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Development of a Continuous Electric Coalescer of W/O Emulsions in Liquid Surfactant Membrane Process

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Abstract

The development of an efficient, continuous demulsification apparatus is needed in the industrial separation process utilizing the liquid surfactant membrane technique. A test apparatus for continuous demulsification was constructed in which a pulsed dc high potential is applied to W/O emulsions flowing between parallel copper electrodes of a perforated plate. Efficient demulsification was achieved by an operation in which the demulsified oil phase was absent between the emulsion and the upper electrode. When the feed rate of emulsion was adjusted to be equal to the demulsification rate, the latter value was proportional to the square root of the applied potential and independent of the distance between the electrodes.

INTRODUCTION

In the separation and concentration process employing the liquid surfactant membrane technique, it is necessary for a W/O emulsion to be separated after the permeation and concentration step into an enriched aqueous phase, which is recovered, and into an oil membrane phase, which is reused repeatedly. Therefore, the development of a technique which can demulsify W/O emulsions continuously and efficiently is needed in the industrial application of this separation process.

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Of the many different methods of demulsification, the electrical method was suggested to be effective in demulsifying a W/O emulsion in the liquid surfactant membrane process (*1*). Since then, several fundamental and experimental studies have been carried out to investigate the effects of operating conditions on the degree of demulsification by applying a dc or ac electric field (*2–9*). Continuous opertions using an electrical demulsification apparatus have also been studied (*10, 11*), but more research is needed to develop an efficient coalescer applicable to the industrial liquid membrane process.

The authors studied the demulsification of W/O emulsions by application of pulsed dc high potential in batchwise operation, and indicated the usefulness of this method (*7, 8*). In the present study, with the objective of developing a continuous demulsification apparatus, a test apparatus was constructed on the basis of our above-mentioned electrical method. Batch demulsification experiments were carried out by using the apparatus to obtain fundamental information for a continuous operation. On the basis of these results, continuous demulsifications were subsequently performed to investigate the effects of operating conditions on the demulsification rate.

EXPERIMENTAL

Apparatus

The demulsification apparatus tested in this study is schematically shown in Fig. 1. The apparatus consists of an acrylic resin box with inside dimensions of 13 cm length, 5 cm width, and 7 cm height. On each side wall, six grooves of 1 mm for inserting an electrode are equipped at a distance of 1 cm. Two bare copper electrodes made of a perforated plate were installed parallel to each other in the box. They had 72 or 18 holes of 3 mm diameter in a regular triangle arrangement. The distance between them could be changed from 1 to 5 cm by inserting the lower electrode in the lowest groove and the upper one in the appropriate other groove. Figure 1 represents the 3-cm distance case. The upper electrode was connected to a high voltage dc generator (Noise Laboratory Co., Ltd.) and the lower one was grounded. Demulsification experiments of W/O emulsions were performed by applying a pulsed dc high potential for a period of 0.6 s between the upper and lower electrodes.

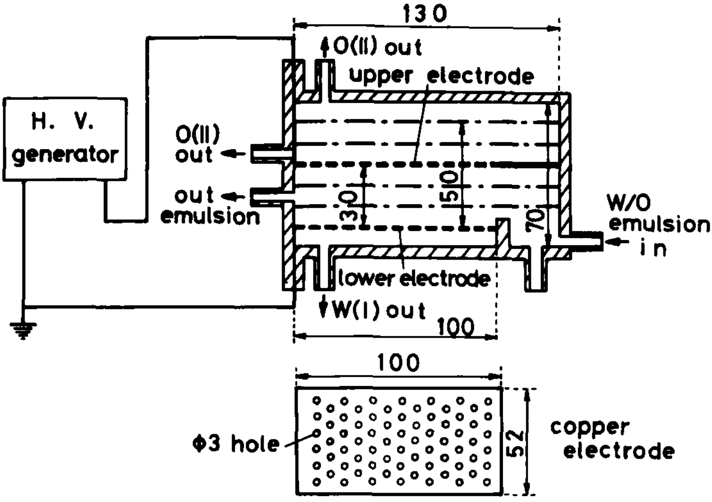

FIG. 1. Experimental apparatus for demulsification.

