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Surface modification to improve powder bulk behavior under humid conditions

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ABSTRACT

Humid environment often creates various problems in fine powder processing and frequently poses a substantial threat to the final product quality. The present study focuses on improvement of bulk behavior of fine powder under humid environment using surface modification. Corn starch powder was surface modified using Aerosil R972 grade of hydrophobic nano-silica through dry coating. It was found that flow and packing properties of uncoated powder deteriorated at elevated relative humidity (RH) conditions with prominent changes observed above 60% RH. Also, at low RH conditions (30%), increased interparticle frictional forces owing to the diminished lubricating moisture layer resulted in decreased flow of uncoated powder. In contrast, surface-modified powder exhibited resistance to such detrimental effects of moisture loss and gain at low and high RH respectively. The present study showed that surface modification of powders helps to reduce the interparticle cohesion in fine powders and prevents the detrimental effects of humidity on its bulk behavior.

1. Introduction

Fine powder handling and processing is a complex operation across industries, including pharmaceutical industry. Manufacturing of pharmaceutical solid dosage forms involves handling of fine active pharmaceutical ingredients (API) and excipient powders. The bulk behavior of these powders is governed by multiple factors such as particle size and its distribution, shape, surface area, density, surface roughness as well as by extrinsic parameters like humidity, temperature, static electricity etc. [1]. For dry powders, existence of strong interparticle force (mainly van der Waals) among fine particles often results in number of processing and manufacturing problems with respect to their flowability, packing, dispersibility and compressibility etc. In fact, these properties are integral part of pre-formulation and formulation studies during the pharmaceutical product development. Poor or variable powder flow and packing can result in various processing problems in pharmaceutical industry such as incomplete die fill resulting in weight variation and content uniformity problems during tabletting and capsule filling operations [2,3], dispersibility of a dose from dry powder inhalers (DPI's) [4], and blend uniformity issues during mixing [5].

Atmospheric humidity is an added complexity in powder handling. Moisture present at high humidity conditions can affect the physical and chemical properties of starting materials as well as the final product. For instance, at high humidity conditions, capillary forces become more significant and can control the flow behavior of powder [6,7]. Amidon and Houghton [8] observed a decrease in the flow as well as in bulk and tapped densities of microcrystalline cellulose with an increase in moisture content, especially at moisture contents above 5%. Humidity can also influence the van der Waals forces. Coelho and Harnby [9] found that the van der Waals forces are strengthened by adsorbed moisture because the added thickness of moisture layer decreases interparticle distance by increasing the apparent diameter of the particle. In addition to this, sometimes it is the moisture content of an excipient which can pose a considerable threat to the performance and quality of the final product [10]. Bravo-Osuna et al. observed that the blend with methyl methacrylate co-polymers exposed at high humidity (75% RH) led to decrease in flow, required higher compaction pressures for tabletting and also resulted in increased drug release [11,12]. Further, increased moisture may cause stability and incompatibility problems with moisture sensitive APIs or excipients in solid dosage forms [13]. Majority of the APIs or excipients employed in manufacturing gain or lose moisture when exposed to a particular humidity condition [14,15]. The resultant effect may be either an increase in the interparticle capillary force at high humidity conditions or increase in other forces such as friction, van der Waals and electrostatic at low humidity [6,16], all of which could create complications in powder processing or handling.

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Thus, protection of fine powder from humid or moist conditions has always been an area of prime importance in pharmaceutical industry and in other industries. Various approaches such as moisture-resistant polymer coatings, use of moisture-resistant packing or simply avoiding the use of hygroscopic materials in the product manufacturing can be used as escape routes to avoid or minimize the deleterious effects of moisture or humidity on final product quality. However, these protection approaches have numerous limitations. Moreover, none of them provides a suitable solution to the problem of moisture sensitivity of a material during manufacturing stages.

Use of glidants such as fumed silica is a common practice in pharmaceutical industries for improving powder flow. However, owing to their nano-size and aggregation tendencies, proper mixing in the powder blend is critical and often very difficult. Pfeffer (2001) proposed surface modification technique for addition of these glidants in a particular fashion so as to get their maximum effect. Surface modification through dry coating using nano-particle (nano-coating) is one such technique [17] which has been explored for numerous applications ranging from powder flow improvement [18–23] to improvement of dissolution of poor water soluble drug [24–26]. Mujumdar et al. (2004) studied the effect of dry coating using carnauba wax and hydrophobic fumed silica to produce humidity-resistant magnesium powder. However, the study was exclusively limited to the moisture sorption and its chemical interaction with the particles at 100% RH [27]. Powder bulk behavior, which is an important aspect of powder handling, is a special concern under varying humid environment. This requires special attention and needs to be addressed properly.

