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ABSTRACT

Researchers have begun to study a batch column with simultaneous top and bottom products, sometimes called a middle vessel column. The column is similar to a continuous column in that it has both rectifying and stripping sections. However, instead of a feed tray, the middle vessel column has a tray with a large holdup that acts like the still pot. Meski et al. (1993) showed that this column has several advantages over the rectifying and stripping batch columns including faster processing times and increased separation flexibility. We show that one can identify the feasible product and possible column profile regions for the batch rectifier, stripper and middle vessel columns using methods developed for continuous distillation.

Extractive distillation has been used for continuous and rectifying batch columns in separating azeotropic mixtures by feeding an extractive agent, normally a high boiler, near the top of the column. Using insights developed for continuous distillation, we compare extractive distillation using the batch rectifier and middle vessel column and show that these columns can theoretically recover all of the pure distillate product from an azeotropic feed. However, the batch rectifier requires a still pot of infinite size. It is possible to "steer" the still pot composition in the middle vessel column by adjusting column parameters such as the product and extractive agent flow rates. This steering enables the middle vessel theoretically to recover all of the distillate product without the need for an infinite still pot.

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1 Introduction

With the renewed interest in batch distillation, some interesting work has appeared in the literature revealing novel types of batch distillation columns. One such column, sometimes called the middle vessel column, is very similar to a continuous distillation column in that there is a rectifying section above the feed tray and a stripping section below the feed tray. In the case of the middle vessel column shown in Figure 1 (disregard the extractive section for the moment), the feed tray can be thought of as a tray with a very large holdup, similar to the still pot in normal batch distillation. Meski et al. (1993) showed that for a 3 component, constant relative volativity system, depending on certain column parameters (reflux and reboil ratio, relative volativities, number of trays), that the middle vessel column can accomplish many interesting types of separations. In particular, one can remove the light component as a distillate product, the heavy component as a bottoms product, while enriching the intermediate component in the middle vessel. At the end of the distillation operation, only the intermediate component would be left in the middle vessel, thereby separating a three component mixture into its pure components with only one column. They also showed that the middle vessel column could process the same mixture twice as fast as a typical batch column. However, they based their results for constant relative volativity mixtures. Hasebe et al. (1994) also studied the middle vessel column. They compared the separation of a three component, constant relative volativity system using a batch rectifier and stripper, shown in Figures 2a and 2b, and the middle vessel column. They optimized the operation of these columns using as an objective function the amount of product recoveied per processing time, and showed that the middle vessel column performed better than the rectifier in almost all cases.

For azeotropic mixtures, the work done by Bernot et al. (1990,1991) identified the product sequences for azeotropic mixtures in batch rectifiers (strippers) at infinite reflux (reboil) and infinite number of trays. Using only residue curve maps, they could predict the order of the distillate (bottoms) products. However, it is possible that one will remove a number of the products as azeotropes or at near azeotropic compositions. One has to process these products in some further processing step, recycle them, or dispose of them as waste.

The problem of azeotropic products in continuous distillation has been studied quite extensively. One technique widely used in breaking azeotropes is that of extractive distillation, in which one feeds a heavy component, called an entrainer, close to the top of the column. This component changes the relative volativities of the components and pulls some of the components down the column that normally show up in the distillate. Wahnschafft et al. (1993) carried out a graphical analysis using residue curve maps where they show why extractive distillation is possible for an appropriate entrainer. They also showed the limits of the extractive distillation.

There has been limited work in the literature regarding azeotropic batch extractive distillation. Yatim et al. (1993) simulated a batch extractive distillation column using a rectifier. They compared their simulations to experimental data that was collected, and got favorable results. They were able to recover approximately 82% of their main distillate product in relatively pure form.

2 Basic Concepts

2.1 Batch Column Product Sequences and Still Paths

A distillation region is a region of still compositions that give the same product sequence when distilled using batch distillation (Ewell and Welch, 1945). Using residue curve maps, Bernot et al. (1990) identified these distillation regions and predicted the product sequences for azeotropic mixtures using a batch rectifier at infinite reflux and infinite number of trays. In identifying these products, they were also able to predict how the still composition changed versus time, sometimes called the still path. In developing their model for a batch rectifier, they showed the overall component mass balance to be:

$$\frac{dx_s}{d\xi} = x_s - x_d \tag{1}$$

where x_s and x_d are the still and distillate mole fractions and £ is a pseudo time. Xg and x_d must lie on a line that is tangent to the instantaneous change of the still composition. The instantaneous change in still composition will be in a direction opposite that of the direction pointing to x^ from Xg. Also, the column profile must follow the residue curve due to the assumption of infinite reflux, where they have approximated the distillation curves with the residue curves in their analysis. Their analysis also only applies at zero holdup in the column.

In determining the product sequence, Bernot et al. pointed out that the first product obtained is the local minimum temperature or unstable node of the distillation region where the still composition currently resided. This product, whether one of the components or an azeotrope, is obtained in pure form because of the assumption of infinite number of trays. The column will continue to produce this product, with the still composition moving in a direction opposite that of the product, until the still path intersects a distillation region boundary or an edge of the composition space. At this point the product will switch to the next lowest temperature node. Figure 3 shows an example of the product sequence and a still path for a batch rectifier. It shows two different distillation regions. The column profile will follow the residue curve through the still composition (total reflux curve) until it runs into the acetone-methanol azeotrope, which is the lowest temperature node in this particular distillation region and the first distillate product The still path will move directly away from the azeotropic product until one has depleted all of the acetone from the system. The column profile will now lie along the methanol-water binary edge, with methanol being the next distillate product. The still path will move away from the product, towards the water vertex. The batch rectifier will continue to produce methanol as a distillate, until one has depleted all of the methanol from the system, at which point only water will remain in the column.

2.2 Feasible Product and Possible Column Profile Regions

Several researchers have worked on identifying the feasible product regions for continuous distillation. Wahnschafift et al. (1992) were able to predict these regions for a specified feed composition using a graphical analysis of the residue curve map of the system. They were able to map out these feasible product regions using product and feed pinch point curves. A pinch point curve is the collection of points that are tangent to a residue curve and point back through the product or feed. For the product pinch point curves, these points correspond to pinch points in the column where a vapor and liquid stream that pass each other are in equilibrium, requiring an infinite number of trays (or increased reflux ratio) to carry out the specified separation. See Wahnschafft et al. (1992) for more details. They also were able to identify the regions of possible column profiles for both column sections, given product specifications. Each column profile region is bounded by the total reflux curve (approximated here as the residue curve that passes

through the product composition) and the product pinch point curve. For a continuous column, there is a distillate and bottoms product resulting in distillate and bottoms product pinch point curves. If the rectifying and stripping column profile regions intersect in at least one point, then a tray by tray calculation can be performed from one product to the other resulting in a feasible column specification. If these regions do not intersect, then the specifications made on this column are not feasible, and this column will not carry out the desired separation. Also, the feed composition does not necessarily need to lie in any of the possible column profile regions. But the feed composition must lie on a mass balance line between the distillate and bottoms compositions.

