

RECOVERY OF METAL WORKING FLUIDS USING CHELATION-ULTRAFILTRATION PROCESS

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ABSTRACT: Metal working (MW) fluids are used to control friction and temperature, improve workpiece surface quality, and reduce tool wear. The batch-life of the MW fluid investigated was controlled primarily by the concentration of metallic soaps (complexes between the oil emulsifier and Al/Mg. Ethylenediaminetetraacetic acid, a strong metal chelator, was added to the MW fluid to break the oil-Al/Mg complexes. The aqueous-phase ethylenediaminetetraacetic acid-Al/Mg complexes were then separated from the oil phase by ultrafiltration (UF). Al levels were reduced between 39 and 49%, and Mg levels were reduced between 67 and 77%. Al transfer from the oil phase to the aqueous phase was slow and increased during UF concentration. Rinsing the concentrated MW fluid with deionized water decreased the ash content but had a lesser impact on Al/Mg removal. Al mass balances produced errors of 2.8% for run 3 and 6.2% for run 4. The UF system (0.11- μm ceramic membrane) effectively separated the oil and aqueous phases. Permeate turbidity was generally <1 nephelometric turbidity unit and permeate flux ranged from 390 to 220 L/m²-h (230 to 130 gal./ft²-day) depending on the degree of concentration.

INTRODUCTION

Metal working (MW) fluids are used to control friction and temperature, improve workpiece surface quality, and reduce tool wear. For the MW fluid investigated in this study, the batch-life is controlled primarily by the concentration of complexes of anionic emulsifiers (e.g., oleic acid) and Al/Mg. These complexes are referred to as "metallic soaps." Al/Mg enters the MW fluid during normal milling operations through contact with the workpiece. Increasing the metallic soaps concentration increases the lubricating ability of the MW fluid. Initially, the increase in lubricating ability is desired but eventually the MW fluid becomes too "slippery," and the rolling process is adversely affected because of unwanted slippage between the workpiece and the rolling mill.

If the concentration of metallic soaps can be reduced in the MW fluid, reuse of MW fluid is possible. The addition of a strong metal chelator [e.g., ethylenediaminetetraacetic acid (EDTA)] to a MW fluid containing metallic soaps should transfer the Al/Mg from the oil phase to the aqueous phase. The aqueous-phase chelating agent-Al/Mg complexes and oil droplets can then be separated using a membrane filtration process. In Fig. 1, a schematic of the chelation-ultrafiltration (CUF) process is presented. A batch of spent MW fluid enters a well-mixed feed tank where an appropriate amount of chelating agent is added. When the aqueous phase Al and Mg concentrations approach a maximum, the emulsified oil droplets are separated using a UF membrane. The UF permeate would contain high concentrations of chelating agent-Al/Mg complexes and very low concentrations of oil and is considered a waste. The concentrate would contain almost all the original oil/additives, would have a lower metallic soap concentration, and could be reused. If necessary, the concentrate can be rinsed with deionized (DI) water to further remove

aqueous-phase chelating agent-Al/Mg complexes and to lower the conductivity. DI water would be added to the concentrated MW fluid after which the MW fluid is processed through the UF membrane. The rinsed/concentrated MW fluid can be brought back to its original oil content using DI water and then reused.

EDTA was chosen as the study chelating agent. Based on previous research (Reed et al. 1997; Viadero 1997; Viadero and Reed 1999), a high-shear rotary ultrafiltration (HSRUF) system, equipped with a 0.11- μm ceramic membrane, was used to separate emulsified oil from the aqueous phase. Goals of this study were to (1) determine the ability of EDTA to sequester the Al/Mg associated with emulsified oil droplets; and (2) ascertain the feasibility of using the HSRUF system to separate the aqueous-phase EDTA-Al/Mg complexes from the oil droplets. Waste MW fluids were acquired from an actual rolling operation, and several EDTA concentrations were used. Permeate Al and Mg concentrations, permeate flux, and quality (turbidity and oil/grease) were measured. The effect of concentrating and rinsing the treated MW fluid was investigated. A mass balance on Al was also performed.

BACKGROUND

Chemistry

For the MW fluid investigated in this study, the primary emulsifier was sodium oleate, an anionic emulsifier. In Fig. 2, schematics of an emulsified oil droplet, oleate molecule, and breaking of metallic soap complexes through EDTA addition are presented. The oleate carboxyl group is hydrophilic, and the remaining hydrocarbon chain is lipophilic. The hydrocarbon chain penetrates the oil droplet, and the negatively charged carboxyl group resides in the water phase. The negatively charged oil droplets repel each other, producing a kinetically stable fluid that resists the agglomeration of finely dispersed oil droplets into larger, less stable droplets. The average size of an oil droplet varies between 1 and 10 μm .

