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Theoretical and Experimental Study of a Non-stationary Isotope Separation Process in a Gas Centrifuge Cascade

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ABSTRACT

Non-stationary separation processes of multicomponent isotope mixtures seem to be efficient in enriching components of small abundance. Using the separation of $^{36}SF_6$ from its multicomponent isotopic mixture SF_6 as an example, a numerical simulation method is applied to predict a non-stationary separation process occurring in a short gas centrifuge cascade, and also a corresponding experiment is carried out. The concentrations of the components in the SF_6 isotope mixture are measured at different times during the separation process. The simulation results are in good agreement with the experimental ones. As an example of application of the simulation method to practice, a cascade is designed to separate

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the $^{36}SF_6$ component to a concentration of over 99% from the isotopic mixture of natural abundance.

Key Words: Multicomponent isotope separation; Gas centrifuge cascades; Numerical simulation.

INTRODUCTION

The fast development of sciences and technologies has produced new applications of stable isotopes in many areas, such as physics, medicine, bioscience, nuclear science, and material science, which has sprung increasing demands on stable isotopes. Centrifugation has been successfully used in the separation of uranium isotopes and proves to be the most advanced technology in large-scale isotope separation so far. It is then a natural extension of the technology of centrifugation to the separation of stable isotopes. In fact, in Europe and Russia, there are a number of gas centrifuge facilities dedicated to separation research and/or production of stable isotopes. Unlike the separation of uranium isotopes, which normally treats the uranium isotope mixture to be composed of only two components, separation of stable isotopes encounters more complicated cases: different number as well as different concentrations of components, different physical and chemical properties, and separation of end or middle components. Therefore, all of these necessitate the development of new separation techniques, in which this article focuses on a non-traditional separation method, that is, a non-stationary separation process.

Separating components of small abundance in a multicomponent isotope mixture is a quite lengthy process with a gas centrifuge cascade operating in a conventional manner. Operating in a conventional manner here means that a cascade in operation has two withdrawals at the two ends of the cascade and one feed in between, and the flows are steady anywhere in the cascade. To enrich a component to the required concentration, the separation process usually consists of several successive runs, using the product or the waste of a run as the feed of the following run. When the concentration of the component to be separated is small, some problems arise. The first problem is that the amount of feed for the last run or the last few runs may not be sufficiently large to allow a steady flow to be established in the cascade. To explain these problems, we used SF_6 as the process gas and take the separation of $^{36}SF_6$ as an example. Sulfur is composed of 4 isotope components in its natural isotope mixture and the concentration of ^{36}S is 0.00013. Assume that there is a demand for 1 g of SF_6 with a concentration of $^{36}SF_6$ greater than 90%. Clearly, to obtain 1 g of such SF_6, the cascade needs to manipulate at least

$0.9/0.00013 \approx 6923\,\mathrm{g}$ of the natural isotope mixture. Suppose that four separation runs are carried out, with the cascade cuts being 0.9 for the first three runs. Such a separation process is not unusual in multicomponent separation. Here, it is the waste withdrawal of a run that is used as the feed for the next run. Then obviously, the amount of feed left for the fourth run is only about $6923 \times 0.1^3 \approx 6.9\,\mathrm{g}$, which is probably not enough to establish a steady flow in the cascade. To avoid this problem, one may separate more SF_6 in the first three runs to provide enough amount of feed for the fourth run. But doing it this way means an increase in the amount of material for separation and, increase in the separation time and thus leads to an increase in the separation costs. Of course, this problem does not exist for a fairly large-scale production of $^{36}SF_6$. But the demands on stable isotopes are varied; it is well possible that a request for stable isotopes is just a matter of a few g, or even μg. The second problem is that, in different runs, the operation status, that is, the feed position, the cascade cut, the shape of the cascade, and so forth, may need to be adjusted accordingly to the changes of the components concentrations to achieve the best separation performance. This is a complicated process and needs careful planning. The third problem is that the intermediate products may need to be stored. In the example here, the product withdrawal may have a higher concentration of ^{36}S than the natural one, especially in the later runs, and therefore is worth being stored. Obviously, the more the runs are carried out, the more storage is required for intermediate products of different concentrations, which is inconvenient.

These problems can be resolved if a non-stationary separation process, as proposed by Rudnev and Colleagues,[1] is employed. In this process, there is only one withdrawal, instead of two in a conventional process. Depending on which component, the lightest or the heaviest, is to be separated, either the product or the waste withdrawal is switched off. A theoretical investigation of the non-stationary process has shown that for the separation of end components, it is almost optimal[2] in terms of the material recovery and the operation time efficiency in comparison with a sufficiently long cascade. However, the use of non-stationary separation is very rarely studied in research work and its further development would be very beneficial if a theory, being capable of not only qualitative analysis but also better still quantitative analysis, was available. Here, we present briefly the idea of a numerical simulation method, which was developed by Zeng and Ying,[3] and also perform a non-stationary separation experiment to make comparisons between the numerical simulation and experimental results. The simulation demonstrated its usefulness in studying non-stationary separations and can be applied to cascade design and separation performance analysis.

