Infusion of Fluid into Powder Beds

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Abstract

When it is desirable that a product contain the maximum volume percentage of selected solids within a fluid matrix, which in turn gels or solidifies, it may be desirable to consider an infusion process. An infusion process permits achievement of a higher solids loading than can be obtained utilizing a pourable premixed slurry. However, infusing a viscous fluid into a bed of fine powder is a daunting task. The wide size distributions and non-uniform shapes frequently encountered with many powders make the classical equations for fluid flow through packed beds difficult or impossible to use. An alternate approach is proposed whereby the mechanism is characterized as flow through parallel capillary channels, rather than just as flow over the particle surfaces. A simple test system can be used to define the effective capillary size, from which the pressure and time required to infuse a fluid into powder mixtures can be predicted as a function of the viscosity of the fluid and the geometry of the vessel into which it is being pumped. The predictability is particularly important when the fluid is a polymerizable monomer with only a narrow operating window before it sets up, such as some potting compounds and energetic applications. Examples are given with various fluid/powder systems.

Introduction

The current art of manufacturing energetic products, such as propellants and explosives, usually involves mixing of the ingredients for long periods to achieve a uniform distribution of the solid ingredients in a binder matrix that is still pourable but eventually slowly polymerizes or gels into a solid mass. The slurry paste becomes very viscous because of the generally high loading of solids. The increase in viscosity caused by the solids loading frequently sets the maximum solids content that can be processed. The high viscosity of the mixture requires substantial torque to blend. The power to mix combined with long mixing times result in inefficient mixing and high power consumption, and can lead to potentially undesirable and dangerous heat buildup.

For example, mixing for the solid propellants such as used in the space shuttle program typically contain 12.6 % by volume aluminum powder as fuel (10 to 20 microns), 63.5 % ammonium perchlorate as oxidant, generally bimodal size distribution (about 20 microns and about 300 microns), and 23.9 % polymerizable mixture of prepolymer and plasticizer. Mixing the above formulation may be done in large planetary change-can mixers.

The recent ACE^{TM} wet manufacturing process described by Gogos et al [1] involves similar energetic products for the opposite end of the size spectra, namely long narrow metal tubes known as burster tubes. The ACE process achieves higher solids loading by replacing the slurry-mix-and-pour step by preconditioning the solids and dry filling the burster tube with the solid mixture, followed by infusing and curing of the binder fluid. The work described herein concerns the time and pressure required to achieve complete infusion before gelling occurs.

Pressure Drop through Packed Beds

Flow of fluids through packed beds has been well investigated by the Ergun [2] equation in the creeping flow regime.

$$\frac{\Delta p}{L}=\frac{p_1}{p}\left[150\frac{(1-\varepsilon)^2}{\varepsilon^3}\frac{\mu U_1}{d_{sv}}+1.75\frac{1-\varepsilon}{\varepsilon^3}\frac{\rho_{g1}U_1^2}{d_{sv}}\right]\approx 150\frac{(1-\varepsilon)^2}{\varepsilon^3}\frac{\mu U_1}{d_{sv}^2} \qquad (1)$$

Where, μ, viscosity of the fluid, E, fraction void, Δp, pressure drop across bed, d_{sv}, surface volume of the packing particles, U, superficial velocity, ρ, fluid density, L, bed depth.

Since the superficial flow velocity $U_1=\varepsilon\frac{dL}{dt}$, thus infusion time t can be rearranged as:

$$t=\left\{75\frac{(1-\varepsilon)^2}{\varepsilon^3}\frac{\mu}{\Delta p\times d_{sv}^2}\right\}L^2=kL^2 \qquad (2)$$

Appling the Ergun equation to predict the time *vs.* infusion length for packed beds of widely differing particle sizes and shapes is difficult. Figure 1, 2 and 3 show size distributions representive of two of the solids used in these tests.

