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Original Research

Reactive Extraction of Acetic Acid from Aqueous Sodium Acetate Waste

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ABSTRACT

Recovery of acetic acid from aqueous sodium acetate using traditional methods is cost and energy intensive. Current work emphasis on exploring synergetic reactive extraction methodology to extract acetic acid from the aqueous sodium acetate waste. Physical extraction experiments demonstrated methyl isobutyl ketone (MIBK) and xylene to be effective diluent among MIBK, xylene, octanol, and toluene. Further extraction efficiency was enhanced by adding Aliquat 336 as an extractant and MIBK and xylene diluents in independent runs. Parameters such as initial acid concentration in aqueous phase, Aliquat 336 concentration in organic phase and temperature of the reaction mixture, were investigated to optimise operating conditions. In all conditions MIBK yielded better results, for high acid concentration in aqueous phase (0.5 mol/lit), 60% extraction efficiency was observed in physical extraction experiments. Adding Aliquat 336 as an extractant (0.729 mol/lit) to the mixture in identical experimental conditions 73% extraction efficiency was achieved. Average extraction improved by 10% for 0.2-0.5 mol/lit of initial acid concentration using reactive extraction technology. Such a recovery from aqueous sodium acetate using reactive extraction is rarely reported and hence presented in this paper.

1. INTRODUCTION

Recovery of chemicals from waste streams is gaining importance both due to economic and environmental concerns. The effluent of process industry comprises of salts, acids, alcohols, pesticides, dyes, and chemicals which cause severe pollution to the freshwater streams. The development of advanced separation technologies for wastewater treatment is imperative to minimize environmental impact and maximize resource recovery. Previous decades have witnessed intensive work on the recovery of valuable chemicals such as carboxylic acid, alcohols, esters, and metals from wastewater solution (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 source is crucially important in coming years.

Sodium acetate is an important chemical entity produced as a waste from many pharmaceutical, insecticide, pesticide, metal, fermentation and dye manufacturing industries. This waste is either dumped in land, incinerated or released in water bodies to create pollution. Release of sodium acetate in freshwater streams causes acetate to hydrolyse and form acetic acid thereby affecting the pH of the local water bodies over the due course of time. Removal of this acetate at source itself 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 minimise pollution and utilise the recycle resources. Literature mentions few articles on recovery/removal of sodium acetate waste utilising technologies such as electro dialysis, crystallisation, membrane separation, reverse osmosis and ozone oxidation. In the synthesis of dially alcohol ketone acetate, sodium acetate is found as one of the major ingredients in the waste streams. It is further separated and recycled back with sequence of unit operations involving precipitation, filtration, washing, crystallisation and drying. This method reduces energy consumption and is claimed to have zero emission or discharge (Liu et al., 2013). Similarly, recovery of sodium acetate (76.6 wt%) from dithianon manufacturing industry is studied using bipolar membrane electrodialysis (Xue et al., 2015). These experiments are studied under different current density and membrane stacks to optimise the parameters and maintain the purity of the acid formed. This method is energy consuming and recovers only the dissociated acid ions from the slurry. Later coupling of diffusion and electrodialysis with bipolar membrane were investigated to achieve pure acetic acid and NaOH. This coupling reduced energy demand as compared to bipolar membrane electrodialysis alone (Xue et al., 2018). Modeling studies on electrodialysis method for acetic acid separation from sodium acetate slurry helps in optimising parameters for maintaining purity of acid separated (Fidaleo & Moresi 2005). Sodium salts of organic acids are recovered from the fermentation broth by discontinuous evaporation crystallisation method without need of further purification. Sodium acetate is produced as a byproduct in fermentation broth and is wisely utilised for intermediate crystallisation to fetch other salts along with it and reduce further purification needed (Plate & van Esch 2018). This method is claimed to save the operation cost of the upstream processes of fermentation broth. However, efficiency of the process is not clear as it depends upon the solubility of the salt. Recent study addresses the removal of sodium acetate and sodium chlorides salts from the wastewater of pigment producing industries using nanofiltration membranes and reverse osmosis technology (Chu et al., 2020; Gubari et al., 2023). Advance technology like ozone oxidation is also investigated for maximum degradation of sodium acetate wastewater (Yang et al., 2012; Yang &Yuan 2014).

Among all the technologies reported, an attempt is made to recover sodium acetate from waste or recycle to the plant. All these reported methods are energy and cost intensive, thus, an urge for alternate less energy consuming technologies to process the effluent containing sodium acetate waste for acid recovery is to be explored Since sodium acetate is readily soluble in water and dissociates to form acetic acid ions, extraction is suggested as one of the less energy consuming and cheaper methods available as compared to above-mentioned technologies. These limited studies barely report reactive extraction technology applied for acetic acid removal from aqueous sodium acetate waste. A single study on acetate waste of chloromycetin production for removal of sodium acetate, acetic acid and sodium chloride mixture using Aliquat 336, Alamine, tributyl phosphate and tri octyl phosphine oxide as extractant and xylene as diluent were interrogated for better extraction efficiency (Juang &Wu 1999). Through this work, an attempt is made to recover acetic acid from aqueous sodium acetate waste.

