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Scale-up of Liquid / Liquid Centrifuges

ABSTRACT

Liquid / liquid separations and extractions are complex processes and several parameters determine the type of equipment to be used. Centrifugal contactors are one of the technologies employed for liquid / liquid separation and liquid / liquid extraction processes. Every liquid / liquid system has its own specific characteristics and in order to successfully predict behavior, testing must be performed with small scale equipment to predict industrial performance. Parameters such as surface tension, viscosity, temperature, mutual solubility, selectivity, and liquid densities are a few of the factors which influence the performance of a liquid / liquid centrifuge.

Process intensification studies can be performed using laboratory scale centrifugal contactors to evaluate these parameters. One of the goals of process intensification is to make large changes in operating conditions on a laboratory scale. By imparting large changes on a small scale, the robustness of the equipment can be determined. If the centrifugal contactor can tolerate the large change on a small scale, confidence in scale-up to larger scale equipment is increased. The different mixing configurations of centrifugal contactors influence equipment performance, and the characteristics of a liquid / liquid system determine the details of equipment design.

Pilot scale testing is performed with larger equipment to perform more thorough and specific testing. At this point, the process is "fine-tuned" by evaluating conditions such as the phase flow rate ratio, the maximum throughputs that can be attained, and range of acceptable rotational speeds, for example, to accurately predict industrial throughputs and maximize product recovery.

WHAT IS THE PROCESS GOAL?

The first step in development of a liquid / liquid separation or extraction process is to know the ultimate process goals, and to determine the extraction equipment that will be the most practical and economical to satisfy the process requirements. Knowing the industrial goals alone can be a limiting factor as to the equipment to be implemented:

What are the desired industrial throughput requirements?

What are the steps required in achieving the end-product? Is only extraction to be implemented? Or are impurities present that need to be removed, for example?

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What are the environmental and health hazards associated with the process? What containment levels are required?

What are the economic justifications?

Asking these questions alone may determine the type(s) of equipment to be used in an extraction process.

PRELIMINARY LABORATORY TESTING

When choosing a solvent for a solvent extraction process, it is desirable to have a solvent that yields a high distribution coefficient [>1, preferably >>1]. The higher the distribution coefficient of the desired solutes in the selected solvent in comparison to the parent liquid, the less solvent will be required to perform the extraction, and fewer theoretical extraction stages are required to achieve the desired recovery of solute. The distribution coefficient of any solvent can be quickly determined by performing separatory funnel tests with equal volumes of solvent and feed material, and using the Kremser equation. These tests can be used to construct an equilibrium curve for the system and McCabe-Thiele plots can be constructed to determine the best combination of solvent / feed ratio and number of theoretical stages.

To determine if a centrifugal contactor is a fit for the liquid / liquid system, it is necessary to perform some simple laboratory tests:

Gravity shake testing – This testing can be performed in a test tube, separatory funnel, or other simple laboratory glassware. The two liquid phases should be shaken vigorously for 5-10 seconds, and then allow the two liquid phases to separate by gravity. Note the time that it takes to, see two distinct liquid phases, and note the time when all interfacial emulsion is gone.

As a rule of thumb, if this gravity settling process takes less than 10 minutes, it is a good candidate for a centrifugal contactor. If it takes less than 1 hour, it is a marginal candidate for a centrifugal contactor. If it takes several hours to separate by gravity, it is not a good candidate, and other liquid / liquid separation equipment is better suited for the process.

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Densities of the liquids – The densities of the two liquids to be processed must have a difference of 0.05 or greater for the process to be feasible with a stagewise, discrete liquid / liquid centrifugal contactor.

LABORATORY TESTING WITH A CENTRIFUGAL CONTACTOR:

Bench scale centrifugal contactors are offered in throughputs ranging from 2 liters / hr [micro scale] to several liters/min [macro scale]. With the low throughput requirements, it is possible to perform a variety of experiments while minimizing the consumption of valuable R & D materials. One of the major advantages of a centrifugal contactor is that equilibrium and steady state operation are achieved rapidly, and this further reduces the amount of material consumed during testing.

