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Reactive Extraction of Acetic Acid from Aqueous Sodium Acetate Waste

Ganesh Bhoj[1], Sunita Patil[2], Sharada Gadale[3] and Shambala Gadekar-Shinde[1]†

[1]Department of Chemical Engineering, Bharati Vidyapeeth (Deemed to be University) College of Engineering, Pune-411043, Maharashtra, India

[2]Department of Chemical Engineering, School of Engineering Management and Research, D Y Patil International University, Akurdi, Pune -411035, India

[3]Yashwantrao Mohite College of Arts, Science and Commerce, Bharati Vidyapeeth (Deemed to be University), Pune, India

†Corresponding author: Shambala Gadekar-Shinde; shamlashinde11@gmail.com

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ABSTRACT

The recovery of acetic acid from aqueous sodium acetate using traditional methods is costly and energy-intensive. The current study focuses on exploring a synergetic reactive extraction methodology to extract acetic acid from aqueous sodium acetate waste. Physical extraction experiments demonstrated that methyl isobutyl ketone (MIBK) and xylene are effective diluents among MIBK, xylene, octanol, and toluene. The extraction efficiency was further enhanced by adding Aliquat 336 as an extractant and MIBK and xylene diluents in independent runs. Parameters such as the initial acid concentration in the aqueous phase, Aliquat 336 concentration in the organic phase, and temperature of the reaction mixture were investigated to optimize the operating conditions. Under all conditions, MIBK yielded better results than the other solvents. For a high acid concentration in the aqueous phase (0.5 moL.L^{-1}), a 60% extraction efficiency was observed in the physical extraction experiment. The addition of Aliquat 336 as an extractant (0.729 moL.L^{-1}) to the mixture under identical experimental conditions resulted in a 73% extraction efficiency. Average extraction improved by 10% for 0.2-0.5 moL.L^{-1} of initial acid concentration using reactive extraction technology. Such a recovery from aqueous sodium acetate using reactive extraction has rarely been reported, and hence, it is presented in this paper.

INTRODUCTION

The recovery of chemicals from waste streams is gaining importance owing to economic and environmental concerns. The effluent of the process industry comprises salts, acids, alcohols, pesticides, dyes, and chemicals that cause severe pollution to freshwater streams. The development of advanced separation technologies for wastewater treatment is imperative to minimize environmental impact and to maximize resource recovery. In recent decades, intensive work has been conducted on the recovery of valuable chemicals such as carboxylic acids, alcohols, esters, and metals from wastewater solutions (Reyhanitash et al. 2017, El-Nadi 2017, Kong et al. 2022, Petersen et al. 2018, Wang & Ren 2014). These chemicals are harmful to aquatic flora and fauna, and their removal from water sources will be crucial in the coming years.

Sodium acetate is an important chemical entity produced as waste from many pharmaceutical, insecticide, pesticide, metal, fermentation, and dye manufacturing industries. This waste is either dumped on land, incinerated, or released into water bodies, causing pollution. The release of sodium acetate in freshwater streams causes acetate to hydrolyze and form acetic acid, thereby affecting the pH of the local water bodies over the course of time. The removal of this acetate at the source is necessary to maintain the ecological environment. The quantum of acetate loss in waste streams is significant, and its recovery in one or the other form is essential to minimize pollution and utilize the recycled resources. The literature mentions a

few articles on the recovery/removal of sodium acetate waste using technologies such as electro dialysis, crystallization, membrane separation, reverse osmosis, and ozone oxidation. Sodium acetate is one of the major ingredients in waste streams generated during the synthesis of diallyl alcohol ketone acetate. It is further separated and recycled back using a sequence of unit operations involving precipitation, filtration, washing, crystallization, and drying. This method reduces energy consumption and is claimed to have zero emissions or discharges (Liu et al. 2013). Similarly, the recovery of sodium acetate (76.6 wt%) from the dithianon manufacturing industry was studied using bipolar membrane electrodialysis (Xue et al. 2015). These experiments were conducted under different current densities and membrane stacks to optimize the parameters and maintain the purity of the acid formed. This method is energy-consuming and only recovers the dissociated acid ions from the slurry. Later, the coupling of diffusion and electrodialysis with a bipolar membrane was investigated to achieve pure acetic acid and NaOH. This coupling reduces the energy demand compared to that of bipolar membrane electrodialysis alone (Xue et al. 2018). Modeling studies on the electrodialysis method for acetic acid separation from sodium acetate slurry help optimize parameters for maintaining the purity of the separated acid (Fidaleo & Moresi 2005). Sodium salts of organic acids were recovered from the fermentation broth by the discontinuous evaporation crystallization method without the need for further purification. Sodium acetate is produced as a byproduct in the fermentation broth and is wisely utilized for intermediate crystallization to obtain other salts and reduce the need for further purification (Plate & van Esch 2018). This method is claimed to reduce the operating costs of the upstream processes of the fermentation broth. However, the efficiency of the process is not clear, as it depends on the solubility of the salt. A recent study addressed the removal of sodium acetate and sodium chloride salts from wastewater in pigment-producing industries using nanofiltration membranes and reverse osmosis technology (Chu et al. 2020, Gubari et al., 2023). Advanced technologies, such as ozone oxidation, have also been investigated for the maximum degradation of sodium acetate wastewater (Yang et al. 2012, Yang &Yuan 2014).

Among all the reported technologies, attempts have been made to recover sodium acetate from waste or recycle it in the plant. All these reported methods are energy- and cost-intensive; thus, there is an urgent need to explore alternative, less energy-consuming technologies for processing effluents containing sodium acetate waste for acid recovery. Since, sodium acetate is readily soluble in water and dissociates to form acetic acid ions, extraction is suggested as one of the least energy-consuming and cheapest methods available compared to the aforementioned technologies. These limited studies have barely reported the application of reactive extraction technology for the removal of acetic acid from aqueous sodium acetate waste. A single study on the acetate waste of chloromycetin production for the removal of sodium acetate, acetic acid, and sodium chloride mixture using Aliquat 336, Alamine, tributyl phosphate, and tri-octyl phosphine oxide as extractants and xylene as a diluent was conducted to achieve better extraction efficiency (Juang &Wu 1999). In this study, we attempted to recover acetic acid from aqueous sodium acetate waste.

