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COMPARATIVE EVALUATION OF EXTRACTION SOLVENTS FOR THE SEPARATION OF AROMATIC HYDROCARBONS

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Abstract:
The article investigates the regularities of high-purity benzene recovery from arena-rich distillate fractions using liquid-liquid extraction and extractive distillation methods, alongside the effect of extractant nature on the process. Within the framework of the study, a comparative analysis of the efficiency of polar heteroatomic solvents widely used in industry (sulfolane, N-methylpyrrolidone, N-formylmorpholine, diethylene glycol, and triethylene glycol) was performed. Additionally, the effects of extraction temperature (20−65 °C), feed-to-solvent mass ratio (1:1 to 5:1), and extraction duration (35−75 min) on the separation degree of aromatic hydrocarbons were experimentally studied using TEG, NMP, and 30 % (by weight) NMP solution. The results indicate that under the investigated conditions, the 30 % NMP solution provides the highest extraction efficiency (64-65) for separating aromatic hydrocarbons, outperforming pure NMP and TEG. The optimal temperature range for the process was determined to be 40−50 °C, with an optimal duration of 55 minutes.

Keywords: Aromatic hydrocarbons, benzene, extraction, sulfolane, N-methylpyrrolidone (NMP), triethylene glycol (TEG), separation degree, mass ratio, extraction temperature.

INTRODUCTION

Within the contemporary petrochemical sector, aromatic hydrocarbons, particularly benzene, represent indispensable feedstocks. Benzene serves as a primary building block in the manufacturing of plastics, synthetic fibers, rubbers, detergents, and various pharmaceuticals. Liquid fractions derived from catalytic reforming and pyrolysis operations are highly enriched with aromatic compounds (arenas), making the isolation of high-purity benzene from these streams a critical technological objective. Because benzene shares extremely close boiling points with its co-existing n-paraffins and naphthenic hydrocarbons, standard fractional distillation is economically unviable and technically inefficient due to the formation of close-boiling azeotropic mixtures. Consequently, extractive distillation and liquid-liquid extraction techniques are widely adopted in commercial operations.

The economic viability and operational efficiency of the extraction process are fundamentally dictated by the chemical nature of the chosen solvent (extractant). An ideal solvent must concurrently possess high selectivity (separation capability) and substantial capacity (solubility towards target compounds). However, an inverse relationship is frequently observed between selectivity and solvent capacity, wherein highly selective agents exhibit deficient solubility traits, or vice versa. This phenomenon necessitates the systematic optimization of operational parameters – such as temperature, operating pressure, and solvent-to-feed ratios– alongside the formulation of blended solvent systems.

The primary objective of this study is to perform a comparative assessment of various solvents and their formulations employed in retrieving benzene from arena-rich distillates. Furthermore, it aims to elucidate the fundamental kinetic and thermodynamic impacts of solvent characteristics on the system dynamics to establish the optimal processing configuration.

Materials and Methods

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In industrial practices directed at isolating aromatic fractions from paraffinic streams, several polar heteroatom-containing solvents have been extensively researched and deployed, including sulfolane, N-methylpyrrolidone (NMP), N-formylmorpholine (NFM), diethylene glycol (DEG), and triethylene glycol (TEG).

Comparative Evaluation of Industrial Solvents

The operational performance indicators of predominant commercial solvents evaluated for benzene extraction are summarized in Table 1:

Table 1. Benchmarking of commercial extraction solvents in benzene recovery

Solvent (Extractant)	Selectivity (S)	Capacity (C)	Thermal Stability	Industrial Implementation
Diethylene Glycol (DEG)	Exceptionally High	Low	Adequate; requires extensive circulation rates	Early stages of Udex operations
Sulfolane	High	Optimal	High (up to 220 °C)	Shell Sulfolane process (most prevalent)
N-Methylpyrrolidone (NMP)	Moderate	Exceptionally High	Sustained; low corrosive tendencies	Arosolvan processing units
N-Formilmorpholine (NFM)	High	High	Excellent; exhibits low vapor pressure	Formex and Morphylane extractive distillation

Global market demand for aromatic hydrocarbons, specifically single-ring structures like benzene, toluene, and xylene, as well as polycyclic derivatives like naphthalene and anthracene, has experienced progressive growth. This surge is driven by their expanding role as base raw materials and auxiliary reagents across nearly all industrial sectors.

