Development of experimental design and image processing methods for drops diameter measurement in an annular centrifugal solvent extraction contactor (ECRAN)

P. Rivalier, J-L. Bourrier, F. Lamadie, M. de Lorenzi

Development of experimental design and image processing methods for drops diameter measurement in an annular centrifugal solvent extraction contactor (ECRAN)

P. Rivalier, J.L. Bourrier, F. Lamadie, M. De Lorenzi

ATALANTE 2016 nuclear chemistry for sustainable fuel cycles

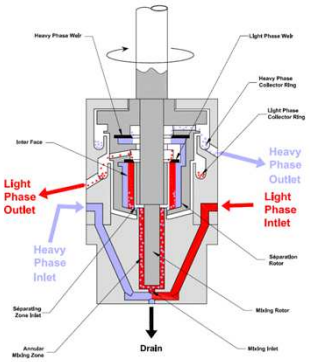
Abstract

Liquid-liquid extraction is a well-known method used to separate compounds based on their relative solubilities in two different immiscible liquids. Among the different types of liquid-liquid extraction apparatus, centrifugal extractors have advantages of low liquid hold-up and high mass transfer efficiency. As a consequence, many types of industrial monostage or multistage centrifugal extractors are currently used in nuclear but also pharmaceutical, food, chemical or hydrometallurgy industries with capacities from a few liters per hour to 100 cubic meters per hour.
In order to determine these industrial applications, testing must generally be performed on small scale equipment to evaluate liquid-liquid extraction relevant parameters such as **drop size distribution** which is the objective of this investigation.
Among the miniature extraction contactors developed since 1970, CEA has conceived a new annular centrifugal contactor referred as **ECRAN** with two original properties :

- 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,
- 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 the entire mixing zone and its volume is constant at any experimental conditions.

The CEA chose the ECRAN to measure the drop size distribution of liquid/liquid systems with the shear rate which depends on the rotor speed and the gap in the annular mixing zone. Images have been processed using three different image analysis software : **ImageJ, Matlab and DaVis**.
In order to ensure best possible drop detection, every processing technique has involved several steps : crop, denoising, background removal, thresholding, contour detection, particle analysis.
Results are presented by histograms of the drop diameter and descriptive statistics (mean diameter,…). The number of detected drops by the three image processing methods is generally different ; despite these differences, the deviation of the mean diameters in a same test calculated by the three techniques is of around ±20%.

Principle design of ECRAN



Schematic of ECRAN

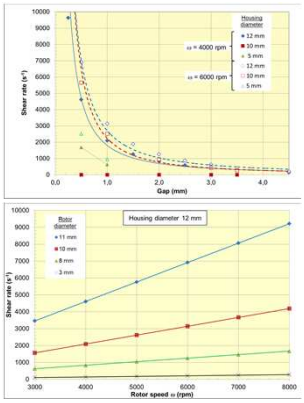
Aqueous and organic phases are fed at the bottom of the mixing zone by means of two channels. They are pumped by the mixing rotor and flow into the annular region between the mixing rotor and the stationary cylinder where they are mixed by shearing.

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 respectives 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.

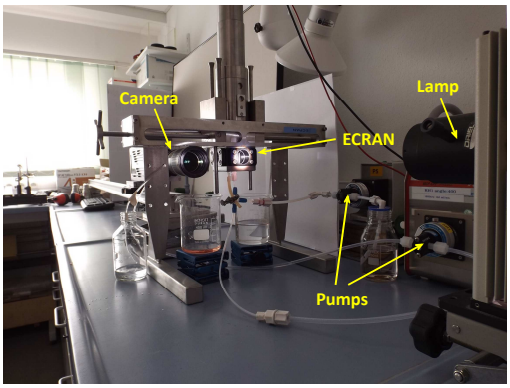
Actually, the maximum inner diameter of the housing is 12 mm the minimum diameter of the mixing rotor is 3 mm; so, the gaps are between 0.5 and 4.5 mm.

Under this wide range of rotor speeds, rotor diameters and annular gaps,With this variety of rotation speed and gaps, the shear rate values vary from 100 to 9000 s^{-1} ; this maximum value is greater than the shear rates in industrial centrifugal extractors.