THEORY

We consider an isotope mixture of N_c components, and number the components sequentially from the lightest to the heaviest for easy reference. The waste end of a rather general cascade for separation is illustrated schematically in Fig. 1. Here, H_n, H'_n and H''_n are, respectively, the holdups in the gas centrifuges, the upstream pipes, and the downstream pipes at the nth stage; $m_{n,i}$, $m'_{n,i}$ and $m''_{n,i}$ are the rates of material losses for the ith component in the centrifuges and the pipes, respectively. F_n, P_n, and W_n are the feed, and the head and tail flows, respectively, and G_n is the entering flow, at stage n. L'_n and L'^a_n are the upstream flows of the nth stage before and after passing through the pipe connecting stages n and $n+1$; L''_n and L''^a_n are the downstream flows before and after passing through the pipe connecting stages n and $n-1$. Assuming that a cascade is to be operated at a state with constant feed rates F_n and constant feed concentration $C^F_{i,n}$, and the withdrawal rates P_n and W_n, the separation process can be divided into three stages. In stage 1, the process gas is fed into the empty cascade, and then as the hold-up increases, the flows inside centrifuges and connecting pipes gradually become steady. Note that in this stage, not only do the flows change, but also the concentration distributions of the components. In stage 2, the concentrations of components keep changing, although the flows are steady. But eventually the concentration distributions will reach a steady state, provided that the cascade has been running for a long enough time. In stage 3, this is, at the end of the separation operation, the feed is cut off, and then the holdup inside the cascade decreases

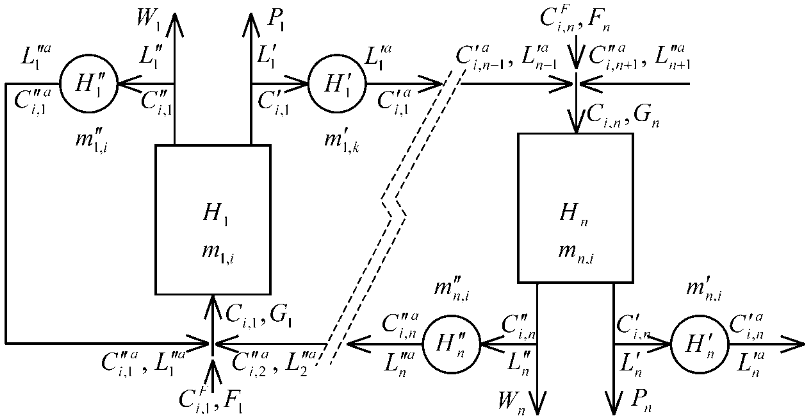

Figure 1. A schematic illustration of part of a separation cascasde.

and eventually the cascade is evacuated. The flows and the concentrations also both change in this stage. Because the change of flows is involved, the first and the third stages are very complicated and are very difficult to study. However, the two stages are short, compared with the second stage, and are of less interest because the most separation work is done during the second stage. Therefore, our study focuses on this stage. The equations describing the concentration distribution in the cascade can be established by consideration of mass conservation. L''^{a}_{n} satisfies

$$L''^{a}_{n} = L'^{a}_{n-1} - \sum_{l=1}^{n-1} \left[F_l - P_l - W_l - \sum_i (m_{l,i} + m'_{l,i} + m''_{l,i}) \right]$$

or

$$L''^{a}_{n} = L'^{a}_{n-1} + \sum_{l=n}^{N} \left[F_l - P_l - W_l - \sum_i (m_{l,i} + m'_{l,i} + m''_{l,i}) \right]$$

$C'_{i,n}$ and $C''_{i,n}$ are, respectively, the concentrations of the *i*th component in the heads and tails flows just leaving the centrifuges at stage *n*, and $C'^{a}_{i,n}$ and $C''^{a}_{i,n}$ are, respectively, the concentrations in the head and tail flows just before the mixing points. At the two ends of the cascade, the following relationships hold:

$$L'^{a}_{0} = L''^{a}_{1}, \quad C'^{a}_{i,0} = C''^{a}_{i,1} \tag{1}$$

$$L''^{a}_{N+1} = L'^{a}_{N}, \quad C''^{a}_{i,N+1} = C'^{a}_{i,N} \tag{2}$$

with *N* the total number of stages. At the *n*th stage, considering the *i*th component in the centrifuges we have

$$\begin{aligned} \frac{\partial}{\partial t} H_n[\theta_n C'_{i,n} + (1-\theta_n)C''_{i,n}] = L'^{a}_{n-1}C'^{a}_{i,n-1} + L''^{a}_{n+1}C''^{a}_{i,n+1} \\ + F_n C^{F}_{i,n} - (L'_n + P_n)C'_{i,n} \\ - (L''_n + W_n)C''_{i,n} - m_{n,i} \end{aligned} \tag{3}$$

where θ_n is the stage cut and is defined by

$$\theta_n = \frac{L'_n + P_n}{L'_n + P_n + L''_n + W_n}$$

For the pipes, the equations of mass conservation are

$$\frac{\partial H'_n C'^a_{i,n}}{\partial t} = L'_n C'_{i,n} - L'^a_n C'^a_{i,n} - m'_{n,i} \tag{4}$$

$$\frac{\partial H''_n C''^a_{i,n}}{\partial t} = L''_n C''_{i,n} - L''^a_n C''^a_{i,n} - m''_{n,i} \tag{5}$$

The separation property of a gas centrifuge is described by the following empirical relationship[4]:

$$\frac{C'_{i,n}/C''_{i,n}}{C'_{j,n}/C''_{j,n}} = \gamma_0^{M_j - M_i} \tag{6}$$

with γ_0 being the unit separation factor, and M_i and M_j respectively the molar weights of the ith and jth components. Equations (1) through (6) are the equation system describing the transient behavior of concentrations in a cascade, with the following constraints:

$$\sum_{i=1}^{N_c} C'_{i,n} = \sum_{i=1}^{N_c} C''_{i,n} = \sum_{i=1}^{N_c} C'^a_{i,n} = \sum_{i=1}^{N_c} C''^a_{i,n} = 1 \tag{7}$$

The temporal terms in the above partial-differential difference eqs. (3) through (5) are discretized by using the Crank–Nicolson scheme. The resulting algebraic equations together with eqs. (1), (2), and (6) are solved at every time step by the so-called Q-iteration method such that

$$\max\left(\left|\sum_n C_{i,n} - 1\right|, \left|\sum_n C'_{i,n} - 1\right|, \left|\sum_n C''_{i,n} - 1\right|\right) \le \varepsilon_1 \tag{8}$$

Here ε_1 is the solution accuracy tolerance.