23 Extractive Distillation Feasibility and Operation

In a continuous or middle vessel extractive column, there are three tray sections: rectifying, extractive, and stripping, shown in Figure 1 for the middle vessel column. The rectifying section is responsible for separating the intended distillate product from the entrainer, while the extractive section breaks the azeotrope. Wahnschafft et al. (1993) carried out a graphical analysis for continuous extractive distillation containing a three component mixture. As pointed out earlier, if the rectifying and stripping profile regions do not intersect, then the column is infeasible. Wahnschafft et al. showed that, with the appropriate entrainer, the extractive section can "join" a rectifying profile region with a stripping region that do not intersect. Without the extractive section, the column would be infeasible.

Wahnschafft et al. showed that there are areas in the residue curve map in which the extractive section will carry out the required separation, and areas in which the section will not work. Figure 4 shows an example of these regions for the acetone/methanol/water system, with the shading denoting areas where the extractive section will not work. If any of the compositions in the extractive section lie within these shaded regions, the column will not produce the intended distillate product, acetone in this case. The A pinch point curves mark the boundaries of these areas. The A point is a constant composition point seen by all of the trays in the extractive section. An overall mass balance for tray j in the extractive section produces the following equations:

$$V_{j-1} + E = D + L_j \tag{2}$$

$$V_{j-1} - L_j = A \quad (D > E) \tag{3}$$

$$W\ l = {}^{A} \ldots (E>D) \qquad (4)$$

where Vj and Lj are vapor and liquid flow rates from tray j and E and D are the entrainer and distillate flow rates. A is located on a line connecting E and D but outside of the composition triangle. The higher the ratio of E to D, the closer A is to E and vice versa. For example, we have a A point and some arbitrary extractive tray composition, L_k, shown in Figure 4, and we take an equilibrium step to produce V_k, the vapor coming up from this tray. Then using equation 4, the liquid coming down from the tray above this, Lfc_{+1}, must lie on a line between the V_k and A. We can repeat this analysis for tray k+1 and see that, as we move up the column towards the acetone-water binary edge, the temperatures associated with each tray decrease. In this manner, we can "connect" a bottoms profile section with a distillate profile that did not intersect before, resulting, in a feasible column specification. Figure 5 shows an infeasible column specification. The extractive section will step from the stripping profile region to the rectifying profile region. There is a minimum E in which the infeasible extractive regions occupy the entire residue curve map, resulting in no feasible space for the extractive section. The A pinch point curves are generated by finding the tangent points on all of the residue curves that lie on a line through A, as shown in Figure 4. In the regions of infeasible extractive sections shown in Figure 4, the stepping we did above proceeds in the wrong direction, one of increasing temperature as we move up the column.

3 Insights into Batch Distillation

3.1 Feasible Product and Possible Column Profile Regions

The analysis presented in section 2.2 for feasible product and column profile regions in continuous columns can also be carried out for batch distillation with some differences. These regions will only apply at one instance in time (still composition). Also, the still composition, S, is like any other tray composition (when holdup effects are ignored) and must lie on the column profiles from each product, while in continuous distillation, the feed tray composition does not have to lie on the same column profiles as the products. For the batch rectifier, there is only one product, the distillate. Shown in Figure 6, the feasible product region is bounded by the residue curve through the specified still composition S (total reflux curve) and the tangent to the total reflux curve through S. The tangent to the total reflux curves gives the distillate compositions whose column profiles will pinch at S (product pinch point curve ends at S). The shaded region

for the batch rectifier shows all of the possible distillate products for the specified still composition. Figure 6 also shows the feasible product region for a batch stripper column, which is found in a similar manner as above.

The feasible product regions for the middle vessel column can also be found in the same way as the batch rectifier and stripper. The middle vessel column is basically a batch rectifier on top of a batch stripper, with only the still pot in common. The same arguments made above for the rectifier and stripper feasible products apply for the rectifying and stripping sections of the middle vessel column. But while in continuous distillation the distillate; feed, and bottoms compositions must all lie on the same mass balance line, these compositions do not have to lie on a mass balance line due to the dynamic behavior of die column. However, if the products do lie on a straight line through S and remain there, the still composition will not change, resulting in a constant composition column. So Figure 6 also shows the feasible product regions for the middle vessel, by adding the rectifier and stripping regions together. Again, this only applies at the current still composition, S.

The regions of possible column profiles for the batch distillation are found exactly as for continuous distillation in section 2.2. For each specified product, the region of possible profiles is bounded by the total reflux curve through that product and the product's pinch point curve (see Wahnschafft et aL, 1992). Figure 5 shows these regions for a specified distillate and bottoms product. These profiles will again only apply at the current product compositions. If the products change, the regions of profiles will change. And as mentioned earlier, for a column specification to be feasible, the distillate and bottoms profile regions must intersect in at least one point. However, for batch distillation, there is one more necessary condition for the column specification to be feasible. S must lie on the column profile from D and from B. S must therefore be contained in the intersection of the two column sections.

An example of an infeasible middle vessel column specification can be found in Figure 5. Here, the distillate and bottoms products have been specified as D and B with still composition S. Also shown are the regions of possible column profiles for each product. These regions are bounded again by the total reflux curve through each product and the product pinch point curve. S

in Figure 5 is contained in the region of possible column profiles for B, so the bottom section of the column is feasible. However, S is not included within the region of possible column profiles for D. It is not possible to perform a tray by tray calculation from S to D, resulting in an infeasible distillate product specification. For the column to be feasible, the rectifying and stripping profile regions must intersect, and S must be contained in that intersection.

3.2 Steering the Middle Vessel Still Composition

As mentioned earlier, it is possible to separate a three component mixture into its pure components using a middle vessel column. This was due to the accumulation of the intermediate component in the still But column parameters (e.g. product withdrawal rates) must be chosen in such a manner that the still composition does accumulate in the intermediate component As the still path for the batch rectifier was a function of the distillate and still compositions, the still path for the middle vessel still path is a function of the distillate, bottoms, and still compositions. From the overall component mass balance:

$$\frac{d}{dt}(Hx_s) = -(Dx_d + Bx_b) \tag{5}$$

we see that the direction of the still path is in a direction opposite that of the combined directions of x_s to x_d and Xg to x_b. How these directions are combined are determined by the magnitude of D and B, using vector addition. So depending on the magnitude of the product flow rates, it is possible to "steer* (he still composition in a variety of directions. For example, Figure 6 shows the residue curve map of pentane/hexane/heptane. In a middle vessel column with infinite reflux and reboil ratios and number of trays, pentane will be the distillate product, while heptane will be the bottoms product. The directions DD and DB show the directions the respective product withdrawals force the still composition to move. The region of possible directions is between DD and DB.

