

# Technique for Measuring Liquid Penetration into Single Granules

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## ABSTRACT

*Wetting experiments were performed for the first time on single granules using an optical imaging technique. This technique involves dyeing the liquid and then tracking the movement of this liquid through the granule. The results allow the liquid velocity profile and the total penetration time to be measured. The effect of liquid properties, namely viscosity, surface tension, the liquid velocity and the total liquid penetration time were investigated. The experimental results were then compared with theoretical data predicted by the Washburn Equation and found in literature. The Washburn equation was found to predict the relationship between the liquid and granule properties and the penetration time.*

**Keywords,** liquid viscosity, liquid surface tension, wetting, granule

## 1. INTRODUCTION

Liquid spreading and penetration into porous solid surfaces is an important process both in everyday life and in industry. It is exhibited in everything from rainwater penetrating into soil to ink into paper. Areas of application are wide ranging from inkjet printing and paints, to the food industry where this research has its foundation. Within granulation, it is an important part of the wetting process and can have significant impact on the granule size distribution. The majority of research in this area has done on porous solids or powder beds with no work done on single granules (Popovich et al., 1999 Hapgood et al. 2002). In granulation it influences the further agglomeration of small aggregates. In coating applications liquid requirements are effected by the amount of liquid penetration occurring. In the food industry liquid interaction with the granule can affect the perception of the product.

The Washburn Equation, (Washburn, 1921), is one of the most common equations for describing liquid penetration into porous solids. The Washburn Equation approximates the penetration process to liquid penetration into a single capillary with characteristic pore radius  $R$ . It states that the liquid penetration rate is directly proportional to that pore radius, the liquid surface tension  $\gamma_{LV}$ , the cosine of the contact angle  $\theta$ , and inversely proportional to the liquid viscosity  $\mu$ .

$$\frac{dx^2}{dt} = \frac{R \cdot \gamma_{LV} \cdot \cos \theta}{2\mu}$$

With  $x$  being the liquid penetration distance, thus  $x^2$  the liquid penetration area and  $t$  the penetration time. Work has been done that shows that the Washburn Equation is relevant for describing liquid penetration into porous solids. Hapgood et al., 2002 studied the effect of liquid properties on droplet penetration time using different powder beds. The investigation method used was similar to that of measuring sessile contact angles, with liquid droplets dropped onto a flattened powder bed surface and total droplet penetration time recorded using a video camera. Liquid viscosity was found to be directly proportional to penetration time and liquid surface tension was found to be inversely proportional to the penetration time. Liquid viscosity was the more dominant parameter as it can be changed by several orders of magnitude and can thus alter the penetration time by similar orders of magnitude. Work by (Popovich et al., 1999) found the relationship between liquid viscosity and penetration time to be slightly different to that of Hapgood; Popovich found normalized liquid viscosity ( $\mu/\mu_{\text{water}}$ ) to be directly proportional to the normalized penetration time ( $t/t_{\text{water}}$ ).

In this work, liquid penetration experiments were performed for the first time on single granules utilizing a new method. The objective was to show that the developed method can measure liquid penetration into single granules well. The results obtained for changing liquid properties were then compared with the results predicted by the Washburn Equation and those found in literature.

## **2. MATERIALS AND EXPERIMENTAL PROCEDURE**

### **2.1 Granules**

The granules for this study were prepared by fluidised bed granulation. The granules were made using Maltodextrin with dextrose syrup powder as binder. The granules used were of equivalent diameter 500 - 1000 microns and an irregular shape.

## 2.2 Liquid

The penetrating liquid, solution of hydroxypropylcellulose (HPC), was prepared by dissolving HPC powder in distilled water. The viscosity of solution was altered by adding various amounts of HPC powder. For changing the surface tension, varying amounts Sodium Dodecyl Sulphate (SDS) was mixed with distilled water. The dye used was Erythrosin B. All solutions were stirred for at least 3 hours to ensure good mixing.

## 2.3 Experimental Setup

The technique developed involves analysing a high speed recording of a dyed liquid penetrating through a single granule. The progress of the dyed liquid through the granule is tracked using image analysis of the high speed recording of the process. The area occupied by the liquid with time can be measured and from this penetration times and rates can be obtained. The images were recorded using a Photron FastCam 1024 PCI High Speed Camera. Consistent drops were produced using an EFD 2415 Ultra droplet dispenser. Image analysis was performed using software Image J.

## 3. RESULTS AND DISCUSSION

Figure 1 shows a typical recording obtained for the process. A red droplet can be seen penetrating into a Maltodextrin granule of approximately 600 $\mu\text{m}$ . Penetration starts in Image 1 ( $t=0\text{s}$ ). It ends in Image 6 ( $t=0.07\text{s}$ ), when the liquid droplet has completely penetrated into the granule. The granule is red as a result. The colour is what is used to measure the area occupied by the liquid with time.

