Development of a new annular centrifugal solvent extraction contactor

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Abstract – *CEA has conceived a basically new annular centrifugal contactor referred to as ECRAN (initials of French words for centrifugal extractor with filled agitated rotor) with two original designs :*

- there are two rotors driven by the same shaft and positioned the one under the second; the upper accomplishes the separation of the phases, the lower the mixture. Depending on process operation, it is possible to design a separation rotor with high diameter and high centrifugal force and a mixing rotor allowing good mixing at a given rotor speed,

- the two phases are fed at the bottom of the mixing zone by means of two channels and discharge into the separation rotor after mixing ; so the emulsion fills all the mixing zone and its volume is constant at any experimental conditions.

INTRODUCTION

Since 1973, few miniature annular centrifugal solvent extraction contactors have been conceived and developed essentially by American, Chinese and French researchers [1], [2], [3]. All these contactors are composed of an outer stationary cylinder and a suspended inner rotating cylinder; the phases are mixed by skin friction between the two cylinders.

Nevertheless, the experience of CEA demonstrated that this type of equipment have two deficiencies; first the mixing hold up is dependent of flowrates, rotor speed, material of cylinders (wettability); second, it cannot be used in process situations where a low mixing and a high-capacity separation are simultaneously needed.

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PRINCIPLE DESIGN

The main features of a single stage contactor are shown in Fig. 1.

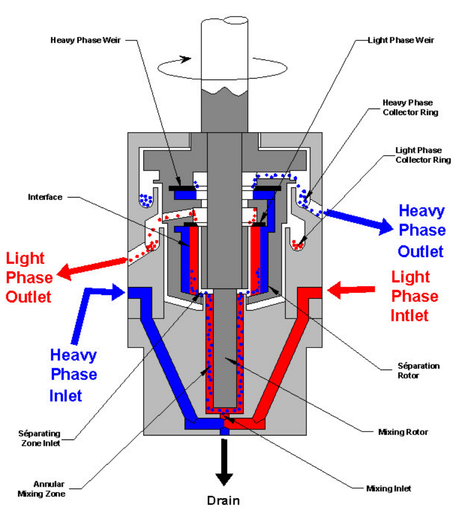

Fig. 1. Schematic of ECRAN

Aqueous and organic phases are fed at the bottom of the mixing zone by means of two channels. They are pumped by the mixing rotor and flow into the Couette mixing zone which is the annular region between the mixing rotor and the stationary cylinder where they are mixed by shearing.

Then the emulsion discharges into the separation rotor where it breaks rapidly under the centrifugal force (up to 1,000 g with rotor speed of 8,000 rpm and diameter of 30 mm)

The separated phases flow over their respective weirs and are thrown by centrifugal force from the rotor into their respective collector rings in the housing, from which they flow through tangential exit ports.

Actually, the maximum inner diameter of the housing is 12 mm the minimum diameter of the mixing rotor is 3 mm; so, the gaps are between 0.5 and 4.5 mm.

As the CEA conventional annular extractor, the multistage group contains single stages supported on two rails and intermediate parts links exit ports and their corresponding inlet ports.
A twostages group is shown in Fig. 2.

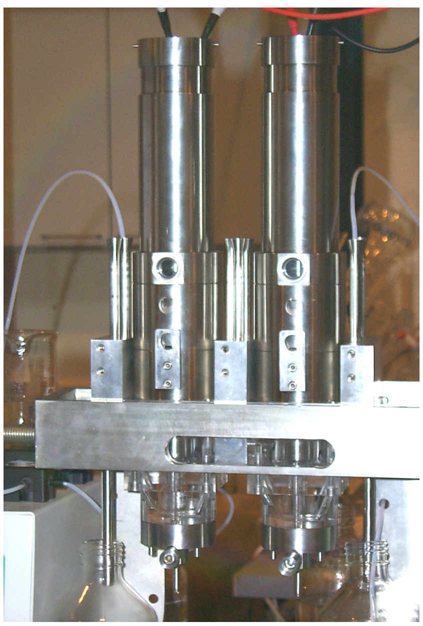

Fig. 2. Assembly of two ECRAN

MASS TRANSFER TESTS

A prototype has been built and a number of tests were performed to measure the extraction efficiency : HNO_3 was extracted in 30% tri-n-butyl phosphate (TBP) in hydrogenated tetrapropylene (TPH) from an aqueous feed of 4.5 M HNO_3. The runs were conducted at A/O 0.2, total throughputs ranging from 0.2 to 7.2 $L.h^{-1}$, rotor speed between 4000 and 5000 rpm and different rotors with diameter from 4 to 8 mm.

