Separation Sequencing in Batch Distillation with Slop Recycle *

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Abstract— **Batch distillation is a critical separation process in industries requiring operational flexibility, such as pharmaceuticals and specialty chemicals, but it often suffers from energy inefficiency and material losses during transition cuts (slop). While industrial practice involves recycling slop to subsequent batches to improve recovery, traditional separation sequencing methodologies based on heuristics often neglect this recycling, assuming fixed feed compositions. This work extends the existing marginal vapor method, to include the effect of slop recycle in separation sequencing. By utilizing a recursive material balance framework, the proposed approach determines the cyclic steady state of the system where feed and slop compositions become invariant, eliminating the need for computationally expensive dynamic simulations. A case study involving a ternary mixture is used to assess the impact of slop recycling on separation sequencing using the proposed approach and the results are verified using dynamic simulations.**

I. INTRODUCTION

Batch distillation is widely employed in fine chemicals, pharmaceutical, and specialty chemical industries due to its operational flexibility and handling of small volumes and changes in product specifications. Unlike continuous distillation, batch operation allows multiple components to be separated sequentially in a single column; however, this flexibility comes at the expense of increased energy consumption and material losses due to intermediate impure cuts (slop) [1], [2]. In multicomponent batch distillation, the complexity of a separation increases significantly with the number of components. For a mixture containing N components, the separation requires N–1 cuts, where each cut represents the withdrawal of a fraction enriched in one component or is an impure mixture of two components. The sequence refers to the specific order in which these component fractions or cuts are withdrawn during the batch operation. For a ternary mixture, there are two possible sequences of separation, direct and indirect. In a direct sequence, the feed is fed to the reboiler. The products are withdrawn in the decreasing order of their volatility from the reflux drum. Whereas in an indirect sequence, the feed is fed in the reflux drum and the least volatile component is collected first from the reboiler. For more complex mixtures the possible sequences expand drastically, necessitating systematic approaches to sequence selection [3]. As a result, the design and sequencing of batch distillation operations remain an active area of research.

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A major source of inefficiency in batch distillation arises during cut transitions, where an impure mixture must be withdrawn before the desired product purity is attained. This impure mixture, commonly referred to as slop (Fig. 1), is unavoidable in practical operation and typically consists of a binary or near-binary mixture collected between adjacent product cuts. In industrial practice, slop is rarely discarded; instead, it is commonly recycled to the subsequent batch in order to improve overall component recovery and reduce the waste generation [4-5]. Such recycling, while beneficial from a material efficiency standpoint, introduces coupling between successive batches and alters the effective feed composition over time.

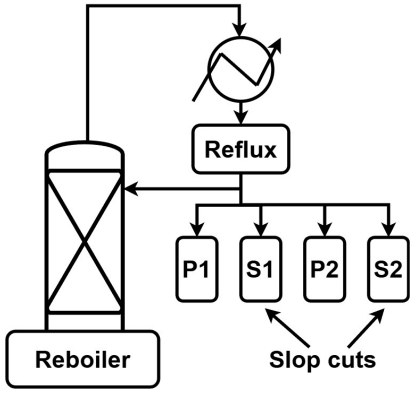

Fig. 1. Conventional Batch Distillation

Several approaches exist for selecting separation sequences in multicomponent distillation, broadly categorized into heuristic rules, shortcut methods, and rigorous optimization techniques [7-8]. A superstructure-based framework has been developed for the synthesis of batch distillation systems that simultaneously evaluates column configurations and separation sequences. The problem is formulated as a mixed-integer nonlinear programming (MINLP) model and solved using a hybrid optimization approach combining simulated annealing and sequential quadratic programming to identify cost-effective designs [2]. Another work established a rigorous framework for feasible separation sequencing using thermodynamic principles and residue curve maps, defining admissible composition trajectories under batch operation and clarifying how complex mixtures can be fractionated through successive cuts [9]. These concepts were later extended to azeotropic and highly non-ideal systems through phase-equilibrium-based synthesis methods, enabling the identification of feasible operating regions and separation structures beyond conventional distillation lim-

