16 Case study

In this concluding chapter the application of certain aspects of the preceding knowledge will be illustrated. The case study is entirely fictional, but it includes many aspects appropriate to a large number of industries. Recently, process engineering industries have moved towards high value, relatively low throughput and batch identifiable compounds and this is the strategy that will be used for this example.

An organic compound is produced in a batch reaction between a mixture of liquids. The solvent in the reaction is an alcohol. The organic compound forms a precipitate that needs to be recovered and cleaned of unreacted species, and the presence of residual solvent must be minimised, before being stored as a dry powder, or granule. The solubility of the precipitate in the reactor is temperature sensitive; dissolving at elevated temperature. Each batch produces approximately ½ tonne of dry powder product. The product is to be put into a 2 tonne storage vessel and then mixed with other powder products to provide a particulate product with total mass of 20 tonnes, per batch. The intention is to sell this product in 50 kg drums, or kegs, as a chemical intermediate for further processing. The specifications on the product state that it must flow easily and reliably, and that it must not provide a serious health risk from dust during any further processing. Given the nature of the organic compound produced in the batch reaction, it is desirable to minimise operator contact with the reactor product and subsequent operations.

Let's assume that the particle size distribution of the crystallised product has been analysed and is as shown in Figure 16.1, marked before recrystallisation. Being a batch system, there is probably the intention of moving the product out of the reactor as quickly as possible to allow for another batch, or cleaning, or a different product to be formed in the reactor. This would make good economic sense as the most expensive capital item could be highly utilised. However, *downstream processing*; i.e. unit operations following on from the reactor may provide a bottleneck, if fed with material that is difficult to process. In general, finer particles are more difficult to process than bigger ones and one possibility is that the original precipitate could be reheated, to dissolve the particles, then gently cooled again to grow larger crystals. On reheating the finer particles will preferentially dissolve, as they have a high surface area to volume ratio. On cooling, the particles remaining in suspension will provide sites on which the solid material, coming out of solution, can deposit onto thus forming bigger particles. This is represented by the second curve shown in Figure 16.1. Appropriate data for the two curves are included in the caption. The effectiveness of this strategy can be evaluated by considering the downstream process of filtration.

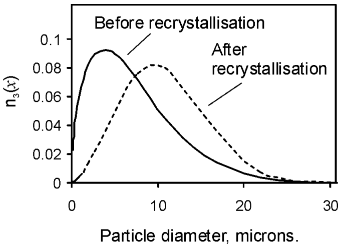

Fig. 16.1 Comparison of two size distributions provided by initial crystallisation and after recrystallisation by heating and cooling – RRB parameters are: $x_{63.2\%}$ of 8 and n of 1.5, and $x_{63.2\%}$ of 12 and n of 2.5, respectively, see equation (2.5)

The permeability of the resulting filter cake from these two suspensions can be determined from the specific surface area and the cake porosity, as described in the box on page 24. The specific surface can be calculated from equation (2.17) and is 1.74 and 0.74 μm^{-1} for the before and after recrystallisation size distributions respectively. The spreadsheet to calculate this is available at:

http://www.midlandit.co.uk/particletechnology

In the absence of testing, the cake porosity will have to be assumed and a reasonable value for a non-biological product with a regular particle shape (i.e. not dendritic) is about 60%, or 0.6 as a fraction. Hence, the cake permeabilities are 8.9x10^{-14} and 5.0x10^{-13} m^2 respectively. These can be converted into specific resistance to filtration using equation (4.12), remembering that solid concentration and porosity must equal one – see Figure 3.1. The organic product has a solid density of 1400 kg m^{-3}; hence, the specific resistances to filtration are 20.0x10^9 and 3.59x10^9 m kg^{-1} for the two size distributed materials. Using a liquid density and viscosity of 980 kg m^{-3} and 0.001 Pa s respectively, and an assumed constant pressure forming the filter cake of 1 bar and 0.15 w/w feed slurry the productivity (filtrate volume with time) on a filter 1 m^2 in area can be calculated using equation (4.18). In this calculation the filter medium resistance (R_m) is assumed to be negligible. The resulting productivity curves are shown in Figure 16.2.

