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Abstract

The article presents the detailed workings of a population balance model by mass developed for studying a shrinking-core dissolution process that occurs inside the fluidized bed. The behaviour of the model is discussed through examination of model predictions for monodispersed and polydispersed size distributions. The model is then applied for estimating the shrinking-core dissolution rate constant from measured conversion data. Finally, the model is used to predict the change in particle size distribution during reaction, which is pivotal for optimization of fluidized bed performance.

Keywords: Population balance model, fluidization, shrinking-core reaction

1. Introduction

Fluidized bed reactors are used throughout the chemical industry for conducting heterogeneous reactions. Their outstanding mixing and isothermal properties guarantee high conversions even with very large reacting volumes and masses (Kunii and Levenspiel, 1991). When solid particles are being consumed through surface reactions between the solid phase and the surrounding gases, the problem of changing particle size is a significant process parameter that one must control in order to optimize the throughput and the performance of the fluidized reactor (Harshe and al., 2004). Indeed, the extent of reaction is related to the changes in size distribution, which impact both the hydrodynamics of the reactor and its overall performance.

By predicting the evolution of particle size distribution during reaction through modelling, one may be able to understand the actual impact of particle size distribution on fluidized bed performance, which may lead to more efficient operation in practice. The population balance model (PBM) is one of the best tools available for investigating complex particulate systems. PBMs have been used extensively for describing the evolution of many types of particulate systems as they provide a mathematically rigorous framework capable of tracking the behaviour of individual particles within large populations of particles (Hounslow, 2002). Originating back to the 1960's (Hulburt and Katz, 1964), population balance models have been applied to virtually all particulate processes and are widely documented in the metallurgical, chemical and biological engineering literature (Herbst, 1979; Randolph and Larsen, 1988; Ramkrishna, 2000). In particular, population balances have been used to investigate fluidization processes. In recent years, many researchers have developed PBM solutions by number to their problems, essentially based on the discretization technique developed by (Hounslow and al., 1998). For example, (Saleh and al., 2003) applied the PBM as a diagnostic tool to investigate the coating of monosized particles in a fluidized bed. More recently, (Peglow and al., 2006 ; Peglow and al., 2007) analysed the mass distribution of a tracer in a fluidized bed reactor, from which they predicted the size distribution, the moisture content and the temperature of particles during drying and agglomeration. (Harshe and al., 2004) clearly demonstrated the value of coupling the population balance model with reaction kinetics models, accounting for material and energy balances in a gas/solid reaction. In particular, they used the coupled approach to study the

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influence of operating parameters such as gas feed temperature and catalyst feed rate on polymer properties and particle size distribution in the case of polypropylene reactor.

The details of the chemical reactive fluidization process investigated by the authors cannot be disclosed for confidentiality reasons. Nevertheless, results presented are readily applicable to similar problems. The behaviour of the dense section of the fluidized bed is here within taken as a surface-controlled dissolution process that consumes reacting particles inside the fluidized bed. Detailed analyses of solids consumption mechanisms at the particle scale have revealed that particle dissolution follows a shrinking-core behaviour (Szekely and al., 1976).

2. Derivation of a population balance model by mass

This section presents the detailed workings of a discretized PBM by mass applicable to a surface-controlled dissolution process that occurs inside the fluidized bed. Assuming that the fluidized bed provides well-mixed conditions (Kunii and Levenspiel, 1991), it is not necessary to consider the distribution of particles within the geometrical space, such that only internal coordinates are relevant. Under such conditions, geometric averaged property distribution is all that is required to describe the process. Since the process depends on particle size only, particle size x is the only internal coordinate that must be considered. Finally, the dissolution process is a process whereby particles undergo gradual changes only, meaning that there are no sudden changes in particle properties. Consequently, death and birth processes can be neglected from the population balance formulation. With these assumptions, the PBM can be formulated as follows (Herbst, 1979):

$$\frac{1}{V_T(t)}\frac{\partial\left(V_T(t)\psi(x,t)\right)}{\partial t}+\frac{\partial\left(v(x)\psi(x,t)\right)}{\partial x}=\frac{1}{V_T(t)}\left(\sum_{\substack{\text{inlet}\\ \text{streams}}}\dot{Q}_{in}(t)\psi_{in}(x,t)-\sum_{\substack{\text{outlet}\\ \text{streams}}}\dot{Q}_{out}(t)\psi_{out}(x,t)\right) \tag{1}$$