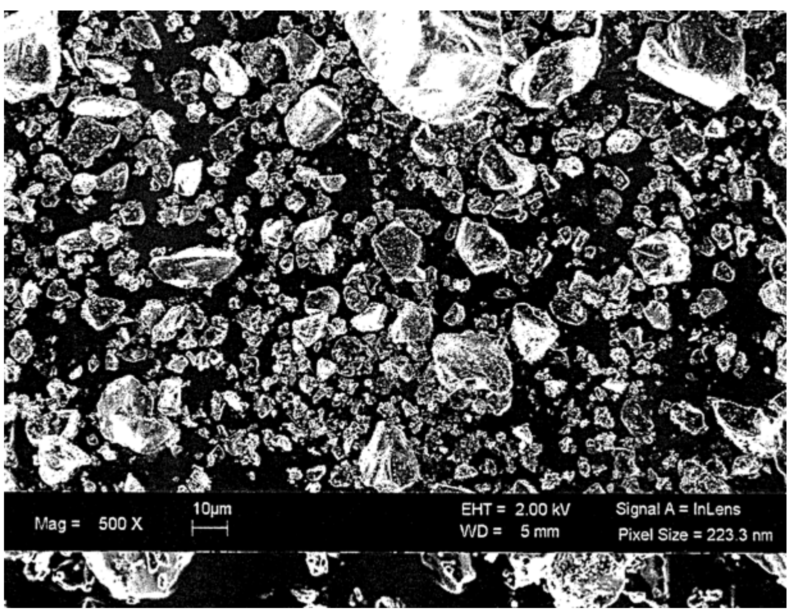

Figure 1 Photomicrograph of Dechlorane Plus 515 particles

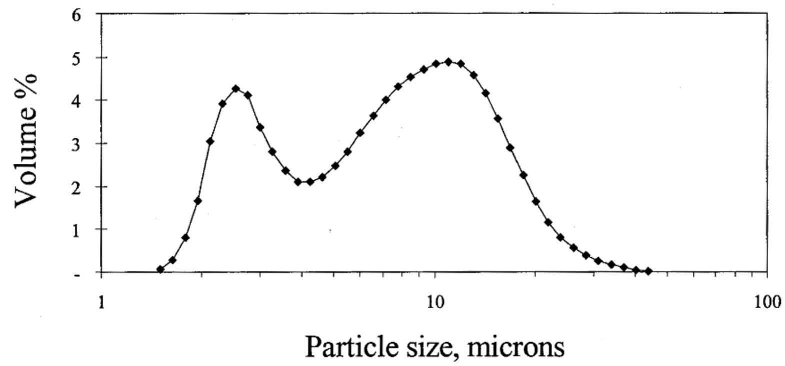

Figure 2 Particle size distribution of Dechlorane Powders

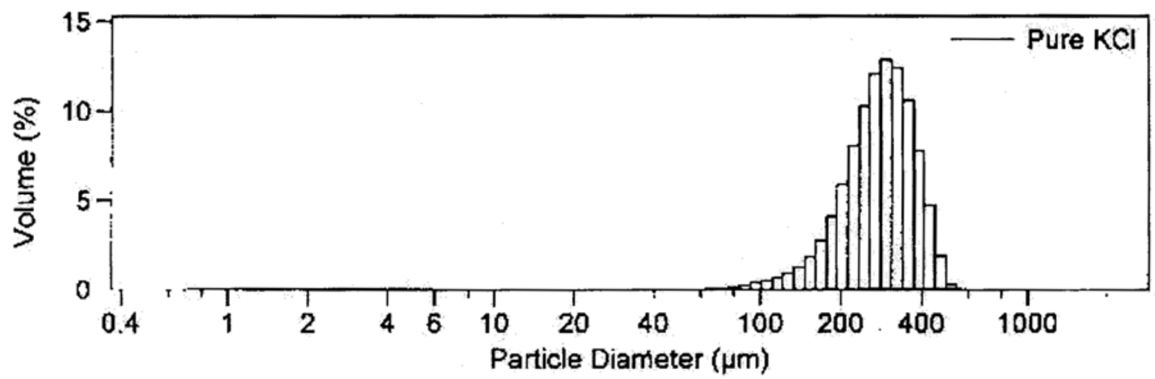

Figure 3 Particle size distribution of KCl crystals

Instead of trying to describe the flow pattern based on the sizes of the particles around which the binder fluid has to flow, we decided to simplify the model for any packed bed of powder by characterizing the flow as many parallel passages with an equivalent capillary diameter, Φ, which can be determined from the same Δp *vs.* L data. For a Newtonian fluid, the flow in a capillary channel follows the formula:

$$\frac{\Phi \Delta p}{4L} = \left(\frac{8V}{\Phi} \right) \mu \quad (3)$$

in which Φ is the effective capillary diameter, and V is the velocity of the fluid

$$V = \frac{dL}{dt} \quad (4)$$

where t is the time. Equation (3) would be further converted into:

$$dt = \frac{32 \mu L dL}{\Phi^2 \Delta p} \quad (5)$$

Integrating Equation (5) along the filled length L results in:

$$\Delta t = \frac{16\mu L^2}{\Phi^2 \Delta p} \quad (6)$$

Rearranging Equation (6), the effective capillary diameter Φ can be calculated.