Another way to look at the direction of the still path is to combine the distillate and bottoms product into a "net product", again depending on the magnitudes of the product flow rates. In Figure 6, if the net product was the intersection of the dotted line from S to the pentane-heptane binary edge, the instantaneous change of the still path would be in a direction directly opposite that of the net product, exactly as was seen in the case of the batch rectifier. This would

point the still path at the hexane vertex. So if the product flow rates were continually adjusted in such a manner that this net product drove the still path towards the hexane vertex, only hexane would be left in the column, operating at total reflux and infinite number of trays, after all of the pentane and heptane were removed as distillate and bottoms products, respectively.

So by steering the still composition towards the intermediate component, it is possible to produce three relatively pure products using only one column. Steering the still composition also shows the flexibility of the middle vessel column, in that by setting the bottoms withdrawal rate to 0, the column now acts like a batch rectifier, and vice versa for a batch stripper. The middle vessel column can be used as a rectifier, stripper, or middle vessel column, depending on the particular separation needed. This flexibility makes the middle vessel column an excellent choice for equipping a batch separation system.

33 Batch Extractive Distillation

While continuous distillation will have a constant A point, batch distillation has a constantly changing A point due to changing still and produa compositions and flow rates. So it is possible for the batch extractive column to work for a period of time, but then stop working because the still composition has intersected the A pinch point curves. When this happens, the extractive section can no longer perform its required separation task, and the column will not produce the desired product. Yatim et al. (1993) simulated a batch extractive distillation using a rectifier, and also compared the results to experimental data they collected. They mention that they were able to recover approximately 82% of the distillate (acetone) from an azeotropic mixture with methanol, using water as an entrainer. The distillate that was obtained was approximately 96% acetone. In Figure 4, their A point would lie along the acetone-water binary edge, but outside the composition triangle near the water vertex. S marks the initial still composition they used. As mentioned earlier, the still path for a batch rectifier is a function of the distillate and still compositions. But now that there is an entrainer feed, the still path is also a function of the entrainer composition and flow rate. The direction of the still path will be some combination of the distillate withdrawal driving the still composition directly away from the acetone vertex (DD in Figure 4) and the entrainer pulling the still composition towards the water vertex (DE in Figure 4) as water is continually added to the system and never removed. Since the

entrainer addition is normally several times greater than the distillate withdrawal, the direction of the still path is more towards the water vertex. Yatim et al. were able to draw off a marly constant composition distillate product for the main operational step. However, their A point and A pinch point curves will only be constant if their entrainer and distillate flow rates were also constant If we were to assume that these flow rates were constant, their still path would eventually intersect the A pinch point curve from the methanol vertex to the acetone-water binary edge. At this point, the extractive section would no longer be feasible, and methanol would come over in the distillate product. This could be one explanation of their limited acetone recovery of 82%. The intersection of the still path and A pinch point curve can be overcome in the rectifier by increasing the entrainer to distillate flow rate ratio, which will move the A pinch point curve towards the methanol-water binary edge. At an infinite entrainer to distillate flow rate ratio, the A pinch point curve will lie exactly on the methanol-water binary edge. In this case, it is theoretically possible to recover all of the distillate product. However, increasing the entrainer flow rate will also increase the size of the still pot that is required, because the entrainer, water in this case, is never removed from the system and only accumulates in the still pot So a 100% distillate recovery requires an infinite size still pot

The middle vessel column can be used to overcome many of the problems that the batch rectifier has in extractive distillation. The still path is now a function of the distillate, bottoms, still, and entrainer compositions. The still path direction will be a combination of the distillate and bottoms withdrawal driving the still away from the respective products and the entrainer addition pulling the still towards the entrainer. In this case, the bottoms product will be the same as the entrainer, because the bottoms will be recycled back as entrainer. If the entrainer addition and bottoms withdrawal are exactly the same, the net still path will move directly away from the distillate product, eventually intersecting a A pinch point curve. However, if we use the still pot steering ideas described earlier, the addition and removal of entrainer could be adjusted so that the still path never intersects the A pinch point curves. A 100% recovery of the distillate product is theoretically possible in a three component mixture when the still path is steered towards the intermediate component. In reality however, a 100% recovery would not be feasible due to high number of trays, long processing times, and high reflux. But the ability to steer around these A

pinch point curves can be very helpful in increasing the distillate product recoveries. Also, because the entrainer is continually pulled off the column, the still pot will not accumulate with the entrainer like the batch rectifier. The still pot size will remain small with decreasing capital costs resulting.

4 Simulation Results

We simulated both the batch rectifier and middle vessel columns using water as the entrainer for the azeotropic mixture of acetone and methanol. Both models ignored holdup effects and a pseudo steady state was assumed on all of the trays except for the still. We used Wilson correlations in modeling the thermodynamics of the system. We integrated the columns using ASCEND (Piela et. al, 1993), an equation based modeling system, and the integrator LSODE (Hindmarsh, 1983).

We broke the distillation operation for the middle vessel column into three sections: a period of entrainer (water) addition with no entrainer removal but with distillate (acetone) removal, period of normal distillate and bottoms (water) removal with the bottoms recycled back as the entrainer, and a period with no entrainer addition and distillate product with the intermediate component (methanol) coming out in the distillate. However we show results for the second step only because this is the main operational step. The initial still contains ISO moles each of acetone and methanol before the first operational step. Figure 7 shows the product compositions versus time for the middle vessel extractive column during the main operational step. The distillate was ~96% acetone as seen in Yatim et al. (1993) and the bottoms was ~99% water, which is recycled back as the entrainer addition. The still product, methanol, was obtained at the end of the distillation because 99.5% of the acetone was recovered as the distillate product and all of the water was removed as the bottoms product. Again, we see that it is possible to separate a three component mixture using only one column. Steering the still pot made this possible by avoiding the infeasible extractive section regions of the composition space. In order to properly steer the still, the following equation was added to the model:

$$\frac{D x_d\,(\text{acetone})}{x_{c_s}\,(\text{acetone})} = \frac{(B - E)\, x_e\,(\text{water})}{x_{c_s}\,(\text{water})} \tag{6}$$

where D, S, B, and E denote the distillate, still, bottom, and entrainer compositions and flow rates. Equation 6 calculates the bottoms flow rate so that the still path is directed towards the methanol vertex, away from the infeasible extractive regions. It also assumes that there is a negligible amount of water in the distillate, negligible amount of acetone in the bottoms and entrainer, and the entrainer and bottoms compositions are the same. But while the three components were separated into relatively pure components, we observed large reflux and reboil ratios and diminishing distillate flow rates resulting in long processing times.

Figure 8 shows the still path for the middle vessel column. Notice that the still path is continually planted directly at the methanol vertex. Also shown is the still path for the latch rectifier that was simulated. All conditions and column parameters for the extractive and rectifying sections of the middle vessel column were repeated for die batch rectifier. The still pattr for the rectifier does reach the methanol-water binary edge. The still path never went into an infeasible extractive region because as the still path continually approached the methanol-water binary edge. We decreased the distillate flow rate in order to meet the distillate product specification. This moved the A point which moved the A pinch point curve towards the methanol-water binary edge, until the A pinch point curve coincided with the binary edge. Also, the final still composition is not pure methanol as in the middle vessel column. The mixture will have to be processed further. Figure 9 shows the still holdup versus time for the middle vessel column and the batch rectifier. The holdup for the middle vessel column decreases continually until it reaches approximately 150 moles, the initial amount of methanol in the column. But the holdup for the rectifier increases during the entire operation, ending at an amount almost 13 times that of the middle vessel column. The middle vessel column not only kept the required still pot size small, it also separated the mixture into all of its pure components.

