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Barega, E.W.; Zondervan, E.; de Haan, A.B.

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Entrainment Reduction in a Static-Mixer Settler Setup by Electric Field Enhanced Coalescence

Esayas Barega a, Edwin Zondervan a & André de Haan a

a Department of Chemistry and Chemical Engineering, Eindhoven University of Technology, Eindhoven, The Netherlands

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This paper investigates the application of electric field enhanced coalescence for entrainment reduction in a static-mixer settler setup for a caprolactam-toluene-water and an ethylbenzene (EB)-x methyl benzyl alcohol (MBA)-water systems. The mixing of the organic and aqueous phase was done in a static-mixer and an AC/DC electric field was applied directly at the end of the static-mixer. The drop size and entrainment were measured at different flowrates, with and without the electric field, at a field strength of 0.4 kV/cm and 1 kV/cm, and at frequencies ranging from 3 to 100 Hz. Significant drops coalescence was observed which resulted in 4–5 times less entrainment. The AC field was more efficient than the DC field and an optimum frequency of 3–10 Hz was obtained. Additionally, the role of phase ratio and temperature was examined. For the caprolactam system, the entrainment reduction efficiency increased up to a phase ratio of 10% v/v and decreased again at 20% v/v. For the MBA-EB-water system, the efficiency did not change much between a phase ratio of 5% v/v and 10% v/v but decreased at a phase ratio of 20% v/v. Furthermore, entrainment decreased at high temperature for the MBA-EB-water system. However, it increased for the caprolactam-toluene-water system.

Keywords coalescence; electric-field; entrainment; frequency; static-mixer

INTRODUCTION

Entrainment

In solvent extraction processes, one phase is usually dispersed in the other immiscible phase to increase the mass transfer area and induce mass transfer. This often results in a difficult to separate dispersion that negatively influences the subsequent separation step and limits the process capacity. In such cases, a separation by gravity settlers is not sufficient. First, the separation requires a long residence time, which means larger settlers with high solvent inventories. This in turn translates into large capital and operating cost and increased safety risk, when the solvents are hazardous. Secondly, due to their size, the small droplets are easily entrained by the continuous phase. This entrainment of the dispersed phase can cause a loss of expensive solvent and/or product, accumulation of impurities in the final product, and a decrease in product quality and capacity. Consequently, the operational costs increase further in removing entrainment, and the solvent added to maintain the same capacity (1,2). Therefore, a new separation principle that avoids the disadvantages of the traditional process is desired to intensify and improve the phase separation.

Thus far, different technologies have been developed in the oil and gas industry to enhance coalescence and separation of the dispersed phase (3–5), which eventually reduce entrainment. Among the different techniques, the application of an electric field has attracted attention for a number of reasons. First, the application of the electric field does not require much space and need for modification of the existing process infrastructure. Therefore, it can easily be incorporated in the process. Second, it has low power requirements due to the very low electrical current across the dispersion. Third, it does not require intensive maintenance, as no moving mechanical parts are involved (6,7). And finally, it has been used frequently for crude oil dehydration (4,8–10) with proven and significant enhancement of phase separation; hence, it should be easily adaptable to bulk extraction processes.

Electric-Field Enhanced Coalescence

The use of the electric field enhanced separation dates back to the early work of Cottrell in 1911 (4,11), where an electrostatic precipitator was used to remove dust particles from an air stream. Afterwards, the electric field was extensively used in the petrochemical industry for the separation of emulsified water droplets from crude oil. Many mechanisms such as chain formation, dipole coalescence, electrophoresis, dielectrophoresis, and random collision were reported to account for the electric field coalescence effect (12). In the electric field, the aqueous drops acquire...
induced charges, which create a dipole-dipole attraction force between them, and this eventually causes their coalescence. Moreover, once the drops acquire charge, they move under the action of electrostatic attraction of the charged electrodes, which leads to collisions and coalescence enhancement of drops.

