MULTISTAGE STIRRING, LEACHING AND WASHING TOWER & ITS APPLICATION IN Re ORE EXTRACTION①

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ABSTRACT

The multistage stirring, leaching and washing tower (MSLWT) was designed to make it possible to finish leaching and washing of ore in one equipment. The residence time distributions (RTD) of MSLWT were studied. Peclet number, $\bar{t}$, σ^2 were calculated based on the diffusion model, the results indicate the diffusion model is suitable to MSLWT. The application of MSLWT to the leaching of rare earth ore increases the recovery to above 92% and reduces the washing factor to less than 0. 06.

Key words: RTD rare earth leaching and washing

1 INTRODUCTION

In the chemical and hydrometallurgical industry there are several types of reactors to deal with the leaching of solid ore. The stirring tanks are used in solid-liquid system [1-5]; Pachuca tanks are used in gas-liquid-solid system[6]; fluidizeds bed are used in gas-liquid or liquid-solid system [7-9]. The operation circuits will be complicated if the reactors mentioned above are used because the leaching and washing processes are carried out in two or more reactors independently. The purpose of this research is to develop a new type of tower in which the leaching and washing processes can be finished simultaneously to simplify the operation process.

2 EXPERIMENTAL APPARATUS AND METHOD

The experimental apparatus is shown in Fig. 1. The tower was made of perspex with an internal diameter of 82 mm and a height of 1,148 mm. It was partitioned by 10 rings whose internal diameter are 50 mm into 9 mixing chambers and two settlers. The settlers at the top and bottom of the tower are twice as big as the mixing chamber. The solid ore goes down chamber by chamber from the top of the tower. In the leaching section which consists of 6 mixing chambers, the powder of ore is mixed with the leaching agent by stirring and the rare earth elements of the ore are extracted . In the washing section which consists of 3 mixing chambers, the leached ore was washed with clean water. Finally the waste ore was concentrated at the bottom settler and was drained out of the tower. The washing liquid entered into the bottom settler through the distributor. The leaching agent entered into the tower through the inlet between the two sections. The leaching solution flowed out through the

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outlet at the top of the tower.

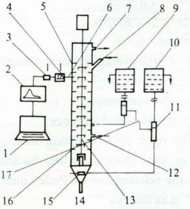

Fig. 1 Sketch of experimental apparatus

1—Computer; 2—Recorder; 3—Amplifier and A-D transmitter; 4—Conductometer; 5—Tower body; 6—Washing solution distributor; 7—Leaching solution tank; 8—Washing solution tank; 9—Flowmeter; 10—Inlet for tracer; 11—Inlet for pulp; 12—Overflow outlet; 13—Slag outlet; 14—Stirring shaft; 15—Conductor electrode; 16—Dummy plate;

The trace pulse method was applied to determine the RTD. The saturated NaCl was injected as tracer into the sixth mixing chamber. A conductometer was set at the first mixing chamber from the top to measure the concentration responses. The electrical signals were sent to the computer with amplifier and A-D transmitter. According to the concentration response curve, the RTD of this equipement was obtained.

In the leaching experiment , the size of the raw ore of rare earth ranged from −40∼−120 mesh, the size distribution of the ore is shown in Table 1.

Table 1 The size distribution of the rare earth ore

size (mesh)	+40	40∼60	60∼80	80∼100	100∼120	−120
wt.−%	44.40	18.30	9.50	15.96	5.93	5.92

Batch operation was applied. When the operation time is t, the samples of the overflow were taken and the rare earth concentration in it was analysed. The concentration of rare earth in residual liquid was also analysed. ICAP 9000 SP was used to measure the concentration of rare earth elements.

