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Comparative Study of Separation Techniques for Multi-Component Organic-Inorganic Mixtures Using Extraction, Distillation, and Evaporation

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Abstract

The separation and purification of chemical compounds from multi-component mixtures is a fundamental process in chemistry and pharmaceutical sciences. This study evaluated the effectiveness of liquid-liquid extraction, simple distillation, evaporation, recrystallization, and acid-base extraction techniques for separating four different ternary mixtures: (i) chloroform, methanol, and sodium acetate; (ii) salicylic acid, diethyl ether, and sodium chloride; (iii) benzoic acid, distilled water, and diethyl ether; and (iv) salicylic acid, ethanol, and distilled water. Each mixture was systematically separated using appropriate techniques based on differences in polarity, boiling point, and solubility. The results demonstrated that the percent error values varied considerably across different compounds and methods, ranging from 2.30% to 93.60%. Recrystallization and distillation showed higher accuracy for non-volatile solids and low-boiling-point liquids, respectively, while liquid-liquid extraction efficiency was influenced by polarity differences and procedural factors. These findings provide a comprehensive comparison of classical separation techniques applicable to pharmaceutical and chemical laboratory practice.

Keywords: separation techniques, liquid-liquid extraction, simple distillation, recrystallization, percent error.

Abstrak

Pemisahan dan pemurnian senyawa kimia dari campuran multi-komponen merupakan proses fundamental dalam ilmu kimia dan farmasi. Penelitian ini mengevaluasi efektivitas teknik ekstraksi cair-cair, destilasi sederhana, penguapan, rekristalisasi, dan ekstraksi asam-basa untuk memisahkan empat campuran terner yang berbeda: (i) kloroform, metanol, dan natrium asetat; (ii) asam salisilat, dietil eter, dan natrium klorida; (iii) asam benzoat, akuades, dan dietil eter; dan (iv) asam salisilat, etanol, dan akuades. Setiap campuran dipisahkan secara sistematis menggunakan teknik yang sesuai berdasarkan perbedaan polaritas, titik didih, dan kelarutan. Hasil menunjukkan bahwa nilai persen ralat bervariasi secara signifikan antara senyawa dan metode yang berbeda, berkisar dari 2,30% hingga 93,60%. Rekristalisasi dan destilasi menunjukkan akurasi lebih tinggi untuk padatan non-volatil dan cairan bertitik didih rendah, sedangkan efisiensi ekstraksi cair-cair dipengaruhi oleh perbedaan polaritas dan faktor prosedural. Temuan ini memberikan perbandingan komprehensif teknik pemisahan klasik yang dapat diterapkan dalam praktik laboratorium farmasi dan kimia.

Kata kunci: *teknik pemisahan, ekstraksi cair-cair, destilasi sederhana, rekristalisasi, persen ralat*

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INTRODUCTION

Purification and separation of chemical compounds from complex mixtures represent fundamental processes in chemistry and pharmaceutical sciences. In most chemical reactions, the desired product is rarely obtained in a pure state; instead, it is accompanied by by-products, unreacted starting materials, solvents, and catalysts. Therefore, effective separation and purification methods are essential for obtaining pure substances suitable for characterization, analysis, and application. Common separation techniques include extraction, filtration, centrifugation, recrystallization, distillation, and sublimation, each exploiting specific physical or chemical properties of the mixture components.

Liquid-liquid extraction is one of the most widely employed separation techniques in both analytical and preparative chemistry. This method operates on the principle of differential solubility, where a solute distributes itself between two immiscible solvents according to its partition coefficient. The technique is particularly effective for separating compounds with significantly different polarities, as governed by the fundamental principle of *like dissolves like*. In a separating funnel, a mixture of two immiscible phases is vigorously shaken and then allowed to settle, forming distinct layers that can be separated by controlling the stopcock.[1,2]

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Simple distillation is another classical separation method based on differences in boiling points between mixture components. In this process, a liquid mixture is heated until the more volatile component evaporates preferentially. The vapor is then condensed through a Liebig condenser and collected as distillate, while the less volatile components remain in the distillation flask as residue. This technique is particularly effective when the boiling point difference between components exceeds 25°C.[3,4]

Evaporation and recrystallization are complementary techniques frequently used for isolating solid compounds from solutions. Evaporation involves the removal of solvent through heating, leaving behind the dissolved solute in solid form. The process is influenced by temperature, pressure, solvent type, and the chemical properties of the dissolved substance. Recrystallization, on the other hand, operates on the principle of differential solubility at different temperatures, enabling the purification of a solid by dissolving it in a hot solvent and allowing it to crystallize upon cooling.[5,6]

