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자극-反應法을 利用한 混合沈降型 抽出裝置의 RTD 모델 開發†

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Development of RTD Model of the Mixer-Settler-Type Extractor Using the Stimulus-Response Method†

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요 약

자극-반응(stimulus-response method)법을 이용하여 1단과 다단 혼합침강형 추출장치의 물질흐름특성을 규명하기 위한 실험을 수행하였다. 실험 결과, 1단 추출장치의 혼합조의 반응특성은 CSTR과 동일한 결과를 나타내었으며 혼합-침강조의 반응특성에서는 시간 지연 효과가 나타나며, 단수가 증가 할수록 시간지연효과는 증가하였다. 1단과 다단 추출장치의 반응특성 실험결과를 RTD모델 해석프로그램인 K-RTD를 이용하여 분석하여 1단 및 다단 추출기의 RTD을 개발하였다. 본 연구에서 개발한 1단 및 다단 혼합침강조형 추출기의 RTD 모델에 의한 계산값과 실험값에 대한 상관 계수는 각각 0.963과 0.995로 비교적 높은 것을 확인하였다.

주요어 : 잔류시간분포 모델, 용매추출, 자극-반응법, 다단 추출기

Abstract

This study presents the findings of the experiments that were conducted on single- and multi-stage solvent extractors using the stimulus-response method, with the aim of identifying flow characteristics of the material inside the mixer-settler-type extractor. The results of this study show that the response characteristics of a single-stage mixer is the same as that of a completely stirred tank reactor (CSTR), and that the lag time of a mixer-settler-type extractor increases with the number of its extraction stages. The experimental data for the single- and multiple-stage extractors were analyzed using K-RTD, a response analysis program, to obtain a retention time distribution (RTD) model of one-stage and four-stage extractors. The correlation coefficient between the calculated values and the experimental data was 0.963 for the one-stage extractor and 0.995 for the four-stage extractor, showing quite a good correlation.

Key words : RTD model, solvent extraction, stimulus-response method, multistage extractor

1. Introduction

Since a pump-mix principle was proposed by Mensing in the 1940s, the application of the mixer-setter-type extractor has been widened owing to its various operational advantages. Moreover, it has come to be used as a representative apparatus for the separation and purification processes of some rare earth elements. It is very important to predict the dynamic behavior of the fluid flow of multi-stage extractors to ensure their startup and shutdown, to determine their optimum operation condition, and to establish automatic control for their continuous operation. In the analysis of the solvent extraction process, it is very important to investigate the transient response characteristics, which predicts the temporary difference that takes for the material or organic phase used in the first stage to affect the succeeding stages during the solvent extract process, with N stages.

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In general, an RTD model for fluid inflowing into the reactor is needed to analyze the dynamic behavior of the reactor with fluid. To develope such RTD model, it is necessary to obtain data on the flow characteristics of the fluid inside the reactor. Such data can be obtained by using the stimulus-response method[1].

In the stimulus-response method, a certain stimulus (a tracer, such as a certain material or an isotope) is inputted at the entrance of the extractor, and its response (the effluent rate of the tracer) is measured at the outflow of the reactor to analyze the dynamic behavior of the fluid in the reactor.

The early studies on the RTD model, which indicate the response characteristics of the mixer-settler-type extractor, considered only the mixer and analyzed the settler as a simple PFR to explain the dynamic behavior of the apparatus, but the obtained results were not satisfactory. The dynamic behavior of the multi-stage mixer-settler-type extractor was simulated using the CSTR model, which incorporates the linear equilibrium equation and Murphree efficiency, and the pseudo-random binary sequence (PRBS) was applied in the 1970s[2]. Based on the results these studies, it was concluded that the settler must also be incorporated into the dynamic-behavior model. As the mainstream of an early dynamic-behavior model for the typical mixer-settler-type extractor, a CSTR and plug flow model, which uses the mixer as a continuous stirred tank reactor (CSTR) and the settler as a plug glow, has been mainly used. A very complex model, however, which consists of multiple CSTRs and PFRs that connected directly or parallel to the single-phase extractor, is currently also being used[2,3].