Oleic acid is an unsaturated fatty acid ($\text{pK}_a \approx 4.5$) that is sparingly soluble in water. Over the life of an MW fluid, Al and Mg will form complexes with the oleic acid that resides predominately in the oil phase. Consider the metallic soap formation reactions and the reactions between EDTA and aqueous-phase Al and Mg



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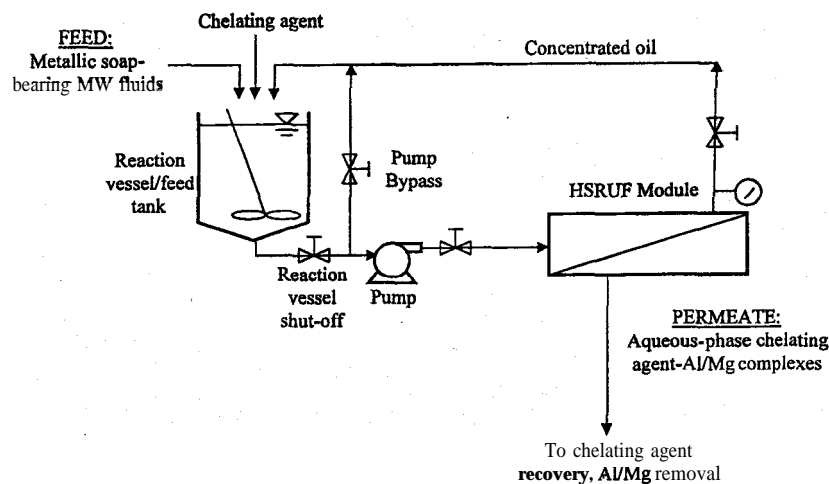


FIG. 1. Schematic of CUF Process

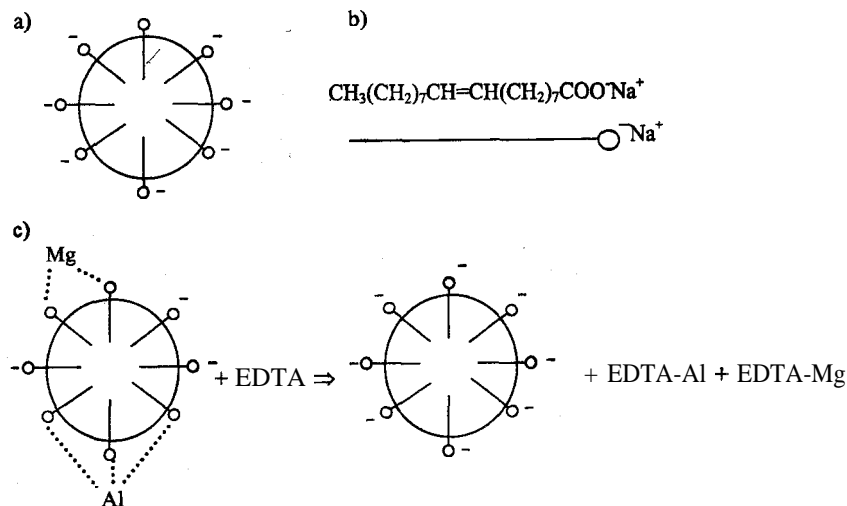


FIG. 2. Schematic of: (a) Emulsified Oil Droplet; (b) Oleate Molecule; and (c) Breaking of Metallic Soap Complexes by EDTA Addition



where $\equiv\text{OA}$ represents oil-bound oleic acid, and pK is the formation or stability constant (Dean 1979). The larger the formation constant, the more stable the complex. The formation constants for Al and Mg with oleic acid are not known. However, if $\text{pK}_{\text{EDTA-Al}}^{\text{F}}$ and $\text{pK}_{\text{EDTA-Mg}}^{\text{F}}$ are greater than $\text{pK}_{\equiv\text{OA-Al}}^{\text{F}}$ and $\text{pK}_{\equiv\text{OA-Mg}}^{\text{F}}$, then the metallic soap complex can be broken, and Al/Mg will reside in the aqueous phase as EDTA complexes. Because these reactions occur at the oil-water interface mass transfer phenomena will affect the overall process kinetics.

For a nonoil system, all aqueous-phase Al will be complexed by EDTA under the following conditions (Westall et al. 1976): (1) pH is greater than -2; (2) EDTA/Al molar ratio ≥ 1 ; and (3) only 1:1 EDTA-Al complexes are formed. For pHs less than -2, H^+ successfully competes with the Al for the EDTA. For Mg, the EDTA-Mg complex becomes significant at pH -4 and all aqueous Mg is associated with EDTA at pHs greater than -6. For both Al and Mg, the formation of hydroxide solids is prevented at EDTA/metal molar ratios ≥ 1 .

