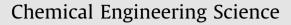
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Experiment and simulation of dry particle coating

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HIGHLIGHTS

- ► Study of the influence of operating conditions on dry coating process.
- ▶ Particle properties changed with rotational speed and operating time.
- ► The DEM simulation of the mixer shows convective movements of particles.
- ▶ The calculated rotational distance increases with an increase of rotational speed.
- ► The simulation results agree with experimental ones qualitatively.

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ABSTRACT

The objective of this study is to get a better understanding of dry powder coating process using experiments and numerical methods. Materials chosen as host particles are SUGLETS® (granules mainly consisting of sugar) and as invited particles, magnesium stearate (MgSt). These two materials are introduced into a high shear mixer (Cyclomix). Operations were performed at various mixing time and rotation speed of the Cyclomix. The surface morphology analysis has confirmed that Suglet particles are coated with MgSt. The product properties such as flowability, wettability and particle size distributions were also characterized. The particle motion in the Cyclomix has been simulated by using Discrete Element Method (DEM). Both number of collisions and the collision force frequency are calculated. The simulation shows an increase of the collision number with the rotational speeds. This result indicates that choosing higher rotation speeds should be better for the dry coating process as long as the particles are not broken down.

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1. Introduction

Dry particle coating is a method used to modify the properties of powders by attaching the fine particles (guest particles) onto the surface of core particles (host particles) by mechanical forces. As such neither aqua binder nor any organic solvents are required in the process. It is a simple process with low environmental impact and will receive considerable industrial attention. Even though a lot of experimental work on dry particle coating has been reported in the literature (Alonso et al., 1989; Honda et al., 1994; Watano et al., 2000; Ouabbas et al., 2009a, Lefevre et al., 2010; Lefevre et al., 2011), the technique is not yet in commercial use. This is because the experiments are still in the trial and error state. In other words, it is difficult to predict the optimum operating conditions for commercial use and/or to scale up the

process for industrial application. Few researchers have studied the theoretical approaches for dry coating mechanisms with the aim of optimizing and improving the understanding of the process. For instance, Mei et al. (1997) developed an extended Johnson-Kendall-Roberts (JKR) particle model to include the effect of particle coating on the force-displacement relationship due to surface energy and elastic deformation. The method for determining the optimum operating conditions of equipment on dry coating processes based on the energy requirement for immobilizing the guest particles on the surface of host particles was also established (Iwasaki et al., 2002). The theoretical modeling of dry coating is a very important requirement for the optimization of the process and the design of new, more efficient apparatus geometries. However, this area of research is found to be less comprehensive. Hence, the objective of this study is to better understand dry powder coating by experimental and numerical methods. Since dry coating is generally achieved by mechanical forces, the particle motion would be the most important factor in determining the optimum operating conditions.

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Discrete Element Method (DEM), proposed by Cundall and Strack (1979), is one of the most popular techniques for simulating and analyzing the solid particle behavior and has been successfully applied in many fields (Kano et al., 1997; Matchett et al., 2000; Cleary and Sawley, 2002). Thus DEM will be applied in this research to simulate the motion of the particles inside the coating apparatus. This research aims to reveal and hence describe the effect of operating conditions such as mixer rotation speed on particle coating, including a numerical approach.

2. Experiment

2.1. Materials

For the powders, Suglets and magnesium stearate (MgSt) were chosen as the host and guest particles, respectively. The Suglets were provided by NP Pharm Company. They are elliptical particles with a majority and minority composition of sucrose and maize starch, respectively. They have hydrophilic characteristics and are mainly used as an excipient in capsule and tablet formulation, particularly in multi-particulate systems. They can form cores upon which Active Pharmaceutical Ingredients (APIs) are coated for controlled or sustained release, drug delivery technologies. The use of such sugar spheres in multi-particulate drug delivery system is an area of increasing interest in the pharmaceutical industry. This is largely due to the clinical and formulation advantages sugar spheres have over single-unit dosage forms, including the ease of proceeding in modified release applications. An especially useful trait is that Suglets are able to withstand the process of drug lavering (coating) an accurate amount of the API in the products. Fig. 1a shows a SEM image of Suglet particles. They are relatively elliptic shape and their surface seems to be angulated. The magnesium stearate (MgSt) guest particles are a fine, white, greasy, cohesive and hydrophobic powder widely used in pharmaceutical formulations as a lubricant. The shape of the MgSt is relatively random, including needle-like and plate-like configurations (Fig. 1b). Fig. 2 shows the particle size distributions of Suglets and MgSt obtained with a Malvern dry feed system (Mastersizer 2000). The volume distribution of Suglets shows one narrow population with median diameter (D50) about 250 µm. However, a wide size distribution is observed for MgSt. The size of MgSt particles varies from less than $1 \mu m$ -20 μm . The D50 for MgSt is about $4 \,\mu m$. These properties of the samples are summarized in Table 1.

2.2. Coating process

A 1 L Cyclomix has been chosen to perform a dry coating in this study. This apparatus is defined as a high shear mixer/granulator, manufactured by Hosokawa Micron B.V. As seen from Fig. 3, the device consists of a conical vessel with an axial impeller in the centre equipped with four sets of specially designed blades. It applies high mechanical impact and shearing forces on the particles in order to break the guest agglomerates and coat them on the host particles. This device has previously been used successfully for dry particle coating experiments (Ouabbas et al., 2009b). The mass fraction of guest particles to host particles required in a coating experiment is calculated using a simplified model in order to guide the experimental work. This model is based on the two following assumptions permitting a theoretical calculation: the guest and host particles have uniform sizes and are spherically shaped (even though in this case, the SEM images in Fig. 1 shows that they are not): and a 100% monolaver surface coverage of guest onto the host particles surface is achieved by the process. This mass ratio of guest to host particles, w can hence be expressed using the following equations (Thomas et al., 2009):

$$w = \frac{4C_{2D}(k_H + 1)^2}{4C_{2D}(k_H + 1)^2 + \frac{\rho_H}{\rho_I}k_H^3}$$
(1)

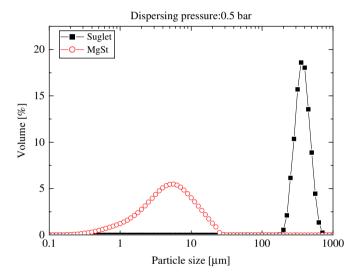


Fig. 2. Particle size distributions of host (Suglets) and guest (MgSt) particles.

Table 1

Properties of Suglets and magnesium stearate particles.

Particles	D ₅₀ (μm)	Density (kg/m ³)	Water interaction
Suglets	250	1580	Hydrophilic
MgSt	4	1140	Hydrophobic

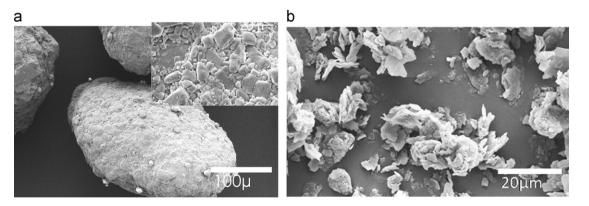


Fig. 1. SEM images of Suglets (a) and MgSt (b).

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