Overview

Powder wettability measurements

Powders have a significant role in many applications such as coatings, pharmaceutical drugs, and food products. Wettability of the powder is important when the processes and products are being developed. For this reason, several methods have been developed to measure the contact angle of the powder. Most used measurements are Washburn method and sessile drop. An extensive review of these methods was done by Alghunaim et. al [1]. Here a short overview of the both is given and the methods are compared.

Washburn method

Capillary rise or Washburn method can be done by using force tensiometer and a special type of powder holder. The measurement set up is shown in Figure 1. The powder holder, with small holes in the bottom, is immersed into the liquid and the amount of liquid absorbed into the powder bed is measured against time.

Capillary rise method is based on the Poiseuille law related to the liquid flow through a capillary

$$v = \frac{R_D^2}{8\eta}\frac{\Delta P}{l}$$

,where v is the kinetics of flow, R_D the mean hydrodynamic radius of the capillary, η the viscosity of the liquid, l the length of the capillary and ΔP the pressure difference. ΔP can be expressed as a sum of a capillary pressure and a hydrostatic pressure. Thus equation can be written

$$\frac{dh}{dt} = \frac{R_D^2}{8\eta h}\left[\frac{2\gamma_l cos\theta}{R_S} \pm \rho g h\right]$$

h is the liquid front height, g the gravitational constant, R_S the mean static radius of the capillary, and ρ the liquid density. Now if hydrostatic pressure is neglected and we suppose that $r = R_D^2/R_S$, integration of the equation with boundary condition h=0 when t=0 gives

$$h^2 = \frac{r\gamma_l cos\theta}{2\eta}t$$

Equation is called Washburn equation.

The porous bed is considered as a bundle of parallel capillaries of constant radius. The use of this equation would require a visual observation of the moving liquid front. Since this is much more difficult to do than measuring the gained weight, the weight w is related to the height in the cylinder by

$$w = \varepsilon\rho\pi R^2 h$$

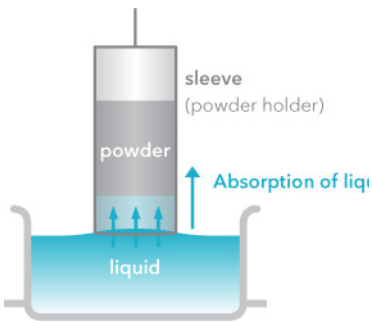

Figure 1: Schematic of a powder holder immersed in the liquid

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Figure 2: A typical powder wettability curve

,where ε is the porosity of the packed powder column, ρ the density of the liquid, and R the inner radius of the tube. And finally combining the equations

$$w^2 = (r\varepsilon^2(\pi R^2)^2)\frac{\rho^2\gamma_l cos\theta}{2\eta}t$$

,which leads to

$$w^2 = c\,\frac{\rho^2\gamma_l cos\theta}{2\eta}t$$

Equation above is often referred as modified Washburn equation. The term c is a geometric factor and is constant as long as packing and the particle size remains similar [1]. It has to be experimentally determined to each type of packing and powder combination. Precise and accurate determination of the constant c is critical for the correct application of the Washburn equation. In practice, to solve the material constant the measurement has to be done first with the completely wetting liquid such as hexane, heptane or octane. The contact angle can thus be assumed to be zero (cos0 =1) and the material constant c can be calculated if the properties of the liquid are known. After definition of the material constant the contact angle measurement with the liquid of interested can be done. A powder packing method has to be repeatable and carefully controlled since it is not possible to measure the material constant and then re-use the same powder for actual contact angle measurement. A typical powder wettability curve is presented in Figure 2.

Sessile drop

Sessile drop offers the most straightforward method for contact angle measurements. The drop of liquid is placed on the sample surface and the contact angle is optically defined. For powder wettability measurements, the powders are most often compressed into tablets by applying high pressure. Hydraulic pellet press used typically to prepare KBr pellets for IR studies is a suitable tool. Another way would be to spread a thin layer of powder on a substrate (with a help of adhesive if needed), and then measure contact angle on top of that.

The method is simple compared to the Washburn method where reference liquids need to be used. However, compression of powder or spreading it on the substrate, introduces roughness on the surface which should ideally be considered in the measurements [2]. High compression when producing the tablets can also alter the powder so that it does not represent the nature of the loose powder.

In general, measurement of advancing and receding angles with the sessile drop method is possible but in case of powders the measurement can be challenging due to adsorption of a liquid.

Conclusions

Contact angle measurements on powders are important but due to the nature of the material they are also challenging to conduct. It is good to keep in mind that different types of powders might require different measurement methods due to the properties of the powder and application. Comparison of the results obtained with different measurement methods is typically not possible as several aspects can affect the contact angle data obtained.

Washburn method is the only method available when wettability of loose powders need to be evaluated. Care needs to be taken as the packing of the powder can have a significant effect on the contact angle values. The first step in powder wettability analysis with Washburn method is the development of the packing method for that particular powder.

Sessile drop method offers a slighly straighforward method for powder wettability analysis but the compression of powder can potentially change the surface properties of the material. This is something that needs to be considered for each powder type separately. Some powders are also impossible to compress.

References

[1] A. Alghunaim, S. Kirdponpattara, and B. Z. Newby, "Techniques for determining contact angle and wettability of powders", Powder Technology 287 (2016) 201.

[2] S. Laurén, "Attension Theta Flex Optical Tensiometer with 3D Topography Module"