lap arising from degree of substitution as well as substituents of diverse chemical structures attached to the monoaromatic as well as polycyclic aromatic compounds.

Future Possibilities

At present, relevant analytical data on crude oil composition are very useful in crude oil processing as petroleum refiners are increasingly using blended feeds instead of limiting the plant operation to a single type of crude oil. The compositional data will be more important as the depletion of light and sweet crude oils continues and refineries process more heavy and sour crude oils. For process control, product quality, catalyst performance and environmental compliance, faster and better analytical approaches to crude oil characterization will become a vital part of the refinery operation. As far as liquid chromatographic methods are concerned, multistep column-liquid chromatography will be less attractive due to limitations such as lengthy turnaround times, high level of uncertainties in the compositional data, excessive solvent consumption and costs involved in their disposal. Modern HPLC, SEC and TLC-FID methods will continue to provide valuable information on crude oils. Most likely, faster and better results will be achieved through major developments in system automation for coupling these techniques with other chromatographic and spectroscopic methods. Such hyphenated techniques are already being pursued to find better approaches to the characterization of fossil fuels, including crude oils.

See Colour Plate 71.

See also: **II/Chromatography: Liquid:** Detectors: Ultraviolet and Visible Detection; Mechanisms: Size Exclusion Chromatography. **III/Flame Ionization Detection: Thin-Layer (Planar) Chromatography. Flash Chromatography.**

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DECANTER CENTRIFUGES IN PHARMACEUTICAL APPLICATIONS

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Centrifugation continues to perform a vital role in pharmaceutical process operations. In the 1990s new enhancements to the technology attracted many new areas of application. Growing demands for higher yields and dryer cell cake, punctuated by the pursuit of lower cost solutions, continue to keep high performance centrifuges at centre stage in the quest for better separation procedures. In addition, tighter regulations and inspection procedures continue to act as catalysts for the development of fail-safe

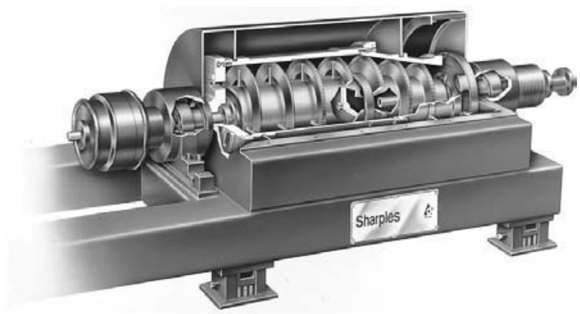

Figure 1 Basic decanter.

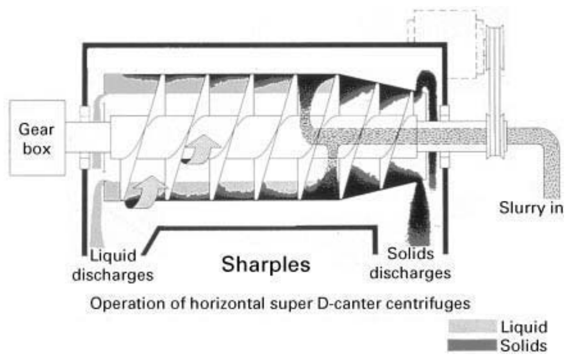

Figure 2 (See Colour Plate 72). General decanter operation.

equipment for sterility and containment to meet more stringent user demands. Within this environment, a decanter (**Figure 1**), which is the most compact type of separation equipment available, is often the centrifuge of choice for producing pharmaceuticals such as erythromycin, insulin (human and animal) and many other large bulk products. These workhorse machines perform a wide range of separation duties, including liquid–solid, liquid–liquid, and liquid–liquid–solid separations and liquid–liquid extractions. Decanters have become most useful for many pharmaceutical operations including recovery and solids purity. These reliable and versatile machines are active in many of industry's typical process steps, e.g. cell harvesting, concentration and washing, cell debris removal, inclusion body recovery and purification, solvent extraction, dewatering and recovery.

Currently, the trend in solid–liquid separation is towards higher solids fractions in the fermentation process. This translates into greater productivity and lower capital costs per pound of product produced. Typical fermentation processes involve amino acids, antibiotics, bacteria, enzymes, ethanol, fungi, inorganic salts, organic acids, polypeptides, polysaccharides, steroids, vaccines, vitamins and yeasts. Decanter centrifuges are also used in extraction and fractionation processes for hormones, alkaloids, blood separation (animal) and natural product isolation.

