

Measuring the wettability powders

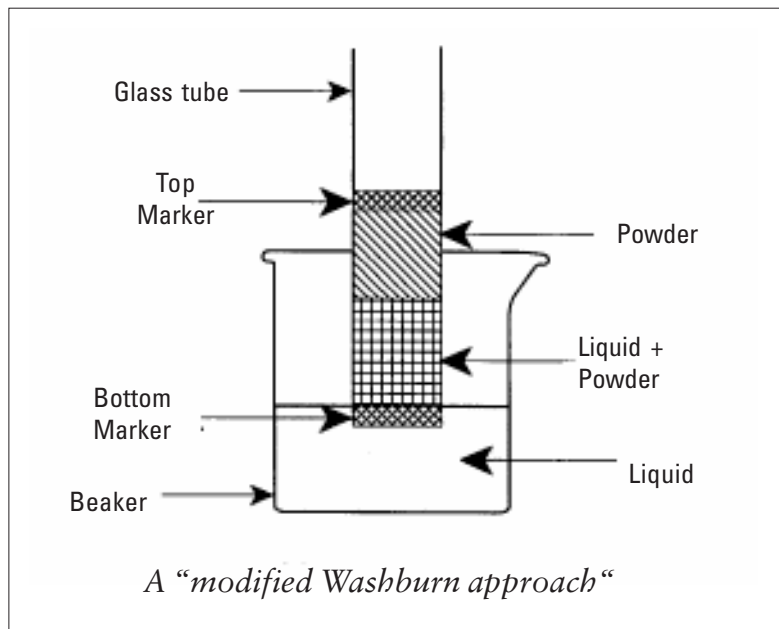
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The wettability of powdered materials is an area of widespread interest throughout many industries and an important element in the formulation of many products including pharmaceuticals, paints, printing inks, photographic film, audio tapes, and adhesives to name a few. Since powdered materials are typically highly absorbent, attempts to separate wettability from absorption with an optical (sessile drop) contact angle measurement have been largely unsuccessful and difficult to reproduce. Gravimetric methods offer a promising alternative to optical methods by accounting for both absorption and wetting properties simultaneously while minimizing the subjectivity introduced by operator error. In this note, three methods will be discussed:

1. an optical method to record the wicking or penetration rate of a liquid through a packed column of powder;
2. a gravimetric method to measure the wetting of a compressed sample of the powdered material via the Wilhelmy plate technique; and
3. a hybrid technique proposed by the author to incorporate the packed column concept (1) with the gravimetric technique (2)

The optical packed column method

Several ideas have been advanced in the literature to measure the wettability of powder samples. One is based on a visual determination of wicking flow in a capillary tube using the Washburn equation to relate the contact angle of a liquid to the wettability of a powder. Using this approach, the rate of penetration or flow of liquid inside a capillary tube of known radius is recorded as the time it takes for a liquid of known surface tension to penetrate a certain distance within a packed column



of powder. The distance is measured typically with the aid of a light source and a visual determination of the meniscus front is made with the naked eye. The Washburn equation relates this penetration rate (L^2) to the surface tension of the probe liquid (γ), the viscosity of the liquid (η), the capillary radius (r), and advancing contact angle (θ) as follows:

$$L^2 = (C * r) * \gamma * \cos\theta / 2 * \eta$$

Where:

L = the length of flow in time t

C = constant to account for randomly oriented capillaries

r = the radius of the capillary

γ = the surface tension of the liquid

θ = the advancing contact angle the viscosity of the liquid

η = the viscosity of the liquid

From an operator's perspective the method can be tedious and is by nature very subjective because the critical parameter (L^2) is measured by a visual determination of the advancing liquid front in a measured time t . If a way could be

found to transfer the measurement responsibility from the operator to a more objective device (i.e. an instrument), perhaps this technique would have a greater appeal.

The compressed disk gravimetric method

An alternative approach to measure contact angle using the gravimetric principle of the Wilhelmy plate technique requires the powder sample to be compressed into a solid (pellet or flat plate configuration). By choosing liquids that do not penetrate the pores of the powder too quickly, a dynamic contact angle scan of the powder surface can be recorded and the wettability of the powder directly correlated to the dynamic contact angle. This approach has been applied with some success in the pharmaceutical industry where pure drug compounds have been compressed in a KBr press at high compression rates to form Wilhelmy plate samples. Measurements can typically be made of the advancing contact angle before wicking becomes a problem. Whenever the powder can be com-

pressed readily into a solid form and remain intact this method can be used. Concerns about the effect of compression on the surface chemistry of a powder have been raised when evaluating this method. On a relative basis, when comparing similar samples that have all been prepared the same way, the effect of compression may not be a major concern, however, on an absolute basis it is worth considering when interpreting data from compressed samples. The gravimetric method can be quite effective and easy to use given the sample prep considerations discussed are satisfactorily considered. In some cases, however, compression is not compatible with the powder of interest and alternative methods must be considered.

The hybrid packed powder-gravimetric method

In trying to come up with a way to incorporate the best qualities of each method, the possibility of a hybrid method arises that offers an alternative to either method independently. This method, conceived in the author's laboratory, uses a combination of the two techniques described above – a “modified Washburn approach” that combines the best of both techniques.

The Washburn equation is used to determine the advancing contact angle as described in (1), however, instead of measuring the penetration rate visually as a function of distance, the rate is measured by recording the weight gain over time. Two “markers” are used in preparing a column- one at the bottom end of the column to hold the powder in place and initiate the liquid penetration, and another at a predetermined distance from the bottom marker. The penetration rate is recorded as the time it takes for the liquid to pass through the column from one marker to the other. When the liquid level is first brought in contact with the bottom marker, the liquid penetrates the first marker readily and then proceeds to penetrate through the powder sample. The rate at which

the powder penetrates through the powder is recorded until the meniscus reaches the top marker. When the liquid front reaches the top marker, the time required to reach this point is recorded and used in the Washburn equation (above). To “calibrate” the column, the constant ($c \cdot r\text{-bar}$) can be measured for each powder packing density by choosing a liquid that readily wets the powder ($\theta = 0$). Once the constant has been determined, θ and the Work of Adhesion for each liquid can be readily determined from the Washburn equation and thus the wettability of the powder can be calculated.

By incorporating a highly absorbent “marker” at a known distance along the column, the distance traversed by the liquid can be recorded independently of the operator traversed the need for a visual measurement. In this way, the balance serves as the analytical instrument in place of the operator's vision. Using this method, the operator can compare the wettability of a number of powder samples in a semi-automated approach without the subjectivity of method (1) or the compression requirements of method (2).

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