

Centrifuge safety and scale up.

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INTRODUCTION.

All centrifuges have the potential to be hazardous; their fundamental method of operation makes this unavoidable in all but a few special cases. Similar comments apply to many items of equipment used in the process industry such as pressure vessels, dryers, pumps etc. For typical centrifuges such as: (i) a 1.2m diameter batch centrifuge basket spinning at 1,200 RPM; or (ii) a 1.0x3.0m decanter centrifuge operating at 1,300 RPM; the stored energy within the rotating element is approximately 4 MJ and the peripheral speed of the basket/bowl is approximately 160 mph (260 km/hr). This stored energy is equivalent to that of a family car travelling at 175 mph (280 km/hr) or a 2.5m³ vessel pressurised with gas to 16 bar.

With speeds and energies such as these, perhaps coupled with a chemical hazard, there is the potential for a dangerous situation to develop. It is important that the operation, design and maintenance of the centrifuge reduces this risk to an acceptable level. The first part of this paper considers some important aspects of centrifuge safety. For a more complete discussion see reference 1.

Centrifuges used in the chemical and pharmaceutical industries are often selected based on pilot scale trials. The scale up from a pilot plant using a small test centrifuge to a full plant scale unit is an important step in the overall plant design. The final part of this paper highlights some of the important aspects of scale up together with some common pitfalls encountered in practical situations.

SAFETY.

This section considers some of the general safety aspects of centrifuges relating to : (a) Standards & certification, (b) Control systems, (c) Basket design & inspection. The information presented here is of a general nature and in practical situations always review and adhere to the documentation relating to the specific centrifuge in question.

(a) Standards & certification.

Adherence to international standards is a first step to ensure the safe installation and operation of any equipment. The main standard which covers industrial (excluding laboratory) centrifuges is BS.EN12547:1999 'Centrifuges - Common safety requirements'. All equipment sold in the European Economic Area must have a declaration of conformity (CE mark) showing compliance with all relevant Directives and the Essential Health and Safety Requirements (EHSR) detailed within them. For centrifuges, the Machinery Directive and the Low Voltage Directive always apply. Depending on the hazards present and the type of centrifuge, other directives may also apply such as the ATEX (explosive atmosphere) Directive, the Pressure Equipment Directive (PED) and the EMC (electromagnetic compatibility) Directive. Compliance with the centrifuge standard BS:EN12547 is one way to ensure compliance with the Machinery Directive and the Low Voltage Directive.

It should be noted that BS.EN12547 does not consider hazards such as :

- Thermal hazards.
- Micro biological hazards.
- Corrosive & erosive chemical hazards.
- Flammable or explosive hazards.
- Hazards caused by unsuitable hygiene involving food products.
- ... plus other application specific hazards.

Centrifuge safety and scale up.

It is highly likely that one or more of these potential hazards may be present in a pharmaceutical or chemical application.

It is a legal requirement to supply equipment with the correct certification and it is the responsibility of the manufacturer, importer or end user of the equipment to ensure that the necessary certification is in place. This applies to both new equipment, second-hand equipment and equipment that has been significantly overhauled or modified even if the modification or significant overhaul is undertaken by the owner of the equipment. This has had a significant effect on the sale of second-hand equipment and the refurbishment of older equipment as generally it is only a manufacturer's centrifuges who have sufficient information and knowledge to provide adequate certification.

(b) Control systems.

Control systems and their associated interlocks play a vital part in the reduction of the risks associated with the hazards inherent in process equipment. In the case of centrifuges the hazards present depend in part on the type of centrifuge being considered. For a typical batch pharmaceutical basket centrifuge (see figure 1) the safety critical aspects include :

- Allowing human access only under safe conditions.
- Operation of product discharge mechanisms.
- Operation of feeding/washing systems.
- Basket integrity.

