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Continuous flow demulsification of a water-in-toluene emulsion by an alternating electric field

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ABSTRACT

In this study, the in-flow demulsification of a water-in-toluene emulsion by an alternating electric field was investigated. A fluorocarbon tube, chosen for its flexibility and resistance to toluene, was positioned between copper electrodes attached to a voltage supply and a wave generator. With this design, it was possible to apply a strong electric field of 1000 kV/m as the emulsion was not in direct contact with the electrodes. The demulsification rate increased on increasing the frequency of the alternating electric field up to ~100 Hz, and a square wave was more effective than a sine wave at low frequencies. Furthermore, the demulsification rate increased on increasing the content of the organic phase and intensity of the electric field. We investigated the effects of an electrolyte dissolved in the aqueous phase, and found that it promoted demulsification in relation to the valence of the cation used. Additionally, demulsification was strongly affected by the ionic strength of the aqueous phase. Our results show that demulsification by the application of an alternating electric field can be very useful in the continuous flow treatment of extraction mixtures in organic synthesis.

1. Introduction

In recent years, the intentional production of emulsions for use as industrial chemicals, cosmetics, food additives, and medicinal materials has become widespread. However, emulsions are often generated as unwanted byproducts from the treatment of oil and aqueous solutions. For example, after conducting an organic reaction in toluene, widely used as a reaction medium for organic synthesis, liquid–liquid extraction may be carried out to separate and recover the product. Emulsions often occur on mixing of the organic and aqueous phases. Once an emulsion forms in the reaction vessel, the separation of the organic and aqueous phases becomes very difficult and time consuming, and requires bulky equipment. Consequently, the development of a rapid, convenient, and continuous method for demulsification would be of enormous benefit to the organic chemist.

Several methods have been developed for demulsification, such as heating [1,2], centrifugation [3,4], chemical treatment [5–7], and electric field treatment [8–16]. The advantages of electrical separation are (i) no additional material is added, eliminating the need for further treatment before or after the process, (ii) the demulsification can be carried out at room temperature, and (iii) the demulsification device is structurally and operationally simple. However, it also has several disadvantages, which include the need for a high electric voltage and the possible occurrence of electrochemical reactions when the electrode is in direct contact with the emulsion. If these deficiencies could be overcome, the electric field method could potentially show good demulsification performance.

In this study, we examined the demulsification of a water-in-toluene emulsion by the application of an alternating current (AC) electric field. A fluorocarbon tube of 1 mm diameter was used for emulsion flow. A strong electric field of 1000 kV/m could be applied as the tube prevented the emulsion from direct contact with the electrodes.

2. Experimental

2.1. Experimental setup

The experimental setup used in this study is shown in Fig. 1. The apparatus comprises five devices, i.e., a plunger pump, the demulsification device, a measuring cylinder, a voltage supply, and a function generator. A fluorocarbon tube through which the

Abbreviations: AC, alternating current; DC, direct current; η, demulsification rate; V_{oil}, volume of the separated toluene phase; V_0, original volume of a respective phase.

