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Recovery of 1-Butanol from Chemical Laboratory Waste through Salting-Out, Dehydration, and Distillation

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ABSTRACT (9 pt)

Chemical laboratory activities generate waste streams such as butanol–water mixtures that are potentially recoverable because they still contain residual 1-butanol. In the present work, a simple process was developed to recover 1-butanol from chemical laboratory waste mainly consisting of a 1-butanol–water mixture. The recovery process involved salting-out using either solid NaCl or saturated NaCl solution to enhance liquid–liquid phase separation, followed by separation of the butanol-rich phase using a separatory funnel, dehydration with anhydrous $MgSO_4$, and vacuum-assisted distillation using a rotary evaporator. The recovered fractions were characterized by gas chromatography with flame ionization detection (GC-FID), Fourier-transform infrared spectroscopy (FTIR), Karl Fischer titration, acidity determination, residue on evaporation, and density measurement. The recovered fractions contained 57.536% and 57.156% 1-butanol. The residual water content was 3.345% for solid NaCl and 7.650% for saturated NaCl solution. The acidity, expressed as acetic acid, was 0.00035% and 0.00034%, respectively. Neither recovered fraction had measurable evaporation residue. The recovered fractions had densities of 0.809 and 0.815 for solid NaCl and saturated NaCl solution treatments. Both recovered fractions showed characteristic O–H, C–H, and C–O absorption bands consistent with 1-butanol. Overall, both treatments enabled the recovery of a 1-butanol-rich fraction from laboratory waste; however, solid NaCl produced a fraction with substantially lower residual water content and a density closer to that of 1-butanol. This simple recovery approach demonstrates potential for reducing organic solvent waste and supporting solvent reuse in chemical laboratories.

INTRODUCTION

Laboratory activities are an indispensable part of education and scientific research, but they generate chemical waste which needs to be managed properly (Gonzales & Bronce, 2019; Ramadhani, 2020). Practical work in the area of two-component phase equilibria, for example, in the 1-butanol-water system, is one possible source of liquid waste in chemistry laboratories. The resulting waste may still contain significant amount of 1-butanol which is often treated as chemical waste despite its potential for recovery and reuse. The improper disposal of organic solvent waste may increase the environmental burden of laboratory activities, while recovery can offer the opportunity to reduce waste generation and improve resource efficiency.

1-Butanol (butan-1-ol or n-butanol) is a primary alcohol widely used as an organic solvent and as a raw material in chemical, pharmaceutical, and material-related applications. Although residual 1-butanol requires appropriate handling because of its flammability and potential adverse effects following exposure, its remaining economic and functional value makes recovery preferable to direct disposal. Solvent recovery is also consistent with green chemistry principles that emphasize waste prevention, resource efficiency,

and the reuse of chemicals whenever technically feasible (Kasare, 2025; Ratnani & Bargujar, 2022).

Recovery of 1-butanol from aqueous laboratory waste requires effective reduction of its water content prior to final purification. In this study, a simple multistage process consisting of salting-out, dehydration, and distillation is proposed. Salting-out using NaCl is applied to promote phase separation and obtain a butanol-rich phase (Xie et al., 2017; Yi et al., 2014). Residual water in this fraction is subsequently reduced using anhydrous $MgSO_4$ before the fraction is subjected to vacuum-assisted distillation. This combination is expected to provide a practical recovery approach without requiring complex separation equipment, while improving the quality of the recovered solvent compared with the original laboratory waste. However, most previous studies have focused on model solutions, fermentation-derived systems, or specific equilibrium systems rather than actual waste generated from educational laboratory activities.

The present study therefore focuses on the application of these established separation principles to actual 1-butanol–water waste generated from a physical chemistry laboratory. The originality of the present work lies not in introducing a new separation principle, but in integrating salting-out, $MgSO_4$ dehydration, and vacuum-assisted distillation at laboratory scale and directly comparing two modes of NaCl addition, namely solid NaCl and saturated NaCl solution. The recovered fractions were evaluated based on 1-butanol content, residual water content, FTIR characteristics, acidity, residue on evaporation, and relative density. The study aims to provide preliminary evidence for the feasibility and limitations of a simple solvent-recovery strategy applicable to educational laboratories.