3 RESULTS AND DISCUSSION

3.1 *Residence Time Distribution*

In MSLWT, RTD changes with the change of operation condition. Because residence time (t) distribution density function $E(t)$ is $E(t)=Q/Cq$ and the concentration of tracer C in equipment is the linear function of voltages on the conductometer probe, the $E(t)$ can be calculated from the voltage response curves. Where Q is the blowrate (L / h), and q the mass of tracer(kg). The concentration response curves at the different stirring speeds and flow rates are shown in Fig. 2−8. Fig. 2∼4 show that at constant stirring speed the peaks move toward the left, the curves become narrower and narrower and the backmixing decreases as the flow rate increases. Under the experimental conditions, the increase of flow rate makes it possible that more and more fluid micro units flow away before they mix with fresh fluid, so the possibility of mixing of micro units with different RTD with each other becomes less. Therefore the backmixing also becomes less. Fig. 5∼8 shows that as the stirring speed increases, the peaks move towards the right and the curves become wider and wider, the convection-diffusion becomes large, the fluid micro units with the different RTD mix fully in the turbulent field and result in a great backmixing.

As fluid flows slowly, such as 80 L / h, the curves have long tails due to the dead zone in the mixing chambers.

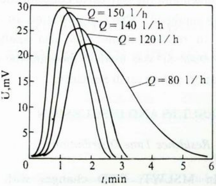

Fig. 2 Concentration response curves
($n=150$ r / min)

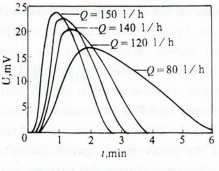

Fig. 3 Concentration response curves
($n=300$ r / min)

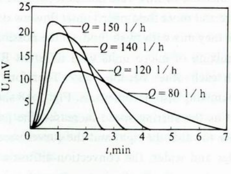

Fig. 4 Concentration response curves
($n=450$ r / min)

With the help of computer sampling, the RTD function $E(t)$ can be calculated from the experimental data according to the following equation

$$t=\int_0^{\infty} t\,E(t)\mathrm{d}t \tag{1}$$

$$\sigma_t^2=\int_0^{\infty} t^2E(t)\mathrm{d}t \tag{2}$$

$$\sigma^2=\sigma_t^2 / t_2 \tag{3}$$

where $\hat{t}$—the average residence time,s;

t_2—time, s;

σ_t^2—variance;

σ—Standard variance

The open pulse injection method is applied in this research, when backmixing is great, the relationship of Peclet number (Pe) and σ^2 is as follows [10]

$$\sigma^2=2 / \mathrm{Pe}+8(1 / \mathrm{Pe})^2 \tag{4}$$

If the backmixing is less the boundary conditions have little effect on the shape of $E(t)$ curves. In the case of open operation or close operation, the following relationship exists[10].

$$\sigma^2=2 / \mathrm{Pe} \tag{5}$$

The experimental conditions and calculation results under different operating conditions are shown in Table 2.

Under the condition of constant stirring speed, as the flow rate increases, Pe_1, Pe_2 increase and σ_t^2, σ^2 decrease. It indicates that

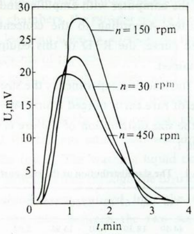

Fig. 5 Concentration response curves
($Q=150$ L / h)