To isolate benzene and toluene from liquid pyrolysis byproducts, conventional rectification schemes have been proposed. To accomplish high-purity separation, the integration of advanced mass-transfer contact columns equipped with cross-corrugated structured packing blocks featuring maximized specific surface areas is recommended. Such specialized contact devices maintain superior efficiency while accommodating wide fluctuations in vapor-liquid hydrodynamic loads, thereby ensuring the precise fractionation of pyrolysis tar streams.

Extracting high-purity aromatics (99.0 % − 99.9 %) from catalytic reforming products remains technically challenging. This difficulty arises because arenas form azeotropic configurations with adjacent-boiling naphthenes and paraffins. To circumvent these limitations, azeotropic distillation, extractive rectification, and selective liquid extraction are deployed, with the latter being the most prevalent configuration in commercial refineries. In this approach, a composite aromatic fraction is first isolated via extraction before being sequentially fractionated into high-purity benzene, toluene, and xylene isomers.

The principle of selective extraction relies on the preferential solubility of target components (arenas) within a solvent, contrasted against the low affinity or absolute insolubility of the non-aromatic matrix. Hence, precise solvent selection is critical. Industrial frameworks rely extensively on compounds like DEG, TEG, sulfolane, NMP, and NFM.

Comprehensive insights gathered from scientific literature reveal the following specific behaviors for these extractants:

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Glycols (DEG, TEG): Diethylene glycol saw widespread use in pioneering extraction processes like the Udex configuration. While glycols display excellent selectivity toward aromatic rings, their solvent capacity is noticeably constrained. This limitation requires massive solvent circulation rates (1:8 to 1:12 solvent-to-feed volumetric ratios), which directly causes high energy expenditure during the solvent regeneration (desorption) phase.

Sulfolane (Tetramethylene sulfone): Modern global operations rely on sulfolane for more than 50 % of all aromatics extraction applications. Research indicates that sulfolane possesses a high density and an elevated boiling point (285 °C), preventing the formation of azeotropes with benzene. Its well-balanced selectivity and capacity allow operations to scale down the solvent-to-feed ratio to a conservative 3:1. Its primary disadvantages include high operating viscosity and a susceptibility to thermal degradation when processing temperatures exceed 220 °C.

N-Methylpyrrolidone (NMP) and N-Formylmorpholine (NFM): Recent investigations highlight the distinct advantages of NFM and NMP within extractive distillation regimes. NFM, characterized by its diminished vapor pressure and superb thermal durability, exhibits outstanding kinetic performance during benzene recovery from pyrolysis gasoline. Conversely, NMP possesses the highest absolute capacity for absorbing arenas, though it typically requires a controlled water fraction (4 % − 8 %) as a co-solvent to raise its selectivity to acceptable levels.

Results and Discussion

Advanced Combined and Blended Solvent Frameworks

A review of academic literature from the past 5 – 6 years shows a clear shift toward binary blended solvent systems (such as sulfolane + NFM or sulfolane + DEG + water) over single-component agents to maximize process throughput. Introducing water as an anti-solvent modifier systematically enhances the extractant's selectivity and lowers the necessary thermal threshold for aromatic desorption. Nonetheless, excessive water fractions are documented to escalate the thermal load on the stripper (regeneration) column while amplifying corrosion risks within the infrastructure.

Concurrently, within green chemistry frameworks, ionic liquids (ILs) and deep eutectic solvents (DES) are being explored as sustainable alternative extractants, though their commercial scalability remains limited by high viscosities.

Influence of Extraction Temperature on Separation Efficiency

Figure 1 illustrates the comparative impact of extraction temperature on the recovery of aromatic hydrocarbons utilizing TEG, pure NMP, and a 30 % (by weig*h*t) aqueous NMP solution. The graphical profiles indicate that temperature variations exert a substantial, yet solvent-specific, influence on mass-transfer efficiency.

