A new conical pump technology reduces solvent entrainment in solvent extraction plants

Guillaume FENOL (1), François DAROUX (2)
1. Process and Technology department, Technip, France
2. Mechanical Engineering department, Technip, France

For the past three years, Technip has invested for improving its proprietary Technip-Krebs Mixer-Settler equipment, operated worldwide in solvent extraction plants (U, Ni, Co, Cu, rare earths etc.). Krebs Mixer-Settlers are pioneering modules that use conical pumps and pre-settling launders, allowing a settling surface reduction by two to three, compared to conventional equipment.

The main objective in this development program is to reduce solvent entrainment in the aqueous phase leaving the settler, responsible for important OPEX loss for our clients. Computational fluids dynamics showed that novel mixing impeller and conical pump design could generate less shear and less solvent entrainment. This was proven with the construction of a new fifteen square meter equivalent settling surface pilot plant, operating with high performance Total Fluids’ Elixore® diluent.

Pilot plant trials compare different configurations of mixing impeller: standard four straight blades, disk and turbine technologies. New in-house impeller sizing criteria have been defined and allow, with a better control of the mixing power, a reduction by twenty percent of the entrainment at the settler outlet.

Significant progress was also made on the conical pump technology itself, above and beyond impeller improvements. Initially, Krebs technology was made of straight blades rotating in a fixed cone. Now, the bottom of the blades have been curved and trials have shown that, combined with a new impeller configuration, entrainments are reduced by nearly forty percent in the pilot plant compared to Krebs standard. Very good results have been achieved in a revamped SX plant in 2015. In the future, we will focus our attention on a new third generation patented concept: a rotating cone located at the bottom of the fixed cone, reducing by fifty percent entrainment at settler outlet compared to Krebs standard.

These encouraging results allow Technip to either remove after-settler tanks for future designs, or to further reduce the size of its Krebs Mixer-Settlers.
INTRODUCTION

Five hundred Technip-Krebs Mixer-Settler are operating around the world. These equipment are different from industry standard «conventional» Mixer-Settlers as they are equipped with dedicated conical pumps - located in the mixer tank - and pre-settling launders. These special features allow for a settler size reduction of between two to three (less footprint, compact design) and allow maximum production with low operational costs.

Solvent extraction plants are recognized as being simple, reliable and easy to operate. However, most of the SX plant users are facing solvent entrainment problems, generating non-negligible OPEX loss and disturbance of downstream processing units. This is particularly true when operating with cruds and where the use of after-settlers is not sufficient to recover all of the organic phase.

“It is generally accepted that entrainment is primarily generated as a result of the formation of fine droplets in the mixer (Eckert, 1984). Key issues are to provide adequate pumping head and mixing power, while minimizing the formation of fine droplets, which can lead to entrainment losses (Taylor and Jansen). Low-shear mixer design and optimized settler design have enabled plants operating with design flows and clean pregnant leach solution to operate with entrainment losses of less than 10 ppm and with very low interfacial crud generation (Tinkler, 2007).” Literatures agrees on the fact that the shear phenomenon is responsible for droplet size in the mixer, and the droplet size—especially fine droplets—are the main contributor to the organic losses observed at settler outlet. Technip ambitions to tackle the problem at the source, which means reducing the shear created by the impeller and the conical pump, and take control of the droplet size.

The purpose of this study is to improve impeller and conical pump design in order to reduce shear, take control on droplet size, and then reduce organic losses. Direct measurement of droplet size into the mixer tank is a difficult task, which requires costly and very specific equipment. Instead, Technip approach was to measure the consequences: organic loss. A fifteen square meter settling surface pilot plant was built and makes it possible to measure organic losses. Different configurations of conical pumps and impeller were tested until the best configurations were finally identified.