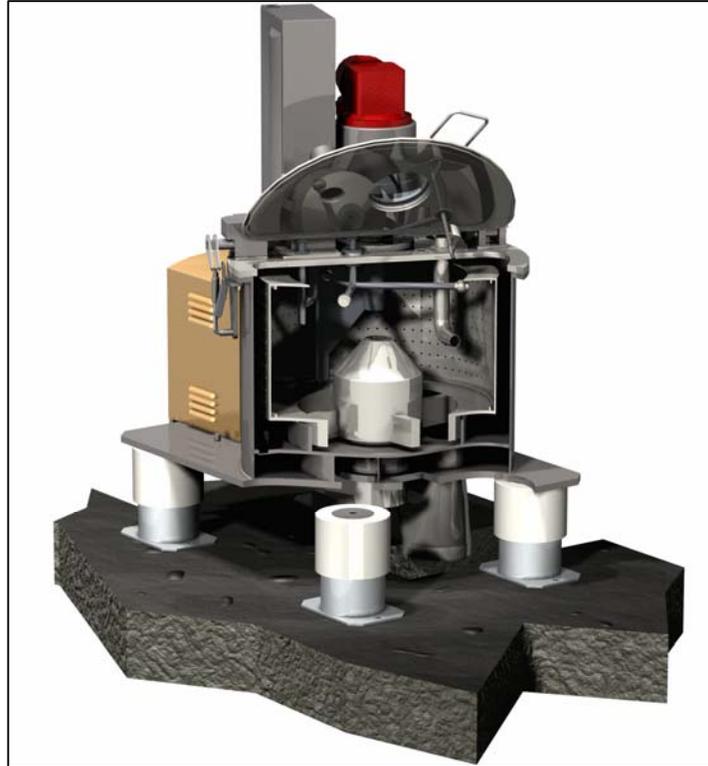
Routine operation of a centrifuge involves certain risks - such as locking access covers when the centrifuge is spinning etc. However additional risks occur when the centrifuge is not used in 'routine operation'. This can occur during : power outages, centrifuge maintenance periods, operation by untrained staff, unexpected behaviour of ancillary equipment etc. There is a growing trend for end users of centrifuges to provide their own control systems and in these cases it is important to have close links with the centrifuge supplier and/or a good knowledge of the non-routine risks to reduce the risks to an acceptable level. Note that if the centrifuge end user provides the control system, the centrifuge manufacturer can only give a Declaration of Incorporation and the end user is responsible for the Declaration of Conformity and CE marking the centrifuge complete with controls.

Risk reduction generally follows several steps. Firstly the risk should be designed out if practical, secondly protective measures should be adopted such as guarding or control interlocks as appropriate. Finally, where no other practical alternative exists, warning labels can be used. Clearly the favoured approach is to design out the risk wherever practical.

Basket integrity is a crucial element in the long term safety of any centrifuge and whilst control systems can ensure the maximum speed of the basket is not exceeded, the risk of a basket or bowl bursting in 'normal' operation cannot be reduced by an electronic control system - it can only be minimised by good mechanical design and manufacture of the basket. The final section on safety below provides basic guidelines on these mechanical aspects of basket integrity.

Centrifuge safety and scale up.

Figure 1. Typical batch centrifugal for the pharmaceutical industry.



Courtesy of Broadbent Centrifuges Ltd.

(c) Basket design & inspection.

BS.EN12547 deals in detail with the design of the basket or bowl of the centrifuge. For a batch centrifuge the basket rotational speeds are typically in the range 1,000-2,000 RPM with small diameter decanters and disc bowl centrifuges rotating at speeds in excess of 4,000 RPM. Failure of the basket whilst rotating at high speed will destroy the centrifuge and possibly anything or anybody near it. It is often assumed that the casing of large industrial centrifuges is designed to act as a containment device in the event of rupture of the basket or bowl. As mentioned in the introduction, the energy stored in a centrifuge basket/bowl at full speed is considerable and the vast majority of centrifuge casings are not designed to contain a ruptured basket. For example a casing made of mild steel would need to be at least 50 mm thick to contain a rupture of a 1.2m diameter basket rotating at 1,200 RPM. Most centrifuge cases are typically 6-12 mm thick depending on the application.

