FROM BATCH TO CONTINUOUS PROCESSING

SUMMARY

an improved annular centrifugal contactor design is being commercially employed in numerous liquid-liquid extraction applications. It is mechanically driven by a directly coupled motor at relatively low rotor speeds. The combination of interchangeable heavy phase weirs and variable rotor drive makes this centrifuge applicable to a wide range of processes. Single stage efficiencies of 90% or higher are typical for chemical systems with rapid kinetics. Mixing and disengaging times range from 10 to 30 seconds each, dependent on the feed rate to the unit and the unit size. Efficient two phase mixing is achieved in the annulus between the spinning rotor and fixed housing. For versatility, a low mix sleeve can also be used to process shear sensitive liquids, often encountered in washing applications. Annular centrifugal contactors with rotor diameters of 5 to 51 centimetres which range in throughput from 2 to 750 liters per minute are now readily available. The criteria used to select the proper size and operating parameters needed will be discussed. In addition, convenient methods of using this technology to convert **batch to continuous processing** will be given. Advantages in yield improvement and waste minimization will be discussed, and process equipment footprint will be given. Finally, some field examples which describe the versatility of this liquid-liquid centrifugal
contactor will be presented.

INTRODUCTION

General Centrifuge

Clarification of process streams has been one of the niches in the process arena carved by liquid-liquid centrifuges, especially whenever emulsions or liquids close in density have been involved (Davies et. al., 1972). Difficulties that often arise in separation of immiscible liquids include: poor or slow phase separation, emulsion or rag layer formation, and poor process control in batch systems. Centrifuges accelerate separation processes by enhancing the specific gravity differences. Liquid-liquid dispersions requiring hours to separate at 1G will proceed much faster at 100 to 1000 G, with greatly improved efficiency and outflow quality. The efficiency of the physical separation of two phases can be several percent higher using centrifuges versus decanting from tanks.

Contactors as Extractors and Washers

Liquid-liquid centrifuges are valuable separation devices because of their small size and the rapid, yet efficient operation. However, they become even more valuable when employed as liquid-liquid contactors. The ability of a centrifuge to thoroughly mix two phases in the annular zone prior to separation in the rotor broadens its scope. Good mixing is very important to ensure optimal mass transfer and to minimize solvent or water usage. Chemical processes requiring extraction and washing (or neutralization) as well as separation can be performed in one step utilizing liquid-liquid centrifugal contactors. Better process control, low retained fluid volume during processing, and reduced plant space usage are realized when using these devices in place of traditional tanks, mixer settlers, and extraction columns.

ANNULAR CENTRIFUGAL CONTACTORS

History

Annular centrifugal contactor design and development has been pursued by various Department of Energy labs for more than 30 years. It has been employed in solvent extraction processes for metals valuable to the nuclear industry. Commercialization of this technology began in 1990 when a patent was granted for continuous separation of hydrocarbons from water (Meikrantz, 1990). In the past years the centrifuge design has been further improved and scaled up to flow rates of several hundred gallons per minute (Meikrantz et. al., 1997). In addition, a low mixing sleeve which enhances the washing and separation of shear sensitive liquids has been developed (Meikrantz et. al., 1996).

