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Membrane Filtration Studies of Inversely Soluble Model Metalworking Fluids

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ABSTRACT

Model metalworking fluids, characterized by phase separation of functional components at elevated temperatures, were studied. Results of membrane filtration experiments with three oils of differing chemical nature confirm the feasibility of specific removal of finely dispersed contaminant oils without the simultaneous loss of active components from adequately formulated fluids. Prerequisites are that membrane materials and pore sizes be suitably chosen and that operating temperatures be kept below the cloud point of the least soluble component. The most suitable filter in the present study was a hydrophilic regenerated cellulose membrane with a NMWL of 100,000 dalton. Complete oil removal was attained if membrane pore sizes did not exceed 0.1 μm. It was also found that intrinsically water-soluble boundary lubricants of the polyglycol ether type are retained by membranes in the presence of PPG-1800, which serves as a precipitation promoter above the cloud point. This is of considerable practical importance since it offers the possibility of designing water-soluble boundary lubrication and extreme pressure additives which are activated by the presence of a inversely soluble component above its cloud point. The latter compound also acts as the principal hydrodynamic lubricant of the present model formulations. The usefulness of a new analytical tool for the rapid acquisition and imaging of data pertinent to changes in molecular aggregation and phase conditions was demonstrated.

INTRODUCTION

The manufacturing of industrial goods often requires the input of some type of metalworking operation, either to remove some material or to reshape

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the material. In most of these operations, metalworking fluids (mwf's) are used in order to reduce friction and tool wear at the same time as heat is extracted and chips are removed from the cutting zone. The application of mwf's thus serves to save energy through friction reduction while simultaneously the work precision and surface quality of the finished products will be improved; in addition, the tool lives are extended (1–3).

Mwf's can be based on straight oils or may be water-based. The former type is used in operations where lubrication is the key issue or when water could cause damage to the worked material, while the latter type is used when cooling is essential for a good result. An aqueous mwf is usually a mixture of lubricating and corrosion-inhibiting components designed for specific or general applications. These basic components are supplemented with surfactants in the form of wetting agents and emulsifiers for insoluble lubricity agents as well as pH buffers, defoaming agents, biocides, and coupling agents. The most common lubricants used in emulsion-type mwf's are mineral oils with oil-soluble additives which act to reduce friction and wear by forming adsorbed layers or reaction compounds on the surfaces of work materials and tools (4–6). The emulsion form as such promotes oil deposition on tools and work pieces, a mechanism crucial for the lubricity of rather dilute media (7).

Synthetic solutions contain water-soluble components only. A key drawback of normal solution-type coolants is that their lubricating performance is limited compared to emulsion-type fluids. On the other hand, they are much easier to keep clean, e.g., by filtering. As a result, microbial infections are easier to control. An infection in a single-phase synthetic solution does not have the same adverse effects on the physical stability as in an emulsion-type fluid (8–10). Microbial infections are routinely combated by the use of biocides and high pH values. These measures are not desirable from an operator's point of view (11–14).

In the processes of metalworking, lubricating oils and greases from machines and work material will contaminate metalworking fluids. Contaminants may be free floating, in which case they may be skimmed off, or they may be emulsified with the surface-active components of the metalworking fluid. Since emulsion-type mwf's do contain oil and emulsifiers for oils, and since constituent emulsified oils appear in the form of droplets, it is not possible to selectively remove tramp oils or fine particulate contaminants without a simultaneous loss of active matter. With properly designed synthetic solutions, on the other hand, tramp oils, microorganisms, and abiotic contaminant particles, e.g., metal oxides and wear debris, can easily be removed without an appreciable loss of functional components. This has important implications on operator's health and safety conditions as well as on environmental aspects on the use of the fluid. Thus, a coolant which can be effectively purified potentially does not need the same input of biocides, corrosion inhibitor excess, or alkalinity reserves since the probability for microbial infections with a concomitant pH drop, bad odors, and iron corrosion problems decreases. At the same time, an extended coolant life will reduce the environmental impact and save money through reduced disposal costs. A small selection of the numerous papers found in the literature on the topic of health and safety as well as coolant housekeeping bears witness that these are considered to be important issues in the metalworking industry (15–23).