where $\psi(x,t)$ represents the volume averaged number density distribution of particles. It is assumed that the number density distribution $\psi_{out}(x,t)$ of particles that leave the fluidized bed is not equal to $\psi(x,t)$, since discharge from the bed is dictated by disengagement mechanisms (bubbles roofs exploding at reaching the surface, entrainment of the wake solids contained in the surface bubble or in the trailing bubble) (Kunii and Levenspiel, 1991). It is noted that Equation (1) does not make any particular assumption about the total volume V_T of the fluidized bed, which is expected to vary over time. Although Equation (1) can be solved analytically in special cases *e.g.* (Leblanc and Fogler, 1987), the system can only be solved analytically under more general assumptions. Solving the PBM numerically with number density distributions as per Equation (1) can lead to uncontrollable numerical oscillations caused by huge numbers of particles flowing in and out of fine particle size classes. (Hounslow and al., 1988) have reported this problem and proposed very sound solutions that permit solving the PBM *by number* with a high degree of reliability. Nevertheless, since the dissolution process is expected to produce very large quantities of fine particles, this work proposes a solution for solving the population balance *by mass* instead, which has been recognised by many as a particularly favourable and meaningful option (Scarlett, 2002).

Let us define m(x,t) such that m(x)dx is the mass in kilograms of particles of size x to x+dx per unit volume of the system. One worthy advantage of using particle size distribution by mass in the PBM is that it can be determined experimentally with accuracy. Introducing m(x,t) into Equation (1) yields an equation for the population balance by mass.

$$\begin{aligned}\frac{\partial m(x,t)}{\partial t}&=-v(x)\cdot\frac{\partial m(x,t)}{\partial x}+\left[3v(x)x^{-1}-\frac{\partial v(x)}{\partial x}-\frac{(1-\varepsilon(t))}{M_s(t)}\cdot\frac{\partial}{\partial t}\left(\frac{M_s(t)}{(1-\varepsilon(t))}\right)\right]m(x,t)\\&+\frac{(1-\varepsilon(t))}{M_s(t)}\left(\sum_{\substack{\text{inlet}\\ \text{streams}}}\frac{\dot{M}_{in}(t)}{(1-\varepsilon_{in})}m_{in}(x,t)-\sum_{\substack{\text{outlet}\\ \text{streams}}}\frac{\dot{M}_{out}(t)}{(1-\varepsilon_{out})}m_{out}(x,t)\right)\end{aligned} \tag{2}$$

In Equation (2), all terms are homogeneous to a mass per unit time per unit volume of the system. The shrinking core velocity ν(x) will need to be specified. In general, one may use a dissolution velocity $\nu(x) = \nu_0 \times x^{n_0}$ in order to have a dissolution rate that depends on particle size. Bed voidage ε is a function of time as bed volume may vary during the process. In order to solve Equation (2), one may consider size-continuous and size-discretized models. In this work, a size-discretized solution was selected. This choice was motivated by the fact that experimental size distributions are measured in discrete size intervals, so that the measured size distribution information is not necessarily sufficient to convert to continuous density function. Particle size is discretized by dividing the size interval of interest into a finite number N of size classes. Class i is bounded by L_i and L_{i+1} as shown in Figure 1. In order to describe a wide range of sizes, the width of the size classes follow a geometric progression with common ratio $L_{i+1}/L_i = r$.