Reactive extraction is a mature technology for the recovery of commodity chemicals; however, the recovery of acids from aqueous sodium acetate waste using the reactive extraction method has not been extensively studied. Although carboxylic acid extraction from waste streams is not new and has been extensively studied in the literature (Djas & Henczka 2018, Uslu & Kirbaslar 2013, Asci & Lalikoglu 2021, Erdas & Marti 2024, Kumar et al. 2021), the reactive extraction of aqueous sodium acetate requires a more in-depth analysis. The recovery of acetic acid from wastewater has been investigated using different extractants, such as trioctylamine (Cascaval et al. 2011, Mungma et al. 2019). triisooctyl amine (Yang et al. 2013), tributyl phosphate (Shakya et al. 2022), trioctylphosphine oxide (Abdulrahman et al. 2019), Alamine 336 (Marti 2016), and Aliquat 336 (Chakraborty et al. 2023). Tertiary and quaternary amines can extract carboxylic acids at both acidic and basic pH values; hence, they are preferred in many examples (Kar et al. 2017, Hussein and Shinde 2012, Gadekar-Shinde et al. 2023). Diluents, such as alcohol, hydrocarbons, ketones, and green solvents, such as oils, are mixed with extractants to alter their properties and improve efficiency. In acetic acid extraction, diluents such as hexane, cyclohexane, toluene, MIBK, propanol, benzyl alcohol, chloroform, xylene, octanol, 1-decanol, cyclopentyl methyl ether, n-undecane, 2-methyltetrahydrofuran and natural oils are investigated in different acetic acid extraction studies (Shakya et al. 2022, Inci 2002, Senol 2004, Marti 2016, Turk et al. 2020, Mungma et al. 2019). Comparing diluents benzyl alcohol, chloroform, MIBK, 1,2-dichloroethane, xylene and 1-octanol, the chemical interaction and solvation ability of dichloroethane and MIBK with amine extractant is found to be maximum for acetic acid removal (Senol 2004). Liquid-liquid extraction of acetic acid, water, and organic solvents such as methyl benzene, heptanol, methyl propyl ethanoate, 2-ethyl hexanol, ethyl acetate, cyclopentyl methy ether, cyclohexane, isobutanol, MIBK, m-xylene, o-xylene, sec-butyl acetate, and toluene was investigated. Among all these solvents, toluene exhibited higher selectivity for acid extraction (Mohadesi & Rezaei

2020). The recovery of acetic acid in industrial processes is of prime importance, and less energy-consuming technologies, such as solvent extraction, have been extensively studied for this purpose (Karunanithi et al. 2023). In summary, acetic acid was extracted using a combination of the extractant-diluent mixture to change the solvation properties of the organic layer and maximize the acid content in the organic layer.

The proposed work highlights the removal of acetic acid formed due to the hydrolysis of sodium acetate and water from the aqueous sodium acetate layer using reactive extraction technology. The diluents selected to explore the extraction ability and compatibility with the extractant were polar and nonpolar. The solvents MIBK, toluene, xylene and 1-octanol were selected to assess their extraction ability and to enhance the properties of the extractant. The diluents MIBK and toluene demonstrated superior efficiency compared to the best-performing solvents, that is, chloroform, octanol, and ethylacetate, in independent studies (Senol 2004, Mohadesi & Rezaei 2020). Hence, MIBK and toluene were selected to check their compatibility with this reaction. In an earlier study (Juang & Wu 1999), xylene was used as a diluent in combination with the extractants Aliquat 336, tri-octyl amine, tri-butyl phosphate, and tri-octyl phosphine oxide, and the efficiency of Aliquat 336 was found to be superior. Hence, Aliquat 336 and xylene were selected for comparison with other diluents to test their extraction ability in this study. In addition, Aliquat 336 has the potential to extract both dissociated and undissociated acid ions from the aqueous phase, thus making it the most suitable amine extractant (Wang et al. 2019). Although many solvents, such as ethyl acetate and chloroform, have high distribution ratios, their reactivity with NaOH and toxicity to aquatic life are the major parameters of concern. The proposed work aims to extract acetic acid from sodium acetate waste. If ethyl acetate is considered, it may enter the aqueous phase and react at temperatures above of room temperature. Moreover, isolating acetate/acetic acid ions with the help of acetate ions would become more tedious in such cases. The hydrolysis of chloroform under basic conditions to produce carbanions has been reported (Stamou et al. 2024). Because we have sodium acetate in our aqueous phase, the reactivity and affinity of chloroform with sodium hydroxide in the aqueous phase cannot be avoided. Hence, this solvent was also avoided in the study. Also, chloroform is more toxic in nature as compared to MIBK and may harm aquatic life. To the best of our knowledge, reactive extraction of aqueous sodium acetate for recovering acetic acid using Aliquat 336 and diluents MIBK, toluene, xylene and 1-octanol is not detailed in literature and attempted through this work.

The main goal of this research was to physically extract acid using toluene, xylene, MIBK, and 1-octanol diluents to determine if the extraction procedure was feasible. Furthermore, the process is intensified by using a suitable extractant (Aliquat- 336) to maximize the efficiency. A parameter study was performed to optimize the process, estimate the maximum extraction efficiency, and enhance the recovery of the acid. The current study focuses on achieving maximum extraction efficiency using both physical and chemical extraction methods. Preliminary investigations were conducted to select the appropriate diluent using physical extraction experiments. Diluents with high efficiency were further selected to examine their performance in the reactive extraction study. In this study, Aliquat 336 + MIBK and Aliquat 336 + xylene were examined in batch reactive extraction experiments for process optimization. The experimental results of this study are presented in terms of the extraction efficiency (E) and distribution coefficient (K_D).