A possible way to suit both the need for good lubrication and the demand for increased coolant recycling is to design inversely soluble mwf's, i.e., fluids which behave like solutions in the tank and recirculation systems but which are converted into a synthetic emulsion under the influence of the high temperatures generated in the work zone. Thus a lubricant-depositing mechanism can be created specifically at the spot where it is needed.

Inversely soluble components are found among organic compounds containing a considerable number of ether linkages, i.e., polyoxyalkylene derivatives (24, 25). Polyoxyalkylene fragments, most commonly from the reaction of ethylene oxide or propylene oxide with a nucleophilic reagent, are common building blocks of many nonionic surfactants and polyglycol ethers. These have found their most widespread use as emulsifying, dispersing, and wetting surfactants in detergents. Polypropylene glycols and derivatives thereof are often used as low foaming wetting agents in machine dishwashing products and are not uncommon as lubricants in aqueous mwf's. It is believed, however, that the use of inversely soluble components in metalworking coolants can be extended considerably and that their performance can be substantially improved by added knowledge from detailed studies of the inverse solubility phenomenon under mwf conditions (26).

There are several methods available for the separation of leak oils and particulate contaminants. Membrane filtration appears to be a viable option (27), especially in combination with other techniques for pretreatment. In this paper an evaluation is also made of which restrictions on separation possibilities may be imposed by the chemical and mechanical nature of some membrane materials. Hence, the experiments were conducted to look specifically into the effects of membrane materials and pore sizes as well as operating conditions, such as temperature and pressure, on the use of ultra- and microfiltration for the selective separation of contaminant oils from model metalworking fluids containing inversely soluble components.

EXPERIMENTAL

1. Chemistry

All reagents were used as received. 2,2,5-Trimethyl hexanoic acid (isononanoic acid, INA) as well as neodecanoic acid (NDA) were provided by Exxon Chemicals as 97.5 and 98.5% pure, respectively. The triethanol amine

(TEA) used was 98% certified reagent grade (Fisher Scientific Co.), while the alkanolamine derivative (TED) was a polypropylene glycol ether derivative of TEA. The other anionic surface-active compound (H5P) was a technical grade phosphate ester based on the reaction of polyphosphoric acid with a pentaethylene glycol ether derivative of hexanol.

As a model for an inversely soluble lubricating component, technical grade polypropylene glycol of molecular weight 1800 (PPG-1800) was used. This compound, the phosphate ester (H5P), and the alkanol amine derivative (TED) were supplied by Akzo Nobel Surface Chemistry AB.

Due to their manufacturing process TED, H5P, and PPG-1800 are all of a polydisperse nature. This implies that each of the products consists of homologues which differ in the number of ethylene or propylene oxide monomer units per molecule (24). A practical effect of this is that they contain homologues with a spectrum of water solubilities. Ethoxylation will increase water solubility while propoxylation will have the opposite effect.

Soybean oil (Sigma Chemical Co.), dodecane (J. T. Baker), and a commercial diesel motor oil (Statoil A/S) were used as model contaminant oils. A red, oil-soluble dye (Sudan Red B, Aldrich) was used as an indicator for the quantification of residual oil content in membrane-filtered fluids.

2. Model Metalworking Fluids

Three model metalworking fluids were formulated. First, base solutions without polypropylene glycol were made according to the following recipes:

Base 1	Base 2	Base 3
11.7% H5P 11.7% INA 23.3% TED	11.7% H5P 11.7% NDA 23.3% TED	11.7% H5P 11.7% INA 7.0% TEA

The balance of materials in the above recipes were distilled water and KOH to make pH 9.0.

Surface-active compounds and cosolvents also exert a considerable influence on important parameters for the application of membrane separation as a purification technology. Thus, in addition to their intended effects, these additives may also affect surfactant aggregation and leak oil emulsification besides possible direct interaction with membrane materials. In the present work these potential side effects were minimized by a careful choice of surface-active components and a minimum use of cosolvents.