UF and CUF Process

UF is a pressure-driven membrane technique that uses porous membranes for the separation of material in the 1-nm to 10- μm size range or compounds with molecular weights in excess of 1,000 (Cheryan 1986). In all membrane processes,

a solute boundary layer will form at the membrane surface due to convective mass transport, which is one reason why the permeate flux for a waste is lower than the clean water flux. The buildup of solute near the membrane surface is reduced by diffusion processes, and the solute boundary-layer thickness is reduced through hydraulic turbulence. In conventional tubular UF systems, the thickness of the solute boundary layer is reduced by recycling a large portion (-98%) of the feed back to the membrane unit, producing large liquid velocities near the membrane surface. In high-shear UF systems, the energy required to produce the "cleaning action" is applied directly to the membrane surface by rotating or vibrating the membrane. In this study, a HSRUF system equipped with a ceramic membrane was used to separate oil droplets from the aqueous phase. The reader is referred to Reed et al. (1997) and Viadero and Reed (1999) for a detailed description of the high-shear system.

MATERIALS AND METHODS

Two batches of MW fluid were received directly from a rolling mill operation in 55-gal. drums. The first batch was used for runs 1 and 2, and the second batch was used for runs 3 and 4. The MW fluid contained soil ($\approx 5\%$ volume-to-volume ratio), oleic acid (an emulsifier), film strength additives, and antifoaming agents. Specific contents of the mixture cannot be disclosed for proprietary reasons. The first batch of MW fluid contained 212-mg/L Al (7.86×10^{-3} M) and 14.8-mg/L Mg (6.1×10^{-4} M). The second batch contained 230-mg/L

every 30 min for the first 5 h of a dosage interval and every hour thereafter and analyzed for Al within 2 h of sampling, and when the permeate Al concentration stabilized for a given EDTA concentration, the next EDTA dose was added; and (3) after final EDTA addition, the system was operated in batch-down mode until the CF reached 4X.

Runs 3 and 4

In runs 3 and 4, a single EDTA concentration (0.05 M) was used. Following EDTA addition, the system was operated in recycle mode with frequent permeate sampling. After the permeate Al concentration had reached a constant value, the system was operated in batch-down mode. The concentrated MW fluid was rinsed by bringing the contents of the feed tank back to the original volume using DI water, operating in recycle mode (for mixing purposes), and then operating in batch-down mode (to remove the rinse water as permeate). The rinsing

procedure was repeated twice. Sampling was conducted in a manner similar to runs 1 and 2 except for the following: (1) Mg was not measured in runs 1 and 2 because it was demonstrated that Mg contributed relatively little to the production of metallic soaps; (2) permeate and concentrate conductivity were monitored frequently; and (3) mass of Al in the permeate and in the final UF concentrate was determined so that an Al mass balance could be conducted.

RESULTS AND DISCUSSION

Run 1

In Fig. 3, permeate Al and Mg concentrations versus time are presented for run 1. Prior to the addition of EDTA, the permeate Mg and Al concentrations were 1.5 mg/L and <0.1 mg/L, respectively. At an EDTA concentration of 10^{-4} M, the permeate Mg concentration doubled, but there was little

