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LIQUID-LIQUID EXTRACTION OF ACETIC ACID FROM AN AQUEOUS SOLUTION USING A LABORATORY SCALE SONICATOR

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ABSTRACT

Extraction of acetic acid from an aqueous solution was carried out in a laboratory scale sonicator using ethyl acetate as a solvent. The results were compared with and without the effect of sonication. A significant separation of acetic acid was found as a result of sonication. The effect of mixing time, feed composition, and solvent to feed ratio was investigated. The effect of time required for extraction using varying feed concentrations was found to be same. Increasing solvent to feed ratio improved the performance of the extractor system. It was observed that the extraction was improved by a factor of two with an eight times increase of solvent to feed ratio (vol/vol).

Keywords: *Acetic-acid, Ethyl-acetate, Liquid-liquid extraction, Sonication and Ultrasonic bath*

1) INTRODUCTION

Acetic acid is widely used as a solvent and participates in important chemical reactions (Saha et al., 2000). Aqueous acetic acid is produced during the course of various chemical reactions and it is important to recover from both an environmental and economic perspective. Moreover, dilute acetic acid produced by microorganisms in a fermentation broth requires separation from the aqueous phase. However, a binary mixture of water and acetic acid has relative volatility close to unity and normal distillation requires a large number of plates and high reflux ratio to separate the two components effectively (Kurum et al., 1995). Several techniques depending on economics are available for

the recovery of acetic acid from aqueous phase. Extractive distillation (Demiral & Yildirim, 2003), reactive distillation [Saha et al., 2000; Xu et al., 1999; Singh et al., 2006), and liquid-liquid extraction (Usman et al., 2006; Golob et al., 1981; Yang et al., 1991) are common ways used in separation processes. Extraction of acetic acid has been studied by a number of researchers in various kinds of liquid-liquid extractors. The technological use of sound waves has significant effect on both chemical and physical operations. It has been exploited for degasification of solvents and inks. It has been extensively used for the extraction of biological materials (Sargenti & Vichnewski, 2000; Retamal et al., 1999) such as proteins and other important leaching systems (Purcaro et al., 2009; Brilis & Marsden, 1990). In the present study, a new extractor system has been devised and studied for the liquid-liquid extraction of acetic acid from an aqueous phase using ethyl acetate as a solvent. A laboratory scale sonicator (ultrasonic bath) was employed to study the effect of various operating conditions for the separation of acetic acid from its aqueous solution. To the best knowledge of authors, the use of sonication for the extraction of acetic acid from aqueous phase using liquid-liquid extraction has never been studied.

2) MATERIALS AND METHODS

A laboratory scale batch type sonicator was used at a frequency of 60 Hz (FS 100 B, Decon Laboratories, Sussex, UK). Ethyl acetate and glacial acetic acid (99 % analytical grade, supplied by Fisher Scientific, UK) were used. All solutions were made using de-ionized water. Experiments were performed under various operating conditions and the effect of time, feed composition and the solvent to feed ratio was studied. In a typical procedure, the quantities of aqueous acid solution and solvent were measured out into a 250 ml Schott bottle, here, called an extractor vessel. The extractor vessel was placed in the water-filled sonicator bath. The sonicator bath was filled with 2000 ml tap water which ensured a stable and upright position of the extractor vessel. The sonicator was set for a specified time and turned on to start the experiment. After the required time, the extractor vessel was taken out of the sonicator and the two phases were allowed to settle. A settling time of 5 min was allowed for the separation of two phases. 2.0 ml of aqueous phase was drawn from the extractor vessel and titrated against N/10 NaOH solution. After taking a sample, the extractor vessel was placed back into the sonicator and the procedure was repeated.

3) RESULTS AND DISCUSSION

Fig. 1 shows the effect of time and compares the acetic acid extraction with and without sonication effect. Both extractions reached to and equilibrium; however, the effect of sonication is quite evident in figure 1 showing efficient separation efficiency of acetic acid from aqueous solution.