Procedure

After an internal water phase W(I) was supplied until it came in contact with the lower electrode, a W/O emulsion was fed through the emulsion inlet into the apparatus by means of a microtube-pump. As soon as the emulsion came in contact with the upper electrode, a high potential was applied between the electrodes. The emulsion flowing between the electrodes was demulsified by the electric force. The demulsified oil phase O(II) passed through the holes of the upper electrode and then effused from the apparatus through the O(II) outlet, and the demulsified water phase W(I) passed through the holes of the lower electrode and then through the W(I) outlet. The undemulsified emulsion was removed through the emulsion outlet from the middle of the emulsion phase height at a rate which was adjusted according to the feed rate because, for efficient demulsification it was necessary to operate in a condition where the emulsion just kept filling the space between the electrodes. The amount of demulsified emulsion, i.e., the demulsification rate, was determined by measuring the effusing rates of the oil and water phases, respectively, or the feed and removal rates of the emulsion in the steady-state. The demulsification rates were measured for different applied potentials and distances between the electrodes, and the residual water content in the demulsified oil phase was analyzed by the Karl-Fisher method. In addition, batch experiments were performed by using the same apparatus in order to obtain fundamental information for continuous operation.

The following system for concentrating copper ion by LIX64N was adopted as an experimental system.

W(I): 0.5 kmol/m^3 H_2SO_4
O(II): 10 vol% LIX64N–4 vol% Span 80 or ECA4360J (polyamine)–kerosene
W(III): 10 mol/m^3 $CuSO_4$–0.25 kmol/m^3 ($CH_3COOH-CH_3COONa$) buffer solution

The experimental procedure for the permeation and concentration of copper ions by liquid surfactant membranes was the same as described in previous papers (*12*, *13*), and the experimental conditions are shown in Table 1.

CIRCUMSTANCES OCCURRING DURING DEMULSIFICATION OPERATION

When continuous demulsification is performed as mentioned above, the two cases represented in Fig. 2 appear depending on the operating

TABLE 1
Experimental Conditions for Liquid Surfactant Membrane Permeation

W(I)/O(II) emulsion:
- Volume ratio, W(I):O(II) = 100:100 cm^3
- Stirring rate, 66.7 rps
- Stirring time, 180 s

W(I)/O(II)/W(III) emulsion:
- Volume ratio, W(I):O(II):W(III) = 50:50:700 cm^3
- Stirring rate, 3.3 rps
- Stirring time, 900 s

Mean diameter of internal water droplets, about 4.8 μm
Temperature, 298 K

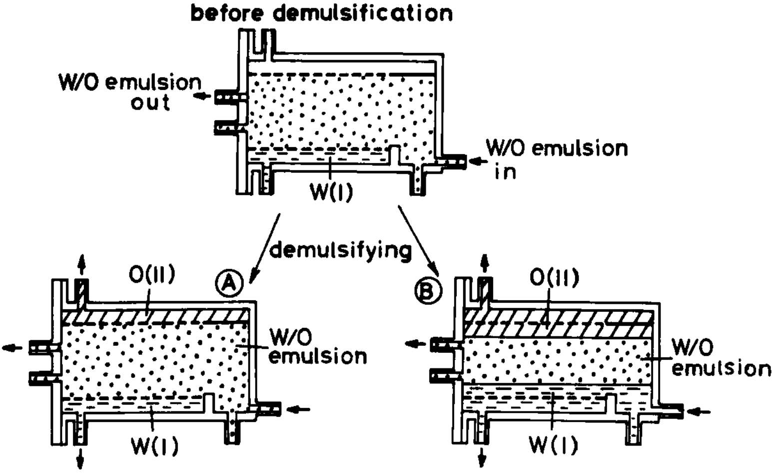

FIG. 2. Cases occurring in demulsification operation.

method and conditions. Case A occurs when the demulsification rate of emulsion just balances with the feed rate, and only the W/O emulsion phase exists between the electrodes. Case B occurs when three phases—W(I), W/O emulsion, and O(II)—coexist. Therefore, the effects on the demulsification rate of the W(I) or the O(II) phase present between the electrodes were investigated by batch experiments. Under the conditions when the W(I) phase (initial thickness t_w) or the O(II) phase (initial thickness t_0) coexisted with the emulsion phase (initial height H_0) between the electrodes, as illustrated in Fig. 3, the height H of the emulsion phase, which decreases with the progress of demulsification, was measured. A