Clearly the basket design must be such as to avoid rupture. BS.EN12547 considers two cases; steady loading and cyclic (or fatigue) loading of the basket. In the case of batch centrifuges, one example of which is shown in figure 1, the centrifuge start/stop cycle (one per batch) produces a cyclic fatigue loading on the basket. The more start/stop cycles a centrifuge performs during its life the greater the significance of fatigue. For example a batch centrifuge in the pharmaceutical industry operating with ten cycles per week for perhaps a 15 year life of the centrifuge equates to 500 cycles per year and 7,500 cycles in total. Other industries, such as the sucrose industry, process at the rate of 20-25 cycles per hour for 23 hours per day and 300 days a year for 25 years. This equates to 155,000 per year and 3,875,000 over the life of the centrifuge. Most centrifuges operate with 5,000 to 50,000 cycles per year and designing to resist fatigue loading is important.

Centrifuge safety and scale up.

Decanter and disc bowl centrifuges are designed for continuous operation and the number of fatigue cycles is correspondingly less. However it is not uncommon for continuous centrifuges to be stopped once per 8 hour shift and to have an operational life of 30 years. This equates to around 30,000 cycles over the 30 year life of the decanter/disc centrifuge.

BS.EN12547 contains specific design guidelines based on 'factors of safety' below yield or tensile strength for the static loading case *but does not contain any specific guidelines* for the cyclic case. The figures above show that designing to avoid fatigue failure is important in all cases - particularly batch centrifuges.

Experience has shown that the design guidelines for the static case normally give an acceptable design for cyclic cases where the number of stress cycles is limited and the materials of construction used are known to have a good fatigue limit. In these cases the simple static design can often be used as a guide for the suitability of a basket subjected to cyclic loading but additional design calculations and/or material type testing may also be required.

There is no automatic method to check a centrifuge basket for impending failure while it is rotating and therefore static periodic manual inspections are required. When designing a basket or bowl it is necessary to consider the expected intervals between inspection - typically a year. Whilst baskets are generally designed for an infinite or extremely long fatigue life the design should be such that if for any reason a fatigue crack becomes visible the day after an inspection it doesn't grow to a point where failure will occur before the next inspection.

Repairs may be necessary during the 15-25 year life of the centrifuge due to erosion, corrosion or mechanical handling damage. Correct repair procedures are vital to ensure that no crack is introduced by an unsuitable repair procedure and that the original design calculations and assumptions are not invalidated by the use of unsuitable repair materials.

Inspection should always be in accordance with the equipment manufacturer's latest procedures, which for an old centrifuge may be different from those included with the original manual. Basket inspection procedures are specific to individual centrifuge types and manufacturing methods. However procedures generally focus on :

- The general loss of material from the basket (e.g. by erosion or corrosion).
- The presence of cracks. Common crack detection methods employed are ultrasonics, magnetic particle inspection and dye penetrant. Any basket found to contain a crack in the basket shell should be taken out of service.
- Damage to any inert coatings.

Basket inspections should be carried out regularly at the specified intervals by qualified and experienced personnel. With good design, maintenance and regular inspection centrifuges of all types can be expected to give many years of safe and reliable operation. For a more detailed discussion on the safety aspects of centrifuges see reference 1.

Centrifuge safety and scale up.

SCALE UP.

This section considers some aspects of scaling up from a laboratory or pilot plant test to full plant scale. The specific points discussed are : (1) The basics of centrifugal filtration, (2) The importance of particle size, (3) Limitations on the maximum size of centrifuges, (4) Representative test samples, and (5) Small scale testing. For a more complete discussion on scale up and selection of centrifuges refer to references 2 to 7 inclusive.

(1) The basics of centrifugal filtration.

An understanding of centrifugal filtration is useful when attempting to scale-up from laboratory or pilot plant trial to full plant scale. This section attempts to provide a basic introduction and makes the assumption that the particles in the solid/liquid mixture to be separated are more dense than the liquid.

