Positron Emission Particle Tracking Applied to High Shear Mixer Processing

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Positron emission particle tracking with positron annihilation spectroscopy was used in order to visualize particulate motion within a high shear mixer. As a result, it was possible to visualize the high-speed behavior of particulates within a fluid bed in a mixer, which is an important basic operation in materials processing. It was demonstrated that this method can be useful in improving process design or in improving the quality of the resultant product. [doi:10.2320/matertrans.M2009176]

(Received May 15, 2009; Accepted July 9, 2009; Published August 25, 2009)

\textbf{Keywords:} positron emission particle tracking, positron annihilation spectroscopy, high shear mixer, particulate motion, powder mixing

1. Introduction

High performance dry powder mixing, wet powder mixing and granulation can be achieved through the use of a high shear mixer. These types of powder mixing technologies have been advantageously used in a variety of industries in order to reduce production time, improve process hygiene and powder/liquid mixing, produce stable emulsions, disperse powders or disintegrate solids, blend liquids of different viscosities, disperse and hydrate gums, and dilute high active surfactants, thickeners and stabilizers. In powder mixing, which is a fundamental technology used in the production of pharmaceuticals, clay and ceramic, detergents, and agricultural chemicals, the quality of the product depends on the degree of mixing of the constituent materials, and the homogeneity of the resulting mixture depends on the nature of the mixing procedure. Moreover, the physical properties of the materials processed influence the design of the mixer, and the properties of the source materials affect the flow pattern within the mixer. Compared to conventional mixing methods such as a fluidized bed, a high shear mixer has the advantages of being able to handle fine cohesive powders as well as high viscosity binder liquids. Further, high shear mixer granulation usually results in more spherical, better-compacted granules with a wider particle size distribution than does fluid bed granulation.

A complex mechanism dominates high shear mixer granulation, owing to the fact that there is an enormous range of geometries and designs for the mixer, with a very wide range of agitation intensities (i.e. shear rates). The mechanisms for powder flow behavior within a high shear mixer are still poorly understood because mixers are considered to be the most complicated of all granulators for analyzing product attributes.\textsuperscript{17} Positron Emission Particle Tracking (PEPT) can provide quantitative information for particle motion in a mixer, and is a powerful tool for non-invasively exploring the dynamic behavior of a single particle in an opaque system. Similarly, Positron Annihilation Spectroscopy (PAS) is a versatile and non-destructive technique that has been used to study voids and defects as small as atomic scale in a wide range of materials, including metals, semiconductors, insulators and polymers. PAS can be used to probe the bulk properties of materials as well as to examine the properties of layer structures.\textsuperscript{20}

No PEPT measurements for a high shear mixer have been reported to date because no suitably small tracer particles have been available. In recent years, however, finer resin tracer particles have been developed, which has made it possible to mimic the fine cohesive particles used in a high shear mixer. In this study, positron emission particle tracking with positron annihilation spectroscopy is used in order to visualize the particulate motion in a high shear mixer.

2. Experimental Method

Microcrystalline cellulose powder (MCC; Avicel PH102, Merck, UK) was used as a starting material. The high shear mixer used in this study was fitted with a stainless steel bowl with an internal diameter of 210 mm and vertical side height of 200 mm, which was attached by using a centrally mounted impeller rotating around the vertical axis (Fig. 1). Figure 1 also shows that PEPT involves the use of a radioactive tracer particle, which produce back-to-back $\gamma$-rays through collision of positrons with local electrons. Two beveled blades with a length of 87 mm, a thickness of 10 mm and an angle of the leading edge of 13° were used in the granulation (Fig. 2), with a rotational speed of 600 rpm.