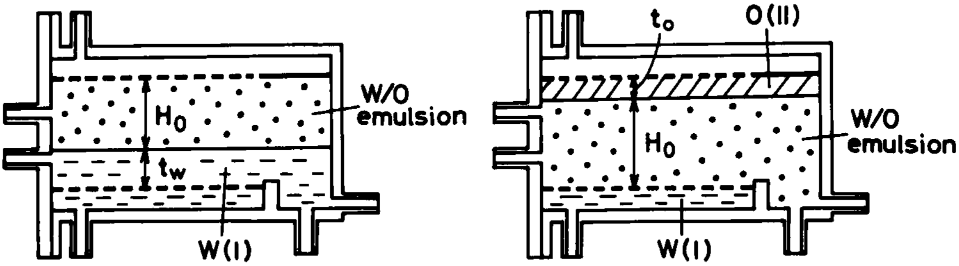

FIG. 3. Conditions for measuring the extent of demulsification.

variation of the extent of demulsification, $B = (H_0 - H)/H_0$ (7), with time was thus obtained compared with the result measured under the existence of the emulsion phase alone. The effect of the applied potential on the extent of demulsification was also investigated. Span 80 was used as the surfactant in all batch experiments.

RESULTS AND DISCUSSION

Examination of Influential Parameters with Batchwise Operation

(1) Effect of Applied Potential

The variation of extent of demulsification B with time was measured under different applied potentials V with a distance $L = 5$ cm between the electrodes and an initial height $H_0 = 5$ cm of the emulsion phase, that is, in the condition where only the emulsion phase initially existed between the electrodes. The results are shown in Fig. 4. $(W/O)_{Af}$ in the figure represents the result in which the W(I)/O(II) emulsion separated from the W(I)/

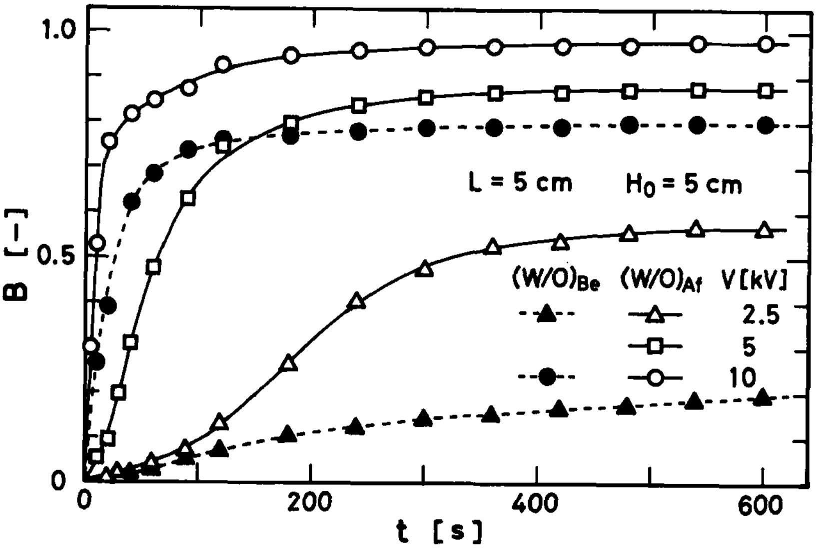

FIG. 4. Effect of applied potential on demulsification.

O(II)/W(III) emulsion after permeation and the concentration of copper ion was demulsified, and $(W/O)_{Be}$ represents the W(I)/O(II) emulsion before being supplied to the permeation step. The value of B increases and the emulsion is demulsified faster as the value of V increases. $(W/O)_{Af}$ emulsion is almost thoroughly demulsified at $V = 10$ kV, and is easily demulsified in comparison with $(W/O)_{Be}$. The above results are similar to our previous results (7) with a cylindrical, batch demulsification apparatus.

(2) Effect of Water Phase between Upper and Lower Electrodes

To investigate the effect of a water phase existing between the electrodes, variations of B with time were measured at $H_0 = 3$ cm for the case ($t_w = 0, L = 3$ cm) of the water phase being absent between the emulsion phase and the lower electrode, and the case ($t_w = 2$ cm, $L = 5$ cm) of the water phase being present in a thickness of 2 cm. There is little difference between these results, as shown in Fig. 5. It is therefore believed that the water phase plays the role of the electrode.