Model concentrate formulations with PPG-1800 were prepared from the base solutions. To obtain clear concentrate solutions at ambient temperature as needed, diethylene glycol monobutyl ether (butyl diglycol, BDG) was used as a cosolvent. In Formulation 2 below, no BDG was needed with a maximum of 3.8% of PPG-1800. The cosolvent-free composition was preferred to the more analogous composition achieved by the addition of BDG. The formulations were made according to:

Formulation 1: 85.0% Base 1 + 5.0% PPG-1800 + 10% BDG
Formulation 2: 96.2% Base 2 + 3.8% PPG-1800
Formulation 3: 85.0% Base 3 + 5.0% PPG-1800 + 10% BDG

All formulations had a cloud point of about 23°C at 5% in distilled water. Micellar aggregation was characterized by surface tension, conductivity, and pH measurements as a function of concentration at 22°C (28–33). The complex nature of the present formulations, with several polydisperse components, makes the determination of critical micelle concentration (CMC) values difficult. However, based on an evaluation of the data shown in Table 1 from the three independent methods, the CMC values were estimated to be 5.5, 4.0, and 4.0% for Formulation 1, 2, and 3, respectively. An uncertainty of ±1.0% is assigned to these values. The most important implication from these data is that only a small fraction of the active material exists in micellar form in the present experiments.

3. Model Contaminant Oils

Dodecane and soybean oil were used as models for a nonpolar- and a polar-based oil, respectively. The commercial motor oil is an example of a

TABLE 1
CMC Values for Formulations 1–3 at 22°C.
Uncertainties Are Estimated to ±1 Concentration Unit

CMC method		CMC (% w/w)
Formulation 1:	Surface tension	6.5
	Specific conductivity	4.5
	pH	4.5
Formulation 2:	Surface tension	3.5
	Specific conductivity	4
	pH	3.5
Formulation 3:	Surface tension	—
	Specific conductivity	4.5
	pH	3

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formulated product containing detergents, which are potential emulsifiers for leak oils in metalworking fluids. The presence of surface-active components in contaminants may pose a serious difficulty for the successful removal of leak oils. They may promote the solubilization of active components of the metalworking fluid into the removed leak oil or work to disperse adventitious oil in the metalworking fluid.

The concentration of oil in permeates from membrane filtration of model metalworking fluids with model oils was estimated by the use of an oil-soluble dye. Thus the color intensities of permeate solutions were compared with standards of known oil content. These standards were prepared with the same dyed oil. In the absence of oils, the present model metalworking fluids were shown not to dissolve any visible amount of the dye.

Even if particulate matter constitutes an important group of contaminants in mwf's in industry, the possibility of removing leak oils was considered crucial for the successful performance of recyclable products. This feature was therefore given first priority.

4. Equipment and Membrane Filtration Procedure

All the experiments were carried out using a stirred cell supplied by Millipore Corporation. Flat circular membranes with a diameter of 47 mm were used, and the cell volume was 75 mL. The pressure inside the cell was maintained using nitrogen gas from a cylinder equipped with a standard N_2 pressure regulator (AGA Gas AB). It has been demonstrated that a stirred cell can be used as a simple-to-operate model for crossflow filtration (34). Since the input of stirring energy results in rather high shear forces along the surface, the flux values obtained should only be taken as relative. Rejection properties are, however, believed to reflect real crossflow conditions. The same stirring rate was used throughout this work.

The membranes used in this study were also supplied by Millipore Corporation. A description of the membranes is given in Table 2 together with the assigned acronyms.

Solutions were kept at desired temperatures by submerging the stirred membrane filtration cell into a thermostated water bath. Deionized water was used to measure the pure water flux rate for each membrane. Fifty milliliters of each base solution or formulation at 5% dilution was used in each experiment. Runs were terminated when 40 mL of permeated solution had been collected. Flux values were determined by timing and weighing samples of the permeate. Changes in the refractive index as well as the change in the cloud point of solutions were used to monitor the loss of active matter.