General Operation

Normally decanters handle two- or three-phase separation of high-solids slurries (**Figure 2**). The slurry enters the centre of the bowl, and denser solid particles are sedimented against a rotating bowl wall. The less dense liquid forms a concentric inner layer. Centrifugal force compacts the solids as they are 'ploughed' out of the pond and up the conical 'beach' by a helical screw conveyor. Solids exit through discharge ports, while clarified liquid or centrate overflows the plate dams situated at the opposite end of the bowl. While decanters can handle a wide range of particle sizes, dryer cake is always produced when particle size is maximized – preferably to 2–10 μm depending upon G-force. It is important to note that in biotech lysed *Escherichia coli* separations, cakes that are 30–40% dry have been achieved with particle sizes of 1 μm or less.

In practise, decanting centrifuges provide excellent performance levels when handling slurries with significant amounts of solids (generally $> 10\%$ by volume), and are often the only choice for solids concentrations above 40% by volume. With appropriate conveyor modifications they can also be used to separate two liquids from each other or from a solid phase. The scroll of the decanter provides a continuous discharge of solids. By interposing nozzles near the beach section it is possible to provide a washing step before the solids are discharged. This method can be used to wash various crystals. In contrast to basket or disc-stack centrifuges,where liquid flow is laminar and flow patterns are relatively simple and contained, the flow patterns and consequent design calculations are more complex for decanters. Separation performance is more subject to analysis by experience and pilot testing.

Vertical Units

Most decanter centrifuges are horizontal, but vertical orientations (**Figure 3**) are also available. The same centrifugal operating principles apply to both types, and process performance is not affected by whether the axis of rotation is vertical or horizontal. The main difference between the two types lies in the seal design. A vertical machine has only one high speed rotating seal plus one low speed (42–52 rpm) or static seal, while a horizontal unit has at least three high speed seals. For this reason, a vertical configuration is

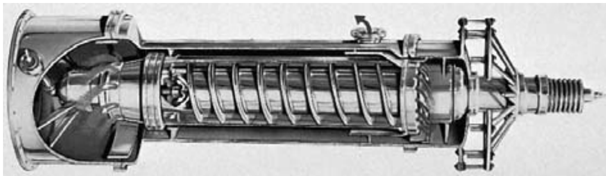

Figure 3 (See Colour Plate 73). Vertical decanter.

easier to seal. With only one sealing surface this unit is often the preferred choice when handling hazardous materials or toxic materials. Vertical centrifuges are often used in high temperature, high pressure applications for handling pressures from vacuum up to 10 bar with process temperatures from -10 to 700°F.

If floor space is a concern, vertical decanters offer distinct advantages over horizontal machines as they require 60–80% less floor area. They also provide more production per square foot with less investment in piping, foundations and real estate.

Factors of Acceptance

For small batch fermentations (100 L or less) membrane technology and mechanical filtration remain the prevalent technologies for processing diverse stream arrays. However, for large volume runs requiring environmental measures and control, decanters are normally specified for several reasons. First, the enclosed seal design of this type of centrifuge type offers inherent advantages. Decanters can be sealed hermetically for the protection of operating personnel, and offer clean-in-place operation and sterilization. Second, the efficient operation of a decanter can produce a dry continually discharged solids phase without contamination of either the product stream or the centrate. Also advancing work on new metallic finishes continues to offer promise for new sterility solutions and safeguards.

Additional factors also work in favour of these machines. Possibly foremost is the growing need to produce dryer cake. As expected, when there is less water solids require less energy for drying. Depending upon the application, decanters can produce dry solids as high as 30–95% for dry solids. The high G-force capabilities of the units can separate out even the finest particles, resulting in cleaner centrate.

The ability to operate at higher G-forces remains a goal. Indeed, high G-force separation continues to expand. Five years ago decanter centrifuges could only generate forces up to 5000 G. During that period disc-stack centrifuges were primarily used for applications, from pilot to full production. Today decanters are capable of 10 000 G operation, making the machines competitive with disc-stack centrifuges for specific pharmaceutical applications.