A plethora of literature exists on electric field coalescence with applications generally directed to the separation of water-in-crude oil emulsions. In these applications, pulsed DC and AC fields were used more frequently. In addition, different designs of electro-coalescers were employed including rectangular duct, circular vessels, and columns (12). Some new designs, for example, spinning type electro-coalescers were also recently investigated to combine the action of electric and centrifugal fields (6,13). The electro-coalescer was mostly placed between the stirred vessel and the settler, albeit in some applications, the electrodes were directly integrated into the settler. Different studies have also been made focusing on the effect of different electrical and process parameters on the efficiency of the electric field. Authors such as Bailes (13,14), Drellich et al. (8), Eow et al. (15), and Kim et al. (7) investigated the influence of electric field frequency. Different optimum frequencies were found depending on the electrical properties of the systems used, and the type and thickness of insulation used. Bailes (14) and Drellich et al. (8) found an optimum frequency below 10 Hz, while others found the efficiency to increase at higher frequencies. Additionally, field strength, hold-up fraction of the dispersed phase, and operational conditions such as temperature and physical properties, for example, viscosity and interfacial tension were found to have influence on the separation.

**Problem Statement**

Despite the abundant literature on the use of an electric field in the petrochemical industries, only few studies focus on the application to solvent extraction processes. As pointed out by Bailes (13), the application of an electric field to solvent extraction systems is hampered by two factors:

1. the high water content of solvent extraction systems causing electrode bridging due to the large number of conducting drops, and
2. the loss of electro-coalescence force between the electrodes due to modification of the conductivity of the continuous phase by the conducting drops, which lowers the efficiency and consequently results in high power consumption.

Therefore, they used insulated electrodes and pulsed DC as a means of energizing to solve the short-circuiting problem.

However, an additional challenge is encountered when the electric field is used for solvent extraction systems with low interfacial tension and in the presence of surface active components. First, the low interfacial tension reduces the drop size; accordingly, the electrical attractive force responsible for the coalescence decreases considerably. Second, the surface active compound adsorbs at the interface between the drops and the continuous phase. This hinders the coalescence process by acting as a physical barrier in preventing direct coalescence of drops by electric field. To our knowledge, the coalescence of this kind of extraction systems by the electric field in a continuous process was not investigated except for the application in emulsion liquid membranes (16–18) and in the work of Bailes and Larkai for sulphuric acid-20% LIX64N in the Escaid system (11).

The solvent extraction systems investigated in this work are characterized by low interfacial tensions, with one of their components being surface active. The systems of interest are caprolactam-toluene-water and ethyl benzene (EB)-z-methyl benzyl alcohol (MBA)-water. In the caprolactam-toluene-water system, the interfacial tension decreases to less than 5 mN/m (19) depending on the concentration of the surface active component caprolactam. Commercial fiber-grade caprolactam requires high purity (>99.9%w/w) (20). During the extraction step, while extracting the aqueous-caprolactam phase with toluene, entrainment of the aqueous phase into the organic solvent phase occurs as a result of the low interfacial tension and the formation of micro droplets. This has a number of consequences such as product dilution, decrease in product quality and production capacity, and an associated increase in entrainment of aqueous-phase soluble impurities (20). For the ethyl benzene (EB)-z-methyl benzyl alcohol (MBA)-water system, the interfacial tension can be as low as 10 mN/m (21), where MBA acts as a surface active component. This system is part of the industrial propylene oxide-styrene monomer process (22). The process step of interest for this research is the caustic (NaOH) wash and its function to remove acidic components from the major EB-MBA stream. Acidic components are first extracted by a caustic solution (pH = 12) in a static-mixer followed by a phase separation. The long residence time required in the settlers causes fouling and product loss due to caustic catalyzed side reactions. Moreover, the salty water phase entrainment to the organic phase results in the fouling of the equipment downstream, and product loss due to side reactions.

A novel concept that was not reported yet in the existing literature is the unique combination that can be achieved with static-mixers and an electric field, which is shown in Fig. 1. All experimental research on electric field coalescence was based on dispersions created by stirred vessels since these are the most widely used mixing devices in extraction processes. Nevertheless, static mixers have now gained attention as mixing devices, because they provide
a uniform droplet size distribution (23–26), require relatively less energy (23,24), and lead to a huge (23) equipment intensification with strongly reduced solvent inventory. Additionally, the use of static-mixers offers a unique opportunity since the electric-field can directly be incorporated in the same pipe as the static-mixer without the need for a separate electro-coalescer. Thus, intensification of both the mixing and the separation step can be achieved without introducing extra equipment. This is not the case in stirred tanks due to their large size; consequently, the electro-coalescer is either placed separately between the mixer and settler or integrated into the settler. Furthermore, since the diameter of the static-mixer is small compared to the dimension of the settler, placing the electric field in the static-mixer pipe enables maintaining high field strength with relatively small potential difference (voltage).