Inversely soluble solutions were characterized by the use of a new analytical technique (35) which permits rapid scanning of turbidity, specific conduc-

TABLE 2
Description of Membranes Used

Membrane designation	Pore size/ NMWL	Membrane material	Chemical nature
PLGC	10,000[a]	Regenerated cellulose	Hydrophilic
PLHK	100,000[a]	Regenerated cellulose	Hydrophilic
PLMK	300,000[a]	Regenerated cellulose	Hydrophilic
VVLP	0.10[b]	Modified polyvinylidine fluoride	Hydrophilic
VVHP	0.10[b]	Polyvinylidine fluoride	Hydrophobic
GSWP	0.22[b]	Mixed cellulose esters (nitrate and acetate)	Hydrophilic

[a] Nominal molecular weight limits (NMWL; dalton).
[b] Pore size in micrometers (μm).

tivity, pH, or other readily measurable physical parameter values over a wide range of temperatures and compositions of liquid mixtures. The results are usually presented in the form of separate diagrams for each measured parameter value. The measured data are thus displayed by projection of a 2-dimensional contour map or a 3-dimensional wire-frame surface, respectively, onto an orthogonal temperature vs concentration base surface.

RESULTS AND DISCUSSION

1. Effects of the Chemical Nature of Membranes and Pore Sizes on Separation Efficiency and Flux

Six types of membrane materials, differing also in their nominal molecular weight limit (NMWL) values and pore sizes, were used to separate contaminant oils from Formulation 1. Initial experiments were done to assess possible retention of active matter with Formulation 1 at 18°C, i.e., below the cloud point of the solution. As may be seen from the results given in Table 3, the concentration of the permeate from the filtration of a 5% solution of Formulation 1 is affected by only one of the membranes as indicated by the lowered refractive index and the increased cloud point. The exception is the PLGC material, a hydrophilic regenerated cellulose with an NMWL of 10,000 dalton. The lowering of the refractive index, measured in Brix units (Bu; the unit is based on a scale of percent sucrose in water), shows that in absolute terms approximately 0.7 mass percent of the active components of the formulation ($5 \times 0.4/2.9$) are removed by the smallest pore size membrane tested, while the observed increase in the cloud point indicates a loss of PPG-1800.

Figure 1 shows PPG-1800 concentration and temperature-dependent turbidity data for 5% of Base 1. The data in Fig. 1 indicate that the cloud point

TABLE 3
Effect of Various Membrane Types on the Separation of Dodecane as a Contaminant from Formulation 1 at 18°C and at 1 bar Pressure[a]

Membrane designation	Refractive index[a] (Bu)	Cloud point[a] (°C)	Concentration of dodecane in permeate (ppm)	Flux ($dm^3/m^2/h$)
PLGC	2.5	29	NIL	13
PLHK	2.9	24	NIL	64
PLMK	2.9	24	NIL	40
VVLP	2.8	23	<50	30
VVHP	—[b]	—[b]	675	59
GSWP	—[b]	—[b]	>5000	296

[a] The table shows values for the permeate. The refractive index and cloud point for the feed formulation were 2.9 Bu and 23°C, respectively.

[b] Brix and cloud-point values are not reliable in the presence of significant amounts of oil.