RESEARCH METHOD

Salting-Out and Phase Separation

A volume of 100 mL of homogenized 1-butanol-water laboratory waste was used for each treatment. Two different methods of NaCl addition were investigated. 2. Treatment with saturated NaCl solution (T1) 25 mL of saturated NaCl solution was added to 100 mL of laboratory waste. The mixture was stirred/shaken for 30 min and then transferred into a separatory funnel. The mixture was allowed to stand until the two liquid phases were formed. Solid NaCl treatment (T2) 9 g of solid NaCl was added slowly directly to 100 mL of the lab waste with continuous stirring. The mixture was stirred for 30 min and then transferred into a separatory funnel and allowed to stand min for separation of phases. After phase separation, the butanol-rich phase was gently collected and its volume was determined. The collected fraction was subsequently subjected to the dehydration stage.

Dehydration

The butanol-rich fraction from each salting-out treatment was dried with anhydrous $MgSO_4$. Anhydrous $MgSO_4$ (10 g per 50 mL of the butanol-rich fraction) was added and mixed at room temperature for 30 min. Then the mixture was allowed to stand for 10 min for the removal of water. The MgSO4 was subsequently removed by filtration, and the resulting dried butanol-rich fraction was collected for the distillation process.

Vacuum-Assisted Distillation

The dehydrated fraction enriched in butanol was vacuum distilled in a rotary evaporator. The distillation was carried out at a water-bath temperature of 60 ℃, a pressure of 25 mbar and a rotation speed of 75 rpm. The condensed volatile fraction was collected in the receiving flask and was called the recovered 1-butanol fraction. The volume and/or mass of the recovered fraction was recorded for each treatment prior to characterization. The mean of the process resulted around 63 mL from 100 mL 1-butanol-water waste.

Determination of 1-Butanol Content by GC-FID

The 1-butanol content of the recovered fractions was determined by gas chromatography with flame ionization detection (GC-FID). 1-Butanol analytical grade was used as reference standard. For the calibration range, covering the expected concentration of the samples, a series of 1-butanol standard solutions were prepared by appropriate dilution with methanol. 1-butanol was accurately weighed and diluted with methanol to prepare the concentrations within the calibration range. A volume of 1 µL of each standard and sample solution was injected into the GC-FID system with a suitable capillary column for the separation of low molecular weight alcohols. The carrier gas was helium. Temperatures of the injector and detector were set to 250 °C and oven temperatures were held at 45 °C for 5 min, then heated at 10 °C/min to 80 °C then heated at 20 °C/min to 220 °C where it was held for 10 min. The peak of 1-butanol was identified by comparing the retention time of the sample with the retention time of analytical grade 1-butanol standard. The peak areas obtained from the standard solutions were used to construct a calibration curve, and the 1-butanol concentration in each recovered fraction was calculated from the corresponding regression equation and expressed as percentage (% w/w). The GC-FID analysis was performed in duplicate (n = 2), and the results were expressed as mean ± standard deviation.

FTIR Analysis

The chemical characteristics of the recovered 1-butanol fractions were evaluated using Fourier-transform infrared (FTIR) spectroscopy. The spectra were recorded over the wavenumber range of 4000-400 cm^{-1}. Analytical-grade 1-butanol was analyzed as a reference and compared with the recovered fractions obtained from the solid NaCl and saturated NaCl treatments. The spectra were evaluated based on the characteristic absorption bands associated with O–H stretching, aliphatic C–H stretching and bending, and C–O stretching. The FTIR analysis was used to support the identification of characteristic functional groups of 1-butanol in the recovered fractions rather than to determine their purity.

