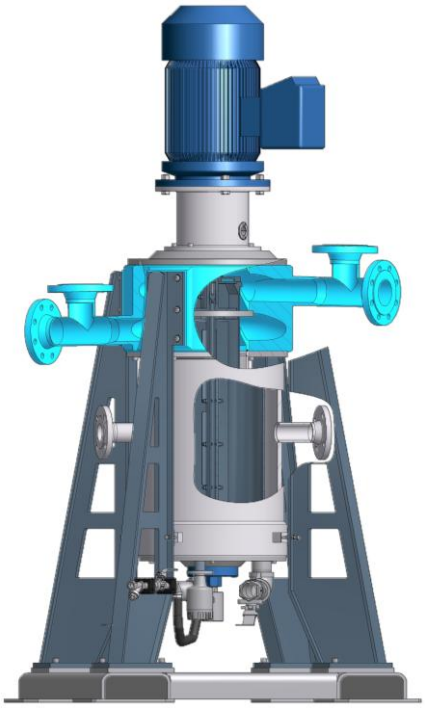
CINC Deutschland GmbH & Co. KG

Annular centrifugal extractor
FROM BATCH TO CONTINUOUS PROCESSING



1. 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. Annular centrifugal contactors with rotor diameters of 5 to 50 centimetres which range in throughput from 2 to 750 liters per minute are available. The criteria used to select the proper size and operating parameters needed will be pre tested with the lab. scale piloting units. In addition, convenient methods of using this technology to convert **batch to continuous processing** will be given.

2. INTRODUCTION

2.1 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. 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 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, mixer settlers or extraction columns.

2.2 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.

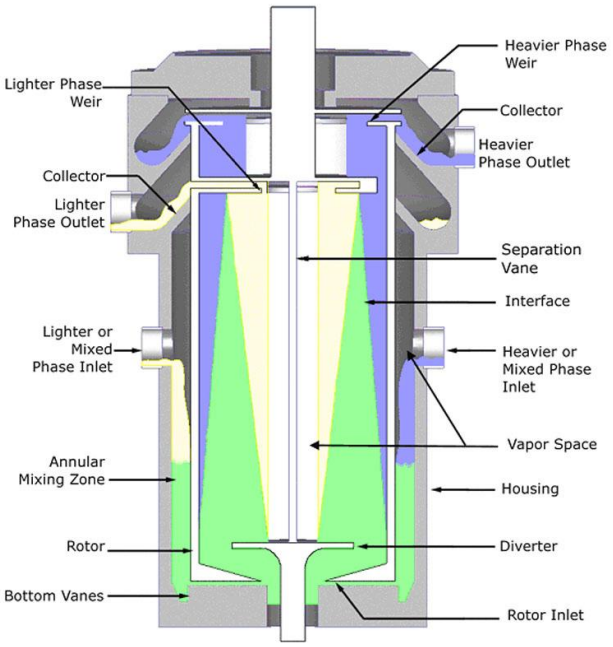
3. Principle of Operation

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.

3.1 Cutaway view

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



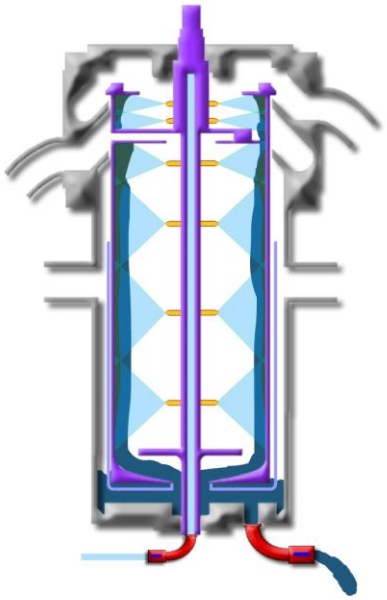
Two immiscible liquids of different densities are fed to the separate inlets and are rapidly mixed in the annular space between the spinning rotor and stationary housing. Please note that the areas above the liquid levels are vapour space. The mixed phases are directed toward the centre of the rotor 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.

3.2 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 models which process up to 120 liters per minute. All 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.

3.3 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.