Reactive extraction is a mature technology for the recovery of commodity chemicals however, recovery of acid from aqueous sodium acetate waste applying the reactive extraction method is under-researched. Though carboxylic acid extraction from waste streams is not new and extensively studied in literature (Djas & Henczka 2018; Uslu & Kirbaslar 2013; Asci & Lalikoglu 2021; Erdas & Marti 2024; Kumar et al., 2021), reactive extraction of aqueous sodium acetate needs in depth analysis. Recovery of acetic acid from wastewater is investigated using different extractants like trioctyl amine (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). It is reported that, tertiary and quaternary amines can extract carboxylic acids at both acidic and basic pH, hence 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 dichloro ethane, xylene and 1 octanol the chemical interaction and solvation ability of dichloro ethane 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, isobuatnol, MIBK, m xylene, o xylene, sec-butyl acetate and toluene were investigated. Among all these solvents toluene exhibited higher selectivity to acid extraction (Mohadesi & Rezaei 2020). Recovery of acetic acid in industrial processes are given prime importance and less energy-consuming technologies like solvent extraction are extensively studied for the same (Karunanithi et al., 2023). In a nutshell, extraction of acetic acid is done by the combination of the extractant-diluent mixture to change the solvation properties of the organic layer and fetch maximum acid to the organic layer.

The proposed work highlights the removal of acetic acid formed due to hydrolysis of sodium acetate and water from the aqueous sodium acetate layer utilising reactive extraction technology. Diluents selected to explore the extraction ability and compatibility with extractant are among the polar and non-polar diluents. Solvents MIBK, toluene, xylene and 1 octanol were selected to assess their extraction ability and to enhance the properties of extractant. The diluent MIBK and toluene demonstrated superior efficiency as compared to best performers chloroform, octanol, and ethyl acetate in independent studies (Senol 2004; Mohadesi & Rezaei 2020). Hence MIBK and toluene were selected for checking their compatibility for this reaction as well. In earlier study (Juang &Wu 1999), xylene was used as a diluent in combination with extractant Aliquat 336, tri octyl amine, tri butyl phosphate and tri octyl phosphine oxide and efficiency of Aliquat 336 was found to be superior. Hence, Aliquat 336 along with xylene were selected in comparison with other diluents for testing their extraction ability in this work. Also, Aliquat 336 is potential of fetching both dissociated and undissociated acid ions from aqueous phase, thus making it most suitable amine extractant (Wang et al 2019). Though many solvents like ethyl acetate, chloroform are having high distribution ratio, yet the reactivity with NaOH and toxicity to aquatic life are the major parameters of concern. The proposed work is meant to extract acetic acid from sodium acetate waste, if we consider ethyl acetate, at above room temperature, there is possibility it may enter the aqueous phase and react. Moreover, isolating acetate/acetic acid ions with help of acetate ions would become more tedious in such case. Literature mentions (Stamou et al., 2024) hydrolysis of chloroform under basic condition to produce carbanion. Since we have sodium acetate in our aqueous phase, the reactivity and affinity of chloroform to sodium hydroxide in aqueous phase cannot be avoided. Hence this solvent is also avoided for 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 is to physically extract utilizing toluene, xylene, MIBK, and 1-octanol diluents to determine if the extraction procedure is feasible. Further the process is intensified by using suitable extractant (Aliquat- 336) to maximise the efficiency. Parameter study is done to optimise the process and estimate maximum extraction efficiency and enhance the recovery of the acid. Current work stresses on achieving maximum extraction efficiency using both physical and chemical extraction. Preliminary investigations are done for selecting the appropriate diluent by physical extraction experiments. The diluents with high efficiency are further selected to examine their performance in reactive extraction study. In this work, Aliquat 336 + MIBK and Aliquat 336 + xylene was examined in batch reactive extraction experiments for process optimisation. The study's experimental results are presented in terms of extraction efficiency (E) and distribution coefficient (K_D).

2. EXPERIMENTAL

2.1 Materials and Method

The Extractant Aliquat 336 a quaternary amine with chemical name-methyl tri-capryl ammonium chloride is a mixture of C_8-C_{10} and has 0.888 g/cm^3 density. It is 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. The sodium acetate anhydrous with an assay of 99.0% was supplied by Merck specialties, India. The HCl and NaOH used for the titration was of analytical grade. Each experiment was analysed with freshly prepared 0.01 N HCl for aqueous phase and 0.01 N NaOH for organic phase. Distilled water was used to prepare synthetic sodium acetate solution of various concentrations (0.2 to 0.5 mol/lit). The list of compounds used in equilibrium experiments is shown in Table 1.

