ELSEVIER

Chemical Engineering Science 60 (2005) 3805–3816

Chemical Engineering Science

www.elsevier.com/locate/ces

Structure of powder flow in a planetary mixer during wet-mass granulation

B.F.C. Laurent*

Department of Engineering, Institute for Manufacturing, Cambridge University, Mill lane, Cambridge CB2 1RX, UK

Abstract

Wet massing granulation, a widely used industrial process, is difficult to monitor and control and the structure of the flow is poorly understood. Flow patterns in a planetary mixer were investigated using positron emission particle tracking. Both dry and wet powders of a model pharmaceutical formulation were studied to develop understanding of the influence of moisture content on the flow structure during granulation. The flow structure was characterised using the distributions of the velocity components in different cross-sections of the mixer. Fourier analysis showed that the dry system is essentially dissipative and disordered whereas the wet system, being more inertial, shows signs of being more ordered with a periodic recirculation within the bowl. In both systems, radial and axial displacements are strongly correlated. For the dry system, within a central radial core region, the behaviour of the particle was determined by the rapid movement of the agitator, forming a single toroidal recycling cell. The radial and axial velocities of the tracer were up to two orders of magnitude lower than the tangential component. However, in the regions close to the wall, the particle was found to exhibit small movements dictated by the planetary rotation. For wet systems these two main regions were again observed. However, velocity field and velocity distribution showed the presence of two toroidal circulation loops, one above the other. In the wall region, the small movements governed by the planetary motion were again found, but with the amplitude of the displacements reduced by an order of magnitude.

Keywords: Granulation; Mixing; Planetary mixer; Positron; Powder

1. Introduction

Wet massing granulation is a particle size enlargement process widely used in a wide range of industrial applications such as food processing, the manufacture of detergents, fertiliser, pharmaceuticals and the processing of minerals. Its prime function to is to ensure that the flow properties of a granulated product, once dried, are adequate for operations such as tabletting. Wet granulation should yield homogenous, dense and compressible granules with good flow characteristics that avoid segregation (Record, 1980). The granulation process will also predominantly determine the size distribution of the product. The steps involved in wet granulating, as listed by Leuenberger (1993), are the dry blending of the primary excipient or active substance followed by the wetting of the particles by adding liquid binder. The wet mass is then mixed to ensure even distribution of the binding liquid. The liquid content required to produce granules with the desired properties is usually slightly less than the amount that causes over-massing, that is the formation of a dough-like mass. The critical quantity of granulating liquid is sensitive to a wide range of parameters including, it is believed, some that are related to the design of the granulator. At the end of the process, the product is screened to eliminate excessively large aggregates and then dried.

Fig. 1 shows the different states of a sample of lactose mixed with PVP and water as granulation progresses in the planetary mixer described in the next section. During the early stage, here after 4 min, clusters of small granules of diameter around 0.5 mm are formed from primary feed particles. This stage is usually referred as nucleation (Sherrington and Oliver, 1981). As granulation progresses, after 8 min, the size of granules has increased to an average size around a few mm. Some clusters of granules are visible. The small granules may have come into contact with further primary

* Tel.: +44 1223 332 729; fax: +44 1223 338 076.
E-mail address: bfc1100@eng.cam.ac.uk.

0009-2509/$ - see front matter © 2005 Elsevier Ltd. All rights reserved.
doi:10.1016/j.ces.2005.02.011

Fig. 1. Granulation test in a planetary mixer: Influence of granulation time on product; 18% water (wt solids). Water addition to lactose (96 wt%) PVP (4 wt%).

feed particles and undergo layered growth. Alternatively, coalescence may have taken place, whereby two granules collide and adhere to produce a larger granule. After 12 min, the granules have undergone further size enlargement and now have an average size of about 5 mm. The shape of particles is approximately spherical. The action of the mixer causes consolidation of the granules which may in turn lead to forming a single coherent body of dough, the phenomenon termed over-massing.

Newitt and Conway-Jones (1958) classified wet massing granulation in terms of the percentage of volume occupied by the interparticular liquid bridges, i.e., the level of liquid saturation. For a perfectly wetting system, it was suggested that the assemblage of spherical particles that constitutes a granule exhibit several distinct stages. These were observed experimentally by a number of different researchers such as Leuenberger (1994) and were defined as follows: for low moisture contents, liquid is held in the granule as discrete lens-shaped rings at the point of contact of the particles; this is termed the pendular state. At a higher liquid content the rings coalesce and there is a continuous network of liquid interspersed with air; this is the funicular state. With yet further increase in the liquid content the capillary state is reached where all the pore spaces in the granule are completely filled with liquid. It is between the funicular and capillary state where the typical size and strength properties of granules are at an optimum, and where the granulation should be stopped. Further increases in liquid saturation, beyond the capillary state, then produce a dough-like paste, and finally a slurry. In industrial practice, the binding liquid is added continuously which means the regime corresponding to the dispersion of the liquid in the bulk is confused with the regime corresponding to granule growth which renders analysis of results difficult, as observed by Knight (1993). This has been partly solved by a novel approach proposed by Iveson and Lister (1998) who constructed a map giving the progress of granulation in relation to the viscosity of the binder and to the saturation of the bulk which is a more general control parameter than the agitation time to which it is proportional in the case of continuous addition of liquid binder. More recently, Iveson (2002) suggested the use of four independent variables, the particle size, the porosity, the binder content and the composition in order to predict an accurate population balance model.

Insufficient understanding of the steps in the granulation process in relation to independent variables such as agitator rotational speed, fill and moisture content can be linked to the lack of powerful techniques of investigation. This has led to significant difficulties with control and monitoring of processes on the industrial scale. Indeed, the review of Knight (2004) on advances and challenges in granulation technology clearly identifies the characterisation of powder flow within granulators as an area of key importance for industrial applications.

The use of tomographic techniques to investigate opaque systems has provided detailed information not available hitherto, as recently presented in Mann et al. (2001). Positron emission particle tracking (PEPT), a non-invasive on-line technique, has proved recently to provide a unique way of investigating granular mixing (Broadbent et al., 1995; Parker et al., 1997a,b; Laurent et al., 2000). PEPT tracks a radioactive tracer in an opaque medium. The positron camera used here was constructed in 1984 at the Rutherford Appleton Laboratory and is constituted of two detectors containing a stack of 20 cathode–anode planes with a sensitive area of 600 mm $\times$ 300 mm and an efficiency for detection of γ rays of 7%. When the data acquisition system is aware of two γ rays within 25 ns of each other, these are assumed to issue from the same annihilation (Parker et al., 1997a,b). The location of the impact of the two γ rays on each of the detectors permits the construction of the line on which the annihilation (termed the event) occurred. In principle, two events give the location of the positron emitter. However, due to γ ray scattering by interaction with material of the system, for example, some events are corrupted and need to be rejected. Parker et al. (1994) give an account of the reconstruction of the position of the tracer and report that the spatial resolution of the position of the tracer, around a few mm, increases with the tracer speed to 4 mm at a speed of 1 m/s for the camera used in this work. The choice of the tracer is such that its properties are close enough those of the bulk particles so that information gathered on the behaviour of the tracer is representative of the bulk flow. By definition, the technique yields the Lagrangian coordinates of a