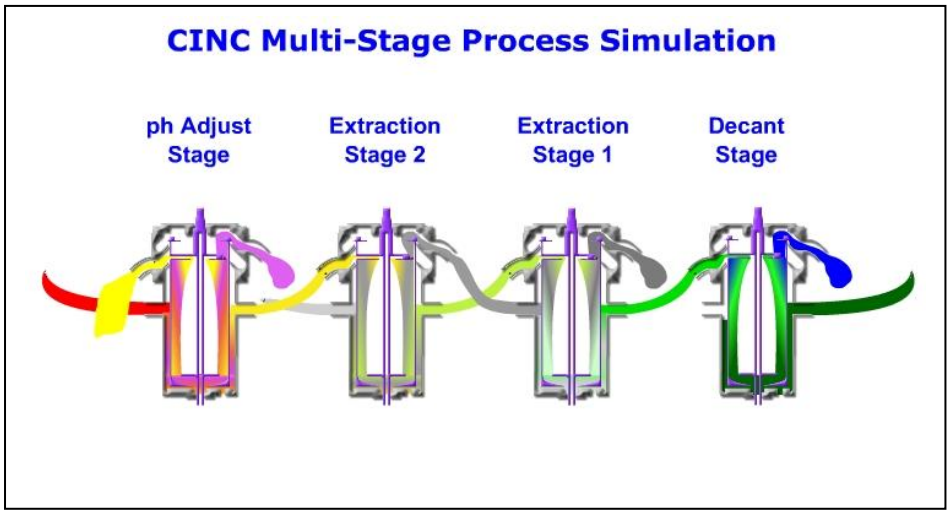
3.4 Processing Principles

The annular centrifugal contactors are low rpm, moderate gravity enhancing (100-2000 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.

3.5 Multi Stage Process

Figure 3

An example of a multistage process is given in Figure 3. In this case, four inter-connected stages provide a continuous metal extraction, scrub, and strip process. No intermediate pumps or tanks are required for the continuous (from the right side) decant, counter current extraction and last a pH cross current extraction or wash.



4 FIELD APPLICATIONS

4.1 Piloting:

Effective piloting can be insured with the CS 50 lab scale continuous centrifugal extractor, the gained physical and chemical characteristics of the relevant liquids will be used to upscale to process size. This way of piloting ensures quick reliable process data and confidence into the technology.

Figure 4 shows a three stage counter current extraction.

Example “Steroid extraction”

Very effective is the production of active pharmaceutical ingredients in fermentation processes. The active pharmaceutical ingredients have to be extracted with organic solvents like

Ethylacetat $H_3C\text{-}OOC_2H_5$ Density 0,9 kg/l Boiling temperature 77 ^oC.

The efficiency is dependant on the selectivity of the organic solvent.

4.2 Pre test: Shaking test

Shaking test can be used to determine the required theoretical stages. The customer used a ratio of 1/4 . The final concentration of the active ingredient in the fermentation broth was set to < 0,05 g/l.

Tab. 2: Shaking test 1/4

Test	Temp.	Ethylacetat	Fermentation broth	Conz. before	Conz. after	Faktor
1	20^{o}C	400 ml	1600 ml	0,94 g/l	0,30 g/l	Faktor 3,13
2	20^{o}C	400 ml	1600 ml	0,30 g/l	0,1 g/l	Faktor 3
3	20^{o}C	400 ml	1600 ml	0,1 g/l	0,02 g/l	Faktor 5

4.3 Piloting CS 50:

The process was piloted with 3 CINC lab. scale centrifuges. Each centrifuge is a centrifugal extractor and mixes both liquids for the mass transfer outside the rotor and separates the liquids inside the rotor. Each unit represents one theoretical stage. The phase separation achieved was excellent and the liquid ratio was given by the customer with 1/5. The extraction efficiency was measured after each unit.

Tab. 3: CS 50 Piloting 1/5 (ethylacetat / fermentation broth) at 1000 g

Test	RPM	Temp.	Flow - Ethylacetat	Flow Fermentation-broth	Conz. before	Conz. after	Faktor
1	6000 RPM	20°C	100 ml/min	500 ml/min	0,96 g/l	0,35 g/l	Faktor 2,74
2	6000 RPM	20°C	100 ml/min	500 ml/min	0,35 g/l	0,11 g/l	Faktor 3,18
3	6000 RPM	20°C	100 ml/min	500 ml/min	0,11 g/l	0,03 g/l	Faktor 3,66

4.4 Up scaling

The required residence time for a complete separation of both liquids inside the rotor was calculated with the max. achievable flow rate at the tested 1000 g.

