EFFECT OF HIGHLY AND LEAST ADSORBED COMPONENT ON SANDWICHING RECOIL CHROMATOGRAPHIC SEPARATION

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The two kinds of effects due to least and highly adsorbed components that limits the separation efficiency of target components in sandwiching recycle are examined. It is illustrated that the strongly adsorbed component interferes the product as the transported impurity caused by successive injections and hence makes the width of feed inputs significantly narrow. There is no complication due to least adsorbed component as it tends to accumulate as unseparated impurity in the product can be easily eliminated by optimizing the recycle periods without affecting the feed intake capacity of the process

INTRODUCTION

There is a growing interest on chromatography as a means of separation in pharmaceutical and biochemical industries. In this connection, we have investigated a new recycling technique called as single-column sandwiching recycle technique (Ghimire and Ishida, 1996) and shown its potentiality for the separation of middle component from a three-component mixture. We have further explored this potent idea by combining two columns in series. It has been observed that the production rate of the target component in the product can be increased by the application of two column sandwiching recycle chromatography (Ghimire and Ishida, 1996).

In the present investigation, we have examined the possible effects taking place in sandwiching recycle. There are mainly two kinds of effects that influences on the impurities in the product. One of them is transported impurity. The strongly adsorbed components proceed very slowly in the column and are easily reached by the target component introduced in the next injection, resulting in their accumulation in the product. On the other hand, those components which are least adsorbed also to some extent affects the separation process and this kinds of impurity is called as unseparated impurity. In the previous paper, we have demonstrated the effectiveness of sandwiching recycle. It is assumed that the target component diethyl phthalate is separated from the more-adsorbed or the less-adsorbed component. When some of the component in the mixture is either least or strongly adsorbing properties, there may be significant effect on the separation performance. The purpose of this paper is to examine the effects of those far neighbor components on the separation performance of target component.

COMPARISON OF THREE AND FIVE COMPONENT CHROMATOGRAM

Our main objective is to separate the target component B (diethyl phathalate) from a three component mixture containing A (di-n-butyl phthalate) and

C (dimethyl phthalate). The single and two-column sandwiching recycle apparatus is used to separate the intended component and a volumetric ratio of 3:2 mixture of hexane and ethyl acetate as eluent. The superficial velocity is set at 1.667 x 10^{-3} m/s and the column used for separation are 40 cm. long having 400 number of plates. The distribution coefficient and capacity coefficient of the mixture have been obtained experimentally as shown in Table 1 and the agreement between experiment and simulation has been confirmed (Hatanaka and Ishida, 1992).

Figure 1 is the schematic flow diagram of two-column sandwiching recycle apparatus and the chromatogram obtained for the separation of middle component is shown in Fig. 2 (Ghimire and Ishida, 1996). Under these conditions the yield, productivity and consumption of eluent fractions are 0.901, 0.392 and 0.351, respectively. This condition is the optimal feed intake and product achieving situation from the corresponding two-column sandwiching recycle apparatus.

However, if the target component is mixed with the far neighbors, say, least and strongly adsorbed components, the separation becomes relatively difficult. To incite such effect we have chosen arbitrary distribution coefficient values of those least and strongly adsorbed components as shown in Table 2. Under this condition the yield productivity and the fraction of eluent consumption are 0.849, 0.285 and 0.526, respectively.

In comparison the yield has been decreased by 0.05 and the productivity as 0.107. Similarly the fraction of eluent has been increased by 0.176. In general this reduction in yield is due to presence of strongly adsorbed component and there is not much contribution of least adsorbed component in particular for this effect can be seen in a typical chromatogram in Fig. 3. Initially, there was no presence of strongly adsorbed component within the product period. But during recycling, it has been transported to the product period as the strongly adsorbed component came out from the column outlet lately. This tendency made the usage of more eluent to elute it out from the column.

SEPARATION PERFORMANCE FOR A THREE-COMPONENT FEED

The effect on yield and productivity at various number of plates for the separation of the target component for a three component mixture is demonstrated in Fig. 4. The effectiveness is compared by considering four separate sets of equipment with different number of plates. As usual, increasing the number of plates of a chromatographic system is a way to increase the separation efficiency. For this reason we have compared the results obtained from simulation of those four individual sets containing 100, 200, 400, 800 number of plates per column. Since 400 number of plates need 40 cm., it is assumed that 100, 200 and 800 plate columns are of 10 cm., 20 cm., and 80 cm., respectively.