This paper investigates a novel way of entrainment reduction by applying electric field enhanced coalescence to a static mixer-settler combination. We examine the potential application of the technology for two bulk solvent extraction systems, which are characterized by a low interfacial tension and a small density difference. Additionally, the electro-coalescer principle is applied in dispersions created by static-mixers, which offer a new possibility of incorporating the electric field in-line. Furthermore, we examine the influence of field strength, type of field (AC or DC), frequency of the electric field, temperature, and phase ratio on the entrainment reduction. For both systems, the drop sizes are measured to determine the extent of coalescence by the electric field.

MATERIALS AND METHODS

Materials
Toluene (purity > 99.9%) was supplied by Merck Germany. ë-caprolactam (purity > 99%), ethylbenzene (EB) (purity > 99%), and z-methyl benzyl alcohol (MBA) (purity > 99%) were supplied by Sigma-Aldrich (USA). MilliQ super distilled water was used for all the experiments.

Experimental Set-Up
The experimental set-up developed for the entrainment quantification and reduction is depicted in Fig. 2. It consists of two gear pumps for the organic and aqueous phases, variable-area flow-meters to measure the flow, a static mixer to mix the phases, an electro-coalescer in the static-mixer pipe to coalesce the drops produced by the static mixer, a settler for separation of the phases, a coalescer to enlarge the entrained droplets from the settler, and buffer tanks (secondary settlers). The static mixer is of the Kenics type and has an inner diameter of 14.6 mm and a length of 400 mm. The static mixer is placed in a glass tube of inner diameter 14.6 mm and length 500 mm. The length of the tube from mixer to settler is 100 mm and the electric field is placed at this position as shown in Fig. 2. The electric field consists of two electrodes connected to a voltage source. The electrodes are made of stainless steel and have a rectangular shape with dimensions of length 70 mm, width 10 mm, and a thickness 1 mm. The distance between the electrodes is 10 mm. The electrodes are insulated with a Kynar material, which has a thickness of 0.25 mm, and is resistant to toluene and ethylbenzene. In addition, it is non-wetting to water, and prevents short-circuiting. To determine the influence of the electric field on the drop size, pictures are also taken between the static mixer and settler.
using a canon 50D camera which are analyzed by manual drop counting in Adobe Photoshop CS5. The drop size measurement by this technique can lead to error due to the curvature of the pipe. A maximum error of 3% was found by measuring the size of known spheres placed in a static-mixer pipe at different locations from the center (29). The electrodes are connected to the voltage source that can provide a maximum voltage of 1 kV, and a square wave with a frequency between 3–100 Hz. The voltage and frequency are measured with a Fluke 175 multimeter. The settler has a volume of 0.009 m$^3$ (length = 0.3 m, width = 0.2 m, height = 0.15 m). The settler has glass windows at the front and back sides to enable drop size measurement and to have a visual observation of the separation in a settler. The interface level in the settler is maintained at 0.05 m. The settler inlet of 17 mm inner diameter is in the organic phase, at a height of 0.12 m. The coalescers are made of glass wool and Teflon fibers to enlarge the size of entrained water and toluene droplets, respectively, and to enable recycling of both phases. A visual check demonstrated that the recycled phases are non-hazy, and the coalescers operate adequately.

