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Microemulsion Liquid Membranes. I. Application to Acetic Acid Removal from Water

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Abstract

A separation technique utilizing nonionic microemulsions as emulsion liquid membranes has been successfully applied to the removal of acetic acid from an aqueous feed phase. The surfactant systems were carefully characterized in order to assure that they were truly microemulsions. The effects of mixing intensity, feed concentration, treat ratio, and microemulsion viscosity on the separation kinetics were investigated. The microemulsions did not typically display leakage and had negligible swell over 5-minute duration. The reversible phase behavior of the microemulsion was utilized to demulsify the liquid membrane phase and recover the acetate ion via a temperature change of approximately 40°C. Material balances closed to within 10% and rates of separation were faster than the sampling rates when the microemulsion was fully dispersed in the aqueous feed phase.

INTRODUCTION

The emulsion liquid membrane technique, invented by Li (*1*), addresses some of the generic research areas which have been tagged as critical needs by a special committee of the National Research Council (*2*). The process has high selectivity, can easily concentrate solutes from parts per million to a few percent, has very fast rates of separation, and is energy efficient compared to classical thermal separations such as distillation. In an extension of Li's original work, microemulsions have been utilized instead of coarse emulsions as the liquid membrane emulsion. As discussed below, microemulsions exhibit several advantages over coarse emulsions when

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used as liquid membranes, including even faster rates of separation, low leakage, and ease of demulsification. To implement microemulsions as emulsion liquid membranes, a detailed understanding of the thermodynamic and kinetic behavior of microemulsions is needed.

BACKGROUND

Microemulsions

A microemulsion may be defined as a thermodynamically stable dispersion of oil and water stabilized by a surfactant. A microemulsion has a microstructure (typically droplets) with small oil and water domains (approximately 100 Å) separated by a monolayer of surfactant (*3*). The variables which affect the phase behavior and microstructure of microemulsions have recently been reviewed in several publications (*4–7*). Such variables include surfactant type and concentration, cosolvent type and concentration, temperature, and pH. Microemulsions can be water-continuous [lower phase or oil-in-water (o/w) type], bicontinuous (middle phase) or oil-continuous [upper phase or water-in-oil (w/o) type]. Microemulsions exhibit ultralow interfacial tensions (less than 0.01 dyn/cm) under appropriate conditions of temperature and salinity when three phases exist in equilibrium: a middle phase microemulsion, an excess oil phase, and an excess brine phase. This property is of particular interest to enhanced oil recovery where ultralow interfacial tensions are necessary to displace oil ganglia trapped underground by capillary forces (*4*). In summary, microemulsions are thermodynamically stable while coarse emulsions are not.

Coarse Emulsion Liquid Membranes

Coarse emulsion liquid membranes employ an immiscible liquid which separates two mutually miscible phases. The desired chemical species (referred to as the solute) is selectively transported from the feed phase across a thin liquid film of the immiscible phase and enriched in the receiving phase. The phases involved are stabilized by forming an emulsion of the membrane and one of the other phases (i.e., the feed phase, or as assumed in Fig. 1, the receiving phase). The emulsion is then dispersed as macrodrops (typical diameters of 0.1–5.0 mm) into the feed phase which contains the solute. The solute is transported across the thin membrane film to the internal receiving phase droplets (see Fig. 1). The membrane phase contains surfactant as a stabilizing agent and a solvent (e.g., hydrocarbon) which might contain complexing agents which help facilitate mass transport across the membrane phase. The main advantage of emulsion liquid membranes over other separation techniques is the large surface area available

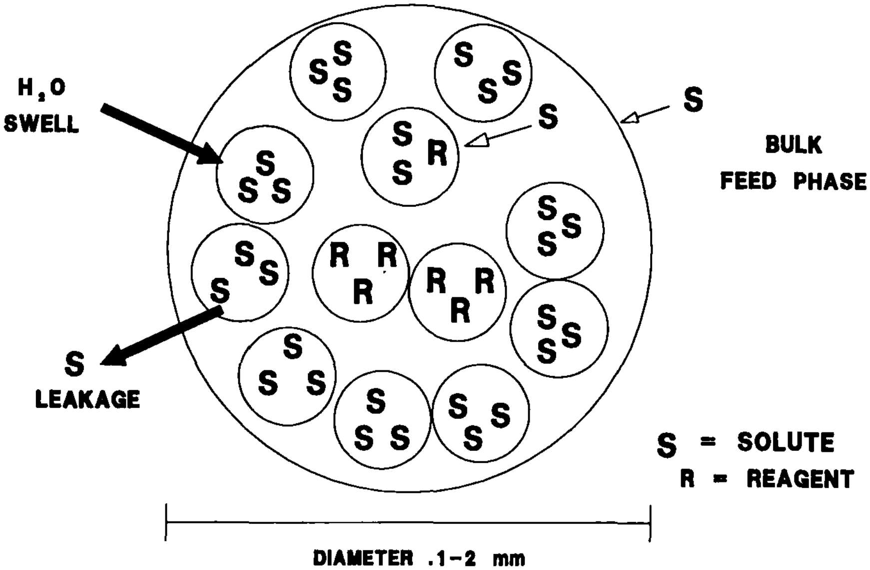

FIG. 1. A typical emulsion liquid membrane. The larger circle represents a macrodrop (typical diameters of 0.1 to 2 mm) which is dispersed into a feed solution containing the solute (S) to be separated. Microdrops are encapsulated within the macrodrop and contain an internal reagent (R) which binds the solute (S). The emulsion is stabilized by surfactant. Swell occurs when water permeates the drop in addition to the solute. Leakage occurs if the microdrops release their content into the external feed phase.