EXPERIMENT

The cascade used in the experiment consists of four stages, as shown in Fig. 2. The feed position is at the fourth stage and so is the product withdrawal. The length of the cascade is too short for practical use, but this does not matter as long as the separation effect is large enough to be able to be compared with the results of the numerical simulation. The process gas is SF_6, and the concentrations of the sulfur components in the feed are the natural ones and

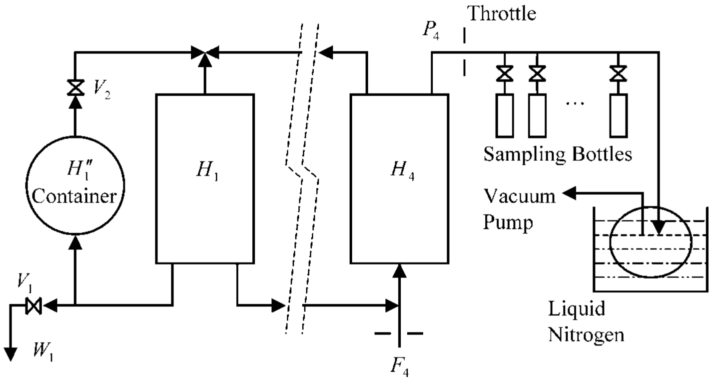

Figure 2. A four-stage cascade used in the experiment for the separation of $^{36}SF_6$.

are listed in Table 1. For simplicity, all quantities specified are the corresponding actual physical quantities divided by the feed rate (g/h) and are presented without giving dimensions. So, the feed rate $F_4 = 1$. A container is installed on the recirculating pipe at the waste end with a holdup of $H_1'' = 0.09$. The holdups in other pipes are negligible compared with H_1'' and are simply taken to be zero. The holdups $H_1 = 0.02$ and $H_n = 0.01$ for $n = 2, 3, 4$, in the four stages, respectively. The unit separation factor γ_0 of the centrifuges is experimentally measured to be 1.5. The flow rates between centrifuges, $L_n' = 1$ for $n = 0, 1, 2, 3, 4$ (in other words, the stage cut is 0.5).

The experiment is conducted as follows. At the beginning of the experiment, the valve V_1 is on and the valve V_2 is off. So $W_1 \neq 0$, $L''^{a}_{1} = 0$, and the cascade is operating now in a conventional manner. Doing this allows the centrifuges as well as the container H_1'' to be filled with some amount of gas such that the actual initial condition is as close as possible to the initial condition used in the calculation. At a certain time, switch off V_1 and on V_2 simultaneously, so $W_1 = 0$ and $L''^{a}_{1} = L_1'' \neq 0$. Now the cascade is operating at the designed status. (To be precise, it takes some time for the flow in the cascade to be established after the change of V_1 and V_2. But the time spent on the flow transition is very short and, therefore, can be ignored.)

Table 1. Natural concentrations of stable sulfur isotope components.

Components	^{32}S	^{33}S	^{34}S	^{36}S
Concentration	0.95006	0.00760	0.0422	0.00013

This moment is counted as the start of the non-stationary separation process. During the process, the product withdrawal is sampled at given time intervals to measure the concentrations by switching on and off the valves on the sample bottles one by one at the given times.

NUMERICAL SIMULATION

The simulation uses the theory presented previously. All quantities are specified according to the experimental setup. The initial condition for the concentration distribution is constant and is simply taken to be the feed concentrations. This condition should not be too far away from the real situation, because at stage 1 of the three stages of a non-stationary separation process, the separation performance of the cascade is not fully put into use. The reasons are, first, the flow is not steady yet and, second, the cascade setup for running in a non-stationary manner is not suitable for running in a conventional manner. Considering that the SF_6 gas is very stable and does not react with its contacting materials, such as centrifuges and pipes, material losses do not exist and, therefore, $m_{n,i} = m'_{n,i} = m''_{n,i} = 0$. The solution accuracy tolerance ε_1 is specified at 10^{-6}.

The readers may refer to Zeng and Ying[5] for details about the solution of the nonlinear equation system involved in the theory.

COMPARISON AND DISCUSSION

The experimental and the simulation results for $^{32}SF_6$, $^{33}SF_6$, and $^{34}SF_6$ components are plotted together in Figs. 3 through 5 for comparison. Because the concentration of $^{36}SF_6$ is too small and hard to be measured to a satisfactory accuracy, it is not plotted here. It is clearly seen that the experimental results are in very good agreement with the theoretical ones. The results from other experiments also showed that the simulation can predict the transient processes satisfactorily. This gives us some confidence that the numerical simulation is usable and can be of practical significance, such as in cascade design, an example of which is given in the following section.

Although the agreement between the experimental and simulation results looks good, a discrepancy is clearly noticed. These experimental points at the time interval between about 3 to 10 hours after the beginning deviate rather significantly from the simulation values. $C'_{1,4}$ and $C'_{3,4}$ in the experiment vary more slowly and $C'_{2,4}$ in the simulation has an overshoot. Two main possible reasons are that the unit separation factor γ_0 and the holdups are not very

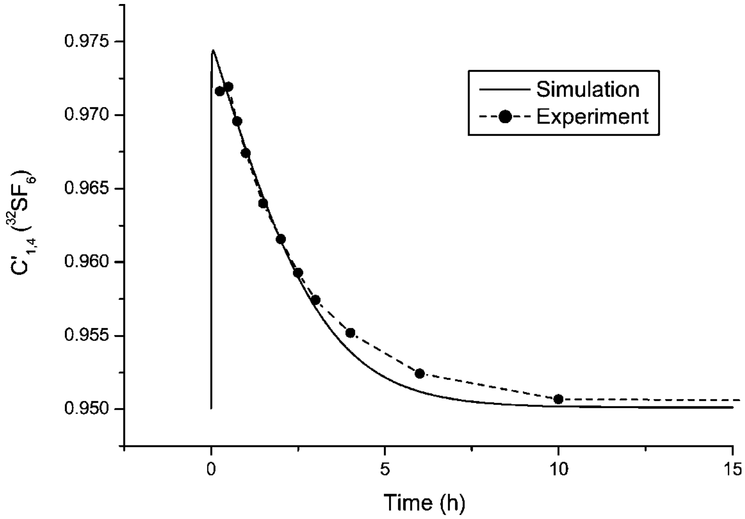

Figure 3. Comparison between the simulation and experimental results for $^{32}SF_6$ at different times during the transient process in the four-stage cascade, $\gamma_0 = 1.5$.