$$\Phi = \left[\frac{16\mu L^2}{(\Delta t)(\Delta p)}\right]^{0.5} \quad (7)$$

Testing Procedure and Results

A test device was assembled (Figure 4) which could hold a metal burster tube or its polycarbonate equivalent. Tube dimensions are listed in Table 1, and test conditions in Table 2. With packed powder tubes, plots of time *vs.* the square of infusion-filled length in the plastic tubes are shown in Figures 5 to 11. The data indicate that the filling time is 3 to 10 times longer than that predicted by the Ergun equation. Table 1 also indicates the hypothetical capillary diameter Φ calculated from Equation (7). Figures 12 and 13 illustrate the time and length observations for both 100 and 300 microns glass beads and for both sizes of the plastic burster tube.

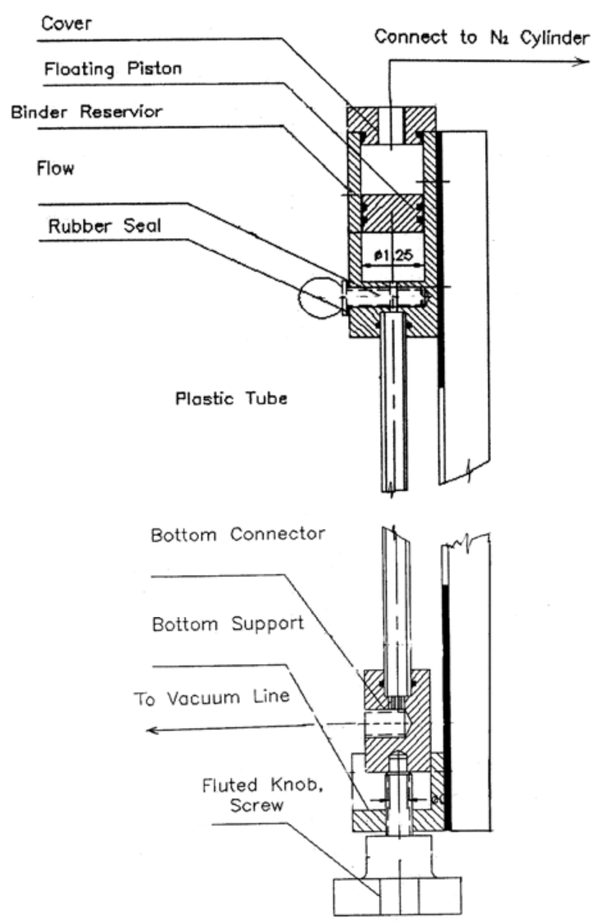

Figure 4 Infusion test device

Table 1 Burster Tube Dimensions

Designation	1 D (mm)	L/D
120-Aluminum	11.11	31.1
155-Steel	15.49	33.2
Plastic-Small	9.52	36.3
Plastic-Large	15.88	32.4

Table 2

Test	Tube Diam (mm)	Solids	%	Shape	Fluid	Viscosity (mPa·s)	Pressure (kPa)	Fraction Void	d_{sr} (mm)	Φ (mm)	Figure
1	9.5	KCl	100	Crystal	SC-B	62	80	0.37	280	30	5
2	9.5	KCl	100	Crystal	SC-B	62	827	0.37	280	29	6
3	9.5	KCl Dechlorane	80 20	Cube Irreg	MMA	0.55	827	0.27	280 23	2.5	7
4	9.5	KCl Dechlorane	80 20	Cube Irreg	SC-A+B	47	1184	0.32	280 23	4.5	8
5	9.5	KCl	100	Cube	SC-A+B	47	1255	0.36	220	49	9
6	9.5	KCl	100	Cube	SC-A+B	47	1360	0.37	220	55	10
7	15.9	KCl	100	Cube	SC-A+B	47	1017	0.37	220	55	11
8	9.5	Glass	100	Beads	Lube	35	680	0.36	100	15	12
9	9.5	Glass	100	Beads	Lube	35	680	0.36	300	52	12
10	15.9	Glass	100	Beads	Lube	35	1296	0.36	100	16	13
11	15.9	Glass	100	Beads	Lube	35	1316	0.36	300	53	13

*Notes: Smooth-cast 327 consists of two components, A and B to form polyurethane. Lube is a non-reactive lubricant.