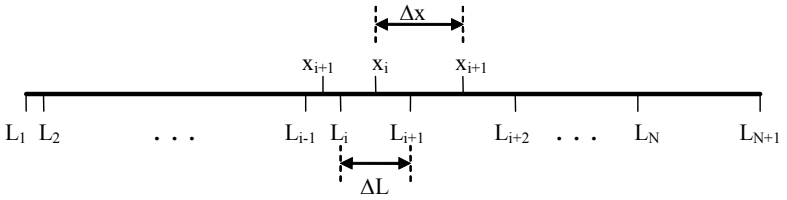

Figure 1. Convention for discretization of the particle size domain

Even though experimental size distributions may not necessarily follow a geometric progression, measured distributions will have to be converted to such a distribution by interpolation so that the proposed model can be applied. With the above discretization scheme for particle size, one would have to apply a numerical scheme that can be used with a nonuniform grid, since the particle size increment $\Delta x = x_{i+1} - x_i$ is not constant. For sake of accuracy and reliability of the numerical results, it is sound to modify Equation (2) further so that a uniform grid can be used with both time and size. Typically, one may chose the representative size x_i of size class i to be either the arithmetic mean diameter or the geometric mean diameter. In both cases, the value of the ratio $\frac{\Delta x}{x} = r - 1$. Let us now introduce a variable y such that $y = \ln(x)$. It is then simple to show that $\Delta y = \ln\left(\frac{\Delta x}{x} + 1\right) = \ln(r)$. Therefore, y can be discretized along a uniform grid with constant step $\Delta y = \ln(r)$. Substituting variable y into Equation (2) gives:

$$\frac{\partial m(y,t)}{\partial t} = -\nu(y)e^{-y} \cdot \frac{\partial m(y,t)}{\partial y} + \left[3\nu(y)e^{-y} - e^{-y}\left(\frac{\partial \nu(y)}{\partial y}\right) - \frac{(1-\varepsilon(t))}{M_s(t)} \cdot \frac{\partial}{\partial t}\left(\frac{M_s(t)}{(1-\varepsilon(t))}\right)\right] m(y,t)$$
$$+ \frac{(1-\varepsilon)}{M_s(t)}\left(\sum_{\substack{\text{inlet} \\ \text{streams}}} \frac{\dot{M}_{in}(t)}{(1-\varepsilon_{in})} m_{in}(y,t) - \sum_{\substack{\text{outlet} \\ \text{streams}}} \frac{\dot{M}_{out}(t)}{(1-\varepsilon_{out})} m_{out}(y,t) \right) \quad (3)$$

Depending on the numerical scheme selected for solving Equation (3), it may not be always possible to guarantee that the unknown vector m(y,t) takes positive values only. In order to eliminate this problem, a new variable w(y,t) such that $m(y,t) = w^2(y,t)$ is introduced into Equation (3). This yields the final PBM equation proposed by the authors.

$$\frac{\partial w(y,t)}{\partial t} = -\nu(y)e^{-y} \cdot \frac{\partial w(y,t)}{\partial y} + \frac{1}{2}\left[3\nu(y)e^{-y} - e^{-y}\left(\frac{\partial \nu(y)}{\partial y}\right) - \frac{(1-\varepsilon(t))}{M_s(t)} \cdot \frac{\partial}{\partial t}\left(\frac{M_s(t)}{(1-\varepsilon(t))}\right)\right] w(y,t)$$
$$+ \frac{1}{2w(y,t)} \times \frac{(1-\varepsilon)}{M_s(t)}\left(\sum_{\substack{\text{inlet} \\ \text{streams}}} \frac{\dot{M}_{in}(t)}{(1-\varepsilon_{in})} w^2{}_{in}(y,t) - \sum_{\substack{\text{outlet} \\ \text{streams}}} \frac{\dot{M}_{out}(t)}{(1-\varepsilon_{out})} w^2{}_{out}(y,t) \right) \quad (4)$$

A number of numerical algorithms can be used to solve Equation (4). In this work, Equation (4) was discretized on a uniform (Δt, Δy) grid with the Crank-Nicholson scheme (Patankar, 1980), which is second-order accurate in time and unconditionally stable (Press and Teukolsky, 1982). This scheme evaluates all the terms of Equation (2) at time $t + \Delta t/2$, where Δt is the time increment used to propagate the solution in time. The overall discretized PBM is given in Equation (5), where the indices i and n refer to size variable y and time t respectively.

