



Effect of phosphogypsum on saline-alkalinity and aggregate stability of bauxite residue

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Abstract: A column experiment was conducted to investigate the effect of phosphogypsum (PG) on the saline-alkalinity, and aggregate stability of bauxite residue. Results showed that: with increasing leaching time, the concentrations of saline-alkali ions decreased while the SO_4^{2-} concentration increased in bauxite residue leachate; compared with CK (control group) treatment, pH, electric conductivity (EC), exchangeable sodium percentage (ESP), sodium absorption ratio (SAR), and exchangeable Na^+ content of bauxite residue were reduced following PG treatment; average particle sizes in aggregates following CK and PG treatments were determined to be 155 and 193 nm, respectively. SR- μ CT test results also confirmed that bauxite residue following PG treatment acquired larger aggregates and larger pore diameter. These results indicate that the PG treatment could significantly modulate the saline-alkalinity, and simultaneously enhance aggregate stability of bauxite residue, which provides a facile approach to reclaim bauxite residue disposal areas.

Key words: bauxite residue; phosphogypsum; saline-alkalinity; aggregate stability

1 Introduction

Bauxite residue, acting as a high alkaline waste, is generated during the production process from bauxite ore to alumina [1,2]. The land-based disposal areas, used to store large scale bauxite residue, usually produce harmful effects on the surrounding environment, including water and soil contamination [3,4]. High salinity and alkalinity of bauxite residue result in the difficulties of pedogenesis [5,6], further suppressing plants growth in bauxite residue disposal areas. Therefore, it is vital to adopt effective measures to regulate the saline-alkali properties of bauxite residue. Generally, pH, electrical conductivity (EC), sodium absorption ratio (SAR), and exchangeable sodium percentage (ESP) are the main parameters to

evaluate the saline-alkali characteristics of bauxite residue [7]. These parameters are high in bauxite residue, i.e., pH: 9.2–12.8, EC: as high as ~ 28.4 mS/cm, ESP: 42%–80%, and SAR > 30%, which are similar with the characteristics of saline-alkali soil [8–10]. Amendment application has been widely applied in saline-alkali soil to improve the physico-chemical properties and enhance fertilizer properties [11,12], which may be considered as a promising way to ecological remediation of the bauxite residue disposal areas, based on the similar characteristics between bauxite residue and saline-alkali soil.

Also, amendment application has a series of advantages, such as low cost, simple operation, easy transportation, and remarkable remediation effect in reducing saline-alkali characteristics of soil. Usually, gypsum and phosphogypsum were

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considered to be one of the promising candidates in the remediation of saline–alkali soil [7,12]. Gypsum addition into saline–alkali soil can elevate soil permeability by increasing ion concentration. With the increase of gypsum proportion, the significant decrease of EC and SAR in soil could be achieved [13]. ZHAO et al [12] investigated the effect of gypsum addition on the saline–alkali soil of Songnen plain, and found that the values of pH, EC, ESP, and SAR in saline–alkali soil are reduced by 14.6%, 38.6%, 61.2%, and 84.8% after two years, respectively. In addition, the aggregate stability also plays an important role in the pedogenesis process and saline–alkali modulation of bauxite residue [14,15]. Calcium ions of gypsum or phosphogypsum could displace sodium ions, which increases aggregate stability of saline–alkali soil, further improving the physical structure of soil [11,12]. However, so far, the bauxite residue studies in amendment application have been focused on only saline-alkalinity performance or single aggregate stability. Few reports on systematical investigation into amendment effect on the saline–alkali properties and aggregate stability of bauxite residue have been found [14].

The main goals in this research were to investigate variation of the saline–alkaline characteristics in bauxite residue following addition of amendments, reveal the effect of amendments on the evolution of aggregate structure, and inspect whether phosphogypsum can enhance remediation processes of bauxite residue.