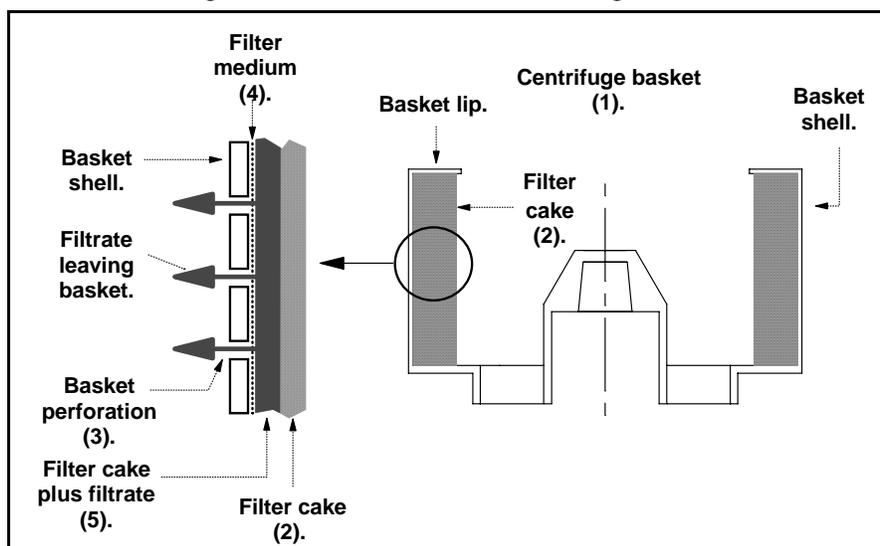
Filtration starts as soon as the feed mixture of particles (solids) and liquid enters the rotating basket (see item 1 in figure 2). As filtration proceeds the particles are pressed together by the centrifugal forces to form a 'cake' (item 2) and the liquor flows through the gaps between particles and then through the holes in the filter screen or cloth (item 4) and out of the basket perforations (item 3) thereby effecting the separation of solids from liquid.

The process of filter cake formation and drainage of the liquor is a complex process occurring in several phases. Only two of the phases will be considered here. These are : Initial Cake Drainage and the approach to Final Dryness. See references 2, 3 & 4 for more details on filtering centrifuges and reference 9 for details on filtration in general.

Initial cake drainage : is taken as that period when the liquor is draining through the bed of particles forming the filter cake and gaps between the particles are no longer all filled with mother liquor. This is shown schematically as item 5 'filter cake plus filtrate' in figure 2.

Approach to final dryness : Is governed by the slow removal of the thin film of liquor adhering to the surface of the particles within the cake after the bulk of the liquid has drained away. Note that non filtration effects such as drying caused by the passage of gas through the cake are being ignored here. See references 1,2 & 3 for more details of filtering centrifuges and filtration in general.

Figure 2. Section of a batch centrifuge basket.



Centrifuge safety and scale up.

Based on a simple analysis the basic dependencies of initial filtration and the approach to final dryness can be derived in terms of the physical characteristics of the feed material (see chapter 7 of reference 2). The results of this analysis give a non rigorous but useful understanding of the physical mechanisms involved and allow the centrifuge user to appreciate the effects of various physical characteristics on centrifugal filtration.

On this basis the initial filtration rate is proportional to :

$$\frac{(\text{Average particle size})^2 * (\text{Centrifuge G})}{(\text{Liquor viscosity}) * (\text{Filter cake thickness})}$$

Eqn 1.

Equation 1 does not take into account the spread of particle size either side of the average, particle shape and the compressibility of the cake formed within the centrifuge.

Regarding the approach to final dryness, the ultimate final moisture content after a long spin time is proportional to :

$$\frac{(\text{Liquor surface tension})}{(\text{Centrifuge G}) * (\text{Average particle size})^2}$$

Eqn 2.

It is clear from equations 1 & 2 that there is the strong dependence on particle size, with a lesser dependence on centrifuge G (i.e. basket/bowl RPM) and other physical properties of the feed material and centrifuge.

(2) The importance of particle size.