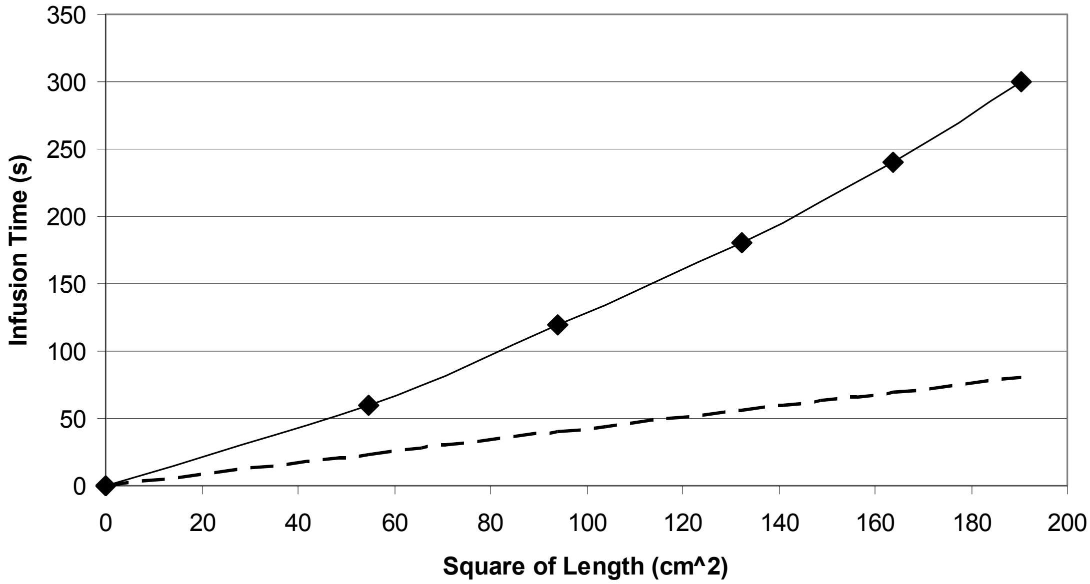

Figure 5 Infusion length *vs.* time for Test 1 with KCl crystals and Smooth-cast B fluid at 80 kPa Δp (-■- experimental, --- predicted by the Ergun equation)

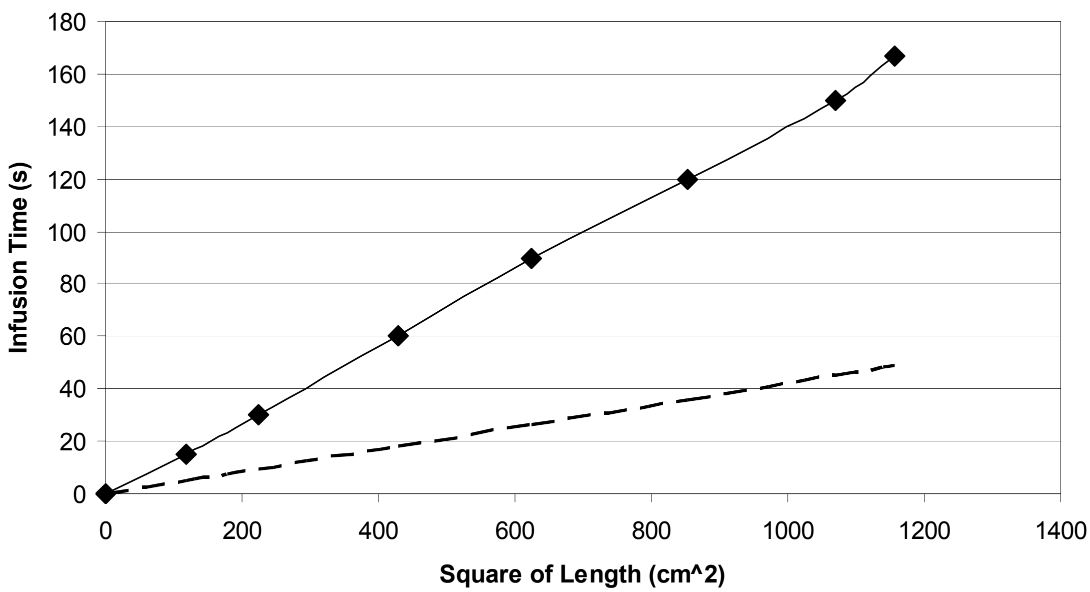

Figure 6 Infusion length *vs.* time for Test 2 with Smooth-cast B fluid at 827 kPa Δp (-■- experimental, --- predicted by the Ergun equation)