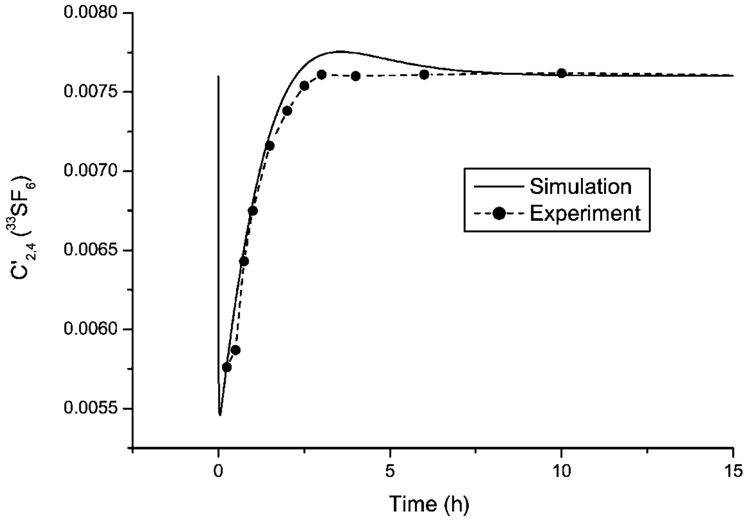

Figure 4. Comparison between the simulation and experimental results for $^{33}SF_6$ at different times during the transient process in the four-stage cascade, $\gamma_0 = 1.5$.

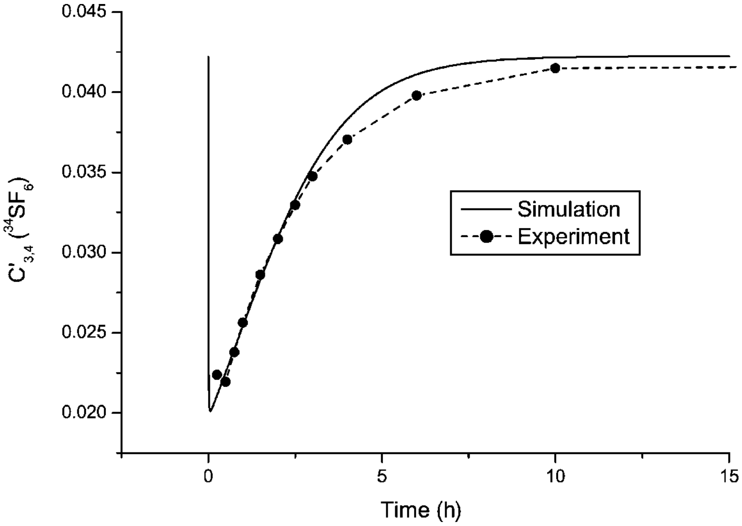

Figure 5. Comparison between the simulation and experimental results for $^{34}SF_6$ at different times during the transient process in the four-stage cascade, $\gamma_0 = 1.5$.

precisely given. The effect of γ_0 on the C' curves at stage 4 is demonstrated in Figs. 6 through 8. These curves are obtained with the same parameters as in the experimental setup except for the varying γ_0. Looking at Fig. 7, obviously a larger value of γ_0 than 1.4 causes the overshooting of the $C'_{2,4}$ values between 2 and 8 hours, which means that in the simulation, the value 1.5 for γ_0 is somewhat too large. Another phenomenon we noticed is that the quick transition part of the transient process occurs before 8 hours and the value of γ_0 does not affect the time for this part very much. But the larger the value of γ_0, the larger the extent of change of the C' values. The time for the quick transition part is affected by the holdups. Figs. 9 through 11 (with H_4 fixed) and Figs. 12 through 14 (with H_1 fixed) present the results of simulation for different values of H_1 and H_4, with other parameters fixed at the values in the experimental setup. (The solid curves are actually the simulation using the experimental setup.) Apparently, H_1 influences the concentrations more than H_4, that is, the holdups at the closed end (including the H' s and the H'' s) play a more important role in the transient process. From Figs. 12–14, it is seen that larger holdups result in slower concentration changes. So it appears that the holdups specified in the simulation are somewhat too small. Just in contrast with γ_0, the holdups do not affect the extent of change of the C' values at stage

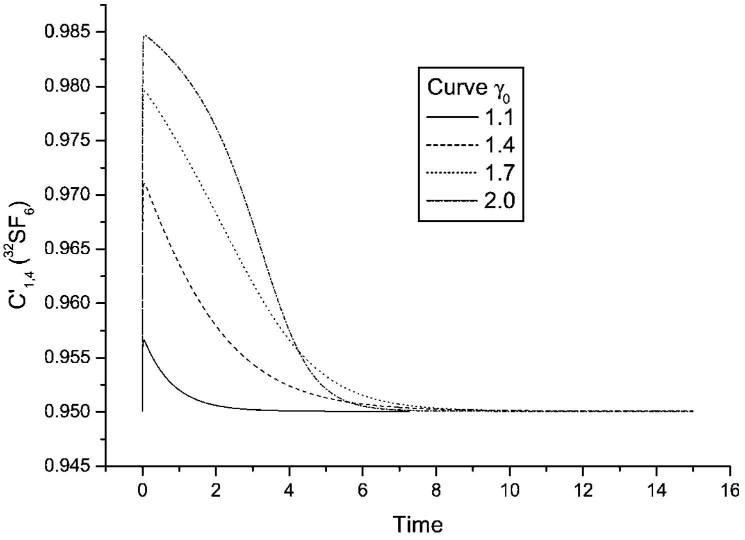

Figure 6. History of concentration change ($C'_{1,4}$) with time for different values of γ_0.