$$
\begin{aligned}
0 = {} & \frac{w_{i,n+1} - w_{i,n}}{\Delta t} + \nu(y_i)_i \; e^{-y_i} \left(\frac{w_{i+1,n+1} + w_{i+1,n} - w_{i-1,n+1} - w_{i-1,n}}{4\Delta y} \right) \\
& - \frac{1}{4} \left(3\nu(y_i) e^{-y_i} - \left(e^{-y_i} \cdot \frac{\nu(y_{i+1}) - \nu(y_{i-1})}{2\Delta y} \right) - \frac{2}{\Delta t} \left(\frac{\left(\frac{M_s}{1-\varepsilon}\right)_{i,n+1} - \left(\frac{M_s}{1-\varepsilon}\right)_{i,n}}{\left(\frac{M_s}{1-\varepsilon}\right)_{i,n+1} + \left(\frac{M_s}{1-\varepsilon}\right)_{i,n}} \right) \right) \times \left(w_{i,n+1} + w_{i,n} \right) \\
& - \frac{(1-\varepsilon)}{4\left(w_{i,n+1} + w_{i,n} \right) \left(\left(\frac{M_s}{1-\varepsilon}\right)_{i,n+1} + \left(\frac{M_s}{1-\varepsilon}\right)_{i,n} \right)} \times \left(\begin{array}{l} \displaystyle\sum_{\substack{\text{inlet} \\ \text{streams}}} \frac{\left(\dot{M}_{in}\big|_{n+1} + \dot{M}_{in}\big|_{n} \right) \left({w_{i,n+1}}^2 + {w_{i,n}}^2 \right)_{in}}{(1-\varepsilon_{in})} \\ \displaystyle - \sum_{\substack{\text{outlet} \\ \text{streams}}} \frac{\left(\dot{M}_{out}\big|_{n+1} + \dot{M}_{out}\big|_{n} \right) \left({w_{i,n+1}}^2 + {w_{i,n}}^2 \right)_{out}}{(1-\varepsilon_{out})} \end{array} \right)
\end{aligned}
\tag{5}
$$

In Equation (5), the term $\left(\frac{M_s}{1-\varepsilon}\right)_n = \frac{\sum_{i=1}^{N} {w_{i,n}}^2}{1-\varepsilon_n}$, noting that $\sum_{i=1}^{N} {w_{i,n}}^2$ is the total mass M_s of particles per unit volume at time n.

3. Population balance model testing

Equation (5) is a system of N nonlinear equations that must be solved simultaneously at every time step. In this work, Equation (5) was implemented in MATLAB (The Mathworks Inc., 1992). The system of nonlinear equations was solved using the *fsolve()* function with the Levenberg-Marquardt algorithm. It is worth noting that no additional equation besides Equation (5) was used for calculating the results presented hereafter, thereby confirming that the proposed solution is numerically robust.

Equation (5) was tested in batch mode ($\dot{M}_{in} = \dot{M}_{out} = 0$) with a constant bed porosity. Under such conditions, the 3rd line of Equation (5) vanishes altogether. Figure 2 shows the predicted evolution of a nearly monodispersed size distribution and a polydispersed size distribution, noting that the polydispersed distribution is taken from the industrial process itself. The shrinking-core dissolution rate was set to $\nu_0 = -9.8\times10^{-10}$ m.s^{-1}, which is realistic for the process. Figure 6 shows the initial and simulated mass distributions, which correspond to the value of the variable m(x,t) in each size class rather than the mass frequency distribution. This mode of representation is interesting as it shows both the shift in particle size distribution that takes place during the process and the actual mass of solids that remains in the system, which is the integral under the curve. It appears clearly the solution is numerically stable up until the entire mass has been consumed. This confirms that the choice of computational scheme is suited for the problem at hand. Not surprisingly, the behaviour of the dissolution is completely different between the two size distributions. The monodispersed case clearly shows the appearance of fine particles and disappearance of coarser particles, which is what one would expect during a shrinking-core process. This behaviour is not so obvious with the polydispersed case, which already contains large amount of fine particles, hence surface area, at the start of the process. Because of the presence of fine particles right from the start, the model predicts that the rate of conversion is higher with the polydispersed particles at the start of the reaction, as shown in Figure 3. Nevertheless, it appears that the gradual increase in fines with the monodispersed size distribution eventually become larger after several hours.