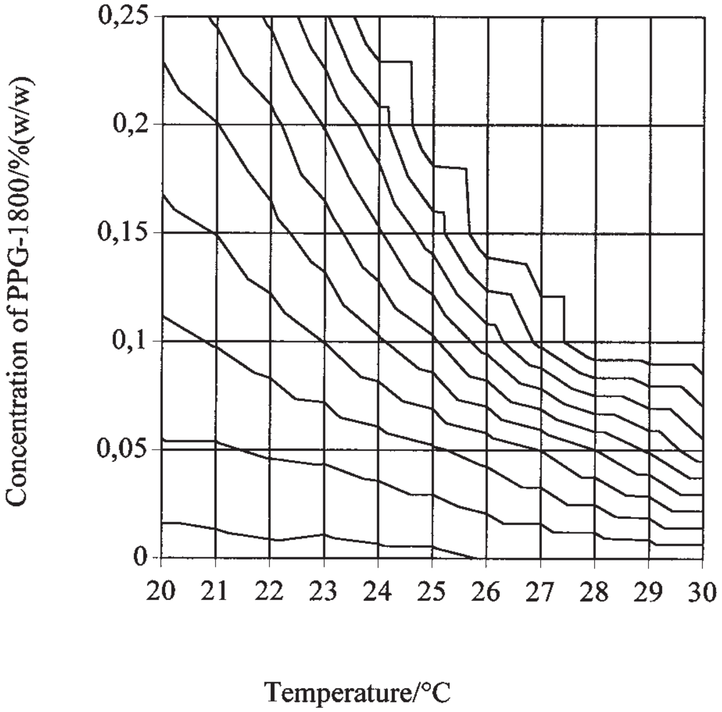

FIG. 1 Turbidity vs temperature and concentration of PPG-1800 in a 5% aqueous solution of Base 1. Shaded area marks turbidities over 90 NTU.

increase from 23 to 29°C after membrane separation of dodecane in Table 3 corresponds to a loss of about 0.2% of PPG-1800 (from 0.25 to 0.07%) from Formulation 1. Cloud points determined visually were found to correspond to a turbidity of around 90 NTU.

When the corresponding PPG-1800-free solution was subjected to the same treatment, the loss of active matter was found to be about 0.4%. This is somewhat unexpected since the critical micelle concentration (CMC) of Formulation 1 is 5.5%. A closer investigation showed that the additionally retained material mainly consisted of the TED compound. This may be explained by the fact that the propoxylated alkanol amine is highly branched and has a polydisperse molecular weight. Polydispersity leads to the existence of species of considerably higher molecular weight than the average (ca. 500 dalton). The higher molecular weight, as accomplished by propoxylation, also renders the molecules less water soluble. Cosolubilization of high molecular weight fractions of the TED in the retained PPG-1800 phase may explain the possible loss in addition to size exclusion in the membrane.

As far as separation of dodecane is concerned, membranes designated PLHK, PLMK, and VVLP seem to be useful options. PLHK stands out as a favored choice because it offers a relatively high flux combined with total rejection of dodecane, as shown in Table 3. The hydrophilic polyvinylidene membrane (VVLP) appears to be more effective than the hydrophobic type (VVHP) in retaining hydrophobic contaminants such as dodecane despite comparable pore sizes. This is probably an effect of the expected superior oil-repelling nature of the former membrane. Complete separation of dodecane was not attained if the pore diameter size exceeded 0.1 μm because this low viscosity compound is easily sheared into small droplets which may pass the membrane freely.

2. Effect of the Chemical Nature of Contaminant Oils on Membrane Separation Efficiency and Flux

In separate experiments the effect of the nature of contaminant oils on separation efficiency and rate of filtration at 1 bar and 18°C was evaluated using the regenerated cellulose membrane, PLHK. From an inspection of Table 4 it is clear that all three types of oils tested in this study can be effectively removed without appreciable loss of the active components of Formulation 1. This is indicated by the insignificant changes in the refractive index and in the cloud point of the 5% solution observed after filtration.

Effects on flux of the addition of the different oil types were more complex as may also be seen from data in Table 4. In fact, as compared to the blank solution without any contaminants, an increase in the measured flux was actually observed in the ultrafiltration of Formulation 1 in the presence of soy-

TABLE 4
Effect on the Separation of Various Oils as Contaminants from Formulation 1 Using PLHK Membranes at 18°C and at 1 bar Pressure

Type of oil	Refractive index[a] (Bu)	Cloud point[a] (°C)	Concentration of oil in permeate (ppm)	Flux ($dm^3/m^2/h$)
Distilled water	0	—	—	359
Blank[b]	2.9	23	—	46
Soybean	2.9	23	NIL	104
Dodecane	2.9	24	NIL	65
Motor oil	2.8	24	NIL	30

[a] The table shows values for the permeate. Refractive index and cloud point for the feed solution of Formulation 1 were 2.9 Bu and 23°C, respectively.