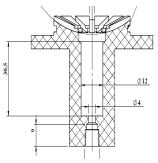
Shear rates as a function of rotor speed, rotor diameter and gap

Material and methods

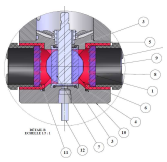


Experimental set up

Compared to a conventional ECRAN, the prototype of this investigation is composed of a stationary cylinder made of transparent materials (Plexiglas or borosilicate glass) and a mixing inox or Plexiglas rotor .

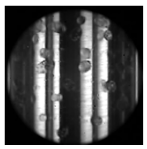


Plexiglas housing

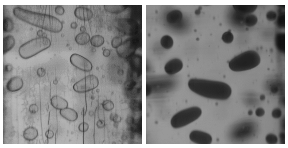


Glass housing

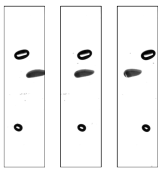
The experimental setup is completed by a high speed camera Photron modèle FastCam SA3 and quartz tungsten halogen lamps ORIEL model 60000 coupled with a power supply of 50-200 w to ensure clear sharp images of projected area of droplets inside the annular mixing zone.



Plexiglas housing Inox rotor



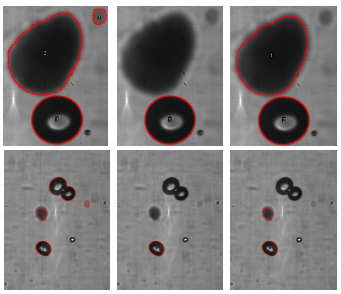
Plexiglas housing Plexiglas rotor



Glass housing Plexiglas rotor

Image processing and analysis

Images have been processed using three different image analysis software : ImageJ, MATLAB and DaVis.



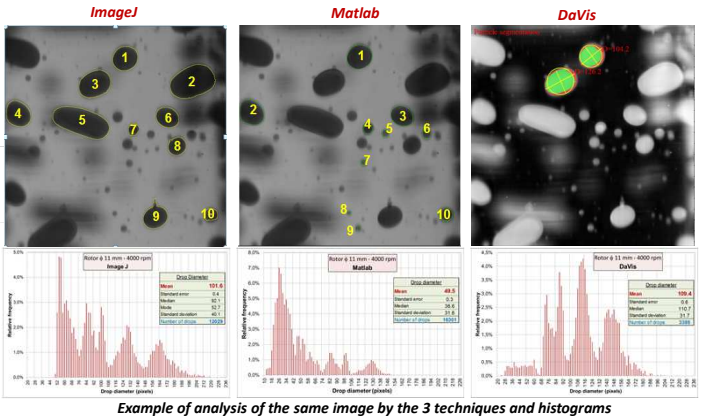
Circularity 0,45 Autothreshold | *Circularity 0,75* | *Circularity 0,75 Open*

Example of analysis of 2 images by ImageJ with different parameters

In order to ensure best possible drop detection, every processing technique has involved several steps: crop, denoising, background removal, thresholding, contour detection, particle analysis.
The shape, the size and the number of detected drops can be different :

- with a same image analysis software using different functions ; for example, whether or not the drops on the borders of the image or drops overlapping are taken into account, the size or circularity ranges are too restrictive.
- with the three techniques, even if a parameter "optimization" has been defined for each other.

Results are presented by histograms of the drop diameter over and descriptive statistics (mean diameter,…).



Example of analysis of the same image by the 3 techniques and histograms

Conclusion

Despite these differences between image analysis protocols, a careful examination of the images and a solid understanding of the parameters of the software minimize the disleading information and generally the deviation of the mean diameters in a same test calculated by the three techniques is of around ±20%. So, this experimental design and these image processing methods are considered reliable and our next objective is to characterize emulsions for a wide range of liquid-liquid processes according to the shear rate.

Commissariat à l'énergie atomique et aux énergies alternatives
Centre de Marcoule | BP17171 | 30207 Bagnols-sur-Cèze Cedex

DIRECTION DE L'ÉNERGIE NUCLÉAIRE
Département de Technologie du Cycle du Combustible