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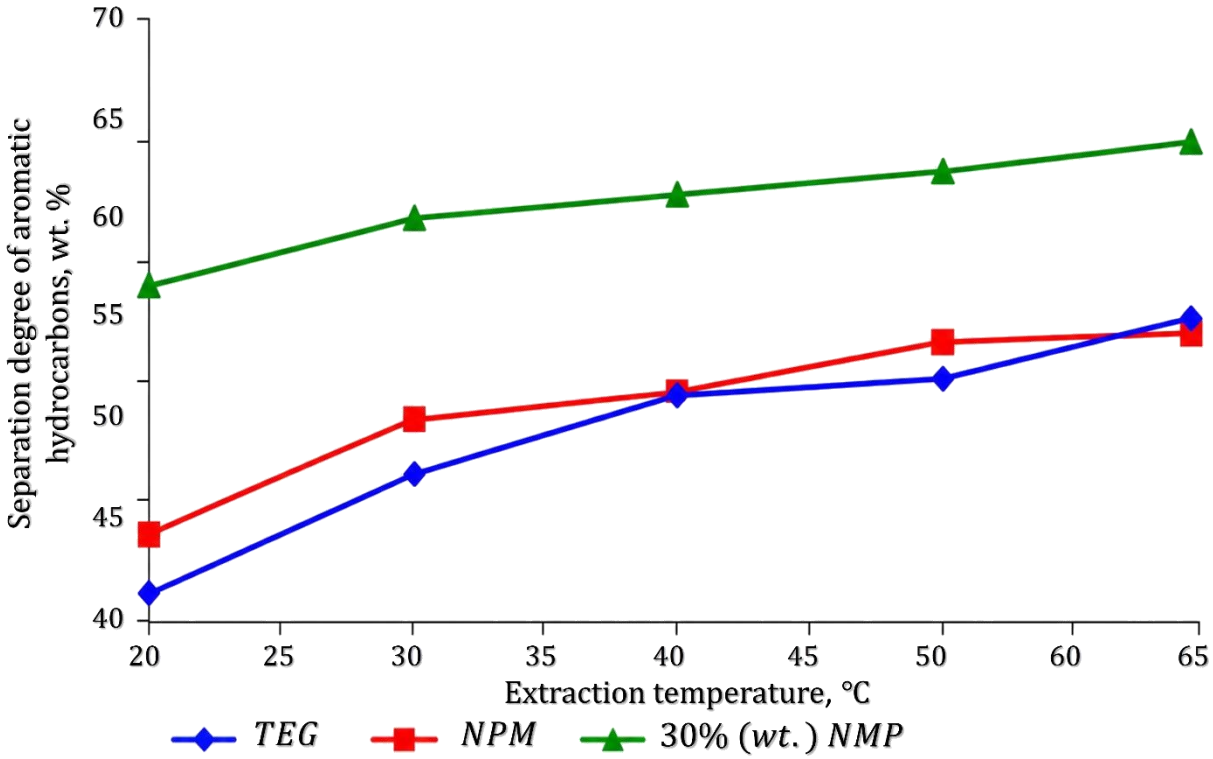


Figure 1. Effect of extraction temperature on the separation degree of aromatic hydrocarbons

When employing TEG, elevating the operating temperature from 20 ℃ to 40 ℃ enhances the recovery degree from approximately 47 % to 51 %. However, beyond 40 ℃, the extraction curve plateaus, showing a slight decline at 65 ℃. This inversion is attributed to the reduced selectivity of TEG toward aromatics at elevated temperatures as the system converges toward thermodynamic equilibrium.

For the pure NMP system, the recovery degree demonstrates a steady linear progression with temperature, rising from roughly 50 % at 20 ℃ to approximately 56 % at 65 ℃. This trend verifies that higher thermal states enhance NMP's solvating power toward aromatic molecules while intensifying interfacial mass-transfer kinetics.

The maximum extraction performance is achieved using the 30 % (by weight) NMP solution. Transitioning the temperature from 20 ℃ to 40 ℃ results in a sharp increase in recovery from 55 % to 64 %. The extraction efficiency remains stable within the 40−50 ℃ window, followed by a marginal reduction at 65 ℃. This behavior marks the 40−50 ℃ range as the optimal operating zone, beyond which thermal forces begin to compromise the solvent's selectivity.

The overall analysis confirms that higher extraction temperatures accelerate mass-transfer kinetics, driving the migration of aromatic species into the solvent phase. However, once the optimal temperature threshold is crossed, the recovery curve stabilizes or decreases due to equilibrium limitations and selectivity losses. Based on these experimental outcomes, the extractant efficacy ranks in the following descending sequence:

30% (wt.) NMP>NMP>TEG

Consequently, the 30 % aqueous NMP formulation delivers the highest performance under the evaluated conditions, and an operating temperature of 40−50 ℃ is recommended to maximize recovery without incurring unnecessary thermal penalties.