A comparison among various sets of recycling apparatus with different column length is made so as to recognize their efficiency. This comparison is simply done with the idea that several shorter single and two-column apparatus set can be operated instead of a longer column. For this reason, we have demonstrated the operated instead of a longer column. For this reason, we have demonstrated the individual and a collective productivity value for each plate number equivalent to the number of sets corresponding to that particular plate numbers. As for example in the case of 200 plate numbers in Fig. 4, the individual productivity is 0.3097. Therefore, with four sets of such equipment productivity equivalent to 1.238 can be obtained collectively. Thus the values mentioned in the Fig. 4 describes both the individual as well as collective productivity value for each of them. In the latter case, the results obtained from a 800 plate number column is compared with two sets of column containing/400 number of plates.

It is observed that the two column sandwiching recycle is found to be more effective as compared to single one. A large production rate can be achieved by using four sets of shorter columns each containing 200 number of plates as shown in Fig. 4. Alternatively, the individual productivity value can also be increased by increasing the number of plates of the single-column in sandwiching recycle system.

SEPARATION PERFORMANCE FOR FIVE-COMPONENT FEED

Let us suppose that there are five components namely, A^-, A, B, C, C^+ components in a mixture and our interest is to separate the middle component B. In this case the component to be separated is in association with the two far neighbors, the most strongly adsorbed component C^+ and the least adsorbed component A^-.

In the following discussion, we examine the separation efficiency of the single and two-column apparatus by considering the distribution coefficient value as has been mentioned in Table 2. This value is chosen for the fact that one third feed input is accepted by the two-column apparatus under such condition. A chromatogram obtained at such condition for single column case is shown in Fig. 5 and for two-column case in Fig. 3. The productivity has been steeply decreased as compared to the case as has been mentioned in Fig. 1, when there is no presence of far neighbor components. The individual productivity for the component B in the product is 0.392 from two-column recycling apparatus while after the presence of most strongly adsorbed neighbor, it is declined to 0.285 as shown in Fig. 6. It means that the production rate under the influence of a strongly adsorbed component is significantly decreased.

As in section 2, a comparison among different sets of recycling apparatus of same column length and plate number is made for five component mixture. It is observed that the apparatus containing 400 plate numbers of two sets of two-column recycling is the best situation as it has the highest collective productivity, 0.57006, while the individual productivity is 0.285 can be seen in Fig. 6. However, there is not much complication due to least adsorbed component and can be optimized without affecting much the production rate of the target component.

CONCLUSION

(1) The strongly adsorbed component is transported during each successive injections and thus interfered the separation.

(2) There is no additional complication due to least adsorbed component on separation and can be easily optimized to exclude it from the target component.

(3) The separation efficiency of two column apparatus with respect to single one is high but the yield observed is low. Thus there is a choice of operation either for high yield or productivity value.

(4) Usage of relatively shorter columns increases the separation performance of sandwiching recycle.

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ANNEX

Table-1: Characteristics of Column for Three-component Mixture

Key	Compound	Capacity Coefficient [1/s]	Distribution Coefficient [-]
A	Di-n-butyl phthalate	1×10^2	0.65
B	Diethyl phthalate	1×10^4	1.034
C	Dimethyl phthalate	1×10^6	1.55

Table-2: Characteristics of Column for Five-component Mixture

Component	Capacity Coefficient [1/s]	Distribution Coefficient [-]
A^-	1×10^2	0.21
A	1×10^2	0.65
B	1×10^4	1.034
C	1×10^6	1.55
C^+	1×10^6	2.3