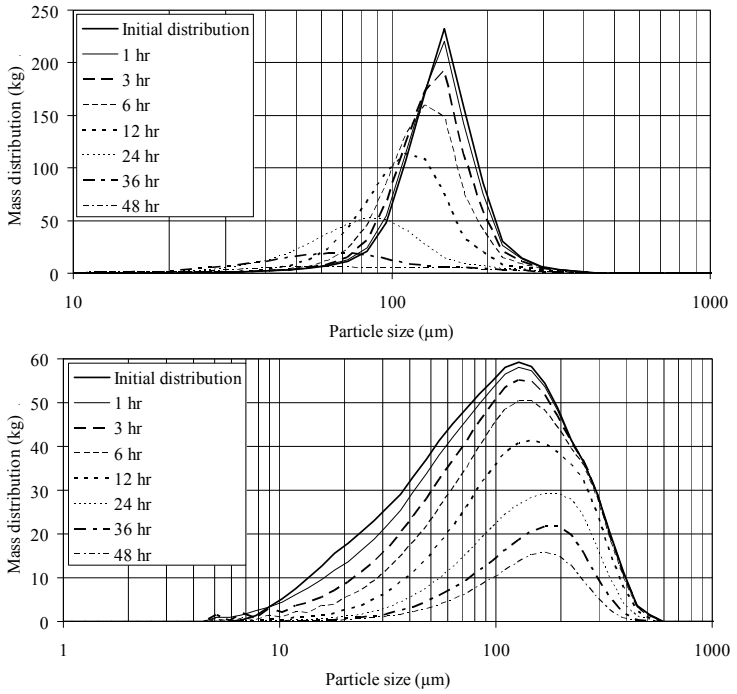

Figure 2. Results from Equation (5) with monodispersed (top) and polydispersed (bottom) particle size distributions

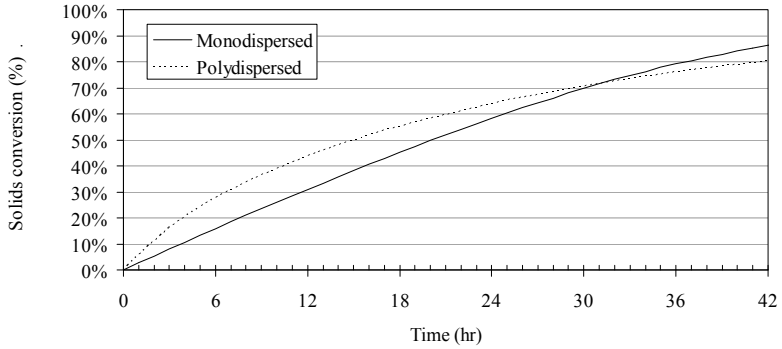

Figure 3. Predicted conversion for monodispersed and polydispersed particle size distributions

Quantitatively, these results must be treated with caution as they apply only to the dissolution rate and size distributions used in the simulations. Nevertheless, the drastically different behaviour between the two size distributions emphasizes that size distribution inside the reactive bed is a critical parameter that must be controlled precisely in order to optimize the production rate.

Contrary to other situations such as agglomeration for example (Hounslow and al., 1988), it is not simple to test the validity of the simulated outputs by examining moments. Indeed, neither the mass nor the number of particles is conserved during the dissolution problem, unless the population balance is coupled with an overall material balance. A given size class with representative size x must become empty after a time equal to $-\frac{x}{2\,v(x)}$, which is the time it takes to completely dissolve a particle of size x. It was verified that individual size classes would become empty precisely at the expected time for a given dissolution rate, thereby confirming that particle mass propagates at the correct rate. Moreover, Equation (5) proved very stable, even with large time increments. The largest time step used during this work was $\Delta t = 60$ seconds. Figure 4 shows the predicted evolution of the Sauter mean diameter x_{32} that corresponds to the previous figures. The model predicts that the specific surface area increases for the monodispersed case and decreases for the polydispersed case, which has also been reported by (Leblanc and Fogler,

1987). However, a simulation over 4 days shows that x_{32} reaches a maximum after 40 hours with the polydispersed size distribution, then decreases monotonically until the entire mass is dissolved. A conversion rate of 97% is predicted after 4 days with the polydispersed distribution.