Continuous operation is another reason why the industry often opts for decanters. The feed and discharge operations (solids and liquids) never cease. There is less downtime and maintenance is substantially lower than with other separation alternatives. At the same time decanters are less affected by feed variations, such as varying particle size, than filter systems which require changing media mesh sizes. The centrifuges also more efficiently separate adhesive, slimy, high viscosity or fluffy materials, which often blind or impair filters. They also provide dryer cake results for these materials than do filters.

Minimal liquid loss is also associated with decanters. Permeable filters lose liquid through absorption as well as losing fluids via filter disposals. This loss becomes critical if a high value is attached to the liquid phase. Also, filters usually separate only two phases simultaneously, requiring another pass for a third separation, while centrifuges are capable of one-step, three-phase liquid–liquid–solid separations (e.g. oil, water, solids).

Improvements to Decanters

Several changes to decanter technology have affected the performance of the machines in the last 10 years.

Settling Vanes

New conveyors have been fitted with unique incline vanes to enhance the collection area of the disc unit. Settling vanes (**Figure 4**) reduce turbulence and stratify feed flow in the clarification zone to promote settling and prevent re-entrainment of solids. This

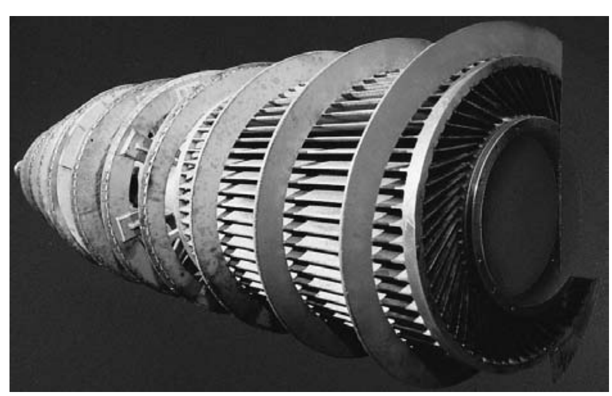

Figure 4 Conveyor with settling vanes.

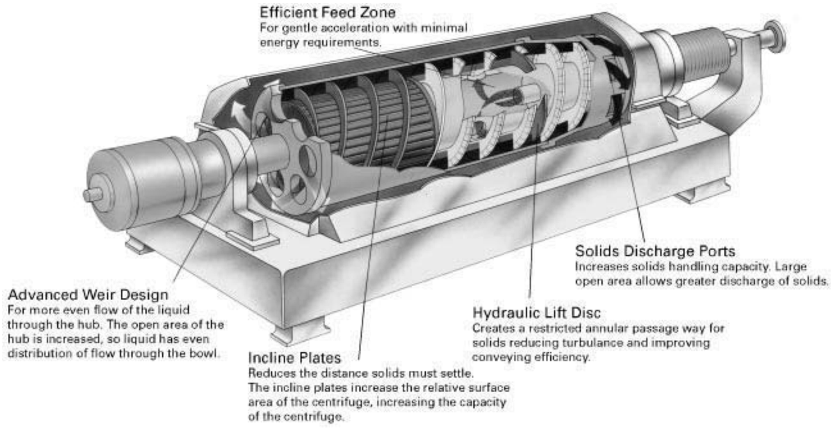

Figure 5 (See Colour Plate 74). Performance-enhancing features of a decanter centrifuge.

results in more efficient solids recovery and a 50% improvement in centrate clarity.

Hydraulic Disc (Baffle Disc)

This feature shown in **Figure 5** enhances the separation of soft, gelatinous materials such as proteins, enzymes, and fermented products. The disc is used to increase the separation efficiency on hard-to-scroll materials (materials that tend to slip on the conical section of a decanter). Acting like a dam, solids build up around the disc, holding in the liquid centrate to provide ultra-deep pond settlings above the level of the solids discharge. The resulting hydrodynamic pressure compacts solids, assists scrolling, and extrudes the compacted solids past the disc and out of the centrifuge.

Dryer Cake: Plough Tile/Conveyor Innovations

New designs now provide purer and dryer cake. Recent innovations permit the centrifugal retention of solids for longer periods of time under maximum G-force. A new conveyor handling compressible solids, such as fibrous materials, has been able to produce up to 35% dryer solids in vitamin, gluten, and cornstarch processing applications.