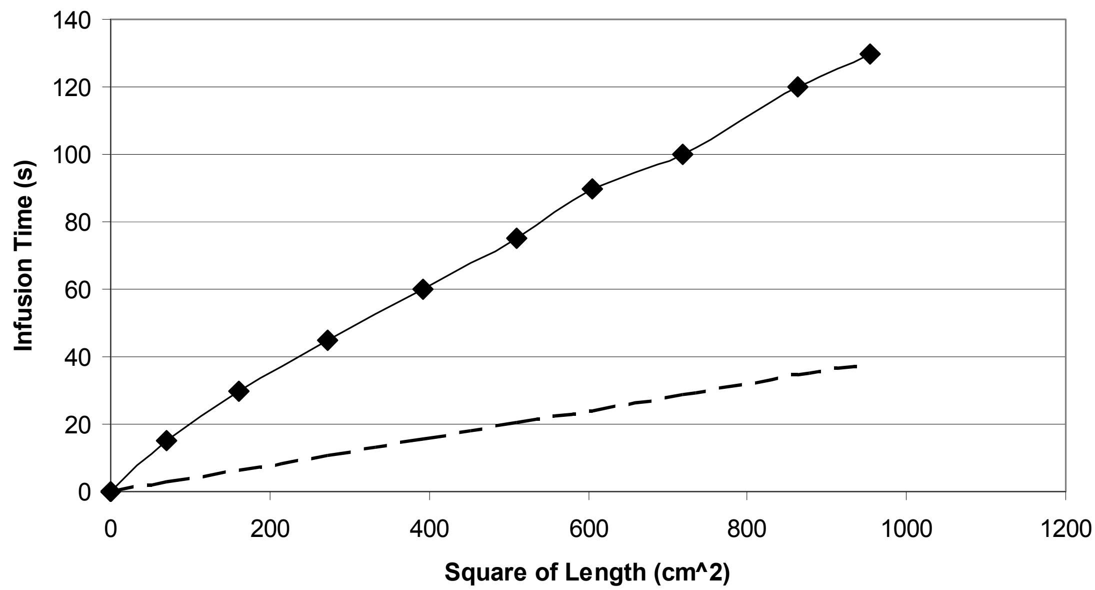

Figure 7 Infusion length *vs*. time for Test 3 for 80 % KCl and 20 % dechlorane with methyl methacrylate fluid at 827 kPa Δp (-■- experimental, --- predicted by the Ergun equation)

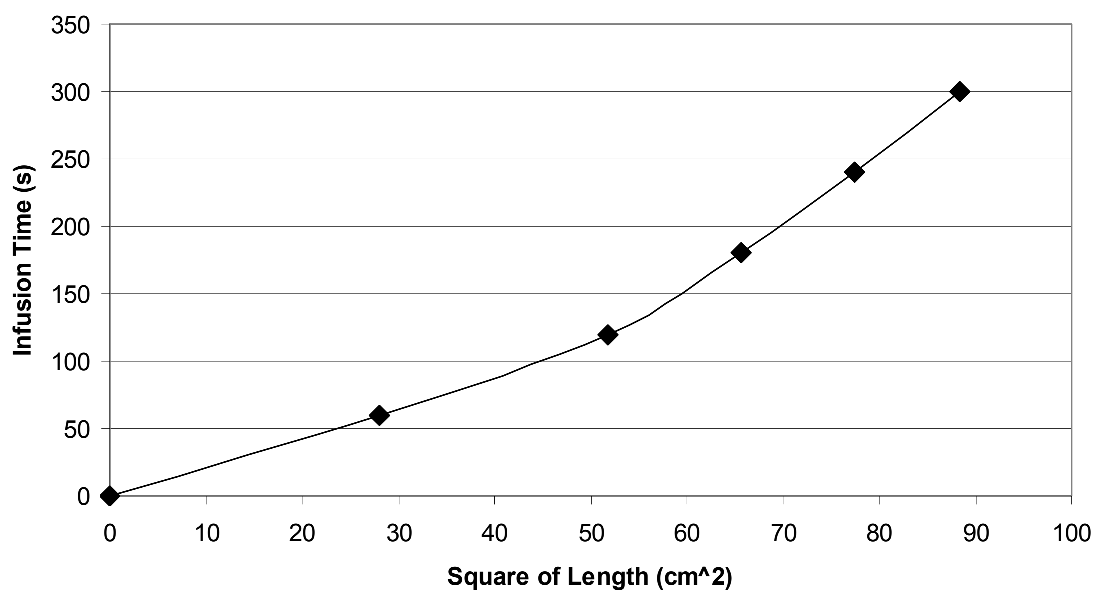

Figure 8 Infusion length *vs*. time for Test 4 for 80 % KCl, 20 % dechlorane with reacting Smooth-cast 327 A and B components