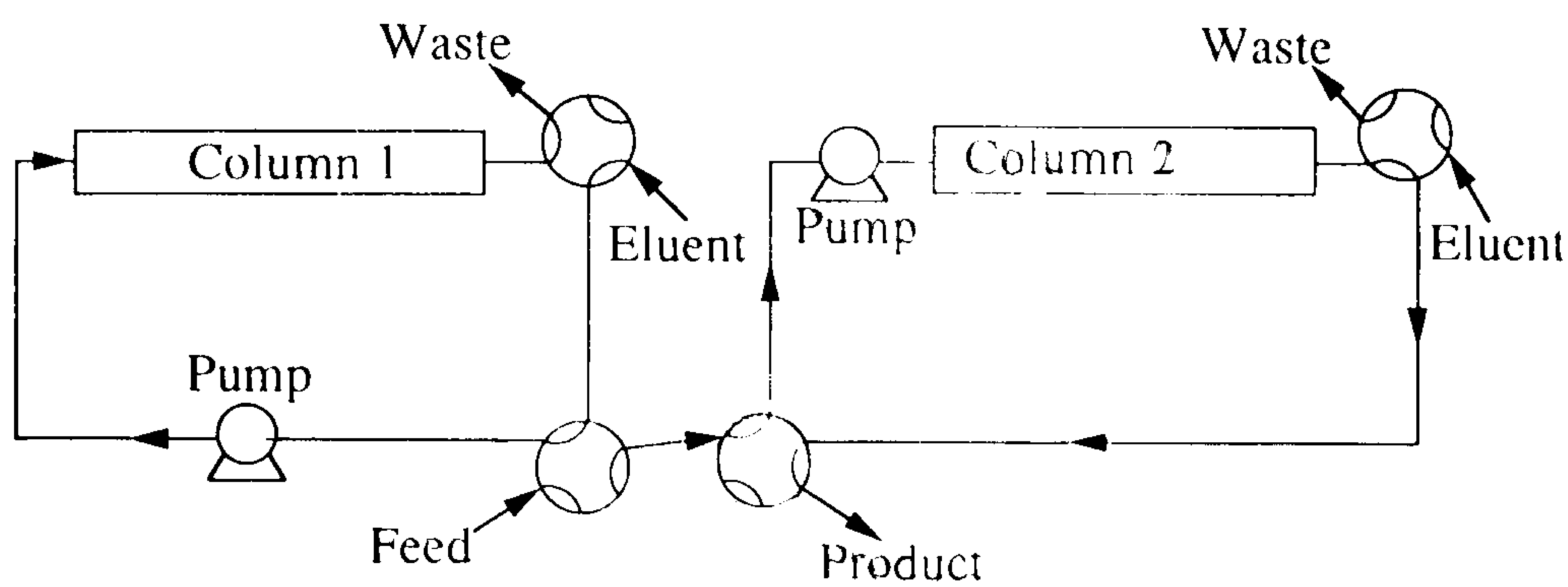


Figure-1: Schematic Flow Diagram of Recycling Chromatograph