**Procedure**

Before starting the experiments for the caprolactam-toluene-water system, an equilibrated amount of caprolactam in toluene and MiliQ water was prepared to avoid mass transfer effects. A concentration of 28.51% (w/w) caprolactam in the aqueous phase was used. This concentration was chosen since it gives considerable entrainment from our earlier experiment without electric field (29). Corresponding to this concentration in the aqueous phase, the equilibrium caprolactam concentration in the organic phase and the physical properties are given in Table 1. Similarly, for the EB-MBA-water system, 33% (v/v) of MBA in EB was prepared and used as organic phase. This concentration was chosen for the same reason as for the caprolactam system. For this system, MiliQ water was used as an aqueous phase. The corresponding equilibrium solubility of water in the organic phase and the physical properties are given in Table 1. Once the organic and aqueous phases were prepared for each system, the organic and aqueous tanks, the settler, and the secondary settlers were filled with equilibrated organic and aqueous phases. The static mixer was primed with the organic phase to pre-wet the internals with the continuous phase. Once the other part of the system was filled with each phase, the organic pump flow was varied between 54 l/hr to 96 l/hr in 5 levels, and the aqueous flow was adjusted to 10% of the organic flow. To study the phase ratio effect, the aqueous flow was also adjusted to 5% and 20% of the organic flow. After the flow rate was set to the required value, the voltage source was switched on. For each flow, the experiment is run for three times the residence time in the settler to ensure equilibrium. The equilibrium was checked by measuring the water content of the continuous phase at the outlet of the settler at different residence times until no change in the water content was observed. For both systems, first the effect of the DC field strength of 1 kV/cm was evaluated at each flow. Subsequently, at the maximum flow, experiments were performed with a DC field of 0.4 kV/cm and a square wave of the same strength at different frequencies; 3, 5, 10, 25, 50, and 100 Hz. It was not possible to evaluate the influence of frequencies below 3 Hz and above 100 Hz with the source used.

**TABLE 1**

| Caprolactam-toluene-water system (19) | | | | | |
|---|---|---|---|---|
| Caprolactam concentration in the aqueous phase [wt%]/T (C) | Equilibrium caprolactam concentration in the organic phase/water solubility [wt%] | Interfacial tension [mN/m] | Density organic continuous phase [kg/m$^3$] | Viscosity organic continuous phase [mPa.s] |
| 28.51/20 | 3.39/0.16 | 7.3 | 877 | 0.635 |
| 28.51/40 | 4.96/0.28 | 5.3 | 857 | 0.513 |
| EB-MBA-Water system (21) | | | | | |
| MBA concentration in the organic phase [vol%]/T(C) | Equilibrium water solubility in the organic phase [wt%] | Interfacial tension [mN/m] | Density organic continuous phase [kg/m$^3$] | Viscosity organic continuous phase [mPa.s] |
| 33/20 | 0.93 | 11.7 | 914.8 | 1.195 |
| 33/30 | 0.98 | 11.5 | 906 | 1.088 |
| 33/40 | 1.02 | 11.4 | 897 | 0.919 |
Entrainment Quantification

For the systems used in this work, the aqueous phase was dispersed in the organic phase. The dispersed aqueous phase entrainment to the organic stream can then be determined by water content measurements. Under equilibrium conditions, some water is present in the organic phase due to the physical solubility. This was determined by circulating the two phases at a low flowrate (no droplet entrainment) until equilibrium was established. This was validated with separate equilibrium data from literature for the caprolactam system (19). Once this was determined, the droplet entrainment was measured by subtracting the solubility from the total water content. To determine the amount of entrainment, 30 mL of the organic stream was taken by opening the organic outlet valve from the settler. From this, 1 mL of organic sample was analysed six times for water content. The mass fraction of water in the organic phase was measured by a coulometric Karl Fischer titration with a 652 KF Coulometer (Applikon, The Netherlands) with an accuracy of ±0.005% (w/w).

RESULTS AND DISCUSSION

Coalescence with DC Field of 1kV/Cm

The influence of a 1 kV/cm DC field on the entrainment reduction for the caprolactam-toluene-water and MBA-EB-water systems at a temperature of 20°C and phase ratio of 10% (v/v) is presented in Figs. 3a and 3b, respectively. It can be observed that a considerable amount of entrainment is present especially at high shear rates (flowrates) for both systems when no electric field was applied. However, by applying an electric field most of this entrainment was removed. For instance, at the maximum flow rate (shear rate) used in the experiment, the entrainment decreased from 1373 ppm to 275 ppm for the caprolactam-toluene-water in the presence of an electric field, while it decreased from 2785 ppm to 628 ppm for the MBA-EB-water system. Additionally, for both systems, the entrainment tends to increase with an increase in flow rate (shear rate) in the presence and absence of the electric field. The figures reveal that without the electric field the entrainment increases steeply as the flow rate approaches the flooding limit. Nevertheless, this increase of entrainment was dampened.

