Recycling Emulsified Oils Using Percentrifugation Technology

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Percentrifugation is the combination of centrifugation with solvent injection and flocculation agents. This technique was used to separate metallic sludge, oil and grease, cutting oil and water contained in the used emulsified cutting oil. The centrifugal force plays two important roles: The first is to favor precipitation of metallic sludge, and the second is to force the oil to reach the surface. After centrifugation, the sample shows three phases: Metallic sludge can be seen at the bottom of the tube; oils are observed at the surface; and water is in the intermediate phase. That result is obtained after the addition of a flocculating agent to favor the separation, to decrease the amount of solvent required and to minimize the need for fast rotation speed. The solvent used is either a cycloalkane or a heavy alcohol. The additive is a polyaluminosilicate.

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Introduction

Cutting oils are used on a regular basis in many different industries. Their function is either to lubricate and/or to cool machining tools. Their composition is variable but they are all more or less stable emulsified oils in water. They contain bactericides and fragrances to minimize degradation and odour. Once used, cutting fluids usually look like a liquid sludge because they contain small metal particles and *dirt* coming from the process. Many technologies are available to treat those fluids using simple physical separation, chemical separation or a combination of both. A universally applicable technology was proven efficient to separate stable emulsions, the most difficult case.

Many different approaches are available for the treatment of used cutting fluids. But they work only if all their specific requirements are encountered. Table I shows those separation techniques with their advantages and disadvantages.

Table I
Classification of available oil/water separation methods.

Separation Technique	Domain of application	Advantages	Disadvantages
Skimming	The oil layer should float.	Simple and low cost.	Almost never applicable.
Thin film oil contaminant	The oil layer should float.	Simple and more efficient than skimming.	As limited as previous technique.
Decantation	Oil/water separation can be achieved only if there is no stable emulsion.	Easy to start up and low cost.	Not efficient if there is an emulsion.
Salt assisted decantation	Separation can be achieved when cutting oils are ionic type solutions.	Easy to start up and low cost.	Generates salt containing residues. Requires the purchase of salts.
Simple filtration	Limited to solutions charged with slag and metallic particles.	Easy to start up, well-known technology.	Filtration media gets clogged, heavy maintenance.
Coalescence	Can be used only if oil agglomerates in big size droplets in short enough time (80 minutes).	Low cost and requires less space than decantation. Electro-coalescing process is more efficient than the basic process.	Limited efficiency for emulsions. Requires additives.
Centrifugation	Oils and water must not form a stable emulsion.	Relatively low cost. Relatively efficient. No need for prefiltration.	Limited efficiency for emulsions. Requires additives.
Evaporation/distillation	No limitation.	Perfect separation.	High energy costs especially for large volumes.
Membrane filtration	Can be applied to any cutting fluid.	Concentrates effluents.	Requires a pre-treatment step to remove the sludge. Membranes are fragile and detergents have a tendency to clog up the membranes.
Ozone/peroxide treatment	Can be used for solutions with medium organic contaminant level.	Mineralizes the residues.	Requires a pre-treatment unit to remove the sludge.
Percentrifugation (centrifugation+extraction)	Can be applied to stable emulsions.	Combination of simple physical and chemical processes.	Requires additives.

Results and discussion

The composition of the cutting fluid selected to use in this project is as follows: an emulsifier (30-40%), a corrosion inhibitor and an anti-fungus agent (0.5-1%), a fragrance agent (0.1%), a chlorinated paraffin (5-15%) and a stabilizing agent (2%). Since the solution is a proprietary one, part of the composition is not mentioned on the technical data sheet or on the material safety data sheet (MSDS).

When that product is mixed with water in a ratio of 3-5% of fluid, a stable emulsion is formed with a pH around 8. To promote a phase separation water and/or solvent and/or additives should be added. Heating or cooling can sometimes facilitate that operation. Screening tests were made in order to choose the combination of technologies, to find out which conditions could help in promoting the phase separation and finally to determine the separation conditions. Table II describes some of the tests and gives a short explanation of the positive or negative results. A positive result indicates that a phase separation was observed. A negative result indicates that no separation was obtained.