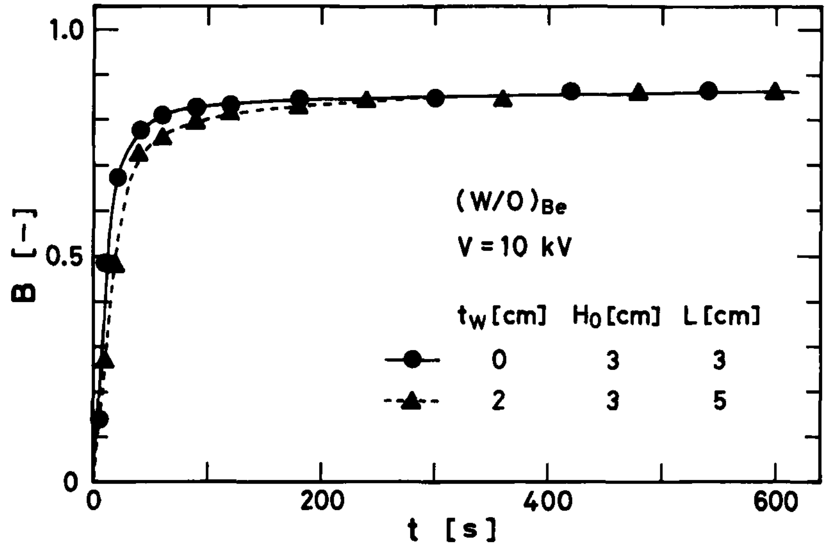

FIG. 5. Effect of water phase between upper and lower electrodes.

(3) Effect of Oil Phase between Upper and Lower Electrodes

Figure 6 shows the results when the effect of the thickness t_0 of the oil phase between the emulsion phase and the upper electrode on demulsification was similarly investigated with $L = 3$ cm. The effect of the oil phase on B is minimal when $t_0 = 1.5$ mm, but an increase in t_0 retards demulsification. A similar result was observed with $L = 5$ cm. Such a retardation may be caused by the fact that the bare electrode acted as an insulator because of the oil phase being in contact with the electrode. In continuous demulsification, therefore, it is preferable to operate in a condition where the oil phase does not existe between the emulsion phase and the upper electrode. Such an operation was performed in the following continuous experiments.

(4) Effect of Hole Fraction of Electrode

The effect of hole fraction p of the perforated electrode, which is a ratio of the area occupied by the holes to that of the electrode, was examined with $V = 10$ kV and $L = 3$ cm, and is shown in Fig. 7. By using electrodes

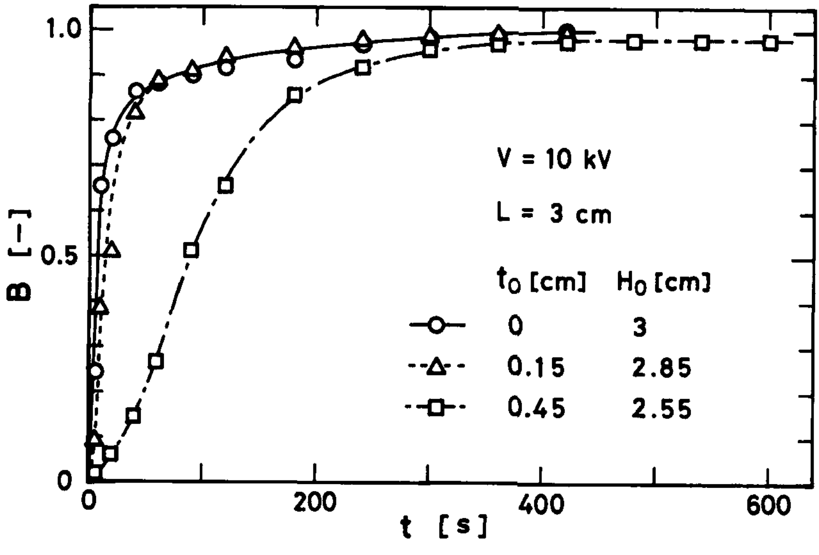

FIG. 6. Effect of oil phase between upper and lower electrodes.