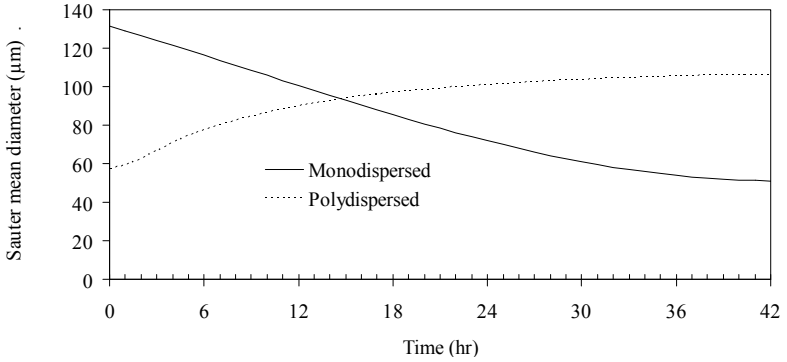

Figure 4. Predicted variation of Sauter mean diameter for monodispersed and polydispersed size distributions

The results that have been presented above all seem to validate the model. In order to further test its validity, the authors are currently planning numerical tests to compare the solution proposed in Equations (5) and (6) with analytical results such as those published by (Leblanc and Fogler, 1987), who derived analytical solutions to the population balance for particle dissolution under specific conditions. These results will be presented in a forthcoming publication.

4. Application of the PBM as a tool for studying a reactive fluidization process

The full-scale plant consists in a large fluidized bed reactor that holds several tons of solids and operates at elevated temperature under highly corrosive atmosphere. Hence, it is not feasible to run any types of tests on such a large unit operation. Over the years, plant engineers have developed a small semi-continuous agitated reactor to help them study and improve the process. The reactor was designed such that its behaviour closely approximates that of the dense phase inside the full scale reactor as far as reaction kinetics are concerned. With this reactor, it is possible to measure the evolution of the conversion rate during the reaction. The data points in Figure 5 show the measured evolution of the conversion rate over a 12.5 hour period. It is noted that this information is the only reliable piece of information that can be obtained from the pilot fluidization reactor at present. The shape of the measured variation is typical of solids consumption by a shrinking-core reaction (Szekely and al., 1976), which is what is expected for the reaction under study. Consequently, the rate of consumption of solid particles was assumed to be size independent, so that $v(x)=v_0$. The PBM was used to determine the dissolution velocity v_0. By varying the dissolution rate v_0 in Equation (5), it was found that the value $v_0=-18.3\times10^{-10}$ m.s^{-1} would yield the 58% conversion measured at the end of the experiment. Figure 5 shows the predicted evolution of conversion during and beyond the duration of the experiment with $v_0=-18.3\times10^{-10}$ m.s^{-1}.

With the dissolution constant determined by matching the measured conversion and that calculated with the PBM, the latter was used to simulate the evolution of the size distribution during the reaction, which is plotted in Figure 6. It is first pointed out that the proposed PBM solution is capable of simulating the evolution in particle size distribution without any numerical difficulty until all particles present in the feed have dissolved. Noting that the initial size distribution is bimodal, with a large quantity of fines, the model predicts that x_{32} increases initially. This is understandable as fine particles, which present the largest surface area, dissolve at a fast rate initially. Once this “excess” fines is dissolved, x_{32} decreases monotonically as the specific surface area increases. Such a simulated behaviour will eventually have to be compared with experimental measurements.

