Advanced Powder Technology 28 (2017) 2875–2885

Contents lists available at ScienceDirect

Advanced Powder Technology

journal homepage: www.elsevier.com/locate/apt

Original Research Paper

Production of composite particles using an innovative continuous dry coating process derived from extrusion

Fanny Cavaillès, Romain Sescousse, Alain Chamayou, Laurence Galet *

Centre RAPSODEE, UMR CNRS 5302, Université de Toulouse, Ecole des Mines d'Albi-Carmaux, Campus Jarlard, 81013 Albi CT Cedex 09, France

ARTICLE INFO

Article history:
Received 9 March 2016
Received in revised form 18 July 2017
Accepted 23 August 2017
Available online 2 September 2017

Keywords:
Dry particle coating
Continuous process
Twin-screw extrusion
Wettability
Talc

ABSTRACT

Dry particle coating is used to modify surface properties and monitor the end use properties of powders. These processes are mainly running in batch mode. In certain cases, continuous processes may present interest for specific applications (limitation of investments, stability, versatility…). In this study, the feasibility of dry coating particles by an innovative way derived from the well-known extrusion process was investigated. Adhesion between host and guest particles is induced by mechanical shear stress during processing. A preliminary parametric study on microcrystalline cellulose particles as host particles was carried out in order to determine the operating condition range. Then, coating was successfully performed using talc and a microcrystalline cellulose system, which demonstrates the feasibility of this novel process and led to different morphologies according to the operating conditions.

1. Introduction

Numerous industrial areas (Pharmacy, Biology, Food, Fertilizers, Minerals, Materials, Inks industries…) require to transform or to produce powdered materials. In such activities, coating processes (dry particle coating in particular) are used for monitoring several end-use properties of the obtained composite particles [1].

Dry particle coating processes employ coating agents in particulate solid form. These particles, generally called "guest particles" (0.1–50 μm size) are located onto the surface of "host particles" (1–500 μm size) by means of mechanical actions provided by the process. In a first step relatively stable structures (ordered mixing) are obtained due to the Van der Waals or electrostatic interactions. In a second step, the mechanical forces act, according to the physical/mechanical properties of the involved materials, to increase the surface adhesion modifying the composite particles. This forms new structures varying from discrete coating – bound or embedded guest particles – to continuous film coating [1] – mono or multilayer coating [2]. The main interest of such processes compared to wet and melt coating is to avoid the use of solvents and subsequent heating and drying steps. These points rank dry coating as an economically efficient and environmentally friendly process: energy costs are reduced as well as organic solvent emission and toxicity risks linked to residual solvent or binder. Moreover, the risk of product degradation due to heating of materials is avoided as well.

Many examples of dry coating applications are mentioned in the literature concerning food, cosmetic or chemical industries: for example controlled release, flame retardant, protection of materials against humidity, oxidation or oil absorbed, improvement of dispersibility, taste, and odour. Owing to their wide interests, composite particles find their place also in the pharmaceutical industry. For instance they can be used to mask the flavour of active pharmaceutical ingredient (API), to produce dry powder inhaler carrier and to improve their properties, to control the surface properties or end uses properties such as flowability [3], air or liquid dispersibility [4], bulk density [5], wettability [3,6,7].

In the current literature, different dry coating processes, generally based on devices diverted from their traditional use, are mentioned [1,8]. For example, Hybridizer, Mechanofusion and Mechanomill processes are some advances of grinding machines or Cyclomix® [9–11], are used for dry particle coating. New mixers are used for coating, such as the resonant acoustic mixer (RAM) in which an acoustic field spreads to provide the necessary energy for coating [12]. During the past five years, several researchers have realized processes that can simultaneously achieve multiple functions, such as milling and coating. Fluid energy mills (FEM) allow micronization of particles thanks to compressed air or gas [13]. This kind of process has been developed in the pharmaceutical field to increase the specific surface and dissolution rate of API. Nevertheless, this single operation often leads to a decrease of

* Corresponding author.
E-mail address: Laurence.Galet@mines-albi.fr (L. Galet).

https://doi.org/10.1016/j.apt.2017.08.014
0921-8831/

flowability due to a relatively strong cohesion between fine particles. To solve this problem, API may be coated with glidants (as nanosilica or talc) to improve various properties: packing density [3], flowability [3,14], de-agglomeration or dispersion [15]. Han et al. [7] have micronized and coated ibuprofen with hydrophilic nano silica in order to produce a formulation for direct compression. They show that the API flowability, bulk density and compressibility are improved after milling and coating by FEM compared to the uncoated API. An additional process step, a premixing between the guest and host particles, is necessary to allow an increase of the flowability in the feeder or a better coating efficiency in the FEM process [16].

All the previous mentioned processes are batch processes, which are time consuming, with limited productivity, and so economically less favourable than continuous processes [17]. Moreover, continuous processes allow a decrease of material handling and avoid scale up problems [18]. They allow greater flexibility concerning products and volumes with a limited product rejection [19]. Concerning the dry particle coating processes, some recent studies have employed a continuous co-milling process in order to improve the API or pharmaceutical excipient performances [3,20,21]. The co-milling processes are generally used for size reduction and improvement of various properties: flowability, tabletability [5,21,22]. These processes require the pre-mixing of particles in order to obtain the desired properties.