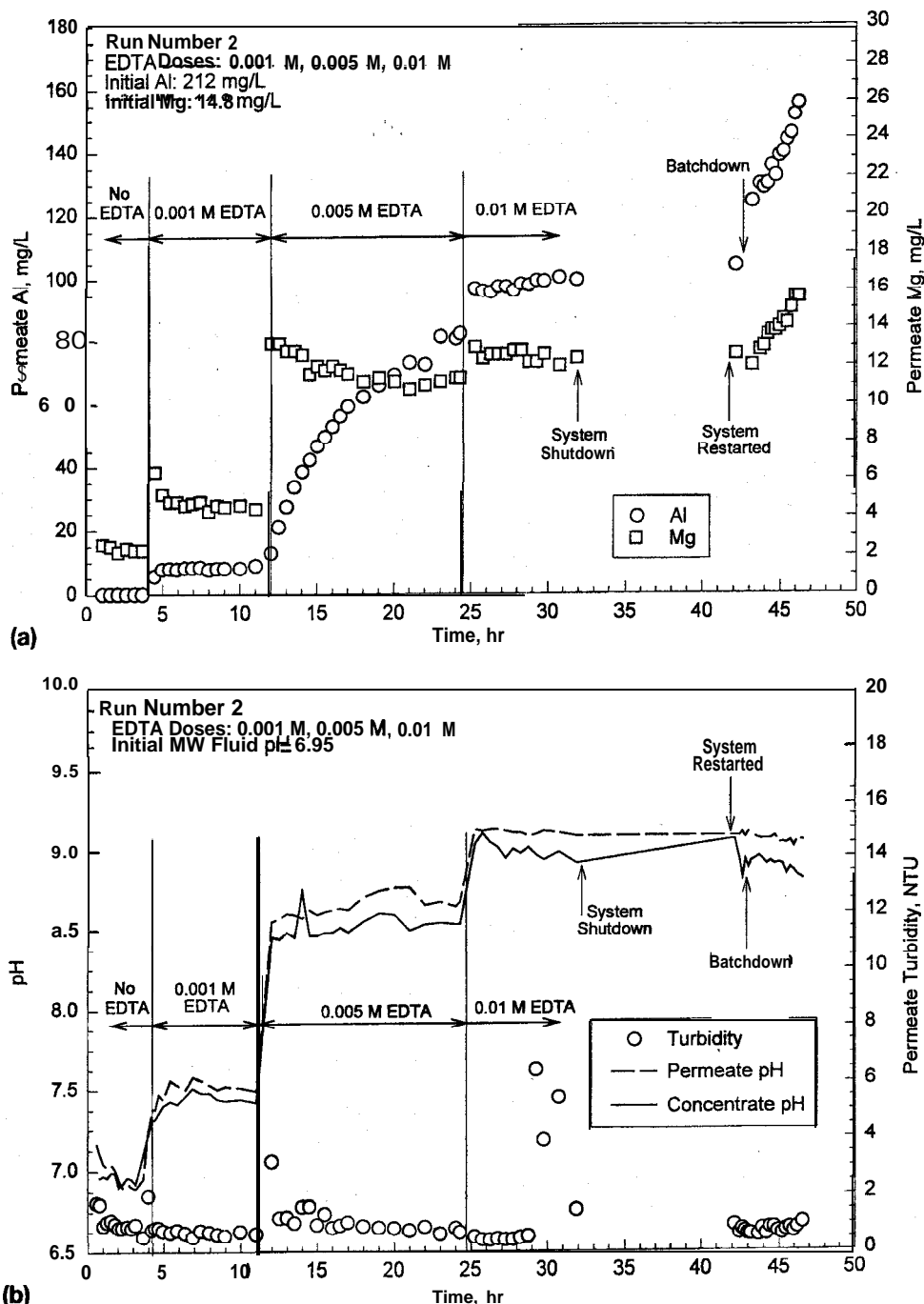


FIG. 4. Run 2: (a) Permeate Al/Mg concentrations; (b) Permeate and Concentrate pH and Turbidity; (c) Permeate Flux and CF

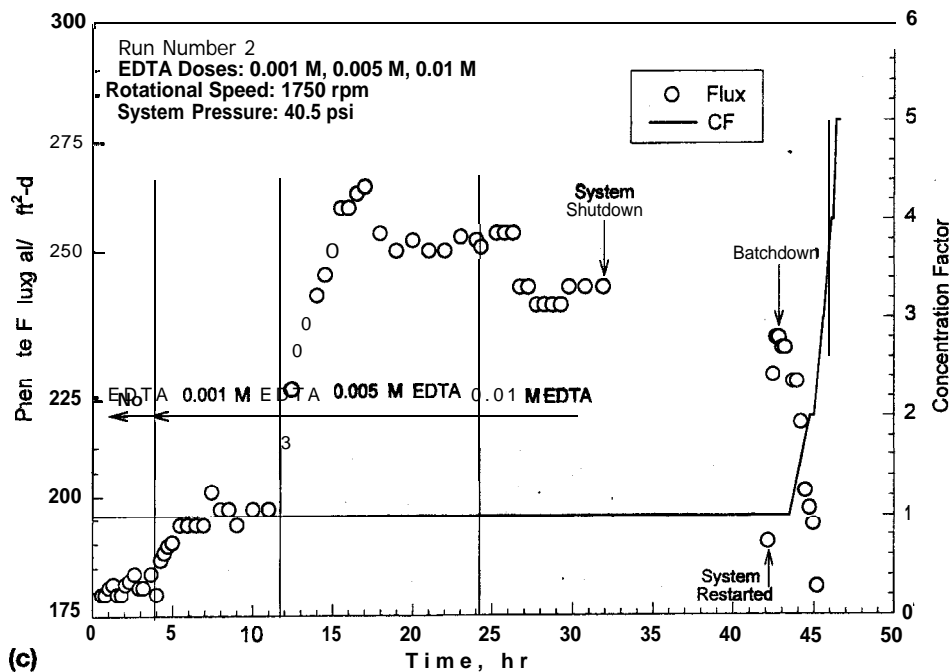


FIG. 4. (Continued)

