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Application Report

Application report: AR293

Industry section: Pharmaceuticals, Food

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Date: March 2020

Force Tensiometer – K100

Method: 

Keywords: adsorption, contact angle, dispersability, fillers, microcrystalline cellulose, pharmacy, powder wetting, tablets, Washburn, wettability

Improving the reliability of powder contact angles with the Washburn Direct method and a tool for uniform sample preparation

A reproducibility study based on measurements of the water wettability of microcrystalline cellulose powder

In many industrial sectors, homogeneous mixtures between powders and liquids, so-called dispersions, must be produced. For ready meals, for example, spice powder is stirred-in on a large scale, in paint formulations pigments are the most important component, and in pharmaceuticals powders are often mixed with water by the patient himself before administration. Whether these processes are successful or whether lumps are formed depends on the wettability of the powder.

The powder contact angle, which reflects the wettability, has been measured for a long time using the Washburn sorption method. This Application Report uses the more recent Washburn Direct approach for analyzing cellulose microcrystalline powder as a model sample. To ensure the particularly important homogeneity of the samples, a special preparation set was utilized. We could show that the thus measured water contact angles exhibit high reproducibility, which is hard to achieve with the classic Washburn approach and conventional preparation methods.

Background

About the Washburn method

The Washburn methodology [1,2] is the method of choice in analyzing the wetting behavior of porous solids, which include powders, woven and non-woven textiles, and composite fibers. Typically, the intrusion of a liquid into a porous solid through capillary action is monitored over time and is governed by the Washburn equation:

$$\cos\theta = \frac{\mathrm{m}^2}{t}\frac{\eta}{\rho^2 \sigma c},$$

where σ, η and ρ are the surface tension (SFT), viscosity and density of the liquid, respectively. These are easily accessible properties of the liquid used. In order to measure the contact angle θ, the second unknown, i.e. the capillarity constant c, must first be determined. c is a material constant of the particular powder package which can be determined using low SFT liquids such as hexane or heptane. These liquids are expected to fully wet the sample so that the contact angle will be zero. The thus measured value for c is then used to determine the contact angle with other liquids.

A plot of a typical Washburn Direct measurement as displayed in the ADVANCE software is shown in Fig. 1, where cos**θ** is plotted versus time. When the sample is properly prepared, which means that the packing density does not change with height, cos**θ** shows a plateau. By moving sliders, the required plateau region can be selected, i.e. the one with the lowest error for the determination.

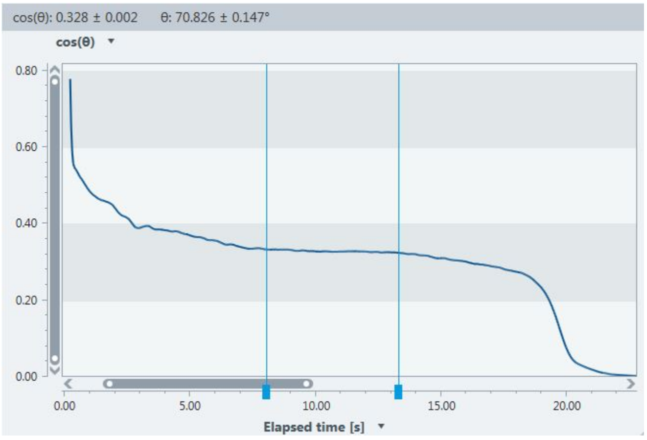

Fig. 1: Example plot of a Washburn Direct curve

How to achieve homogeneous and reproducible powder packings

Porous solids such as powders exhibit measurement variability depending on how consistently the powders are compressed. Although jolt and centrifuge instruments can be used to pack these porous solids, when dealing with powders with variable particle sizes, smaller particles tend to migrate and congregate. This means that particle size separation is evident when packing with either of these methods. In the Washburn Direct plot, this would result in a curve that has no plateau region.

Herein, we demonstrate how a recently developed Washburn preparation set (Fig. 2) helps in carrying out precise and reproducible contact angle measurements with the Washburn Direct method. The set provides for uniform sample compression using weights of a precise mass.

