Seepage mechanism during in-situ leaching process of weathered crust elution-deposited rare earth ores with magnesium salt

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Abstract: To reveal seepage mechanism during in-situ leaching process of weathered crust elution-deposited rare earth ores with magnesium salt, the effects of particle gradation, particle migration, Atterberg limit on the permeability coefficient were investigated, and the relation between the particle size and rare earth content was discussed. The results showed that the ore in the humic layer (HL) with high porosity and permeability was uniformly graded particles. The ore in the completely weathered layer (CWL) with low porosity and permeability belonged to dense-graded particles. The ore in the partly weathered layer (PWL) was open-graded particles, whose permeability fell in between the HL and the PWL. The change of -0.075mm particles content was the largest in the leaching process. When -0.075mm particle content was less than 30%, the migration ability of fine particles and the permeability coefficient decreased gradually. On the contrary, the migration ability of fine particles gradually remained stable, and the change in the permeability coefficient was not obvious. The liquid limit (LL) in the Atterberg limit of HL, CWL and PWL was inversely proportional to the permeability coefficient, and followed the order: LL_{HL} < LL_{PWL} < LL_{CWL}. With the -0.075mm particle content increasing, the LL of the ore samples increased gradually and finally tended to be stable. The peak value of rare earth concentration appeared earlier and the rare earth content decreased gradually with the increase of the ore particle size. This work provided a theoretical basis for achieving high-efficient mining of weathered crust elution-deposited rare earth ores.

Keywords: seepage mechanism, weathered crust elution-deposited rare earth ores, in-situ leaching, magnesium salt

1. Introduction

Weathered crust elution-deposited rare earth ores are rich in middle and heavy rare earth and belong to national strategic resources, which are widely distributed in eight southern provinces in China, for example, Jiangxi, Fujian, Guangdong, Guangxi, Zhejiang, Hunan, Yunnan and Guizhou provinces (Chi and Tian, 2008). The weathered crust elution-deposited rare earth orebody is divided into three layers (Fig. 1), humic layer (HL), completely weathered layer (CWL) and partly weathered layer (PWL), from top to bottom according to the degree of weathering (Chi et al., 2005). In order to achieve the highly efficient mining of weathered crust elution-deposited rare earth ores, the mining technology has developed the pool leaching (Chi and Wang, 2014), heap leaching (Li, 2014) and in-situ leaching (Huang et al., 2010) since studied in 1969. Nowadays in-situ leaching technology has been gradually developed into mature and widely applied in mines in recent years. However, there are some problems existed in this process, including the slow seepage rate of leaching fluid and the low leaching rate of rare earth, etc., which can not efficiently leach the rare earth from the weathered crust elution-deposited rare earth ores.
In view of the above problems, a lot of research on the factors influencing the seepage in the leaching process of rare earth ore have been carried out. In order to optimize the leaching process, Tian et al (2010) performed the leaching kinetics experiments using three-necked experimental apparatus. It was found that particle size had a big impact on the permeability and leaching rate. The effects of concentration and pH, temperature on the seepage of the leaching agent solution were investigated, and the optimum conditions for the high permeability were also proposed (He et al., 2016; Chen et al., 2019). Due to the rare earth mainly absorbed on the clay minerals, Zhang et al (2018) studied the swelling properties of the weathered crust elution-deposited rare earth ores and showed that cation had an effect on the seepage of the leaching solution. It also indicated that the leaching agent played a key role in the process of ion-exchange reaction. Nowadays ammonium salts widely used in the mines as leaching agents were easy to lead to the enrichment of ammonia nitrogen causing water eutrophication (Li et al., 2010). So lots of scholars have been exploring the new leaching agent in recent years, such as magnesium salt (Xiao et al., 2015), carboxylate salt (Zhou et al., 2019) and compound salt (Lai et al., 2018), etc. Huang et al (2005) found that magnesium salt has high permeability and leaching rate, which was recognized as the most likely substitute for ammonium salt in the proposed new leaching agent because of no ammonia nitrogen. A new technology of unsaponified extraction and separation of rare earths by her and her team, which has been applied in industry. In addition, Wu et al (2007) found that preferential flow had a great influence on the effective pore structure determined by particle size, which controlled the seepage of leaching solution and extraction. It was also indicated that the permeability properties of rare earth ores were mainly influenced by the pore channels influenced through the pore size, pore direction and pore distribution (Wu et al., 2009).