The aim of this study is to conduct a response characteristics experiment for single- and multi-stage extractors to analyze the RTD model of the mixer-

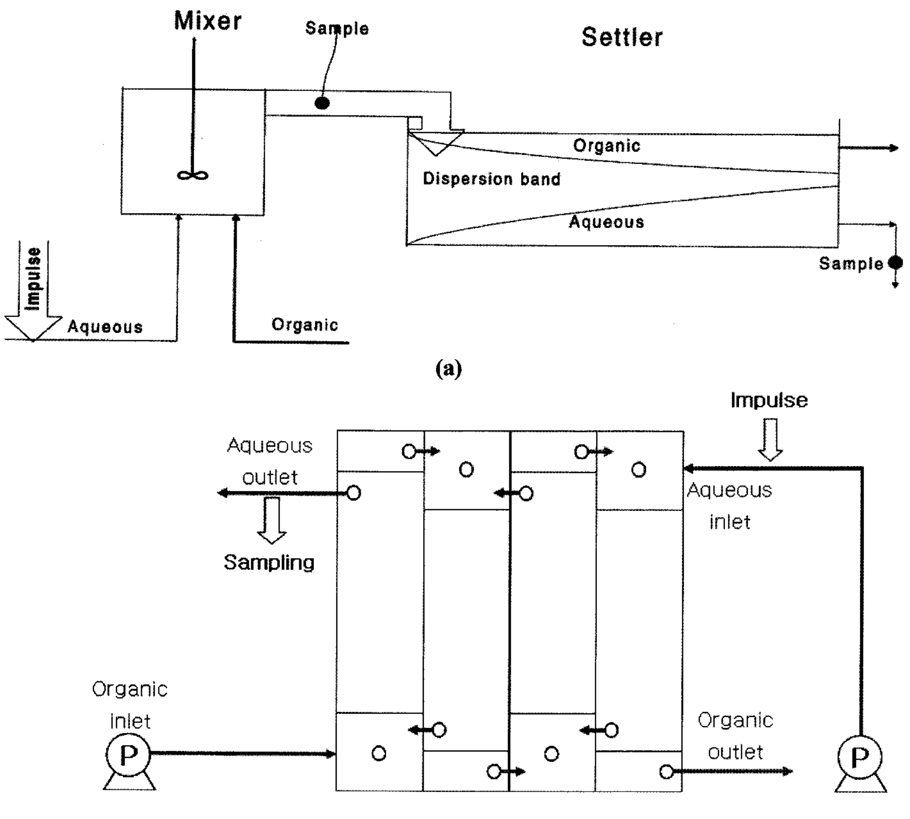


Fig. 1. Tracer injection and sampling points: (a) one-stage extractor and (b) four-stages extractor.

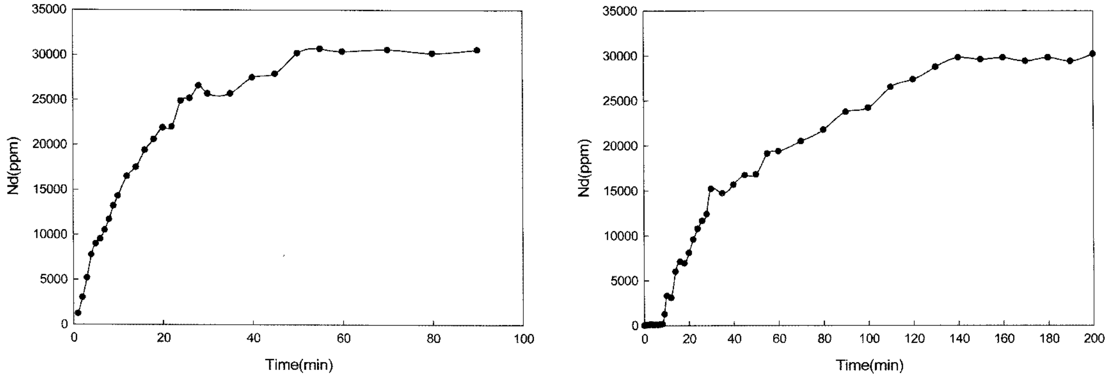
settler-type extractor and to develop an RTD model using K-RTD based on the results of the experiment.