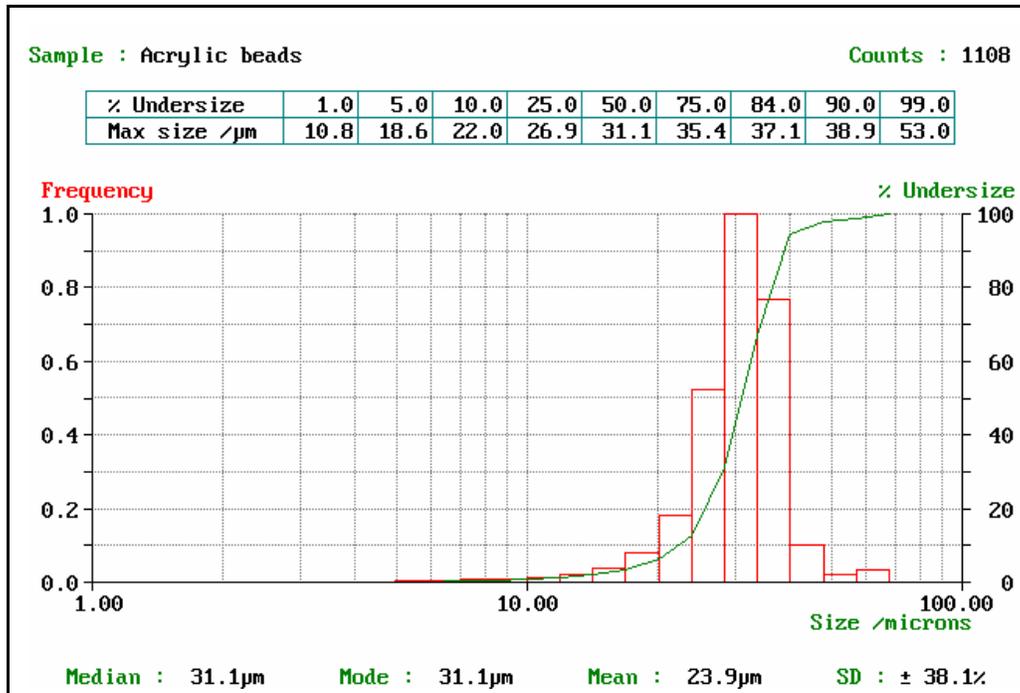
Centrifuge users generally size centrifuges to produce a given mass of the discharged product. Most centrifuges operate by accelerated removal of liquor from the surface of the particles using centrifugal force. So the centrifuge is 'processing' surface area whilst the user is primarily interested in mass of discharge product and the link between the mass and surface area of the particles is the particle size distribution (PSD). This makes the PSD a key parameter for any centrifuge application. As might be expected the particle size distribution - and things that can alter it - are highly significant when predicting centrifuge performance.

The particle size is often quoted as a single figure or range such as a D₅₀ of 80-100 microns and whilst this is useful information it is not the complete picture. Ideally the PSD would be specified as a table or graph showing the proportion of particles present in the sample as a function of size. Figure 3 shows such a distribution where the D₅₀ is 31.1 microns and 10% w/w of the sample has a particle size of 22 microns or less. This additional information is important as it has a large effect on the overall surface area of the sample and therefore a large effect on the separation performance of any centrifuge.

In addition to the size distribution, the shape of the particles is also important. Needle shaped particles have a larger surface area for a given volume, are more difficult to discharge from a centrifuge and are prone to breakage during pumping, feeding or discharging.

Centrifuge safety and scale up.

Figure 3. Typical particle size distribution.



(2) Limitations on the maximum size of centrifuges.

The maximum diameter of a centrifuge is limited by the materials of construction. The basket or bowl material must be strong enough to support itself when spinning empty with any remaining material strength (after due allowance for safety factors etc) being used to support the filter cake and liquor contained within. For a given centrifugal 'G' the proportion of the basket material strength required to support it's own weight increases with basket diameter. So as the basket diameter increases a smaller proportion of the basket material strength is available to support the filter cake and liquor.

For a basket or bowl capable of running at a separating force in the range 500-1000G the practical limit is around 2000-2200 mm diameter for current materials of construction. Similar considerations apply to all centrifuges regardless of type and these basket material limitations explain why the maximum 'G' available from centrifuges drops as the basket or bowl diameter increases.

(4) Representative test samples.

Representative test samples are needed to achieve an accurate scale up from small tests. The ideal situation occurs when a full scale plant already exists, good sampling techniques are followed (see reference 8) and the feed material is stable over time. In practice the small scale sample is usually taken from a pilot scale plant where the feed material volume is limited and produced using different equipment from that planned for the full scale plant. Some of the aspects to consider when taking samples are :

Time delays

Samples that may decompose, recrystallise, agglomerate or otherwise change their physical characteristics over time need to be tested rapidly. In such cases it is generally better to take the centrifuge to the samples rather than the other way round.

Centrifuge safety and scale up.

Sampling techniques.