In summary, it could be seen that there were many factors influencing the seepage of leaching solution in the weathered crust elution-deposited rare earth ores, which could be divided into chemical factors and physical factors according to two major phases, such as ion-exchange process and seepage diffusion movement. Chemical factors contained the leaching agent type, concentration, pH and temperature, etc. Physical factors included injection strength, liquid-solid ratio, particle size and pore structure, etc., in which the particle size played a key role in determining the permeability of the rare earth ores (Zhou et al., 2019). But there are few findings on how the particle size affects the permeability of the weathered crust elution-deposited rare earth ores. There were three factors, such as particle gradation, particle migration and Atterberg limit, which were directly related to the particles size. It was known that the pore structure of rare earth ores was determined by the particle gradation which controlled the effective seepage channel. The phenomenon of fine particle migration occurred during the leaching process (Yang et al., 2014), which had a big impact on the seepage and diffusion movement of the leaching solution. The liquid limit (LL) in the Atterberg limit determines the critical point of ore particle flow (Mbonimpa et al., 2002), which controlled the critical hydraulic gradient of the leaching solution. However, it was rarely reported that the effects of the particle gradation, particle
migration and Atterberg limit on the permeability and seepage mechanism of the weathered crust elution-deposited rare earth ores in the leaching process.

In view of this, the rare earth ores of HL, CWL and PWL in the weathered crust elution-deposited rare earth ores 2# and 4# in Dingnan, Jiangxi province in China, were taken as research objects in this study. In order to reveal the seepage mechanism during in-situ leaching of the weathered crust elution-deposited rare earth ores, the effects of particle gradation, particle migration and the LL in the Atterberg limit on the permeability of rare earth ores were investigated, and the relation between particles size and rare earth content was discussed as well. The research results are helpful to provide a theoretical basis for achieving high-efficient mining of the weathered crust elution-deposited rare earth ores.

2. Experimental

2.1. Experimental samples

Experimental samples were selected from the ores of HL, CWL and PWL in the weathered crust elution-deposited rare earth ores 2# and 4# in Dingnan, Jiangxi province in China. Collected rare earth ore samples were screened according to the particle size of -0.075 mm, 0.075-0.15 mm, 0.15-0.45 mm, and +0.45 mm, respectively. The weight percentage of different types of ores samples with four kinds of particle size as shown in Fig. 2. Where purple represented -0.075 mm particles, blue represented 0.075-0.15 mm particles, red represented 0.15-0.45 mm particles, and black represented +0.45 mm particles.

![Graph showing weight percentage of different types of ores samples with four kinds of particle size](image)

As shown in Fig. 2, there were differences in the quality distribution of the rare earth ores with different particles in three ore layers. The weight percentage with +0.45mm particles in the weathered crust elution-deposited rare earth ores followed the order of HL>PWL>CWL. However, the weight percentage with -0.075mms particles followed the reverse order of HL<PWL<CWL.

2.2. Experimental methods

2.2.1. Column leaching experiment

In this study, 0.2mol /dm³ leaching solution was prepared using MgCl₂ as the leaching agent. Selected ore samples of the weathered crust elution-deposited rare earth ores were measured and put into a glass column with an inner diameter of 45mm. The leaching agent solution was injected into the glass column
at a constant flow rate using a constant peristaltic pump. The lixivium was collected with a measuring cylinder and recorded.

### 2.2.2. Calculation method of the permeability coefficient and non-uniformity coefficient

#### 2.2.2.1. Permeability coefficient

The seepage diffusion movement of leaching fluid belonged to laminar flow, which conformed to Darcy’s law. Therefore, the permeability coefficients of the ore particles can be obtained by the following expression (He et al., 2017):

\[ k = \frac{v}{j} \]  \hspace{1cm} (1)

where \( k \) was the permeability coefficient, cm/s; \( v \) was seepage velocity, cm/s; \( j \) was the hydraulic gradient.

#### 2.2.2.2. Non-uniformity coefficient

Non-uniformity coefficient was used to reflect the uniformity of the particles in the soil. It was expressed through the ratio of restricted particle size to effective particle size (Bunzl, 2001):

\[ Cu = \frac{d_{60}}{d_{10}} \]  \hspace{1cm} (2)

where \( Cu \) was the non-uniformity coefficient; \( d_{60} \) was the particle size whose weight percentage was less than 60%; \( d_{10} \) was the particle size whose weight percentage was less than 10%.