2. Experiment Setup and Method

The extractor that was used in this experiment consisted of a mixer with a 288 ml capacity, a settler with a 1,080 ml capacity, and a four-stage extractor. Fig. 1 shows the sampling point of a mixer-settler-type extractor. Sampling was performed in the mixer and settler, separately, for the single-stage extractor. For the multi-stage extractor, however, sampling was performed without differentiating the mixer and the settler (they were considered as a single reactor), and the response characteristics that were gathered during the experiment on the one-stage extractor were expanded in the experiment on the four-stage extractor.

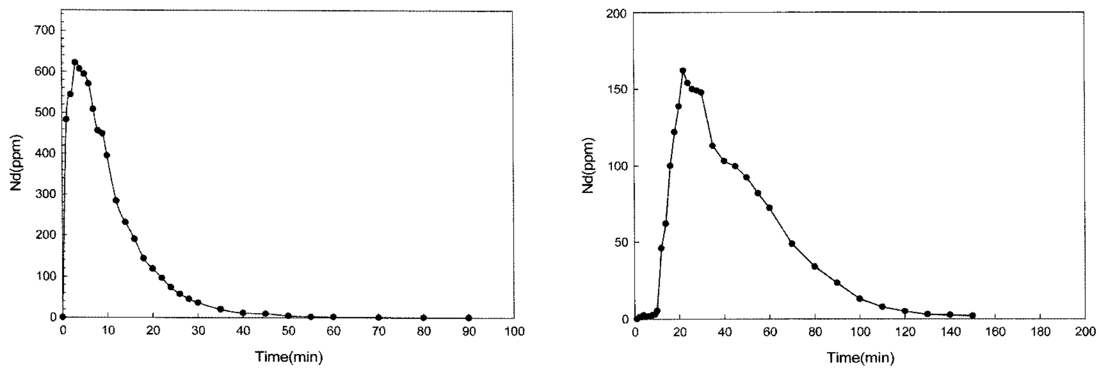
The tracer that was used in this experiment was analyzed using a $CuSO_4$ solution, which can be easily analyzed, and an $NdCl_3$ solution, which was the target for extraction. The same results were obtained regardless of the type of tracer that was used. Therefore, this study reported and examined the data that had been obtained using Nd as a tracer.

The organic and aqueous phases were injected in the mixer, respectively, under the extraction condition and with the same flow rate of 14 ml/min. The extractor was operated for a sufficient time to attain a steady state. After a steady state was attained, a proper concentration of the $NdCl_3$ solution was injected into the mixer, then samples were taken at the sampling points at regular intervals, and these were analyzed using ICP-AES (JY38PLUS, JOBIN YVON).



(a) (b)

Fig. 2. Response curve (F-curve) with step input: (a) mixer and (b) mixer-settler.



(a) (b)

Fig. 3. Response curve (C-curve) with impulse input: (a) mixer and (b) mixer-settler.

3. Results and Discussion

3.1. Response Analysis through an Impulse-Response Experiment

An impulse-response experiment was conducted for the single- and multi-stage extractors, as shown in Fig. 1, using Nd, as a tracer, in order to determine the characteristics of the fluid flow of the mixer-settler-type extractor.

3.1.1 Response characteristics of the single-phase extractor

Fig. 2 shows the F-curves (response curves) of the mixer, and the mixer-settler obtained through the step injection of a 0.2-M $NdCl_3$ solution as a tracer, to ensure a şequential stimulus input for the reactor. As shown in the figure, the response curve of the mixer showed the characteristics of CSTR, and a steady state was attained about 60 min after the impulse. Meanwhile, when the sample was taken and analyzed by considering the one-stage mixer-settler extractor as a single reactor, an about 10-min lag time was identified, and a steady state was attained in the reactor about 150 min after the impulse.

Fig. 3 shows the C-curves that were obtained by applying the impulse to the reactor, where the impulse that was applied was the instant injection of 1.0 ml of a 1-delete M $NdCl_3$solution. As shown in the figure, the response curve of the mixer was the typical curve of a continuous mixer reactor, except in the initial stage of 1 to 2 min, similar to the F-curve, and it attained a steady state after 60 min, which is also similar to the F-curve. When the response curve was obtained by considering the mixer-settler extractor as a single reactor, an about 10-min lag time was identified, similar to the F-curve, and it attained a steady state about 150 min after the impulse.

