Particle Destruction in Centrifuges

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Industrial processes in the food, chemical and pharmaceutical industries require high and reliable product qualities over long periods and high mass throughputs. Like in salt dehumidification, mechanical dewatering of the product, i.e. the separation of the solid particles from the surrounding liquid phase, is an important unit operation that saves energy and process time in downstream drying apparatus. However, some unwanted effects like the destruction of the separated particles in these machines often impair product quality to a not negligible degree.
In this paper a recently started research project is presented that investigates the sources and reasons of particle destruction in solid-liquid separation apparatus, especially in centrifuges.

1. Motivation

Continuously working centrifuges are required for efficient and economical solid-liquid separation of large amounts of products. During this unit operation, in many processes with a product consisting of crystalline particles with low mechanical strength, like organic crystals, the danger of particle damage or particle destruction exists during the whole course of process. Thus, the amount of fine particles increases severely, at the same time the mean particle size decreases. As a consequence, demanded product qualities cannot be reached, e.g. the width of particle size distribution, mean particle diameter, proportion of coarse and fine material, filtering properties, flowability etc.
Fig. 1 shows an example of a crystallization process in which particle damage occurred. The product was discharged from crystaller 1 and 2 at almost the same particle size distribution. The mean fraction diameter was about 520 µm. During the transport to the solid-liquid separation apparatus this value has changed to about 460 µm. In the pusher centrifuge the mean size decreased severly from the entry to the discharge to 310 µm. The last unit operation, a thermal fluidised bed dryer, could not provide for any improvement. It was only possible to reduce the amount of fine particles by agglomeration. As a matter of fact, the mean particle diameter in this process was reduced from the beginning to the end to almost half of its original size.

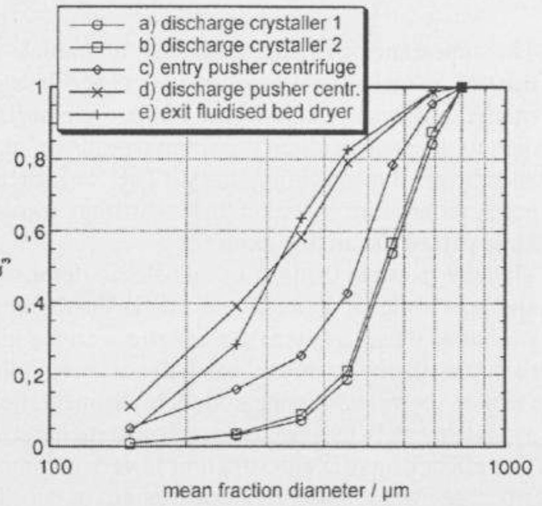


Fig. 1: Particle size distribution of a crystallization process: a), b) discharge of crystaller; c) entry of pusher centrifuge; d) discharge of pusher centrifuge; e) exit thermal dryer

These effects are a considerable problem for the chemical and pharmaceutical industry that must not be underestimated. The solid-liquid separation of a suspension consisting of organic crystals, available as intermediate or final product, from the mother liquid or (after reslurrying) from the washing liquid is interfered by the damaging of the particles due to high mechanical stress during the process. Conveying and transport cause them to crumble. Thus, the operating parameters of the following unit operations are influenced in a negative way (high solids concentration in the filtrate, blocking of filter cloth, very long residence times, even stoppage of a plant section).

Therefore, this research project was recently initiated to localize the sources of particle damage in production processes. The aim of these examinations is to develop a simple method to determine whether the mechanical strength of a crystalline product is sufficient for the stress during its production process. So, by lab-scale tests with few samples of newly developed substances, predictions of the particle behaviour in solid-liquid-separation apparatus could be done to select suitable and optimal apparatus for each unit operation.

2. Theoretical Background

The appearance of particle damage or particle destruction in centrifugal processes is related to a high mechanical energy input into the particle structure due to the acceleration to drum speeds of up to 80 m/s at the machine entry. The influence of accelerating forces within the centrifuge causes a large part of the arising damages.