for mass transfer which results in a fast rate of separation. Two disadvantages of the technique have been: 1) the lack of stability of the emulsion which allows leakage of the solute and unreacted internal reagent back into the feed phase; and 2) swelling of the internal microdrops with water from the feed phase. These problems are illustrated in Fig. 1. Both effects result in reduced separation efficiency. Other separation mechanisms have been outlined elsewhere (*8*) and will not be repeated here.

Microemulsion Liquid Membranes

A microemulsion suitable for use as a liquid membrane must fulfill several constraints. The microemulsion must be oil-continuous if utilized to separate species from an aqueous feed phase. The receiving phase should constitute a significant volume fraction of the microemulsion in order to allow for high separation capacity. The microemulsion should be tolerant to large pH changes because pH is often used as the driving force for separation. Therefore, nonionic surfactants are ideal candidates for this application due to low sensitivity to pH. Finally, the microemulsion should not contain components which easily partition into the aqueous feed phase. This requirement limits the use of cosurfactants to those which are water immiscible. Finding a microemulsion which fulfills all of the above constraints is nontrivial. Coarse emulsions can be formulated to contain any concentration of the various constituents. Microemulsions, on the other hand, attain a thermodynamic equilibrium which often limits the amount of receiving phase which may be incorporated into the microemulsion. Phase behavior studies have been conducted and used to optimize the microemulsion formulations for separations discussed here (*9*, *10*). Such studies are necessary in view of the thermodynamic constraints on solubilization in microemulsions.

Advantages of Microemulsion Liquid Membranes

An obvious question arises, "Why use microemulsions if coarse emulsions are already proven to be effective?" The answer lies in the advantages afforded by replacing coarse emulsions with microemulsions. The low interfacial tensions which are characteristic of microemulsions will lead to smaller macrodrops which implies faster mass transfer rates due to increased surface area per unit volume. The microdrops dispersed in a coarse emulsion will coalesce over time and phase separate. Such phase separation will undesirably result in leakage of the receiving phase into the feed phase. Microemulsions do not show such phase separation due to their thermodynamic stability and may offer a more stable liquid membrane. Another key advantage is the ease of both emulsification and demulsification provided by the microemulsion system. A simple adjustment of temperature can cause spontaneous emulsification or demulsification of the microemulsion. Formation of the microemulsion requires no special mixing; however, gentle mixing is usually provided in order to minimize the time required for the microemulsion to form spontaneously. The experimental results presented below support all of these claims.

Related Work

The removal of acetic acid via a coarse emulsion liquid membrane was originally studied by Li, Terry, and Ho (*11*). This sytem was chosen for investigation in this work in view of the ease of quantitative analysis and its well-defined chemistry. The mechanism of separation is depicted in Fig. 2. Acetic acid (HAc) is soluble in the organic phase of the emulsion liquid membrane in its undissociated form. Thus, HAc partitions from the aqueous phase into the liquid membrane macrodrop and diffuses inward. Upon encountering the receiving phase microdrops which contain NaOH,