Fig. 2: Washburn sorption cell and preparation set with packing weights

We carried out water contact angle measurements using microcrystalline cellulose powder as a model sample. This substance is a nature-based polymer used in various products as an anti-caking agent, a fat substitute, or an emulsifier in food production. It is also extensively utilized as an excipient in the pharmaceutical industry to facilitate tablet disintegration. The wetting behavior of this powder in both water and biological media is of great interest. For example, it affects the release and delivery of the active ingredients in tablets.

Experimental section

Materials

Cellulose microcrystalline powder (Avicel® PH-101) and n-hexane (ACS reagent grade) were purchased from Sigma-Aldrich. For all capillarity constant and contact angle measurements, n-hexane and distilled water were used, respectively.

Powder compression

Cellulose microcrystalline powders were compressed using the SH0824 Washburn sorption packing weight set shown in Fig. 2. Filter paper was fitted onto the bottom of the Washburn cell and a sorption glass tube was then inserted in the cell. The cap was screwed on top of the Washburn cell, thereby pressing the glass tube against the bottom. An amount of 0.75 g of the microcrystalline cellulose powder was then weighed directly into the sorption glass tube (Fig. 3A). The cell was placed in a fitted shape metal container. The plunger with the attached circular weights was inserted inside the sorption glass tube and the powder sample was compressed for 30 seconds. The cell containing the compressed powder (Figure 3B) was used for either capillarity constant or contact angle determination. In order to compare reproducibility, the procedure was carried out with a 1000 g and a 1500 g weight.

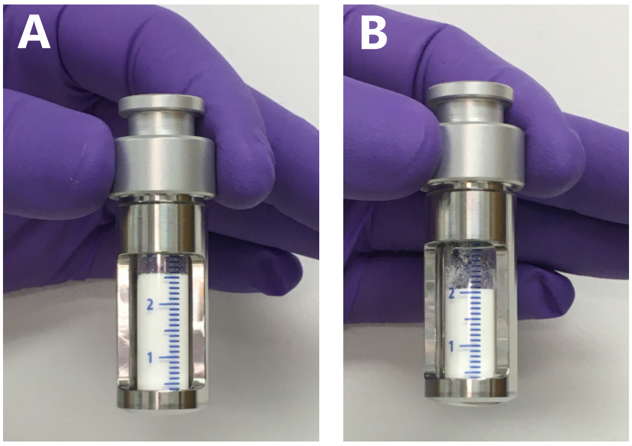

Fig. 3: Microcrystalline cellulose powder before (A) and after (B) compression.

SFT, capillarity constant, and contact angle measurement

The SFT of water and n-hexane was initially measured with the Wilhelmy plate method using a K100 Force Tensiometer.

For analyzing the solid sample, the Washburn cell containing the compressed powder was attached to the force sensor of the K100 Force Tensiometer (Fig. 4). The liquid uptake for n-hexane (for capillarity constant) and water (for contact angle) was recorded until no or almost no more adsorption (i.e. mass increase) could be detected. All measurements were carried out at 25 °C.

Fig. 4: The Washburn cell containing the compressed powder sample is attached to the force sensor

Results

Liquid data required for Washburn evaluation

Table 1 shows the results of the SFT measurements as well as data for the viscosity and density as taken from the ADVANCE software database.

Table 1: Liquid data at 25 °C as required for the calculation according to Washburn

Liquid	n-hexane	water
Mean SFT (mN/m)	18.28 ± 0.28	72.63 ± 0.43
Viscosity (mPa s)	0.3131	0.9000
Density (g/cm^3)	0.6594	0.9970

Determination of the capillarity constant

The uptake of n-hexane into the microcrystalline cellulose powder packed using a 1000 g weight already exhibited comparable Washburn Direct curves. The calculated capillarity constants for 10 determinations (Table 2) gave a mean value of 4.11 ± 0.38 mm^5, which corresponds to a relative standard deviation (RSD) of 9.3%. A packing regime using 1500 g weights was then investigated to assess whether the heavier packing weight resulted in higher precision in the capillarity constant determination. The resulting curves show a good agreement (Fig. 5).