The originality of the present work is to study the possibility to perform dry particle coating in a continuous process using twin-screw extruder, which could provide major advantages. In pharmaceutical industries, the flexibility and polyvalence of processes are of great interest. In this case, the possibility to use an existing extruder in order to produce in a continuous way, a significant amount of composite particles is a significant advantage independently of the initial investment. Extrusion is widely used in polymer industry for shaping thermoplastics polymers or for compound formulation. In the pharmaceutical industry, this process allows the production of solid dispersion: an API is usually molecularly dispersed in a water-soluble polymer in order to enhance its solubility. Extrusion can also be found for food processing (cereals, pastas, cheese etc.). Due to the flexibility of screw configurations, temperature and pressure control, extrusion processes offer a variety of applications.

In conventional use, feed materials in powder, granule or pellet form are introduced through a hopper and are conveyed by the rotation of a single or twin-screw in a heated barrel. During the transport, the matter is melted and mixed and finally forced through a die. Such a process exposes treated materials to high levels of shear stress and may also be adapted in an innovative way to a continuous dry particle coating process. A co-rotative twin-screw extruder without a die could also be employed as a dry coating device. The aim of this work is to study and to demonstrate the feasibility of dry particle coating using a twin-screw extrusion derived process.

As particle breakage is an important issue in pharmaceutical drug powders or cosmetics, this may be performed by choosing operating conditions with enough shearing to perform coating but soft enough to avoid particles damaging, particularly in case of brittle host particles which could be broken by the process. A preliminary study on the effect of operating conditions on particles will always been required.

We avoid the use of abrasive materials (example: nano-siliica particles [21]) according to the recommendation of the equipment provider. This exploration and innovative work was for a particle couple well-known as excipients in the pharmaceutical industry: cellulose/talc, and previously used in batch process [23]. We chose to highlight the influence of process parameters on the coating quality for this couple of particles in order to test the coating method.

Microcrystalline cellulose granules (Cellet®) were chosen as host particles. These particles display a high abrasion resistance and biocompatibility. They are commonly used in pharmaceutical industry as an excipient for controlled release formulations [24–26]. Cellet® particles were also studied as an adsorbent for removing dyes in water treatment [27]. Talc was chosen as the guest particles. It is employed in the pharmaceutical industry as an anti-sticking agent for tablet production [28] and as a dermatological protector [29]. Both materials are nonabrasive, which is important to avoid damaging the equipment.

These materials are interesting both in regard to their industrial applications and to their physico-chemical differences. Composition difference leads to a favourable contrast for scanning electron microscopy (SEM) observations and Energy Dispersive X-ray measurements. The difference in surface properties (hydrophilic and hydrophobic behaviour for cellulose and talc respectively) allows the wettability as an end-use characterization depending on the quality of the coating.

The first part of this paper focuses on the impact of the operating conditions, screw configurations and rotation speed, on the host particles morphology. The second part concerns the production of composite particles using such a process to perform dry coating. Influence of screw rotation speed on coated particles was also examined.

2. Material and methods

2.1. Materials

Cellet® particles (microcrystalline cellulose provided by Pharmatrans Switzerland, Cellet®100) were used as host particles. They display a narrow size distribution range from 100 to 200 μm, with a 160 μm median size (D_H) and a 1.47 g cm^{-3} solid density (ρ_H). The talc used as guest particles is Talc00 lamellar magnesium silicate $Mg_3Si_4O_{10}(OH)_2$ powder from Luzenac quarry, France, with a 12 μm median size (D_G) and a 2.82 g cm^{-3} solid density (ρ_G). See part 2.3. for characterization methods. SEM images of raw materials are shown in Fig. 1. Fig. 2 shows images of a water drop on the surface of Cellet® and talc particles as a function of time. On the pure Cellet® particles, the water drop takes less than 1 s to be completely absorbed, showing the high hydrophilic property of this cellulosic material. On the contrary, after deposition, the water drop is very stable on the bed of talc particles for several minutes demonstrating its hydrophobic property with a contact angle of 130° (Fig. 2D). To illustrate those two different water affinities of the materials and the water drop behaviours, videos are proposed as supplementary data (Appendix A). Description of measurement equipment is described in Section 2.3.

2.2. Coating process

Dry particle coating has been carried out using a co-rotative twin-screw extruder Pharmalab 16 (Thermo Scientific, Germany). This device is characterised by a 40:1 L/D ratio, where L (640 mm) and D (16 mm) are the length and the internal diameter of the barrel respectively. The external diameter of the screws is 15.6 mm allowing an air gap of 200 μm. Host particle size is small enough to allow their transport through the air gap and large enough to provide shearing in this region. Two gravimetric feeders (Brabender, Germany), with capacities of 0.05–2.5 kg and 0.5–10 kg, were used for guest and host particles respectively. These feeders are both placed upstream of the extruder screws. Screw configurations, composed of conveying elements and