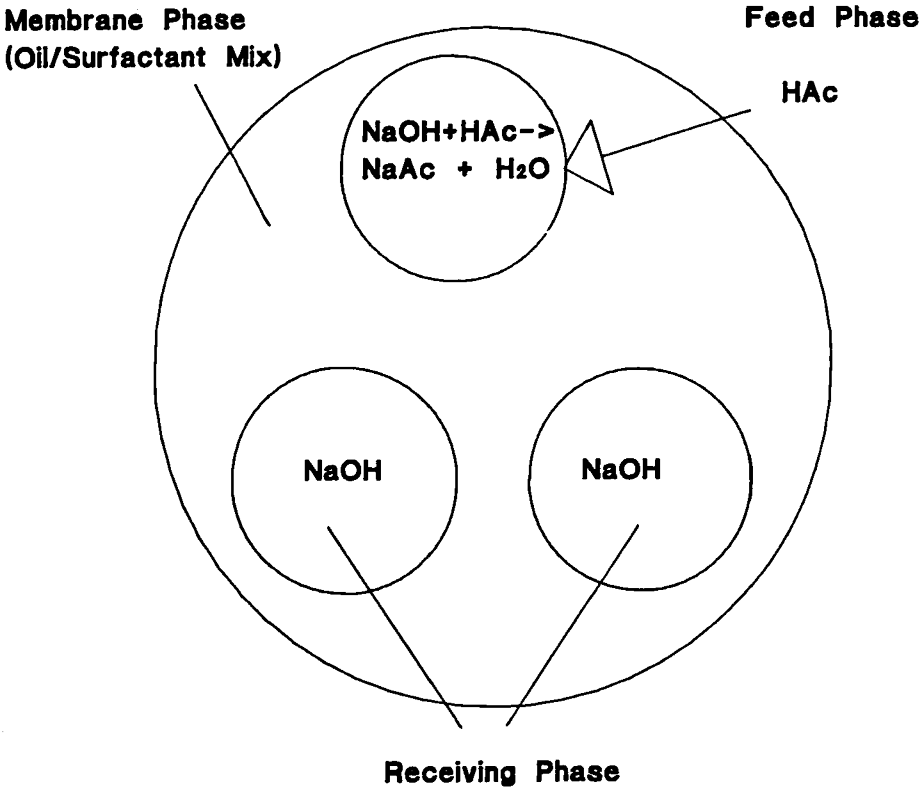

FIG. 2. Mechanism for acetic acid (HAc) removal from an aqueous feed solution. The internal reagent is sodium hydroxide (NaOH) which "binds" the acetic acid by reacting to form sodium acetate (NaAc) which has negligible solubility in the oil portion of the emulsion.

the HAc reacts to form sodium acetate (NaAc). NaAc is insoluble in the organic membrane phase and is isolated from the original feed phase.

The utilization of surfactants in various separation schemes is the focus of much research (*12*). For example, Bauer and Komornicki (*13*) utilized the large surface area per unit volume available in microemulsion phases to increase the rate of germanium extraction from aqueous solution into an organic, microemulsion-forming phase. In fact, many investigators (*13–15*) suggest that the extraction of metals into an organic phase involves the formation of reverse micelles or microemulsion droplets. Robbins obtained a patent in 1969 describing a separation technique which purified hydrocarbons based on selective solubilization into a microemulsion phase (*16*). Later, Nagarajan and Ruckenstein (*17*) realized that the solutes in the aqueous phase may selectively partition into a micellar phase and thus effect separation. Goklen and Hatton (*18*), based on earlier work by Liusi and coworkers (*19*), found that even proteins will selectively separate into a reverse micellar phase based on simple electrostatic interactions. Dunn, Scamehorn, and Christian (*20*) utilized micelles to enhance the removal of organics from aqueous streams via ultrafiltration. In this scheme, the organics dissolve into the large surfactant aggregates which are not small enough to penetrate the ultrafiltration membrane. Most of the above-mentioned investigators have utilized ionic surfactant systems. However, the nonionic surfactant systems may be utilized in similar schemes. For example, Flaim and Friberg (*21*) demonstrated that a middle phase nonionic microemulsion provides an extraction media with high surface area per unit volume and, thus, fast rates of extraction. In addition, owing to the temperature sensitivity of such systems, the various phases are easily demulsified and recovered.

Most of the investigations mentioned above rely, to some extent, on the equilibrium properties of the surfactant solution, and none of these studies involves a scheme which is analogous to the emulsion liquid membrane technique. Experimental studies of transport through microemulsion phases (i.e., microemulsion serving as a membrane) as opposed to into microemulsion phases (i.e., microemulsion serving as a receiving phase) are limited. Tondre and Xenakis (*22*) utilized both oil-continuous and water-continuous microemulsion phases as membrane phases. However, the investigators limited their studies to a U-tube apparatus which displayed very low rates of mass transfer due to the relatively small surface area per unit volume. Wiencek and Qutubuddin (*9*) discussed the characteristics a microemulsion should possess if utilized as an emulsion liquid membrane and presented preliminary results of a microemulsion liquid membrane system utilized to separate acetic acid from water. These preliminary results will be embellished here. Rautenbach and Machhammer (*23*) mistakenly termed their emulsion liquid membrane systems microemulsions. The data in their paper clearly indicate the system to be a macro- or coarse emulsion (*24*).

EXPERIMENTAL PROCEDURE

Solution Preparation

A microemulsion was formulated by taking a 10% v/v stock solution of a twin-tailed, nonionic surfactant (ethoxylated dinonyl phenol, Emery DNP-8) in an alkane (decane, dodecane, tetradecane, or hexadecane) and equilibrating it with an equal volume of 0.25 *M* NaOH(aq). After equilibration, the composition of the microemulsion was estimated by noting the phase volumes (i.e., the volume of microemulsion phase and the volume of excess aqueous phase) and assuming all of the surfactant is in the microemulsion phase. This microemulsion was utilized as a liquid membrane in subsequent experiments. The feed solution containing the acetic acid which was to be separated (i.e., the solute) was prepared from glacial acetic acid and the concentration confirmed via titration with 0.01 *M* NaOH. All experiments and solutions were maintained at 21.5°C except as noted.

Separation Vessel and Sampling Procedure

All separations were conducted in a 2-L resin flask using a marine-type impeller for agitation. The feed phase (aqueous solution of acetic acid) was first placed in the flask and the impeller speed calibrated by using a phototachometer. The mixer was stopped after calibration and the microemulsion liquid membrane added. As soon as the membrane was added, the mixer and a stopwatch were started. Samples were taken over time through a stopcock located in the bottom of the flask. The number of samples was kept to a minimum in order to minimize the change in feed volume. Sample size was approximately 10 mL. These samples were immediately centrifuged to remove any residual microemulsion liquid membrane.