Efficient diluents were selected from the result table of the physical extraction experiments and were utilized for the reactive extraction study. Reactive extraction experiments were attempted independently, for both the prepared organic phase Aliquat-336 + MIBK and Aliquat 336 +xylene.

Table 1. List of chemicals required in 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	
Range of initial aqueous phase for reactive extraction	20%, 25% and 33.33% on volume basis in case of reactive Extraction 0.2 to 0.5 mol/lit

2.1.1 Batch extraction

A synthetic sodium acetate solution and organic solution were mixed in equal volumes i.e., 15ml each in a conical flask having capacity 100 ml and constantly swirled for three hours at a steady temperature using a magnetic stirrer (Remi 2MLH 500 rpm). Thermocouple PT 100 of accuracy ± 2 K recorded temperature of the mixture. The equilibrium mixture was transferred to a separating funnel for separation of aqueous and organic layer after stirring for stipulated time. After overnight settling the two immiscible layers were withdrawn separately for sample analysis. 0.01 N NaOH was used to titrate organic layer whereas sample from 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 the experimental results were tabulated as distribution coefficient ($\mathbf{K_D}$), extraction efficiency or degree of extraction (E %), and loading ratio (Z).

Distribution Coefficient: The distribution coefficient, or KD, is determined by 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)

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

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

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

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

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

3. RESULTS AND DISCUSSION

3.1 Physical Extraction

Extraction performed using diluent alone as organic phase is termed as physical extraction. 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's basicity), which in turn increases the distribution coefficient of the AA. Mechanism of the sequential reactions taking place are represented by equations (4) to (9). Ionization of sodium acetate in aqueous phase, is represented by eq (4). it gets dissociated as Na^+ ion and CH_3COO^- ion.

$$CH_3COONa + H_2O \longrightarrow CH_3COO^- (aq) + Na^+ (aq) \tag{4}$$

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

$$CH_3COO^- (aq) + H_2O \longrightarrow CH_3COOH + OH^- (aq) \quad ...(5)$$

$$Na^+ (aq) + OH^- (aq) \longrightarrow NaOH \quad ...(6)$$

Stoichiometry of the reaction suggests equivalent moles of acetic acid (AA) and NaOH is formed in aqueous layer. The amount of NaOH formed in aqueous layer is estimated by titrating it against 0.01 N HCl. AA present in aqueous phase is fetched to the organic phase due to the high salvation capacity of used organic solvents and estimated by 0.01 N NaOH. Thus, during the physical or reactive extraction, this AA is extracted in organic phase. Ionisation of acetic acid in aqueous phase is Ka and is represented by eq (7).

$$HAAaq + H_2O \leftrightarrow H_3O^+ + HA^-$$

$$K_a = \frac{[H3O]^+[HAA]^-}{[HAA]} \tag{7}$$

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

$$[HAA]_{aq} \leftrightarrow [HAA]_{org} \tag{8}$$

$$P = \frac{[HAA]_{org}}{[HAA]_{aq}} \tag{9}$$

3.1.1 Effect of sodium acetate/ acetic acid concentration

In this parameter study, the effect of change in initial concentration of sodium acetate/ acetic acid in aqueous phase on extraction efficiency was studied. Physical extraction experimental observations were made for aqueous phase sodium acetate/acid concentration ranging from 0.1-0.5 mol/lit and extraction efficiency with diluents MIBK, xylene, one octanol and toluene were estimated in independent experiments. Table 2 displays the results for physical extraction at equilibrium conditions in terms of degree of extraction. The extraction efficiency was observed for all diluents within the range of minimum 27% to maximum 60 %. At all acid concentration range (0.1-0.5 mol/lit), the diluents, MIBK and xylene exhibited better extraction efficiency whereas toluene and 1-octanol exhibited the least efficiency among all (See Fig. 1). This is specifically due to disparities polarity and dielectric constant, in particular, are diluent physicochemical characteristics that influence the solvation and extraction behaviour of AA ions from the aqueous phase.

Table 2. Physical extraction efficiency of diluents at 303 K and initial acid concentration range 0.1 - 0.5 mol/lit.

Initial concentration $[HAA]_o$mol/Lit	**% 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

The distribution coefficients of diluents i.e., 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 concentration $[HAA]_o$ from 0.1 to 0.5 mol/lit, percentage extraction recovery increases for all the diluents. Comparing average extraction efficiency for all diluents, MIBK and xylene were found to be superior to 1-octanol and toluene and hence, selected further for reactive extraction experiments (see Table 3).