2 Experimental

2.1 Sample characteristics

The tested bauxite residue was collected in a bauxite residue disposal area (surface layer 0–20 cm), which was located at Guangxi, China (24.075°N, 108.356°E). In this work, bauxite residue means a recently deposited sample which has been stacked about 60 d from the production to sample collection. The physical and chemical characteristics were recorded as follows: pH, EC, ESP, exchangeable Na content, exchangeable Ca content, exchangeable Mg content, and exchangeable K content showing 10.43, 197.30 $\mu\text{S}/\text{cm}$, 58.89%, 2.07×10^3 mg/kg, 1.26×10^3 mg/kg, 1.81 mg/kg, and 180.03 mg/kg, respectively. Phosphogypsum material was collected from a phosphate–fertilizer

workshop at Yunnan Province, China, and its characteristics are as follows: the values of pH, EC, ESP, exchangeable Na content, exchangeable Ca content, exchangeable Mg content, and exchangeable K content are 2.75, 165.20 $\mu\text{S}/\text{cm}$, 5.89%, 122.09 mg/kg, 1.94×10^3 mg/kg, 41.29 mg/kg, and 160.48 mg/kg, respectively. Bauxite residue and phosphogypsum were air-dried for 7 d, and passed through a 2 mm sampling sieve prior to the incubation experiment.

2.2 Experiment design

The incubation experiment was carried out in a soil column (internal diameter of 12 cm, height of 100 cm) with several holes at the side wall, which was described in our previous work in detail [16]. Two treatments, including CK (control group, untreated bauxite residue) and PG (mixing with 2% phosphogypsum and bauxite residue), were set to fill the column at 0–25 cm (surface layer). From top to bottom, bauxite residue was uniformly filled to the column at 25–45 cm (middle layer) and 45–65 cm (sub-layer) for comparative study, followed by natural compaction. The bottom (>65 cm) of the column was padded with gauze to prevent the leakage of residue particles (Fig. 1). All of the treatments were in triplicate. In order to humidify bauxite residue samples, deionized water was continuously supplied from the bottom of the soil column until achieving the maximum water holding capacity of bauxite residue, staying for 48 h. Then, 175 mL water was calculated through the local average rainfall, leaching the residues from the top layer once every 2 d. A 500 mL conical bottle was placed at the bottom of the column to obtain the leachate every 3 d. The leaching process lasted for 30 d. At the end of the leaching process, residue samples of 0–25 cm, 25–45 cm,

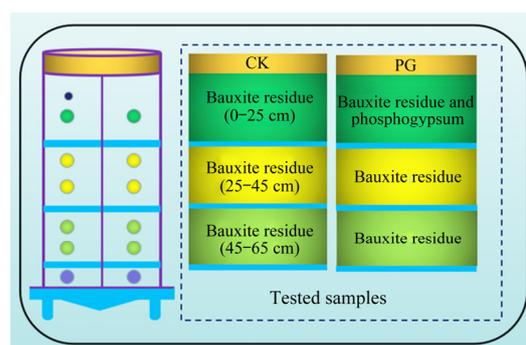


Fig. 1 Schematic diagram of experimental design

and 45–65 cm layers were collected, then the oven-dried process was carried out at 65 °C and the screened operation was conducted using a 2 mm sieve prior to physical and chemical analysis.

2.3 Characterization methods

2.3.1 Tested parameters

The tested parameters in leachates of bauxite residue included pH, EC, and concentrations of water-soluble ions (Na^+ , Ca^{2+} , Mg^{2+} , K^+ , HCO_3^- , CO_3^{2-} , SO_4^{2-} , Cl^-). Meanwhile, pH, EC, cation exchange capacities (CEC), ESP, SAR, exchangeable cation contents, and aggregate stability of bauxite residue were analyzed for all samples.

2.3.2 Physico-chemical analysis

The values of pH and EC for all samples (solid/water mass ratio of 1:5) were determined by pH indicator (PHS-3C-pH, INESA, Shanghai, China) and conductivity meter (DDS-307, INESA, Shanghai, China), respectively [17]. Exchangeable cation contents were extracted with 1 mol/L ammonium acetate (pH=7), and were measured using inductively coupled plasma atomic emission spectrophotometry (ICP-AES). CEC value was determined by sodium acetate-flame photometry method [18]. Plasma emission spectrometer (ICPE-9800, Shimadzu, Japan) was used to examine the water-soluble cation concentrations (Na^+ , Ca^{2+} , Mg^{2+} , K^+). Anion concentrations of HCO_3^- and CO_3^{2-} were measured by means of titration of sulfuric acid [19]. The concentration of Cl^- was titrated by using AgNO_3 solution, and SO_4^{2-} concentration was measured by BaCl_2 addition treatment and titrated by means of ethylenediaminetetraacetate (EDTA) solution [19].