METHODOLOGY

The Technip-Krebs Mixer-Settler technology is unique by the use of a specific conical pump technology installed in the mixer tank. Mixing impeller and conical pump blades are installed on the same rotating shaft, forming the “rotor”. Conical pump blades are rotating into a fixed cone, forming the “stator”. The conical pump has two functions:

- Ensure a proper mixing of both aqueous and organic phases, with the impeller located on shaft bottom (in green color, Figure 1).
- Set the mixture in rotation using the rotor blades (in red color, Figure 1), centrifuge it in the cone (in grey color, Figure 1), and finally pump it up to the pre-settling launder. Conical pump technology ensures gravity flow from one Mixer-Settler to the next.
A new conical pump technology reduces solvent entrainment in solvent extraction plants

Computational Fluid Dynamics (CFD)

Shear phenomenon, responsible for organic losses, is generated at each place of the conical pump where the liquid is subjected to a speed gradient. Firstly, the use of CFD for the Standard Conical Pump (figure 1) enables to improve our knowledge of where the shear precisely takes place, and identify alternative low-shear configurations.

- CFD show the following shear distribution: 50% of the total shear is generated by the impeller, 30% is generated in the spacing between rotor and stator, and finally 20% is generated when the liquid slows down in the pre-settling launder.
- Interesting alternatives to reduce shear were quickly identified: They consist in optimizing impeller technology and changing the shape of the rotor blades. Ultimately, the solution is to operate with the bottom part of the cone in rotation.

Figure 1 Krebs Standard Conical pump – Generation 1

Figure 2 Visualization of shear in mixer tank and shear intensity vs. configuration
Having taken note of these observations, Technip has taken the decision to build an industrial size fifteen square meter settling surface pilot plant. This pilot plant will serve as a basis to confirm CFD results and to develop low shear conical pump generations, based on organic entrainment measurements at the outlet of the pilot plant settler.

**Mixer-Settler Pilot plant**

The Technip-Krebs Mixer-Settler pilot plant is designed to process 41 m³/h of liquid. It contains 4 m³ of Total Fluids’ Elixore® diluent (density=820 kg/m³, viscosity= 3cP @ 20°C), assimilated as the organic phase, and 6 m³ of water assimilated as the aqueous phase.

Its industrial dimensions are giving us a great confidence level for transposition of results for larger sized equipment. Pilot plant is operating in full recycle: Aqueous and organic phases going out from the settler are directly re-introduced into the mixer tank.

The pilot plant is equipped with sight glasses on the mixer and settler tanks to observe the fluid behavior. Settler covers are removable. A conductivity meter is installed on the mixer tanks as well as level gauges. The conical pump is removable and is equipped with a variable speed drive system. Impellers are removable. Those special features ensure a high flexibility for pilot plant operation. The pilot plant is operated in aqueous continuous phase to artificially increase organic entrainments in aqueous at settler outlet, and increase our level of confidence when comparing tests results.

![Figure 3 Technip-Krebs Mixer-Settler Pilot Plant](image)

Based on CFD results, an exhaustive trials plan was developed to design and improve:

- The impeller: Based on fluid behavior through the mixer sight glass, diameter and mixer type is modified to reduce shear, but still ensuring thorough phase contact. A new method for impeller sizing was developed.

- Conical pump blades shape: Initial straight blades are curved, to take benefit of a “spoon effect” and reduce conical pump rotation speed. Different bending angles are tested.

- A brand new concept: The bottom part of the cone, instead of being fixed, is now in rotation. It is equipped with internal baffles whose angles and shapes are to be optimized in order to reduce shear.
Organic losses are measured in the settler’s aqueous outlet piping, after two hours of pilot plant operation at 41 m³/h flow. Samples are taken in two liters separating funnels and rest for decantation over one night.

Each configuration is compared to the others based on one single sample (screening process). The objective is to find out the best assembly Impeller–Conical pump. For statistical validation, the final best configuration test is repeated four times (two hours tests for each) with two samples each time.

STUDIES AND RESULTS

Study of the agitation

Agitation represents 50% of the shear phenomenon. A good understanding of impeller sizing is fundamental to avoid the generation of un-settleable fine droplets.

Technip worked in parallel on two problematics: What is the best impeller technology for the mixing of two inhomogeneous phases? How to challenge the historical impeller sizing criteria?