#### 2.2.3. Type of particle gradation

Particle gradation was divided into four types, such as uniformly graded, open-graded, dense-graded and gap-graded, according to the distribution curve of soil particle grading (Mamlouk and Zaniewski, 2001). Uniformly graded particles were mainly composed of large particle size and few fine particles, and the gradation curve was steeper. Open-graded particles were constituted from large particle size and small particle size in appropriate proportion. Dense-graded particles were continuous, intercalated and compact, and the gradation curve was even and smooth. Gap-graded particles were discontinuous without one or more grades in the particle gradation composition, and the gradation curve was approximate flat in the middle.

#### 2.2.4. Analysis procedure of particle migration

After the column leaching experiments, the ore sample was divided into upper, middle and bottom parts according to the height of the ore sample. The divided ore samples were dried, screened and weighed to analyze the migration law of ore particles.

### 3. Results and discussion

#### 3.1. Effect of particle gradation on permeability

100g of the ores samples of HL, CWL and PWL in the weathered crust elution-deposited rare earth ores 2# and 4# were packed into the column, respectively. Leaching experiments were carried out with 0.2 mol/dm$^3$ of MgCl$_2$ solution and wherein the hydraulic gradient was 0.25, 0.75, 1.25, 1.75 and 2.25. The results were shown in Fig. 3, where the slope of the line represented the permeability coefficient according to Eq. (1).

Fig. 3 showed that the permeability coefficient of the rare ores in the HL was the largest, while the rare ores in the CWL was the smallest. The permeability coefficient of the ores of HL, CWL and PWL in the weathered crust elution-deposited rare earth ores 2# and 4# followed the order of $k_{4\#,2\#,HL} > k_{2\#,4\#,HL} > k_{2\#,4\#,PWL} > k_{4\#,4\#,PWL} > k_{2\#,2\#,PWL} > k_{2\#,2\#,CWL} > k_{4\#,4\#,CWL}$. Compared with Fig. 1, it was easily found that there was a good correlation between the particle gradation and permeability coefficient (Sheikhzadeh et al., 2001).

In order to explore the effect of particle gradation on the permeability coefficient, the type of particle gradation and non-uniformity coefficient were analyzed from qualitative and quantitative perspectives respectively in this study.
3.1. Type of particle gradation

Particle gradation of the ores of HL, CWL and PWL in the weathered crust elution-deposited rare earth ores 2# and 4# were investigated by using laser particle analyzer (Winner2308). The particle gradation curves were shown in Fig. 4.

The results showed that the rare earth ores particles in the HL, CWL and PWL belonged to uniformly graded, dense-graded and open-graded particles respectively according to the judgment standard of the type of particle gradation in section 2.2.3. Compared with the permeability coefficient of three weathered layer ores in Fig. 1, it was found that uniformly graded particles showed the characteristics of high porosity and permeability due to the high proportion of large particle size. Dense-graded particles had low porosity and permeability because the ore particles were continuous and embedded in each other. Permeability of open-graded particles fell in between uniformly graded particles and dense-graded particles.

3.1.2. Non-uniformity coefficient

Particle parameters \(d_{10}\) and \(d_{60}\) and non-uniformity coefficient \(Cu\) of the rare ores in different weathered layers can be obtained according to Fig. 4 and Eq. (2), the results were shown in Table 1. In addition, the relation between the non-uniformity coefficient and permeability coefficient was displayed in Fig. 5.

It could be seen from Fig. 5 that the permeability coefficient of the rare earth ores was inversely correlated with the non-uniformity coefficient, which demonstrated that the permeability coefficient of
the weathered crust elution-deposited rare earth ores decreased with non-uniform coefficient increasing. The reason was that the smaller the non-uniform coefficient of the ore particles, the more uniform particle size distribution and the greater the intergranular porosity. With the non-uniform coefficient decreasing, the particle skeleton became more stable gradually and particles migrated fewer gradually (Fiès, 1992). Therefore, the rare earth ores with low non-uniform coefficients showed good permeability. On the contrary, with the non-uniform coefficient increasing, the stability of the particle skeleton became worse gradually and the phenomenon of fine particle migration was easy to occur, which led to gradual attenuation of the permeability coefficient.