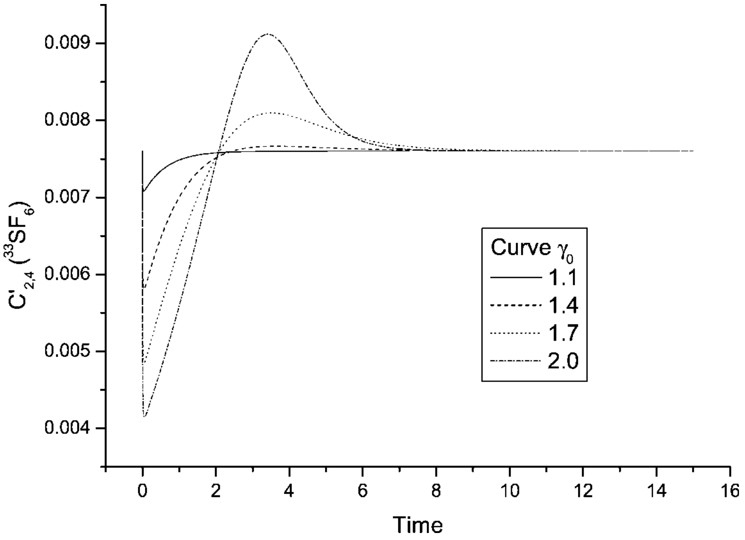

Figure 7. History of concentration change ($C'_{2,4}$) with time for different values of γ_0.

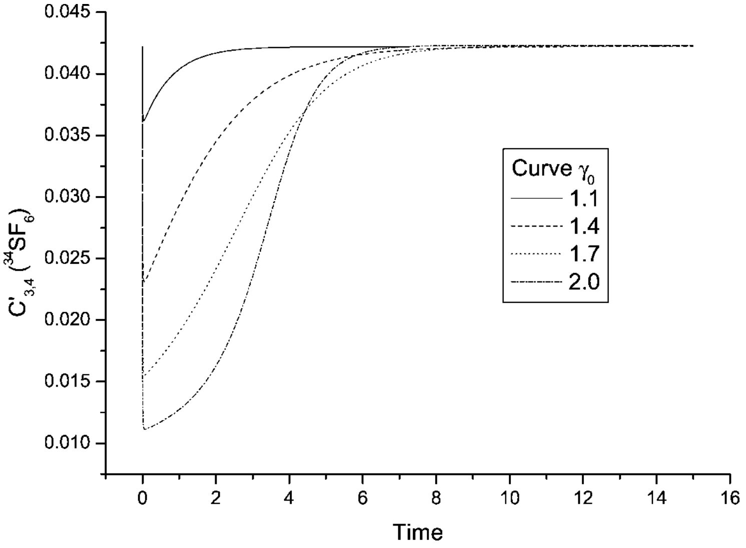

Figure 8. History of concentration change ($C'_{3,4}$) with time for different values of γ_0.

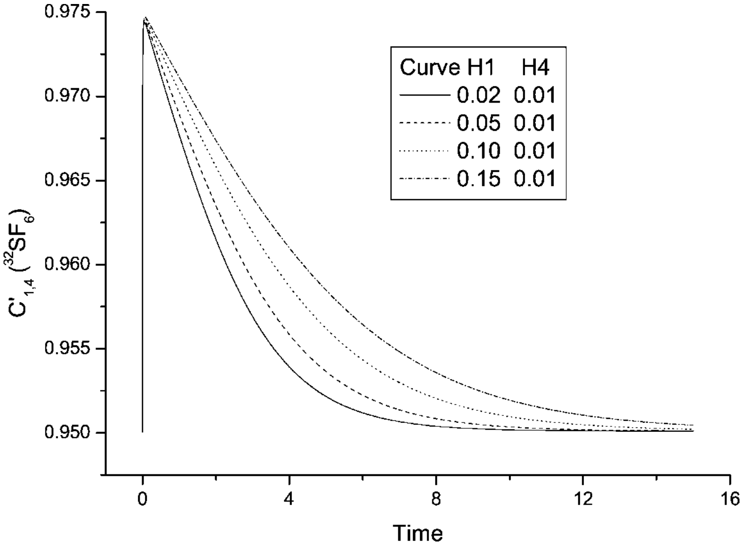

Figure 9. History of concentration change ($C'_{1,4}$) with time for different values of H_1.

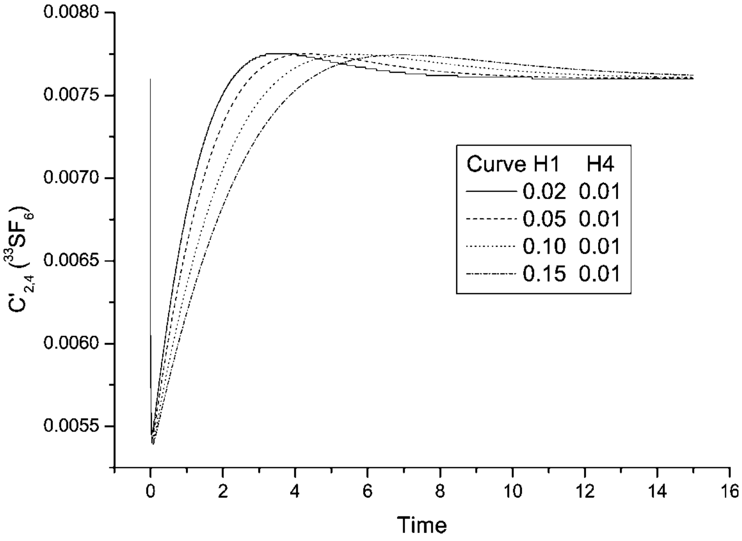

Figure 10. History of concentration change ($C'_{2,4}$) with time for different values of H_1.