Thereby, particle damage means elastic deformation and spreading of defects and cracks in the crystal structure. When applying further stress on the grains they are likely to break partially or even totally. Particle destruction is the complete disintegration of a single particle into two or more separate parts.

The effect of particle destruction is very common in processes where pusher centrifuges are in use. They are standard in many areas of solid-liquid separation, e.g. mineral dressing processing, chemical and pharmaceutical industry, fertilizer and plastics industry. Mass products with good filtering properties can be dehumidificated to low levels of residual moisture and high degrees of elutriation.

Several different types of these machines are manufactured. The adaption to a certain product or any

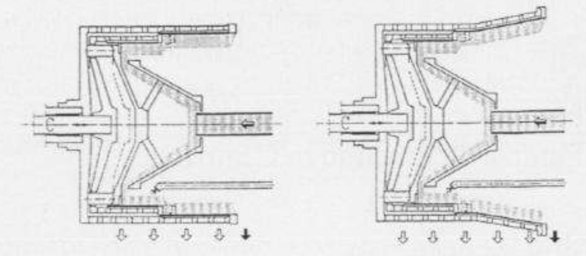


Fig. 2: Pusher centrifuge with a) cylindrical and b) cylindrical-conical drum

special demand is easy due to component kit systems. For example, two types of drums, with cylindrical and cylindrical-conical geometry are shown in fig. 2.

Regarding a cycle in a pusher centrifuge, it can be divided into five phases (fig. 3).

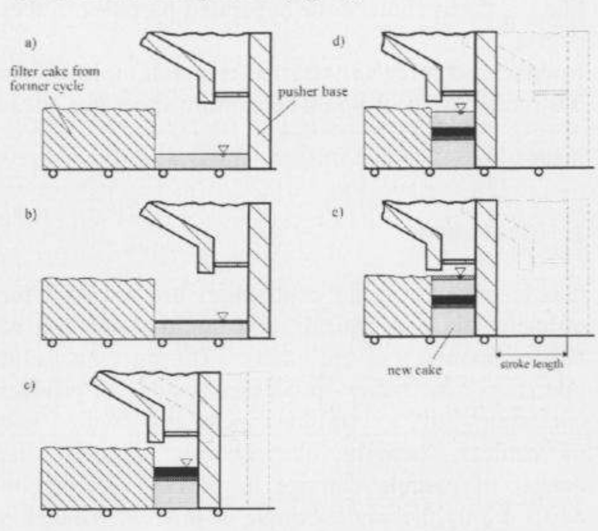


Fig. 3: Process cycle in a pusher centrifuge:
a) back stroke; b) rear dead point;
c) forward stroke at break-off point;
d) forward transport of cake;
e) front dead point

The suspension is fed continuously by a rotating funnel through an annulus onto the interstice sieves (a, b). As the pusher base moves forward again the arising filter cake is compressed in axial direction until it has enough inner strength to push forward the cake built during former cycles (c-e). In all phases the dewatering of the particles in the filter cake continues until, at the end of the sieve drum, the dehumidificated particles are thrown off into the discharge. Thus, three main sources of particle damage can be found in the above process. At the machine entry the particles hit the interstice sieves in radial direction. Additionally, they are accelerated to the drum speed causing a shear stress. Moving in axial direction by the pusher base also causes shear stress in the third dimension. During the transport along the drum axis in the centrifuge the grains are scrubbed over the sieves under the load of the centrifugal force. Finally, at the solids discharge the particles are catapulted at full circumferential speed into the discharge housing. This huge stress can only be stood by strong and brittle crystals. However, a problem is how to determine the necessary strength of particles for a certain process and how to measure it.