its [10]. Most of the existing sequencing methodologies, however, implicitly assume that each batch is processed independently with a fixed fresh feed composition. Under this assumption, slop generation is either neglected or treated as an individual operational issue rather than as an integral part of the separation strategy. When the process of slop recycling is practised, this assumption is no longer valid, since the recycled material progressively modifies the feed composition until a cyclic steady state is reached [11]. At this steady state, the compositions of the feed, products, and slop remain invariant from cycle to cycle. The cyclic steady state feed conditions depend on the slop generated and hence, a methodology is required to approximately estimate the slop generated in any sequence for the given feed conditions. Consequently, sequencing decisions based solely on the fresh feed may no longer be optimal under practical operating conditions as shown later in this paper. In our earlier work, a method of marginal vapor, an analytical measure of the additional vapor required due to the presence of non-key components relative to an equivalent key-only binary system, was introduced to select optimal batch distillation sequence [12]. While this approach provides a computationally efficient criterion for selecting optimal separation sequences based on vapor requirement, it focuses on energy consumption rather than material efficiency and does not account for slop generation or recycle across batch cycles. The objective of the present work is to extend this sequencing framework to explicitly incorporate the effects of slop generation and recycle in batch distillation. By relating the amount of slop generated to the column feed, a batch-to-batch material balance framework is developed that captures the evolution of feed composition under slop recycle and enables the determination of a cyclic steady state without requiring detailed dynamic simulation. The marginal vapor concept is then used for this cyclic steady-state system to pursue separation sequencing in the presence of slop recycle. The proposed approach provides an analytical framework that combines thermodynamic efficiency, product yield and batch production campaign. By accounting the effects of slop recycle on the separation sequencing, the proposed approach retains the simplicity of the original framework while enabling more realistic sequencing decisions for batch distillation processes. This approach moves beyond single-batch operation to address the cumulative effects of slop recycle over multiple cycles, a factor that significantly influences energy consumption, and overall yield.

II. Theory

In conventional multi-cut batch distillation, pure product fractions are withdrawn when the distillate composition satisfies the desired purity specification. Between two such product withdrawals, the distillate composition falls below the specified purity level, resulting in an intermediate impure fraction commonly referred to as a slop cut. Several approaches have been proposed to handle these intermediate cuts generated during batch operation[13]. One of the most commonly employed techniques is slop recycle, wherein all intermediate cuts are recycled and mixed with the fresh feed of the subsequent batch[14]. Using this strategy, a cyclic steady state is eventually attained after several operating cycles, such that the quantities of products and slops generated in two successive cycles become identical.

Within this framework, the effect of slop recycle is manifested solely through a change in the initial feed composition from one cycle to the next. Consequently, the slop generated in cycle n, denoted by C^n, depends on both the fresh feed contribution and the recycled slop carried over from the previous cycle, C^{n-1}. The cyclic operation can therefore be represented using recursive component mass balances, enabling the analytical derivation of C^n[11].

A ternary mixture is considered here to illustrate the proposed approach. The material balances associated with this problem are schematically represented in Fig.2. In the first batch, the fresh feed is denoted by B_1 with composition x_{B_i}. The first operating step produces a light product cut P_{11} with composition x_{P1_i}. The material remaining in the still is S_{11} with composition x_{S1_i}.

The second operating step produces the light intermediate cut C_{11} with composition x_{C1_i}, while the material remaining in the still is S_{21} with composition x_{S2_i}. The third operating step produces a medium product cut P_{21} with specification x_{P2_i}, and the material remaining in the still is S_{31} with composition x_{S3_i}. The fourth and final operating step produces the heavy intermediate cut C_{21} with composition x_{C2_i}. The material remaining in the still constitutes the heavy product cut P_{31}. This completes the first batch cycle.