change in the Al concentration. Similar behavior was observed for 5×10^{-4} M and 10^{-3} M EDTA concentrations. Not until the EDTA concentration was increased to 5×10^{-3} M was there a significant amount of Al released from the oil phase. The initial Al and Mg concentrations in the MW fluid were 212 mg/L (7.86×10^{-3} M) and 14.8 mg/L (6.1×10^{-4} M), respectively. The amount of Mg complexed at 5×10^{-3} M EDTA was approximately 10 mg/L (4.1×10^{-4} M) leaving 4.6×10^{-3} M of EDTA available for Al complexation. Assuming the EDTA-Al complex was 1:1 and the remaining EDTA is 100% efficient in sequestering Al, [Al], should equal 4.6×10^{-3} M [124 mg/L, see (4)]. The highest Al concentration reached at 5×10^{-3} M EDTA was 91 mg/L (3.4×10^{-3} M), which corresponded to a chelating efficiency of about 74%. The percentage of the total Al and Mg residing in the aqueous phase at 5×10^{-3} M EDTA were 43 and 67%, respectively.

At 10^{-2} M EDTA there was enough EDTA added to form 1:1 complexes with all Al and Mg present in the MW fluid. However, the permeate Al concentration increased by only 7 mg/L to 98 mg/L (3.6×10^{-3} M), and the Mg concentration decreased slightly. Under these conditions, the Al chelation efficiency decreased to about 46%. The percentage of the total Al and Mg residing in the aqueous phase at 10^{-2} M EDTA were 46 and 61%, respectively. The incomplete sequestration of Al and Mg when the EDTA/Al and EDTA/Mg ratios were >1 gives some insight into the strength of the metallic soap complexes (especially for Al) but also may indicate that a portion of the Al and Mg is inaccessible to the aqueous-phase EDTA. For example, oil may have coalesced around inert particles (e.g., dust and workpiece fragments) shielding the oil droplet from contact with the aqueous phase.

Run 2

In Fig. 4, permeate Al/Mg concentrations, permeate and concentrate pH, permeate turbidity, and flux and CF versus time are presented for run 2. As in run 1, prior to the addition of EDTA, the permeate Al concentration was negligible, and the Mg concentration was approximately 2.5 mg/L. Based on manufacturer's information, the concentration of oleic acid was always larger than the combined Al/Mg content (i.e., [oleic acid] $>$ [Al + Mg]). Comparing permeate Mg (2 mg/L)

and Al (<0.1 mg/L) concentrations prior to EDTA addition, the formation constant for Mg-oleic acid is significantly less than that for Al-oleic acid, and the Mg-oleic acid complex should be easier to break. Following the first EDTA addition (10^{-3} M), the aqueous-phase Mg increased immediately but then declined as the Al concentration increased from ≈ 0.1 mg/L to about 8 mg/L. This phenomenon was more apparent at 5×10^{-3} M EDTA. The Al-EDTA chelation efficiency for run 2 at CF = 1 X was similar to that in run 1. The percentages of the total Al (Mg) residing in the aqueous phase at 5×10^{-3} M and 10^{-2} M EDTA were 39% (77%) and 46% (81%), respectively. Given the small increases in aqueous-phase Al and Mg concentrations when the EDTA concentration was increased from 5×10^{-3} M to 10^{-2} M, an EDTA concentration of 5×10^{-3} M was chosen for use in runs 3 and 4.

Permeate Al and Mg concentrations increased with CF with the vast majority of total Al/Mg removal occurring as the CF was increased from 1 X to 3 X (Table 1). Actual Al and Mg mass removal efficiencies, based on the mass of Al and Mg in the permeate collected as the CF was increased from 1 X to 5X, were 49% for Al and 86% for Mg. These values were much higher than the removal efficiencies predicted using permeate Al and Mg concentrations observed during operation in recycle mode (37 and 64% for Al and Mg, respectively). Either additional Al/Mg was released as the oil content was increased or oil droplets containing Al/Mg passed through the membrane into the permeate. The latter explanation is unlikely given that the permeate turbidity and O/G did not increase substantially during batchdown. It is more likely that during batchdown the oil droplets coalesced into larger droplets that required less

TABLE 1. Percentages of Total Mass of Al and Mg Removed during Batchdown

| CF change (1) | Percent of Total Al or Mg Present | |
|---------------------|-----------------------------------|------------------|
| | Al (%) (2) | Mg (%) (3) |
| 1x \rightarrow 2x | 61 | 59 |
| 2x \rightarrow 3x | 23 | 23 |
| 3x \rightarrow 4x | 12 | 13 |
| 4x \rightarrow 5x | 4 | 5 |

emulsifier (oleic acid) to maintain emulsion stability. The formation of larger oil droplets caused the oil-bound Al and Mg to be released more easily.