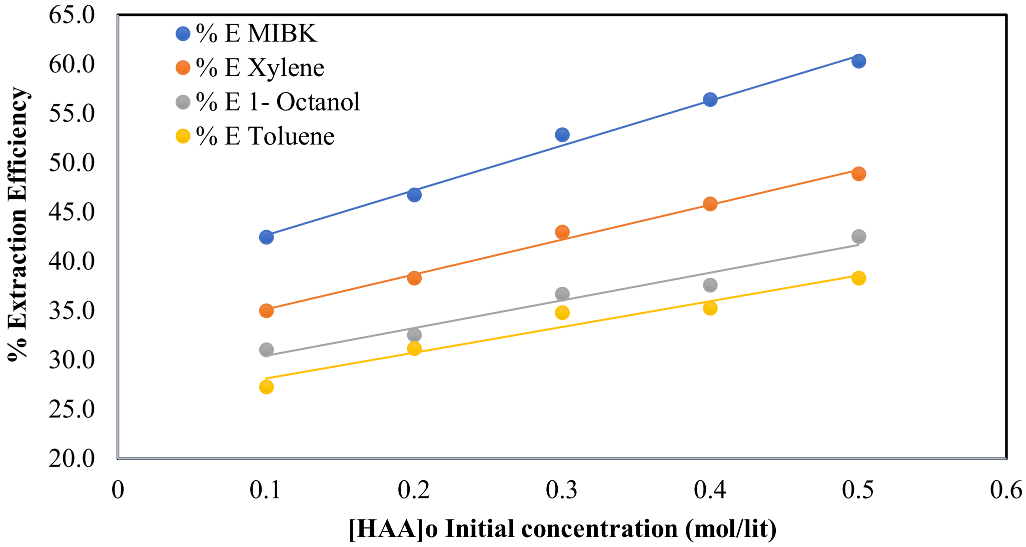


Fig. 1. Diluent extraction efficiency at 303 K for 0.1-0.5 mol/lit of initial acid concentration.

Table 3. Average Distribution coefficient and extraction efficiency in physical extraction at initial acid concentration of AA 0.1 - 0.5 mol/lit 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

3.2 Reactive Extraction

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

The amine extractant is dissolved in diluents like ketone, alcohol or hydrocarbons to the desired concentration and then applied for extraction. 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 known to be added to improve the physical properties of the extractant, such as its solubility and viscosity. They also influence the extractant's extraction power 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 broader extraction range than Alamine 336 as it extracts both dissociated and undissociated forms of the acids. This means that extraction efficiency is independent of pH of aqueous solution (Kar et al., 2017).

The selected extractant Aliquat 336 is a highly viscous (1500 mPa.s) and dense (0.8884 gm/cm3) in nature and hence is to be mixed with selected diluents to increase its solvation capacity. MIBK and xylene diluents were mixed with Aliquat 336 in independent runs and 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.

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

n =1,2 or 3

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

Reactive extraction of AA using Aliquat-336 with diluents MIBK and xylene as an organic phase were experimented at temperatures 303 K, 313K and 323K and Aliquat-336 concentrations of 0.437, 0.547 and 0.729 mol/lit (20, 25 and 33.33% Vol) at atmospheric pressure in independent studies.

3.3 Parameter Study

The parameters such as AA concentration, extractant to diluent ratio and temperature were investigated for optimisation of process.

3.3.1 Effect of Initial sodium acetate or acetic acid concentration

Initial acid/sodium acetate concentrations of 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 the experiments were performed for higher Aliquat 336 concentrations (25 vol% 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/lit the extraction efficiency raised 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 distribution coefficient, K_D 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 interphase increases and strong affinity of Aliquat 336 fetches the acid ions to the organic phase forming the reaction complex.

3.3.2 Effect of extractant to diluent ratio

Extractant to diluent ratio was differed by maintaining other parameters such as initial concentration, and temperature constant for this study. The concentration of the extractant Aliquat 336 was adjusted between 20 - 33.33 vol%. Diluents MIBK and xylene were evaluated for the change in the extraction efficiency in individual experiments. Aliquat 336 a quaternary amine is highly viscous liquid and needs to be diluted with solvents to decrease its viscosity and increase solvation capacity. Literature mentions (Wasewar et al., 2011), 10-30% by volume range of extractant is sufficient to experimentally analyse its feasibility. Higher extractant concentration leads to decrease in extraction efficiency of the extractant. This is mainly due to inability of the extractant to fetch acid ions at the interface, possibly due to co bonding of extractant ions among themselves in highly viscous liquid, Also, higher extractant concentration alters the physical properties of the organic phase leading to third phase formation, which further lowers down the extraction efficiency.