In the second batch cycle, the feed consists of the total slop generated in the previous cycle (C_{11} and C_{21}) together with the fresh feed B_2, such that the total feed amounts to F. The second batch is operated in a manner similar to the first batch, and the slop fractions generated are collected and mixed with the fresh feed for the subsequent cycle. This procedure is repeated for each operating cycle until cyclic steady state is achieved, i.e.,

$$B_{n-1} = B_n, \qquad C_{1,n-1} = C_{1,n}, \qquad C_{2,n-1} = C_{2,n}$$

Applying material balances over the column, the total feed to the column in the i^{th} cycle is maintained constant as

$$F = B^i + C_1^{i-1} + C_2^{i-1} \tag{1}$$

The overall mass balance over the column after Step 1 is given by

$$F = P_1^i + S_1^i \tag{2}$$

The corresponding component balance after Step 1 can be written as

$$x_{F_1}^i F = x_{P_1}^i P_1^i + x_{S_1}^i S_1^i \tag{3}$$

If f_1^i represents the fraction of material remaining in the still after removal of the light intermediate cut, then

$$f_1^i = \frac{S_2^i}{S_1^i} \tag{4}$$

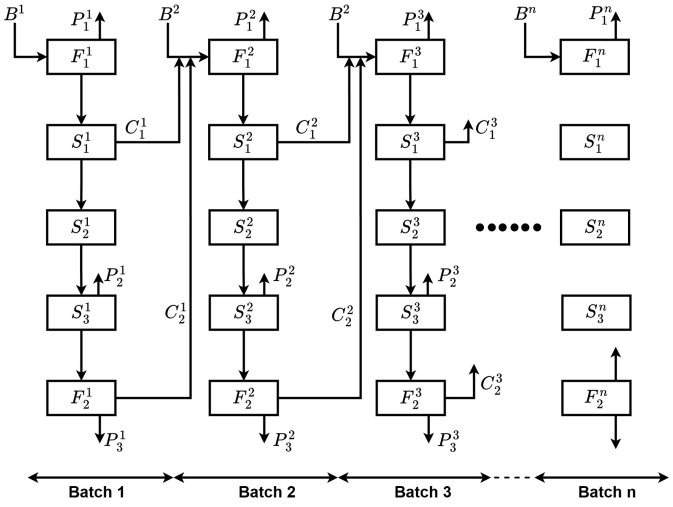

Fig. 2. Consecutive cycles for ternary mixture with complete slop recycle

The amount of light intermediate cut collected is therefore given by

$$C_1^i = S_1^i - S_2^i = S_1^i(1 - f_1^i) \tag{5}$$

Using Eqs. (2), (3) and (5), the light intermediate cut can be expressed as

$$C_1^i = F\beta^i \tag{6}$$

where

$$\beta^i = (1 - f_1^i)\left(\frac{x_{F_1}^i - x_{P_1}^i}{x_{S_1}^i - x_{P_1}^i}\right) \tag{7}$$

Combining Eqs. (1) and (6) gives

$$C_1^i = \left(B^i + C_1^{i-1} + C_2^{i-1}\right)\beta^i \tag{8}$$

Similarly, the second intermediate cut can be expressed as

$$C_2^i = \left(S_2^i + C_2^{i-1}\right)\gamma^i \tag{9}$$

where

$$\gamma^i = (1 - f_2^i)\left(\frac{x_{F_2}^i - x_{P_2}^i}{x_{S_3}^i - x_{P_2}^i}\right) \tag{10}$$

and

$$f_2^i = \frac{P_3^i}{S_3^i} = \frac{S_4^i}{S_3^i} \tag{11}$$

Defining f^i as the fraction of the original feed retained in the still after extraction of the light intermediate cut,

$$f^i = \frac{S_2^i}{F} \tag{12}$$

Eq. (9) can then be rearranged as

$$C_2^i = \left(Ff^i + C_2^{i-1}\right)\gamma^i \tag{13}$$

The recursive application of Eqs. (8) and (13) over n cycles yields a convergent series that can be used to determine the steady-state values of both intermediate cuts[11]. This feature enables the modeling of batch distillation with recycle streams without requiring repeated simulations to achieve cyclic steady state. In other words, it is unnecessary to simulate the distillation process beginning from a fresh batch and extending through multiple subsequent cycles.