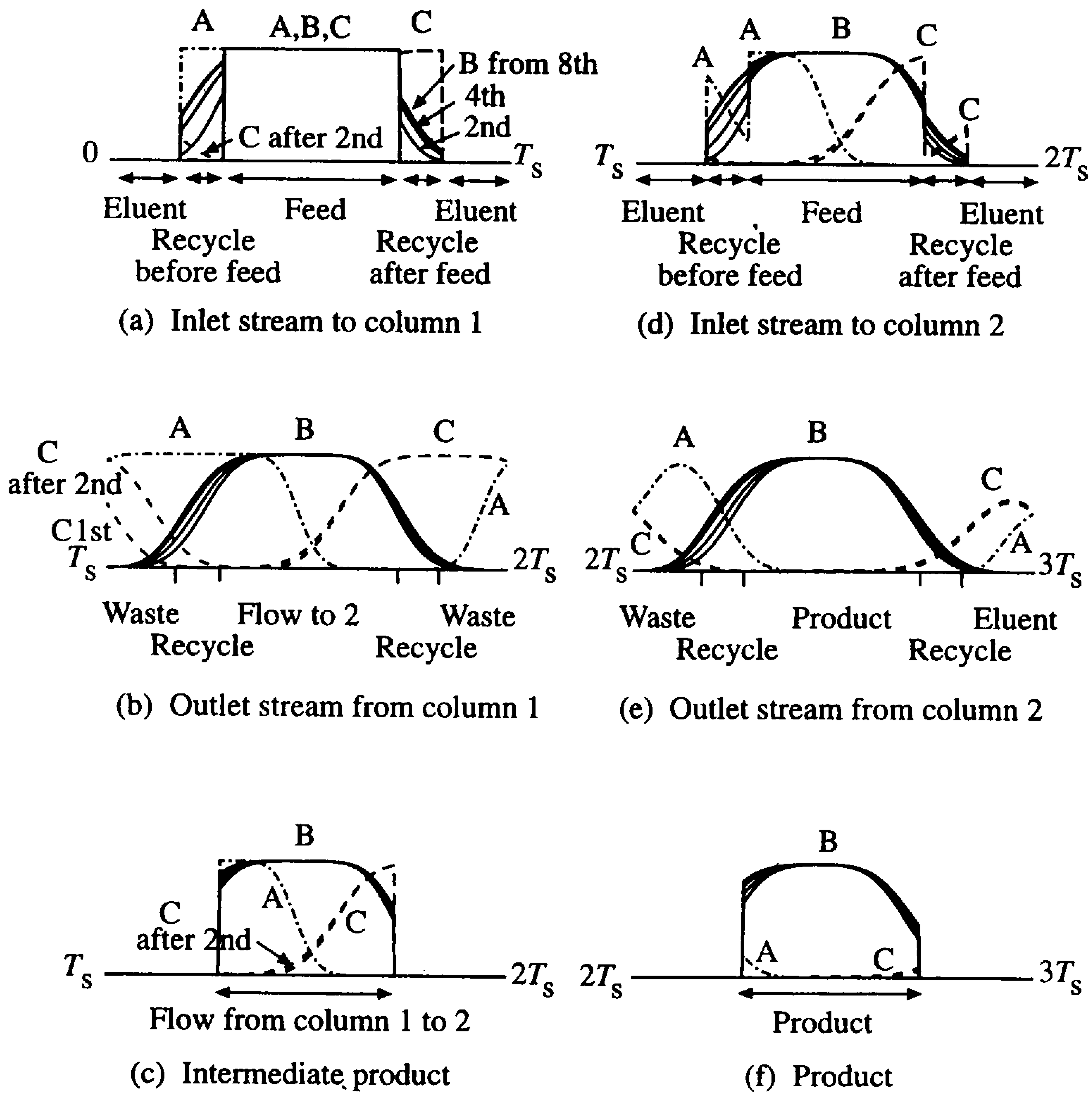
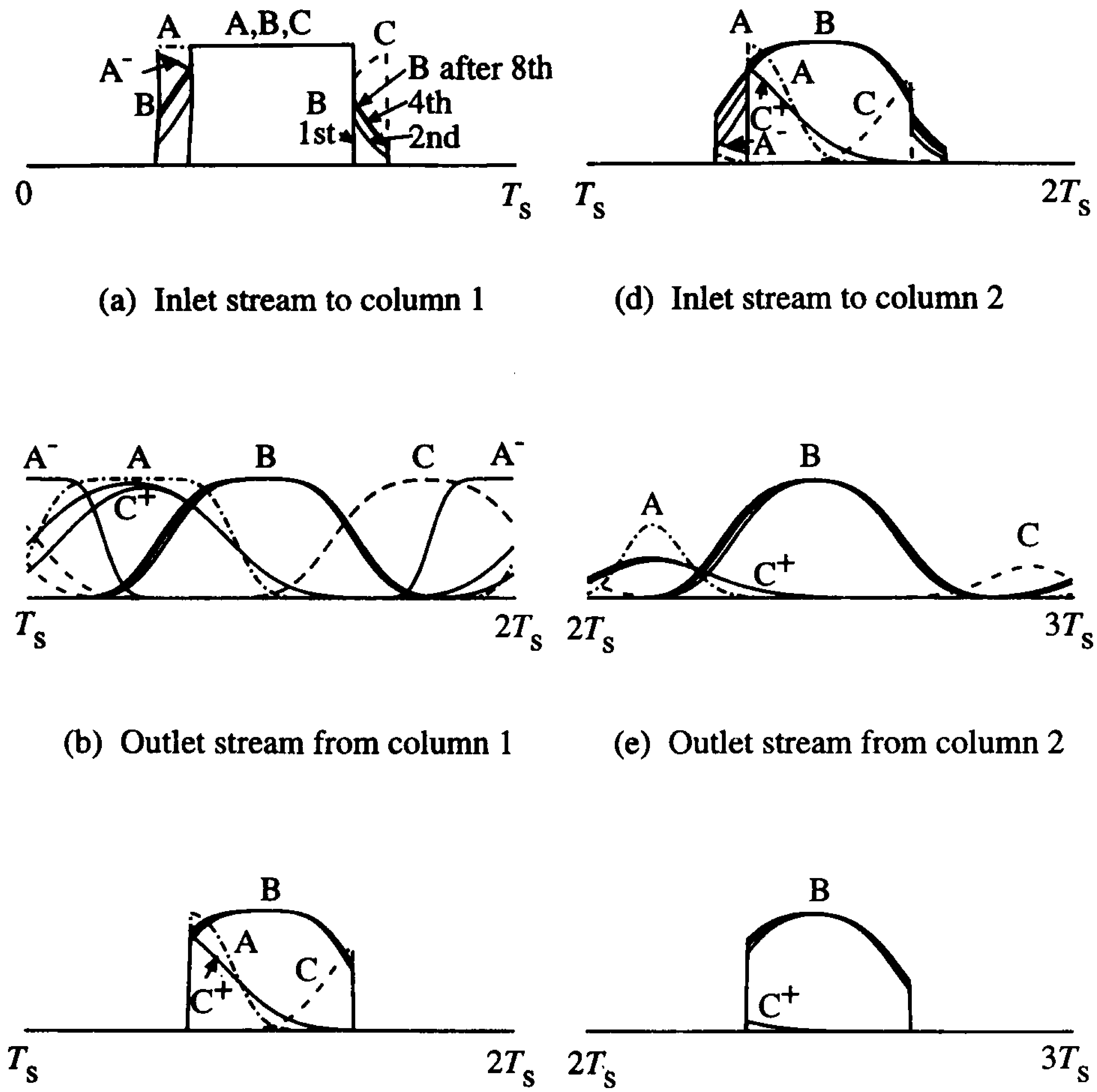