Experiments were conducted for aqueous phase AA concentration from 0.2 to 0.5 mol/lit and 20, 25 and 33.33 vol% (0.437, .547 and 0.729 mol/lit) Aliquat 336 in organic phase at temperatures of 303 K. Table-4, represents effect of variation in acid concentration (0.2-0.5 mol/lit) as well as Aliquat 336 concentration 20, 25 and 33 vol% at constant temperature 303 K. Experimental results revealed that with increase in extractant to diluent concentration, extraction efficiency increases at constant initial acid concentration and temperature. At constant temperature when the concentration of extractant is increased i.e. Aliquat 336 to diluent (MIBK and xylene) ratio is increased from 20 to 25 and then to 33.33 vol%, the experimental results indicate, increase in the extraction efficiency for both the diluents. For higher concentration of Aliquat 336 + MIBK, the extraction efficiency increases and is found in the range of 51-69% for 25 vol % and to 57-73.60% for 33.33 vol% of extractant to diluent ratio. Comparable results were observed for the Aliquat 336 + xylene with 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 33.33 vol% of extractant concentration.

At lower concentration 20% by volume (0.437 mol/lit) of Aliquat 336, extraction efficiency varied between 48 to 64% for MIBK diluent. Selecting extractant concentration below 0.437 mol/lit (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 comparative results of extraction efficiency considering both diluents MIBK and xylene at fixed temperature 303 K and acid concentration of 0.5 mol/lit. From results it can be concluded that MIBK is better diluent than xylene for achieving highest extraction of acid molecules. Higher concentration of Aliquat 336 in MIBK or xylene results in improved 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 dilute [HAA]aq phase together intensify the RE process and forms reaction complex as $[(\text{HAA})_n\text{– Aliquat}_{\text{org}}]$. This necessarily means association of acid ions to extractant, since the value of loading ratio Z<0.5 in all the experiments at 303 K, we can conclude that 1:1 acid: extractant complex is formed. At high initial concentration of acid in aqueous phase (0.5 mol/lit) and for extractant concentration of 0.437 mol/lit it is observed that value of z is 0.581 and goes on decreasing as we increase extractant concentration (z is 0.561 for 0.547mol/lit and z is 0.453 for 0.729 mol/lit of extractant concentration). This suggests that in such situations when initial acid concentration is high in aqueous phase, increase in 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.

$[\text{Aliquat } 336]_{org}$ mol/lit	$[HAA]_o$ Initial mol/lit	MIBK				Xylene			
		$[HAA]_{org}$ mol/lit	K_D	%E	Z	$[HAA]_{org}$ mol/lit	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

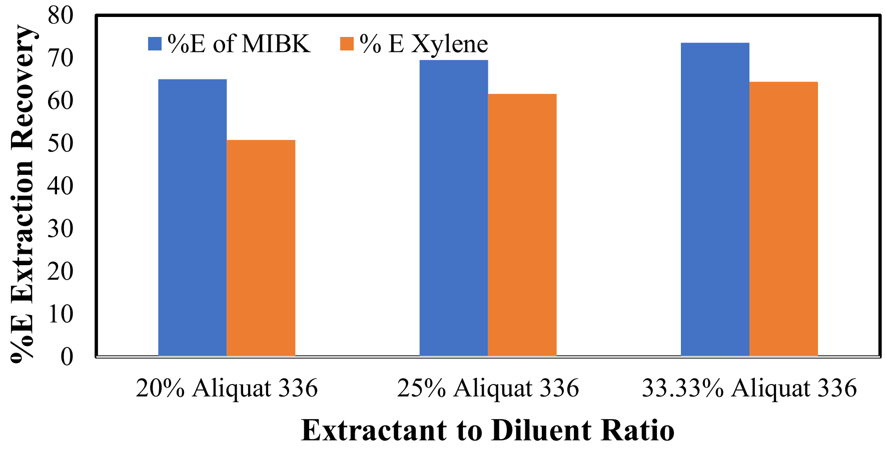
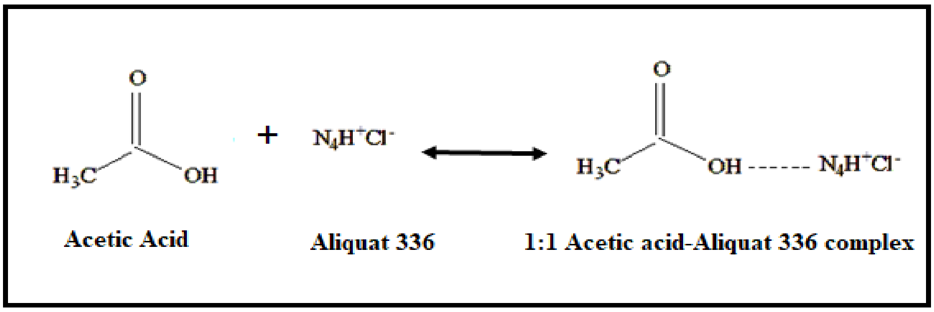


Fig. 2. Extraction efficiency for Aliquat 336: MIBK and Aliquat 336: xylene at concentration range 20, 25 and 33.33 vol% at initial acid concentration of 0.5 mol/lit and 303 K.