One possible approach is to use detailed dynamic simulations to estimate the values of β and γ, followed by the determination of the cyclic steady-state slop conditions. However, such a procedure is computationally intensive. In the present work, the amount and composition of the slop are approximately estimated using the concept of warped time[15].

III. METHODOLOGY

The marginal vapor method[12] can be used to determine the optimal separation sequence. The marginal vapor (MV) is defined as the difference in vapor flow rate (V) of a distillation column in the presence and absence of non-key components. The MV of a separation task $l \dots l_k/h_k \dots h$ is given by

$$MV(l \dots l_k/h_k \dots h) = V(l \dots l_k/h_k \dots h) - V(l_k/h_k) \tag{14}$$

This method requires the initial feed conditions of the mixture to evaluate the vapor generated during batch operation for a given sequence. However, in the presence of slop recycle, the feed composition changes from one cycle to the next until cyclic steady state is attained. Therefore, prior to applying the MV method in such systems, it is necessary to determine the cyclic steady-state feed composition.

To predict the cyclic steady-state behavior of a batch distillation system, the quantity and composition of the slop generated for a given feed condition must be estimated. In the present approach, the concept of warped time is employed to estimate the slop amounts and compositions[15]. This approach eliminates the need for detailed dynamic simulation of the entire batch column. By establishing a relationship between feed and product compositions, it enables direct estimation of the product and slop quantities.

Consider a conventional batch distillation column with N stages, where the feed (F) is charged to the reboiler and products are withdrawn from the top at a rate D mol/min. The overall material balance for the column is given by

$$\frac{dH}{dt} = -D \tag{15}$$

where H represents the holdup in the reboiler at time t.

Assuming negligible tray holdup, the component balance for component i can be written as

$$\frac{d(Hx_{B_i})}{dt} = -Dx_{D_i} \tag{16}$$

Expanding Eq. (16) gives

$$\frac{dx_{B_i}}{dt} = \frac{D}{H}(x_{B_i} - x_{D_i}) \tag{17}$$

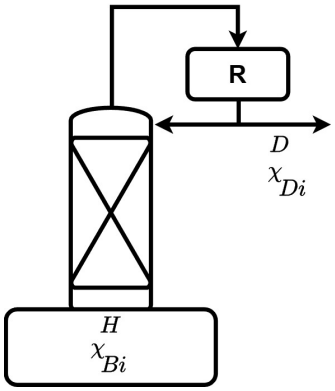

Fig. 3. Conventional Batch Distillation

To simplify integration, a warped time variable ξ[15], defined such that

$$d\xi = \frac{D}{H}dt \tag{18}$$

with $\xi = 0$ at $t = 0$.

From Eqs. (17) and (18), we get:

$$\frac{d(x_{B_i})}{d\xi} = (x_{B_i} - x_{D_i}) \tag{19}$$

For a direct sequence, assuming constant relative volatility and negligible vapor holdup, for an ideal system, the product composition for i^{th} component in the reflux can be related to still composition as:

$$x_{D_i} = \frac{\alpha_i^{(N+1)} x_{B_i}}{\sum_{i=1}^{3} \alpha_i^{(N+1)} x_{B_i}} \tag{20}$$

where α_i is volatility of the i^{th} component and N is the total number of stages in the column.