A great deal has been published about sampling techniques in the chemical industry - see for example reference 8. Efforts should be made to avoid systematic errors caused by sampling at a point that tends to favour the collection of one size of particle over another. Examples of this effect are sampling at the bend of a pipe where large particles predominate at the outside radius of the bend due to centrifugal effects or sampling from a corner of an agitated tank which are often 'dead zones' where there is little mixing.

Design of pilot plant.

It is quite common for samples produced by a prototype small scale pilot plant to be unrepresentative of those produced by a full scale industrial plant. This is likely to result in differences in PSD and/or solids concentration - both of which can lead to errors in scale-up.

(5) Small scale testing.

Scaling up from small scale tests is a judicious mix of theory, experience and black art. Careful testing and detailed analysis minimises the 'black art' element and give the best results. In practice most scale-up investigations are satisfactorily handled by centrifuge vendors or chemical or pharmaceutical company's in-house laboratories. This section lists some of the tests generally undertaken in a typical scale-up investigation. See chapter 7 of reference 2 for a more extensive discussion.

Cake bulk density measurements.

The density of a compacted filter cake in a centrifuge is higher than that of loose packed dry particles but lower than the theoretical solid particle density. Accurate measurement of the density of dried cake within the centrifuge basket is necessary to be able to select a suitable full scale basket volume for the particular application. Measurements are usually performed using a small scale laboratory centrifuge where the weight of the entire basket with and without the cake can be used to calculate the density.

Filtration rate measurements.

Measurement of the filtration rate through the cake per unit area of screen provides information for scale-up of the feed and drying phases of the centrifuge cycle. Correct feeding of a batch centrifuge requires that the rate of addition of the feed liquor is approximately balanced by the feed liquor filtration rate through the cake at the centrifugal 'G' used for feeding.

Once the filtration rate per unit area of screen is known, scale-up to a full sized basket is made by adjusting the feed rate for the larger screen area and thicker cake of the full scale centrifuge.

Cake compressibility and cake thickness.

Filter cakes are often classified as compressible or incompressible whereas in practice all cakes are compressible to some degree. The centrifugal pressure on the particles in the cake forces them closer together reducing the cross sectional area of the pathways for the liquor through the cake and therefore reducing the filtration rate. Equation 1 suggests that filtration rate will increase linearly with 'G' for a given cake thickness; however the increase will be less if the cake is compressible. Measurement of the filtration rate at two or three 'G' levels will provide a clear indication of the degree to which the cake is compressible.

Centrifuge safety and scale up.

Cake discharge tests.

The mechanical discharge of the dried filter cake from a centrifuge is commonly referred to as ploughing or scraping. Testing the ploughing characteristics of the dried cake material is important and test centrifuges fitted with small scale ploughs are available for this purpose. Normally ploughing leaves a residual bed of cake (typically 2-6 mm thick) on the filter screen or cloth (item 4 in figure 2) and unless special actions are taken to remove it before the next batch of feed is processed it will remain in the basket. The residual bed may reduce the cake filtration rate and repeating the small scale test using a basket with the residual bed in place from earlier test(s) allows these effects to be measured and minimized by the use of filter aid etc.

SUMMARY.

This paper discusses some aspects of centrifuge safety and scale up which may be of assistance to users and prospective purchasers of centrifuges. The key points are :

Centrifuge safety :

- BS.EN12547 details the safety requirements for all centrifuges. Special site requirements, use in a hazardous area, etc. require adherence to additional standards.
- A CE declaration is required for second hand equipment that has been modified significantly. Only complete machines can carry the CE mark. Incomplete machines completed by others (e.g. no controls) carry a certificate of incorporation.
- A good understanding of normal and abnormal centrifuge operation is required to design centrifuge controls for safe operation.
- Basket design is a complex issue and BS.EN12547 gives only limited guidance on design for cyclic duties.
- Maintenance and inspection of any centrifuge is necessary for the long term reliability and safety

Centrifuge scale up :

- Obtain representative samples - particularly with regard to the particle size distribution.
- Perform the test as soon after sampling as practical.
- Use the largest test centrifuge possible in the circumstances.
- For filtering centrifuges perform tests to investigate feeding, drying, ploughing etc together with the impact of the residual bed remaining after ploughing.

Centrifuge safety and scale up.

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