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Review Techniques for determining contact angle and wettability of powders

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ABSTRACT

This review summarizes some common methods for determining the wettability of powders, especially the contact angle that a liquid would form on powders. The sessile drop, Wilhelmy plate and liquid penetration methods are then briefly compared, especially on the advantages and disadvantages of these techniques. The later part of the review summarizes some experimental details in determining wettability of powders utilizing liquid penetration into a packed column or the Washburn capillary rise (WCR) method based on liquid mass uptake. The discussion is focused on the selection of capillary tube for packing, bottom support, powder properties, reference wetting liquid, and packing method. How each of these parameters influences mass uptake and subsequent wettability determination are also presented, followed with the applicability of mass based WCR for determining the wettability of some powders. The intent of this review is to provide researchers a guide on the proper selection of the method and then the associated details when they apply the mass based WCR experiments for deducing the wettability information of the powders of interest.

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1. Introduction

Powder wettability plays a significant role in many applications such as coatings, dispersion, as a precursor to dissolution, powder processing such as granulation and other practical usages [1-14]. Such significance has driven scientists to develop ways to assess the wettability of powders. Generally, the wettability of solid surfaces is based on the angle

that is formed by the liquid of interest on a particular solid surface. One of the most common techniques for measuring contact angle is the sessile drop technique where a liquid drop is placed on a surface and the contact angle is measured directly at the three phase (liquid/solid/vapor) contact line of the formed drop. While this technique might be straightforward for flat smooth and non-porous surfaces, several issues such as surface roughness, swelling and liquid penetration into pores make it difficult to measure the contact angle of powders directly and as a result, several indirect methods that relate penetration rate or pressure to contact angles have been developed [15–20]. Nevertheless, sessile drop, Wilhelmy plate and liquid penetration (both column and thin film wicking) are still the most commonly reported







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The common methods used for evaluating wettability of powders (i.e., the contact angle of a liquid formed on a powder).

| Method | Sample | Advantages | Disadvantages/applicability limits | References |
|----------------|--|---|--|-----------------------------|
| Sessile drop | Powder compacted discs (15 mg to <10 g) under high pressure (70-700 MPa) powder layer deposited on a substrate from a solution or using an adhesive | Simple, reproducible | Compacting pressure affects surface properties of powder, including an inevitable change (e.g., plastic deformation) of the sample surface, contact angle strongly depends on the compact pressure Surface structure leads to drop penetration and air trapping, both of which greatly influence the contact angle. Low contact angles lead to fast penetration resulting in unstable drop shape. Air trapping influences liquid spreading. Surface roughness results in only the apparent contact angle, not the actual contact angle, thus mostly qualitative; the porous and rough compact surface causes a much higher hysteresis, and the roughness cannot be predicted correctly Powder could disperse into the drop, when the bed/disc needs pre-saturation, swelling and deformation might result, making the measured contact angle questionable | [1,2,14,21-28,32-37,52,57-6 |
| Wilhelmy plate | Powder (milligrams) coated on a substrate with or without the aid of an adhesive or powder compacted into discs or rectangular shapes | Capable of obtaining a wide range of wettability including low contact angles (<20°), consistency | Liquid imbibition into pores causes swelling, roughness effect → underestimated contact angle measured and a large contact angle hysteresis Compacting/compressing could damage the powders or alter surface properties leading to erroneous results. A serious error could result from unknown true perimeter, especially in the case of a thick adhesive or increased surface roughness When coating powder using an adhesive, not a total surface coverage could result, the adhesive might expose or spread over the powder Compacts need to be coherent before/during the measurement | [27-32,36] |

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