Table 2 Calculation results under different operating conditions

Q / L · h^{-1}	n / r · min^{-1}	t / s	Q_t^2	Q^2	Pe_1	Pe_2
150	150	74.01	588.3	0.1074	22.01	18.62
		78.68	634.1	0.1024	22.93	19.52
		76.13	616.6	0.1064	22.19	18.79
140	150	81.84	711.6	0.1063	22.21	18.81
		81.05	727.5	0.1115	21.43	18.06
		81.83	732.7	0.1092	21.65	18.19
120	150	91.29	931.6	0.1118	21.26	17.54
		91.41	952.5	0.1114	21.90	17.25
		93.24	1 007.8	0.1159	21.64	17.89
80	150	146.5	3228.8	0.1504	16.52	12.87
		142.8	3170.9	0.1554	16.07	12.99
		141.0	3061.1	0.1539	16.20	13.40
150	300	75.49	739.9	0.1298	18.70	16.16
		78.40	760.6	0.1237	19.48	15.40
		78.91	803.2	0.1289	18.80	15.51
140	300	83.48	1014.1	0.1455	16.98	13.74
		85.71	999.8	0.1361	17.97	14.70
		86.04	1 063.7	0.1437	17.16	13.92
120	300	97.14	1519.3	0.1611	15.60	12.42
		99.29	1523.6	0.1554	16.14	12.94
		96.31	1397.7	0.1510	16.49	13.27
80	300	147.7	4195.2	0.1992	13.49	10.41
		148.3	4367.8	0.1986	13.13	10.07
		153.2	4850.4	0.2066	12.72	9.680
150	450	81.26	655.7	0.1291	18.78	15.45
		80.79	717.9	0.1432	17.21	13.96
		85.78	819.7	0.1427	17.25	14.01
140	450	93.80	1320.4	0.1680	15.03	12.72
		94.85	1333.4	0.1482	16.72	13.49
		84.23	1298.7	0.1838	14.04	10.93
120	450	102.3	2175.1	0.2077	12.67	9.629
		107.3	2418.0	0.2098	12.56	9.529
		106.7	2302.3	0.2023	12.94	9.885
80	450	163.1	7556.2	0.2643	10.46	7.567
		163.9	6973.4	0.2595	10.61	7.708
		160.7	6567.4	0.2542	10.79	7.869

*: Pe_1 and Pe_2 were calculated from formula (4) and (5) respectively.

back-mixing becomes less. Under the condition of constant flow rate, as stirring speed increases, σ^2 and E_2 increase and Pe decreases, Which indicates that backmixing has become great. The change of σ_t^2 , σ^2 and Pe_1, Pe_2 is in agreement with the change of RTD curve at the same operation conditions, which indicates that under the given conditions, the diffusion model can satisfactorily describe the fluid flow pattern in MSLWT.

3.2 *Leaching of Rare Earth Ore*

There is no problem of environment pollution and soil impoverishment provided that

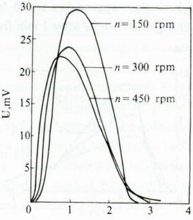

Fig.6 Concenration response curves of the trace pulse method
(Q = 140 L / h)

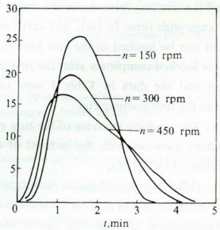

Fig.7 Concentration response curves of the trace pulse method
(Q = 120 L / h)

$(NH_4)_2SO_4$ is used as leaching agent instead of sodium chloride. The concentration of $(NH_4)_2SO_4$ was 2%, the ratio of liquid to solid was 3 : 1. Experiments were carried out under the leaching condition of no stirring and the relationship between the recovery of rare earth elements (R_{Re}) and the leaching time (t) is shown in Fig.9. It can be seen from Fig.9. that the recovery R_{Re} approaches to 90 % and hardly changed with time after 2 min from the beginning.

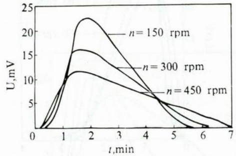

Fig. 8 Concentration response curves

(Q = 80 L / h)

Under stirring condition, it was found that the leaching recovery was more than 90% and the leaching rate was so fast that it did not change with time. In fact, rare earth elements that can be leached in the raw rare earth ore were leached completely after the first leaching run and the data in Table 3 were obtained from the first leaching run.

The leaching recovery of the rare earth elements increases with the increase of concentration of $(NH_4)_2SO_4$.

After a comprehensive investigation on the leaching rate of rare earth elements and the comsumption of chemicals agents and other items, the solution of $(NH_4)_2SO_4$ with concentration of 2 wt.-% was finally selected as leaching agent.