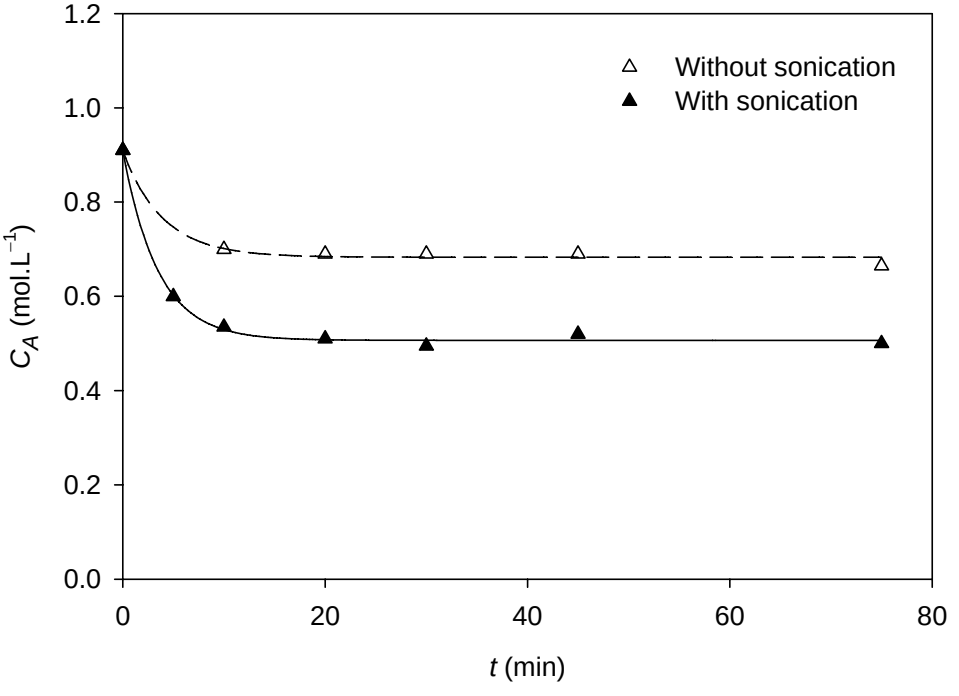

Fig.1: Comparison of acetic acid extraction from aqueous solution with and without sonication using ethyl acetate as the solvent. S/L = 1.0, where S and L are the volumes of the solvent and the feed respectively, t is operating time in min and C_A is concentration of acetic acid at any time t in mol L¬¹.

It is therefore concluded that sonication improved the extraction performance of acetic acid. As an example, after 30 min, the percent drop in concentration, as defined in Eq. 1, is 24.2% in the case of without sonication, while the percent drop in concentration under same conditions with sonication is 45.6% which is twice that obtained without sonication.

$$\text{Percent drop in concentration} = \frac{C_{A0} - C_A}{C_{A0}} \times 100 \quad (1)$$

Where, C_{A0} (mol·L^{-1}) is the initial concentration of acetic acid in aqueous solution at t = 0 min, while C_A (mol·L^{-1}) is the concentration of acetic acid in aqueous solution at any time t (min). The results encouraged further investigations to observe the behavior of the sonicator's effect on the extraction of acetic acid from aqueous solution as shown in Fig. 2 and Fig. 3.

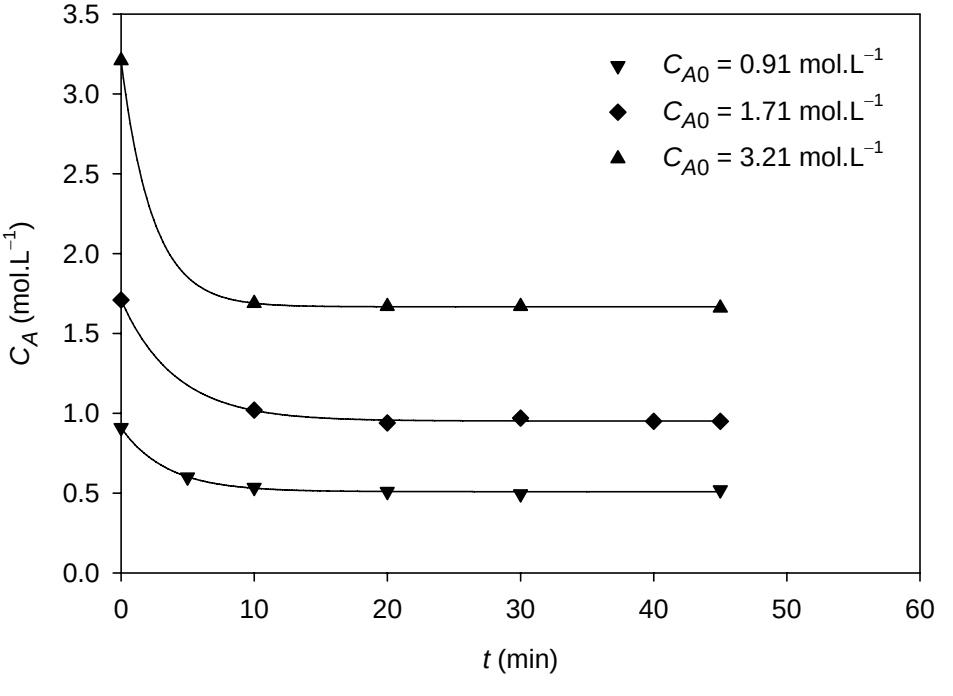

Fig. 2 Effect of feed composition on acetic acid extraction from aqueous solution using ethyl acetate as solvent. S/L = 1.0, where S and L are the volumes of the solvent and the feed respectively. t is operating time in min, C_A is concentration of acetic acid at any time t in mol·L^{-1}, and C_{A0} is initial concentration at time t = 0 min.

