the density of measuring liquid, ε the relative porosity and R the inner radius of the measuring tube.

The term in square brackets is a constant, material factor, known as packing constant, C (mm^5). C can be determined, experimentally, determining the 'wicking rate' (defined as the speed of a liquid front as it moves up a packed column of powder) using a totally wetting liquid, which has contact angle of 0° against the powder.

Once the packing constant is determined, it may be substituted into the modified Washburn equation and the advancing contact angle for other liquids can be calculated.

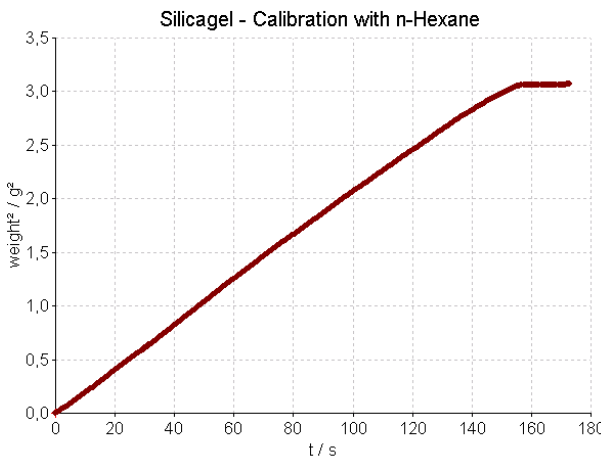
Procedure

To determine the surface free energy of a powder with its polar and dispersive parts, the contact angle is measured with a number of test liquids and evaluated according to the methods of Owens-Wendt or Wu. Crucial to the reliability of this method is a consistent packing of the powder in the series of tubes used for this evaluation. The powder must be packed reproducibly for each measurement and the tubes clean and dry.

The bottom of each tube is closed with a porous ground glass filter or frit. A small circle of filter paper should be placed on top of this frit before filling the tube in order to avoid clogging the pores of this glass frit. Precisely weigh a quantity of the powder to be placed into the tube and add it to the tube in a way that can easily be reproduced for later tubes. Tap the tube, manually, on a hard surface until the powder has found its level. A good general rule for powder packing is that a higher degree of tube to tube reproducibility will be obtained the tighter a packing achieved.

When determining the packing 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°.

(Hexane is generally used in this regard).



Having determined the packing constant, C, with this fully wetting (zero contact angle) liquid, the contact angle(s) with different liquids of different polarities can now be carried out.

Very often water, ethylene glycol, diiodomethane and dimethylsulfoxide (DMSO) are selected (for their range of polarities) as liquids from which the wetting contact angles can be determined and then a subsequent surface energy calculation for the powder, be made.

Results

The contact angles, relative to silica powder (mesh 60), were determined, with water and di-iodomethane. Table 1 details these measured contact angles.

Table 1. Contact angle on silica powder with different liquids

	Di-iodomethane	Water
contact angle [°]	**50.92 ± 2.90**	**47,17 ± 2.58**

When evaluating surface energy from this contact angle data, according to Owens-Wendt model, the values detailed in Table 2 are obtained.

Table 2. Surface free energies and their dispersive and polar contributions in mN/m.

	Surface free energy (total) [mN/m]	Dispersive part [mN/m]	Polar part [mN/m]
silica powder mesh 60	**52.06**	**21.37**	**30.69**

Summary

The DCAT 11 with the software module, SCAT 11, provides an easy way to determine the surface energy of powders.

The procedure described above should provide accurate contact angles and subsequent calculation of the surface free energy of powders within the range of 30 - 60 mN/m.

For powders outside this range, our method is less robust. Even in these case, the method provides useful data for comparing wetting characteristics of liquids on these powders.