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Measurement of particle concentration in a Wurster coater draft tube using light attenuation

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ABSTRACT

Particle concentration in a coating zone of fluid bed coater is important to ensure appropriate coating uniformity and process yield. A transmissive optical setup at the top of the Wurster draft tube was used in a lab scale coater. Measured transmittances were converted to particle chord average volume fraction distributions using results of Monte Carlo calculations. The ranges of measured transmittances were from 0.002 to 0.285 and the range of particle chord average volume fraction was from 1.36% to 5.44%. The effect of gap between the draft tube and distribution plate, fluidizing air flow rate, particle size and load was studied. Comparisons of particle chord average volume fraction results with results of global particle volumetric fraction in the draft tube and particle cordal fraction obtained from CFD simulations are shown. In order to study the dynamics of the system as a function of different process parameters frequency analysis of transmittance signals was performed. Four distinct frequency responses were identified. Findings indicate presented local light attenuation method as suitable for assessing particle chord average volume fraction at the top of Wurster draft tube and with a potential for in-line method development.

1. Introduction

Fluid bed particle coating is a common process in pharmaceutical (Dixit and Puthli, 2009), food (Werner et al., 2007a, b), agricultural (Nienow, 1995) and other industries. The reasons for coating are taste masking, cosmetic, to provide protection or to control the release of the active ingredient or the process is used to apply the active ingredient (Turton, 2008). Two main parameters of coating process are coating thickness uniformity and the yield of the process, which are a result of many design, process and formulation variables (Cheng and Turton, 2000a). Particle volume fraction in the coating zone is a transient parameter that depends on various process parameters, most characteristically on air volume flow rate and draft tube gap and has a big influence on both coating uniformity and process yield (Cheng and Turton, 2000b). This study is focused on assessment of the particle volume fraction downstream the coating zone. Particle volume fraction affects the variability of the amount of deposited mass per pass through the spraying zone, which besides the circulation time and its variability in turn affects final variability of total per-particle mass deposited during the coating process (Turton, 2008). Variability of circulation times have been studied by tracer particles such as magnetic tracer (Cheng and Turton, 2000a) or radioactive labeled particle using PEPT (Chang et al., 2013; Depypere et al., 2009). The mean circulation time can also be expressed out of solids mass flow rate, which can be measured using various techniques, e.g. using a imaging system (Chan et al., 2006) or piezo impact sensor (Luštrik et al., 2013). A number of measuring techniques can be used to measure particle volume fraction of particles, such as tomography (e.g. X-ray and γ-ray tomography) (Van Ommen and Mudde, 2008), capacitance probes (Delmon et al., 1996), pressure measurements (Magnusson et al., 2005) and optical techniques, which are

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Nomenclature

h	denotes the gap height
k	proportionality factor
d	diameter of particles
n	number density of particles
I	intensity of transmitted beam
I_0	intensity of incident beam
L	light path length
D	draft tube diameter
H	the draft tube height
L_{chordal}	chordal length in simulations
$L_{\text{gr,I}}$	chordal line length in particular computational cell
MC	Monte Carlo transmittance probability
T	transmittance
VF	predicted particle chord average volume fraction distribution
V_{cho}	chordal volume
$V_{\text{gr,cho}}$	volume of all particles that are crossing the light beam
V_{gr}	granular volume of particles in the draft tube
V_{noz}	spraying nozzle volume
V_{v}	draft tube volume
$\varepsilon_{\text{chordal}}$	particle chordal fraction along the L_{chordal}
$\varepsilon_{\text{gr,i}}$	granular phase volume fraction in particular computational cell
ε_{vol}	particle draft tube volume fraction
$\varepsilon_{\text{vol,cho}}$	particle chord average volume fraction
σ	represents all constant terms in Beer–Lambert equation

divided into transmissive and reflective (Van Ommen and Mudde, 2008). Fiber based optical probes have been used by various researchers mostly in reflective mode (Bergougnoux et al., 1999; Magnusson et al., 2005; Seachman et al., 2006; Xu et al., 2013). Such measurements provide useful information by themselves or can be used as validation data in conjunction with numerical simulations such as discrete element method (Kuipers et al., 1992; Link et al., 2009). Others have employed imaging analysis to obtain particle volume fraction in fluid bed systems (Casleton et al., 2010; Saadevandi and Turton, 1998; Wang et al., 2010).

Transmission light measurements were performed to study the dynamics of a simple fluidized bed under different process conditions (Shahbazali et al., 2009). Transmissive mode also known as optical beam attenuation for particle systems is described in form of Beer–Lambert equation (Crowe et al., 1998):

$$T = \frac{I}{I_0} = e^{-k\frac{\pi}{4}d^2nL} \tag{1}$$

where T is transmittance, I is the intensity of transmitted beam, I_0 the intensity of incident beam, k proportionality factor, d diameter of particles, n number density of particles and L the light path length.

Eq. (1) can be expressed in terms of particle chord average volume fraction $\varepsilon_{\text{vol,cho}}$, assuming uniform particle size and spherical shape:

$$T = e^{-k\frac{3}{2}\frac{\varepsilon_{\text{vol,cho}}L}{d}} \tag{2}$$

which can for a fixed light path length be further simplified into

$$T = e^{-\frac{\sigma\varepsilon_{\text{vol,cho}}}{d}} \tag{3}$$

where σ represents all the constants ($3kL/2$) in Eq. (2).

Monte Carlo simulations can be used for predicting particle size effect in reflective type optical fiber measurements and the results can be then used in determination of particle volume fraction (Bellino et al., 2001; Tran et al., 2006). Similarly Monte Carlo simulations were used in predication of coating uniformity in fluid bed coaters (KuShaari et al., 2006).

Our aim was to use a simple light transmission setup to measure light attenuation at the top of the Wurster draft tube in a lab scale coater. Light attenuation itself would provide a direct information about inter particle sheltering with its implications to the coating process outcome and additionally light transmittance data would be used for calculation of particle chord average volume fraction and its time characteristics. Verification of presented measurement method would present a good basis for development of an in-line method.

2. Materials and methods

2.1. *Materials*

Neutral microcrystalline pellets (Cellets® 700 and 1000, Harke Pharma GmbH, Germany), which are commonly coated with active pharmaceutical ingredient were used in all measurements. Prior to their use they were sieved into narrower distribution of particles with size fractions of 600–710 μm, 900–1000 μm and 1120–1250 μm. Sieving was performed using a set of laboratory test sieves (Endecotts Ltd, England) and a sieve shaker (AS 200, Retsch, Germany). The density of the pellets determined by helium pycnometry was 1.46 g/cm^3 for all fractions used.

2.2. *Equipment and procedure*

Bottom spray fluid bed coater with Wurster insert (GPCG-1, Glatt GmbH, Germany) was used in the experiments. Light attenuation experiments were performed for the five combinations of particle sizes and bed loads (Table 1). For each experiment light attenuation was measured at all combinations of two fluidizing air flow rates (105 and 130 m^3/h) and three gaps between the draft tube and the distribution plate (10, 20 and 25 mm) yielding a total of 30 measurements. The inlet temperature was set to 30 °C and water was sprayed at 4 g/min to simulate coating conditions. The atomizing air pressure was set to 2 bar.

Table 1 – Experimental plan.

Bed load (g)	Particle size (μm)
500	900–1000
1000	600–710
1000	900–1000
1000	1120–1250
1500	900–1000

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