![Figure 3](https://via.placeholder.com/150)

**FIG. 3.** Effect of 1 kV/cm DC electric field on entrainment reduction and mean sauter diameters for caprolactam-toluene-water (a,c) and for MBA-EB-water (b,d) systems as function of shear rate. Experimental conditions are: temperature 20°C and a phase ratio of 10% (v/v). The error bars are based on duplicate experiments performed for all shear rates. In some data points the error bars are not visible because they are small.
in the presence of the electric field. This indicates that the settler capacity has increased significantly in the presence of the electric field. The increase of settler capacity and decrease of entrainment is attributed to drop coalescence in the presence of the electric field, which increases the size of droplets fed to the settler. As a result, their settling velocity increases; hence, the droplets can be removed before they reach the end of the settler. The influence of the electric field on the droplet size for the two systems is depicted in Figs. 3c and 3d. For all the shear rates used, it can be seen that the drop sizes were considerably increased by applying the electric field, which consequently resulted in the observed intensified reduction of entrainment in both systems.

**Effect of Field Strength**

Generally, the efficiency of electro-coalescence can be increased by using higher field strength. This is because both the droplet-droplet electrical attraction force and drop mobility increase at high field strength, which enhances coalescence between the drops. However, increased electric field strength also means more power requirement, which increases the operating cost. In addition, it might lead to further breakup and dispersion of drops instead of coalescence, when the electrostatic force exceeds the interfacial restoring forces (18). Therefore, lower field strength was used to evaluate the separation efficiency at lower power consumption.

In Figs. 4a and 4b, the entrainment reduction achieved with DC fields of 0.4 kV/cm and 1 kV/cm is compared for the caprolactam-toluene-water and MBA-EB-water systems, at a temperature of 20°C, a phase ratio of 10% (v/v), and at the maximum shear rate. It can be observed that by using electric field strength of 0.4 kV/cm, the entrainment decreased from 1373 ppm to 425 ppm for the caprolactam-toluene-water system, and from 2785 ppm to 1025 ppm for the MBA-EB-water system. At field strength of 1 kV/cm, these entrainment values decreased further down to 280 ppm and 628 ppm for the caprolactam-toluene-water and MBA-EB-water systems, respectively. This is expected since a higher field strength results in enhancement of the electrostatic attraction force responsible for coalescence of drops. In addition, the higher field strength increases the magnitude of induced charges on the drops, which further increases the attraction force between the drops. Furthermore, it can be concluded from Figs. 4a and 4b, that while extra reduction of entrainment was obtained at a field strength of 1 kV/cm, the decrease flattened with the change of field strength from 0.4 kV/cm to 1 kV/cm. Other studies (7,30) also reported a similar dependence of separation efficiency on the field strength, and the saturation of separation efficiencies at high field strength. A possible explanation is that at low field strength, the induced dipole and droplet elongation cause increased separation. However, at higher field strength the performance flattens due to charge saturation and the possibility of breakup of elongated drops.

**Effect of Frequency**

According to past studies by Bailes (13,14), Drelich (8), and Eow (15), the influence of frequency on electro-coalescence results from two effects. First, once the electric field is applied, the build-up of interfacial charge on the drops will not occur instantaneously, but takes some time. There is a lag between the application of the electric field and built up of the charge. This depends on the relaxation time of the various dielectrics (the dispersion and insulation), electrical properties of the continuous phase and the electrode coating material, and its thickness. This overall time constant governs the electric field in the system and the build-up of interfacial charge. Additionally, the frequency dependence results from the way the applied voltage distributes across the insulation and dispersion with time (13,14,31). Initially, upon the application of the