5 Conclusions and Future Work

We showed that one can use previous work in the analysis of continuous distillation for batch distillation. In particular, we used the work of Wahnschafft et al. (1992) to find the regions of instantaneous feasible products for the batch rectifier and stripper and middle vessel columns. From this we showed that the still in the middle vessel column can be steered in many directions

by appropriate choices of various column parameters, normally the product withdrawal rates. We were also able to show the regions of possible column profiles for the specified distillate and bottoms products, and that the still composition must lie in the intersection of these regions for the middle vessel column to be feasible.

We also extended the work done for continuous extractive columns by Wahnschafft et al. (1993) to include the batch rectifier and the middle vessel column. We were able to show graphically one explanation for the limited recovery of the distillate product seen in the work of Yatim et al. (1993). We suggest it may be due to the columns' extractive section becoming infeasible during the column operation. Howeyer, the capability of steering the middle vessel column's still composition enabled the theoretical 100% recovery of the distillate product without an infinite size still pot, as seen in the batch rectifier. We steered the still composition around thcA pinch point curves which limited the distillate recovery. Simulations of the middle vessel column and batch rectifier showed that a near 100% recovery is possible in both columns, but steering the middle vessel column's still path enabled the mixture to be separated into its pure components with a much smaller required still pot size.

While the steering of the still path in the middle extractive vessel column does determine the optimal entrainer withdrawal to addition ratio, there are many other parameters that need to be optimized. We need to investigate the sensitivity of parameters such as reflux and reboil ratios, number of trays, and product withdrawal rates, as well as the optimization of this column and its entire operation.

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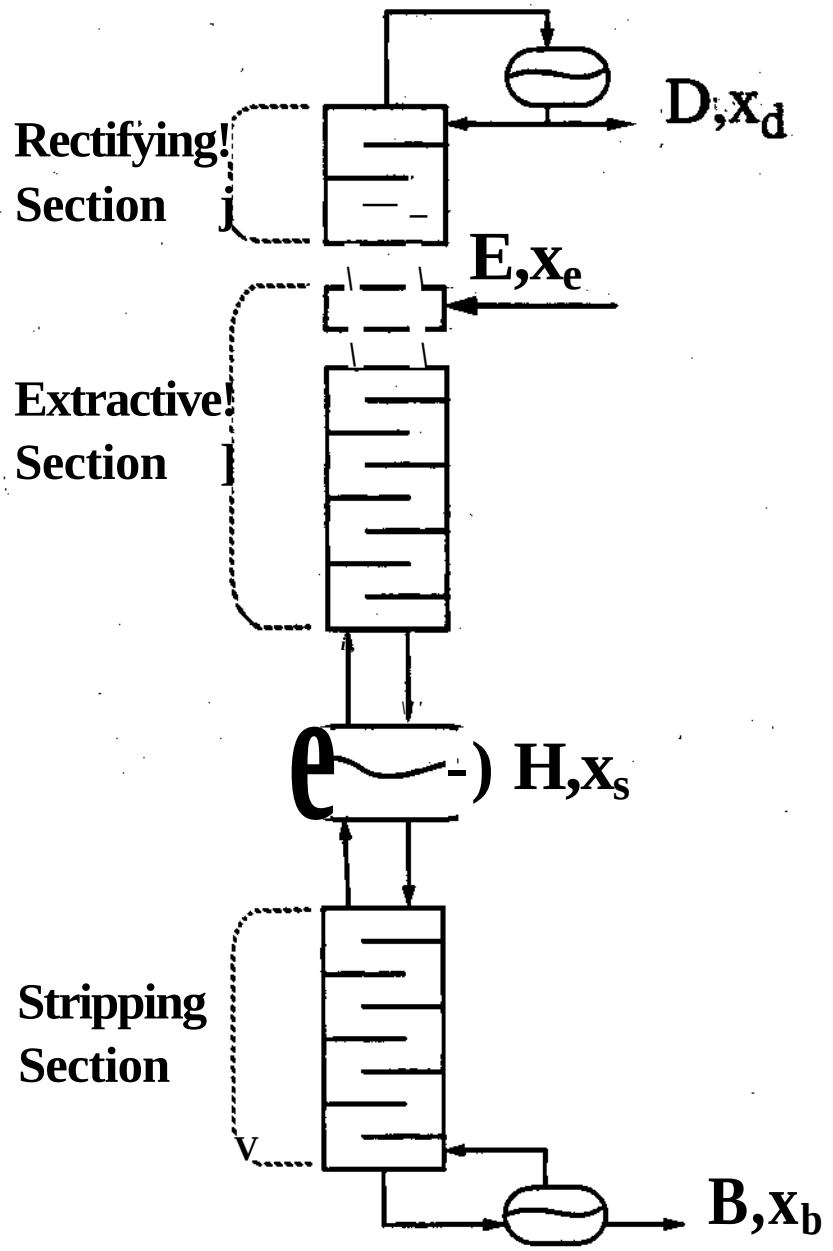