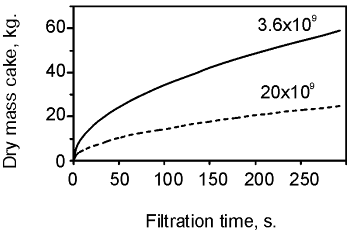

Fig. 16.2 Filter productivity with two particle sizes – full spreadsheet is available at: http://www.midlandit.co.uk/particletechnology

So, by increasing the size of the particles, with the Sauter mean diameter changing from 3.5 to 8.1 μm, the productivity is shown to increase significantly: from about 20 kg in five minutes to 60 kg per m^2 of filter area. Thus, if the filtration capacity is limited it would be better to use the reactor, or a suitable free vessel, to solubilise and recrystallise the solids before filtration to obtain a threefold increase in filtration performance.

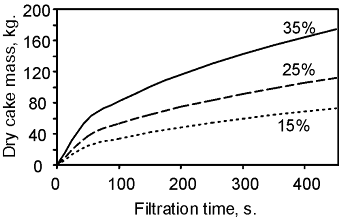

Fig. 16.3 Productivity at different feed slurry concentration marked in % by mass

Productivity in the filtration stage can be improved further by feeding a higher concentration of solids to the filter. This is evident from equation (4.17), where dry cake mass per unit volume of filtrate (c) is seen to be a function of feed slurry concentration. Using different values of feed slurry concentration and equations (4.17) and (4.18) provides the data illustrated in Figure 16.3 – investigating throughput and feed concentration. The feed concentration can be increased in this way by allowing the solids formed in the reaction, at 15% by mass, to settle before passing on to the filtration stage. The time taken for the settling can be estimated from equation (6.1). Firstly, the terminal settling velocity of the particles is required. Using the Sauter mean diameter in Stokes' law, equation (5.5), provides a value of 1.5x10^{-5} m s^{-1}. In order to use equation (6.1) the Particle Reynolds number must be first calculated, equation (5.6), to check which exponent to use in the Richardson and Zaki equation. The Particle Reynolds number is much less than 0.2; thus 4.65 is used in equation (6.1). On investigating solid concentrations between 15%

Filter cake resistance

This example uses a calculated cake resistance from a size distribution. In many instances the measured cake resistance is much higher: due to transfer of fines within the cake to a position of high liquid flow resistance and cake compaction. Ideally, laboratory tests should be used to determine the cake resistance to be used in design studies.

and 35% by mass, 0.11 to 0.27 by volume – see equation (3.16), the settling velocities are found to vary between $1.34\text{x}10^{-5}$ and $1.1\text{x}10^{-5}$ m s^{-1}. Thus, the time taken to settle one metre is between 21 and 25 hours! So, gravity sedimentation is too slow to provide thickening in this case. Clearly, the low density difference between the solid and liquid, and small particle size are factors in this disappointing result.

As there is a wish to minimise operator contact with the product and the need for rapid processing of the batch, one suitable method would be to use an automated filtering centrifuge. One popular type of machine is the inverting bag centrifuge, which can discharge its solids contents directly into a dryer. Washing of the filter cake and spinning dry of residual liquid within the cake is possible prior to cake discharge. These operations will now be considered with the feed concentration of 15% by mass.