MATERIALS AND METHODS

Aliquat 336, a quaternary amine with the chemical name methyl tri-capryl ammonium chloride, is a mixture of C_8-C_{10} and has a density of 0.888 $g.cm^{-3}$. It was purchased from SD Fine Chem. Ltd., India. The diluents toluene (99.0 %), methyl iso-butyl ketone (MIBK) (99.0%), and xylene (99.0%) were purchased from Loba Chemicals, whereas 1-octanol (99.0%) was purchased from HiMedia Laboratories, India. Sodium acetate anhydrous with an assay of 99.0% was supplied by Merck Specialties, India. HCl and NaOH used for titration were of analytical grade. Each experiment was analyzed using freshly prepared 0.01 N HCl for the aqueous phase and 0.01 N NaOH for the organic phase. Distilled water was used to prepare synthetic sodium acetate solutions of various concentrations (0.2–0.5 $moL.L^{-1}$). The compounds used in the equilibrium experiments are listed in Table 1.

Efficient diluents were selected from the results of the physical extraction experiments and used for the reactive extraction study. Reactive extraction experiments were

Table 1: List of chemicals required for physical and reactive extraction experiments.

Material	Quantity details
Aqueous sodium acetate	Synthetically prepared 15 mL
Diluents: MIBK, Xylene, 1-Octanol and Toluene	15 mL each
Extractant: Aliquat-336	20%, 25% and 33.33% on volume basis
Range of initial aqueous phase for reactive extraction	0.2 to 0.5 $moL.L^{-1}$

independently performed for both the prepared organic phases, Aliquat-336 + MIBK and Aliquat-336 + xylene.

Batch Extraction

A synthetic sodium acetate solution and an organic solution were mixed in equal volumes, that is, 15 mL each, in a 100 mL flask and constantly swirled for three hours at a steady temperature using a magnetic stirrer (Remi 2MLH 500 rpm). A thermocouple PT 100 with an accuracy of ± 2 K recorded the temperature of the mixture. The equilibrium mixture was transferred to a separating funnel for the separation of the aqueous and organic layers after stirring for a stipulated time. After overnight settling, the two immiscible layers were withdrawn separately for sample analysis. 0.01 N NaOH was used to titrate the organic layer, whereas the sample from the aqueous layer was titrated with 0.01 N HCl. The sample analysis was repeated to check the accuracy of the results within ± 2%.

Calculations: Distribution coefficient (K_D), extraction efficiency(E), and loading ratio(Z).

All experimental results were tabulated as the distribution coefficient (K_D), extraction efficiency or degree of extraction (E%), and loading ratio (Z).

Distribution Coefficient: The distribution coefficient, or K_D, is determined by the equation and is defined as the ratio of the acid concentration in the organic phase to that in the aqueous phase under equilibrium conditions (1).

$$K_D = \frac{[HAA]_{org}}{[HAA]_{aq}} \quad \ldots(1)$$

Where, $[HAA]_{org}$ the total acetic acid concentration in the organic phase (both dissociated and undissociated in case of reactive extraction with Aliquat 336) and $[HAA]_{aq}$ is the total amount of dissociated and undissociated acetic acid present in the aqueous phase at equilibrium.

Extraction Efficiency: The ratio of the acid mass moved from the aqueous to the organic phase to the original acid mass in the aqueous phase, under constant volumes and equilibrium circumstances, is known as the extraction efficiency or percentage. Equation (2) represents it in terms of the distribution coefficient.

$$E\,\% = \frac{K_D}{(1+K_D)} \times 100 \quad \ldots(2)$$

Loading ratio: The loading ratio, Z, quantifies the degree of saturation of the organic phase with the acid. It is the ratio of the total number of moles of AA extracted from the aqueous phase to the total number of moles of Aliquat 336 initially present in the organic phase, as expressed in Equation (3).

$$Z = \frac{[HAA]_{org}}{[\text{Aliquat } 336]_{org}} \quad \ldots(3)$$

RESULTS AND DISCUSSION

Physical Extraction

Extraction performed using a diluent alone as the organic phase is termed physical extraction. The selected diluents (MIBK, xylene, 1-octanol, and toluene) were tested in individual physical extraction experiments. These highly polar solvents increase their ability to donate electron pairs (Lewis basicity), which in turn increases the distribution coefficient of AA. The mechanism of the sequential reactions is represented by Equations (4)–(9). The ionization of sodium acetate in the aqueous phase is represented by Eq. (4). It dissociates into Na^+ and CH_3COO^- ions, respectively.

$$CH_3COONa + H_2O \rightarrow CH_3COO^- (aq) + Na^+ (aq) \quad \ldots(4)$$

The acetate ion, a strong base, undergoes protonation by water to yield acetic acid (eq. 5) and the OH ion. This extra OH^-(aq) makes the solution basic (Eq. 6)

$$CH_3COO^- (aq) + H_2O \rightarrow CH_3COOH + OH^- (aq) \quad \ldots(5)$$

$$Na^+ (aq) + OH^- (aq) \rightarrow NaOH \quad \ldots(6)$$

The stoichiometry of the reaction suggests that equivalent moles of acetic acid (AA) and NaOH are formed in the aqueous layer. The amount of NaOH formed in the aqueous layer was estimated by titrating it against 0.01 N HCl. AA present in the aqueous phase is transferred to the organic phase due to the high solvation capacity of the organic solvents used and estimated using 0.01 N NaOH. Thus, during physical or reactive extraction, AA is extracted in the organic phase. The ionization of acetic acid in the aqueous phase is K_a and is represented by Eq. (7).

$$HAA_{aq} + H_2O \leftrightarrow H_3O^+ + HAA^-$$

$$K_a = \frac{[H3O]^+[HAA]^-}{[HAA]} \quad \ldots(7)$$

Partition (P) of undissociated acetic acid between two phases is,

$$[HAA]_{aq} \leftrightarrow [HAA]_{org} \quad \ldots(8)$$

$$P = \frac{[HAA]_{org}}{[HAA]_{aq}} \quad \ldots(9)$$

Effect of Sodium Acetate/Acetic Acid Concentration

In this parameter study, the effect of a change in the initial concentration of sodium acetate/acetic acid in the aqueous phase on the extraction efficiency was studied. Physical extraction experimental observations were made for aqueous phase sodium acetate/acid concentrations ranging from 0.1-0.5 $moL.L^{-1}$, and extraction efficiencies with diluents MIBK, xylene, 1 -octanol, and toluene were estimated in independent experiments. Table 2 displays the results of the physical extraction at equilibrium conditions in terms

Table 2: The physical extraction efficiency of the diluents was determined at 303 K and an initial acid concentration range of 0.1–0.5 moL.L^{-1}.