Figure 1: Middle Vessel Column with Extractive Section

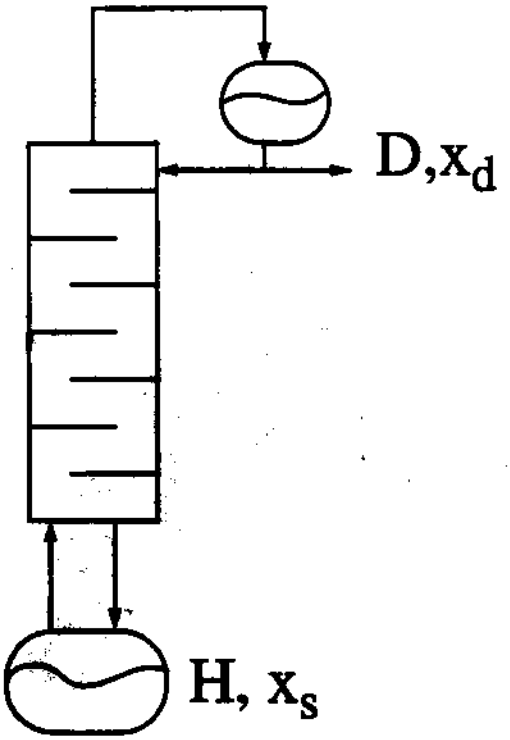

Figure 2a: Batch Rectifier

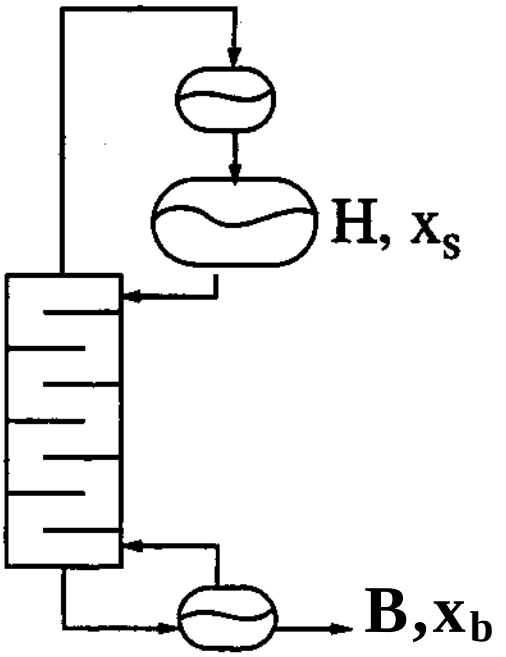

Figure 2b: Batch Stripper

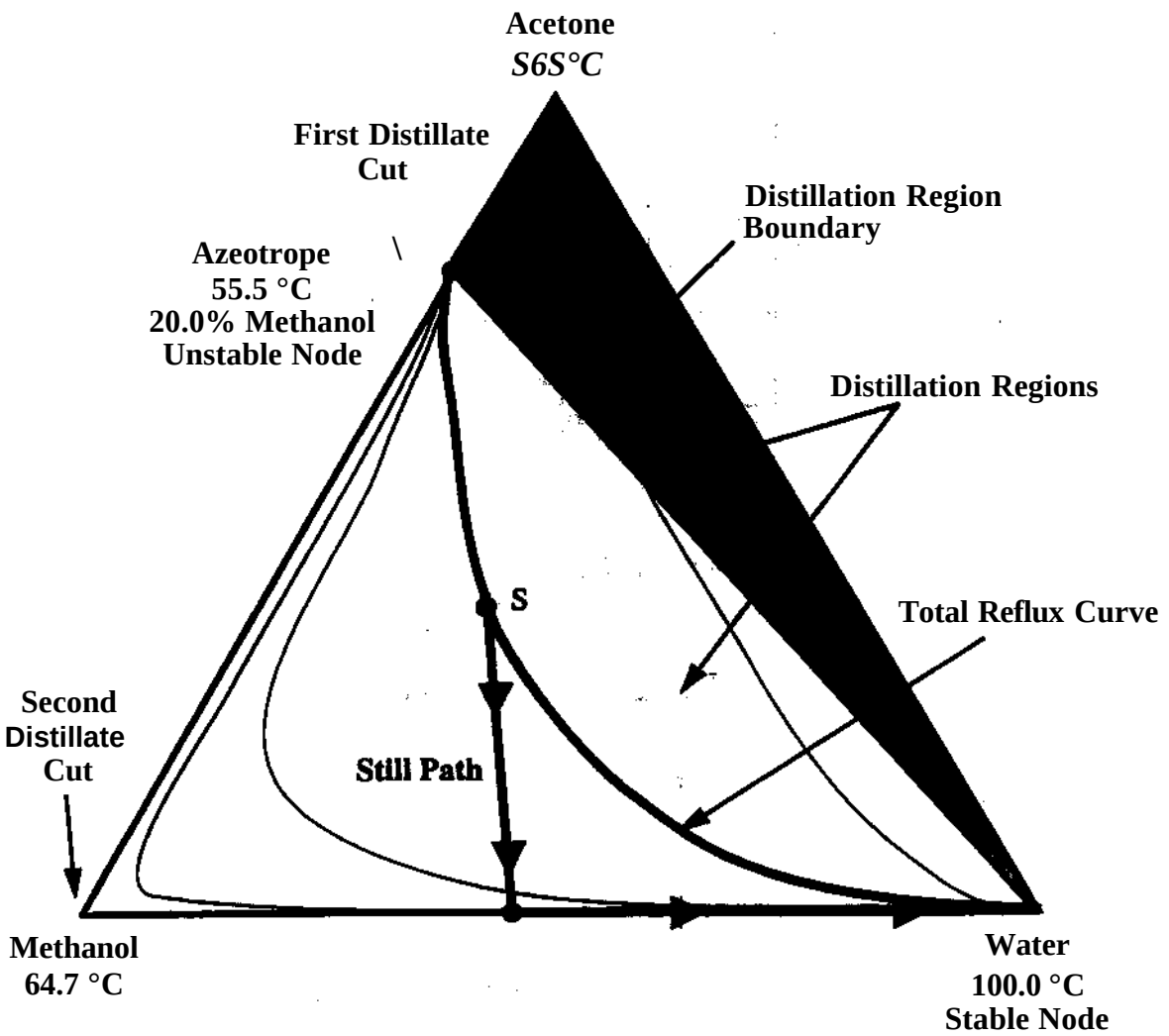

Figure 3: Product Sequence and Still Path for Batch Rectifier at Infinite Reflux and Irays