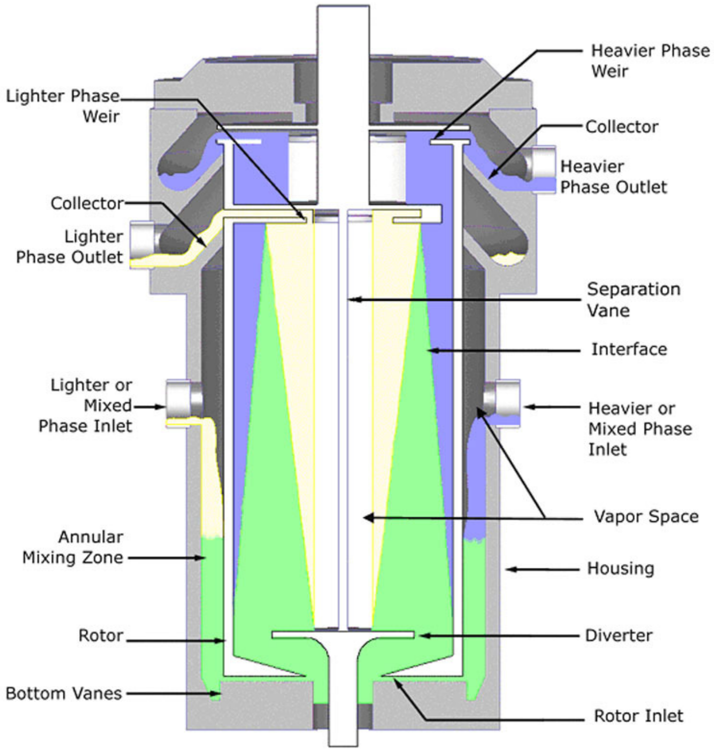
The annular centrifugal contactor possesses many unique design features that distinguish it from other centrifuges on the market today. It has an upright design in which the vertical rotor pumps, thereby feeding itself. A self-pumping rotor maintains separation equilibrium during intermittent feeding because a constant liquid volume is maintained in the rotor. Liquid-liquid separators that require direct feeding to the rotor are not as capable of handling processes where interruptions in flow often occur. Another advantage of a self-pumping rotor is the method by which a process stream is fed to the centrifuge. Because the liquid need only be fed to the annulus, any low pressure pump or gravity feed supply can be used.

COMMERCIAL ANNULAR CENTRIFUGAL CONTACTOR

Principle of Operation

The annular centrifugal contactor operates as both separator and contactor which makes it a valuable tool in numerous types of processes. It's unique design provides mixing and separation in a single, compact unit.

Figure 1. shows a cutaway
view of the centrifuge housing and rotor and details the significant design features including the liquid flow path.



Cutaway View

Figure 1

Two immiscible liquids of different densities are fed to the separate inlets and are rapidly mixed in the annular space between the spinning rotor and stationary housing. Please note that the areas above the liquid levels are vapour space. The mixed phases are directed toward the centre of the rotor bottom by radial vanes in the housing base. As the liquids enter the central opening of the rotor, they are accelerated toward the wall. This self pumping rotor is divided into four vertical chambers which are dynamically balanced by the pumped liquids. The mixed phases are rapidly accelerated to rotor speed once trapped in a quadrant, and separation begins as the liquids are displaced upward by continued pumping. The separating zone extends from the diverter disk to the lighter phase weir, which provides a transit time for the liquid-liquid interface to form and sharpen. The interface should be positioned half way between the lighter phase weir and the heavier phase underflow at the top of the separating zone. This is done by selecting the proper heavy phase weir ring and then adjusting the rotor speed to fine tune position if necessary. Optimum performance is thus achieved despite changes in flow rate or liquid ratios because the interface position can shift a significant distance without loss of separation efficiency. Because the interface is free to adjust in position, it is important to keep the liquid discharges unrestricted in terms of liquid and vapour flow and pressure. Equilibration of pressure between the centrifuge housing, discharge pipes, and receiver tanks ensures trouble free operation over a wide range of process conditions.

Low Mix Option

In process situations where only a two phase separation is being performed or shear sensitive fluids are employed, excess mixing in the annulus needs to be minimized. To accomplish this, a low mixing sleeve can be used, which is a cylinder slightly larger than the rotor. It is permanently attached to the bottom of the housing. By shrouding the rotor, liquids entering the annulus do not come in contact with a high shear surface, but instead enter a primarily static environment. The radial vanes in the bottom of the housing are still present so that the liquid flow path to the rotor is unchanged. Liquid-liquid shear is minimized yet the pumping action of the rotor is not adversely affected. Mixing of the two phases occurs as the liquids are accelerated to rotor speed and pumped. This action is vigorous enough to provide an efficient washing step in many shear sensitive processes.