2.3.3 Micromorphology analysis of residue aggregates

The surface morphology and size of aggregate samples were obtained by a scanning electron microscope (SEM, FET Quanta-200). The powder samples were dispersed on the surface of copper sheet, and then small gold particles were sprayed on the top of samples to serve as a conductive coating. The element distribution was investigated using an energy dispersive spectrometer (EDS). The aggregate structure of bauxite residue was analyzed by synchrotron-related μCT (computer tomography) technique (beam line BL13W1 from Shanghai Synchrotron Radiation Facility

Workstation (SSRF)). Aggregate samples were fixed in a plastic-based tube and mounted at a rotary stage, followed by rotating from 0° to 180° at an interval step of 0.1°, accompanying with scanning the samples with a photon energy of 19.5 keV. In total 1000 slices with 1200×1200 (pixels) in size were acquired. Image segmentation with an ImageJ 1.48 software was used to remove the obvious ring artifacts [20]. To refrain from edge effect, a volume with $300 \times 300 \times 300$ (voxels) was extracted from the obtained 3D aggregates [21].

2.4 Data analysis

All data were expressed as their mean \pm standard deviation and were analyzed in Excel 2010 software. Figures were depicted by Origin 8.5 software.

3 Results and discussion

3.1 Saline-alkali characteristics of bauxite residue leachates

3.1.1 pH and EC

Figure 2(a) shows variation of pH value of bauxite residue leachates with different treatments. With the increase of leaching time, pH value of bauxite residue leachate decreased, which may be attributed to the changes of alkaline anions in bauxite residue leachates [8]. Compared with CK treatment, pH of bauxite residue leachate was reduced after PG application, because phosphogypsum addition resulted in pH reduction of bauxite residue leaching due to releasing Ca^{2+} into solution, further causing precipitation of excessive anions (i.e., OH^- , CO_3^{2-} , and HCO_3^-) to generate $\text{Ca}(\text{OH})_2$, tri-calcium aluminate, hydrocalcumite, and CaCO_3 [8,22]. As shown in Fig. 2(b), with increasing the leaching time, the decrease of EC of bauxite residue leachates occurred. EC value of bauxite residue leachate following PG treatment is higher than that of CK treatment, which indicates that phosphogypsum is beneficial to the enhancement salt elution rate [23].

3.1.2 Anions concentrations

As shown in Figs. 3(a, b), the concentrations of main anions (CO_3^{2-} and HCO_3^-) in leachates were inspected, which agreed with the previous results reported by JONES et al [23]. Compared with the CK treatment, after PG treatment, the

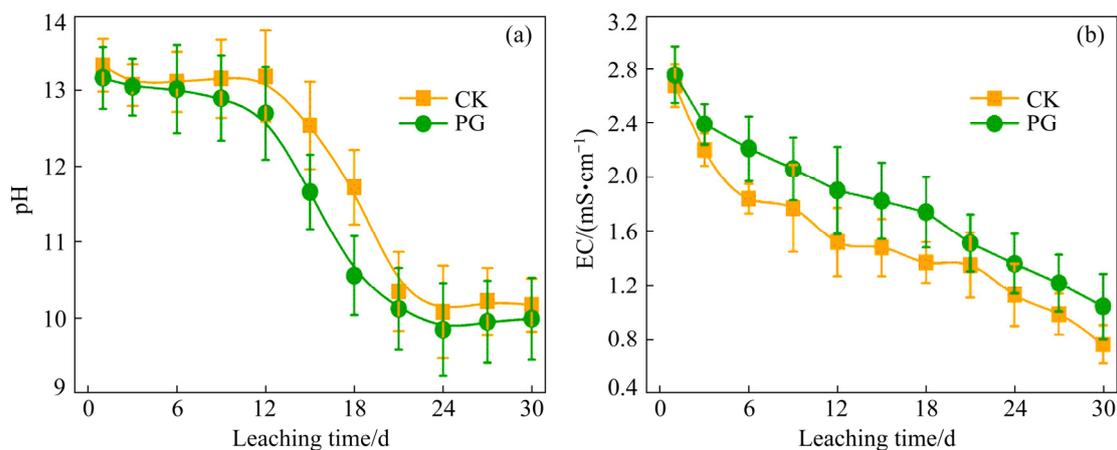


Fig. 2 Effects of different treatments on pH (a) and EC (b) of bauxite residue leachates (Bars were mean±standard deviation (SD, $n=3$))