![FIG. 4. Effect of DC electric field strength on entrainment reduction (a) for caprolactam-toluene-water, and (b) for MBA-EB-water. Experimental conditions are: temperature 20°C, phase ratio 10% (v/v), and at the maximum shear rate. The error bars are based on duplicate experiments performed for all field strengths. In some data points the error bars are not visible because they are small.](image-url)
electric field, the potential difference distributes itself based on the ratios of capacitances of the dispersion and the insulation. This means most of the applied potential (field) will initially distribute itself across the dispersion because the insulation has a higher capacitance. However, with time the ultimate distribution of the potential difference depends on the ratio of the resistances (14,31) and most of the potential difference distributes itself across the insulation. Therefore, the frequency should be high enough to maintain a high field in the dispersion, but at the same time should allow for the field and interfacial charge to fully develop. This determines the optimum frequency that should be used to obtain maximum coalescence.

The effect of electric field frequency on entrainment reduction for the caprolactam-toluene-water and MBA-EB-water systems is shown in Figs. 5a and 5b at a field strength of 0.4 kV/cm, a temperature of 20°C, a phase ratio of 10% (v/v), and at the maximum shear rate. The figures reveal similar dependence of entrainment reduction on the frequency of the electric field for both systems. It can be concluded that all frequencies of the AC field have lower entrainment than a DC field (frequency of 0 Hz). This is expected since using the DC field results in a situation where most of the field will be across the insulation for a significant period of the cycle (9). Additionally, frequencies lower than 10 Hz were found to be more effective in the reduction of entrainment compared to higher frequencies. For the caprolactam-toluene-water system the lowest entrainment was observed at 3 Hz and the entrainment was found to increase gradually as the frequency was increased. Similarly, for the MBA-EB-water system, lower entrainment values were found at frequencies less than 10 Hz. The entrainment, however, increased sharply for frequencies greater than 10 Hz. This can be explained by the fact that less time is available for the charge to fully develop when the frequency of the AC field is increased to higher values. As a result less charge will be induced on the drops, which decreases the attraction force responsible for coalescence. Therefore, the electro-coalescence efficiency decreases and results in an increase of entrainment at higher frequencies as depicted in Figs. 5a and 5b.

**Effect of Phase Ratio**

The effect of the phase ratio on entrainment reduction by electric field results from two opposing trends associated with the increase of holdup at high phase ratios. On the one hand, the distance between drops in the dispersion decreases as the phase ratio increases due to the increased number of droplets at high phase ratio. This increases the dipole-dipole attraction force, since this force is inversely proportional to the fourth power of the inter-droplet distance (12):

$$F = \frac{24\pi\varepsilon_o\varepsilon_1 r^6 E^2}{(d + 2r)^4}$$

where $\varepsilon_o$ is the permittivity of the vacuum, $\varepsilon_1$ the dielectric constant of the continuous phase, $d$ the distance between the near surfaces of the two drops, $E$ the electric field strength, and $r$ the drop radius.

The electro-coalescence efficiency is expected to increase dramatically at increased phase ratio considering only this effect. On the other hand, a high phase ratio will also result in dispersion with a large water holdup, which is described by Sun et al. (16,32) as creating a “sponge phase” containing an abundant water phase at the interface of the water and continuous organic phase. This considerably decreases the electro-coalescence efficiency by creating a conductive path. Therefore, it can be expected that the electro-coalescence efficiency increases with increased phase ratio up to a certain value (30) due to increased attraction force resulting from a reduced distance between the drops. However, a further increase of the phase ratio will reduce the efficiency again as a result of the conduction effect induced by the large number of droplets.

![FIG. 5. Effect of AC field frequency on entrainment reduction (a) for caprolactam-toluene-water (b) for MBA-EB-water. Experimental conditions are: field strength 0.4 kV/cm, temperature 20°C, phase ratio 10% (v/v), and at the maximum shear rate. The error bars are based on experiments performed in duplicate.](image-url)
The influence of the phase ratio on entrainment reduction by an electric field for the caprolactam-toluene-water system and MBA-EB-water is presented in Figs. 6a and 6b, at a field strength of 0.4 kV/cm and a temperature of 20°C. As can be observed for both systems, the entrainment values increase at high phase ratios. This can be explained by the relative increase of the dispersed phase flow (more drops into electro-coalescer) at higher phase ratios. Nevertheless, this is not equivalent to the electro-coalescence efficiency, since the entrainment without the application of an electric field is different for the different phase ratios. The efficiency of the electric field is determined by comparing the entrainment reduction obtained to that of the entrainment without applying an electric field (7,8,14), as presented in Eq. (2).