Substituting Eq. (20) in Eq. (19):

$$\frac{dx_{B_i}}{d\xi} = x_{B_i} - \frac{\alpha_i^{(N+1)} x_{B_i}}{\sum_{i=1}^{3} \alpha_i^{(N+1)} x_{B_i}} \tag{21}$$

These set of ordinary differential equations (Eq. 21 for n number of components) are solved numerically to get the composition profiles over the column. Based on these profiles and the specified product purity requirements, distinct operating phases corresponding to the withdrawal of pure product fractions and intermediate impure cuts (slop) can be identified. The transition between these phases is determined from the composition trajectories, from which the corresponding values of ξ are obtained and used to estimate the holdup in the still at a given instant. From Eqs. (15) and (18), the holdup in the still is given as:

$$H(\xi) = H_0 e^{-\xi} \tag{22}$$

The difference in holdup between successive transitions provides an estimate of the quantities of both pure products and slop generated during the batch operation. This procedure is illustrated in detail in the subsequent section through a representative case study. Both slop cuts are mixed (C) and recycled to the next cycle with constant charge F per cycle:

$$F = B^k + C^{(k-1)} \tag{23}$$

with the updated feed composition:

$$x_F^k = \frac{B^k x_B + C^{(k-1)} x_C^{(k-1)}}{F} \tag{24}$$

with the mixed slop composition:

$$x_C^k = \frac{C_1^k x_{C1}^k + C_2^k x_{C2}^k}{C_1^k + C_2^k} \tag{25}$$

The cyclic steady state is reached when successive cycles produce identical cuts, i.e., $B_{n-1} = B_n$, $C_{1,n-1} = C_{1,n}$ and $C_{2,n-1} = C_{2,n}$. Using these cyclic steady-state conditions, we calculate the marginal vapor. The total vapor generated during a separation process in batch distillation column is calculated using Underwood's equations. The detailed procedure of the same is described in earlier work [12]. The proposed methodology determines the optimal separation sequence for a ternary mixture while accounting for slop generation. The slop quantity is estimated from the initial feed composition and column design parameters, thereby reducing the computational complexity of the problem. Unlike detailed dynamic simulations that require modeling of all trays in the column, the proposed approach relies on simplified dynamic representations, resulting in significantly lower computational effort.

IV. CASE STUDY

Let's illustrate the proposed approach using a ternary mixture of pentane (1), hexane (2), and heptane (3). The initial fresh feed of 1000 mol is charged to a column with 42 stages (Table I) [12].

TABLE I
INITIAL FEED CONDITIONS FOR THE TERNARY MIXTURE

Components	Feed composition	α
Pentane	0.25	7.1352
Hexane	0.50	2.6292
Heptane	0.25	1.0000

For the conventional operation (direct sequence), the approximate composition profile of components in the still and the reflux drum (Fig. 4) for a constant product withdrawal rate of 1.5 mol/min are computed using warped time. The composition of component 2 (hexane) will keep on increasing over time and when it reaches 99% purity, pure hexane is collected in the following stage, after which an impure mixture of hexane and heptane is withdrawn in the fourth

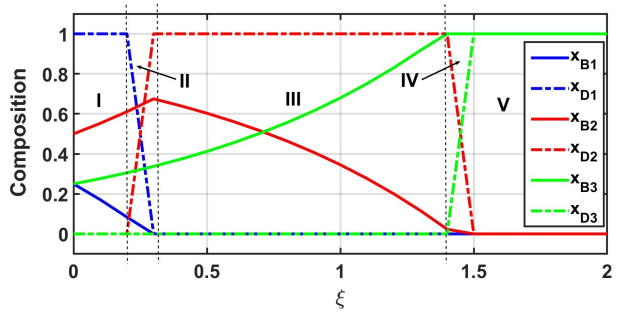

Fig. 4. Composition profile in distillate and reboiler for direct sequence

phase (C_2). The process is completed when the holdup remaining in the still becomes pure heptane. As shown in Fig. 4, Phase 1 terminates at $\xi = 0.23$. This corresponds to a remaining holdup in the still of approximately 788.838 mol after the removal of pentane. Accordingly, the amount of pentane collected is estimated to be 211.162 mol. The amounts of products and slops collected in first cycle and at steady state is shown in Table II for a direct sequence.