Table 1. $d_{10}$, $d_{60}$ and Cu of ore particles in different weathered layer

<table>
<thead>
<tr>
<th></th>
<th>2#</th>
<th>4#</th>
</tr>
</thead>
<tbody>
<tr>
<td>$d_{10}$</td>
<td>HL</td>
<td>CWL</td>
</tr>
<tr>
<td></td>
<td>0.0825</td>
<td>0.0334</td>
</tr>
<tr>
<td>$d_{60}$</td>
<td>0.6103</td>
<td>0.3562</td>
</tr>
<tr>
<td>Cu</td>
<td>7.3976</td>
<td>10.6647</td>
</tr>
</tbody>
</table>

Fig. 5. Relation between the non-uniformity coefficient and permeability coefficient of the ores

3.2. Effect of particle migration on permeability

The phenomenon of particle migration occurred in the leaching process (Yang et al., 2014). It resulted in changes in effective pore channels, which directly influenced the permeability of the rare earth ores. The factors that affect particle migration were particle size and particle content with different particle sizes, which caused a difference in the ores particle content. Therefore, it was essential to study the effect of the particle size and particle content on the particle migration. In addition, the relation between the particle migration and the permeability of the weathered elution-deposited rare earth ores were discussed as well.

3.2.1. Effect of particle size on the particle migration

Screened ores particle of -0.075 mm, 0.075-0.15 mm, 0.15-0.45 mm and +0.45 mm in the CWL of the rare earth ores 2# were taken as 8g, 8g, 8g and 100g, respectively. Leaching experiments were carried out with 0.2 mol/dm$^3$ of MgCl$_2$ solution and wherein the hydraulic gradient was 2. Four kinds of ore particles were mixed evenly and recombined to form a new experimental sample before conducting the column leaching experiments. Before and after leaching, the upper, middle and bottom parts of ore samples were weighed, recorded and statistically analyzed to obtain the migration amount of the ore particles with different particle sizes in the leaching process according to the analysis method of particle migration in section 2.2.4. The results were shown in Table 2.
Table 2. Migration amount of the ore particles with different particle size in the leaching process

<table>
<thead>
<tr>
<th>Particle size / mm</th>
<th>Ore weight after leaching/ g</th>
<th>Weight percentage of the ore particles before and after leaching/%</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Upper part</td>
<td>Middle part</td>
</tr>
<tr>
<td>-0.075</td>
<td>0.11</td>
<td>1.25</td>
</tr>
<tr>
<td>0.075-0.15</td>
<td>2.06</td>
<td>2.91</td>
</tr>
<tr>
<td>0.15-0.45</td>
<td>2.59</td>
<td>2.69</td>
</tr>
<tr>
<td>+0.45</td>
<td>33.31</td>
<td>33.34</td>
</tr>
</tbody>
</table>

As can be seen from Table 2, the migration amount of -0.075mm particles was the largest, and the maximum migration of ore particles within this size range was 149.05%. It indicated that the upper and middle parts of the ores with -0.075mm particle had a large degree of migration. The migration amount of 0.075-0.15mm particles was smaller than that of -0.075mm particles, and the migration rate of the upper ore particles was up to 22.84%. It demonstrated that the ore particles in the range from 0.075 to 0.15 mm particle size migrated to the middle and bottom part with the leaching agent solution. The 0.15-0.45mm particles rarely migrated, and the maximum migration was 2.99%. The migration amount of +0.45mm particle size was the least, and the maximum migration was only 0.07%. The reason was that the large particles with the least migration amount played a skeleton supporting role in the leaching process (Fig. 6a) while fine particles migrated down through the pore channels. Therefore, -0.075 mm particle size was the most likely to occur migration phenomenon, which was easy to block the seepage channels (Fig. 6b) and played a key role in the seepage process of leaching agent solution.

3.2.2. Effect of particle content on the particle migration

The -0.075mm particles with maximum migration were taken as an example in order to explore the influence of particle content on the particle migration. The -0.075 mm and +0.45 mm ore particles were mixed evenly to form new ore samples with the -0.075mm particle content of 5%, 8%, 11%, 14%, 17%, 20%, 25%, 30% and 35%, respectively. The variation law of the relation between -0.075mm particle content and the particle migration percentage was shown in Fig. 7.