The entrainment values without the electric field for the different phase ratios are given in Table 2 for the two systems.

\[
\text{Separation efficiency} \% = \left( \frac{\text{Entrainment without electric field} - \text{Entrainment with electric field}}{\text{Entrainment without electric field}} \right) \times 100
\]  

(2)

Figures 6c and 6d depict the entrainment removal efficiencies calculated using Eq. (2) and Table 2 for the two systems at a field strength of 0.4 kV/cm, and a temperature of 20°C. For the caprolactam-toluene-water system, the electro-coalescer efficiency increased first as the phase ratio is changed from 5% (v/v) to 10% (v/v) phase ratio. However, the efficiency decreased again at a phase ratio of 20% (v/v). As described before, the increase of efficiency from 5% (v/v) to 10% (v/v) phase ratio results from the reduction of the inter-droplet distance between the drops, which increases the electro-coalescence attraction force.

**TABLE 2**

<table>
<thead>
<tr>
<th>Phase ratio [v/v %]</th>
<th>Entrainment [ppm]</th>
<th>Phase ratio [v/v %]</th>
<th>Entrainment [ppm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>280</td>
<td>5</td>
<td>1530</td>
</tr>
<tr>
<td>10</td>
<td>1373</td>
<td>10</td>
<td>2785</td>
</tr>
<tr>
<td>20</td>
<td>1258</td>
<td>20</td>
<td>4678</td>
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</tbody>
</table>

FIG. 6. Effect of AC field frequency and phase ratio on entrainment reduction and separation efficiency (a,c) for caprolactam-toluene-water (b,d) for MBA-EB- systems. Experimental conditions are: field strength 0.4 kV/cm, temperature 20°C, and at the maximum shear rate.
The decrease of efficiency at the phase ratio of 20% (v/v) can be attributed to the conductive loss created by the presence of abundant water phase. In contrast, for the MBA-EB-water system, the efficiency did not change much when a phase ratio was changed from 5% (v/v) to 10% (v/v). However, at a phase ratio of 20% (v/v), the efficiency of the separation declined sharply, particularly, when using a DC field and higher frequencies of 50 Hz and 100 Hz. This indicates that for the MBA-EB-water system, the decrease of coalescence efficiency due to conduction effects occurs already at a lower phase ratio of 10% (v/v). This can be expected since in the MBA-EB-water system, the dispersed phase consists only of water in contrast to the former system, where the dispersed phase also contains caprolactam. This results in a higher conductivity of the dispersed phase for the MBA-EB-water system, which decreases the coalescence efficiency. Additionally, it can be observed from Fig. 6 that for all phase ratios the AC field is more efficient in entrainment reduction compared to the DC field (frequency of 0 Hz).

Effect of Temperature
The influence of temperature on entrainment reduction by the electric field depends on two factors;

1. whether the drop size increases or decreases with temperature, and
2. the decrease in the viscosity of the continuous phase, which increases the separation rate in the settler.

The first factor has two effects. First, the drop size created by the static-mixer governs the effectiveness of the electric field by influencing different electrical forces responsible for electro-coalescence. The electrophoretic, dielectrophoretic, and dipole-dipole attraction forces are proportional to the drop size to the power 2, 3, and 6, respectively (12). And second, the drop size affects the settling velocity according to Stokes’ law. Thus, as the drop size created by the static-mixer decreases, the electric field effectiveness decreases, and the accompanied decrease of settling velocity leads to more entrainment.