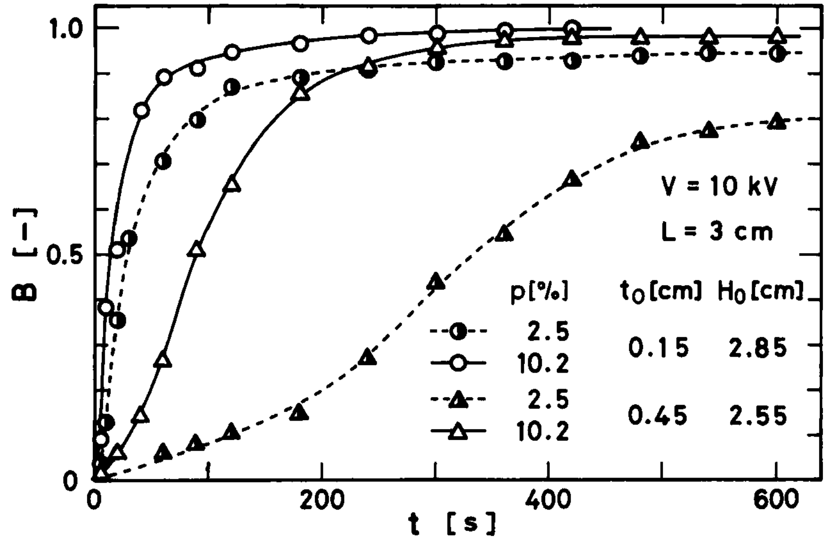

FIG. 7. Effect of hole fraction of electrode.

with a smaller p, the effect of the oil phase between the electrodes increases and demulsification becomes slow. A similar tendency was shown with $L = 5$ cm. In experiments with electrodes of a smaller p, it was observed that the formation of a chain, i.e., the alignment of water droplets with the electric field, and fluidization of the emulsion phase, which occurred upon initially applying an electric potential, did not happen. This caused the demulsification to be retarded. Therefore, electrodes with p of 10.2% were used in the following continuous operations.

Continuous Demulsification

(1) Modeling for Demulsification Process

Figure 8 is a schematic diagram of the steady-state operation for continuous demulsification. When the distance between parallel plate electrodes with dimensions of W in width and l in length is L, the volume v of the space between the electrodes is given by

$$v = LWl \qquad (1)$$

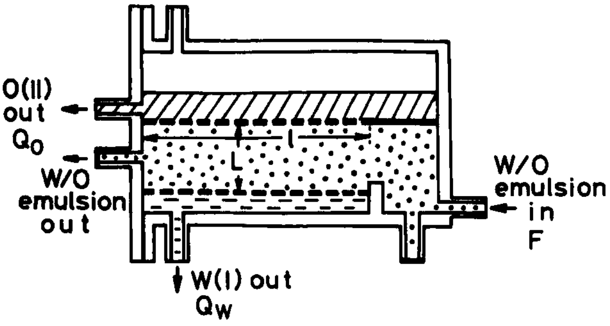

FIG. 8. Model diagram of continuous demulsification.

An emulsion fed at flow rate F is demulsified by an electric force flowing between the electrodes, and the demulsified water and oil phases are continuously effused at flow rates of Q_w and Q_o, respectively. The rate Q of the emulsion demulsified in such an operation, which is the sum of Q_w and Q_o, is assumed to be proportional to electric field strength E (= V/L) and the time needed for the emulsion to pass between the electrodes, i.e., a mean residence time τ (= v/F). The following relation is obtained:

$$Q\ (= Q_w + Q_o) \propto E\tau = (V/L)(v/F) \qquad (4)$$

Substituting Eq. (1) into Eq. (2),

$$Q \propto (V/F)Wl \qquad (3)$$

Because the product of W and l represents the area of the electrode, it is constant once the electrode to be used is determined. It can therefore be predicted that the demulsification rate Q is proportional to the applied potential V and inversely proportional to the feed rate F, and independent of the distance L between the electrodes.

In addition, at the condition of balancing $F = Q$, that is, when the feed emulsion is just demulsified during its flow between the electrodes and the removal of the emulsion is unnecessary, Q becomes equal to F. Consequently, the square of Q is predicted to be proportional to V as expressed by the following equation:

$$Q^2 \propto VWl \qquad (Q = F) \qquad (4)$$

By using $(W/O)_{Af}$ emulsions, the demulsification rates were measured for various V and L at $F = Q$.