Acid-base extraction is a specialized form of liquid-liquid extraction that takes advantage of the ionizable nature of acidic or basic compounds. By adjusting the pH of the aqueous phase, organic acids or bases can be converted to their ionized forms, which are preferentially soluble in the aqueous phase, thereby facilitating their separation from neutral organic compounds.[7]

Several recent studies have investigated the optimization of these classical separation techniques. Chen *et al.* (2019) reviewed the fundamental principles and applications of liquid-liquid extraction, emphasizing the role of solvent selection and phase equilibrium in determining extraction efficiency.[8] Silva and Rodrigues (2019) reported optimization parameters for liquid-liquid extraction to achieve improved analytical efficiency, noting that factors such as solvent ratio, contact time, and agitation speed significantly influenced recovery rates.[9] Tshepelevitsh *et al.* (2017) demonstrated a systematic approach to optimizing liquid-liquid extraction parameters for isolating unknown components, confirming that the physicochemical descriptors of solutes—including polarity, hydrogen bonding capacity, polarizability, and molar volume—are critical determinants of partition behavior.[10]

However, despite the well-established theoretical basis of these methods, their practical application often results in significant deviations from theoretical yields, particularly in educational laboratory settings. Factors such as temperature control, agitation technique, apparatus handling, and compound volatility contribute to variable results. Furthermore, few studies have systematically compared the effectiveness of multiple separation techniques applied to different multi-component mixtures within a single comparative framework.

Therefore, this study aimed to evaluate and compare the effectiveness of liquid-liquid extraction, simple distillation, evaporation, recrystallization, and acid-base extraction for separating four different ternary mixtures: (i) chloroform, methanol, and sodium acetate; (ii) salicylic acid, diethyl ether, and sodium chloride; (iii) benzoic acid, distilled water, and diethyl ether; and (iv) salicylic acid, ethanol, and distilled water. The results are expected to provide a comprehensive comparison of these classical separation techniques and identify the key factors influencing their efficiency.

METHODOLOGY

Equipment and Materials

The equipment used across all four experiments included separating funnels (Iwaki Asahi Glass, 500 mL), round-bottom flasks (Pyrex Glass, 500 mL), Liebig condensers (Gratech), heating mantles (Joanlab), evaporating dishes, analytical balances (Fujitsu), graduated cylinders (Iwaki, 100 mL), Erlenmeyer flasks (Iwaki Pyrex, 250 mL), beaker glasses (Iwaki, 250 mL), thermometers (110°C), filter paper, ring stands with clamps, water baths (Faithful), pipettes, and aluminum foil. Additional equipment for specific experiments included hot plates for recrystallization processes and ice baths for crystallization.

The chemical materials used varied across experiments and included chloroform ($CHCl_3$), methanol (CH_3OH), sodium acetate (CH_3COONa), salicylic acid ($C_7H_6O_3$), diethyl ether ($(C_2H_5)_2O$), sodium chloride (NaCl), benzoic acid (C_6H_5COOH), ethanol (C_2H_5OH), sodium hydroxide (NaOH), hydrochloric acid (HCl), ferric chloride ($FeCl_3$), and distilled water (aquadest). All chemicals were of analytical grade.

Experiment I: Separation of Chloroform, Methanol, and Sodium Acetate

A 100 mL sample containing a mixture of chloroform, methanol, and sodium acetate was transferred into a separating funnel. Subsequently, 50 mL of distilled water was added as a polar solvent. The mixture was shaken vigorously in a horizontal position with periodic venting of gas pressure through the stopcock. After shaking, the mixture was allowed to stand until two distinct layers formed. The lower layer (chloroform) was collected through the stopcock into a vial. The upper layer containing methanol, sodium acetate, and aqueous solution was transferred to a round-bottom flask for simple distillation at 65°C. The distillate (methanol) was collected in an Erlenmeyer flask. The remaining residue containing sodium acetate in aqueous solution was transferred to an evaporating dish and heated on a water bath until all water evaporated, yielding solid sodium acetate.[1,3]

Experiment II: Separation of Salicylic Acid, Diethyl Ether, and Sodium Chloride

The mixture of salicylic acid, diethyl ether, and sodium chloride was subjected to liquid-liquid extraction using a separating funnel with distilled water as the polar solvent. After phase separation, the lower aqueous layer containing NaCl was collected and subjected to recrystallization by evaporation on a water bath at 100°C. The upper layer containing salicylic acid and ether was mixed with ethanol to bind the salicylic acid, then subjected to simple distillation at 36°C to separate ether as the distillate. The residue containing salicylic acid and ethanol was further heated on a hot plate at 78°C to evaporate the ethanol, yielding purified salicylic acid crystals.[2,11]