Improvement of impeller technology

Technip’s partner Mixel is a specialist in agitation issues. They helped us identify two variants for the replacement of the historical Krebs “4 straight blades” impeller.

- a low-shearing 3 blades propeller technology,
- a rotating disk technology.

The following diagrams show the organic losses in aqueous phase at settler outlet, measured with the different types of impellers. Only impeller type is changing (same flowrates, O/A ratios in mixer, phase continuity, and same conical pump). Diameters are optimized in order to get the same impeller pumping flow for all impellers.

Organic losses in Aqueous at 41 m³/h (ppm)

![Diagram](image)

**Figure 4** Effect of impeller technology

The new “3 blades” impeller reduced organic losses by 25% compared to the standard 4 blades straight. Disk technology is abandoned: Shear is very important and fine droplets do not settle.
A new conical pump technology reduces solvent entrainment in solvent extraction plants

**Improvement of impeller sizing method**

Impeller sizing methods are largely developed in scientific reviews. However, our application is specific. Mixers can be circular or square based, with baffles. Their objective is not only to homogenise both phases, but also to ensure optimal droplet size distribution.

Bibliography is presenting two criteria commonly used for liquid/liquid mixing: Peripheral velocity of the impeller and recirculation volume (Reference [3]). This second criteria expresses how many times the mixer volume is going through the impeller during one hour.

Based on our observations at the mixer sight glass, both criteria can be evaluated for different diameters of the “3 blades curved” impeller.

![Figure 5 Effect of the “3 blades curved” impeller diameter](image)

For the pilot plant mixer, Figure 5 shows that the recirculation volume criteria (at 300 mixer/h) is a better sizing criteria than peripheral speed: It remains valid for both impeller diameters.

Based on these results, impeller diameter is now optimized to meet with two new sizing criteria:

- Recirculation volume: targeted value takes into account the mixer characteristics
- Average droplet size is calculated based on Calder-brank equation (1) (Reference [2])

\[
    d_{32} = 2.24 \left( \frac{\sigma^3}{\rho_c} \right)^{0.2} \left( \frac{P}{V} \right)^{-0.4} \sqrt{\beta \left( \eta_d/\eta_c \right)^{0.25}}
\]  

(1)

To ensure thorough phases contact, this droplet size is compared to experimental measures performed on an industrial plant (Reference [1]).

**Development of “Generation 2” conical pump**

CFD showed 30% of the shear is generated in the spacing between rotor and stator. The objective of this paragraph is to present the new technological developments brought to the conical pump itself, especially to the rotor blades.

Shear phenomenon is generated by speed gradients in the mixer. One simple action to reduce speed gradients is to reduce the conical pump rotor speed, without changing the original pumping efficiency. Technip investigated the “spoon effect” (See figure 6) where the fluid collection is improved thanks to curved blades, and shear reduced at the bottom end of the blades.
A new conical pump technology reduces solvent entrainment in solvent extraction plants

Bend radius of the blades has been optimized, with several tests, in order to reduce shear. Following curves are comparing organic losses between the standard Krebs “Generation 1” conical pump, and the best configuration of the newly developed “Generation 2” conical pump, with optimized curved blades and impeller.

Figure 6 Generation 2 and focus on the curved blades

Figure 7 Benefits of Generation 2 compared to Generation 1
A new conical pump technology reduces solvent entrainment in solvent extraction plants

These results obtained on the pilot plant are extremely encouraging. Organic entrainments are reduced by forty to fifty percent when using a Generation 2 conical pump instead of a Generation 1. This result is the combination of two improvements:

- Improvement of the mixing technology and impeller sizing method
- Improvement of the blade shape that allows less shear and conical pump speed reduction.

For clients using Generation 1 conical pumps, it becomes possible to evaluate OPEX gains for one year of operation, depending of their actual aqueous advance flowrate and measured organic losses. Payback time for an upgrade of existing Generation 1 pumps to Generation 2 can be easily calculated. Here, the example (Figure 7) is shown for the Uranium industry. The results are valid for Mixer-Settler units operating in the same conditions as the pilot plant: No cruds and no after-settler.

