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Influence of tube wettability on water contact angle of powders determined by capillary rise

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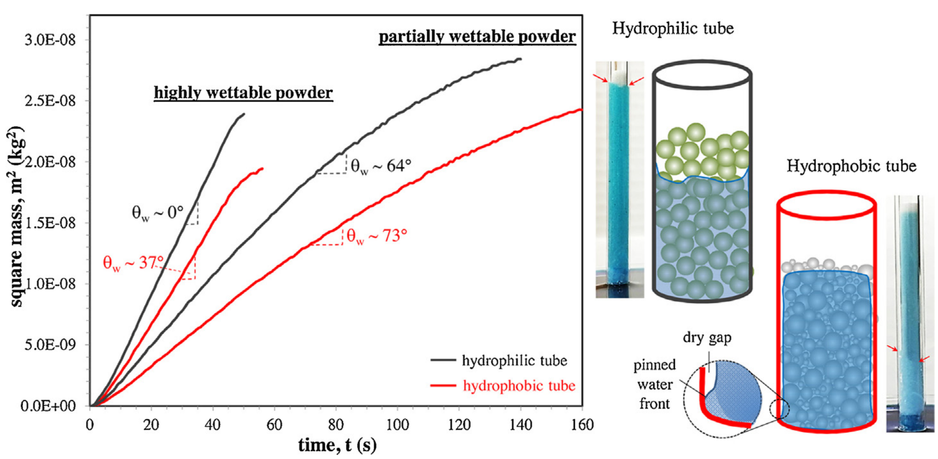
HIGHLIGHTS

- Water contact angle of powder (θ_w) was assessed by Washburn capillary rise (WCR).
- Packing tube wettability affected θ_w determined by WCR.
- A hydrophobic tube increased θ_w of a highly wettable powder.
- The effect of tube wettability on θ_w was more dramatic by using a smaller tube.

GRAPHICAL ABSTRACT



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ABSTRACT

During a recent literature review, we have noticed that researchers used tubes made of materials having a wide range of wettability, including plastics and metals, to pack powders for wettability determination using the Washburn capillary rise (WCR) method. Most studies applying WCR have focused on the effects of powder properties and their packing methods on the contact angle, while neglecting the role of packing tube. Although the ratio of packed powder surface area to tube surface area is large, we believe that tube wettability could have a significant effect on powder wettability determined using the WCR method. In this paper, we report that when water was penetrating the packed powder inside hydrophobic tubes, the water front along the tube wall was severely pinned, and in some cases, it was completely pinned at the support/packing/tube wall interface until the water had penetrated all the way up the packed powder. Using hydrophobic tubes limited the water rise to the powder region, which resulted in contact angles that were larger (up to ~37°) than ones obtained using hydrophilic tubes, especially when the tube size was small.

1. Introduction

From reviewing the existing literature on wettability determination of powders [1], the Washburn capillary rise (WCR) method is one of the most popular methods for powder wettability deter-

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Nomenclature

WCR	Washburn capillary rise
t	Penetration time
m	Mass of penetrating liquid
C	Geometry factor of the packing
ρ	Density of the penetrating liquid
γ	Surface tension of the penetrating liquid
μ	Viscosity of the penetrating liquid
θ	Contact angle between penetrating liquid and the powders
θ_w	Contact anglebetween water and the powders
r_{eff}	Effective radius of voids in the packed powders
A	Cross-section of the tube
ε	Porosity of the packing in the tube
APTES	Aminopropyltriethoxysilane
OTS	Octadecyltrichlorosilane
DI	De-ionized
ID	Inside diameter of the tube
R^2	Coefficient of determination (measure of linearity fit)
m_o	The total force (in term of mass) associated with mass uptake into the support and the force exerted by/on the tube wall on/by the liquid
m_{wcr}	The mass uptake into the packed powder bed
m_{exp}	The total force (in term of mass) associated with mass uptake into the support, powder and the force exerted by/on the tube wall on/by the liquid
m_s	The mass uptake into the support
M	Correction factor
L	Length of the packed bed
D_p	The diameter of the beads/bubbles

mination. Other common techniques, such as sessile drop and Wilhelmy plate, require high compaction pressure to process powders into discs or pellets. Such processing has been shown to have a significant impact on the determined contact angle due to alteration of physical/surface properties of powders [2]. Depositing powders on a substrate avoids such alteration but introduces roughness and air-trapping issues [3]. Using the WCR technique over such techniques has the advantage of maintaining powder properties.