Experiment III: Separation of Benzoic Acid, Distilled Water, and Diethyl Ether

ternary mixture was first separated by liquid-liquid extraction in a separating funnel, yielding an upper layer (ether and benzoic acid) and a lower layer (distilled water). The upper layer was then subjected to acid-base extraction by adding 5 mL of NaOH solution to convert benzoic acid to sodium benzoate (water-soluble). After phase separation, the ether layer was collected. The aqueous sodium benzoate solution was treated with concentrated HCl to regenerate benzoic acid, which was then purified by recrystallization using an ice bath. The crystals were filtered, dried in an oven, and weighed.[7,12]

Experiment IV: Separation of Salicylic Acid, Ethanol, and Distilled Water

The mixture was first subjected to simple distillation at 78°C to separate ethanol (boiling point 78°C) from the aqueous solution containing salicylic acid and distilled water. The distillate (ethanol) was collected. The residual mixture of salicylic acid and distilled water was then separated by liquid-liquid

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extraction using diethyl ether as the extraction solvent in a separating funnel. After phase separation, salicylic acid dissolved in the ether layer (upper) was collected, and the aqueous layer (lower) was tested with $FeCl_3$ reagent to confirm the absence of salicylic acid residue.[13,14]

Percent Error Calculation

The percent error for each separated compound was calculated using the following formula:

$$\%Error = \left|\frac{(Theoretical\ value - Practical\ Value)}{Theoretical\ value}\right| x\ 100\%$$

RESULT AND DISCUSSION

Separation Results

The results of separation experiments for all four ternary mixtures are summarized in **Table 1**. The data present the theoretical values, practical values obtained, and calculated percent error for each compound separated across the four experiment.

Table 1. Summary of separation results for all four ternary mixtures

Exp.	Compound	Theoretical Value	Practical Value	% Error	Method
I	Chloroform	40 mL	20 mL	50.00%	LLE
	Methanol	60 mL	21 mL	65.00%	SD
	Sodium Acetate	2.10 g	1.75 g	16.66%	Evaporation
II	Salicylic Acid	2.125 g	1.86 g	12.47%	Recrystallization
	Diethyl Ether	100 mL	52 mL	48.00%	SD
	Sodium Chloride	3.040 g	2.97 g	2.30%	Recrystallization
III	Distilled Water	40 mL	36 mL	10.00%	LLE
	Benzoic Acid	2.030 g	0.13 g	93.60%	ABE + Recryst
	Diethyl Ether	60 mL	16.2 mL	73.00%	ABE
IV	Distilled Water	40 mL	17.5 mL	56.25%	LLE
	Salicylic Acid	2.048 g	1.90 g	7.22%	LLE
	Ethanol	60 mL	62 mL	3.30%	SD

Note: LLE = Liquid-Liquid Extraction; SD = Simple Distillation; ABE = Acid-Base Extraction; Recryst. = Recrystallization

Experiment I: Chloroform, Methanol, and Sodium Acetate

The separation of the ternary mixture of chloroform, methanol, and sodium acetate was accomplished through a sequential application of liquid-liquid extraction, simple distillation, and evaporation. The liquid-liquid extraction step utilized distilled water as a polar solvent in a separating funnel. Upon addition of water, two immiscible layers formed: the lower layer consisting of chloroform (density 1.49 g/mL), which is denser than water, and the upper layer containing methanol, sodium acetate, and distilled water. The chloroform was collected with a yield of 20 mL from a theoretical volume of 40 mL, resulting in a 50% error. This significant deviation can be attributed to several factors, including incomplete phase separation, loss of chloroform vapor during the venting of pressure buildup, and the inherent difficulty in achieving quantitative separation at the liquid-liquid interface.[1,15]

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The methanol separation by simple distillation yielded 21 mL from a theoretical 60 mL, corresponding to a 65% error. This high deviation can be attributed to the co-evaporation of methanol with water during extraction (due to its volatility), incomplete distillation, and temperature fluctuations during the process. As noted by McCabe et al. (2005), distillation efficiency is significantly affected by the type of solution, temperature, distillation time, and pressure.[3] The sodium acetate recovery by evaporation yielded 1.75 g from a theoretical 2.10 g (16.66% error), demonstrating relatively higher efficiency. Evaporation is generally effective for non-volatile ionic compounds, though losses may occur due to spattering, incomplete solvent removal, or thermal decomposition.[5]

Experiment II: Salicylic Acid, Ether, and Sodium Chloride

Experiment II demonstrated that recrystallization was particularly effective for recovering ionic solids, as evidenced by the remarkably low percent error of 2.30% for sodium chloride. The NaCl was separated from the aqueous layer by recrystallization on a water bath at 100°C, where crystal formation was favored by the gradual concentration of the solution. The factors influencing crystallization rate include the degree of supersaturation, the number and surface area of existing crystal nuclei, solution viscosity, impurity type and concentration, and the relative movement between solution and crystals.[6,16]