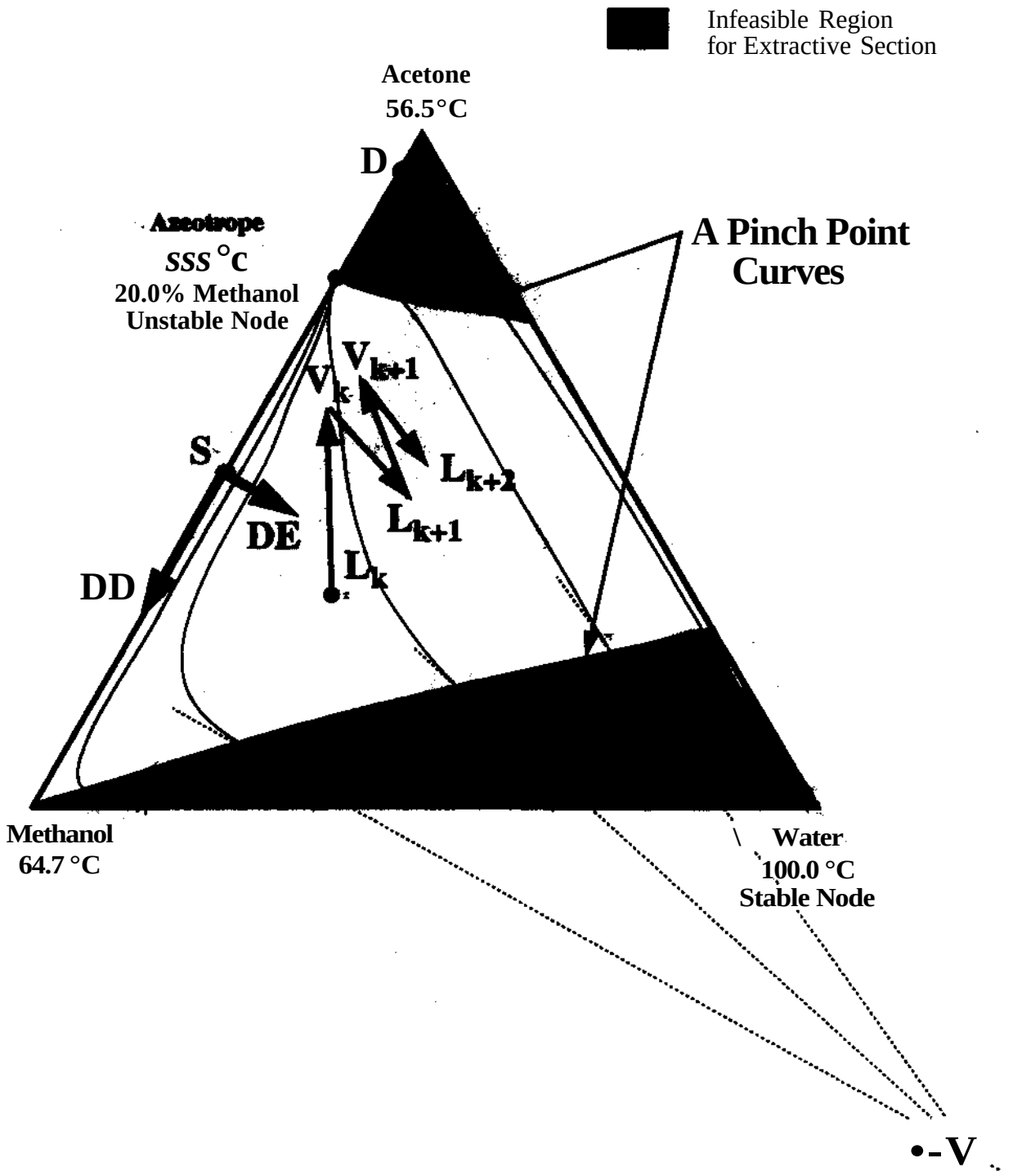

Figure 4: A Pinch Point Curves for Acetone/Methanol/Water

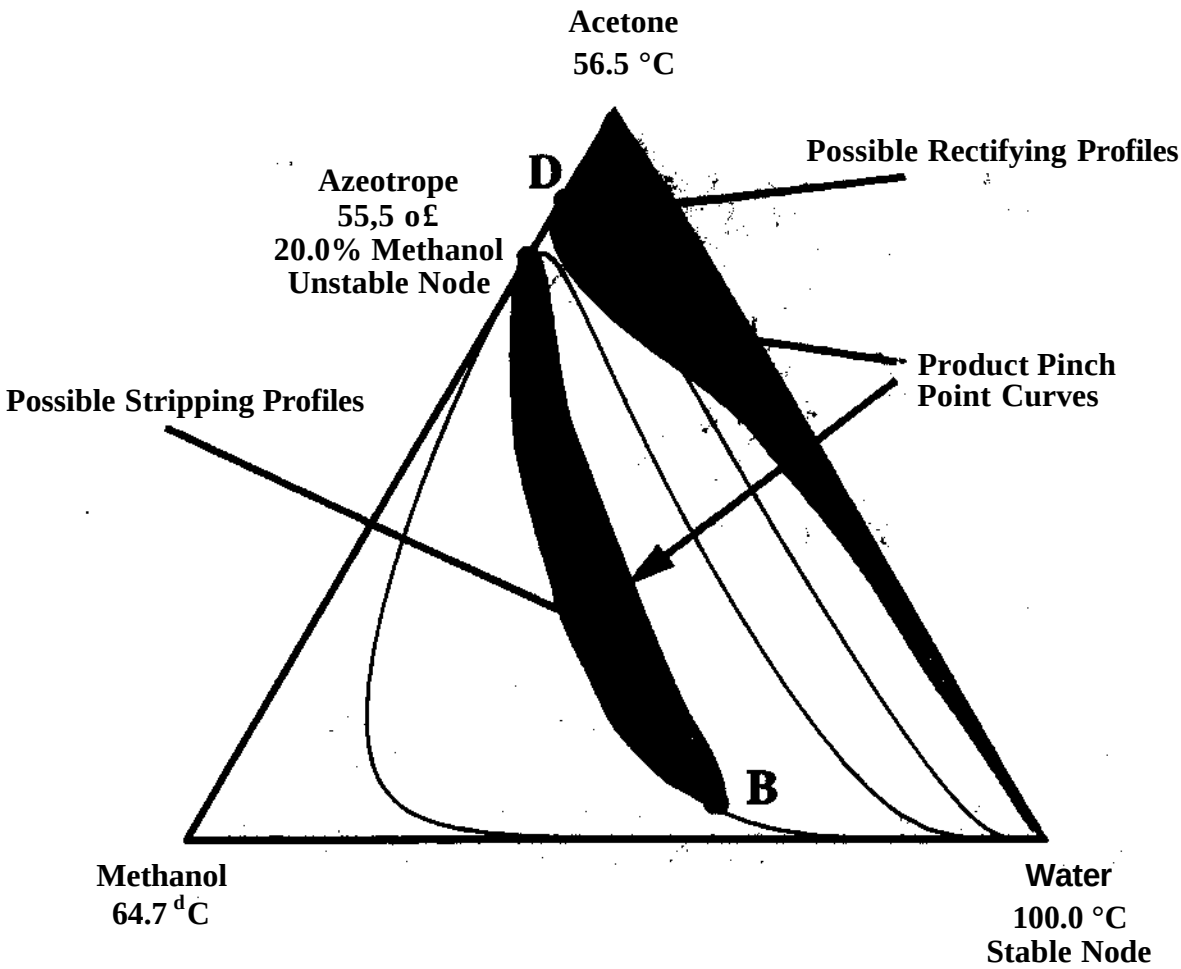

Figure 5: Infeasible Middle Vessel Column Specification