Table 4 shows the experimental conditions and leaching-washing results of rare earth elements in MSLWT. All of the leaching recovery (R_{Re})were more than 92% and the ratio of the concentration of Re in exhausted liquid(C_d) and leaching liquid C_e was less than 0. 06. Under the experimental operation conditions, the diluted and concentrated phases sections in the tower are stable in operation with negligible backmixing action between the two sections. Therefore, better washing efficiency could be gained. The amount of liquid entered in solid phase (L_s) is less than 30% . The MSLWT is proper for leaching and washing operation simultaneously and continuously. The pulp density of overflow (S_L) was also measured. It increased with an increase of the stirring speed.

Table 3 Effects of concentration of $(NH_4)_2SO_4$ on leaching recovery

$(NH_4)_2SO_4$ %	1	2	3	4	5
E_{Re} %	90.8	92.0	95.1	95.7	96.4

According to the results of leaching and washing of rare earth and movement pattern of particles in the tower, stirrer rotation speed of 300 r / min was used in the experiments. It was also found that the washing efficiency changed with the change of the ratio of flowrate of the washing agent to that of the leaching agent. By comparison of experimental results of Series No2、6、4、7、5、8 in Table 4, it was found that as the ratio of flowrate of washing agent to that of leaching agent changed from 3 : 1 to 2 : 1, C_d. C_e had a little increase and washing efficiency decreased. The experiments showed that the range of the ratio of the two kind of agents which was rinsed in the experiments was adaptable to the operations. If the ratio was too large, it would cause the reduction of leaching agent concentration and recovery of rare earth elements.

Table 4 Experimental data of leaching and washing of rare earth ore

Item / Series NO	Q_W / $L \cdot h^{-1}$	Q_L / $L \cdot h^{-1}$	Q_P^* / $Kg \cdot h^{-1}$	C_P^* / %	n / $r \cdot m^{-1}$	C_e / ppm	C_d / ppm	C_{Re} / ppm	S_L / $Kg \cdot L^{-1}$	L_S / vol.-%	C_d / C_e	R_{Re} / %
1	120	40	106	50	450	16.98	0.8922	18.39	0.060	74.0	0.052	92.3
2	120	40	106	50	300	18.68	0.8966	20.13	0.0553	72.1	0.048	92.3
3	120	40	106	50	150	19.30	0.8777	20.75	0.0491	75.2	0.045	93.0
4	90	30	80	50	300	21.60	0.9536	23.20	0.0501	70.7	0.044	93.1
5	60	20	53	50	300	18.42	0.8722	19.89	0.0506	80.2	0.047	92.6
6	100	50	100	50	300	16.77	0.9720	18.21	0.0496	66.3	0.058	92.1
7	80	40	80	50	300	21.05	1.158	22.85	0.0573	70.4	0.055	92.1
8	60	30	60	50	300	20.20	1.151	21.79	0.0369	74.1	0.057	92.7
9	40	20	40	50	300	20.72	0.9862	22.23	0.0289	70.3	0.048	93.2

Note: All samples were diluted with water in equal volume: Q_W—flowrate of washing agent; Q_L—flowrate of leaching agent; Q_P—flowrate of pulp; C_P—concentration of pulp.

4 CONCLUSION

In MSLWT, each mixing chamber is an ideal mixer. Between the neighboring mixing, the degree of backmixing is related to stirring speed and the flowrate of solid phase and liquid phase . The experimental results showed that the flow pattern in MSLWT was quite well described by diffusion model with backmixing, so the model can be used as a basis of designing these kinds of reactors and determining optimum operating conditions.

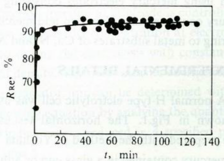

Fig 9 Leaching recovery–time curve of the rare earth ore with non-stirring

MSLWT makes it possible to finish leaching and washing of the rare earth ore simultaneously and continuously in one equipment . It has the advantages of simple structure and convenient operation. Under the experimental conditions, diluted phase zone sections could be formed in the sedimentation settler and the interface between the two zones was stable . The extracted ore of rare earth elements could be washed fully and the loss of the rare earth elements would be minimal. The goal of higher leaching recovery and better washing efficiency will be achieved if MSLWT is used.

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