Initial concentration $[HAA]_o$ moL.L^{-1}	% E MIBK	% E Xylene	% E 1-Octanol	% E Toluene
0.1	42.4	35.0	31.0	27.3
0.2	46.7	38.3	32.5	31.2
0.3	52.8	43.0	36.7	34.8
0.4	56.4	45.8	37.6	35.3
0.5	60.3	48.9	42.5	38.3

of the degree of extraction. The extraction efficiency was observed for all diluents within the range of a minimum 27% to a maximum of 60 %. At all acid concentrations (0.1-0.5 moL.L^{-1}), the diluents MIBK and xylene exhibited better extraction efficiencies, whereas toluene and 1-octanol exhibited the lowest efficiencies (Fig. 1). This is specifically due to the disparities in polarity and dielectric constant, which are diluent physicochemical characteristics that influence the solvation and extraction behavior of AA ions from the aqueous phase.

The distribution coefficients of the diluents, that is, MIBK, xylene, 1-octanol, and toluene, are very low; hence, the % extraction recovery is also low. It was observed that at constant temperature 303 K, increase in the initial acid concentration from 0.1 to 0.5 moL.L^{-1}, the percentage extraction recovery increases for all the diluents. Comparing the average extraction efficiency for all diluents, MIBK and xylene were found to be superior to 1-octanol and toluene, and hence, were selected for the reactive extraction experiments (see Table 3).

Reactive Extraction

The extraction process is considerably intensified with the addition of the third component, the extractant, to the mixture. The synergetic effect of the extractant substantially enhanced the process performance. Long-chain quaternary amines have been found to have a greater ability to extract carboxylic acids at both acidic and basic pH values. Hence, Methyl tri-capryl ammonium chloride (Aliquat-336), a quaternary amine, was selected as a suitable extractant for this study. The selected extractant, Aliquat 336, has a strong affinity to form acid-amine complexes owing to the presence of long-chain amines and specific chemical interactions between the amines and acid molecules, thus causing separation.

Table 3: Average Distribution coefficient and extraction efficiency in physical extraction at initial acid concentration of AA 0.1 - 0.5 moL.L^{-1} and temperature 303 K.

Diluents	K_D Range	K_D Average	% E Range	%E Average
MIBK	0.7 to 1.5	1.11	42.4 to 60.3	51.7
Xylene	0.5 to 1.0	0.74	35.0 to 49	42.2
1-Octanol	0.45 to 0.74	0.57	31 to 42.5	36.05
Toluene	0.38 to 0.62	0.51	27.3 to 38.3	33.35

The amine extractant is dissolved in diluents such as ketones, alcohols, or hydrocarbons to the desired concentration and then applied for extraction. The addition of diluents controls the density and viscosity of the solvent phase, thereby enhancing the mobility and interaction of the extractant with the acid ions. Diluents are added to improve the physical properties of the extractant, such as its solubility and viscosity. They also influence the extraction power of the extractant by providing specific interactions with the solute.

From studies, it was noted that Methyl tri-capryl ammonium chloride (Aliquat 336), a quaternary amine salt, extracts a broader extraction range than Alamine 336 as it extracts both dissociated and undissociated forms of

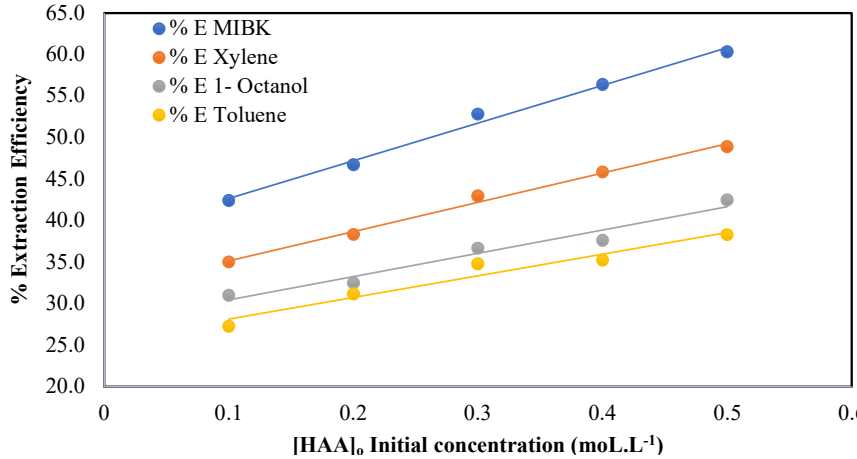


Fig. 1: Diluent extraction efficiency at 303 K for 0.1-0.5 moL.L^{-1} of initial acid concentration.

the acids. This indicates that the extraction efficiency is independent of the pH of the aqueous solution (Kar et al. 2017).

The selected extractant, Aliquat 336, is highly viscous (1500 mPa.s) and dense (0.8884 g.cm^{-3}) in nature; hence, it is mixed with selected diluents to increase its solvation capacity. MIBK and xylene diluents were mixed with Aliquat 336 in independent runs, and the extraction efficiency was estimated in each case. The reactive extraction of AA by Aliquat 336 involves a three-step mechanism: 1) mass transfer of acetic acid from the aqueous phase to the organic phase interface, 2) chemical reaction between acetic acid and Aliquat 336 at the interface, and 3) mass transfer of the formed complex into the bulk organic phase (Yang et al. 2013). See Eq. 10.

$$HAA_{aq} + [\text{Aliquat 336: } (HAA)_{n-1}]_{org} \leftrightarrow [\text{Aliquat 336: } (HAA)_n]_{org} \quad \ldots(10)$$

n =1,2, or 3

This complex formation is an essential step in the separation of AA from the aqueous phase.