At the beginning of the examinations of the mechanical properties of crystals the understanding of the fraction process and the dependence from the particle size and strength is an important topic. Until now the description of the particle destruction procedure is not complete. On the side of solid-liquid-separation it is of great importance to characterize the mechanical strength of given crystals (by measuring or computing) in a way that predictions on the fracture behaviour in centrifuges can be done. For small and brittle crystals the measuring of the hardness (e.g. by methods of Vickers or Brinell) is very difficult to do. Here, the method of single grain compression between two parallel surfaces derives from particle grinding technology and supplies the so-called diametrical pressure test. It is a compression test that provides useable results to estimate the maximum load single grains can stand.

3. Experimental Setup

So far, by means of single grain compression experiments were done at the Institute for Mechanical Process Engineering at the University of Karlsruhe with some industry products, like adipin acid, salicyl acid, refined sugar crystals or sodium chloride. To do so, a simple measuring apparatus, shown in fig. 4, was constructed and built.

Product samples were fractionated by screening. Among every fraction a number of 35 or more particles have to be taken for statistic reliability of the compression tests. The test procedure was the following: a single particle is placed on a glass plate (1) under the piston (2). With a motor-driven micro screw (3) a lever (4) moves down the piston and loads pressure onto the grain. An inductive path sensor (7) measures the movement of lever and piston. The compression force is recorded via a weighing machine with serial interface (6). The measured data is recorded on a PC.

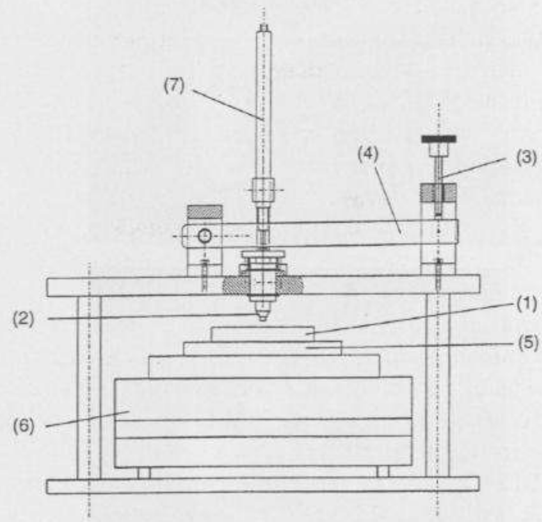


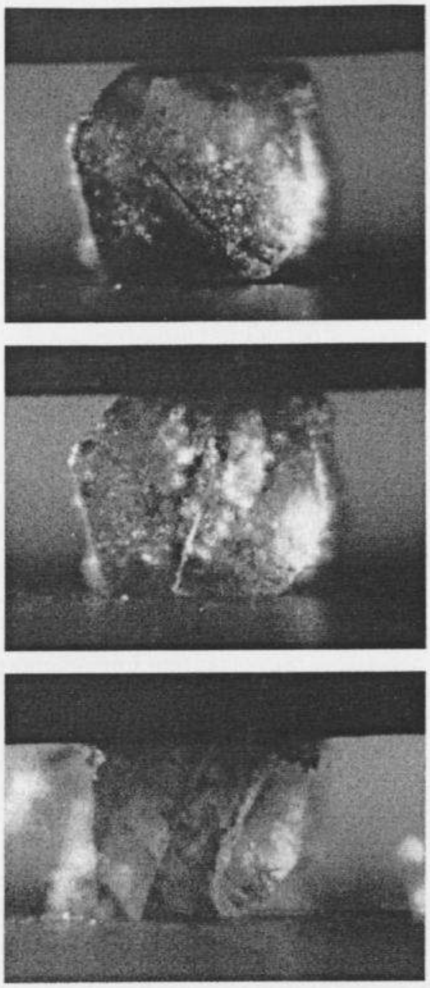
Fig. 4: Apparatus for measuring force-compression curves

With this device combined force-compression curves can be measured (analogous to materials science). With few amounts of product samples it is possible to get statistically covered statements of the products.