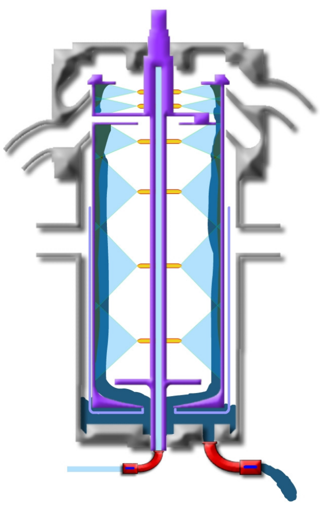
Take Apart Rotor / cGMP Design

Many process streams include small amounts of solids and particulates that build up on the internal surfaces of the rotor even though filtration is used. Eventually these solids will impact the separation efficiency of the centrifuge. Many pharmaceutical and chemical industry applications require thorough cleaning between batches to ensure product purity. Cleaning of the annular centrifugal contactor can be accomplished in two ways. The two litres per minute laboratory scale model has a rotor which can be completely disassembled for cleaning and inspection of the internals. The rotor can be removed from the housing by the operator with simple tools. Removal of the vane
package and heavy phase weir exposes all internal surfaces for cleaning. The frequency of cleaning is dependent on the percentage of particulates in the process stream. These features are also available on the next larger model which processes up to 20 liters per minute. Both units utilize a rotor suspended from the upper bearing housing to enhance disassembly and simplify the design. Good manufacturing practice requirements for these centrifuges are readily addressed by the use of castings to eliminate welds or crevices and by the ability to inspect all wetted areas.

Clean-In-Place Rotor

Figure 2

A hollow through-shaft is employed which starts below the bottom plate of the housing and extends into the upper rotor assembly. It is equipped with a series of high pressure spray nozzles for each quadrant. These nozzles provide complete coverage of the internal wall of the rotor, the aqueous underflow, and the upper rotor assembly. A rotary union that is permanently attached to the tail shaft provides the inlet for the desired cleaning solution and allows the cleaning process to be fully automated. The process steps for cleaning are quite simple. Product feed to the centrifuge is halted and the rotor is stopped, which drains the hold up volume into the annulus. Next, draining the process liquid from the centrifuge exposes all the internal rotor surfaces to the cleaning solution spray. Cleaning solution is then pumped to the centrifuge via the rotary union until the unit is clean. After sufficient cleaning, the process is reversed and the centrifuge is put back in service. The total operation is performed in minutes requiring no disassembly of the unit or connection and disconnection of supply lines.