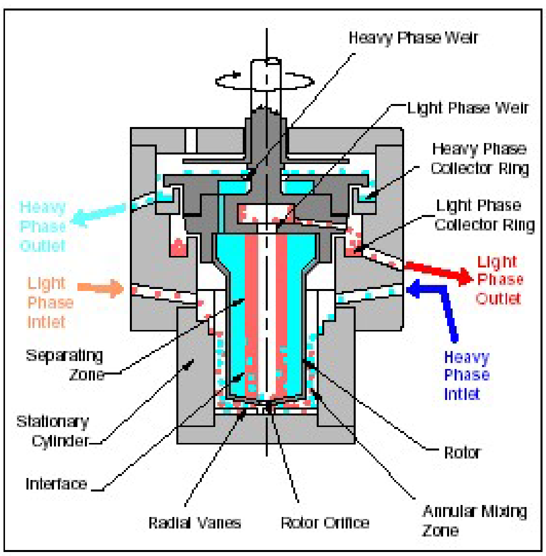

Fig. 1: Rousselet-Robatel annular centrifugal contactor

When testing a centrifugal contactor on a laboratory scale, there are two important factors to study:

1) **Analytical results**:

What is the stage efficiency when compared to the theoretical results? Typically, stage efficiencies of 90% or greater are possible with centrifugal contactors. Samples can easily be taken and analyzed during testing to determine the results.

A major benefit to centrifugal contactors is that countercurrent multistage extraction can be evaluated on a laboratory scale. Traditionally, lab tests have been performed cross-currently using fresh solvent with each extraction. This traditional laboratory extraction consumes excessive quantities of solvent and yields a more dilute extract, whereas centrifugal contactors can be installed in series to perform 2, 3, 4 stages, or more of countercurrent extraction.

Centrifugal contactors can be used on the lab scale for performing batch or continuous extractions. The ability to perform continuous processing on a laboratory scale is a major advantage of the centrifuge.

It is possible to rapidly evaluate changes in phase flow rate ratio and optimize this parameter on a lab scale.

2) Hydraulic performance:

It is important to determine the highest possible throughputs that can be processed through the centrifuge. This is a function of several variables, and following are some of these variables:

- Process temperature
- Liquid viscosity
- Phase continuity
- Surface tension
- Centrifuge rotational speed
- Feeding methods

Process temperature: Process temperature can affect both the hydraulic and analytical results of a centrifugal contactor test program. Changes in process temperature can change the distribution coefficient of a system. This can also change solubility of the two liquids with each other. At the laboratory scale, it is recommended to change the process temperature as widely as possible, as a part of process intensification, to determine the robustness of the centrifuge. Determine the widest change of process temperature swing that the centrifuge can tolerate.

Typically, there is a 1°C temperature rise in process fluids through a single stage centrifugal contactor. As a process intensification step, the centrifuge should be

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operated at 1/10th the nominal capacity, or less, to maximize the affect of temperature as it is magnified as a function of lower throughput.

Liquid viscosity: Typically, the more viscous that any one of the liquids is, this will reduce the maximum throughput possible through the centrifuge.

Phase continuity: This has both hydraulic and mass transfer ramifications. For example, an organic continuous phase [droplets of aqueous dispersed in the aqueous phase] may separate more quickly than an aqueous phase [droplets of organic dispersed in the aqueous phase]. The system that separates more quickly will allow for higher throughputs in the centrifuge. However, one phase continuity may yield better mass transfer. In a liquid extraction system, it is typically desired to have the extracting phase be the continuous phase.

If the phase flow rate ratios are low, it is possible to start up the centrifuge favoring one phase continuity versus the other. This is a good method of testing the robustness of a system. What effect does phase continuity have on the overall process? If the flow rate of one phase is suddenly reduced to 1/10th its target level, e.g. what is the hydraulic result?

Rotational speed: This is an important parameter to evaluate for hydraulic, and analytical reasons. Typically, there will be a range of acceptable rotational speeds that yield good phase separation. There are two dynamics taking place in a liquid / liquid centrifugal contactor; mixing / pumping of the phases, and separation of the phases. Faster rotational speeds are not always better. Because mixing energy is also increased when the rotational speed is increased, higher speeds may cause a stable emulsion to form that cannot be separated. It is important, as part of process intensification, to find the greatest range of acceptable rotational speeds. As a second process intensification step, make sudden changes to rotational speed during processing. Identify the effects on both hydraulic and analytical performance. An important test is to stop the feeds, stop bowl rotation, and start again with the held up liquids in the casing, to determine how quickly the system recovers from the upset.

Feeding methods: Centrifugal contactors can be fed by pumps or gravity. Fluctuations on feed rates [such as pulsing] can affect the results obtained. At the lab scale, it is important to maximize the change in feed rate, to determine the robustness of the centrifuge.

