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Original Research Paper

Compartmental residence time estimation in batch granulators using a colourimetric image analysis algorithm and Discrete Element Modelling

Andrew D. McGuire [a], Kok Foong Lee [a], Maksym Dosta [b], Sebastian Mosbach [a], Jan-Georg Rosenboom [a,b], Stefan Heinrich [b], Markus Kraft [a,c,*]

[a] Department of Chemical Engineering and Biotechnology, University of Cambridge, New Museums Site, Pembroke Street, Cambridge CB2 3RA, United Kingdom
[b] Institute of Solids Process Engineering and Particle Technology, Hamburg University of Technology, Denickestrasse 15, 21073 Hamburg, Germany
[c] School of Chemical and Biomedical Engineering, Nanyang Technological University, 62 Nanyang Drive, Singapore

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A B S T R A C T

In this paper we present an experimental technique and a novel colourimetric image analysis algorithm to economically evaluate particle residence times within regions of batch granulators for use in compartmental population balance models. Residence times are extracted using a simple mixing model in conjunction with colourimetric data. The technique is applied to the mixing of wet coloured granules (binary and ternary systems) in a laboratory scale mixer. The resulting particle concentration evolutions were in qualitative agreement with those from the mixing model. It was seen that the algorithm was most stable in the case of the binary colour experiments. Lastly, simulations using the Discrete Element Method (DEM) were also performed to further validate the assumptions made in the analysis of the experimental results. Particle concentrations from the simulations showed the same trends as the experiment and highlighted the importance of particle size distributions on the DEM residence times.

1. Introduction

Granulation is an example of particle manufacture and is a key process used in the production of foodstuffs, pharmaceutical tablets and fertilisers among others [1]. The granulated product will have an optimum size (typically a distribution), porosity, solubility, mechanical strength, shape and flow-ability amongst other properties dictated by the specific application. Granules have several advantages over a simple mixture of the raw ingredients such as better flow-ability; better transport properties (such as limited separation of components and reduced risk of powder explosions); dissolution behaviour and controlled release of Active Pharmaceutical Ingredients (API) [2,3].

Due to the complexity of granulation as a unit operation and the number of particles involved, Population Balance Models (PBM) [4] are typically employed to simulate these systems [2,5–16]. Here, the individual particle sub-processes such as coagulation and breakage are recast as transformations which act on the particle ensemble to evolve it in time. In this study we define *particle* as any free moving material in the mixer including unbounded precursor powder and agglomerates.

Vital to such models is the compartmentalisation of the processing system, granulators being just one example, to represent spatial inhomogeneities in processing conditions [17–23]. Each compartment is assumed to be well-mixed and may have a different set of sub-process rates. Material may be transferred between compartments as illustrated in Fig. 1. Though large regions of batch granulators cannot generally be considered to be well-mixed at all times during the mixing process, this assumption is nevertheless critical in compartmental population balance models of granular systems. The primary aim of this study is the estimation of compartmental residence times for use in massive scale parameter studies of compartmental population balance models. For such simulations to be feasible, simulations must be computationally inexpensive and thus, compartmental residence times must be known *a priori*. In this study, we assert the well-mixed assumption on the models used to estimate residence times from the experimental data. Due to this simplification we are primarily interested in evaluating the order of magnitude of such residence times in the experimental system.

* Corresponding author at: Department of Chemical Engineering and Biotechnology, University of Cambridge, New Museums Site, Pembroke Street, Cambridge CB2 3RA, United Kingdom. Fax: +44 1223 334796.
E-mail address: mk306@cam.ac.uk (M. Kraft).
URL: http://como.cheng.cam.ac.uk (M. Kraft).

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Nomenclature

Roman symbols

b	blue component of an rgb vector [–]
B	blue component of an RGB vector [–]
C_i	compartment i [–]
g	green component of an rgb vector [–]
G	green component of an RGB vector [–]
H	Heaviside step function [–]
$K_{i,j}$	fitted model gain particle j in compartment i [–]
$\dot{M}_{k\to p}$	mass flow-rate from compartment k to p kg s^{-1}
n	number of compartments [–]
N	number of particles [–]
O	occupancy [–]
$Q_{k\to p}$	volumetric flow-rate from compartment k to p m^3 s^{-1}
$Q^{[j]}_{k\to p}$	volumetric flow-rate of particle type j from compartment k to p kg s^{-1}
r	red component of an rgb vector [–]
R	red component of an RGB vector [–]
RGB	three component digital RGB column vector [–]
t	time s
$V_{i,T}$	total volume of particles in compartment i m^3
V_{particle}	total volume of particles m^3