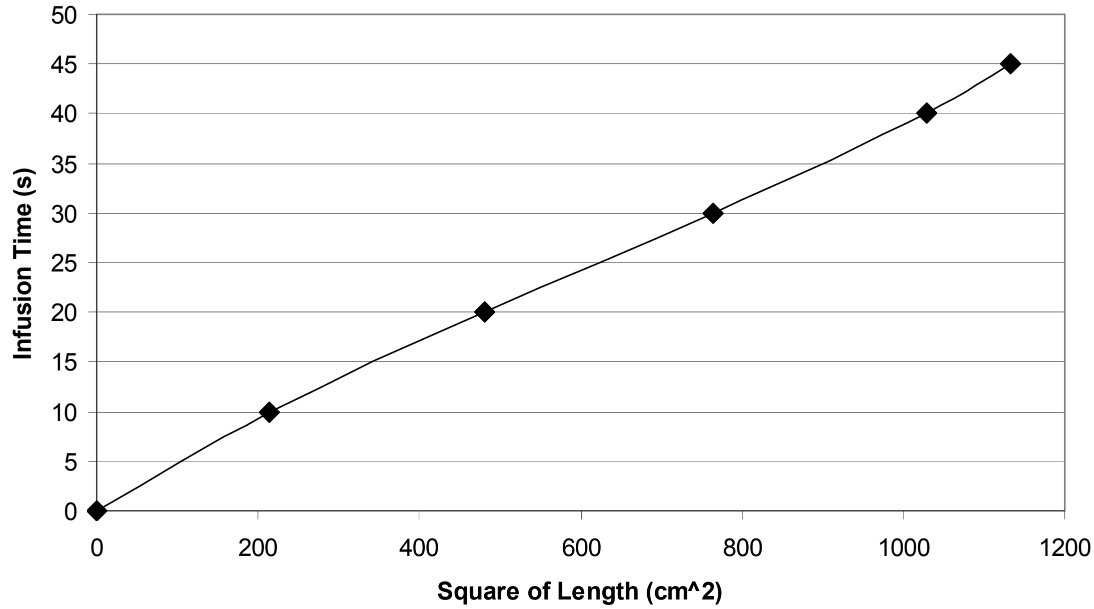

Figure 9 Infusion length *vs*. time for Test 5 with KCl crystals and Smooth-cast 327 binders