Reactive extraction of AA using Aliquat-336 with diluents MIBK and xylene as an organic phase was performed at temperatures of 303 K, 313 K and 323 K and Aliquat-336 concentrations of 0.437, 0.547, and 0.729 moL.L^{-1} (20, 25, and 33.33% vol) at atmospheric pressure in independent studies.

Parameter Study

Parameters such as AA concentration, extractant-to-diluent ratio, and temperature were investigated for process optimization.

Effect of Initial Sodium Acetate or Acetic Acid Concentration

The initial acid/sodium acetate concentrations of the aqueous phase were altered and mixed with a known constant amount of organic phase (20 vol% extractant) in individual experiments. The effect of acetic acid concentration on extraction recovery was noted, and further experiments were performed for higher Aliquat 336 concentrations (25 and 33.33 vol%).

From the results (Table 4), it is evident that at fixed temperature 303 K and 20% volume fraction of Aliquat 336, as acid concentration increased from 0.2 to 0.5 moL.L^{-1}, the extraction efficiency increased from 49.0 to 65.0% for Aliquat 336 + MIBK organic phase, whereas 40.0 to 50.8% for Aliquat 336 + xylene organic phase. Similar results were observed for the distribution coefficient, K_D, which varied from 0.96 to 1.86 for Aliquat 336 + MIBK and 0.67 to 1.03 for Aliquat 336 + xylene. The transfer of acid molecules at the interphase increases, and the strong affinity of Aliquat 336 fetches the acid ions to the organic phase, forming a reaction complex.

Effect of the Extractant to Diluent Ratio

The extractant-to-diluent ratio was varied by maintaining other parameters, such as the initial concentration and temperature, constant. The concentration of the extractant Aliquat 336 was adjusted to 20–33.33 vol%. The diluents MIBK and xylene were evaluated for changes in extraction efficiency in individual experiments. Aliquat 336, a quaternary amine, is a highly viscous liquid that must be diluted with solvents to decrease its viscosity and increase its solvation capacity. The literature, as noted by Wasewar et al. (2011), suggests that an extractant concentration in the range of 10–30% by volume is sufficient for experimental analysis of its feasibility. Higher extractant concentrations lead to a decrease in the extraction efficiency of the extractant. This is mainly due to the inability of the extractant to fetch acid ions at the interface, possibly due to the co-bonding of extractant ions among themselves in a highly viscous liquid. In addition, a higher extractant concentration alters the physical properties of the organic phase, leading to the formation of a third phase that further lowers the extraction efficiency.

Experiments were conducted for aqueous phase AA concentrations from 0.2 to 0.5 moL.L^{-1} and 20, 25, and 33.33 vol% (0.437, 0.547, and 0.729 moL.L^{-1}) Aliquat 336 in the organic phase at 303 K. Table-4, represents the effect of variation in acid concentration (0.2-0.5 moL.L^{-1}) and Aliquat 336 concentration (20, 25, and 33 vol. %) at a constant temperature of 303 K. The experimental results revealed that with an increase in the extractant-to-diluent concentration, the extraction efficiency increased at a constant initial acid concentration and temperature. At a constant temperature, when the concentration of the extractant was increased, that is Aliquat 336 to diluent (MIBK and xylene) ratio was increased from 20 to 25 and then to 33.33 vol%, the experimental results indicated an increase in the extraction efficiency for both diluents. For higher concentrations of Aliquat 336 + MIBK, the extraction efficiency increased and was found to be in the range of 51- to 69% for 25 vol% % and 57 to 73.60% for 33.33 vol% of the extractant to diluent ratio. Comparable results were observed for the Aliquat 336 + xylene with an extraction efficiency of RE in the cumulative range of 49.0 to 64.4 % for 25-33.33 vol% of organic phase. Since, Aliquat 336 interacts more strongly with acid molecules to create a reaction complex, the highest extraction efficiency was recorded at an extractant concentration of 33.33 vol%.

At a lower concentration of 20% by volume (0.437 moL.L^{-1}) of Aliquat 336, the extraction efficiency varied between 48 and 64% for the MIBK diluent. Selecting extractant concentration below 0.437 moL.L^{-1} (20 vol%)

would not justify the purpose and objective of adding extractant to the solution, hence experiments were conducted and analysed at three different extractant concentrations of 20, 25 and 33.33 vol%

Fig. 2 displays the comparative results of the extraction efficiency considering both diluents MIBK and xylene at a fixed temperature of 303 K and an acid concentration of 0.5 moL.L^{-1}. From the results, it can be concluded that MIBK is a better diluent than xylene for achieving the highest extraction of acid molecules. Higher concentrations of Aliquat 336 in MIBK or xylene resulted in an increased number of available active sites for acid ion complexation, leading to enhanced solvation and extraction capacity.

The hydrophobic nature of the Aliquat-AA complex formed, and the concentration gradient developed due to availability of AA in the dilute $[HAA]_{aq}$ phase, together intensify the RE process and form a reaction complex as $[(HAA)_n–$ Aliquat$_{org}]$. This necessarily means that acid ions are associated with the extractant. Because the value of the loading ratio Z is less than 0.5 in all experiments at 303 K, we can conclude that a 1:1 acid: extractant complex is formed. At a high initial concentration of acid in the aqueous phase (0.5 moL.L^{-1}) and an extractant concentration of 0.437 moL.L^{-1}, the value of Z was 0.581 and decreased as the extractant concentration increased (Z was 0.561 for 0.547 moL.L^{-1} and Z was 0.453 for 0.729 moL.L^{-1} of extractant concentration). This suggests that in such situations, when the initial acid concentration is high in the aqueous phase, an increase in the extractant concentration maintains the 1:1 acid-Aliquat 336 reaction complex.

Table 4: Reactive extraction equilibrium results of Acetic acid + (Aliquat 336: MIBK) and Acetic acid + (Aliquat 336: xylene) system at 303 K.