Determination of Water Content by Karl Fischer Titration

Approximately 1.0 g of the recovered 1-butanol sample was accurately weighed and immediately introduced into the conditioned titration vessel. The sample was mixed thoroughly and titrated with standardized Karl Fischer reagent under continuous stirring until a stable electrometric endpoint was reached. The volume of Karl Fischer reagent consumed was recorded, and the water content was calculated from the reagent consumption and its standardized water equivalence factor and expressed as % w/w.

The determination of water content by Karl Fischer titration was performed in duplicate (n = 2), and the results were expressed as mean ± standard deviation.

Determination of Acidity

The acidity of the recovered 1-butanol was determined by a titrimetric method. A volume of 74 mL of the sample (approximately 60 g) was transferred into a suitable flask and titrated with 0.020 N alcoholic potassium hydroxide (KOH) using phenolphthalein as the indicator. The titration was continued until a pink color appeared and persisted for not less than 15 s. The acidity was expressed as percentage of acetic acid. The acidity determination was performed in triplicate (n = 3), and the results were expressed as mean ± standard deviation.

Determination of Residue on Evaporation

The recovered 1-butanol sample (100 mL) was accurately measured and transferred into a previously dried and weighed porcelain dish. The sample was evaporated on a steam bath to remove the volatile components. The dish containing the remaining residue was subsequently dried at 105 °C for 30 min, cooled under conditions that minimized moisture uptake, and weighed. The amount of nonvolatile residue was determined gravimetrically and expressed as percentage residue on evaporation. The determination was performed in triplicate (n = 3), and the results were expressed as mean ± standard deviation.

Determination of Relative Density

Relative density was determined using a pycnometer. The clean and dry pycnometer was first weighed empty, then filled with distilled water to the calibration mark and weighed again. The same procedure was subsequently performed using the recovered 1-butanol sample. Relative density was calculated from the ratio of the mass of the sample to the mass of an equal volume of distilled water at the same temperature. The measurement was performed in triplicate (n = 3), and the results were expressed as mean ± standard deviation.

RESULTS AND DISCUSSION

Waste 1-butanol is an organic solvent waste that still has the potential to be recovered through separation and purification processes. In this study, the waste was generated from a two-phase equilibrium experiment involving the 1-butanol–water system conducted in the Physical Chemistry Laboratory of Universitas Negeri Surabaya. The recovery process consisted of three main stages, namely salting-out, dehydration, and distillation, with the aim of recovering the 1-butanol fraction and improving its quality prior to characterization.

The first stage was salting-out, which was intended to improve the separation of 1-butanol from the aqueous phase. The addition of NaCl increases the ionic strength of the aqueous phase because the Na^+ and Cl^- ions interact strongly with water molecules through hydration. These interactions decrease the availability of water molecules to solvate 1-butanol and decrease the solubility of 1-butanol in the aqueous phase.

Therefore, 1-butanol molecules are prone to migrate and concentrate in the butanol-rich phase, while NaCl is mainly distributed in the water-rich phase (Fu et al., 2020). This phenomenon is called the salting-out effect and it results in a better separation of the two liquid phases. The process is mainly a physical equilibrium phenomenon and does not involve the formation of new chemical compounds. Increased ion hydration decreases the relative solubility of 1-butanol in the aqueous phase and favours the formation of a butanol-rich phase(García-Cano et al., 2016). The two phases were separated using a separatory funnel after complete phase separation and the butanol-rich phase was collected for the subsequent treatment.

The second stage was dehydration, which was performed to decrease the residual water left in the butanol-rich phase after salting-out. Anhydrous magnesium sulphate ($MgSO_4$) was employed as a drying agent because of its hygroscopic properties and its capacity to combine with water molecules by forming magnesium sulphate hydrates (Williams & Lawton, 2010). At this stage, compared with the salting-out process, anhydrous $MgSO_4$ directly binds the residual water present in the organic phase. The hydration process can be expressed in simplified form as:

$$MgSO_4 + nH_2O \rightarrow MgSO_4 \cdot nH_2O$$

Hydrated magnesium sulfate formation reduces the water content of the butanol-rich phase. The $MgSO_4$ and its hydrated forms were then filtered off leaving a drier butanol-rich fraction. This dehydration is important, as residual water may impact on 1-butanol purity and efficiency of the subsequent separation process.