[b] Formulation 1 without any oil.

bean oil and dodecane as model oils. It is well known that surface-active compounds work as foulants by adsorption to the inner pore surfaces of membranes. This is a reasonable explanation for the rather low flux observed with the blank (oil-free) solution as compared with deionized water (359 $dm^3/m^2/h$). Further, it is not surprising that the detergents of the compounded motor oil are more potent foulants than the surface-active components of Formulation 1. At a first glance, the reason for the observed flux increase on adding the soybean oil and dodecane is less obvious. The negligible loss of active matter and the absence of oil in the permeate may not seem to support the removal of foulant surfactants by solubilization into the oil phase, since this would lead to the lowering of surfactant concentration in the solution. It should also be observed that the present experiments were conducted at a concentration below the CMC of Formulation 1. The most likely explanation resides in the fact that the polypropylene glycol derivatives, i.e., PPG-1800 and TED, are polydisperse. This means that there exists a small fraction of high molecular weight of these materials. The more hydrophobic nature of these fractions increases their affinity for the membranes. This phenomenon, in combination with their larger molecular size and the branched shape of TED, leads to an effective flux reduction. A removal of this small fraction by partitioning into an oil phase may thus give rise to a remarkable increase in the flux without a notable change in the total concentration.

3. Effect of Variations in Model Metalworking Fluid Composition on Membrane Separation Efficiency and Flux

Membrane filtration experiments were done with 1% dodecane at 18°C and a pressure of 1 bar to investigate the effect of small changes in metalwork-

TABLE 5

Results of Ultrafiltration Filtrate Using PLHK Membrane for the Separation of 1% Dodecane as Contaminant in Various Model Metalworking Formulations at 18°C and at 1 bar Pressure. Data Refer to Permeate Solutions

Type of formulation	Refractive index (Bu)	Cloud point (°C)	Concentration of dodecane (ppm)	Flux ($dm^3/m^2/h$)
Formulation 1[a]	2.9	24	NIL	64
Formulation 2[a]	2.9	24	NIL	77
Formulation 3[b]	2.3	24	NIL	46

[a] The refractive index and cloud points for these formulations before membrane filtration were 2.9 Bu and 23°C, respectively.

[b] The refractive index and cloud points for the formulation were 2.4 Bu and 23°C, respectively, before filtration.

ing fluid composition on flux and oil separation efficiency. Investigations were done with 5% solutions of Formulations 1–3.

Results in Table 5 demonstrate an excellent coincidence of data for Formulation 1 with the corresponding, independently obtained data for Formulation 1 in Table 4. With Formulation 2, a somewhat higher flux is observed, probably depending on the lower concentration of PPG-1800. The lower flux value found with Formulation 3 indicates a negative influence of TEA as compared with TED in Formulation 1. Differences are not very prominent in the present case and may well be explained by the presence of micelles in Formulation 3, since the CMC of Formulation 3 is only 4.0 ± 1.0%. Due to the polydispersity of the H5P compound, which will determine aggregate dimensions, micelles should have a diameter around at least 200 Å, which coincides fairly well with the present membrane pore size. The retention of a small fraction of these micelles would hardly be noticed as a change in the refractive index or in the cloud point, but may add to the concentration polarization layer (34) sufficiently to further slow the flux.

4. Effect of Temperature on Membrane Separation Efficiency, Flux, and the Loss of Active Matter

In Table 6 are shown the results of membrane filtration experiments with Formulation 1 at a pressure of 1 bar and with PLHK membranes in the presence of 1% dodecane in the well-stirred mixture. Five percent solutions were thermostatted to 8, 18, and 30°C while concentrations and cloud points as well as flux values of permeate solutions were measured.

As expected, solutions filtered below their cloud point of 23–24°C did not display any loss of active matter, whereas a considerable loss was obtained with the solution filtered at 30°C.

TABLE 6

Effect of Temperature on the Removal of Dodecane and the Effect on the Refractive Index and Cloud Point of Dodecane-Free Solutions of Formulation 1:[a] Membrane Type: PLHK; Pressure: 1 bar

Temperature (°C)	Refractive index (Bu)	Cloud point (°C)	Concentration of dodecane in permeate (ppm)	Flux ($dm^3/m^2/h$)
8	2.9	23	NIL	53
18	2.8	24	NIL	63
30	2.6	43	NIL	121

[a] The refractive index and cloud point for this formulation before membrane filtration were 2.9 Bu and 23°C, respectively.