Analytical

Titration of the samples with 0.01 *M* NaOH allowed for the determination of the acetic acid concentration to ±6 ppm. Sodium content in the feed phase at the end of the experiments was determined by a sodium selective electrode which was calibrated with standard solutions. Sodium analysis allowed the determination of leakage rates. The concentration of NaOH in the internal phase was needed to conduct a material balance. This analysis required the removal of the liquid membrane from the flask after a separation was completed. The microemulsion liquid membrane was centrifuged to remove any entrained feed phase. The clarified microemulsion liquid membrane was then heated to 60°C and held at that temperature for 1 day to induce spontaneous demulsification. One milliliter samples of the demulsified aqueous phase were titrated with 0.01 *M* HCl to determine the amount of unreacted NaOH in the internal phase.

Characterization of Microemulsion

The oil–water–surfactant solution described above was analyzed for the presence of microstructure via dynamic light scattering (Brookhaven PCS system) and for low interfacial tension via a spinning drop tensiometer.

RESULTS AND DISCUSSION

Microemulsion Characterization

As mentioned above, the experiments discussed in this paper utilized a twin-tailed surfactant (ethoxylated dinonyl phenol). Single-tailed, nonionic surfactants were also tested but showed unacceptable leakage and were, therefore, abandoned. The twin-tailed system displayed a bluish color (the Tyndall effect) and a high aqueous content (as high as 30% v/v) as compared to neat organic phases. In addition, the system was optically isotropic when viewed through cross-polarized screens. This behavior precludes the presence of liquid crystalline phases and supports the supposition that the systems are microemulsions. Although upon visual observation the system looked like a microemulsion, a more quantitative characterization was conducted in order to assure the system was indeed a microemulsion. Phase diagrams were constructed and compared to other microemulsion systems. The microdrop sizes were measured via dynamic light scattering, and the interfacial tension was measured via the spinning drop method. The details of the experiments are presented below, and the results are positive evidence that the system employed in this study is indeed a microemulsion.

Solubilization Behavior

The solubilization of an aqueous phase into an oil-continuous microemulsion typically displays several characteristics as a function of temperature, oil type, electrolyte type, and electrolyte concentration. A complete investigation of the DNP-8 system has recently been published (*10*) and is typical of the behavior seen in nonionic microemulsions. To summarize, the effect of increasing NaOH concentration at a fixed temperature and oil type is to decrease the amount of aqueous phase solubilized. Increasing alkane chain length (while fixing other variables) results in increasing the amount of aqueous phase solubilized. Finally, increasing temperature results in a reduced solubilization of the aqueous phase. The amount of aqueous phase solubilized ranged from approximate 30% v/v down to essentially zero. In fact, the changes in solubilization caused by manipulation of one of these thermodynamic variables can be utilized to demulsify the microemulsion. For example, the system employed in the studies presented here required a change in temperature from 21.5 to 60°C to decrease the solubilization to essentially zero.

Interfacial Tension and Microdrop Size

Four milliliters of a stock solution containing 10 w/w % DNP-8 in tetradecane was equilibrated with 3 mL of 1.0 *M* NaOH at 35°C. The high electrolyte concentration and temperature resulted in a sample with a significant amount of excess aqueous phase as well as moderate aqueous phase content. These attributes allowed for the determination of interfacial tension by the spinning drop technique as well as the droplet size by dynamic light scattering.

The aqueous volume fraction for this particular sample was determined to be 0.045 ± 0.0025 and the interfacial tension was determined to be 0.18 ± 0.04 dyn/cm. The sample was filtered and centrifuged before conducting light-scattering tests on a Brookhaven PCS unit. The details of particle sizing by light scattering may be found elsewhere (*25*). The data were analyzed using a second cumulant analysis which gives information on the mean particle size and polydispersity. The mean particle diameter was measured to be 156 Å (±1% relative) and the polydispersity index was measured to be 0.176 (±4% relative), which is considered monodispersed for microemulsion systems (*26*). Note that both the size of the droplet and the interfacial tension are of the order of magnitude expected for microemulsions.

Material Balance, Product Recovery, and Leakage

Having obtained a system which could be termed a microemulsion, a separation via the liquid membrane technique was carried out. A material balance was utilized to determine where the acetic acid was accumulating. A tetradecane microemulsion liquid membrane was contacted with an aqueous solution containing 590 ppm acetic acid. The treat ratio (volume of microemulsion : volume of aqueous feed solution) was 1 : 5 and the phases were contacted for 5 min at 200 rpm. The two phases were allowed to settle and were then separated. The tetradecane microemulsion was demulsified and the recovered internal reagent was titrated. There was a net consumption of 2.1 mmol NaOH in the internal phase and a net loss of 2.3 mmol HAc in the external feed phase. The material balance agreed to within roughly 10%, which is quite acceptable in view of the amount of sample handling required for this particular experiment. The leakage in this run was zero and, in general, was very low for all of the experiments. The highest leakage rate was 24 ppm NaOH/h (with a feed concentration of 300 ppm) but typically it remained at zero. The material balance also proved that the separated species can be recovered from the internal receiving phase by thermal demulsification. The material balance calculations assume that no swelling occurs. If swelling does occur, the concentration of the internal NaOH at the end of the experiment would be lower than in the absence of swelling. Thus, if swelling had occurred, the material balance calculations would have resulted in more moles of NaOH apparently consumed than HAc consumed. At least for this short duration experiment, swelling appears to be negligible.

