# **Splitting of Emulsions with Compressed Propane**

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#### **INTRODUCTION**

Beside the recycling of pure lubricants the waste treatment of mixtures like oils and additives or emulsions like cutting fluids are important. In Germany a million tons of oil/water emulsions are disposed each year. Additionally 1.2 million tons of oil sludge, containing water and solid residues, have to be treated. The costs for the disposal of cutting fluids add to up to 10 % of the manufacturing costs. [1,2] The deposition of these products as hazardous waste becomes more and more expensive.

Therefore different classical techniques to split emulsions into fractions of oil and water are in use. It is possible to concentrate the emulsions by ultra-filtration, to separate both components by distillation or to add chemicals like acids to split the emulsions.

Alternatively a separation technique using compressed propane was investigated for the splitting of oil/water emulsions. In the frame of this research, more than 20 industrially used emulsions could be separated successfully into water and oil fractions by using propane. The new technique is characterized by very fast splitting kinetics, compared to the classical processes.

To investigate the mechanism of splitting and the influence of the main process parameters, model emulsions of mineral oil, water and emulsifiers were used. The main focus of the work lies on the behaviour of the surfactants at the boundary of the water and oil phase. The results show, that it is possible to classify the emulsifiers with regard to their stabilizing or destabilizing behaviour of the emulsion, depending on the process temperature. A destabilizing is necessary for the splitting by the use of compressed propane. In this contribution splitting experiments and the phase behaviour of oil/water/emulsifier at different temperatures are explained. In addition measurements of surface tension under high pressure are presented.

## **MATERIALS AND METHODS**

Real cutting fluids and cooling lubricants that were supplied by Minitec Engineering as well as a model emulsion prepared from the commercially available mineral oil BP 400 ® (supplied by German BP) and different surfactants were investigated. Ionic and non-ionic surfactants were used for the experiments. All surfactants were supplied by Cognis Care Chemicals, Germany. The fatty alcohol ethoxylates Dehydol 04 ®, Synative AC 3370 V ®

and Synative AC 3370 V  $\circledast$  as well as Plantacare 1200 UP  $\circledast$  an alkyl polyglucoside are examples of non-ionic emulsifiers. Texapon N 70  $\circledast$  a sodium lauryl ether sulfate belongs to the group of ionic emuslifiers. Propane with a purity of 95 % from Linde was used for all experiments.

Solubility measurements in presence of propane were performed according to a static analytical method in a high pressure view cell developed by NWA, Germany. For the determination of the surface tension the software Drop Shape Analysis developed by Kruess, Germany, was used.

## RESULTS

The aim of research was to investigate if the extraction of mineral oils originating from cooling lubricant with propane as solvent can be applied successfully. For this task more than 20 industrially used emulsions could be separated in a water phase and oil fraction. At the beginning of the project used oil was recycled. Later the applicability on the splitting of emulsions was investigated. First experiments on the thermal stability of cooling lubricants were preformed. Then the influence of propane on the separation kinetics is investigated. The splitting of the emulsion occurs instantly for most investigated emulsions. The splitting process is depending on the process temperature and pressure. The process could even be successfully applied on natural emulsions like butter and cream.



 $T = 65 \ ^{\circ}C, p = 1 bar$ 



$$T = 86 \,^{\circ}C, p = 40 \text{ bar}$$

Figure 1: Instant splitting of a model emulsion prepared from mineral oil, water and Deyhdol 04 (49/49/2 wt. %)

To understand the splitting kinetics a model emulsion of a mineral oil (BP 400 B) and deionised water was prepared. The influence of the chemical nature of the applied surfactant was investigated. For this purpose model emulsions with four non-ionic emulsifiers and Texapon N 70 B as example of an ionic surfactant were prepared. The emulsions prepared with fatty alcohol ethoxylates showed an instant splitting (figure 1). While in the case of the alkyl polyglucoside and Texapon N 70 B the phases could not be separated.

For explanation the phase behaviour of the system mineral oil, water and emulsifier was investigated. The system with Dehydol 04 ® is presented.

Based on these investigations a pilot plant for the treatment of industrial cooling emulsions was designed and built. The plant was built in cooperation with Minitec Engineering, Germany. Figure 2 shows the pilot plant.



Figure 2: Pilot plant 40 kg/h (pictures by courtesy of Minitec Engineering)

The process design is given in Figure 3.



**Figure 3**: Pilot plant designed for a capacity of 1000 kg/h cooling emulsion (pictures by courtesy of Minitec Engineering)

## CONCLUSIONS

The treatment of emulsions with propane was applied successfully. 18 different emulsions have been split. The phased separation is accelerated in the presence of propane. For this process a minimum temperature and pressure has to be applied. The hydrophilic behaviour of surfactants and properties of the oil are influencing the process parameters. Only emulsions containing an emulsifier with temperature dependent solubility in water and oil can be split. The solubility of the emulsifier in propane is important for the success of the process.

The technique is meanwhile used in pilot plant scale with a capacity of 40 kg/h. The engineering of a 5000 t/year plant is finished.

## REFERENCES

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