Greek symbols

θ	model time-delay s
$\boldsymbol{\lambda_i}$	normalised average RGB vector for compartment i [–]
$\nu_{i,j}$	uncalibrated particle volume fraction for particle type j in compartment i [–]
$\boldsymbol{\nu}_i$	vector containing uncalibrated particle volume fractions for all components in compartment i [–]
$\hat{\nu}_{i,j}$	calibrated particle volume fraction for particle type j in compartment i [–]
$\hat{\boldsymbol{\nu}}_i$	vector containing calibrated particle volume fractions for all components in compartment i [–]
τ_i	characteristic residence time of compartment i s
$\phi_{i,j}$	colour calibration constant for particle type j in compartment i [–]
$\boldsymbol{\Phi}_i$	colour calibration array for compartment i [–]

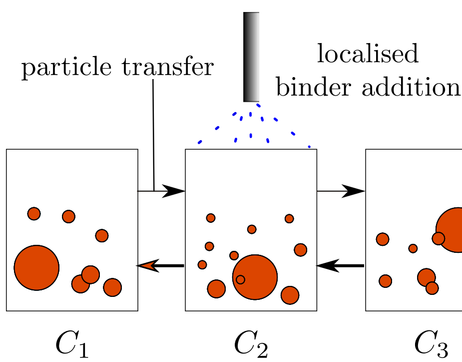

Fig. 1. Compartmentalisation of a batch granulation system.

There is a rich history of mixing analysis in ploughshare mixers. Originally such analysis was performed using positron emission particle tracking (PEPT), in which a radioactive tracer is tracked as the particle bed is mixed. PEPT studies have been used to assess the effect of fill level, particle size and rotor speed in ploughshare mixers and other batch mixing equipment [24–30]. The estimation of residence times in regions of some system has been attempted using PEPT but the ability to track only a single particle generally leads to large degrees of uncertainty in the results [24,25]. Generally these studies focus on the quantification of overall mixing within the entire system by means of a mixing index [31]. Whether or not the movement of the radioactive tracer used in PEPT studies is in fact representative of the bulk material is still controversial.

Particle mixing analysis has also been performed using the Discrete Element Methods (DEM) [32–37]. DEM has allowed for the investigation of variables beyond traditional PEPT, such as the effect of particle shape on mixing [38–40]. It also allows for the degree of mixing to be assessed qualitatively/quantitatively using many particles, as opposed to the single tracer used under PEPT. Mixing effectiveness in DEM is generally assessed by tagging particles with a colour [41]. Particles are initially segregated by colour and the method of centroids is used to produce mixing curves [29,39,42]. Colour particles are also used to aid qualitative visual assessment of mixing in such systems [43,44].The state-of-the art in granulation modelling involves the coupling (uni or bi-directional) of techniques such as DEM [34,35] and computational fluid dynamics (CFD) [45] to particle models such as PBM. In this way, collision frequencies between particle size classes and particle flow characteristics can be updated as the particle ensemble evolves. Similarly, the resulting ensemble properties (such as size and porosity distributions) can be fed back to the DEM simulations to induce new collision/flow behaviour.

Though both PEPT and DEM allow for in-depth analysis of mixing in batch particulate systems, both have significant drawbacks. Under DEM, it has been stressed that particle flow characteristics are strongly dependent on the particles physical properties (wet granule strength and asperity sizes), many of which have to be assumed within a DEM simulation [46,47]. It has also been noted that mixing characteristics derived from ideal systems (monodispersed, spherical, dry materials) have questionable applicability within an industrial context [47]. This highlights the need for mixing experiments to use materials that bear close resemblance (in terms of material physical properties) to those used in application so as to mimic the true granular systems as closely as possible.

Colourimetric methods for the quantitative characterisation of mixing in batch particulate systems have received increased attention in the last decade as imaging equipment such as digital cameras have become relatively inexpensive [47–51]. Data acquisition through digital imaging is often preferred over more experimentally intensive techniques such as PEPT and computationally intensive techniques such as DEM. In comparison to PEPT experiments, digital camera imaging is widely available, low-cost and does not require irreversible modification of the equipment. Colourimetry has been successfully applied to estimate residence time distributions in continuous granulation systems such as twin screw [52,53] by means of a dye impulse at the binder inlet or a variation in the colour of the feedstock. Here, the residence time distribution is assessed by measuring the time-dependent colour concentrations at the device outlet. Under batch operation the assessment of fluxes/residence time becomes more problematic due to the lack of these inlets/outlets. [47] developed a method for quantifying the effective diffusion coefficient (based on a Fickian model) within a batch drum mixer using dry, coloured, non-ideal powders (non-monodisperse size distribution with the potential to break and agglomerate). This employed solidification or freezing of the particle bed prior to extraction and image analysis of axial slices to asses radial concentration gradients. This technique also allowed for a qualitative evaluation of flux patterns within the equipment.

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