The results showed that the particle migration percentage decreased with the content of -0.075mm
Fig. 7. Relation between -0.075mm particle content and the particle migration rate

particles increasing in the leaching process. When the -0.075mm particle content was less than 8%, the particle migration rate reached more than 93%. It indicated that the migration ability of particles was strong as the -0.075mm particle content was in the range from 0 to 8%. As the -0.075mm particle content was between 8% and 30%, the particle migration rate decreased from 93% to 20% and the migration ability of particles was gradually weakened. When the content of -0.075mm particles was greater than 30%, the migration percentage gradually presented a stable trend. It demonstrated that particles would not migrate at all as the content of -0.075mm particles reached a certain level. The reason was that the more the -0.075mm particle content, the smaller the porosity of the ore samples which would result in less particle migration with the leaching agent solution in the leaching process (Shepherd, 1989).

3.2.3. Relation between the particle migration rate and the permeability coefficient

The experimental results in section 3.2 (2) were taken as research objects in order to explore the effect of the particle migration rate on the permeability coefficient. The change law of the relation between the particle migration percentage and the permeability coefficient was shown in Fig. 8. The results showed that the permeability coefficient of the ore samples increased with the migration percentage of -0.075mm particles increasing. It meant that the migration percentage of -0.075mm particles had a big impact on the permeability coefficient of the ore samples. That was because the large particle content increased with the increase of the particle migration percentage so that the ore particles with a strong migration ability showed a high permeability coefficient due to the high stability of the ore particle skeleton (Zhou et al., 2019).

This behavior could be shown by using a fitting equation adopted to predict the permeability coefficient of the ore particles with the -0.075mm particle content in the range from 5% to 35%. According to the distribution characteristics of experimental results in Fig. 8, polynomial function, power function and exponential function were used to fit the experimental data, and the fitting results were shown in Fig. 8 and Table. 3. The “adequate precision” and the reliability of the obtained model was determined by the correlation coefficient ($R^2$). The larger the $R^2$, the fitting equation was close to the actual values (Aracena et al., 2019; Sobouti et al., 2019). Therefore, the relation between the particle migration rate of -0.075mm particles and the permeability coefficient of the ore samples could be expressed as,

$$y = -0.95 + 28.49x + 6.80x^2$$

where x and y represented the particle migration rate of -0.075mm particles and the permeability coefficient respectively.

The permeability coefficient of the 2# and 4# rare earth ores and the corresponding theoretical results predicted by the fitting function were shown in Table 4. It can be seen from Table 4 that the permeability coefficient could be well predicted by the fitting equation.
3.3. Effect of Atterberg limit on permeability

Liquid limit (LL) in the Atterberg limit of soil particles was one of the key factors affecting the permeability coefficient (Mbonimpa et al., 2002). In order to explore the effect of Atterberg limit on the permeability coefficient of the rare earth ores, the relation between permeability coefficient and LL of ore samples with different weathered layers and the -0.075mm particle content were investigated, respectively.

3.3.1. Relation between permeability coefficient and LL of the ores in different weathered layers

The change law of the relation between the permeability coefficient and LL of the rare earth ores in different weathered layers was shown in Fig. 9. It showed that the liquid limit (LL) in the Atterberg limit of the rare earth ores in the HL, CWL and PWL was inversely proportional to the permeability coefficient. The results were consistent with the experimental results of natural soils without fissures obtained by Mbonimpa et al. (2002). In addition, it can be clearly seen from Fig. 9 that the liquid limit (LL) in different weathered layers followed the order of LL_{HL} < LL_{PWL} < LL_{CWL}. The ore in the HL entered a flow state relatively fast, the ore in the PWL was next, and the ore in the CWL was the slowest. This
was mainly because clay minerals content in the HL was the least while that in the CWL was the largest (Chi and Wang, 2014). High content of clay minerals needed to absorb more water to enter a flow state (Grim et al., 2006).