As shown in the above results, the fact that the time lag occurred was identical to the times for attaining steady state means that their average retention times are likewise identical and thus that the average retention time of the reactor is the same regardless of the stimulus. As such, it can be used to reveal the characteristics of the reactor, whatever the stimulus is, as long as the response of the stimulus could be analyzed properly. Accordingly, a stimulus-response experiment was conducted in the following experiments, using an impulse-type stimulus for which numerical analysis could be performed relatively easily.

3.1.2. Response characteristics of the multi-phase extractor

No time lag was observed for the mixer-only-type extractor from the response characteristics of the one-stage extractor using an impulse or step stimulus, whereas a time lag was observed for the mixer-settler-type extractor. Typically, the mixer-settler-type extractor is made by connecting several extractors, and

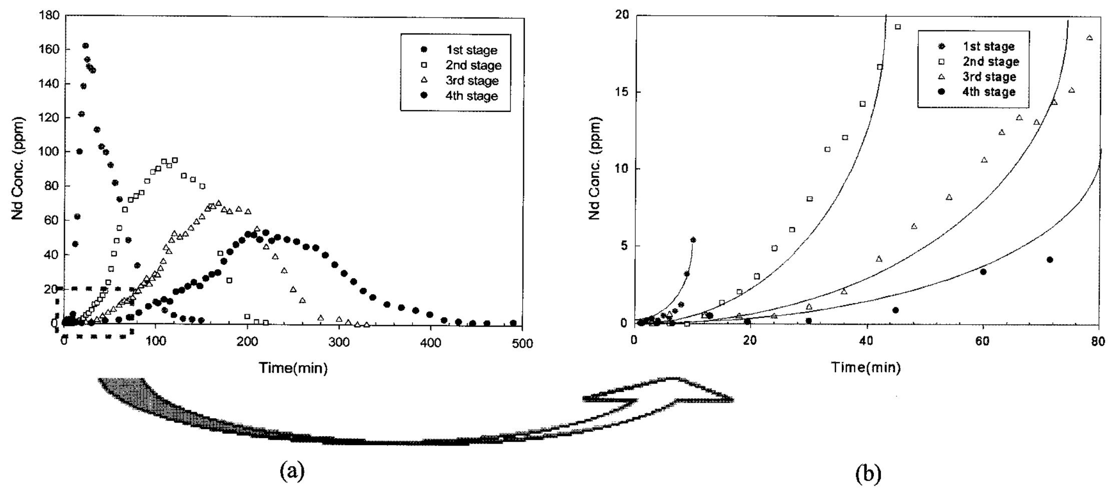


Fig. 4. Response curve of the stage number (a) full scale and (b) initial time lag.

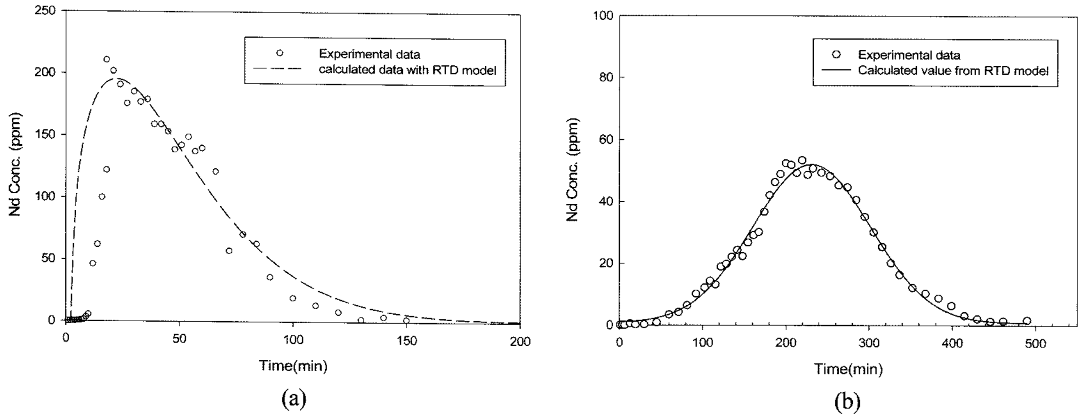


Fig. 5. Impulse response curve and calculated curve using the RTD model: (a) one-stage extractor; and (b) four-stage extractor.

several tens of extractors are connected and used to separate rare earth elements. Thus, it is very important to identify the effect of such time lag as the number of stages increases. Therefore, in the stimulus-response experiment that was conducted in this study, the number of stages of the mixer-settler-type extractor was increased from one to four. The results of the experiment are shown in Fig. 4.