Figure-2: Separation of Target Component from a Three-component Mixture, T_s=249, T_f=108, T_{r1}=T_{r2}=26.6



(a) Inlet stream to column 1

(d) Inlet stream to column 2

(b) Outlet stream from column 1

(e) Outlet stream from column 2

(c) Intermediate product

(f) Product

Figure-3: Separation of Target Component from a Five-component Mixture
T_s=249s, T_f=108s, T_{r1}=T_{r2}=17s

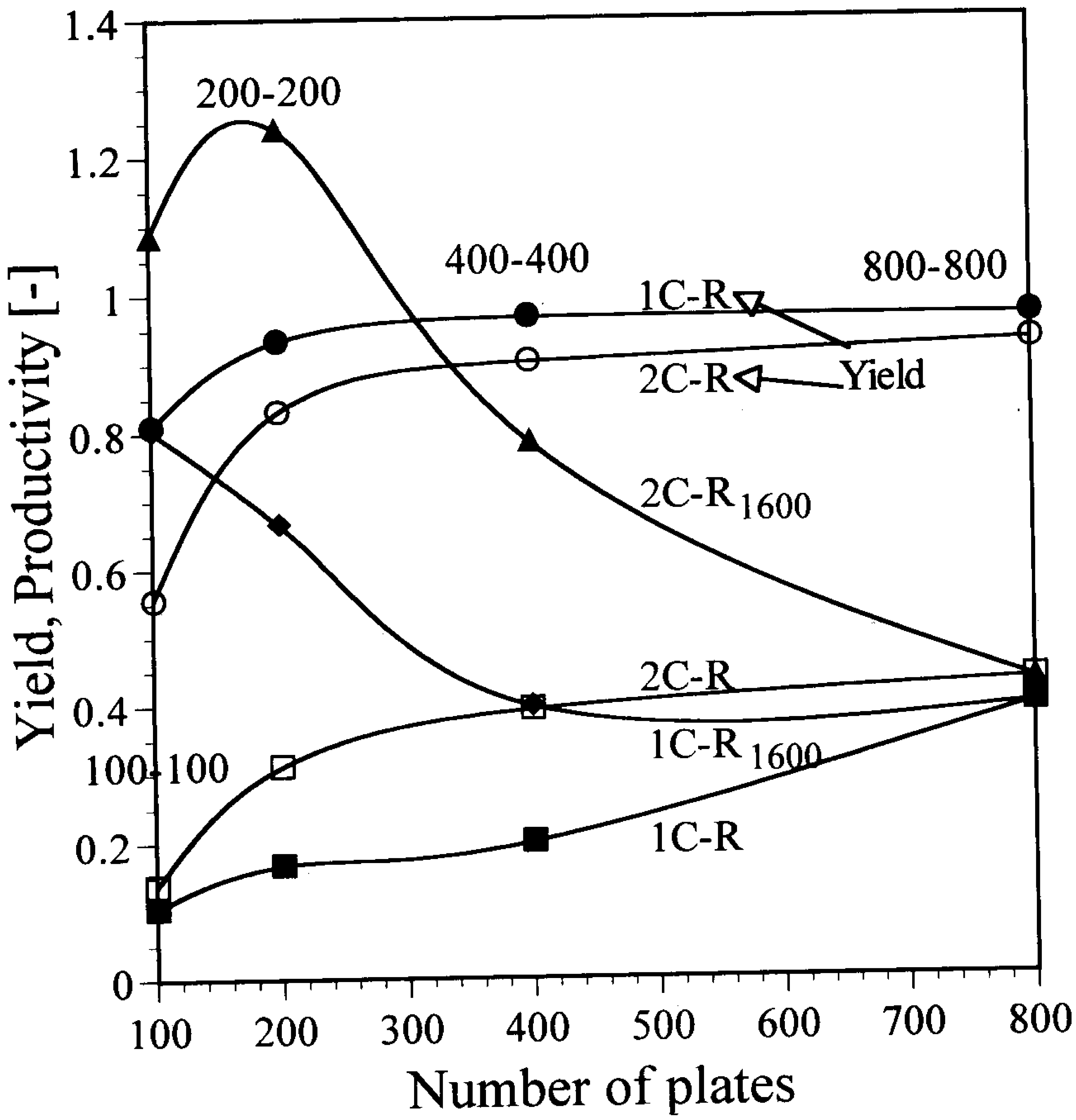
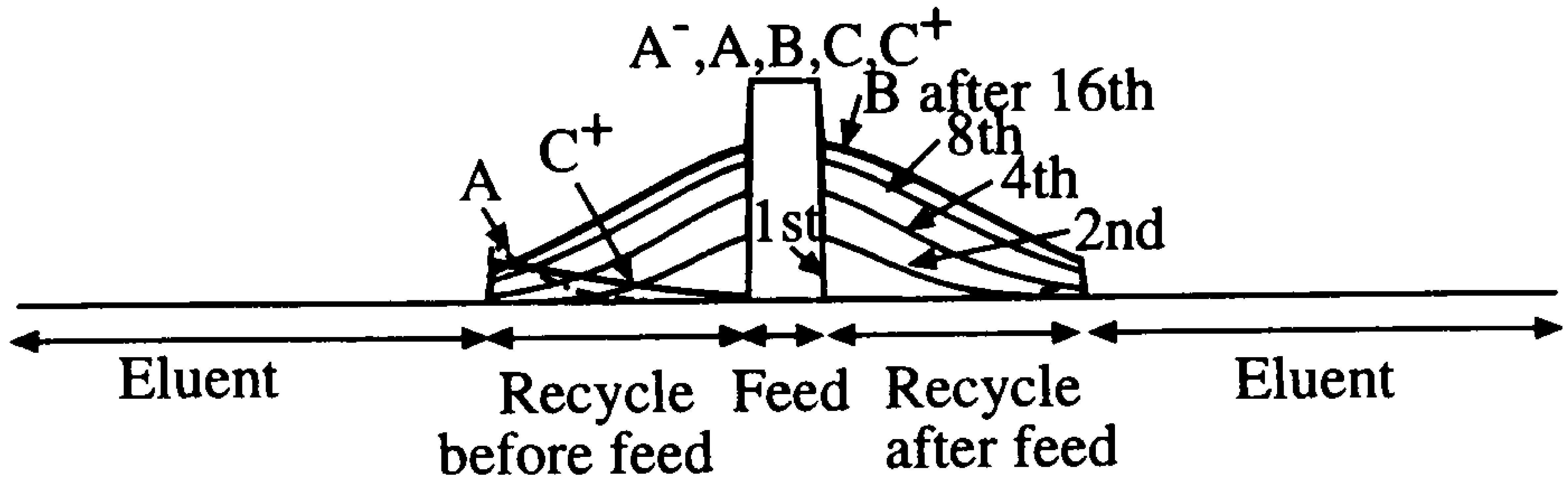
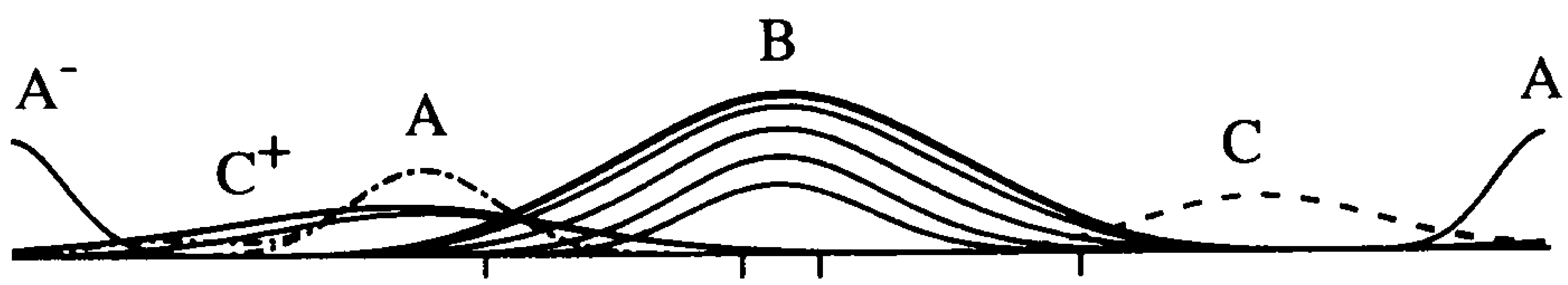