Somewhat unexpected, however, was the excess increase of the cloud point in the permeate of the solution filtered at 30°C. The resulting cloud point, 43°C, was substantially higher than the filtration temperature, indicating that components and homologues of components, which are intrinsically water soluble, are also retained at this temperature as a result of partitioning into the removed dodecane and coacervate (precipitated polyglycol ether) phase. Since the PPG-1800 at the most can account for less than half of the 0.5% loss of matter, as deduced from the decrease in the refractive index in Table 6, other components must be subject to a comparative loss of active matter. Other available components with an inverse solubility constitution are the phosphate ester (H5P) surfactant and TED. In view of the project targets, this is an interesting observation since the phenomenon confirms a synergistic phase separation of potential boundary lubricating surface-active components together with the hydrodynamic lubricant PPG-1800.

As may also be seen in Table 6, an increase in temperature leads to an increase in the flux. It should be observed that this occurred even above the cloud point. As in the experimental results discussed in Section 2, a reasonable explanation is that the extraction mechanism occurs here to remove adsorbed high molecular weight material into the physically separated phases. In the present case the additional presence of a coacervate may further enhance the effect due to increased solubility of polyalkylene glycol derivatives therein. At least for the flux increase at temperatures below the cloud point, a viscosity decrease of the concentration polarization layer is a probable cause of the increased flux in going from 8 to 18°C.

5. Effect of Pressure on Flux

According to basic liquid flow models (36), flux is directly proportional to the differential pressure over a membrane. In real systems, however, this is only applicable when the pressure is very low or the crossflow velocity is very high. In the present case it was found that for the PLHK membranes in the filtration of a 5% solution of Formulation 1, flux was practically independent of pressure. In varying the pressure from 0.5 to 3 bar in steps of 0.5 bar, no significant change in flux was observed. This may be rationalized in terms of compensating mechanisms. Among possible counteracting forces, the most probable at work in the present case are the osmotic counterpressure, established by even a very slight increase in the extent of retention and adsorption of matter in the pore channels, and mechanical compression of membrane pores.

CONCLUSIONS

Experiments with an ultrafiltration membrane, of a pore size which permits the retention of a water-soluble polyoxypropylene polymer, reveal a considerable loss also of smaller molecular mass material. Since no micelles were present under the conditions of the experiment, the results indicated substantial retention of a highly branched alkanol amine derivative with this fine membrane. This was somewhat surprising since the specified NMWL (10,000 daltons) was substantially higher than the mean molecular weight (500 dalton) of the branched alkanol amine derivative. However, the discrepancy arises from the fact that membrane characteristics were established by the use of linear polymers.

In another case a considerable cosolubilization of intrinsically water-soluble substances was found in the retained polyoxypropylene fraction when a model mwf solution was subject to membrane filtration above its cloud point. Under the conditions of the experiment it was expected that the coacervate phase, initiated by the presence of the same polyglycol polymer, would be removed to some extent. It was observed, however, that the obtained increase in the cloud point by far exceeded the temperature of treatment and that the loss of material, as indicated by a decrease in refractive index, was larger than the mass of the total available PPG-1800. These findings support the working hypothesis of the present project, i.e., that suitably designed boundary lubricants, even under conditions where they are soluble by themselves, may coprecipitate with a inversely soluble component like PPG-1800. Thus a synergistic increase in the lubrication performance of the formulation is predicted. In repeat experiments, where the PPG-free solution was subject to the same treatment, no loss of material was observed under the same conditions. Results of initial friction and wear studies with the present formulations and base solutions also support this conclusion.

As a practically important overall conclusion, it was found that three different model oils could be successfully removed without a significant loss of active matter. This proves the assumed feasibility of formulating reverse soluble mwf's which are possible to purify even when they are contaminated with large amounts of leak oils.

Even if flux values varied substantially, with different formulations being filtered with the same membranes, it was found that the exchange of some of the components was not crucial to the possibility of removing tramp oils. It was also found that pressure is not a very important parameter for the control of flux.

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