Optical monitoring enables the observation of the fracture phenomena of brittle crystals. In fig. 5 phases of a compression procedure are imaged. The first picture (a) shows a grain of at the beginning with no load force on it. A diametrical crack, a predamage, is visible diagonally to the force direction. The piston is moved to the contact point. In (b) the upper contact area is oblate and a first fraction has occurred. The fraction direction is the same as the force direction. Picture (c) shows some more fractions that arise when applying further load force unless the grain is completely broken into several small fragments.

4. Results

The different ways of fracturing that are expressed in the different force-compression curves were assigned to four types of particles. Differences exist in the geometrical and crystal structure. The curves for each type are shown in fig. 6. The data from the weighing machine was recorded over the compres-



a)

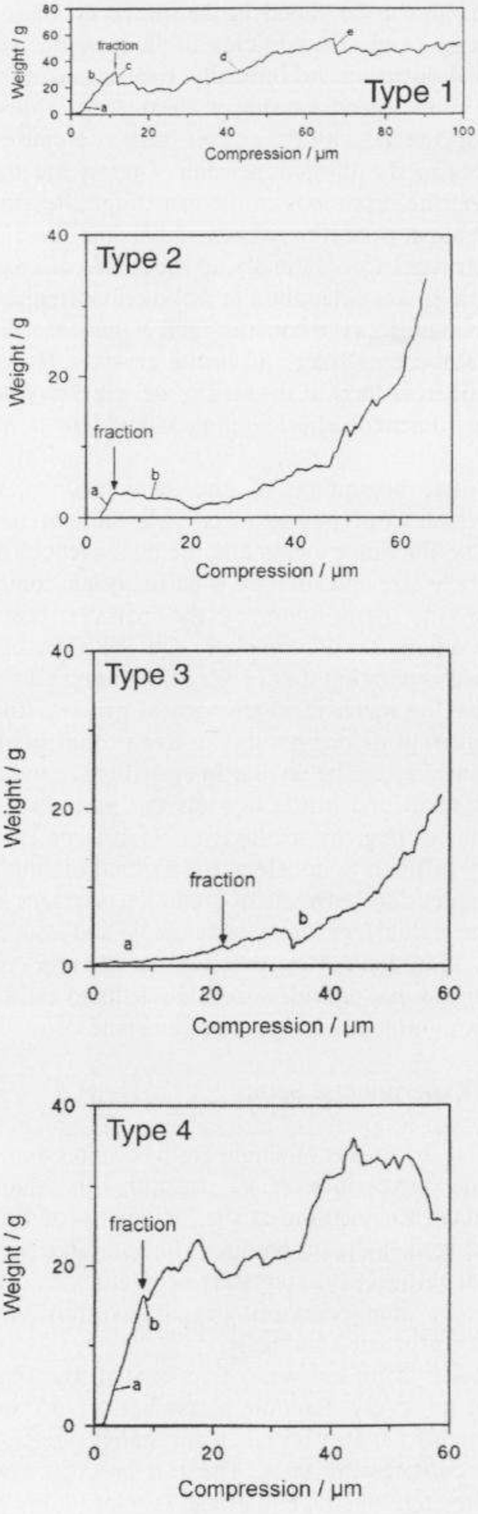
b)

c)

Fig. 5: Phases of single grain compression:
a) before stress; b) after first crack;
c) complete destruction

sion. For example, particle type 1 has a regular crystal structure but an irregular geometry whereas type 2 has regular geometry but irregular crystal structure. The particle types 2 and 3 show plastic deformation while type 1 and 4 have brittle behaviour until the fraction occurs.