[Aliquat 336]$_{org}$ moL.L^{-1}	$[HAA]_o$ Initial moL.L^{-1}	MIBK				Xylene			
		$[HAA]_{org}$ moL.L^{-1}	K_D	%E	Z	$[HAA]_{org}$ moL.L^{-1}	K_D	%E	Z
0.437	0.2	0.096	0.92	48.0	0.22	0.08	0.67	40.0	0.18
	0.3	0.15	1.09	52.0	0.36	0.126	0.72	42.0	0.29
	0.4	0.231	1.37	57.7	0.54	0.185	0.86	46.2	0.42
	0.5	0.321	1.79	64.2	0.74	0.254	1.03	50.8	0.58
0.547	0.2	0.092	0.91	47.6	0.19	0.098	0.96	49.0	0.18
	0.3	0.164	1.22	55.0	0.31	0.16	1.14	53.3	0.29
	0.4	0.25	1.67	62.0	0.47	0.236	1.44	59.0	0.43
	0.5	0.341	2.14	68.2	0.64	0.308	1.60	61.6	0.56
0.729	0.2	0.108	1.17	54.0	0.16	0.109	1.20	54.5	0.15
	0.3	0.182	1.54	60.6	0.25	0.175	1.40	58.3	0.24
	0.4	0.27	2.08	67.5	0.38	0.244	1.56	61.0	0.33
	0.5	0.365	2.70	73.0	0.50	0.322	1.81	64.4	0.44

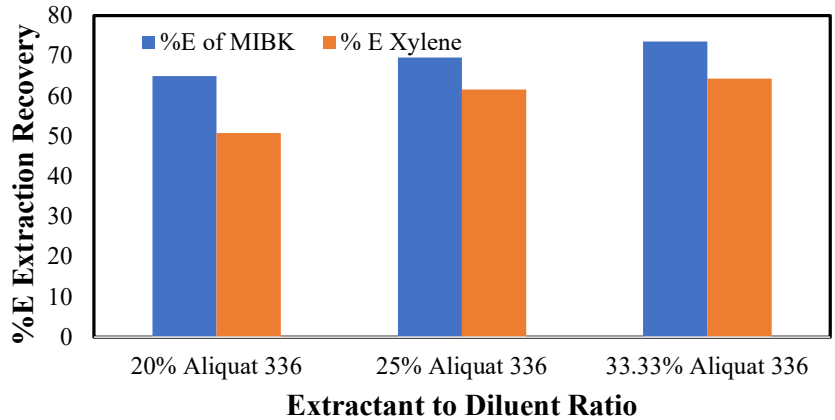
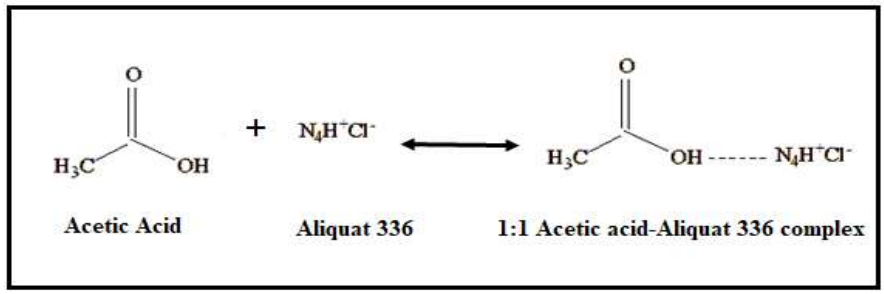


Fig. 2: Extraction efficiencies for Aliquat 336: MIBK and Aliquat 336: xylene at concentrations of 20, 25, and 33.33 vol% at an initial acid concentration of 0.5 moL.L^{-1} and 303 K.

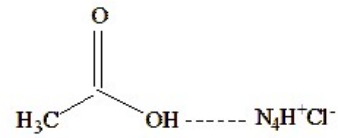
Chemical Reaction Mechanism and Complex Formation

The reaction complex formed between acid and Aliquat molecule is represented by equation (10), and can be represented as 1:1 ([(HAA) – $Aliquat_{org}$]), 2:1 ([$(HAA)_2$ – $Aliquat_{org}$]) or 3:1 ([$(HAA)_3$ – $Aliquat_{org}$]). Fig. 3 shows the structures of the formed complexes. As experimentally investigated and displayed in Table 4, the value of Z<0.5 for almost all observations.

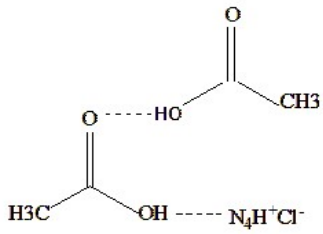
This indicates a 1:1 reaction complex is formed between acetic acid and Aliquat 336, as displayed by the reaction in eq (11).



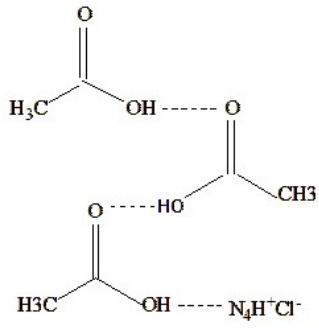
...(11a)



a)



b)



c)

Fig. 3: Acetic acid–Aliquat 336 a) 1:1 b) 2:1 c) 3:1 complex structure.

$$HAA_{aq} + [\text{Aliquat 336}]_{org} \leftrightarrow [\text{Aliquat 336: } HAA]_{org} \quad \ldots(11b)$$

The equilibrium complexation constant (Keq) for the reaction is evaluated by eq. (12)

$$Keq = \frac{[\text{Aliquat 336: } HAA]_{org}}{[\text{Aliquat 336}]_{org}\ [HAA]_{aq}} \quad \ldots(12)$$

Complexation constant is also estimated from loading ratio values as mentioned in literature (Sharma et al. 2017) and displayed by equation (13)

$$Keq[HAA]_{aq} = \frac{Z}{1-Z} \quad \ldots(13)$$

Effect of a Rise in Temperature

This parameter is important for understanding the behavior of the system at elevated temperatures for extraction and back extraction studies. Most of the process wastewater from fermentation broth is at approximately room temperature (303 K), whereas that of the pigment and metal industry may vary from 313-323 K. It is essential to understand the extraction efficiency and degree of extraction at these temperatures. The reaction is exothermic and favorable at lower temperatures (303 K). Studies (Rewatkar et al. 2016, Antony & Wasewar 2020, Demir 2021) mention a stable acid-extractant complex at lower or room temperatures, resulting in a high degree of extraction, whereas high temperatures result in back extraction due to reaction at a high order and lower down the extraction efficiency.