Except for a few samples between 29 and 32 h, permeate turbidity was generally <1 (NTU) and $O/G < 200$ mg/L, demonstrating the effectiveness of the ceramic membrane in rejecting oil droplets. The pH of the permeate and concentrate increased because the EDTA stock solution pH (made from Na_4EDTA) was 11.9. Increasing the pH should not affect the chelation of aqueous-phase Al and Mg because Al and Mg are theoretically fully complexed with EDTA between pH = 7-9. However, pH could affect the relationship between oleic acid and Al/Mg as well as the chemistry of the oil droplet. The permeate flux increased from 195 to 250 gal./ft²-day when the EDTA concentration was increased from 10^{-3} M to 5×10^{-3} M. Additional increases in EDTA concentration had little effect

on the permeate flux. An increase in flux with conductivity (ionic strength) has been reported for the microfiltration of a domestic wastewater (Al-Malack and Anderson 1996) and for fuel and crude oil contaminated waters (Tansel et al. 1995). Al-Malack and Anderson (1996) reported an increase in flux with increasing salt concentration until an optimum dose was reached above which no improvement in flux was observed. During batchdown, permeate flux decreased with increasing CF (i.e., increasing concentrate oil content). These results were consistent with earlier work on the same MW fluid (Reed et al. 1997, 1998). As the oil concentration was increased, the solute boundary-layer thickness increased due to (1) more oil being transported to the membrane surface via convection; (2) decreased back-diffusion of oil from the membrane surface because of a smaller oil concentration gradient between the membrane surface and bulk solution; and (3) less turbulence