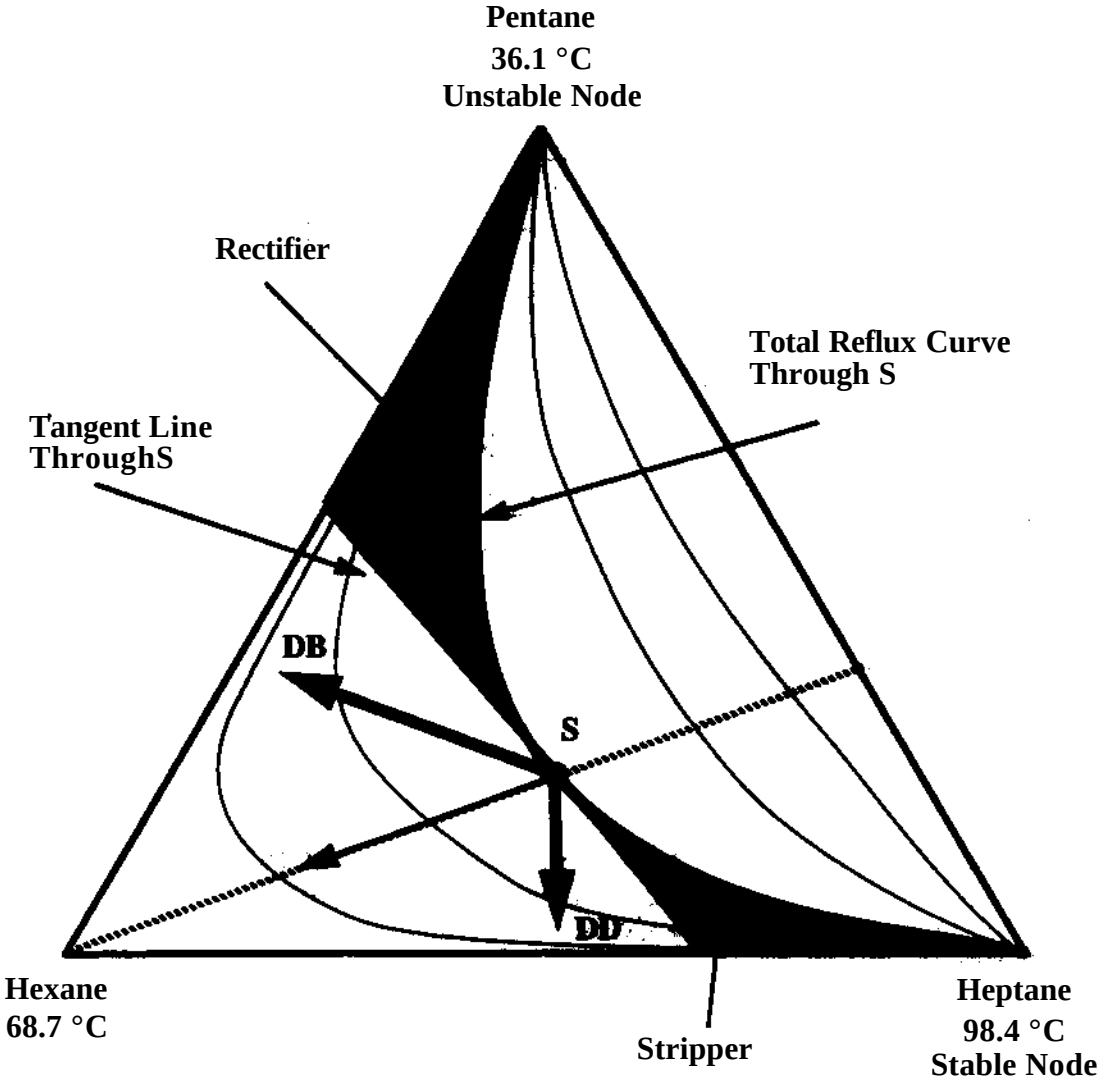

Figure 6: Feasible Product Regions for Batch Rectifier and Stripper

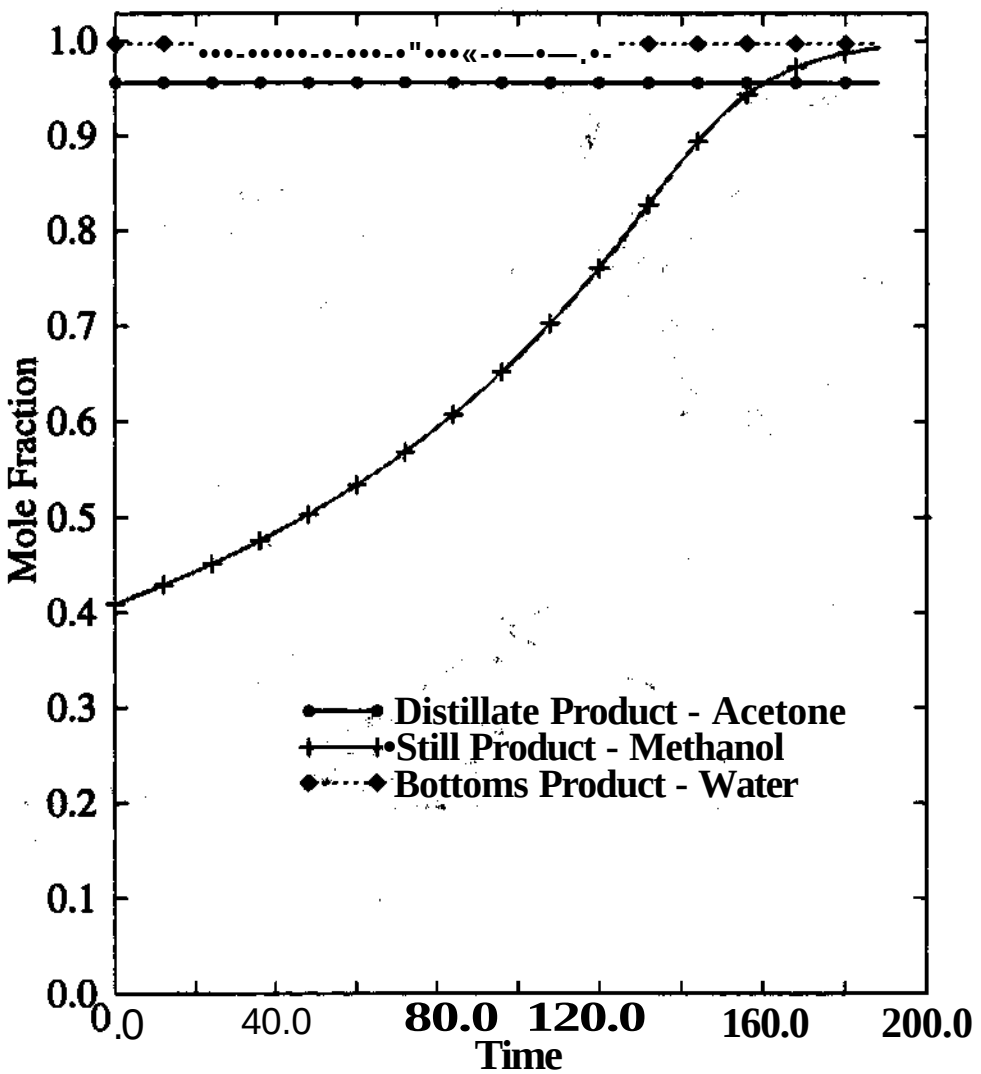

Figure 7: Products from Extractive Middle Vessel Column