The WCR method relates the penetration rate of a liquid into a packed bed of powder within a tube to the penetrating time (t) as:

$$m^2 = \frac{C\rho^2\gamma\cos\theta}{\mu}t \text{ with } C = \frac{r_{eff}A^2\varepsilon^2}{2} \tag{1}$$

where m is the mass of the penetrating liquid, C is the geometry factor of the packing, ρ, γ, and μ are the density, surface tension and viscosity, respectively, of the penetrating liquid, and θ is the contact angle between the liquid and the powder; r_{eff} is the effective radius or the equivalent radius of voids in the packed powders, A is the cross-section of the tube, and ε is the porosity of the packing in the tube.

Most studies applying WCR have focused on the effects of powder properties and their packing methods on the contact angle, while neglecting the role of packing tube. Few researchers have recently investigated the effects of tube shape [4] and tube irregularity [5–7] on the capillary rise, both experimentally and theoretically. Researchers have normally used circular tubes of different sizes and materials, including plastics and metals that could have a hydrophobic surface [8–12]. The effect of tube properties on water contact angle of powder might seem negligible at first due to the fact that the ratio of packed powder surface area to tube surface area being large. However, we believe that tube wettability could have a significant effect on powder wettability determined using the WCR method, and such effect should not be neglected.

In this study, we hypothesize that both tube size and tube surface wettability would affect the determination of water contact angle on the powder, especially when the surface wettability of the powder is substantially different from that of the packing tube. Three powders, slightly hydrophobic glass beads, highly wettable un-treated glass bubbles and intermediate wettable treated glass bubbles, and glass tubes with two sizes (ID = 4 mm and 10 mm) and two different wettabilities (water contact angle 10–30°—hydrophilic and ~100°—hydrophobic) were utilized for the investigation.

2. Experimental

2.1. Materials and equipment

The three powders used were 3 M K15 Glass bubbles/microspheres, BioSpec 100 μm Glass beads and aminopropyltriethoxysilane (APTES) modified glass bubbles. Detailed information on these powders is presented in Table 1 and S1 (Supplementary material). One point worth noting is that the wettability of glass bubbles and glass beads, roughly assessed by the sessile drop method on a layer of bubbles or beads deposited on a scotch tape, is very different (~40–70°). The difference in wettability could be the result of how they were manufactured. Therefore the difference in water contact angles obtained by the WCR method reported in this article is due to the intrinsic properties of the powders. The glass tubes used had a length of 75 mm and an inside diameter (ID) of 4 mm or 10 mm. The modification of glass bubbles with APTES and the glass tubes with octadecyltrichlorosilane (OTS) is provided in the Supplementary material section, De-ionized (DI) water was purified in house (with a conductivity of ≤1 μS/cm) and 99% pure octane was used as the completely wetting liquid.

The main equipment used included a precision balance (Denver Instrument, model P-403 with a measurement accuracy of 1 mg) and a home built micro-manipulator containing two-directional micro-positioning stages to allow all three dimensional positioning. Other equipment included a contact angle goniometer (ramé-hart, model 100-00), an optical microscope (Olympus IX 70) and a desktop computer.

2.2. Packing of powder in tubes

A support was inserted into one end of the tube. The selection of supports for the tubes is detailed in the Supplementary material section. For glass bubbles and glass beads, ~4 mg and ~65 mg, respectively, of the powder per mm^2 cross-sectional area was measured and poured into the tube from the other end, and the tube was manually tapped on a hard surface for two minutes (~500 times) until the desired powder packing height (~45 mm) reached. To avoid any systematic errors, the WCR experiments for one particular powder was packed in one setting into at least six tubes of each of the four different types of tubes (4 mm hydrophobic, 4 mm hydrophilic, 10 mm hydrophobic and 10 mm hydrophilic) in a random order.

2.3. Washburn capillary rise

The four different types of tubes, with powder packed inside, were shuffled prior to the capillary rise experiment. Each packed tube was secured to the micro-manipulator with the tube perpendicular to the surface of the liquid (a schematic illustration of the set-up is shown in Fig. 1). To obtain the geometric factor, octane was