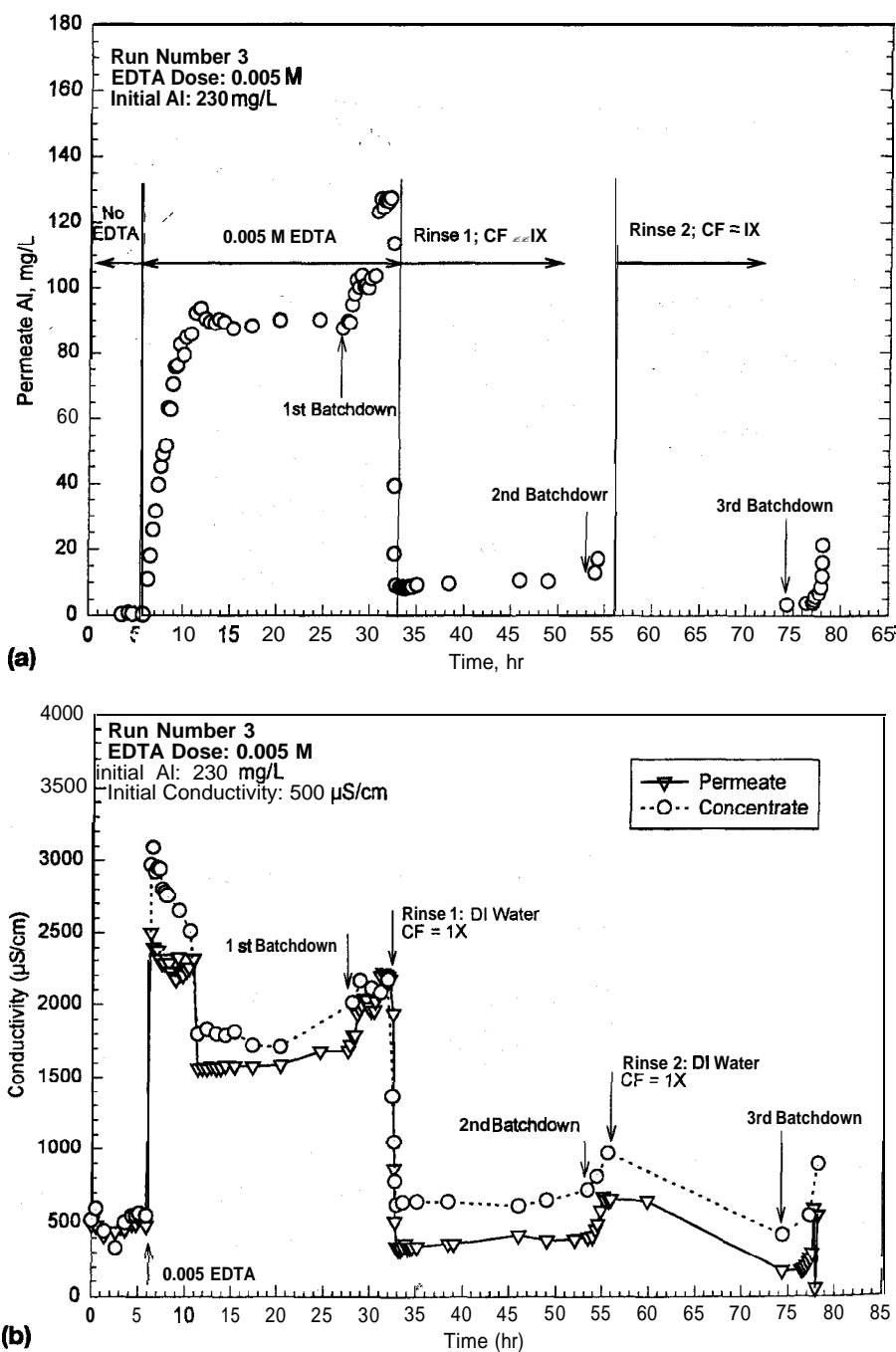


FIG. 5. Run 3: (a) Permeate Al Concentration; (b) Permeate and Concentrate Conductivity; (c) Permeate Flux and CF

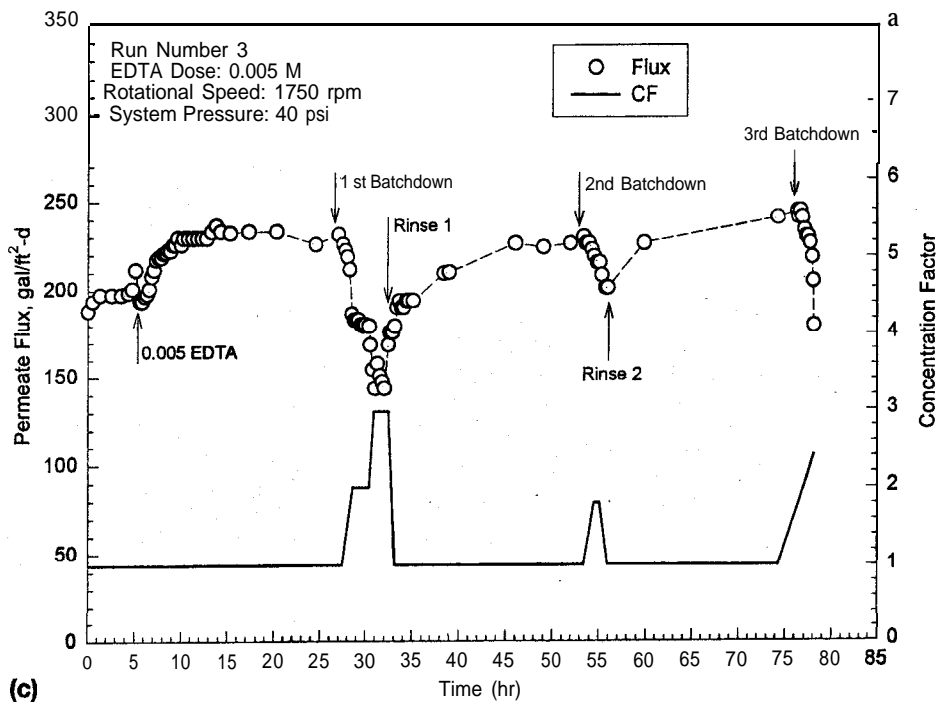


FIG. 5. (Continued)

at the membrane surface due to an increase in viscosity (Reynolds number $\sim 1/\mu$).

Runs 3 and 4

In Fig. 5, permeate Al concentration, permeate and concentrate conductivity, and flux and CF versus time are presented for run 3. Results for run 4 were similar and will not be presented. Following the 5×10^{-3} M EDTA addition, the permeate Al concentration increased over a 5-h period from near zero to 90 mg/L and then remained fairly steady for the remainder of the CF = 1 X portion of the experiment. First- and second-order kinetic models ($r = k[Al]$ and $r = k[Al]^2$) were applied to the data in this 5-period, but neither model adequately described the increase in Al with time. The following multistep process may be occurring:

- Step I-EDTA transport from the bulk aqueous solution to the stagnant liquid layer surrounding the oil droplet
- Step II-Diffusion of the EDTA through the stagnant liquid layer
- Step III-Reaction of the oleic acid-Al and oleic acid-Mg complexes with EDTA
- Step IV-Diffusion of EDTA-Al and EDTA-Mg complexes through the stagnant liquid layer
- Step V-Transport of EDTA-Al and EDTA-Mg complexes to the bulk solution

The bulk solution hydrodynamic conditions in the experimental system were turbulent as the MW fluid and EDTA were well mixed in a number of locations: (1) In the centrifugal pump that provided recirculation between the membrane chamber and feed tank; (2) within the high-shear membrane chamber; and (3) within the feed tank itself where a pneumatic mixer was located. The Reynolds number in the high-shear membrane chamber can be estimated by (Murkes and Carlsson 1988)

$$R = \omega r^2 / \mu \quad (7)$$

where ω = membrane rotational speed (rad/s); r = membrane disk radius (m); and μ = absolute viscosity (kg/m-s). At 1,750