The last step was vacuum distillation using a rotary evaporator. Reducing the pressure will allow the vapour pressure of the liquid to equal the surrounding pressure at a temperature lower than the normal boiling point . That is why volatile components can be evaporated at temperatures lower than their boiling points at atmospheric pressure . The volatile components from butanol-rich fraction evaporated from the evaporation flask, passed through the condenser, and condensed and collected in the receiving flask as the distillate fraction (Zweckmair et al., 2017). The 1-butanol fractions from the two treatments were characterised for 1-butanol content by GC-FID, functional-group identification by FTIR spectroscopy, water content by Karl Fischer titration, acidity determination, Residue on evaporation and density.

The physicochemical characteristics of the recovered fractions were evaluated by measuring the 1-butanol content and other quality parameters. Table 1 shows the results for some quality parameters, such as 1-butanol content, water content, total acidity expressed as acetic acid, residue on evaporation and density. The contents of 1-butanol were 57.536% and 57.156% in the case of solid NaCl and saturated NaCl solution, respectively, showing only a minor descriptive difference. Conversely, a substantial difference in water content was observed. The fraction obtained from solid NaCl contained 3.345% water, whereas the fraction obtained from saturated NaCl solution contained 7.650%. The values of total acidity expressed as acetic acid were nearly the same for both the treatments, that is 0.00035% for solid NaCl and 0.00034% for the saturated NaCl solution. Both treatments resulted in 0% residue on evaporation, indicating that no measurable nonvolatile residue was left under the test conditions

applied. Also a slight difference was observed in the density with values of 0.809 and 0.815 for solid NaCl treatment and saturated NaCl solution treatment respectively.

Table 1. Characterization Result of Purified 1-Butanol Fraction

Parameter	Variations	
	Solid NaCl	**Saturated NaCl**
1-butanol content (%)	57.536 ± 0.2687	57.156 ± 0.1874
Water content (%)	3.345 ± 0.0212	7.650 ± 0.1131
Acidity (%Acetic acid)	0.00035 ± 0.000014	0.00034 ± 0.0000
Residue of Evaporation (%)	0.00 ± 0.0000	0.00 ± 0.0000
Relative Density	0.809 ± 0.000006	0.815 ± 0.00002

The recovered fractions contained 57.536% 1-butanol for the solid NaCl treatment and 57.156% for the saturated NaCl solution treatment, indicating only a minor descriptive difference between the two salting-out approaches. Liquid-liquid equilibrium studies have previously shown that inorganic salts can promote the transfer of 1-butanol from aqueous phase to butanol-rich phase. Chawong et al. (2017) found that inorganic salts including NaCl reduce the mutual solubility of water and 1-butanol and facilitate phase separation, with more pronounced effects at higher salt concentrations (Chawong et al., 2017). Similarly, Fu et al. (2020) demonstrated that salting-out can concentrate 1-butanol in the organic phase while transferring most of the water into the aqueous phase, but their highest enrichment was obtained using highly soluble potassium salts instead of NaCl (Fu et al., 2020). The relatively moderate 1-butanol contents obtained in the present study therefore suggest that NaCl-assisted salting-out followed by $MgSO_4$ dehydration and vacuum distillation was sufficient for solvent recovery but not for high-purity 1-butanol under the conditions employed. This does not necessarily conflict with previous studies as the efficiency of salting-out is highly dependent on the salt species, salt concentration, feed composition and separation conditions. The similar values obtained for solid and saturated NaCl treatment also indicate that the form of NaCl addition had a limited effect on the final chromatographically determined 1-butanol content. Chromatographic characterisation is of particular importance in solvent recovery studies because the recovered solvent may contain other volatile components that cannot be distinguished from water content only, and previous laboratory-scale solvent recycling studies have used GC-based analysis in combination with complementary physicochemical tests to assess the quality of the recovered solvent.