Influence of Solvent-to-Feed Mass Ratio on Aromatic Recovery

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The solvent-to-feed mass ratio (S:F) is a critical parameter governing the material balance, product yield, and overall economic performance of liquid-liquid extraction units. Incremental adjustments to the S:F ratio alter the extraction dynamics through specific mechanisms:

Driving Force Enhancement: Elevating the solvent volume relative to the feed drives a significant initial increase in aromatic recovery (α, %). This increase occurs because a larger solvent phase expands the two-phase heterogeneous region, shifting the equilibrium concentration in favor of the extract phase. This maintains a steep concentration gradient (ΔC), accelerating interfacial mass transfer.

The Upper Threshold (Equilibrium Limit): The solvent ratio cannot be increased indefinitely. Beyond an optimal threshold (typically 3:1 to 4:1 depending on the solvent matrix), detrimental process trends emerge. Excessive solvent volumes lower the mass-transfer resistance for non-aromatic species, allowing paraffinic and naphthenic fractions to migrate into the extract phase. This co-extraction directly degrades the purity of the final benzene product. Furthermore, large solvent volumes impose a heavy thermal load on the subsequent desorption column, increasing the energy required for solvent stripping and raising operating costs.

Solvent-Specific Variations:

Sulfolane: Due to its high capacity for benzene rings, it operates efficiently at low mass ratios ranging from 2.5:1 to 3.5:1, providing a significant energy advantage.

N-Formylmorpholine (NFM): Requires moderate ratios of 3:1 to 4.5:1 within extractive distillation setups.

Glycols (DEG, TEG): Their low capacity requires high operating ratios ranging from 8:1 to 12:1 to achieve equivalent separation performance.

Figure 2 outlines the aromatic recovery profiles as a function of the solvent-to-feed mass ratio for TEG, NMP, and the 30 % NMP solution. While all candidate systems show an upward trend in extraction efficiency as the solvent fraction expands, the rate of increase is highly dependent on the solvent's chemical composition, with the 30 % NMP blend consistently outperforming the alternatives.

For TEG, increasing the mass ratio from 1:1 to 5:1 raises the aromatic recovery from 46 % to 58 %. The most pronounced gain is observed between 1:1 and 3:1, after which the curve flattens as the system approaches equilibrium. Pure NMP demonstrates superior capacity compared to TEG, with its recovery efficiency rising from 49 % to 57 % over the same range, showing a sharp initial surge up to a 2:1 ratio.

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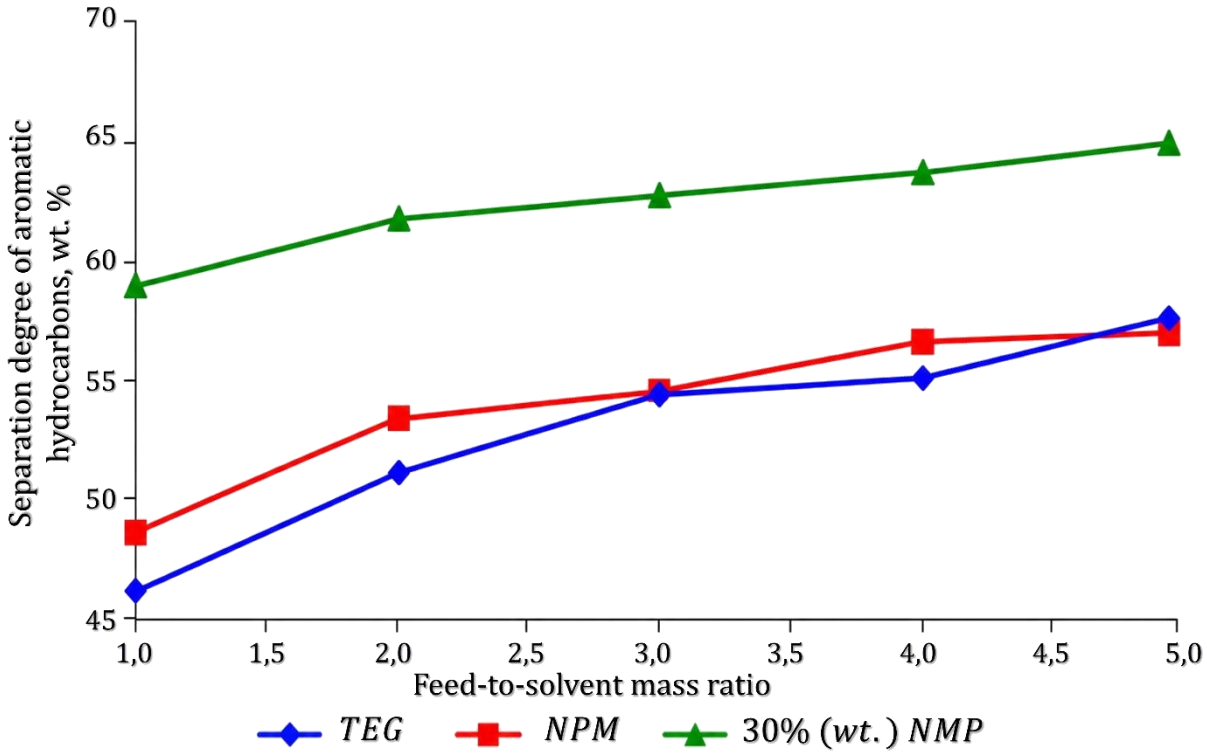