Fig. 2 shows the effect of initial feed concentration on the extraction of acetic acid. Three different molar feed concentrations of acetic acid; 0.91 mol·L^{-1}, 1.69 mol·L^{-1}, and 3.21 mol·L^{-1} were employed. In all the three cases, it requires less than 15 min to reach equilibrium. Moreover in each case, the time required to reach equilibrium is virtually similar.

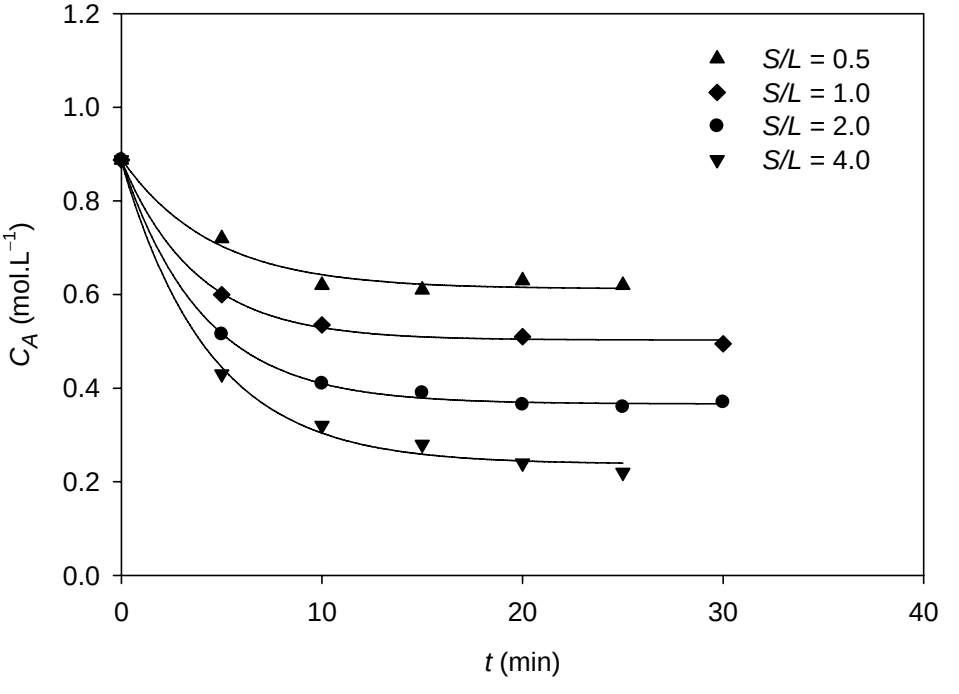

Fig. 3: Effect of solvent to feed ratio on acetic acid extraction from aqueous solution using ethyl acetate as solvent. S and L are the volumes of the solvent and the feed respectively. t is operating time in min, C_A is concentration of acetic acid at any time t in mol·L^{-1}, and C_{A0} is initial concentration at time t = 0 min.

Fig. 3 shows the effect of solvent to feed ratio on the extraction of acetic acid for similar initial concentration of acetic acid. As expected, increasing the solvent to feed ratio has a profound effect on extraction performance and increasing solvent to feed ratio increases the extraction of acetic acid. As an example, after 15 min of operation, the initial concentration dropped to 0.61 mol·L^{-1} for lowest solvent to feed ratio, while it dropped to 0.28 mol·L^{-1} for the highest ratio (8 times the lowest ratio expressed in vol/vol), which is equivalent to more than twice the percent drop in initial concentration as calculated by Eq. 1. It is observed that after about 25 min of operation, the system has reached steady-state equilibrium value except for the highest solvent to liquid ratio, which requires further extraction and more time to reach at equilibrium.

4) CONCLUSIONS

A sonicator (ultrasonic bath) has been investigated for the extraction of acetic acid from aqueous solution using ethyl acetate as the solvent. Comparing extraction efficiency obtained with and without sonication clearly demonstrated the effective use of sonication technique. The time required for the extraction to reach equilibrium for varying feed concentrations was found to be virtually identical. An eight time increase of solvent to feed ratio (vol/vol) produces more than twice the extraction of acetic acid for similar time of operation.

NOMENCLATURE

C_A concentration of acetic acid in aqueous solution at any time, mol m^{-3}
C_{A0} initial concentration of acetic acid in aqueous solution, mol m^{-3}
L volume of the feed, m^3
S volume of the solvent (ethylacetate), m^3
t time, s

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