Table II
Preliminary tests using cutting fluids in water

Tests	Result	Explanation
Centrifugation	Negative	Good mechanical resistance of the emulsion.
Ultrasounds	Negative	Good mechanical resistance of the emulsion.
Heating up to boiling point (212 ^{o}F)	Negative	The emulsion is thermally stable.
Cooling down slowly to 24.8 ^{o}F	Negative	Only part of the solution crystallized meaning that it is thermally stable.
Lowering pH from 7 to 2	Negative	Good chemical stability showing low content of ionisable functions.
Increasing pH from 7 to 10	Negative	Good chemical stability showing low content of ionisable functions.
Addition of oxidizing agents up to 50%	Negative	Slight discoloration of the solution. Good chemical stability of the emulsion.
Addition of sodium chloride	Positive	Phase separation. Medium influenced by a change in ionic strength.
Addition of calcium and iron chloride	Negative/Positive	Phase separation after two days. Medium slightly influenced by a change in charge densities.
Addition of bentonite (colloidal clay)	Negative/Positive	Formation of a dispersed bottom phase separation that cannot be used. Oils and surfactants are only slightly absorbed.

These tests show that the emulsion is chemically, physically and thermally stable. It means that all existing separation technologies are inappropriate except distillation and membrane filtration but they are quite expensive. That leaves room for new technologies like percentrifugation, a technology combining centrifugation and extraction. Used cutting oils are mixed with a solvent to break the emulsion and force the phase separation. The choice of the appropriate solvent is a critical step.

Six solvents were tried because they show an interesting extracting power combined with properties like miscibility or not in water, linear or ramified hydrocarbons, relatively low cost, solvating power, affinity for oils, etc. These solvents are heptane, isooctane, xylene, diesel, octanol and cycloalkane. Samples were centrifuged at 2500 rpm for 30 minutes. Three different concentrations of solvent were added to the used oil samples (10, 20 and 30% v/v). The best separated samples showed three phases for all concentrations. Unfortunately the phases were not well defined. Figure 1 shows a typical sample after the percentrifugation using cyclohexane. The three phases were as follows: metallic particles at the bottom followed by a water phase (milky phase) and finally an oily brown phase at the top. The best solvents were in order of increasing efficiency: heptane, octanol and cyclohexane.

Fig.1. Three phases sample after percentrifugation using cyclohexane as the extracting solvent.

Fig. 2. Example of a well-separated sample showing four distinct phases.

Different flocculating agents were added in order to improve the phase separation. The one selected is an aluminosilicate that combines classical coagulant properties to the coprecipitation action of silica or magnesium oxides. That aluminosilicate is not very expensive and acceptable on an environmental point of view. Its presence decreases the amount of solvent required to separate oils and surfactants from water. In ideal conditions of percentrifugation, 85-87% of the initial amount of water is removed in one single step.

The optimal operating conditions determined after the set of tests are as follows: mix the solvent with the used cutting oils in a ratio of 5% v/v; add 0,5% v/v of aluminosilicate and finally centrifuge the resulting solution.

The result is a well-separated sample showing 4 phases: an oily phase at the surface, a foam-like phase at the interface between oil and water, a clear water phase and finally the metal particles at the bottom of the tube. Best results were observed using cyclohexane, octanol and heptane.

Conclusions

For cutting fluids containing or not oils and greases, an addition of 5% of an organic solvent like octanol, heptane or cycloalkane combined with a polyaluminosilicate will induce a phase separation. By skimming, the organic contaminants are removed. The solvent can be reused few times before it reaches saturation. The remaining water can be reused to dilute new cutting oil. The centrifugation machine is actually the most expensive part of that technology that is expected to be competitive on the market of cutting fluids recycling.

References

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