PEPT is a method in which a single labeled radioactive tracer is tracked in three dimensions with granulation time, where the tracer mixes with a bed of particulate and granules. In this technique, a single tracer particle is labeled with a radioisotope. The radionuclide in the tracer undergoes $\beta^+$ decay, that is, a proton is converted to a neutron, accompanied by the release of a positron.\textsuperscript{3} Following its emission, the positron will slow down over a small stopping distance and will subsequently annihilate with a neighboring electron. This electron-positron annihilation is possible only when the momentum and energy conservation laws are satisfied. Each annihilation will result in a pair of 511 keV $\gamma$-rays which, in order to conserve momentum, are emitted back-to-back, 180° apart (to within approximately 0.3°).\textsuperscript{4} Detection of coincident pairs of gamma photons on the two...
camera detectors provides a straight line along which the particle lies, and lines generated from subsequent pairs will intercept at the source, enabling determination of the particle positrons by geometric triangulation.

It is necessary for the size and density of the tracer to be close to that of the bed materials, and in this study, a resin particle (strong base anion ion exchanger) with a diameter of 200 μm and a density of 1100 kg m⁻³ was used as the tracer particle in order to match the diameter and density of the MCC powder.

A radioisotope of sodium (²²Na) is most commonly used in PAS as the source of the positrons. However, in this study, the tracer particles were labeled using a radioisotope ¹⁸F via the ion exchange technique. During the granulation, the tracer particle rapidly stuck to the finer MCC particles, and the movement of the tracer was therefore identical to that of the bulk of the granules and particles.

When the emitted positrons are in the 0 to 700 keV energy range, as was the case in this study, the result is that the mean range of the positrons in different materials will generally not exceed 150 μm. This indicates that the majority of the positrons will be annihilated in the layers nearest to the surface of the material under investigation.⁴ Therefore, PAS can be used to determine the structure of any defects or voids in these near-surface layers, or in the case of the powder mixing in this study, PAS can be used to determine the behavior of the materials that are being mixed, particularly in the near-surface layers.

3. Results and Discussion

Figures 3 and 4 show the movement of the particles within the mixer during powder mixing at a speed of 600 rpm where the time-averaged occupancy information as a function of position are displayed. The results displayed are averaged over all the passes of the tracer through each individual pixel. The quantity in this occupancy plot represents the fraction of the selected time range that the tracer was located in each pixel. Occupancy $O$ is defined as $O = t_{\text{pixel}} / t_{\text{total}}$ where $t_{\text{pixel}}$ is the time the tracer spends within an individual pixel, and $t_{\text{total}}$ is the total time range. The length of the arrows corresponds to velocity of the flow. Examining the change in the tracer’s position with time gives an estimate of its instantaneous velocity that is accurate to 10%. The X-Y plane is horizontal and the X-Z plane is parallel to the blades. It is
clear that, while the majority of the region such as the pink region within the mixer will show good mixing behavior, there are regions at the boundaries of the mixture that will show poor mixing.

These PAS-PEPT visualization studies provide useful information on powder flow beneath the powder surface in mixers, which will be helpful in more accurately modeling high shear mixing. For the first time, it has been possible to clarify the flow patterns of the particle in three dimensions. In Fig. 4, the powder flow was mainly directed towards the wall of the mixer. Particularly near the blade, there was less upward flow than in the upper part of the mixer. The velocity of the particles near the blade was much higher than in the upper part of the mixer. There was upward flow in the upper part. The difference between the upper and the lower part was significant.

It is clear that there will be different characteristic impact velocities in each region depending on the location within the mixer. Three-dimensional imaging for high-shear mixers, which was previously impossible to perform with CCD cameras and PIV, has now been achieved by using PEPT with PAS. These new findings should prove extremely useful for the design, the operation and the scale-up of high-shear mixers, although similar experiments should be performed using different powders and different types of mixer designs in order to obtain additional corroborative data.

4. Conclusions

(1) Within a high shear mixer during powder mixing, there are a number of different regions where different characteristic impact velocities will be in each region.

(2) The use of PAS with PEPT provided useful information on powder flow beneath the powder surface, which will be helpful in more accurately modeling high shear mixer processing, making it possible to improve mixer design and quality control.

REFERENCES