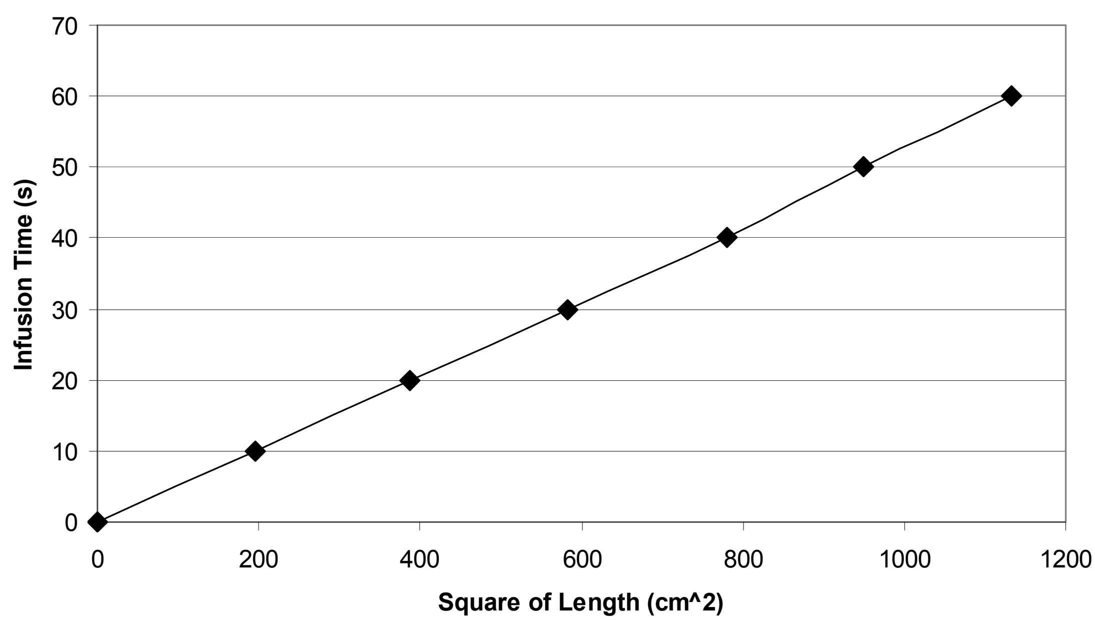

Figure 10 Infusion length *vs*. time for Test 6 for Smooth-cast 327 in 120 mm burster tube

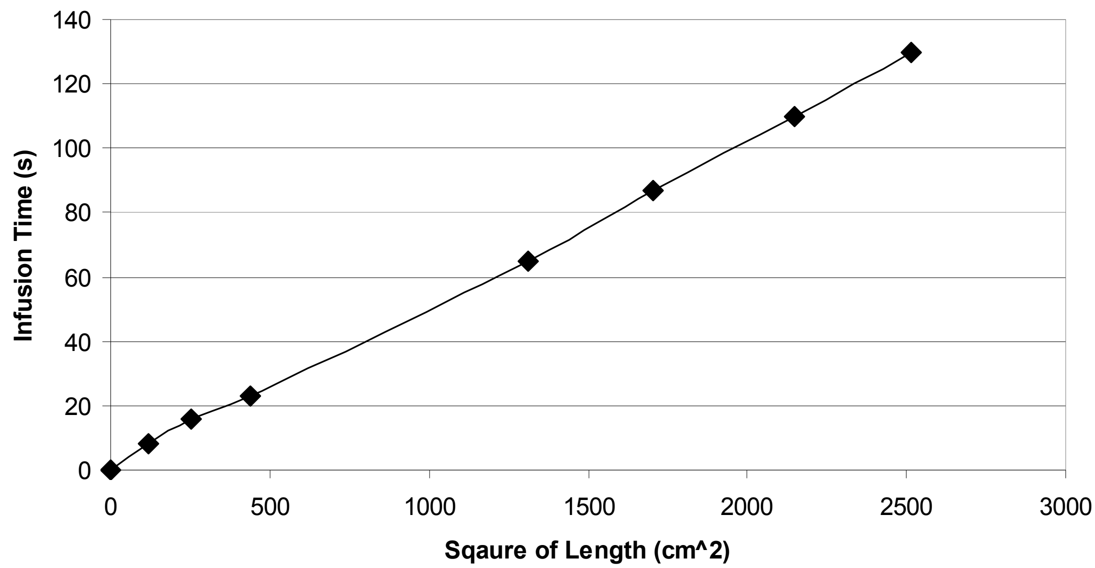

Figure 11 Infusion length *vs.* time for Test 7 for Smooth-cast 327 in 155 mm burster tube