Effect of Mixing Speed

The primary effect of mixing is to disperse the microemulsion phase into the aqueous feed phase. The average size of the macrodrops will decrease as agitation is increased. As can be seen in Figs. 3–5, separation is very

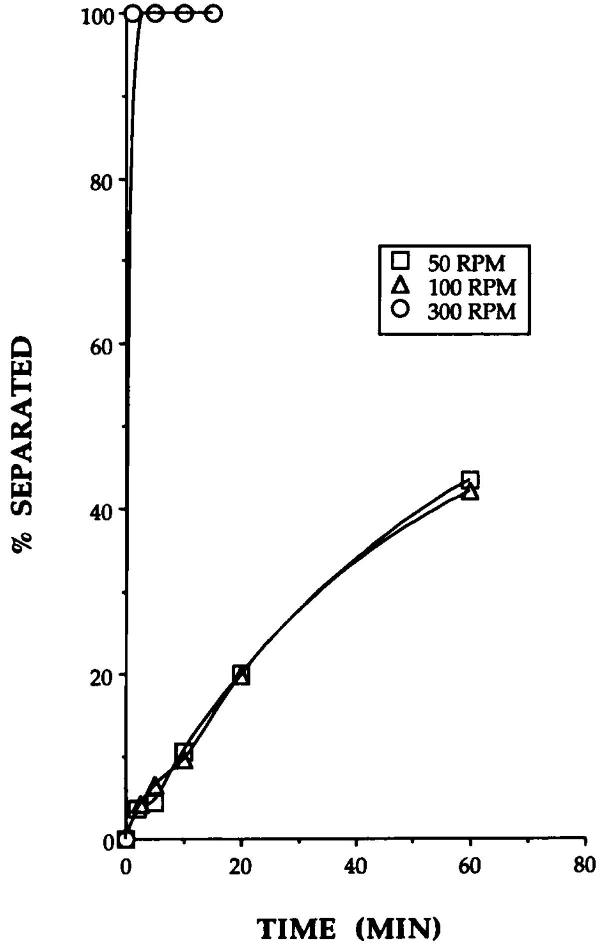

FIG. 3. The effect of mixing rate on separation kinetics for a microemulsion liquid membrane employing hexadecane as the oil phase. Experimental conditions: treat ratio (volume of microemulsion : volume of feed solution) of 1 : 5 and feed solution containing 300 ppm of acetic acid.

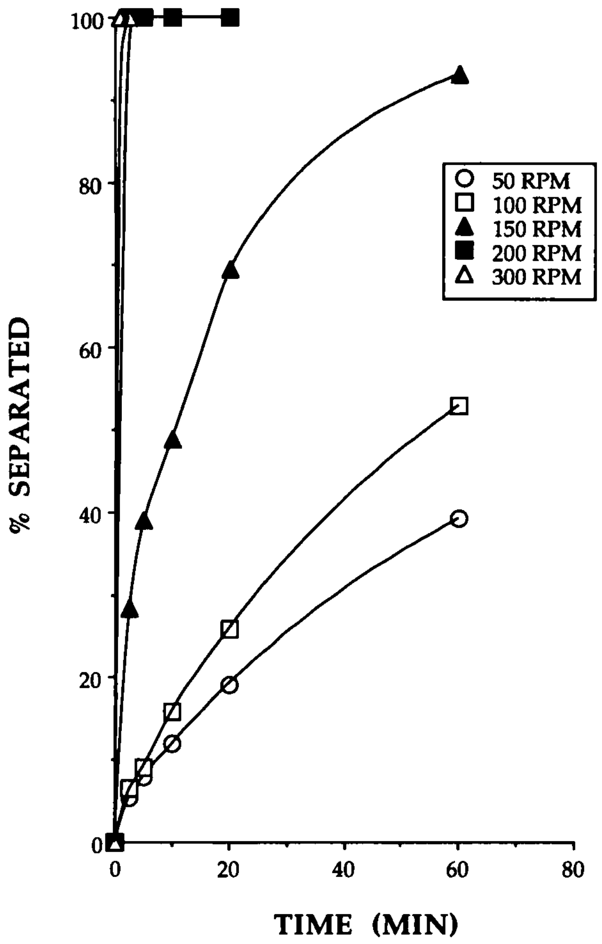

FIG. 4. The effect of mixing rate on separation kinetics for a microemulsion liquid membrane employing tetradecane as the oil phase. Experimental conditions: treat ratio (volume of microemulsion : volume of feed solution) of 1 : 5 and feed solution containing 300 ppm of acetic acid.