3.3.2. Relation between permeability coefficient and LL of the ores with different particle content

From section 3.2 (2), it could be known that the difference in the content of -0.075mm particles led to changes in the migration amount of particles, which caused differences in the permeability of the ore samples. Therefore, it was necessary to explore how fast the ore samples with different content of -0.075mm particles enter the flow state and its relationship with the permeability coefficient. Based on the experimental results in section 3.3 (2), the variation law of the Atterberg limit of the ore samples and its relationship with the permeability coefficient was shown in Fig. 10.

Fig. 10 showed that LL of the ore samples increased and finally tended to be stable with the increase of the -0.075 mm particle content. It indicated that the -0.075mm particle content had a big impact on the LL of the rare earth ores. The reason was that the -0.075mm particles contained more clay minerals compared with other particles size (Zhang, 2018). The -0.075 mm particles ores had high plasticity and consistency, which resulted in greater cohesion among particles. It needed to absorb more leaching agent solution in the leaching process. In addition, the results in Fig. 10 also confirmed the idea that the liquid limit (LL) was inversely proportional to the permeability coefficient.

3.4. Relation between particles size and rare earth content

It could be seen from the above analysis that particle gradation, particle migration and liquid limit were correlated with the particle size which had an effect on the permeability coefficient of the rare earth ores in the seepage diffusion phase in the leaching process. At the same time, the particle size also had an
influenced on the ion exchange process (Qiu et al., 2004). Therefore, it was essential to investigate the effect of particle size on the content of the rare earth. Screened ores particle size of -0.075 mm, 0.075-0.15 mm, 0.15-0.45 mm and +0.45 mm in the CWL of the rare earth ores 2# were taken as 100g, 100g, 100g and 100g, respectively. Four types of new ore samples were used to carry out the column leaching experiments with a constant liquid/solid ration in the leaching process. The concentration of rare earth was determined by adopting EDTA titration. The leaching behaviors of the rare earth were shown in Fig. 11.

It could be clearly observed in Fig. 11 that the concentration of rare earth increased fast to a maximum value and then decreased rapidly and then continue to decrease slowly to a steady trend in the leaching process. The peak value of rare earth concentration appeared earlier with the ore particle size increasing. This was primarily due to the +0.45mm particles with high porosity, low particle migration and small Atterberg limit, which contributed to the ion exchange reaction and seepage diffusion movement. So the +0.45mm particle samples showed high permeability. On the contrary, the -0.075mm particles with low porosity, high particle migration and large Atterberg limit, which would slow down the reaction rate leading to and permeability caused a low leaching rate of rare earth. In addition, Fig. 12 showed that the content of rare earth elements decreased gradually with the ore particle size increasing in the leaching process. This behavior was mainly because the specific surface area of mineral particles increased with the particle size decreasing (Shu et al., 2019). -0.075mm particles with large specific surface area having strong absorption capacity adsorbed a lot of rare earth ions. It also confirmed that fine particles and rare earth ions migrated gradually with the groundwater movement (Zhang, 2016), which caused a higher content of -0.075mm particles having high content of RE$^{3+}$ in the CWL than that in the HL.

![Fig. 11. Leaching behaviors of RE$^{3+}$ (A, B, C and D were the peak point of rare earth concentration)](image)

![Fig. 12. Rare earth content in the rare earth ores with different particle size (A: +0.45mm; B: 0.15-0.45mm; C: 0.075-0.15mm; D: -0.075mm)](image)
4. Conclusions

In this study, the effects of particle gradation, particle migration and liquid limit on the permeability coefficient were studied, and the relation between the particle size and rare earth content was discussed. The results showed that the ores in the HL with uniformly graded particles and low non-uniformity coefficient had high permeability while that in the CWL with dense-graded particles and high non-uniformity coefficient showed low permeability. The ore in the PWL was open-graded particles, whose non-uniformity coefficient and permeability fell in between the HL and the PWL. -0.075mm particle migration was the largest, whereas +0.45 mm particles migrated little downward with the leaching agent solution. The permeability coefficient decreased gradually as the -0.075mm particle content was less than 30%. But when the -0.075mm particle content was greater than 30%, the change of the permeability coefficient was not obvious. The permeability coefficient could be well predicted by the obtained model. There was an inverse correlation between the Atterberg limit and the permeability coefficient in the leaching process of the weathered crust elution-deposited rare earth ores. The maximum value of rare earth concentration appeared earlier with the ore particle size increasing in the leaching process, but the content of rare-earth elements decreased gradually because of the groundwater movement.

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