(2) Effect of Applied Potential

The demulsification rates Q were measured for applied potential V with 3 and 5 cm as the distances L between the electrodes. The square of Q is plotted against V in Fig. 9. When Span 80 was used as the surfactant, as seen from the figure, Q^2 is nearly proportional to V with both L values, which indicates the same tendency as Eq. (4). Emulsion with ECA4360J of a polyamine was unsuccessful at a low V of 2.5 kV, while above 5 kV the proportional relation between Q^2 and V was obtained. However, the value of Q is extremely low compared with that for Span 80, and higher potentials are required for demulsification.

The residual water content in the demulsified oil phase was between 25 and 50 mg/cm^3 O(II) for Span 80 and tended to become higher with L of 5 cm than of 3 cm. For ECA4360J, it was around 20 mg/cm^3 O(II).

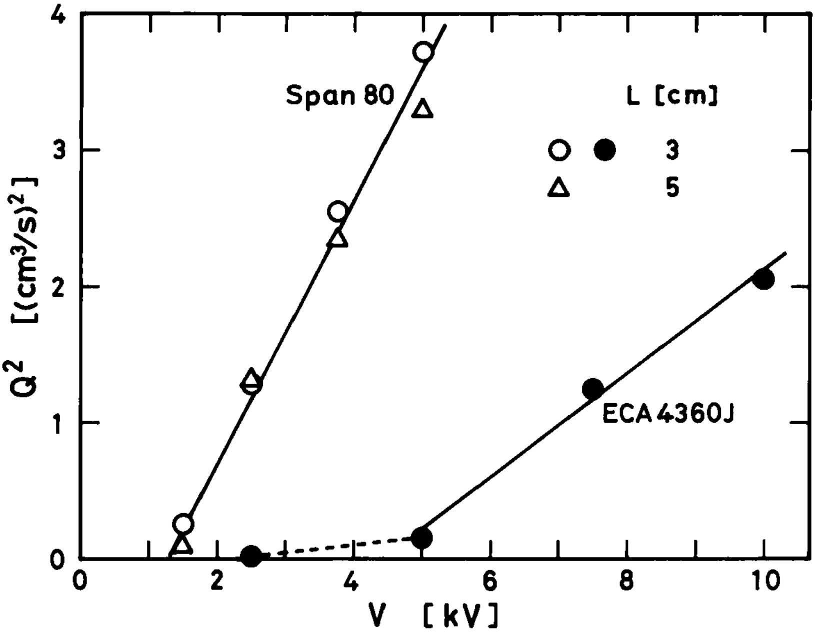

FIG. 9. Relation between demulsification rate and applied potential.

(3) Effect of Distance between Upper and Lower Electrodes

Figure 10 shows the results when values of Q are plotted against L with V as a parameter. In the demulsification of the emulsions with Span 80, the value of Q is independent of L and is nearly constant for each value of V, although there is some scatter in the data. A similar result is obtained for ECA4360J. The tendency predicted above is therefore satisfied in these results.

(4) Effect of Feed Rate

The above results were obtained with $Q = F$. The demulsification rates were next measured under conditions in which the emulsion was fed faster than the demulsification rate, that is, an operation accompanied by removal of the emulsion. The results in which emulsions with Span 80 were demulsified at V of 2.5 kV and with L of 3 and 5 cm are shown in Fig. 11. The result with L = 3 cm indicates that Q increases gradually as F increases, which is a different tendency than expected from Eq. (3). On the