3.3.3 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 is 1:1 ($[(HAA) - \text{Aliquat}_{\text{org}}]$), 2:1 ($[(\text{HAA})_2\text{– Aliquat}_{\text{org}}]$) or 3:1 (

$[(HAA)_3 - \text{Aliquat}_{org}]$). Fig. 3 represents the structure of the complexes formed. As experimentally investigated and displayed in Table 4, value of $Z<0.5$ for almost all observations. This indicates 1:1 reaction complex is formed between acetic acid and Aliquat 336 as displayed by reaction in eq (11)



………(11a)

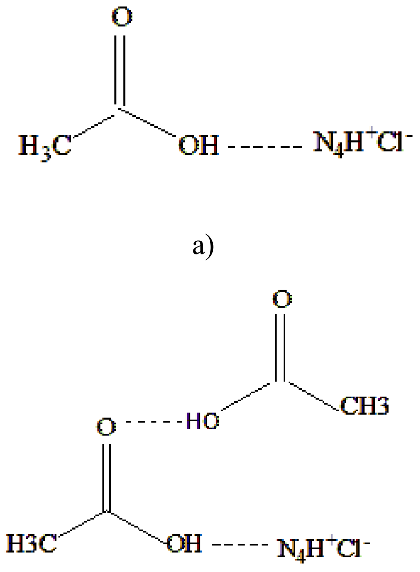
HAA_{aq}+ [Aliquat 336 $]_{org}$ ↔ [Aliquat 336: $HAA]_{org}$ ……………………………………(11b)

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

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

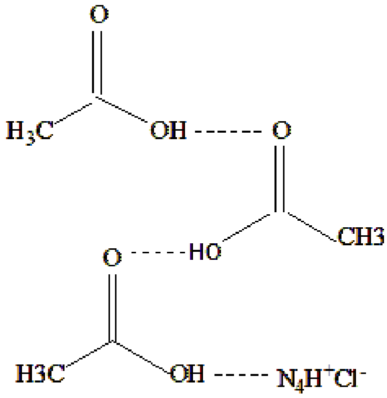
Complexation constant is also estimated from loading ratio values as mentioned in literature (Sharma et al., 2017) and displayed by equation (13)

$$Keq[HAA]_{aq} = \frac{z}{1 - \mathrm{z}} \qquad \text{……………………}(13)$$



a)

b)



c)

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

3.3.4 Effect of rise in temperature

This parameter is important to understand the behaviour of the system at elevated temperatures for extraction and back extraction studies. Most of the process wastewater from fermentation broth is around the room temperature 303K, whereas that of 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 itself is exothermic and is favourable at lower temperature 303K. Studies (Rewatkar et al., 2016; Antony & Wasewar 2020; Demir 2021), mention stable acid-extractant complex at lower or room temperatures resulting in high degree of extraction, whereas high temperature results in back extraction due to reaction at high order and lowers down the extraction efficiency.

Independent batch experiments under identical batch conditions were performed to quantify the impact of temperature on acetic acid reactive extraction with xylene and MIBK diluents. The parameters like initial AA and extractant concentration were also varied in the temperature range of 303, 313 and 323K to estimate extraction efficiency. The experimental findings for 303 K are mentioned in Table 4, whereas the results for 313 K and 323 K are mentioned in Tables 5 and 6 respectively. The results clearly illustrate that, as the temperature of the reacting mixture increases, the distribution coefficient and extraction efficiency also decrease. It is quite visible from the experimental data in Table-4, 5 and 6 and certain that as the temperature increases, percentage extraction decreases. Among the experimental readings at temperature 303 K, 313 K and 323 K, the distribution coefficient and extraction efficiency are found to be lowest at 323K. As the temperature increases reverse phenomenon initiates leading back transfer of acid ions to the aqueous phase. Thus, temperature of the reaction mixture and extraction efficiency are found to be inversely proportional to each other. Fig. 4, reflects the effect of change in temperature (303 to 323 K) on % extraction recovery for initial acid concentration 0.2 mol/lit and 20 vol% of extractant/diluent organic phase. For Aliquat 336-MIBK extractant, % extraction is 49% for 303 K, 48% for 313 K and 47.50 % for 323 K whereas for Aliquat 336-xylene, % extraction is 40% for 303 K, 38.50% for 313 K and 37.00 %. This reduction in extraction efficiency is due to the thermodynamically favoured back-extraction of acid from the organic phase to the aqueous phase at elevated temperatures. This reverse extraction is undesired for AA removal and hence, extraction at room temperature is recommended for most of the reactive extraction processes.