TABLE II

BASE CASE RESULTS FOR DIRECT SEQUENCE

Simulation	First batch		Cyclic steady state	
	Approximate Simulation	Rigorous Simulation	Approximate Simulation	Rigorous Simulation
Product 1 (mol)	232.152	184.167	193.560	181.235
Product 2 (mol)	422.270	344.930	440.130	332.076
Product 3 (mol)	212.018	182.673	211.580	185.837
Total slop (mol)	133.560	138.230	154.730	150.852
Slop composition	[0.220 0.550 0.230]	[0.229 0.563 0.208]	[0.240 0.530 0.230]	[0.232 0.541 0.227]
No. of ODEs	03	128	12	512
Computation time(s)	2.19	34.72	8.48	119.58

The amounts of products and slop predicted using the proposed approach closely match those obtained from detailed dynamic simulations, with deviations arising because of the assumption of negligible holdups in the column. For simulations, a part of the feed is distributed along the trays and reflux drum, and their holdups are kept constant. Also, in the proposed methodology, the operation starts when the reflux drum is pure in the most volatile component.

These cyclic steady-state slop amounts (177.913 mol) require a fresh feed of 822.087 mol. The total charge to the column is 1000 mol. The composition of the mixed feed is $[0.242 \quad 0.511 \quad 0.247]$. To evaluate the marginal vapor of the sequence, the composition profile of the still is first determined (Eqs. (26) and (27)), which is then used to calculate the reflux ratio and vapor generated using Underwood's equations.

$$x_{BA} = \frac{H_0(x_{FA} - 1)}{(H_0 - Dt)} + 1 \tag{26}$$

$$x_{Bi} = \frac{H_0 x_{Fi}}{(H_0 - Dt)} \qquad i \in \{B, C\} \tag{27}$$

The vapor generated is integrated over batch time to compute the total vapor for the column, $V(A/BC)$, which is found to be 4071 mol. In a similar manner, the total vapor required for binary separation of pentane and hexane, $V(A/B)$, is computed as 3155 mol. For a ternary mixture, the marginal vapor in a direct sequence is expressed as:

$$MV = V(A/BC) - V(A/B) \tag{28}$$

The marginal vapor is computed to be 915.80 mol for the direct sequence using these modified feed conditions.

Let us now consider an inverse column configuration (Fig. 5), where the feed is charged in the reflux drum and the products are withdrawn from the reboiler. For this case, the product composition for the i^{th} component in the reboiler can be related to distillate composition as:

$$x_{Bi} = \frac{\alpha_i^{(N+1)} x_{Di}}{\sum_{i=1}^{3} \alpha_i^{(N+1)} x_{Di}} \tag{29}$$

$$\frac{dx_{Di}}{d\xi} = x_{Di} - \frac{x_{Di}/\alpha_i^{(N+1)}}{\sum_{i=1}^{3} x_{Di}/\alpha_i^{(N+1)}} \tag{30}$$

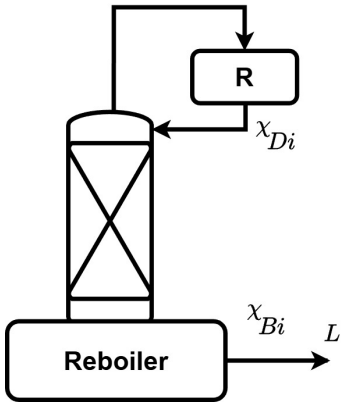

Fig. 5. Inverse batch configuration

Fig. 6 shows the composition profile of the components in the still and distillate for the indirect sequence. The operation has five phases similar to direct configuration. However, the least volatile component is removed first in phase 1.