The results of the experiment show that the lag time between the response and the stimulus increases as the number of stages increass: it was extended from about 8 min for the one-stage extractor to 45 min for the four-stage extractor. Such lag time varies depending on the flow rate of the aqueous phase.

3.2 RTD Model Analysis Using K-RTD

In chemical industry, the dynamic behavior of the reactor must be analyzed so that the process employing the reactor could be effectively controlled. Owing to the demand for such and to the advanced computer simulation techniques avaiable, commercial programs for analyzing the results of stimulus-response experiments and for identifying the characteristics of the reaction through dynamic-behavior analysis using an RTD model, etc. have been developed and are being sold at a high price. In these commercial programs, however, the input data which are required for the dynamic-behavior analysis of the reactor must be derived from the actual operation process. Thus, more time and higher costs are required, and more data must be secured by conducting experiments repeatedly.

In this study, K-RTD, a simulator developed and supplied by the Korea Atomic Energy Research Institute (KAERI), was used for the analysis of an RTD model simply by employing transient responses. K-RTD is a program that can analyze the RTD models of most reactors with fluid flow by using a radiotracer, that can calculate the average retention time, and that is used by local and foreign RTD researchers. It is known that highly accurate results, as accurate as the results that can be obtained using the expensive commercial programs, can be obtained using the K-RTD program.

The response data that were obtained from the one- and four-stage extractors mentioned above were analyzed, using K-RTD, to in turn also analyze the RTD model of the mixer-settler-type extractor used in this study.

The unit number of the complete mixer, which consisted only of a one-stagemixer-settler extractor that was analyzed using K-RTD, was 2.7, and the average retention time was 34.77 min. Therefore, k = 2.7, t_0 = 12.88, and t = 34.77 for the one-stage extractor. Its RTD function *E(t)* can thus be written as follows:

$$E(t) = 0.045(0.077t)^{1.7}\exp(-0.028t) \qquad (1)$$

When the response data that were obtained for the four-stage extractor using K-RTD were analyzed, the number of the complete mixer was found to be 11.1, and the average retention time was 148.22 min. Therefore, k = 11.1, t_0=13.35, and t=148.22 for the four-stage extractor. Its RTD function *E*(*t*) can thus be written as follows:

$$E(t) = 0.099(0.075t)^{10.1}\exp(-0.00267t) \quad (2)$$

Fig. 5 shows the experimental data and calculated values for the one-stage and four-stage extractors, which were obtained using Eq. (1) and Eq. (2), respectively. As can be seen in the figure, the analysis model that was used for the four-stage extractor simulated the experimental data better than for the one-stage extractor (the correlation coefficient for the one-stage extractor was 0.963, and 0.995 for the four-stage extractor). Moreover, the time lag was not represented in the calculated values for the one-stage extractor, but it was represented quite correctly for the four-stage extractor. This can be attributed to the fact that the dead-space error was not included in the experiment data for the one-stage extractor, whereas the dead space was incorporated in the experiment data for the extractor with at least three stages.

4. Conclusions

A response characteristics experiments were conducted for one-stage and multi-stage extractors to identify flow characteristics of the material in the mixer-settler-type extractor. The results of the experiment show that the response characteristics of the one-stage extractor are the same as those of the complete mixer reactor, and that the retention time of the mixer-settler-type extractor becomes longer as the number of extraction stages increases.

The experimental data that were obtained for the response characteristics of one-stage and four-stage extractors were analyzed using K-RTD, a response analysis program, to obtain a retention time distribution model for one-stage and four-stage extractors. The calculated values that were obtained are as follows:

RTD model for the one-stage extractor:

$$E(t) = 0.045(0.077t)^{1.7}\exp(-0.028t)$$

RTD model for the four-stage extractor:

$$E(t) = 0.099(0.075t)^{10.1}\exp(-0.00267t)$$

The correlation coefficient between the calculated values and the experimental data was 0.963 for the one-stage extractor and 0.995 for the four-stage extractor, respectively, showing quite a high precision.

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