The ether recovery by distillation yielded 52 mL from a theoretical 100 mL (48% error). Ether is highly volatile (boiling point ~35°C) and lacks hydrogen bonding capability, which makes it prone to evaporative losses during handling. Unlike alcohols, ethers cannot form hydrogen bonds because they lack hydroxyl groups, resulting in lower boiling points similar to alkanes of comparable molecular mass rather than the corresponding alcohols.[17] Salicylic acid was recovered with a mass of 1.86 g from a theoretical 2.125 g (12.47% error). The loss was attributed to carry-over during distillation, where small amounts of salicylic acid may have been entrained in the vapor phase, and to incomplete recrystallization.[11]

Experiment III: Benzoic Acid, Distilled Water, and Ether

The separation of the benzoic acid mixture revealed the most challenging results, with the benzoic acid recovery showing a remarkably high error of 93.60% (only 0.13 g recovered from a theoretical 2.030 g). This extreme loss can be attributed to multiple factors inherent in the acid-base extraction process. The conversion of benzoic acid to sodium benzoate required precise pH adjustment with NaOH. Insufficient basification may have left a significant portion of benzoic acid in its non-ionized form, which remained dissolved in the organic phase rather than migrating to the aqueous phase.[7,12]

Furthermore, the temperature of the ice bath used for recrystallization may have been suboptimal. Muhammad *et al.* (2020) demonstrated that benzoic acid solubility increases with temperature, meaning that inadequate cooling would prevent effective crystal formation.[18] The ether recovery also showed a high error of 73%, attributable to its extreme volatility and the oxidation process catalyzed by hydroxide ions from NaOH, which accelerated peroxide formation and additional volume loss.[19] The distilled water recovery was the most accurate in this experiment (10% error), consistent with the relatively straightforward nature of aqueous phase separation in liquid-liquid extraction, though some loss was attributed to incomplete phase separation and back-diffusion.[10]

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Experiment IV: Salicylic Acid, Ethanol, and Distilled Water

Experiment IV yielded the most favorable overall results. Ethanol was separated by simple distillation with a remarkably low percent error of 3.30% (62 mL recovered from a theoretical 60 mL). The slightly higher practical value suggests possible co-distillation of a small amount of water, as the distillation temperature may have briefly exceeded the target of 78°C. Salicylic acid was recovered by liquid-liquid extraction with diethyl ether, yielding 1.90 g from a theoretical 2.048 g (7.22% error), demonstrating effective partitioning of salicylic acid into the organic phase due to its favorable solubility in ether.[13,14]

The distilled water recovery showed a 56.25% error (17.5 mL from 40 mL), which was primarily caused by uncontrolled temperature during distillation. The temperature briefly reached 95°C, causing significant water co-evaporation with ethanol. This finding underscores the critical importance of precise temperature control during distillation, as emphasized by Luyben (2006).[20] The qualitative analysis using $FeCl_3$ reagent confirmed the absence of salicylic acid in the recovered distilled water, as the solution remained yellow rather than turning purple-blue, indicating successful separation.[21]

Comparative Analysis

Across all four experiments, the percent error values varied considerably, reflecting the inherent challenges of each separation method and the specific physicochemical properties of the target compounds. The lowest percent errors were achieved for the recovery of non-volatile ionic solids (NaCl, 2.30%) and compounds with well-defined boiling point differences (ethanol, 3.30%). Conversely, the highest errors were observed for highly volatile compounds (diethyl ether) and compounds requiring multi-step processes (benzoic acid via acid-base extraction). These results demonstrate that the selection of an appropriate separation technique must consider the volatility, polarity, thermal stability, and solubility characteristics of each component in the mixture.

CONCLUSION

This study evaluated the effectiveness of five classical separation techniques—liquid-liquid extraction, simple distillation, evaporation, recrystallization, and acid-base extraction—for the separation of four different ternary mixtures. The results demonstrated that the percent error values ranged from 2.30% to 93.60%, with the efficiency being highly dependent on the physicochemical properties of the target compounds and the appropriateness of the selected method. Recrystallization proved most effective for non-volatile ionic solids such as sodium chloride (2.30% error), while simple distillation was highly efficient for compounds with significantly different boiling points, such as ethanol (3.30% error). Liquid-liquid extraction showed variable efficiency depending on polarity differences between the compounds, and acid-base extraction required precise pH control for optimal performance. The critical factors influencing separation efficiency were identified as temperature control, solvent selection, agitation technique, and the volatile nature of compounds such as diethyl ether and methanol. These findings provide a systematic comparison of classical separation techniques and offer practical guidelines for selecting appropriate methods in pharmaceutical and chemical laboratory practice. Future studies should focus on optimizing individual parameters for each technique and exploring alternative solvents to improve recovery rates.

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