Figure 2. Effect of feed-to-solvent mass ratio on the separation degree of aromatic hydrocarbons

The 30 % NMP solution exhibits the highest extraction performance across the entire testing interval, starting at 59 % recovery at a 1:1 ratio and reaching 65 % at a 5:1 ratio. This steady, sustained upward trend underscores the superior selectivity and mass-transfer characteristics of the aqueous blend.

These results indicate that increasing the solvent-to-feed ratio enhances the extraction driving force and optimizes the interfacial contact area. However, the sharply lower growth rate observed between 4:1 and 5:1 confirms that the system is approaching its thermodynamic limit. Balancing extraction yields against solvent utility costs indicates that a mass ratio of 4:1 or 5:1 represents the optimal operational limit for this configuration, following the performance sequence:

30% (wt.) NMP>NMP>TEG

Compared to TEG and pure NMP, the 30 % NMP formulation provides an average efficiency gain of 7−13 % and 5−10 % percentage points, respectively, demonstrating its viability as a high-performance extractant.

Influence of Extraction Duration on Kinetic Performance

Extraction duration governs the mass-transfer kinetics of aromatic species migrating across the liquid-liquid phase boundary. The time variable directly influences interfacial equilibrium and molecular diffusion rates.

During the initial phase of extraction, aromatic recovery increases sharply in direct proportion to time. This rapid kinetic phase is driven by a maximum concentration gradient (ΔC) between the uncontacted solvent and the feed. As benzene and its derivatives contact the polar extractant, rapid mass transfer occurs across the interfacial boundary. Over time, the solvent phase approaches saturation, the concentration gradient narrows, and the system reaches thermodynamic equilibrium, beyond which additional contact time yields no further recovery.

Extending the contact duration beyond this equilibrium point can impair overall selectivity. Light aromatics like benzene and toluene migrate rapidly into the solvent phase within the first

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10–20 minutes . Prolonged contact allows slower-diffusing non-aromatic paraffins and naphthenes to enter the solvent phase via physical absorption, decreasing the purity of the extract. Additionally, in mechanical agitation systems like mixer-settlers, excessive mixing times can induce stable emulsions that complicate subsequent phase separation.

The time required to reach equilibrium is mediated by solvent viscosity. High-viscosity systems like sulfolane and glycols (DEG, TEG) present higher resistance to mass transfer, requiring longer contact times (35−45 minutes). Low-viscosity fluids like NMP and NFM exhibit higher diffusion coefficients, reaching equilibrium within 15−20 minutes.

Figure 3 displays the kinetic extraction curves for TEG, pure NMP, and the 30 % NMP blend over a time horizon of 35 to 75 minutes . The profiles show that recovery increases moderately before stabilizing into a steady plateau.