Type 1 particles were mostly observed. The slightly irregular raise of compression force at the beginning is caused by micro-fractions in the contact area and by oblating of small surface roughnesses. The complete break-off can be determined due to a loss in compression force of more than 40 %. Type 2 particles are characterized by a long, almost horizontal force line with a strong raise of smaller cracks



a)

b)

c)

d)

Fig. 6: Four different types of particle behaviour:
a), d) brittle; b), c) plastic

fractions due to irregularities in the crystal structure and cracks that existed already before. The compression progress of type 3 particles is characterized by a weak increasing of the force over a long range. The exact determination when the particle breaks is very difficult due to the very irregular geometry of these crystals. Here, agglomerations of finest crystal fragments, destruction of surface peaks and cracks in the main crystal appear together. Particles of type 4 have high fracture loads without visibly inelastic behaviour unto a complete meridian fracture. However, the crystal structure is not weakened in a way that a noticeable force loss is registered, so the particle can stand further loads.

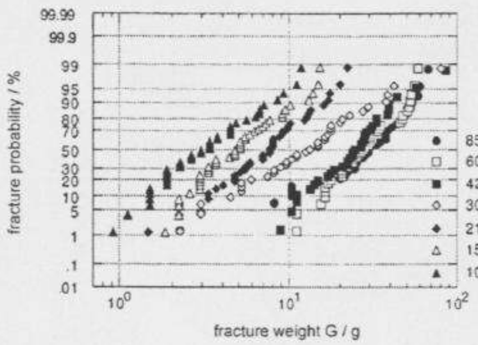


Fig. 7: Fracture probability as function of fracture weight for adipin acid crystals

To describe the particle breakage quantitatively a fracture probability was defined as the amount of particles broken at a certain fracture weight.
In fig. 7 the fracture probability is printed over the fracture weight. In double-logarithmic scale, the fracture probability is nearly linear rising with the fracture weight. The probability gradients are almost parallel. Fig. 8 shows that the mean fracture weight is also increasing with the mean diameter of the screened fractions. Linear approximation fits well to the measured values.

To evaluate the mechanical resistance of the crystals the fracture force was divided by the square of the mean fraction diameter (fig. 9). Here, the smaller particles show higher resistance than the bigger ones in a linear degression. It is also possible that the lower strength of the larger fractions is due to the formation of agglomerates.

For a big particle has a larger contact area and a larger cross-sectional area than a smaller particle, the force needed to break it is higher in comparison to the smaller ones.

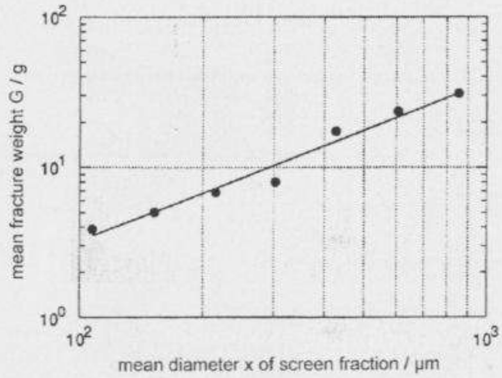


Fig. 8: Mean fracture weight as function of mean fraction diameter for adipin acid crystals

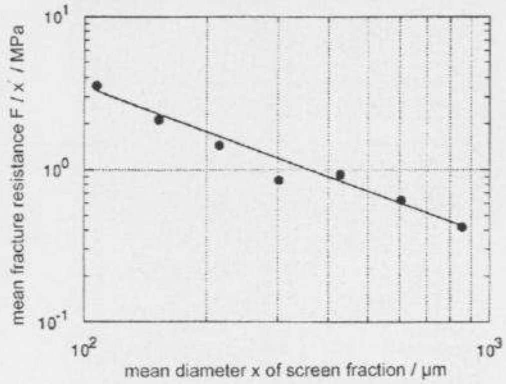


Fig. 9: Mean fracture resistance over mean fraction diameter

The examination of these mechanical particle properties can supply insights into the problem of creating strong and resistant crystals. For the course of the research project it is planned to do further tests to get more information about different products and crystallization processes. Not only single particles will be regarded but also samples of bulk material will be pressed. Measuring the particle stress and the yield energy in a centrifuge provide maximum rotation speeds and centrifugal accelerations for certain types of particles. Additionally, constructive alterations can be realized to reduce the effects of mechanical shocks at the product feed and discharge.