All samples were analyzed using potentiometric titration.

Hausen efficiency is defined as the amount of nitric acid actually extracted divided by the amount of nitric acid that would be extracted if the two phases were in equilibrium.

A shown in Fig. 3, the extraction efficiency exceeded 95% with total throughputs up to 3.6 $L.h^{-1}$ and rotor speed of 5,000 rpm if the gap was 1.5 mm corresponding to a mixing hold up of 2 mL (hold up of conventional laboratory centrifugal extractors); then the efficiency decreased when throughputs increased.

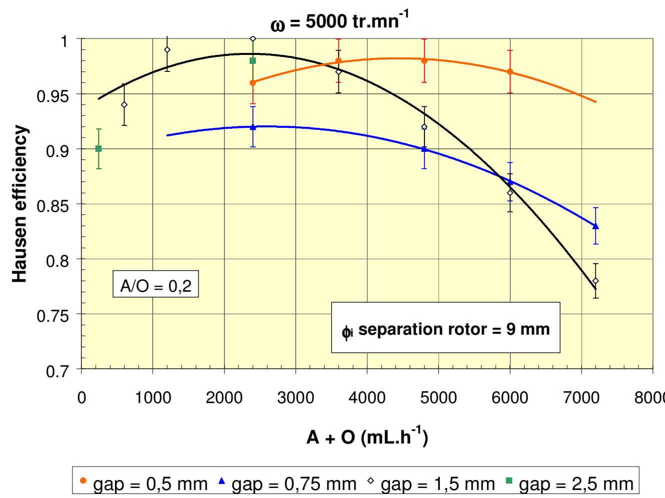

Fig. 3. Mass transfer performances with HNO_3/TBP : effect of total throughput on efficiency

With a gap lower than 1.5 mm, the decrease with total throughput was less when the gap decreased; for example, the extraction efficiency exceeded 95% with total throughputs up to 6 $L.h^{-1}$ if the gap was 0.5 mm.

The gaps up to 1.5 mm are interesting only with particular systems, for example, shear sensitive systems.

SHEAR SENSITIVE SYSTEMS

In process operations where shear sensitive phases are employed, excess mixing in the annular zone causes stable emulsions which can not be pumped and separated in conventional annular extractor, as shown in Fig. 4. text [1].

In the ECRAN, a low mixing can be used with a thin mixing rotor as the high centrifugal separation remains in the standard separation rotor.

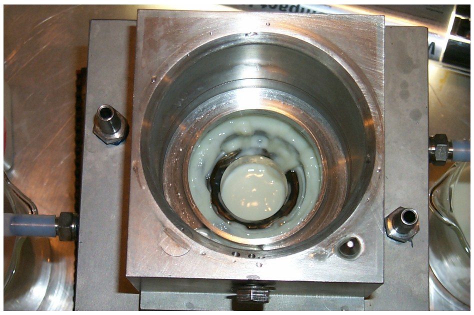

Fig. 4. Diamex spent solvent clean up : stable emulsion in all the parts of conventional annular extractor

For DIAMEX spent solvent clean up with sodium hydroxide, high efficiency was performed at 8,000 rpm with a 3 mm diameter mixing rotor and a gap width of 4.5 mm

CONCLUSIONS

The first tests of the new annular centrifugal extractor designed by CEA have demonstrated that high extraction efficiencies were achieved with total throughputs up to 6 $L.h^{-1}$, higher than with the CEA conventional annular centrifugal extractor at equal mixing hold up.

Moreover, it can be used to mix and separate shear sensitive liquids, for example encountered in solvent washing applications.

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