fast if the mixing speed is 200 rpm or higher. Below 200 rpm, the microemulsion is not fully dispersed, tending to become less dispersed as the mixing speed is decreased. Thus, the increase in the separation rates with increased mixing speed is due to the increased surface area available for mass transfer. Note that once full dispersion is achieved, the rates are faster than the sampling rate. For this reason, other variables were generally tested at a lower mixing speed of 100 or 50 rpm. Note that the

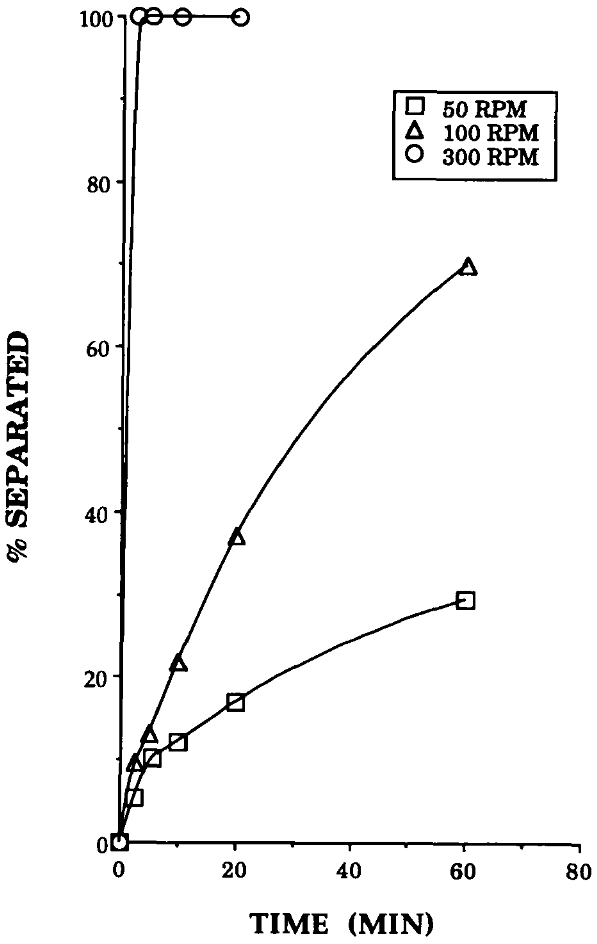

FIG. 5. The effect of mixing rate on separation kinetics for a microemulsion liquid membrane employing dodecane as the oil phase. Experimental conditions: treat ratio (volume of microemulsion:volume of feed solution) of 1:5 and feed solution containing 300 ppm of acetic acid.

experiments are reproducible as shown for a decane microemulsion liquid membrane separation in Fig. 6.

Effect of Feed Concentration

If the initial concentration of acetic acid is increased in the feed, the acetic acid will consume more internal reagent over the course of the separation. Assuming an advancing reaction front model is valid for this

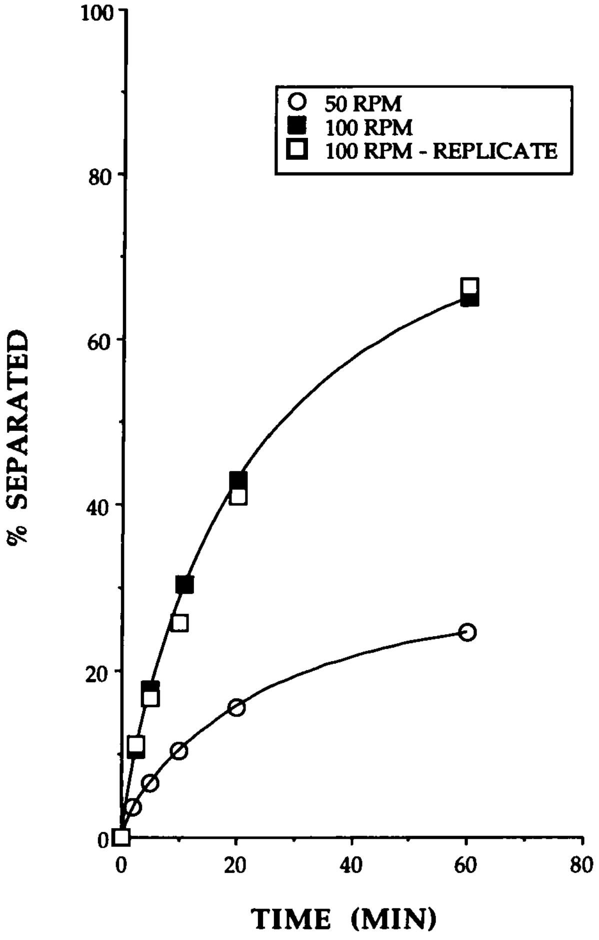

FIG. 6. The effect of mixing rate on separation kinetics for a microemulsion liquid membrane employing decane as the oil phase. Note the reproducibility of the experiment. Experimental conditions: treat ratio (volume of microemulsion:volume of feed solution) of 1:5 and feed solution containing 300 ppm of acetic acid.

separation (*27*), this increased consumption translates into longer diffusion lengths for the acetic acid over time. Thus, the behavior depicted in Fig. 7 is expected; that is, the higher feed concentrations lead to a slower rate of separation as the separation progresses. Although the initial rates may be similar, the average relative rates as shown in Fig. 7 are slower for the higher concentrations of acetic acid in the feed.