The difference between the two treatments for the residual water content was much larger than that for the 1-butanol content. The recovered fraction using solid NaCl contained 3.345% water and the saturated NaCl solution treatment contained 7.650% water. This trend agrees with the basic salting-out behaviour of the water-1-butanol system. Experimental liquid-liquid equilibrium studies have demonstrated that the addition of inorganic salts decreases the mutual solubility of 1-butanol and water and

promotes the formation of separate water-rich and butanol-rich phases. In addition, Fu et al. (2020) demonstrated that salting-out can facilitate the migration of water from the 1-butanol-rich phase to the aqueous phase, thus improving phase separation and dehydration simultaneously (Fu et al., 2020). The considerably lower water content achieved with solid NaCl in this work can reasonably be explained by the direct addition of salt without the introduction of an additional aqueous volume, whereas the saturated NaCl treatment inherently brings water along with the dissolved salt. This interpretation is specific to the present experimental design and should therefore be considered together with the following step of $MgSO_4$ dehydration. Although anhydrous $MgSO_4$ reduced residual moisture, the dehydration efficiency was not improved to fully anhydrous 1-butanol, indicating that the optimisation of salt loading, phase separation, drying-agent dosage or drying time may further improve the dehydration efficiency.

The acidity of the recovered fractions was very low, corresponding to 0.00035% acetic acid for the solid NaCl treatment and 0.00034% for the saturated NaCl solution treatment. The nearly identical values indicate that the method of NaCl addition did not substantially influence the amount of titratable acidic species present in the final recovered fractions. Acidity is an important quality parameter for recovered alcohol solvents because primary alcohols may undergo oxidation to aldehydes and subsequently to the corresponding carboxylic acids under sufficiently oxidative conditions. Previous mechanistic studies on 1-butanol oxidation have demonstrated the formation of butyric acid as an oxidation product, confirming that acidic degradation products are chemically possible for this alcohol (Zigová et al., 2000). Nevertheless, the very low acidity observed in the present recovered fractions suggests that appreciable accumulation of titratable acidic impurities did not occur during the recovery process (Oudshoorn et al., 2011). The similarity between both treatments further indicates that salting-out using either solid or saturated NaCl did not promote appreciable acid formation under the applied conditions.

In both recovered fractions no detectable residuals on evaporation were observed, with both treatments producing a value of 0.00%. This result indicates that the nonvolatile constituents were not detectable at the sensitivity of the gravimetric procedure used. This result is especially relevant as the recovery process involved the addition of inorganic salts and anhydrous $MgSO_4$, which may result in contamination of the recovered product in the case of inefficient phase separation or filtration. The lack of measurable residue suggests that the steps of separation, filtration and subsequent distillation were successful in minimising the carry-over of nonvolatile inorganic materials into the condensed fraction. Distillation has been successfully used for laboratory solvent recycling because volatile solvents can be separated from nonvolatile or less volatile waste components, and the recovered solvents can then be brought to a sufficient quality for reuse when appropriately controlled and characterised. For instance, Zweckmair et al. (2017) showed laboratory-scale recovery of analytical solvents by controlled distillation and highlighted the necessity of analytical verification of the recycled solvent before reuse (Zweckmair et al., 2017).