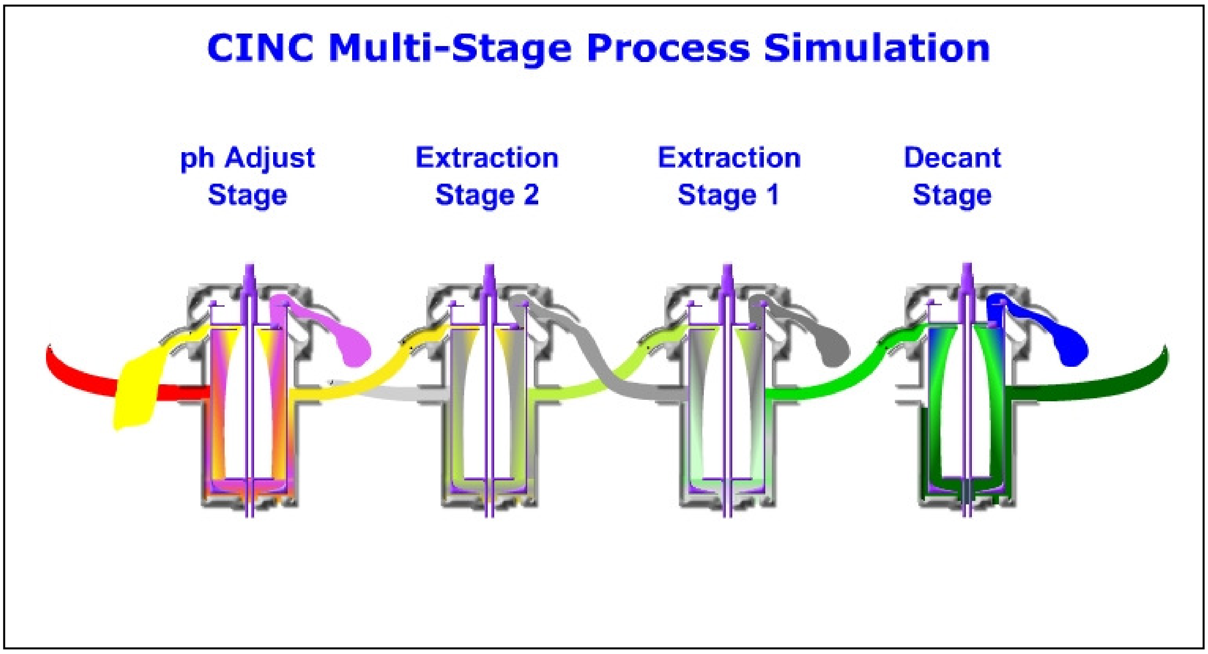
Processing Principles

The annular centrifugal contactors are low rpm, moderate gravity enhancing (100-1000 G) machines, and can therefore be powered by a direct drive, variable speed motor. The effectiveness of a centrifugal separation can be easily described as proportional to the product of the force exerted in multiples of gravity (G) and the residence time in seconds or g -seconds. Achieving a particular g -seconds value in a liquid-liquid centrifuge can be obtained in two ways: increasing the multiples of gravity or increasing the residence time. Creating higher g force values for a specific rotor diameter is a function of rpm only, which is limited by direct drive motor capabilities.

Multi Stage Process

Figure 3

A further example of a multistage process is given in Figure 6. In this case, six inter-connected stages provide a continuous metal extraction, scrub, and strip process. No intermediate pumps or tanks are required for the continuous phase as it traverses the complete separation. A 90% efficiency is assumed and a 1:1 aqueous to organic ratio is used to quantify the interstage metal concentrations in the 3 stage extraction part of this process. Counter current flow in both the extraction and strip stages is employed to gain maximum efficiency while minimizing reagent usage. Counter current flow in both the extraction and strip stages is employed to gain maximum efficiency while minimizing reagent usage.



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FIELD APPLICATIONS

A good example of a commercial installation took place in April, 1997 in Arkansas. An annular centrifuge contactor was installed as the first step in converting a batch process to a continuous operation following the reaction sequence. It replaces a 4,000 gallon decant tank by efficiently separating the brominated polymer product from the aqueous waste at the rate of 45 liters per minute.

The increase in efficiency thus gained has been measured as a 3% improvement in product recovery, which represents 136,000 kilograms of brominated polymer worth $400,000 per year. The contactor has been operating continuously without problems while being fed in batch mode from the multiple production reactors. In addition, off normal, emulsified product batches which previously were processed off-line are no longer a concern. The enhanced separation power of the annular centrifugal contactor operating at 300 times gravity processes all product rapidly and efficiently.

A second Hastelloy C-276 contactor has been purchased for the next process step, hydrochloric washing of the polymer phase. When installed, this unit will remove unreacted amine from the product and will enhance the recycling of this starting material. Coupling the second unit to the first will be simple and will make the process even more cost effective to operate.

CONCLUSIONS

Annular centrifugal contactor designs of this type are a significant improvement over traditional methods of liquid-liquid processing. Increased productivity from continuous or simultaneous multiple step processes as well as improved finished product quality from better process control is realized. Rapid and efficient separation prevents significant product loss at the liquid-liquid interface and from unwanted reactions resulting from prolonged contact times. Multistage separations and extractions utilizing annular centrifugal contactors not only minimize water and liquid reagent usage but also occupy a minimum of floor space compared to the alternatives. Low maintenance due to moderate operating speeds and ease of cleaning means downtime is reduced thereby maintaining process efficiency.

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