The present study is aimed to employ the surface modification technique with hydrophobic nano-silica as a tool to create a humidity-resistant powder system. In this study, flow and packing properties of the uncoated and nano-silica coated corn starch powders exposed to different relative humidity (RH) conditions were examined. Packing and flow behavior were analyzed using FT4 powder rheometer.

2. Experimental

2.1. Materials

Corn starch (Suru Pharma, India) was used as a host particle, and a hydrophobic grade of colloidal silicon di-oxide (Aerosil R972 Pharma) with mean primary particle size of about 16 nm and obtained as a gift sample from Evonik/Degussa Industries, USA was selected as guest particle for surface coating.

2.2. Dry coating process

Dry coating of corn starch powder was performed with hydrophobic fumed silica (Aerosil R972) using Co-mill (Prism Pharma Machinery, India) following a method similar to the one described in literature [28,29]. A guest particle loading of 0.5% w/w of the total blend was used for the nano-coating procedure. The coating process was performed at room conditions of 45 ± 5% RH and 25 ± 2 °C.

2.3. Particle size analysis

It is important to check that high shear mixing process used for dry coating of guest over host does not alter the original particle size either by particle size reduction or by particle aggregation. Laser diffraction particle size analyzer (Cilas, Model 1190) was used for the determination of particle size of dry coated and uncoated starch powders using dry analysis mode. Approximately 1 g sample was taken for analysis, and compressed air supply at 1000 milibar (mb) pressure was used as the dispersing medium to obtain the particle size distribution.

2.4. Scanning electron microscopy (SEM)

The surface morphology as well as the efficiency of dry coating process for starch samples was examined using Scanning Electron Microscope (SEM) (LEO s-440i, Cambridge, UK) at a working distance (WD) of 12-16 mm and a voltage of 10 kV.

2.5. Moisture sorption studies

The uncoated and coated starch powder samples were uniformly spread to a thin layer in a stainless steel tray. Samples were conditioned in a humidity chamber (HMG India, India) to equilibrate at five different humidity conditions of 30%, 45%, 60%, 75% and 90% RH in an increasing order and at constant temperature of 25 °C for a period of 24 h. Percent loss on drying (LOD) was determined by keeping the samples in hot air oven (Equitron 7053-250, India) at 130 ° C for 90 min [30]. Also, visual observations of the uncoated and coated samples exposed to different humidity levels were noted.

2.6. Bulk and tapped densities

The poured bulk density (PBD) of uncoated and coated starch powders was determined by gently filling the powder sample in a 100 ml glass cylinder by keeping the cylinder in a slightly inclined position and pouring the powder into it so as to avoid compaction of the powder during free fall. The poured volume and mass of powder were recorded. The tap density of powder samples were determined following USP type I test with a tap density test apparatus (Veego Instruments Corporation, TAP/MATIC-II model, India). A total of 1250 taps (500 initial taps followed by final 750 taps) were applied to the cylinder at a rate of 300 taps per minute. The procedure was continued till a tapped volume difference of less than 2% was obtained between two consecutive tapping sequences as per USP specification [31].

2.7. Characterization using powder rheometer

The uncoated and nano-coated (surface modified) starch powders conditioned at different relative humidity (RH) conditions were characterized for their flow and packing properties using FT4 Powder Rheometer (Freeman Technology Ltd., Worcestershire, UK). Two tests viz. shear and compressibility tests were performed to characterize them for their failure properties and packing characteristics respectively.

2.7.1. Shear test

The shear test assembly of FT4 powder Rheometer consists of a 25 mm × 10 ml split cylindrical glass vessel, a vented piston for pre-consolidation and a shear cell. In the first step, sample powder was taken in the vessel and was pre-conditioned using a specially designed blade. In the second step, the sample was pre-consolidated at 3 kPa normal load using the vented piston. Finally, the pre-consolidated powder sample was sheared using the shear cell to obtain yield loci. Two Mohr circles were obtained from the best fit line of loci extrapolated to the y-axis using the FT4 analysis software providing the values of unconfined yield strength (UYS) and major principal stress (MPS) for the sample powders. The flow function co-efficient (ff_c), which is the ratio of MPS to UYS was then determined. The better the flowability of a powder and lesser the UYS, the more is the ff_c. Schulze provided the classification of powder flow behavior based on their flow function values. According to this classification, $ff_c < 1$, not flowing; $1 < ff_c < 2$, very cohesive; $2 < ff_c < 4$, cohesive; $4 < ff_c < 10$, easy flowing; and $ff_c > 10$ indicates free-flowing behavior [32].

2.7.2. Compressibility test

Compressibility test of powder sample using FT4 Powder Rheometer is a relatively simple test to perform; however, it can provide a