The volume of the lab scale rotor is 140 ml.

Residence time = rotor volume / flow rate = 140 / 600 x 60 = **14 sec.**

Flow rate required is 3500 l/h = 58,3 l/min.

Required rotor volume = flow rate / 60 x 14 = **13,6 l**

The CS 250 has a rotor volume of 20 l and is the next available size.

Mixing will be intensified with increasing radius because the outer rotor speed is 2 x pi x r x RPM / 60.

Linear velocity at 1000g = 6000 RPM with a CS 50 (radius 25 mm) = 16 m / sec

Linear velocity at 1000g = 6000 RPM with a CS 250 (radius 125 mm) = 35 m / sec

Tab. 4 Mixing

Modell	RPM 1000 g	Rotor Diameter [m]	m/sec at 1000 g
CS 50	6000	0,0508	**16**
CS 125	3600	0,127	24
CS 250	2600	0,254	**35**
CS 400	2040	0,406	43
CS 500	1800	0,508	48

4.5 Prozess

The process was installed with 3 x CS 250 in the EEx Zone 1 with the safety features (vibration, bearing noise, level control und RPM sensor) and 30 mbar Nitrogen blanket.

Startup results Tab. 5: CS 250 ratio 1 / 4 at 1000 g

Stage	RPM G - force	Temp.	Flow - Ethylacetat	Flow Fermentation-broth	Conz. before	Conz. after	Faktor
1	2700 U/min	20°C	800 l/h	3200 l/h	0,94 g/l	0,21 g/l	Faktor 4,47
2	2700 U/min	20°C	800 l/h	3200 l/h	0,21 g/l	0,05 g/l	Faktor 4,2
3	2700 U/min	20°C	800 l/h	3200 l/h	0,05 g/l	**0,01 g/l**	Faktor 5

The max. concentration after the extraction was given to < 0,05 g/l. At the ratio 1/4 this value was far excided. To minimise the solvent required different ratios had to be tested.

Startup results Tab. 6: CS 250 ratio 1 / 5 at 1000 g

Stage	RPM G - force	Temp.	Flow - Ethylacetat	Flow Fermentation-broth	Conz. before	Conz. after	Faktor
1	2700 U/min	20°C	800 l/h	4000 l/h	0,94 g/l	0,25 g/l	Faktor 3,76
2	2700 U/min	20°C	800 l/h	4000 l/h	0,25 g/l	0,09 g/l	Faktor 2,78
3	2700 U/min	20°C	800 l/h	4000 l/h	0,09 g/l	**0,01 g/l**	Faktor 9

Startup results Tab. 7: CS 250 ratio 1/6 at 1000 g

Stage	RPM G - force.	Temp.	Flow - Ethylacetat	Flow Fermentation-broth	Conz. before	Conz. after	Faktor
1	2700 U/min	20°C	650 l/h	4000 l/h	0,94 g/l	0,40 g/l	Faktor 2,35
2	2700 U/min	20°C	650 l/h	4000 l/h	0,40 g/l	0,15 g/l	Faktor 2,67
3	2700 U/min	20°C	650 l/h	4000 l/h	0,15 g/l	**0,02 g/l**	Faktor 7,5

Startup results Tab. 8: CS 250 ratio 1/8 at 1000 g

Stage	RPM G - force	Temp.	Flow - Ethylacetat	Flow Fermentation-broth	Conz. before	Conz. after	Faktor
1	2700 U/min	20°C	500 l/h	4000 l/h	0,94 g/l	0,49 g/l	Faktor 1,9
2	2700 U/min	20°C	500 l/h	4000 l/h	0,49 g/l	0,22 g/l	Faktor 2,2
3	2700 U/min	20°C	500 l/h	4000 l/h	0,22 g/l	***0,08 g/l***	Faktor 2,75

5 Conclusions

The shaking test of the customer and the piloting resulted in the specific separation and extraction characteristics of these liquids.

After the piloting the customer calculated that the process would be very economical due to the extraction results and ordered the 3 centrifuges.

The process start up is even more economical because at the process ratio of 1/6 he saves 33% in the recovery of the organic solvent.

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