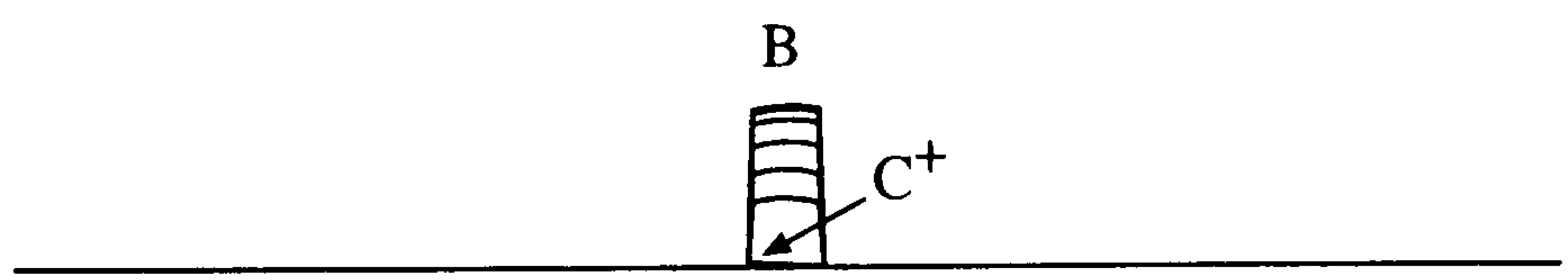
Figure-4: Effect of Plate Numbers on Separation Performance of Three-component Mixture.