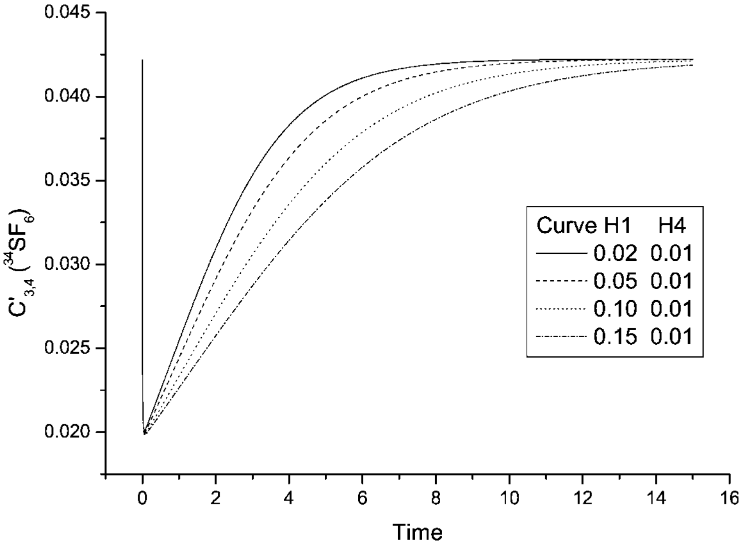

Figure 11. History of concentration change ($C'_{3,4}$) with time for different values of H_1.

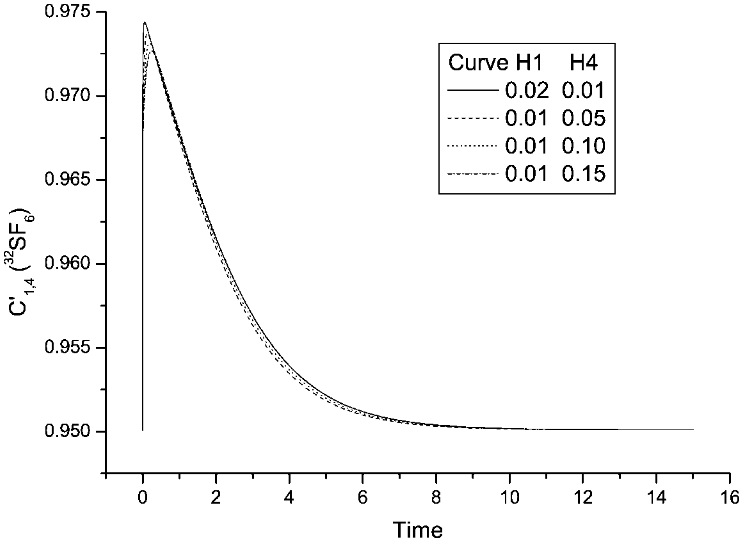

Figure 12. History of concentration change ($C'_{1,4}$) with time for different values of H_4.

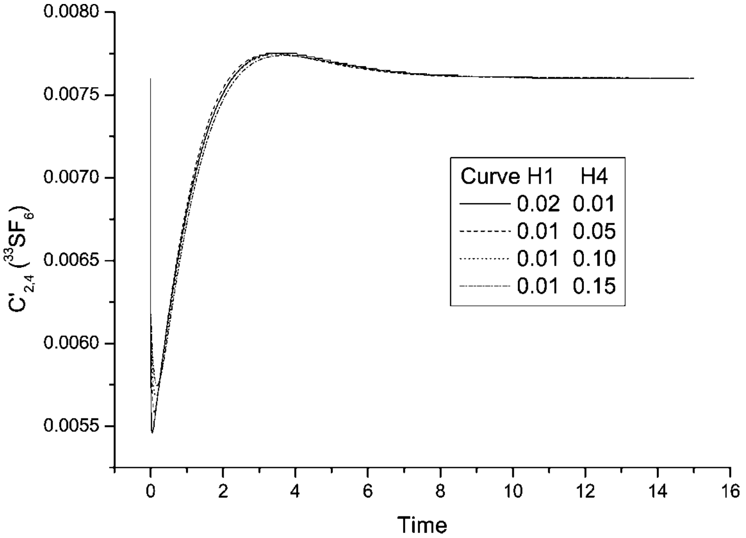

Figure 13. History of concentration change ($C'_{2,4}$) with time for different values of H_4.

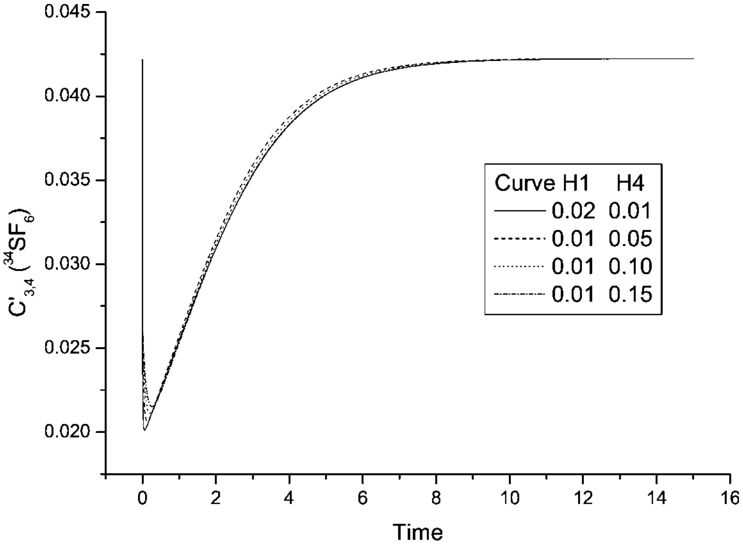

Figure 14. History of concentration change ($C'_{3,4}$) with time for different values of H_4.