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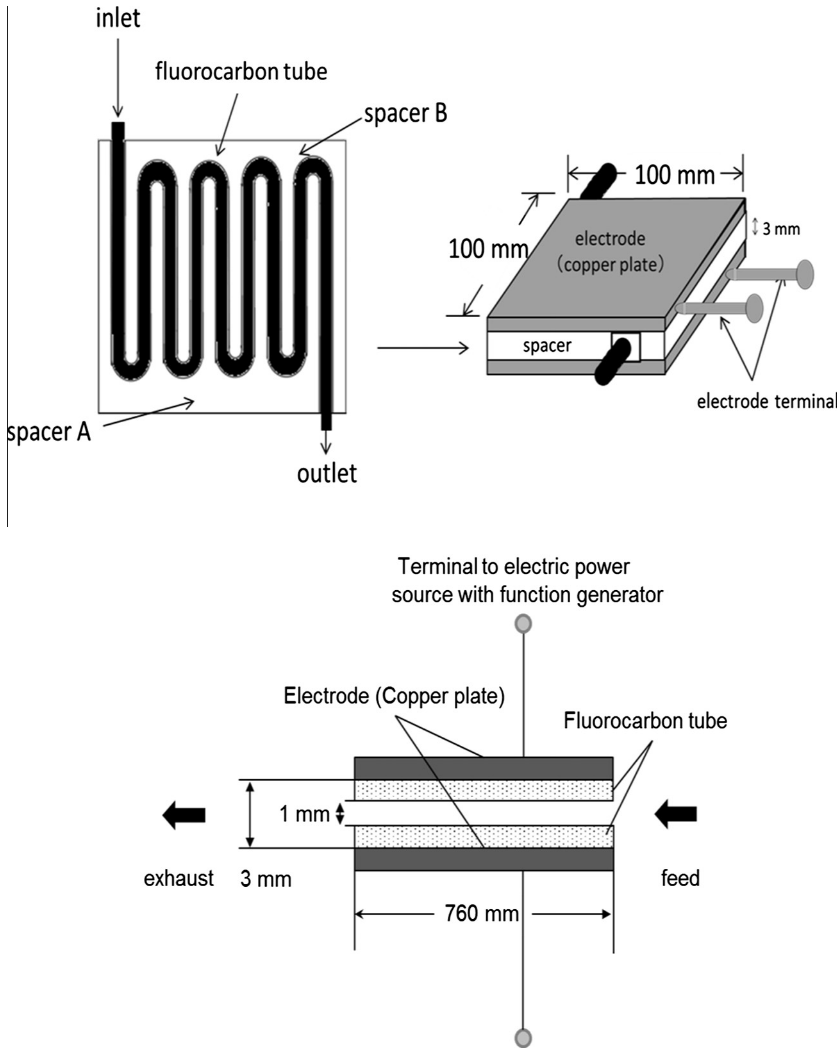

Fig. 1. Experimental design of the demulsification device.

emulsion flows is inserted between two electrodes, which apply the electric field. Two copper plates (100 mm × 100 mm × 5 mm) are used as electrodes and connected to a voltage supply (HEOP-3B10, Matsusada Precision Inc., Kusatsu, Japan) and a wave generator (WF1974, NF Corporation), which controls the waveform and frequency of the alternating electric field. A fluorocarbon tube (ARAM Corporation, Japan, Code No. 1024-01) of internal diameter 1 mm, external diameter 3 mm, and length 760 mm is positioned between the two copper electrode plates. The emulsion is fed into the fluorocarbon tube by a dual plunger pump (YMCK-11-13-P, YMC Co., Ltd.). The flow rate, and therefore, retention time of the device, is chosen to correspond to the duration of the applied electric field. The output from the equipment is collected in a measuring cylinder, in which the volumes of the oil, emulsion, and aqueous phases can be measured.

2.2. Experimental procedure

Span 80 (sorbitan monooleate) was dissolved in toluene (4 vol %) as a stabilizer. Known amounts of toluene and water were stirred at 24,000 rpm for 1 min by a homogenizer (IKA, T18BS1) to prepare a stable emulsion. From preliminary experiments, we found that a reaction time of 185 s (20 mL/h) was sufficient to demulsify a test mixture. Consequently, the emulsion was fed into the demulsification device at constant flow rates of 20, 60, 100, 150, and 200 mL/h with a syringe pump. The volumes of the liquid output and the oil phase generated by demulsification were directly measured in the measuring cylinder. The demulsification experiment was conducted with a 1000 kV/m electric field oscillating as a square wave at a frequency of 100 Hz. The alternating field was applied to a 50 vol% suspension of water in toluene for 62 s. The demulsification experiments were conducted under these conditions unless otherwise specified.

The demulsification rate (η) was defined as

$$\eta = \frac{V_{oil}}{V_{0,oil} + V_{0,water}} \quad (1)$$

where V_{oil} is the volume of the separated toluene phase, and $V_{0,oil}$ and $V_{0,water}$ are the original volumes of toluene and water, respectively, before mixing. The water content of the oil phase was analyzed by Karl Fischer titration (KF-21 moisture meter, Mitsubishi Chemical Analytec).

3. Results and discussion

3.1. Observations of water-in-toluene emulsions

When the prepared emulsions were stored at room temperature, no phase separation was observed over the course of a week,