(a) Change in inlet concentration with respect to time



(b) Change in outlet concentration with respect to time



(c) Change in concentration of product with respect to time

Figure-5: A Chromatogram Showing the Tendency of Strongly Adsorbed Component in Single-column sandwiching Recycle

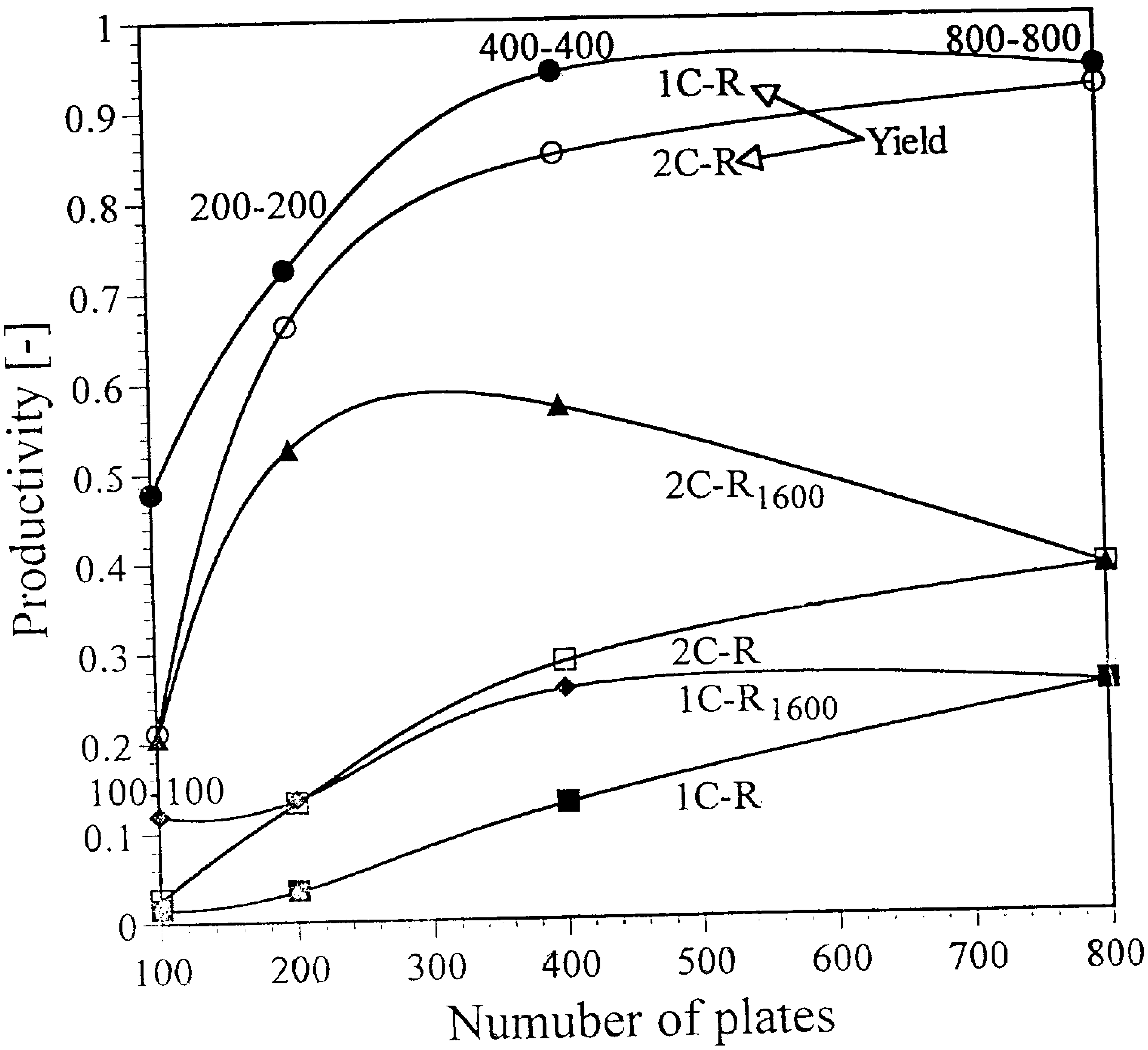


Figure-6: Separation Performance of Sandwiching Recycle in Presence of Far Neighbor Components