(183 rad/s) and 5% oil, R was $> 1.8 \times 10^6$ (Viadero 1997), which is much $> 2.5 \times 10^5$, the cutoff between laminar and turbulent flow (Ketola and McGrew 1968; Murkes and Carlsson 1988). Thus, it is unlikely that Steps I and V were rate limiting. Typically, reaction kinetics are not considered to be rate-limiting. However, little is known about the reaction between the metallic soaps and EDTA. As a side experiment, virgin MW fluid was contacted with Al to determine if a synthetically contaminated MW fluid could be developed. After several weeks of mixing, there was little change in the aqueous-phase Al concentration. In the full-scale MW process, it takes between 6 and 8 weeks before there is a buildup of metallic soaps. Thus, Step III as well as Steps II or IV may be rate-limiting. Complicating the transport of EDTA to the oil droplet and the transport of EDTA-Al and EDTA-Mg complexes away from the oil droplet are the presence of negative charges on the oil droplet, EDTA (-4), EDTA-Al (-1), and EDTA-Mg (-2) complexes. The rapid increase in bulk aqueous-phase Mg concentration compared with Al could be due to Steps III and IV being faster for Mg-the more negative EDTA-Mg⁻² complex may move away from the negatively charged oil droplet faster than the EDTA-Al complex due to differences in electrostatic repulsive forces.

During the first batchdown (CF = 1X to 3X) the permeate Al increased from 90 to 127 mg/L and 30% of the Al was removed. Subsequent DI water rinses removed little additional Al (4.7% for rinse 1 and 4.3% for rinse 2). The total Al removal efficiency was 39% for run 3. The effectiveness of the rinse in removing ions from the aqueous phase is demonstrated by the decrease in permeate and concentrate conductivity. When the system was brought back to its original volume following the first batchdown, the concentrate conductivity was similar to the original value. This is important because the ash content of the MW fluid (defined by the MW industry as the residual remaining after heating at 770°C) would be unacceptably high, due to Na₄EDTA addition, unless the MW fluid was rinsed. During the first batchdown in run 4 (CF = 1X to 4.2X), 38% of the Al was removed from the system. In subsequent rinses, Al removal efficiencies for rinses 1 and 2 were 7.8 and 3.0%, respectively. The total Al removal efficiency for run 4 was 49% and was higher than that observed in run 3

because a higher CF was reached (4.2X compared with 3X in run 3). Al mass balances, conducted at the end of the runs, produced errors of 2.8% for run 3 and 6.2% for run 4.

After the system was brought back to its original volume (CF = 1 X), the flux took over 10 h to reach its prebatchdown level indicating that the solute boundary layer was somewhat stable. Similar behavior was observed during the second rinse. Stability of the solute boundary layer with changes in rotational speed were reported for an actual MW fluid (Reed et al 1997) and for a virgin MW fluid (Viadero 1997).

CONCLUSIONS

For the MW fluid investigated in this study, batch-life is controlled primarily by the concentration of complexes of anionic oil emulsifiers (e.g., oleic acid) and Al/Mg (known as metallic soaps). Metallic soaps increase the lubricating ability of the MW fluid, but eventually the MW fluid becomes too slippery, and the rolling process is adversely affected because of unwanted slippage between the workpiece and the rolling mill. EDTA, a strong metal chelator, was added to a MW fluid containing metallic soaps with the goal of transferring Al/Mg from the oil phase to the aqueous phase. The aqueous-phase EDTA-Al/Mg complexes and oil droplets were then separated using a UF membrane process. The following conclusions are drawn:

- The CUF process was effective in removing oil-bound Al and Mg (metallic soaps). Removal efficiencies for Al (39-49%) and Mg (67 and 86%) were highest at 5 X 10^{-3} M EDTA. Because metallic soaps, up to a certain concentration, impart valuable lubrication properties, higher Al/Mg removal efficiencies may not be required.
- Concentrating the MW fluid following EDTA addition increased Al and Mg removal efficiencies. The beneficial effect was minimal after a CF of 3X was reached, and the membrane separation portion of the CUF process can end at this level of concentration.
- Rinsing the concentrated MW fluid with DI water reduced the conductivity/ash content substantially but had a less significant impact on Al and Mg removal.
- Oil-bound Al release into the aqueous phase was slow.

First- and second-order aqueous-phase kinetics did not adequately describe the data; thus, a multistep, two-phase conceptual model was proposed.

- The UF system (0.11- μ m ceramic membrane) was effective in separating the oil droplets from the aqueous phase. Permeate turbidity was generally <1 NTU, and the O/G content was always <200 mg/L. The separation step does not appear to be the limiting factor in the effectiveness of the CUF process.

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