Independent batch experiments under identical batch conditions were performed to quantify the impact of temperature on the reactive extraction of acetic acid using xylene and MIBK diluents. The parameters, such as initial AA and extractant concentration, were also varied in the temperature range of 303, 313, and 323 K to estimate the

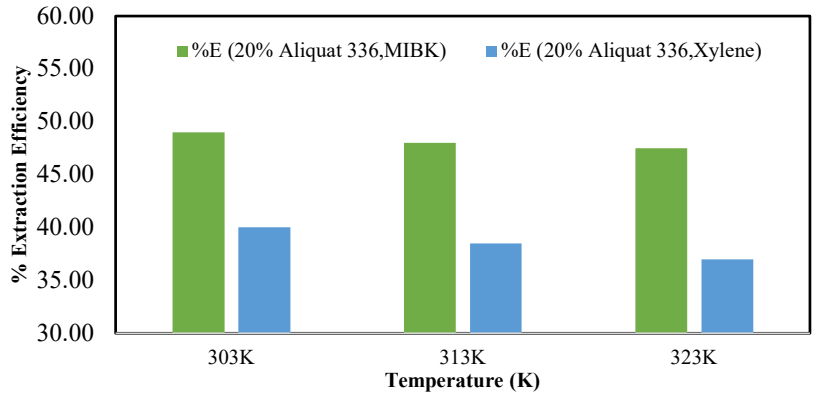


Fig. 4: Effect of change in temperature (303 to 323 K) of 20% Aliquat 336+ (MIBK and xylene) diluents on % extraction recovery of $[HAA]_o$ an initial acid concentration of 0.2 moL.L^{-1}.

Table 5: Equilibrium results for the reactive extraction of Acetic acid + (Aliquat 336: MIBK) and Acetic acid + (Aliquat 336: xylene) systems at 313 K.

$[Aliquat\ 336]_{org}$ moL.L^{-1}	$[HAA]_o$ Initial moL.L^{-1}	MIBK				Xylene			
		$[HAA]_{org}$ moL.L^{-1}	K_D	%E	Z	$[HAA]_{org}$ moL.L^{-1}	K_D	%E	Z
0.437	0.2	0.096	0.92	48.0	0.22	0.077	0.63	38.5	0.18
	0.3	0.15	1.09	52.0	0.34	0.08	0.67	40.0	0.18
	0.4	0.231	1.37	57.7	0.53	0.178	0.80	44.5	0.41
	0.5	0.321	1.79	64.2	0.73	0.244	0.95	48.8	0.56
0.547	0.2	0.092	0.91	47.6	0.17	0.092	0.85	46.0	0.17
	0.3	0.164	1.22	55.0	0.30	0.153	1.03	50.6	0.28
	0.4	0.25	1.67	62.5	0.46	0.246	1.41	58.5	0.45
	0.5	0.341	2.14	68.2	0.62	0.291	1.39	58.2	0.53
0.729	0.2	0.108	1.17	54.0	0.15	0.108	1.17	54.0	0.15
	0.3	0.182	1.54	60.6	0.25	0.171	1.33	57.0	0.23
	0.4	0.27	2.08	67.5	0.37	0.243	1.55	60.7	0.33
	0.5	0.365	2.70	73.0	0.50	0.311	1.65	62.2	0.43

Table 6: Equilibrium results for reactive extraction of Acetic acid + (Aliquat 336: MIBK) and Acetic acid + (Aliquat 336: xylene) system at 323 K.

$[Aliquat\ 336]_{org}$ moL.L^{-1}	$[HAA]_o$ Initial moL.L^{-1}	MIBK				Xylene			
		$[HAA]_{org}$ moL.L^{-1}	K_D	%E	Z	$[HAA]_{org}$ moL.L^{-1}	K_D	%E	Z
0.437	0.2	0.095	0.90	47.5	0.22	0.074	0.59	37.0	0.17
	0.3	0.146	1.01	50.3	0.33	0.079	0.65	39.5	0.18
	0.4	0.226	1.30	56.5	0.52	0.17	0.74	42.5	0.39
	0.5	0.311	1.65	62.2	0.71	0.235	0.89	47.0	0.54
0.547	0.2	0.089	0.80	44.5	0.16	0.088	0.79	44.0	0.16
	0.3	0.161	1.18	54.0	0.29	0.148	0.97	49.3	0.27
	0.4	0.246	1.60	61.5	0.45	0.226	1.30	56.5	0.41
	0.5	0.333	1.99	66.6	0.61	0.283	1.30	56.6	0.52
0.729	0.2	0.104	1.08	52.0	0.14	0.101	1.02	50.5	0.14
	0.3	0.178	1.46	59.3	0.24	0.165	1.22	55.0	0.23
	0.4	0.265	1.96	66.2	0.36	0.233	1.40	58.2	0.32
	0.5	0.359	2.55	71.8	0.49	0.299	1.42	58.6	0.41

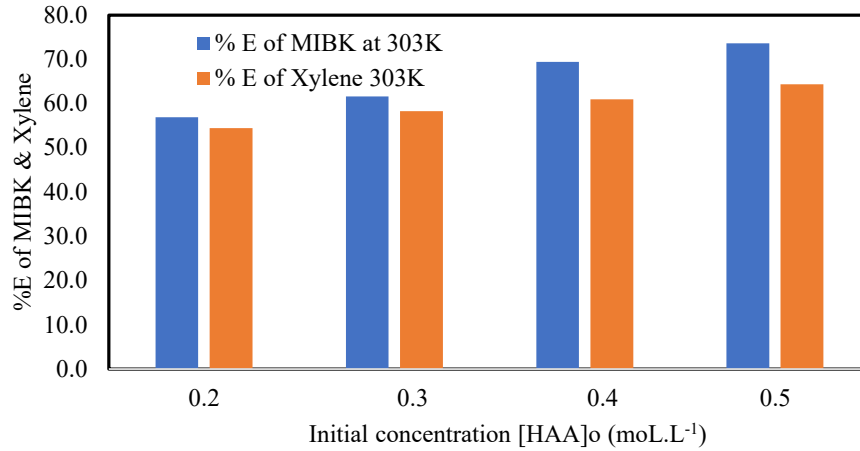


Fig. 5: Comparative extraction efficiency of 0.729 moL.L^{-1} of Aliquat 336 +MIBK and Aliquat 336+xylene at 303K.