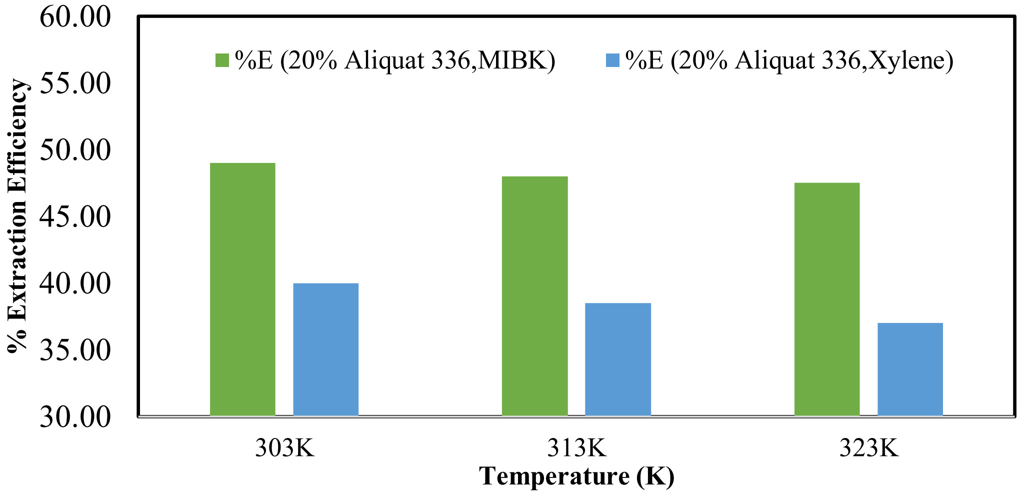


Fig. 4. Effect of change in temperature (303 to 323 K) of 20% Aliquat 336+ (MIBK and xylene) diluents on % Extraction recovery of [HAA]o Initial acid concentration 0.2 mol/lit.

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

[Aliquat 336]$_{org}$ mol/lit	**$[HAA]_o$ Initial mol/lit**	**MIBK**				**Xylene**			
		$[HAA]_{org}$ mol/lit	**K_D**	**%E**	**Z**	**$[HAA]_{org}$ mol/lit**	**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/lit	**$[HAA]_o$ Initial mol/lit**	**MIBK**				**Xylene**			
		$[HAA]_{org}$ mol/lit	**K_D**	**%E**	**Z**	**$[HAA]_{org}$ mol/lit**	**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

Overall observation suggests acid removal from aqueous solution of sodium acetate is possible using reactive extraction technology. From results of 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 extractant i.e. Aliquat 336 in 20-33.33 vol% and their % extraction recovery was studied. Higher initial acid concentration with 33.33 vol% extractant to diluent ratio and 303 K are the optimised parameters for the process. Nevertheless, initial acid concentration cannot be controlled and hence full range from 0.2 to 0.5 mol/lit of initial acid concentration mostly found in industrial wastewater is selected as a base for the study. A comparative graph, (Fig. 5) is displayed for both the organic phases with extractant + MIBK and extractant + xylene at higher extractant concentration 0.729 mol/lit (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, can be concluded that Aliquat 336+ MIBK is suitable organic phase for extraction of acid from aqueous sodium acetate waste.

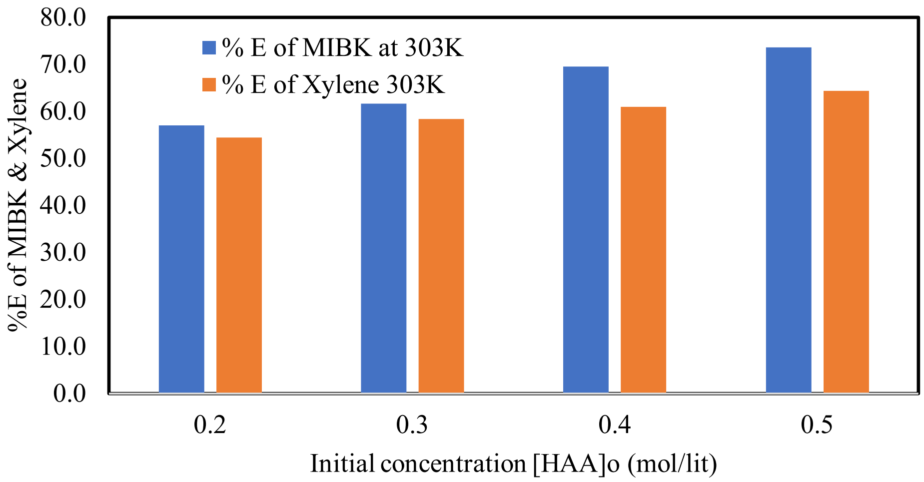


Fig. 5. Comparative extraction efficiency of 0.729 mol/lit of Aliquat 336 +MIBK and Aliquat 336+xylene at 303K.