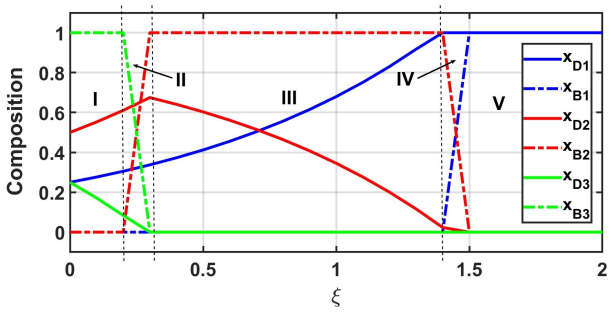

Fig. 6. Composition profile in distillate and reboiler for indirect sequence

Table III gives the amounts of products and slops collected in the cycle 1 and at steady state for the indirect sequence. These steady state slop amount and compositions need fresh feed of 845.27 mol. The total charge to the column is kept 1000 mol. The composition of the mixed feed is [0.248 0.507 0.245].

TABLE III
BASE CASE RESULTS FOR DIRECT SEQUENCE

Simulation	First batch		Cyclic steady state	
	Approximate Simulation	Rigorous Simulation	Approximate Simulation	Rigorous Simulation
Product 1 (mol)	211.162	180.368	181.269	186.317
Product 2 (mol)	425.370	353.942	438.760	327.615
Product 3 (mol)	242.088	185.670	202.058	182.350
Total slop (mol)	121.38	130.020	177.913	153.718
Slop composition	[0.230 0.550 0.220]	[0.230 0.580 0.190]	[0.210 0.560 0.230]	[0.213 0.529 0.268]
No. of ODEs	03	128	12	512
Computation time(s)	2.27	29.41	7.03	113.63

The composition profiles over time for the components in an indirect sequence are given by:

$$x_{DC} = \frac{H_0(x_{FC} - 1)}{(H_0 - Lt)} + 1 \tag{31}$$

$$x_{Di} = \frac{H_0 x_{Fi}}{(H_0 - Lt)} \qquad i \in \{A, B\} \tag{32}$$

These profiles are used to calculate the reflux ratio and vapor generated using Underwood's equations. The vapor generated is integrated over batch time to compute the total vapor for the column, $V(AB/C)$, which is found to be 5986 mol. In a similar manner, the total vapor required for binary separation of pentane and hexane, $V(B/C)$, is computed as 951 mol.

The marginal vapor in an indirect sequence is expressed as:

$$MV = V(AB/C) - V(B/C) \tag{33}$$

Using these modified feed conditions, the marginal vapor is computed to be 5035 mol for the indirect sequence.

TABLE IV
MARGINAL VAPOR (MOL) FOR TERNARY MIXTURE

Sequence	Slop recycling	Single batch
Direct	915.800	774.000
Indirect	5035.082	2968.000

From Table IV, it can be seen that the marginal vapor of the direct sequence is less than that of the indirect sequence for both single batch operation as well as slop recycle operation. Hence, the direct sequence can be considered the optimal sequence for the given mixture when the objective is to minimize the energy requirement of the process. It is interesting to note from Tables II and III that, during a single isolated batch, the total slop generated is lower. However, at cyclic steady state, the indirect sequence produces less slop and therefore results in higher productivity. The proposed framework thus demonstrates a trade-off between energy efficiency and material recovery in the selection of separation sequences for a ternary mixture. For the system considered, the direct sequence results in a lower marginal vapor, indicating lower energy requirement than the indirect sequence. In contrast, the indirect sequence leads to reduced slop generation at cyclic steady state, thereby improving material recovery. This demonstrates that the optimal sequence depends on the relative importance assigned to energy consumption and material efficiency.

V. CONCLUSIONS

This work uses the marginal vapor method to investigate the impact of slop recycle on sequencing of batch distillation through a recursive material balance framework. The proposed approach estimates the cyclic steady-state behaviour of a ternary mixture under slop recycle by considering feed composition evolution across successive batch cycles. The proposed framework offers a systematic and computationally efficient tool for incorporating the effects of slop recycle into separation sequencing. Our future work aims to generalize the proposed framework to the multicomponent mixtures and incorporate alternative slop-handling strategies.

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