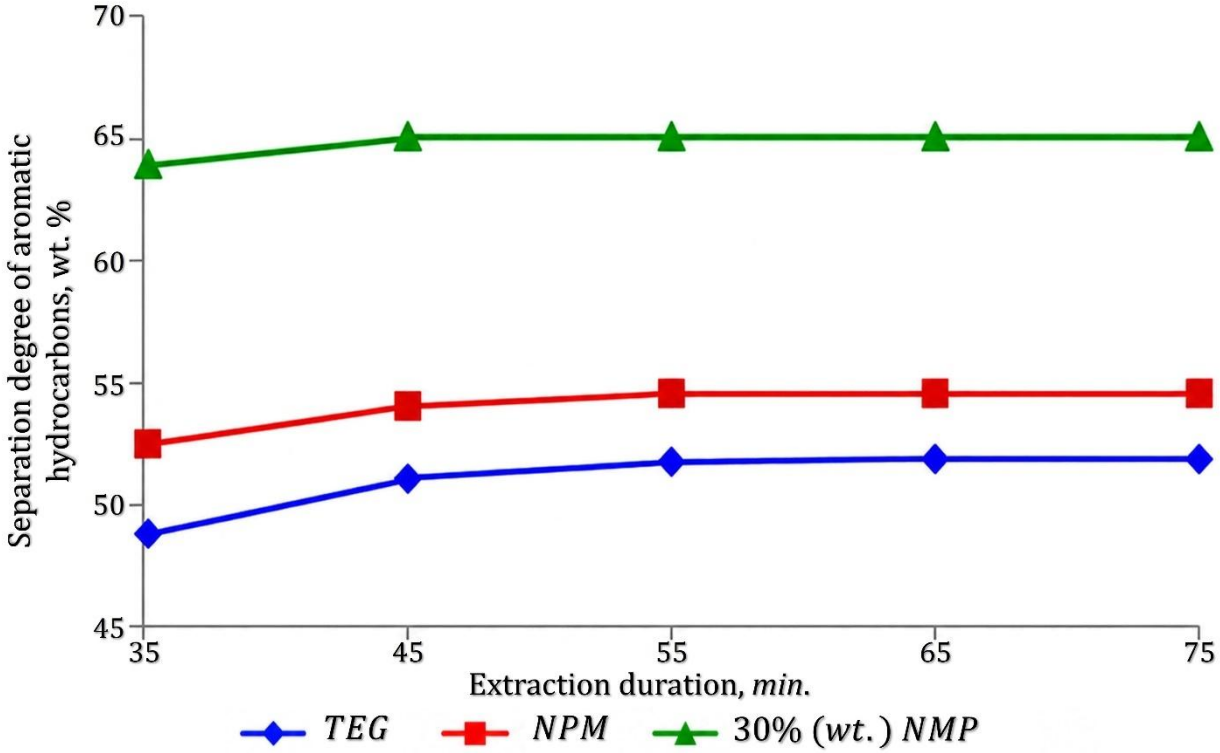


Figure 3. Effect of extraction duration on the separation degree of aromatic hydrocarbons

For the TEG system, recovery stands at 48.5 % at 35 minutes and reaches a maximum of 51.0 % − 51.5 % within the 55–65 minute window, indicating the onset of phase equilibrium. Pure NMP establishes a higher kinetic profile, yielding 52 % recovery at 35 minutes and stabilizing at 54 % at 55 minutes.

The 30 % NMP solution demonstrates superior performance, achieving a high recovery of 64 % at 35 minutes and reaching a stable plateau of 65 % between 45 and 55 minutes. This rapid stabilization indicates that the system reaches equilibrium quickly, making further processing time inefficient.

These kinetic evaluations demonstrate that extending the extraction time up to 55 minutes optimizes mass transfer and enhances aromatic recovery. Beyond 55 minutes, the process reaches a definitive plateau. Therefore, a 55−minute extraction period is identified as the techno-economic optimum, following the efficiency sequence:

30% (wt.) NMP>NMP>TEG

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The 30 % NMP solution outperforms TEG by 13-14 % percentage points and pure NMP by 10−11 % percentage points, demonstrating that optimizing solvent composition is a key factor in improving extraction performance.

CONCLUSION

Based on this systematic evaluation of benzene recovery from arena-rich distillates via liquid-liquid extraction and extractive distillation configurations, the following conclusions are established:

1. **Extractant Efficiency Benchmarking:** Comparative analysis of polar heteroatomic solvents indicates that **sulfolane** and **N-formylmorpholine (NFM)** provide the most effective balance of selectivity and capacity for industrial benzene separation.
2. **Kinetic Optimization:** Mass-transfer rates peak during the initial 15–20 minutes of extraction. Extending contact times beyond 30 minutes facilitates the co-extraction of adjacent paraffins and naphthenes due to late-stage diffusion, which reduces process selectivity.
3. **Mass Ratio Parameterization:** Increasing the solvent-to-feed (S:F) ratio shifts the phase equilibrium favorably, increasing aromatic yields. For sulfolane systems, the optimum techno-economic balance is achieved at an S:F mass ratio between 2.5:1 and 3.5:1. Ratios above this range increase the thermal load on the desorption column without yielding significant recovery gains.
4. **Strategic Directions:** To reduce industrial energy consumption and maximize benzene recovery, the use of binary solvent mixtures modified with small fractions of water (e.g., sulfolane + water or NMP + water) represents a highly effective processing strategy.

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