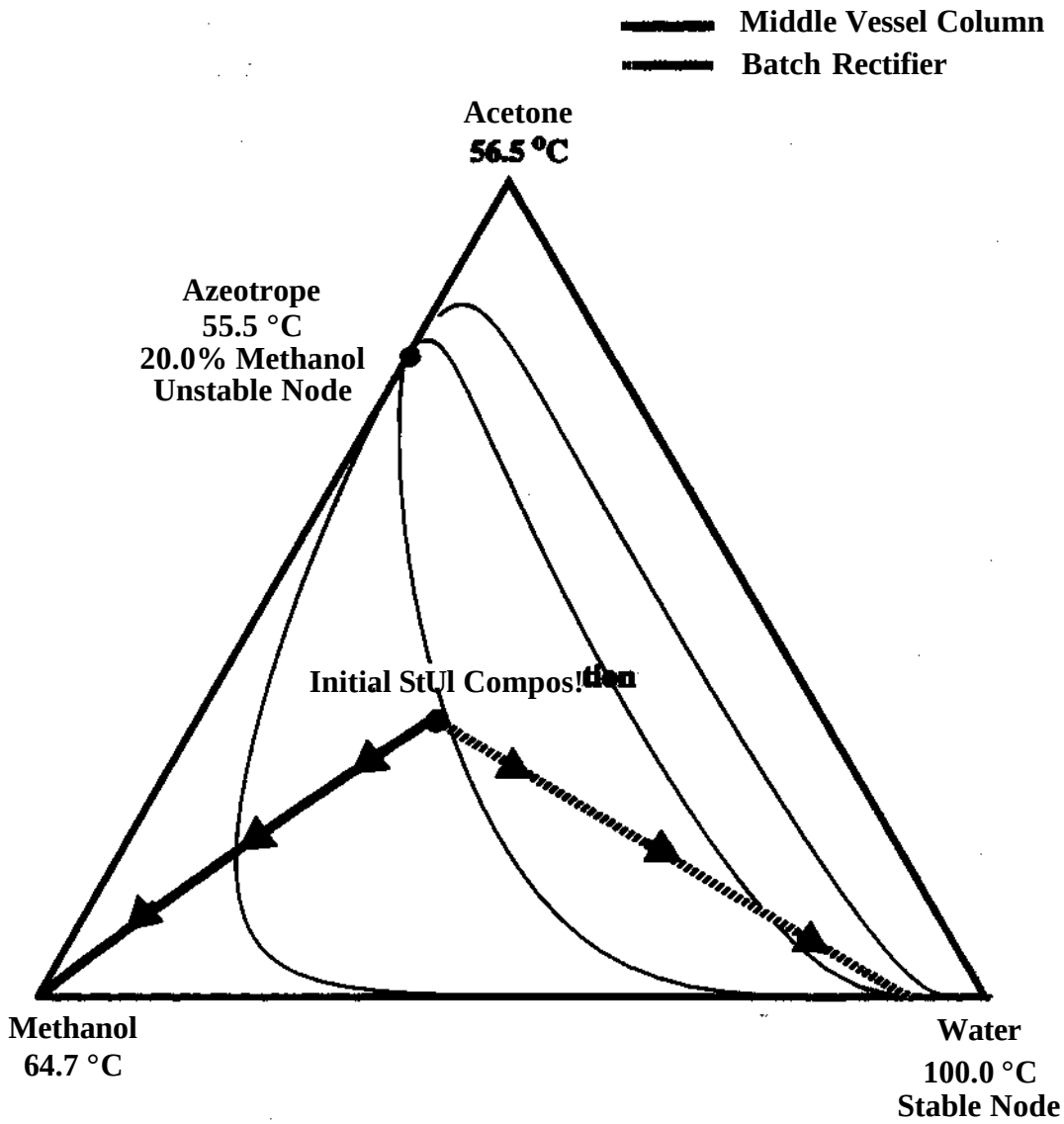

Figure 8: Still Paths for Extractive Batch Columns

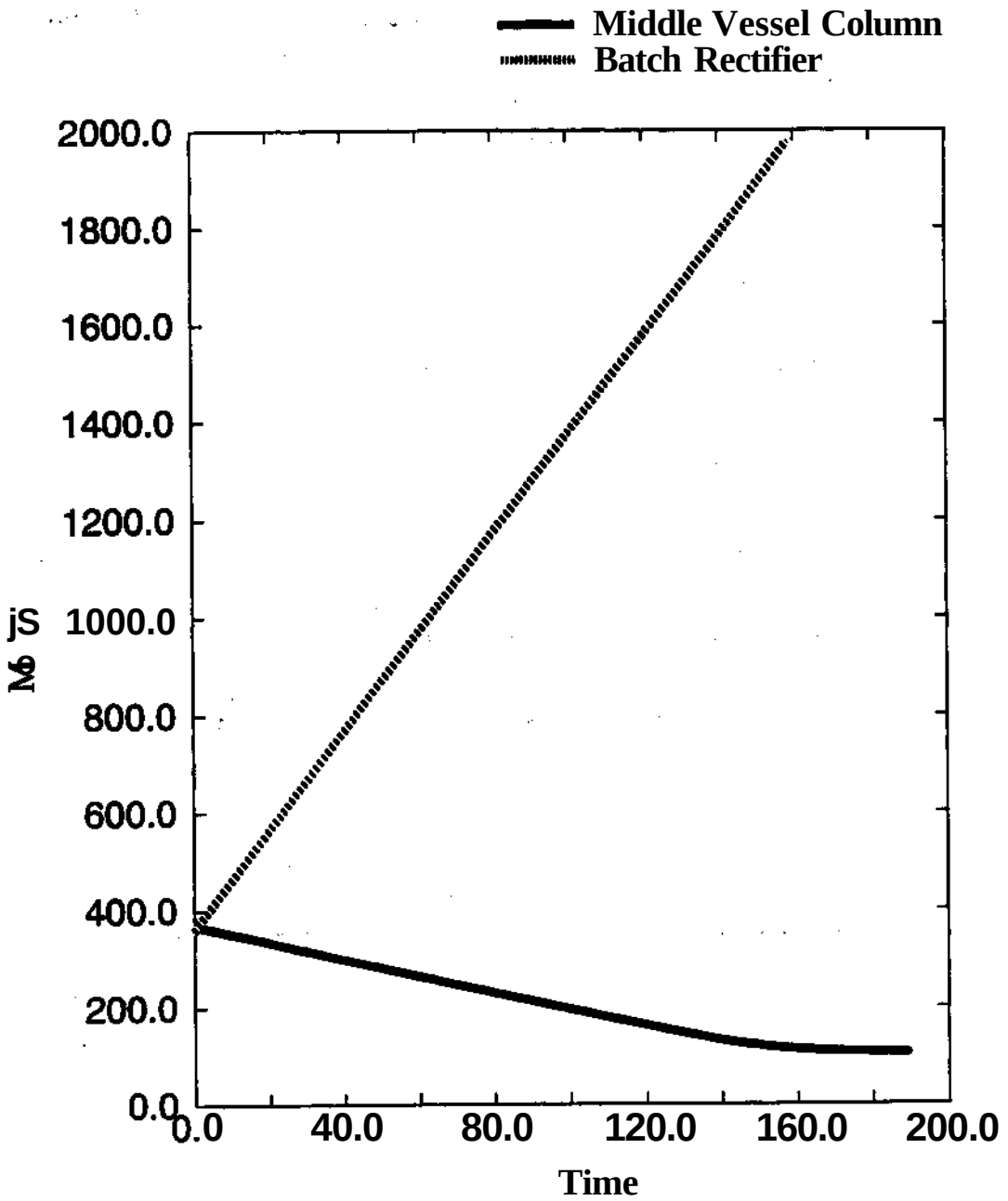

Figure 9: Still Pot Holdups in Extractive Batch Columns