In Fig. 7a, the influence of temperature on entrainment reduction by the electric field is shown for the caprolactam-toluene-water system at a field strength of 0.4 kV/cm, a phase ratio of 10% (v/v), and at the maximum shear rate. As can be observed, higher entrainment values were obtained at 30°C and 40°C compared to 20°C. This increase of entrainment at high temperatures was explained by measuring the drop sizes. As observed from the experimentally measured drop sizes depicted in Fig. 7c, the mean sauter diameters were found to decrease with an increase in temperature. This is due to the decrease in interfacial tension and viscosity ratio of the dispersed to continuous phase viscosity ratio (μd/μc). The decrease in interfacial tension and viscosity ratio of the dispersed to continuous phase viscosity ratio (μd/μc) decreases the interfacial layer, which in turn decreases the interfacial tension and viscosity ratio of the dispersed to continuous phase viscosity ratio (μd/μc).

![Figure 7](image_url)

**FIG. 7.** Effect of AC field frequency and temperature on entrainment reduction and mean sauter diameters for caprolactam-toluene-water (a,c) and for MBA-EB-water (b,d) systems. Experimental conditions are: field strength 0.4 kV/cm, phase ratio 10% (v/v), and at the maximum shear rate. The error bars are based on duplicate experiments performed for all temperatures. In some data points the error bars are not visible because they are small.
tension facilitates drop breakup. Additionally, as shown by Grace (33), the decrease in viscosity ratio further increases the drop breakup when the viscosity ratio is larger than 1. Thus, the smaller drop sizes created by static-mixer for the caprolactam-toluene-water system at higher temperature, and the accompanied decrease of electrical forces responsible for electro-coalescence explain the increase of entrainment at high temperature.

In Fig. 7b, the influence of temperature on entrainment reduction is presented for the MBA-EB-water system at a field strength of 0.4 kV/cm, a phase ratio of 10% (v/v), and at the maximum shear rate. In contrast to the caprolactam system, it can be observed that lower entrainment values were obtained at higher temperatures. This is a result of the way the drop size changes with temperature for the two systems. In contrast to the caprolactam system, it can be seen from Fig. 7d that the mean sauter diameters increase with an increase of temperature for the MBA-EB-water system. As can be seen from Table 1, for MBA-EB-water system, the change of interfacial tension is not significant, while the viscosities considerably decrease at temperatures of 30°C and 40°C. The influence of temperature on drop size thus comes from its effect on the viscosities. The increase of temperature decreases the viscosity ratio and according to Grace (33) favors coalescence since the viscosity ratio is less than 1. Hence, the drop sizes increase at higher temperatures resulting in the decrease of entrainment for the MBA-EB-water system. In addition, it can be concluded from Fig. 7 that the influence of frequency on the entrainment remained unchanged at higher temperatures. The DC field was found to be less efficient than the AC field. Moreover, the efficiency of the electric field was found to decrease at higher frequencies.

CONCLUSIONS

In this work, an electric field was successfully integrated in a static-mixer to intensify entrainment reduction for the caprolactam-toluene-water and MBA-EB-water systems. By applying a DC field of 1 kV/cm, the entrainment was considerably reduced from 1373 ppm to 275 ppm for the caprolactam-toluene-water, and from 2785 ppm to 628 ppm for the MBA-EB-water system. The AC field was more efficient in entrainment reduction compared to the DC field since similar entrainment reduction efficiency could be obtained at field strength of 0.4 kV/cm. The entrainment reduction was found to be frequency dependent with lower frequencies of 3–10 Hz being the optimum for both systems. Moreover, the entrainment reduction was influenced by phase ratio and temperature. Higher temperatures led to less entrainment for the MBA-EB-water system, while more entrainment was found for the caprolactam-toluene-water system. This was explained by the way the drop sizes changed with temperature for the two systems. The effect of the phase ratio on entrainment reduction was expressed in terms of efficiency to account for the change in the dispersed phase flow and entrainment with the phase ratio. For the caprolactam system, the entrainment reduction efficiency increased up to a phase ratio of 10% (v/v) and decreased afterward at phase ratio of 20% (v/v). For the MBA-EB-water system, the efficiency did not change much between a phase ratio of 5% (v/v) and 10% (v/v); however, in a similar way as the caprolactam system, the efficiency decreased again at a phase ratio of 20% (v/v).

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REFERENCES

2. Gergensen, G.V. (1999) Copper Leaching, Solvent Extraction, and Electrowinning Technology; Wiley: NJ.