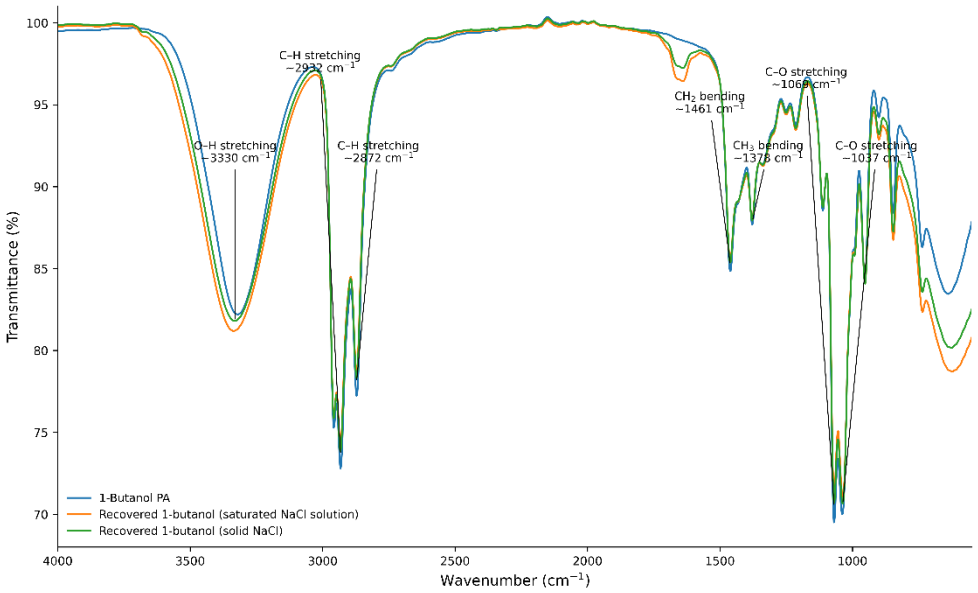


Figure 1. FTIR spectra of analytical-grade 1-butanol and the recovered 1-butanol-rich fractions.

The FTIR spectra also showed that the major chemical features of both recovered fractions were similar to that of 1-butanol. The analytical grade reference exhibited characteristic bands at 3321.05, 2932.07, 2871.33, 1461.08, 1378.09, 1069.28 and 1038.30 cm^{-1}. Bands similar to the saturated NaCl treatment were observed at 3336.37, 2932.88, 2871.92, 1461.33, 1378.80, 1069.22, and 1036.53 cm^{-1}, and for the solid NaCl treatment at 3333.61, 2932.66, 2871.76, 1461.26, 1378.62, 1069.20, and 1036.87 cm^{-1}. These assignments are firmly supported by previous spectroscopic work on 1-butanol. Myers et al. (2022) observed a broad O-H stretching absorption at approximately 3329 cm^{-1}, C-H stretching bands at around 2950-2850 cm^{-1}, and prominent C-O/C-C stretching features within the 1080-1025 cm^{-1} region (Myers et al., 2022). The good agreement between these literature bands, analytical-grade 1-butanol, and both recovered samples supports the presence of the characteristic functional groups of 1-butanol in the recovered fractions. The small shift of the O-H band from 3321.05 cm^{-1} in the analytical-grade 1-butanol to 3333.61-3336.37 cm^{-1} in the recovered fractions may indicate differences in the hydrogen-bonding environment, which is sensitive to intermolecular interactions and residual water.

CONCLUSION

This work demonstrated the recovery of a 1-butanol-rich fraction from laboratory-generated 1-butanol–water waste through sequential salting-out, dehydration with anhydrous $MgSO_4$, and vacuum-assisted distillation. The recovered fractions obtained using solid NaCl and saturated NaCl solution contained 57.536% and 57.156% 1-butanol, respectively, while the residual water content was lower in the solid NaCl treatment (3.345%) than in the saturated NaCl solution treatment (7.650%). Both treatments showed very low acidity, no measurable residue on evaporation, and comparable relative density

values. FTIR analysis supported the presence of the characteristic functional groups of 1-butanol in both recovered fractions. Overall, the process produced a 1-butanol-rich fraction rather than high-purity 1-butanol, and further optimization of the separation, dehydration, and distillation conditions is required before the recovered solvent can be considered for routine reuse.

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