4. DISCUSSION

The study highlights the efficacy of reactive extraction for acetic acid recovery from sodium acetate waste. MIBK, combined with Aliquat 336, showed superior extraction efficiency (73.6%) compared to xylene (64.4%) under optimal conditions. Earlier study (Juang & Wu 1999) mentions removal of acetate/ acetic acid from mixture of sodium acetate, acetic acid and sodium chloride and calculated efficiency for Aliquat 336 with xylene molar concentration of 0.5 M appears to be approximately 30%. Whereas, higher extraction efficiency was observed for all concentration of acid (0.2-0.5 mol/lit) and extractant (0.437-0.729 mol/lit) between the range 47-72% for MIBK and 37-59% for xylene solvent in current work at comparable temperature and extractant/diluent conditions. Temperature inversely impacted efficiency, suggesting room temperature operations for industrial scalability in this work. These findings demonstrate an energy-efficient and environmentally sustainable alternative for waste treatment applications.

5. CONCLUSION

The recovery of sodium acetate/acetic acid from aqueous stream by the reactive extraction process presented in this study offers a promising approach for acid recovery from the industrial waste containing sodium acetate. The synthetic aqueous sodium acetate was prepared, and extraction study was done to recover the acetic acid formed due to hydrolysis reaction. Physical and reactive extraction experiment were performed, and further parameter study was done to optimise the process.

Current work highlights the following key findings.

- Of the diluents used, MIBK, xylene, toluene, and 1-octanol were shown to be the most appropriate by the physical extraction investigations.
- Synergetic effect was observed for reactive extraction with Aliquat 336 in MIBK and xylene, as it enhanced the recovery of AA as compared to physical extraction. At constant 0.5 mol/lit of acid at 303K, physical extraction experiments demonstrated average extraction efficiency of 60% for MIBK and 48.9% for xylene, whereas employing reactive extraction at same conditions of acid concentration and temperature and maintaining 33.33 vol% Aliquat 336 in MIBK and xylene, extraction efficiency was enhanced and reached 73.60% and 64.40% respectively. Overall, the extraction efficiency increased by 10%.
- Higher initial concentration (0.5 mol/lit) of sodium acetate/acetic acid in aqueous solution, exhibits more interaction of acid-organic molecules and thus increases distribution ratio and extraction efficiency.
- Ration of extractant to diluent affects the extraction process, positive result in extraction efficiency is visible when extractant concentration is increased in organic phase. As the concentration of the extractant grew, so did the distribution coefficient and, therefore, the extraction efficiency. The extraction efficiency was determined to be 65% for 0.437 mol/lit of extractant at a constant temperature of 303 K and an acid concentration of 0.5 mol/lit. 69% for extractant at 0.547 mol/lit and 73% for extractant at 0.739 mol/lit.
- The temperature parameter analysis showed that when the temperature rose, 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 could be scaled up and widely implemented in industrial wastewater treatment and resource recovery applications.

6. PATENTS

As no patents have resulted from the work reported in this manuscript.

Author Contributions: The conceptualization of the research was carried out by Ganesh Bhoj and Shambala Gadekar-Shinde, who contributed significantly to the formulation of the study's objectives and framework. The methodology was developed collaboratively by Ganesh Bhoj and Dr. Shambala Gadekar-Shinde, ensuring a robust research design. Ganesh Bhoj was responsible for the experimentation and analysis of the work. Data curation and validation of the findings were done by Ganesh Bhoj, Sunita Patil, Sharada Gadale and Shambala Gadekar-Shinde. Writing the original draft was done by Ganesh Bhoj, with Sunita Patil, Sharada Gadale and Shambala Gadekar-Shinde responsible for reviewing and editing. Overall supervision of the work was done by Shambala Gadekar-Shinde. All authors have read and approved the manuscript for publication.

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Institutional Review Board Statement: This study did not involve human or animal subjects, nor did it require ethical approval. The work is limited to the reactive extraction of acetic acid from aqueous sodium acetate waste, a chemical and environmental engineering experiment, and does not fall under the purview of ethical review.

Informed Consent Statement: Not applicable. This study did not involve human participants.

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Conflicts of Interest: The authors declare no conflicts of interest. The funders had no role in the design of the study; in the collection, analysis, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

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