4. Therefore, with a more accurate specification of the values of γ_0 and the holdups, it seems that the numerical prediction and the experimental results can be matched very well. The experimental confirmation of these two reasons requires modifying the experimental setup and sampling frequently at each stage, which is quite complicated, so further experiments are not carried out.

CASCADE DESIGN

Suppose that we would like to enrich ^{36}S to the concentration of being greater than 99% from the natural isotope mixture. Depending on practical demands, there are a number of questions that a cascade designer needs to answer, such as, with the centrifuges used in the previously described experiment, how many stages and how many centrifuges should the cascade have? And how long does it take to obtain the required concentration? The readers may obtain some inspiration from the following considerations.

To design a cascade, the shape of the cascade (cascade length and stage-flow rates) and the feed position are the important factors to be determined. Without involving complicated optimizations, it is assumed that the cascade is rectangular (L'_n is constant for all stages), the feed position is specified at the two thirds of cascade lengths from the waste end, and only one separation run is carried out. Then, the number of stages is determined based on the required concentration: one can start from a short cascade and gradually increase the number of stages until the required concentration is obtained in the container H''_1. The stage-flow rates are determined here according to the consideration of operation time. In order to maintain the same separation performance in all stages, the upstreaming flow rates L'_n should not be too small (so that the stage cuts do not deviate significantly from 0.5). Here, L'_n is fixed at 10, for $n = 0, 1, 2, \ldots, N$.. The other factors to be determined in a cascade design are the holdups. The holdups H_n in the centrifuges depend on the type of centrifuges and are assumed to be 0.1, and, for simplicity but without loss of generality, the holdups H'_n and H''_n, except H''_1 are assumed to be zero. The size H''_1 of the container at the waste end is given as known according to how much ${}^{36}SF_6$ is wanted in one run. Here, it is set to 1. Figs. 15 and 16 present the variations of the concentrations of ${}^{36}SF_6$ at the waste end and the product end of different cascades in the transient processes of separation from the start to the steady state. A steady state is thought to be reached when the total mass conservation

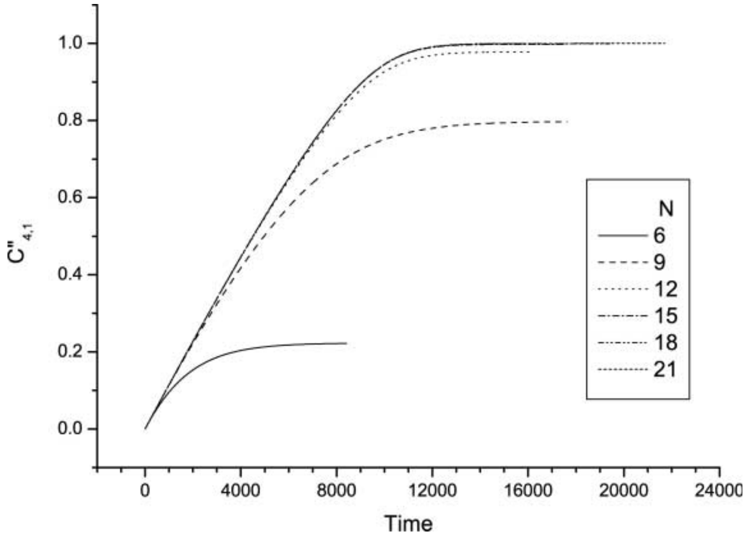

Figure 15. Concentration change of ${}^{36}SF_6$ with time at the waste end for different cascade lengths.

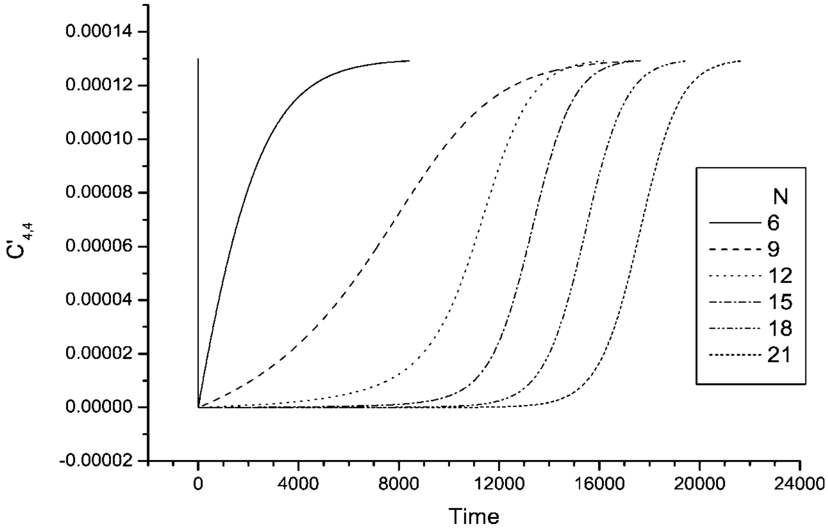

Figure 16. Concentration change of $^{36}SF_6$ with time at the product end for different cascade lengths.

is satisfied to the required accuracy:

$$err = \left(\sum_n \sum_i \left(F_n C^F_{i,n} - P_n C'_{i,n} - W_n C''_{i,n} \right)^2 \right)^{1/2} \leq \varepsilon_2$$

where ε_2 is a small number and is taken to be 10^{-6} in all calculations. Actually, the satisfaction of this condition implies that the concentrations become steady (cf. Figs. 15 and 16). The transient time is taken to be the time used by a separation run. When the cascade length is longer than 12 stages, it is hard to distinguish the curves from each other in the figures. So to make this clear, we give the concentrations at the steady states as well as the transient times in Table 2.