In the operation of a filtering centrifuge with particles less than 10 μm in diameter it would be unrealistic to expect to have no filter medium resistance. The fine particles are likely to penetrate the cloth to some extent and cause a significant resistance. Hence, a value of $1\text{x}10^{11}$ m^{-1} will be assumed. The cake resistance and physical properties are as before and other data is as follows: filter basket height 0.22 m, filter basket diameter 0.45 m, and rotational speed of 1200 rpm. Equations (8.24) and (8.26) must be solved and a critically important factor is the cake radius: this is limited by the geometry of the machine and filtration must stop before the cake becomes too great. Under the stated operating conditions, an analysis shows that the radius starts at 0.225 m, the full radius of the device, and after 5 minutes the cake is at a radius of 0.13 m; providing a cake thickness of 0.1 m. In order to wash unreacted chemicals from the product a wash ratio of 4 has been selected, see Figure 8.13. Clearly, the thicker the cake the greater the wash time will be. The wash time is provided by equation (8.27). A reasonable wash time is provided by the 0.1 m thick cake, which was formed by filtering for 300 seconds. The filter has 14.2 kg of solids in it and has filtered 65.6 kg of filtrate. An additional 15.8 kg of liquid is retained in the cake prior to the *dewatering* stage. The time taken to spin the cake as dry as possible and the residual moisture value are obtained from equations (8.29) to (8.33). One advantage of a low cake resistance is that the dewatering stage is quick and very efficient at removing residual solvent within the cake. Figure 16.5 illustrates the speed with which the cake dewaters at a rotational speed of 3400 rpm. All other properties are as provided before, or stated below the figure.

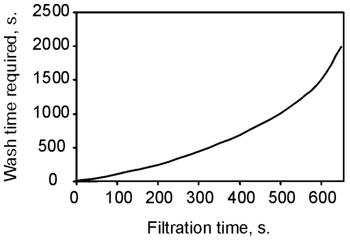

Fig. 16.4 Time required for a wash ratio of 4 on the filtering centrifuge

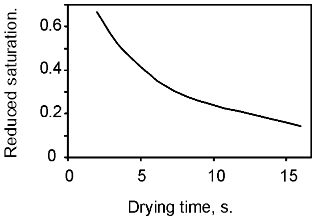

Fig 16.5 Zeitsch's dewatering model – with a surface tension of 0.08 N m^{-1} and 10 degrees solvent to solid surface contact angle and a very low cake resistance

The cycle time, on a single small inverting bag centrifuge is, therefore, 300 seconds for filtration and 500 seconds for washing. Time is also required for speed changes and discharge; thus a total cycle time of 1200 seconds would be anticipated, to provide 14.2 kg of product. This equates to 43 kg per hour and 12 hours to process the entire reactor product using a single machine. This represents best case productivity, as the cake resistance could be higher than we used – see the box on the earlier page.

As the powder must have a low solvent content a drier must be used to reduce the residual saturation of solvent to a very low value and it is likely that agglomerates will probably form. These may need to be broken before storage in the two tonne silos. However, an alternative strategy that fulfils the overall requirement would be to discharge the moist cake from the inverting bag centrifuge into a screw conveyor that feeds a combined fluidised bed drier and granulator. The discharge from the screw conveyor may have to pass through a mesh to form an extrusion that breaks off into the fluidised bed and forms granules during drying. A suitable granule size within the fluidised bed would be about 200 μm.

Under the conditions provided above the required minimum fluidising velocity can be calculated from equation (7.4) and is 0.038 m s^{-1}, for an assumed voidage of 50% at incipient fluidisation and using a gas viscosity and density of 2×10^{-5} Pa s and 1.2 kg m^{-3}, respectively. It is likely that a significant multiple of the minimum fluidising velocity would be used, at least 5 times the value. The settling velocity of the 200 μm granules can be calculated from the Heywood Tables and is 0.84 m s^{-1}, with a Particle Reynolds number of 10, so a fluidising velocity of 0.19 m s^{-1} is safely below the velocity at which significant entrainment of solids in the gas stream above the fluidised bed will occur. This is a low throughout operation and a very small fluidised bed would suffice. Even in a bed of diameter 0.2 m, and height 0.3 m, the average residence time in the bed would be about 9 minutes. The residence time is greater in larger beds and 9 minutes is likely to be sufficient for drying and agglomerate formation. A more detailed analysis of this would require mass transfer data on the particles. An experimental study of this, and the granulation process would be warranted.