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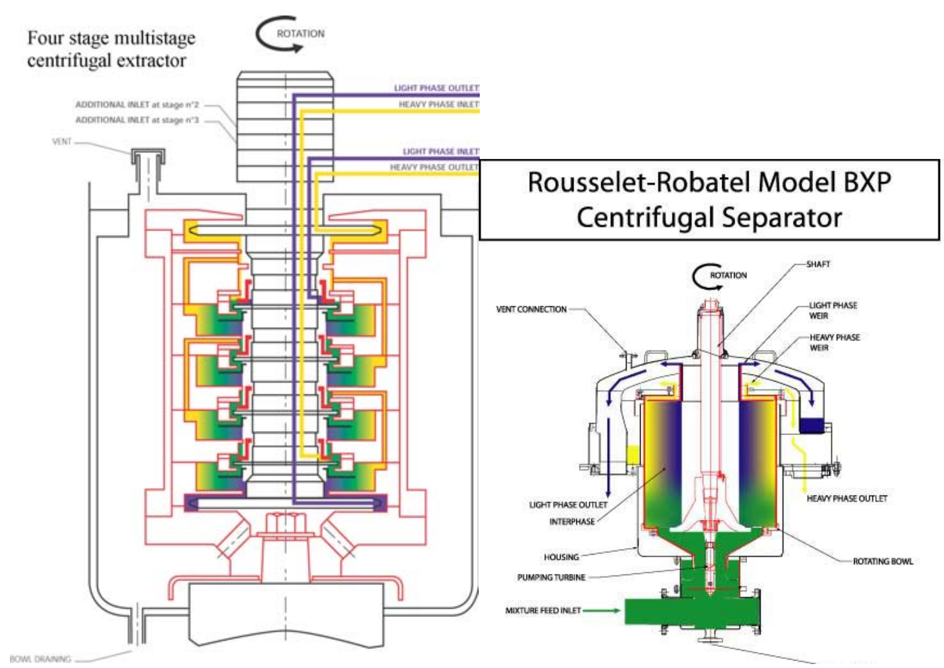
PILOT SCALE TESTING

At this stage of the process development, the goal is to test at a small scale [typically ½ to 1/10th the industrial throughput].

The parameters evaluated on the lab scale are "fine-tuned" to determine the range of optimum operating conditions. These parameters include flow rates, centrifuge rotational speed, and phase flow rate ratios.

However, it should be noted that it is possible, and frequently practiced, to proceed directly from preliminary laboratory tests to a pilot scale. If it is desired to jump directly to pilot scale tests, then it is necessary to perform the same sets of process intensification steps that are highlighted in the **Laboratory Testing** section, which was previously discussed.



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At the pilot scale, it is possible to optimize the heavy phase weir selection. The heavy phase weir diameter in a larger scale centrifugal contactor is chosen based on the density ratio of the liquids and the phase flow rate ratio. In cases where the density difference is low, it may not be possible to further optimize the phase flow rate ratio. All possible heavy phase weirs, that may be acceptable, should be tested to determine the affect of fluctuations in densities of the liquids during processing.

If a liquid extraction process is shear sensitive, or if strictly liquid / liquid separation is the only process goal, then it is necessary to evaluate different pumping configurations. Liquid centrifugal contactors can be equipped with "low shear" pumping configurations to minimize the mixing of liquids and maximize the separation ability.

Some of these low shear configurations include a partially immersed cone, which gently accelerates the two liquids up to the rotational speed of the bowl, and does not cause aeration of the liquids. It is important to evaluate the pumping configuration on a pilot scale, as the pumping designs are very limited on a laboratory scale.

Long duration testing should be performed on a pilot scale. Pilot campaigns should be performed for 24 hrs, or several days continuously to determine if there are buildups of impurities, for example.

If solids are present in the process, how long can the materials be processed before the solids accumulate to a level where the centrifuge does not adequately separate the liquid phases? Pilot scale centrifuges can be equipped with clean-in-place systems to remove the solids, and these cleaning systems can be tested on the pilot scale.

Centrifugal contactors can be fabricated from common alloys such as 316L stainless steel, and exotic alloys such as Hastelloy C. They can also be fabricated from fluoropolymer materials such as Kynar for corrosive processes. It is important to test on a pilot scale with metallic construction, if metallic construction is foreseen on the industrial scale. Metallic centrifuges are typically wetted by aqueous phases, whereas polymeric materials are wetted by an organic phase. This can have a tremendous impact on the separation ability of the centrifuge.

CONCLUSION

It is possible, through both laboratory and pilot scale testing to accurately predict the behavior of a liquid / liquid system on an industrial scale. By maximizing the process changes on a smaller scale, it is possible to predict the [smaller] changes on a large scale.