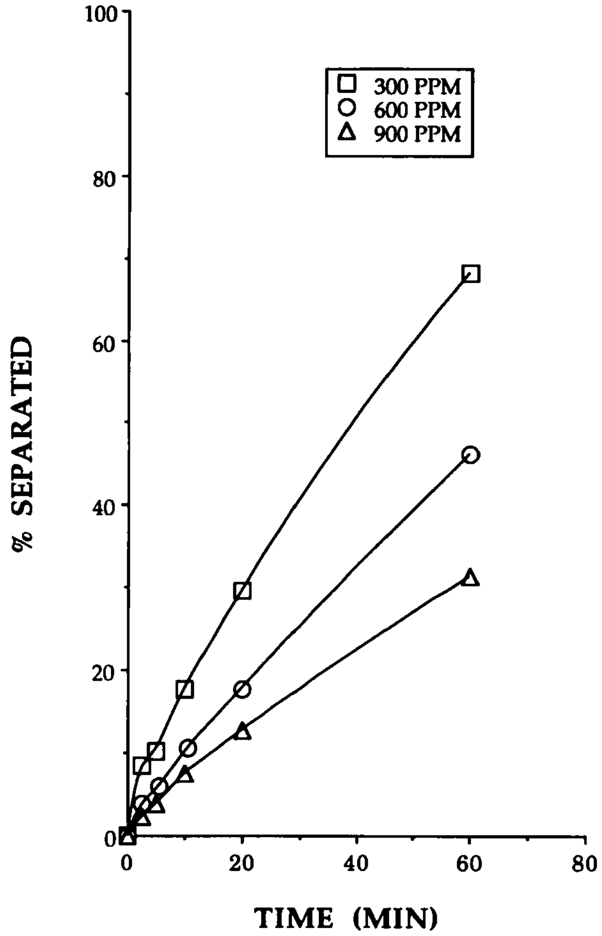

FIG. 7. The effect of feed concentration on separation kinetics for a microemulsion liquid membrane employing tetradecane as the oil phase. Experimental conditions: treat ratio (volume of microemulsion:volume of feed solution) of 1:3 and mixing speed of 100 rpm.

Effect of Treat Ratio

The effect of treat ratio (volume of microemulsion:volume of aqueous feed solution) is depicted in Fig. 8. the higher treat ratios result in faster separation rates due to the favorable shift in stoichiometry (i.e., increase in amount of NaOH per HAc fed). At a low treat ratio such as 1:4, the total amount of NaOH contained within the microemulsion is stoichiometrically equal to the amount of HAc in the feed. Thus, near the end of the separation, the reaction front is located at the center of the macrodrop,

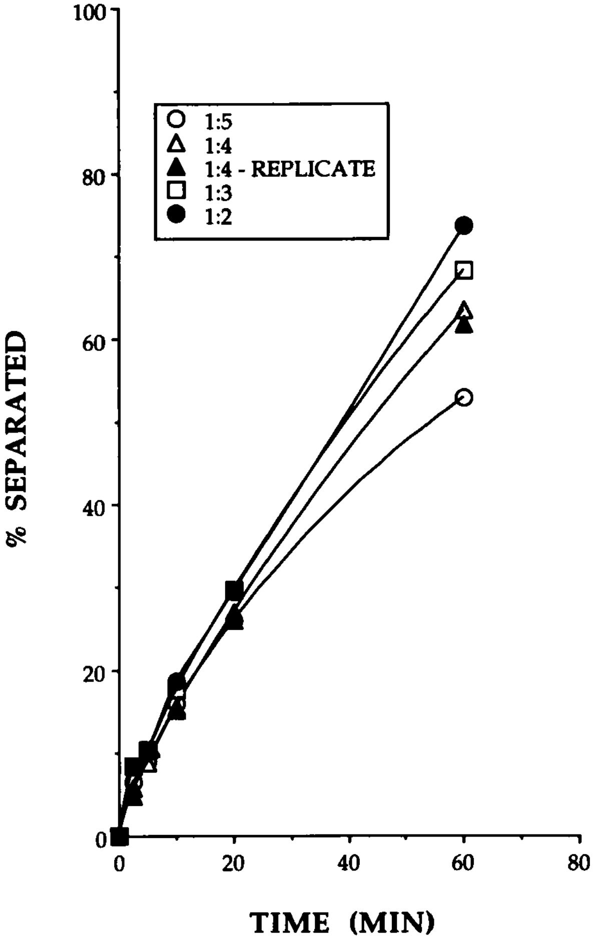

FIG. 8. The effect of treat ratio (volume of microemulsion:volume of feed solution) on separation kinetics for a microemulsion liquid membrane employing tetradecane as the oil phase. Experimental conditions: feed solution containing 300 rpm of acetic acid and mixing speed of 100 rpm.

and diffusion must take place over a length equal to the macrodrop radius. Increasing the treat ratio results in an increase in the number of macrodrops. Thus, the final location of the reaction front is closer to the macrodrop/feed solution interface. This decreased diffusion length results in faster rates of separation for higher treat ratios. Note that the differences between the data sets in Fig. 8 is significant in view of the reproducibility demonstrated in the 1:4 treat ratio experiment.