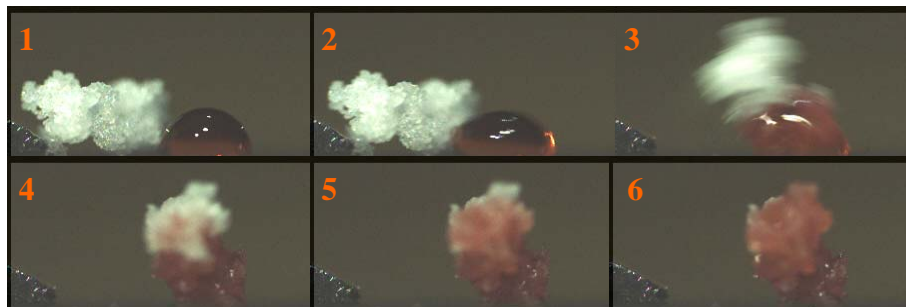
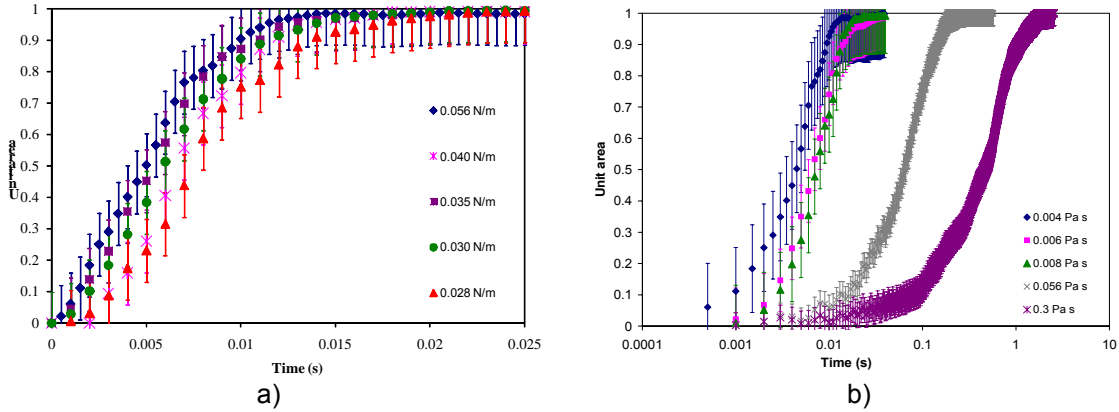
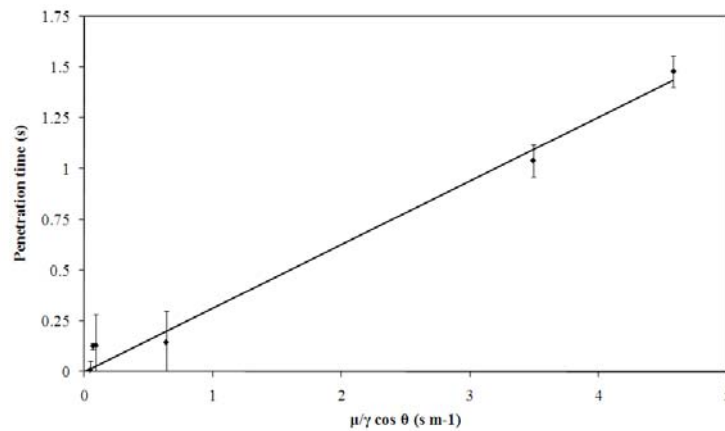


Figure 1: Liquid penetration into a single granule. The liquid surface tension was 0.056 N/m and viscosity was 0.004 Pa s



**Figure 2: Normalised area covered by liquid as a function of penetration time for different a) surface tensions and b) viscosities.**

Figure 2 shows the typical results obtained from the penetration experiments. The y-axis shows the change in unit area with the change in penetration time on the x-axis. For the effect of liquid viscosity on the penetration time graph, the time axis is on a logarithmic scale. The unit area is the area that the liquid occupies at a respective time divided by the total area of the granule. Unit area thus is 0 at the beginning of the process and 1 at the end of the process. This is used as it allowed granules of slightly differing size to be compared. The results show viscosity has a significant influence on the liquid penetration time. Surface tension has a minimal effect on the penetration time. Granule porosity was also found to significantly alter the penetration time. If the Washburn Equation holds true, a plot of liquid penetration time against liquid properties ( $\mu/\gamma\cos\theta$ ) should give a straight line. Figure 3 shows a plot of different wetting liquids and the resultant penetration time. The result is a straight line between liquid properties and penetration time. Granule porosity was also found to be inversely proportional to the liquid penetration time. The results obtained concur with those found by Hapgood et al 2002 on powder beds and the Washburn Equation.



**Figure 3: Effect of changing liquid properties against penetration time. N.B different values of ( $\mu/\gamma\cos\theta$ ) were obtained by varying HPC and SDS concentrations in the solution.**

The same work was repeated using two cameras to look at the process from two perspectives to compare penetration occurring in different directions and to give 3-D information. The liquid penetration rates in the investigated directions were found to be the same. Estimating the penetrated volume from the penetrated area by assuming the granule was a perfect sphere was found to be the best method.

#### **4. CONCLUSION**

Wetting experiments were performed using a novel imaging technique that uses a dyed liquid to track the progress of liquid penetration through single granules. Using this technique, liquid viscosity was found to be linearly related to penetration time. Liquid surface tension was found to be inversely related to penetration time. However liquid surface tension was found have a minimal influence on the penetration time. Both these results correlate to those predicted by the Washburn Equation and results found in literature.

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