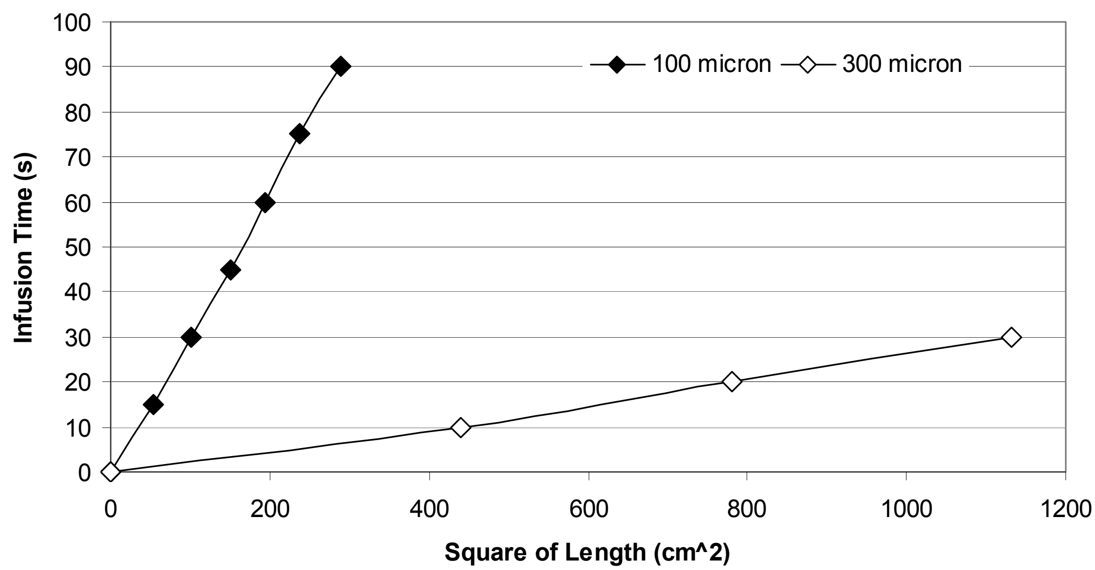

Figure 12 Infusion length *vs.* time for Test 8 with lubricant flow in 120 mm burster tube filled with glass beads

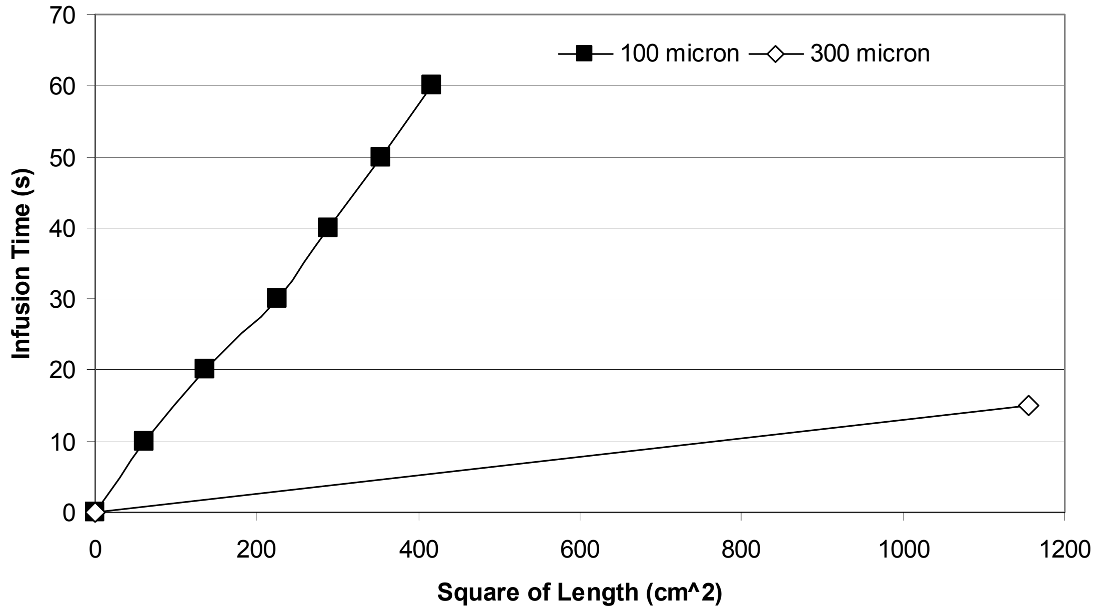

Figure 13 Infusion length *vs.* time for Test 10 lubricant flow through a bed of 100 micron glass beads in a 15.9 mm plastic tube

Conclusion

A simple model based on a simple test device and procedure has been developed to determine pressure (Δp) - time (t) - penetration depth (L) for the infusion of a fluid into mixed beds of powders. From the observed Δp-t-ΔL data, a hypothetical equivalent capillary Φ is calculated:

$$\Phi = \left[\frac{16 \mu L^2}{(\Delta t)(\Delta p)} \right]^{0.5}$$

Testing can be done at room temperature with a known viscosity liquid, and extrapolated for equivalent packed beds of mixed powders for other viscosities and processing conditions.

The hypothetical capillary diameter Φ is about $0.15d$ to $0.18d$ for a bed of uniform diameter (d) spheres. For beds of non uniform powders, Φ is sensitive to sizes and shapes of the particulates, and may range from about $0.1d_{sv}$ to $0.3d_{sv}$, depending on how the average diameter is defined.

Acknowledgement

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References

1. Costas G. Gogos, Ming-Wan Young, Linjie Zhu, Chen Wan, Raj Dave, David Todd "A Novel Net Shape Manufacturing Process for Concentrated Polymer-Particulate Composite Products", SPE ANTEC, Milwaukee, May 6 [2008]

2. Ergun, S., Chem Eng Prog, 48, 89 [1952]