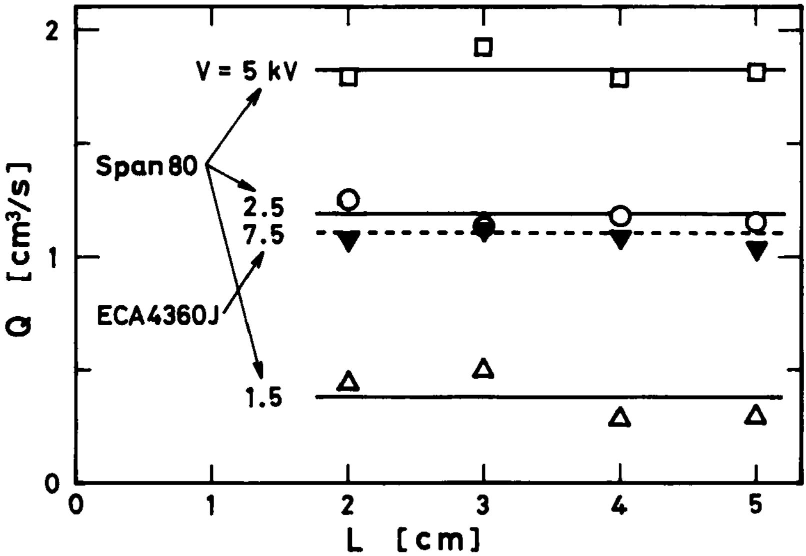

FIG. 10. Effect of distance between upper and lower electrodes on demulsification rate.

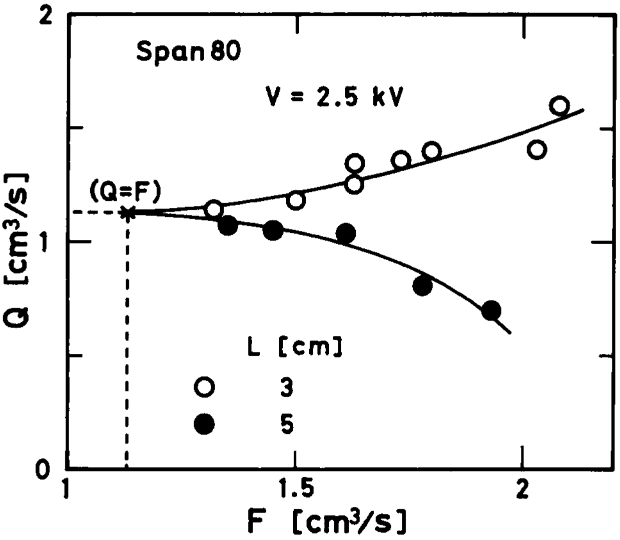

FIG. 11. Effect of feed rate of emulsion on demulsification rate.

other hand, with L = 5 cm, Q decreases with an increase of F, and the tendency is similar to the above prediction. Thus, the difference in the observed results with a change of L may be because the flow of emulsions in the apparatus changes with L due of the removal of emulsion. This is a subject to be studied in the future.

The residual water content in the demulsified oil phase was between 25 and 40 mg/cm^3 O(II). This value is nearly equal to that in the operation without the removal of emulsion, described above.

CONCLUSION

In order to develop a continuous demulsification apparatus for the separation process employing the liquid surfactant membrane technique, a test apparatus that applies an electrical method was constructed. W/O emulsions flowing between the parallel copper electrodes of a perforated plate were demulsified by applying a pulsed dc high potential between the electrodes. The effects of operating conditions on the demulsification rate were investigated.

Efficient demulsification with this apparatus was achieved by operating under the condition in which the demulsified oil phase was absent between the emulsion and the upper electrode. In such an operation, when continuous demulsification was performed under the condition in which the feed rate of emulsion was equal to the demulsification rate, with the use of either Span 80 or ECA4360J as the surfactant, the square of the demulsification rate was proportional to the applied potential and independent of the distance between the electrodes. The residual water content in the demulsified oil phase was less than 5 wt%, and thus the emulsions were found to be satisfactorily demulsified. The effect of the feed rate on the demulsification rate will be investigated in the near future.

SYMBOLS

B	extent of demulsification
E	electric field strength (kV/cm)
F	feed rate of W/O emulsion (cm^3/s)
H	height of W/O emulsion phase (cm)
H_0	initial height of W/O emulsion phase (cm)
L	distance between upper and lower electrodes (cm)
l	length of electrode (cm)
p	hole fraction of electrode
Q	demulsification rate (cm^3/s)
Q_o	effluent rate of oil phase (cm^3/s)
Q_w	effluent rate of water phase (cm^3/s)
t	time (s)
t_o	initial thickness of oil phase (cm)
t_w	initial thickness of water phase (cm)
V	applied dc potential (kV)
v	volume of W/O emulsion between upper and lower electrodes (cm^3)
W	width of electrode (cm)

Greek Letter

τ	average residence time of W/O emulsion (s)

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