Effect of Viscosity at 50 rpm

It is expected that viscosity would directly affect the separation rates via its impact on the diffusivity of the acetic acid in the microemulsion phase. However, increasing the viscosity by a change in the alkane used in the microemulsion formulation also results in increased solubilization of NaOH due to the concomitant change in the microemulsion phase behavior. Figure 9 shows that at 50 rpm the external phase mass transfer resistance is dominating the behavior for all of the microemulsions, independent of viscosity,

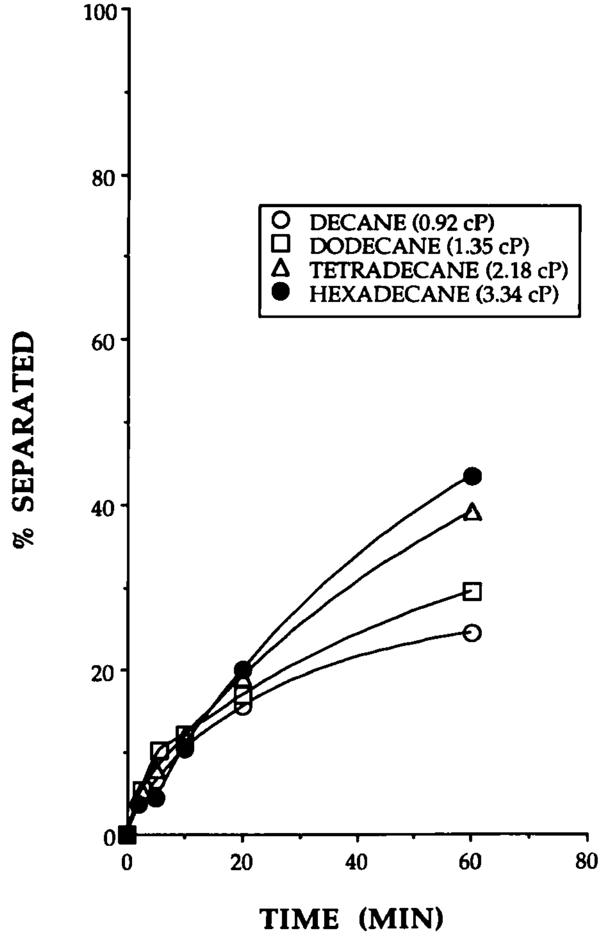

FIG. 9. The effect of viscosity (i.e., oil phase) on separation kinetics. Experimental conditions: treat ratio (volume of microemulsion : volume of feed solution) of 1 : 5, feed solution containing 300 rpm of acetic acid and mixing speed of 50 rpm.

for the first 20 min. At times greater than 20 min. the rates diverge as the globules are more deeply penetrated. This behavior is in accord with the advancing reaction front model. The higher alkanes have a higher loading of NaOH; therefore, the HAc does not have to penetrate the higher alkanes as deeply as the shorter alkanes. On the other hand, it may be argued that the lower diffusion coefficient of the tetradecane phase will more than offset the increased loading in NaOH. However, this argument can be dismissed by simple dimensional analysis. For a given amount of separation, $\mathcal{D}t/L^2$ is constant if diffusion is the rate limiting step ($\mathcal{D}$ is the diffusion coefficient, t is time, and L is the characteristic length). The diffusion coefficients change from 2.36×10^{-5} cm^2/s for decane to 1.34×10^{-5} cm^2/s for tetradecane. However, the amount of NaOH in the decane is only half of that in tetradecane. Assuming a planar geometry (valid at 50 rpm), this solubilization data implies that the diffusion length in the decane microemulsion is always twice that of the tetradecane microemulsion. This type of argument results in the prediction that the amount of time for a tetradecane membrane to obtain a given amount of separation should be roughly 0.44 of the amount of time it takes a decane microemulsion to obtain that same amount of separation. Indeed, comparing the times necessary to obtain 20% separation, a time ratio of roughly 0.5 is observed.

SUMMARY

A separation technique utilizing nonionic microemulsions as emulsion liquid membranes has been successfully applied to the removal of acetic acid from an aqueous feed phase. This work is the first successful utilization of a microemulsion as a dispersed liquid membrane. The systems under investigation were carefully characterized in order to assure that they were truly microemulsions. The effects of mixing intensity, feed concentration, treat ratio, and microemulsion viscosity on the separation kinetics are rationalized on the basis of the advancing reaction front model. Typically, the microemulsions did not display leakage and had negligible swell over a 5-min duration. The reversible phase behavior of the microemulsion was utilized to demulsify the liquid membrane phase and recover the acetate ion via a temperature increase. Material balances closed to within 10% and rates of separation were faster than the sampling rates when the microemulsion was fully dispersed in the aqueous feed phase. The extension of the technique to coupled transport for copper ion separation is the subject of the second paper in this series.

Acknowledgments

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