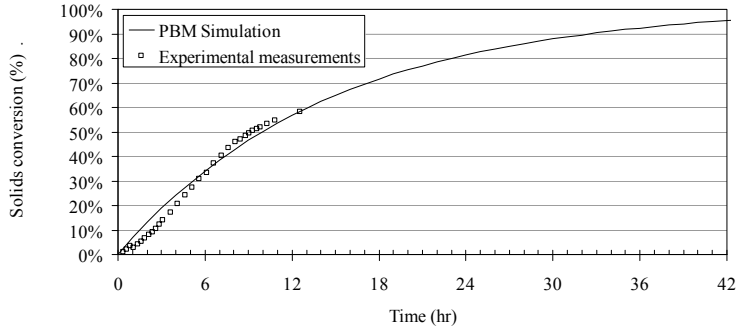

Figure 5. Measured (symbols) and fitted (solid line) conversion

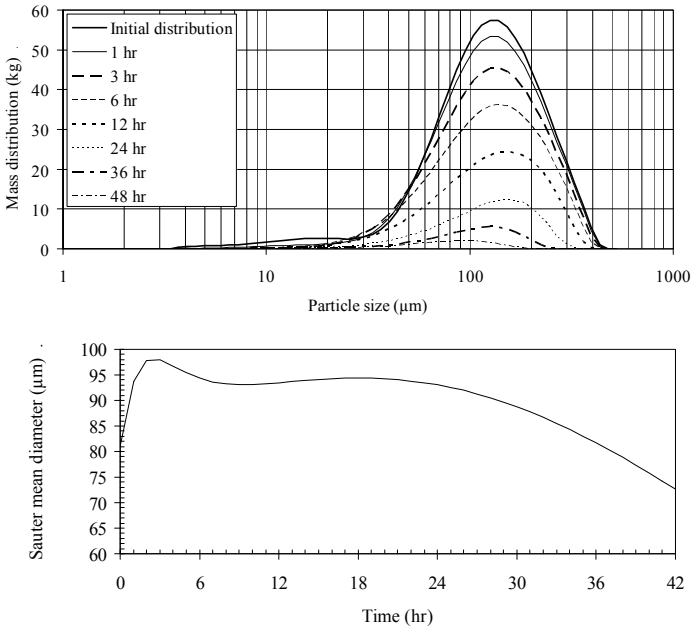

Figure 6. Simulation of particle size distribution (top) and Sauter mean diameter (bottom) variation with the PBM

The PBM model that has been presented is capable of simulating the evolution of particle size distribution and Sauter mean diameter. These variables govern hydrodynamic parameters such as minimal fluidization velocity and terminal settling velocity in the fluidized bed, which dictate bed height and elutriation from the bed. This model will eventually be used in a continuous mode in order to investigate continuous operation of the fluidized bed, and assist plant engineers with optimisation of operating parameters including gas flow, bed height, feed particle size distribution, recycled flow and throughput.

5. Conclusions

This work aimed to develop a reliable population balance model solution as a companion tool for investigating a shrinking-core reaction process inside a fluidized bed reactor. To this end, a population balance model *by m ass* was proposed. Although the model requires a particle size distribution that follows a geometric progression in order to cover a broad range of particle size, the proposed model can be discretized on a uniform time-size grid. This makes the proposed solution attractive and simple to implement. The model was discretized using the Crank-Nicholson scheme. Initial testing and analysis of the predictions indicate that the proposed model and its implementation are reliable.

The PBM was successfully used to estimate the dissolution rate of a shrinking-core reaction that occurs in a pilot fluidized bed reactor. With the estimated dissolution rate, the evolution of a particle size distribution used in the industrial process was simulated, confirming the value of such a model.

A test series is currently underway in order to better quantify the reaction kinetics. It is believed that the PBM will be of great assistance with analysis of experimental data, as for example with identification of unreliable measurements or with analysis of hypothesized reaction mechanisms. Moreover, the PBM is bound to be useful for planning experiments, which is of great value considering that recourse to experimentation must be reduced to a minimum due to safety and cost constraints.

Nomenclature

$\dot{M}$	Solids mass flowrate ($kg.s^{-1}$)
M_s	Mass of solids in fluidized bed (kg)
$\dot{Q}$	Volumetric flowrate ($m^3.s^{-1}$)
V_T	Fluidized bed volume (m^3)
m(x,t)dx	Mass of particles in size range [x:x+dx] per unit volume ($kg.m^{-3}$)
r	ratio between size class boundaries
x	Particle size (m)
x_{32}	Sauter mean diameter (m)
y	Modified particle size = ln(x)
Δ	increment
ε	Bed or stream voidage (fraction)
$\nu(x)$	Size-dependent dissolution rate: $\nu(x) = \nu_0 \times x^{n_0}$ (m/s)
$\psi()$	Volume averaged number density distribution function ($fraction.m^{-3}$)
i	Size class index
in	inlet streams
n	Time index
out	outlet streams

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