Pursuing our proactive effort, commercialisation and fabrication of Generation 2 conical pumps are becoming the rule since 2013.

Organic entrainments have been measured in real industrial conditions in 2015. The newly installed Generation 2 conical pumps are able to process 400 m3/h of mixed phase, and their performances have been compared with the old Generation 1 previously installed. Mixer-settlers operate with a high cruds content, and are normally followed by an after-settler to recover as much organic phase as possible.

The benefits obtained on this solvent extraction plant are spectacular. Organic entrainments are divided by a factor of two to four. Let’s precise that the launder end was also modified, contributing to better liquid distribution and better settling. In all cases, this confirms what was shown on Technip Mixer-Settler pilot plant. Generation 2 conical pumps are a real technological progress.

Development of “Generation 3” conical pump

This new development is considered as a real technological breakthrough. As shear is generated in the fine gap between rotor and stator (Generation 1 & 2), a brand new concept has been developed. It is inspired from the “rotating cup” that centrifuges the liquid, and technical feasibility was confirmed by CFD before proceeding to pilot plant tests.

Figure 8 Generation 3 - From the initial concept to the conical pump
A new conical pump technology reduces solvent entrainment in solvent extraction plants

On figure 8, rotating part with baffles is shown in blue color. It is linked to the rotating brown shaft. The main challenge was to find out the best rotating configuration that actually reduces shear: How high and how many baffles for the rotating part? With what angles and what inclination? Is there any impact on mixer sizing? All these questions have been studied carefully in order to optimize the sizing of the Generation 3 conical pump and obtain its best possible configuration.

This technological breakthrough was the subject of a patent registration in 2015, and will be published in 2016 under the reference FR 15 55085. Performance have been compared to Generation 1 and Generation 2 conical pumps:

- Organic losses are reduced by ten to fifteen percent compared to Generation 2
- Organic losses are reduced by fifty to sixty percent compared to original Generation 1
- Smoother operation, surge reduction, easy to install and adjust
- Possibility to get higher pumped flowrates

This technology is still in development, especially to make the equipment more reliable from a mechanical point of view. A process scale up study is still to be completed. In the short term, Technip will be able to size Generation 3 conical pumps for a high range of flowrates and lift heights.

For the moment, this Generation 3 conical pump technology will be designed for clean processes, without cruds, but its application will be probably expanded based on the first industrial performance tests, expected in 2017.

CONCLUSION

This development program aims to reduce organic losses by influencing the source of the issue: shear. Positive results of reduction of the shear effect induced by the impeller and the conical pump were obtained on the pilot plant and confirmed by industrial operations. The Generation 2 conical pumps are able to reduce by nearly two the organic entrainments and offer interesting alternatives to our clients:

- Replace older Generation 1 by Generation 2 conical pumps, with a short pay-back time thanks to organic loss reduction.
- Consider the possibility to remove after-settlers when operating with Generation 2, or alternatively reduce once again the dimensions of the Technip-Krebs Mixer-Settler.
- Guarantee lower organic entrainments out of the settler.

From a process, mechanical and performances point of view, Generation 2 conical pumps are perfectly mastered. Generation 3 conical pumps are still under development and should be proposed gradually in 2017. They offer interesting alternatives either for aqueous and organic loss reduction, but also for maximizing pumped flowrates.
A new conical pump technology reduces solvent entrainment in solvent extraction plants

ACKNOWLEDGMENTS

Technip would like to thank its support and co-operation partners of this R&D program:

- Total Fluids who have provided the Elixore® diluent used in the pilot plant.
- Mixel, our partner on mixing.
- Proycon, who manufactured the pilot plant and allowed us to operate in their premises.

LITERATURE CITED

[4] TAYLOR (A.) and JANSEN (ML.) - Solvent extraction Mixer-Settler Design, p.2-3 (No date).