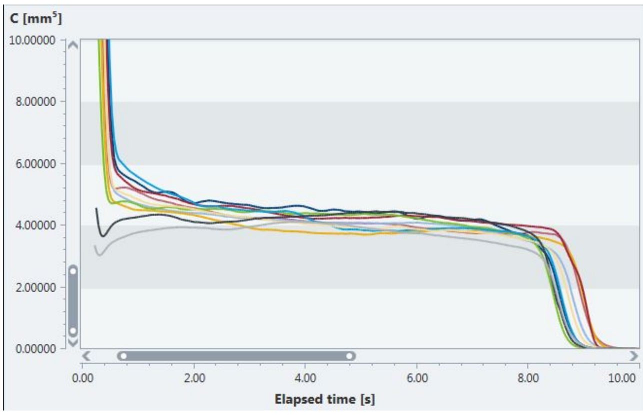

Fig. 5: Washburn Direct curve overlays for capillarity constant determination of packed microcrystalline cellulose powder (1500 g for 30 sec) using n-hexane

Table 2 lists the results for the samples packed with 1000 g and 1500 g, respectively. The values confirm that the 1500 g weights gave very similar, but more precise capillarity constant results.

Table 2: Capillarity constant of microcrystalline cellulose powder using n-hexane

Compression weight (g)	Mean capillarity constant (mm^5)	RSD (%)
1000	4.11 ± 0.38	9.3
1500	4.12 ± 0.22	5.3

Contact angle measurements

Fig. 6 shows the Washburn Direct curves for the uptake of water into microcrystalline cellulose powder, packed with a 1500 g weight.

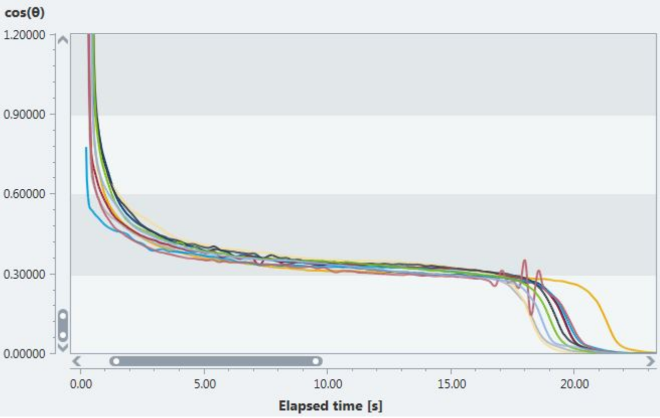

Fig. 6: Washburn Direct curve overlays for water contact angle determination of packed microcrystalline cellulose powder (1500 g for 30 s)

The mean water contact angle for 10 determinations was 71.0° ± 0.8° with an RSD of 1.1% (Table 3). Similar to the capillarity constant determination, this shows that highly comparable contact angles can be obtained.

Table 3: Contact angle of water on microcrystalline cellulose powder

Compression weight (g)	Mean contact angle (°)	RSD %
1000	68.1 ± 1.7	2.5
1500	71.0 ± 0.8	1.1

Summary

Powder contact angle measurements provide information on wettability, which is important for the production of homogeneous dispersions. However, reproducible results have not been easy to obtain so far. This application report shows the way to better comparable contact angle results. Using microcrystalline cellulose powder as a model substance, measurements were carried out using the recently developed Washburn Direct method. To achieve homogeneous powder packages with always the same packing density, samples were uniformly compressed using a newly developed preparation set.

Low relative standard deviation was attained with a tenfold determination of the required capillarity constant using n-hexane. An equally good agreement was achieved in the tenfold determination of the water contact angle. When compressing this specific powder it was also found that reproducibility could be increased with a heavier weight for compressing, i.e. with 1500 g instead of 1000 g.

The measurements indicate that, using the special preparation set with the Washburn Direct method, highly reproducible and precise powder contact angle results can be obtained.

Literature

[1] Edward W. Washburn: The Dynamics of Capillary Flow. Phys. Rev., 17, 374 (1921).

[2] Wettability studies for porous solids including powders and fibrous materials. KRÜSS Technical Note TN302.

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