Three facts from observation of the figures are worth mentioning. First, it can be clearly seen that the longer a cascade is, the higher concentration that can be achieved. But this is at the cost of spending more separation time and consuming more material. Therefore, if the desired component is to be enriched to a very high concentration, it is worth planning carefully the separation strategies. For example, whether or not preliminary separation runs are necessary. Second, when the cascade is fairly long, for example longer

Table 2. Concentrations and transient times for different cascade lengths.

N	3	6	9	12	15	18	21
$C''_{4,1}$	0.001064	0.23672	0.80555	0.97947	0.99815	0.99984	0.99999
T_s	450	8944	17,837	16,530	17,944	20,053	22,242

than 12 stages here, the transient time increases almost linearly as the number of stages, which is due to the fact that the increase of the cascade length causes an almost linear increase of the total holdup in the cascade. This suggests that the holdups in the centrifuges and, especially in the stage connecting pipes, should be kept as little as possible. But it has to be pointed out that the reduction of the holdups in centrifuges may have negative effects on the separation performance of the centrifuges. In case this really happens, to maintain the separation capability of a cascade, the number of cascades has to be increased, which leads to the increase of the number of centrifuges and the increase of the total holdup of the cascade. One may use optimization to make the best planning such that a good performance of the cascade is maintained while the total holdup is reduced. Third, the transient process is exceptionally long for the case of the cascade length $N = 9$, even longer than the case $N = 12$ and comparable with $N = 15$, whereas the final concentration at the steady state is lower than the latter two cases. Such exception, of course, is better avoided. With the help of the theoretical simulations, there is no difficulty for us to discover it.

Now, it is obvious that to have the concentration of $^{36}SF_6$ greater than 99%, the number of stages of the cascade should be around 15, and the time spent is about 17,900. With the number of stages and L'_n known, the total number of centrifuges can be easily calculated according to the operation parameters of a centrifuge. This example is, of course, a very simplified design. In a practical design, there may be many other factors that need to be considered. For example, it should be noted that the effect of feed position is not taken into account here, because the feed positions are always two thirds of the corresponding cascade lengths away from the waste end. Its effect may not be ignored in practice. Figure 17 shows clearly that the transient time and concentration of $^{36}SF_6$ in H''_1 at the waste end have fairly large differences as the feed position changes from 1 to 15. The further away the feed position is from the waste end, the higher the concentration of $^{36}SF_6$, but the more time the separation uses to reach the steady state. In this example, the maximum change in the transient times is about 10%, whereas the maximum change in the concentration is about 3%. These changes would certainly cause an increase or decrease in material and energy consumptions, manpower input,

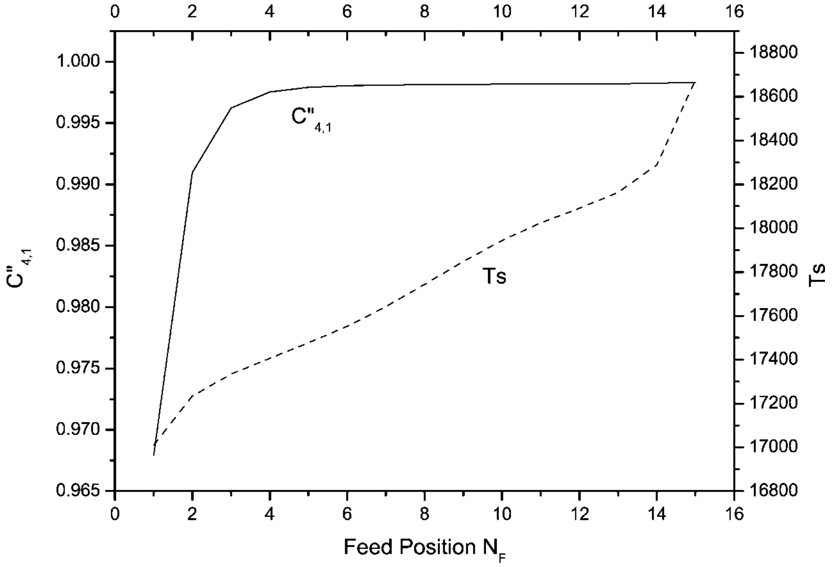

Figure 17. The transient time and concentration of $^{36}SF_6$ at the waste end as functions of feed position for the case $N = 15$.

and so forth, which will be reflected eventually in the product price. Therefore, a good practical design should take into account these factors. The simulation method presented here is an important tool in a prior analysis and would provide useful information in cascade design.

CONCLUSION

A non-stationary separation of multicomponent isotopic mixtures was studied by numerical simulation and experiment using the separation of $^{36}SF_6$ as an example. The experiment was carried out in a cascade of four stages with only a withdrawal at the product end. The experimental results are in good agreement with those of the theoretical simulation, which are obtained by solving the partial-differential difference equations describing the concentration distribution of isotope components in cascades. A simple application of the simulation method was given to the design of a cascade for the enrichment of $^{36}SF_6$ to a concentration greater than 99% from the isotopic mixture of natural abundance to demonstrate the usefulness of the simulation method.

Some important factors, which should be considered at the design stage to make appropriate decisions, such as the choice of the cascade length and the feed position and the time consumption of separation, can be investigated by means of the simulation method.

The study of non-stationary separation here focuses on an end component, and as shown, the non-stationary separation has achieved satisfactory performance. But a similar idea can also be applied to the separation of middle components, which will be studied by using the theory presented here and the results reported in a forthcoming article.

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