extraction efficiency. The experimental findings at 303 K are listed in Table 4, and the results at 313 and 323 K are listed in Tables 5 and 6, respectively. The results clearly illustrate that as the temperature of the reacting mixture increased, the distribution coefficient and extraction efficiency decreased. The experimental data in Tables 4, 5, and 6 show that as the temperature increases, the percentage extraction decreases. Among the experimental readings at 303, 313, and 323 K, the distribution coefficient and extraction efficiency were the lowest at 323 K. As the temperature increased, the reverse phenomenon was initiated, leading to the back transfer of acid ions to the aqueous phase. Thus, the temperature of the reaction mixture and the extraction efficiency were found to be inversely proportional to each other. Fig. 4 reflects the effect of a temperature change (303 to 323 K) on % extraction recovery for an initial acid concentration of 0.2 moL.L^{-1} and 20 vol% of extractant/diluent organic phase. For Aliquat 336-MIBK extractant, the % extraction was 49% at 303 K, 48% at 313 K, and 47.50 % at 323 K, whereas for Aliquat 336-xylene, the % extraction was 40% at 303 K, 38.50% at 313 K, and 37.00 %. This reduction in extraction efficiency is due to the thermodynamically favored back-extraction of the acid from the organic phase to the aqueous phase at elevated temperatures. This reverse extraction is undesirable for AA removal; hence, extraction at room temperature is recommended for most reactive extraction processes.

Overall, the observations suggest that acid removal from aqueous sodium acetate solutions is possible using reactive extraction technology. Based on the results of the physical extraction study, two suitable diluents, MIBK and xylene, were selected for further intensification of the process. Both the diluents, i.e., MIBK and xylene, were mixed with the extractant, i.e., Aliquat 336, in 20-33.33 vol%, and their % extraction recovery was studied. A higher initial acid concentration with a 33.33 vol% extractant-to-diluent ratio and 303 K were the optimized parameters for the process. Nevertheless, the initial acid concentration cannot be controlled; hence, the full range from 0.2 to 0.5 moL.L^{-1} of initial acid concentration, mostly found in industrial wastewater was selected as a base for the study. A comparative graph (Fig. 5) is displayed for both organic phases with extractant + MIBK and extractant + xylene at a higher extractant concentration of 0.729 moL.L^{-1} (33.33 vol% at 303 K. Extraction recovery of Aliquat 336+ MIBK is superior to that of Aliquat + xylene at constant temperature. At all experimental conditions, it is visible, and hence, it can be concluded that Aliquat 336+ MIBK is a suitable organic phase for the extraction of acid from aqueous sodium acetate waste.

DISCUSSION

This study highlights the efficacy of reactive extraction for acetic acid recovery from sodium acetate waste. MIBK combined with Aliquat 336 exhibited a superior extraction efficiency (73.6%) compared to xylene (64.4%) under optimal conditions. An earlier study (Juang & Wu 1999) reported the removal of acetate/acetic acid from a mixture of sodium acetate, acetic acid, and sodium chloride, and calculated the efficiency of Aliquat 336 with a xylene molar concentration of 0.5 M to be approximately 30%. However, higher extraction efficiency was observed for all concentrations of acid (0.2-0.5 moL.L^{-1}) and extractant (0.437-0.729 moL.L^{-1}) between the range 47-72% for MIBK and 37-59% for xylene solvent in the current work at comparable temperature and extractant/diluent conditions. Temperature inversely impact efficiency, suggesting room-temperature operations for industrial scalability in this study. These findings demonstrate an energy-efficient and environmentally sustainable alternative for waste treatment.

CONCLUSIONS

The recovery of sodium acetate/acetic acid from an aqueous stream using the reactive extraction process presented in this study offers a promising approach for acid recovery from industrial waste containing sodium acetate. Synthetic aqueous sodium acetate was prepared, and an extraction study was performed to recover the acetic acid formed due to the hydrolysis reaction. Physical and reactive extraction experiments were performed, and further parameter studies were conducted to optimize the process.

The current study highlights the following key findings.

- Among the diluents tested, MIBK and xylene were found to be the most suitable diluents, based on the physical extraction investigations.
- A synergetic effect was observed for the reactive extraction with Aliquat 336 in MIBK and xylene, as it enhanced the recovery of AA compared to the physical extraction. At a constant 0.5 moL.L^{-1} of acid at 303 K, physical extraction experiments demonstrated average extraction efficiencies of 60% for MIBK and 48.9% for xylene. In contrast, when reactive extraction was employed under the same acid concentration and temperature conditions, while maintaining 33.33 vol% Aliquat 336 in MIBK and xylene, the extraction efficiencies were enhanced to 73.60% and 64.40%, respectively. Overall, the extraction efficiency increased by 10%.
- Higher initial concentrations (0.5 moL.L^{-1}) of sodium acetate/acetic acid in aqueous solution exhibited more interaction of acid-organic molecules, thus increasing the distribution ratio and extraction efficiency.
- As the concentration of the extractant increased, so did the distribution coefficient and, therefore, the extraction efficiency. The extraction efficiency was determined to be 65% for 0.437 moL.L^{-1} of extractant at a constant temperature of 303 K and an acid concentration of 0.5 moL.L^{-1}. The extraction efficiencies were 69% and 73% for the extractants at 0.547 and 0.739 moL.L^{-1}, respectively.
- The temperature parameter analysis showed that when the temperature increased, the extraction efficiency and distribution coefficient for the diluents MIBK and xylene decreased. Hence, extraction at room temperature (303 K) is recommended.
- The findings suggest that with further research and refinement, this process can be scaled up and widely implemented in industrial wastewater treatment and resource recovery applications.

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