The granules from the fluidised bed are sufficiently large and should be spherical in shape; so, they should easily flow and simple gravity discharge from the fluidised bed into the two tonne storage hopper is possible. In this hopper further batches of the reactor product will be added. When the equipment is available, the two tonnes from the hopper need to be mixed with another powder and stored in a 20 tonne hopper before putting into drums. One possibility is to batch mix the powders directly above the 20 tonne storage hopper and to use what is known as *bomb-doors* discharge into the 20 tonne hopper. The wide opening discharge doors of the hopper ensure that the product is discharged with minimal opportunity to segregate. The storage hopper would need to be designed for mass flow discharge so, again, segregation is minimised and the drums should contain a well mixed product.

Pneumatic conveying conditions

mean size 200 μm
Gas density: 1.2 kg m^{-3}
Solid density 1400 kg m^{-3}
gas viscosity 1.8×10^{-5} Pa s
vertical distance 20 m
horizontal distance 30 m
number of bends 8

To raise the granules from the 2 tonne intermediate hopper to the batch mixer a pneumatic conveying pipe may be used, probably with an inert gas. If the conveying rate is 1000 kg h^{-1}, the theory presented in Chapter 9 suggests that a pressure drop of 0.28 bar results from the operating conditions provided in the box, in a 63 mm diameter pipe. The gas flow rate required for this operation is 0.05 $m^3\ s^{-1}$, operating at 50% excess gas over the saltation velocity. Dilute phase conveying should help minimise granule breakup.

Assuming that the powder to be mixed with the granules has a mean size, by mass, of 100 μm the quality of the batch mixture can be estimated from equation (12.5). Note, this will represent the best quality we should expect from the powders; it is possible that the mixture quality will be worse, due to the performance of the equipment. The mass proportion of our granules will be 0.1, i.e. 2 tonnes going to form an overall mixture mass of 20 tonnes, and the mean particle mass of the granules will be $5.9\text{x}10^{-9}$ kg, see box.

Mean particle mass of the granules is:

$$W_p = \frac{\pi}{6}\rho_s x^3$$

where the density is 1400 kg m^{-3} and diameter is 200 μm, but this assumes no internal voidage of the granules. The real density would be slightly less, but the overall mixing result will not significantly change.

The other powder is likely to be a filler and will have a density closer to that of an inorganic material, such as 2000 kg m^{-3}. Thus, the mean particle mass for the other powder will be $1.0\text{x}10^{-9}$ kg. The appropriate sample mass to use in equation (12.5) is the mass of the product leaving the process, which is in 50 kg drums. Hence, the between drum variance of our active ingredient will be $9.7\text{x}10^{-12}$, giving a standard deviation of $3.1\text{x}10^{-6}$. Now, to put these values into context: using the analysis described in Chapter 12, one drum in 1000 will have a proportion of the granules greater than 0.1 plus 3 times the standard deviation, and one in 1000 will have a proportion less than 0.1 minus 3 times standard deviation. This suggests that 998 drums out of 1000 will have masses of the granules varying from 4.9995 to 5.0005 kg. This analysis suggests that a very good mixture quality will be achieved by batch mixing these dry powders. However, segregation as the powder falls from the mixer into the hopper will have to be assessed. Thus, the batch mixing theory provides an indication of the best possible quality that can be obtained, operating procedures and mixer type choice may reduce the mixture quality obtained in practice.

The 20 tonne storage hopper should be designed to provide mass flow, in accordance with Chapter 10. This will minimise segregation during the filling of the 50 kg drums. It is not possible to provide a design here without laboratory data obtained from a shear cell, similar to that provided on page 112. In practice, it is likely that the hopper will discharge directly into the drums which will be positioned on a load cell to weigh the drum contents. An alternative would be to discharge into a screw conveyer, which then deposits the mixture into the drum positioned on a load cell. The latter system is more controllable and less likely to provide a hazard to the process operators.

The above simplified example provides some indication of how unit operations in particle technology are related and how some of the principles covered in the preceding pages may provide an insight, or even an outline design, of the process.