A newly developed plough tile conveyor (**Figure 6**) uses scoop-shaped tiles to reduce the torque produced by the solids moving through the centrifuge. By lifting and turning the solids from the bowl wall these tiles reduce the torque, produce dryer cake, and increase capacity. The addition of this innovation enables the conveyor to achieve up to twice the throughput of standard conveyors.

Another additional improvement is the 'Kiwi conveyor' (**Figure 7**), a patented flightless conveyor that achieves dryer cake to improve clarity. This unique innovation acts as a catalyst to force newer solids to move old solids out of the bowl.

Efficient Feed Zone

Redesign of the feed zone as shown in Figure 5 has helped to reduce the shear forces on the feed material as much as possible. Shear forces on the liquid stream can cause problems, especially when liquid goes from rest to bowl speed. Upgrade designs now allow for gentle acceleration and entrance into the bowl by means of a low-shear feed zone. This improvement has eliminated the need for acceleration blades.

Variable Speed and Differential Control

Programmable Logic Controller (PDC) and digital controllers are now used to control the large speed swings of a decanter's backdrive motor that are associated with differential control. These systems provide the necessary control features to monitor and stabilize a decanter's eddy current brakes, direct current motor, and alternating current variable

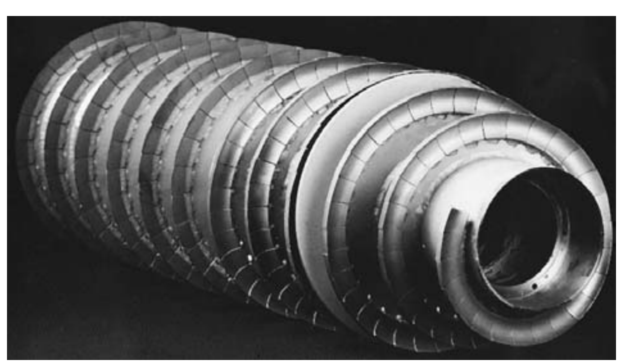

Figure 6 Plough tile conveyor.

Figure 7 'Kiwi' conveyor.

frequency drives to within 0.1 rpm of the desired set points. By maintaining lower differential controls, users achieve dryer cake.

Centripetal Pumps and Containment

Optional centripetal pumps can be added to minimize foaming and aerosols, and gently discharge delicate pharmaceuticals without the risk of oxidation or contamination to the product stream. Such pumps help control liquid cresting and the separation efficiency of the unit.

Vapour Sealant

For applications requiring biohazard-safe, vapour-sealed operation, casing seals are available to contain pressure up to 14.4 kPa inches of water. Lip seals, labyrinth seals, and gas-purged mechanical seals are typically used. If required, decanters can be equipped with steam sterilization for aseptic steam processing.

Clean-In-Place Operation

Self-cleaning units are also available for faster and safer pharmaceutical processing. Built-in motor controls safely reduce rotation to 1 G and automatically clean the decanter's bowl and casing. This eliminates the need to tear down or dismantle the centrifuge.

Conditioning Monitoring

Together with the decanter improvements, the capability to maintain product integrity has been further enhanced by conditioning monitoring programs available from some manufacturers. New software eliminates second guessing and enables users to analyse machines on a continuous basis, e.g. monthly, weekly, etc., by checking for new vibrational patterns due to changes in unit load, balance, or the hydraulic dynamics of the bearing. Conditioning monitoring provides real-time information about the working operation of the centrifuge. Accompanying instrumentation and probes also analyse critical areas such as bearing patterns to note changes, predict failure and prevent unexpected costly shutdowns. Data acquired and stored in the program can also be used to determine what spare parts to keep in stock.

Conclusion

The development of pharmaceutical products requires separation equipment that delivers high recoveries and high solids purity. During the 1990s decanter centrifuges closed the gap on or, depending upon the application, even surpassed the advantages once held by alternative separation devices. In centrifugation, operating costs are notably lower than with filtration devices with capital investment of hardware as the only significant cost. Decanters also require less auxiliary equipment such as secondary pumps, tanks, and extra ventilation machines. Thus large-scale pharmaceutical operations rely on continuous centrifuges to produce maximum possible amounts of dry cake and decanters are well suited to this task.

See Colour Plates 72, 73, 74.

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