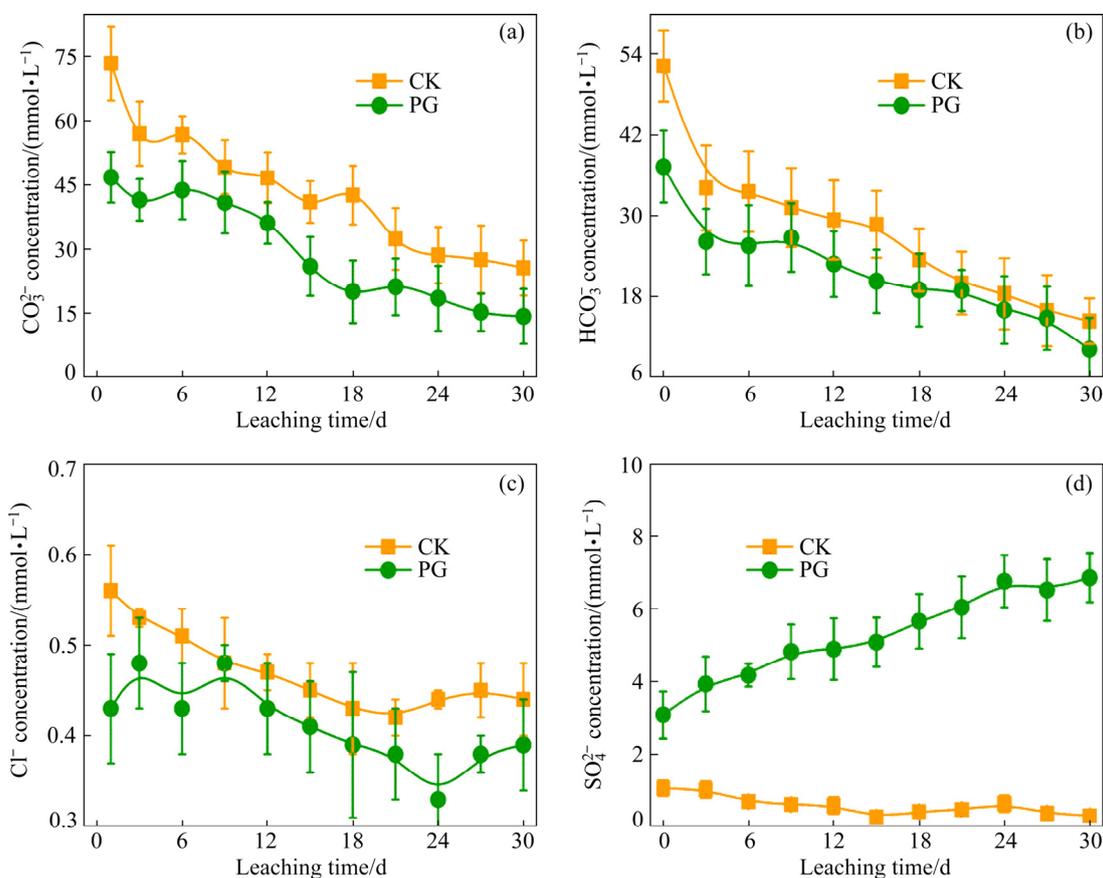


Fig. 3 Effects of different treatments on CO_3^{2-} (a), HCO_3^- (b), Cl^- (c), and SO_4^{2-} (d) concentrations of bauxite residue leachates

concentrations of CO_3^{2-} and HCO_3^- in bauxite residue leachate decrease, which can be ascribed to that precipitation occurs when Ca^{2+} from phosphogypsum suffers from CO_3^{2-} and HCO_3^- in bauxite residue leachates [10]. It can be observed that Cl^- concentration of bauxite residue leachate after PG treatment is lower than that after CK treatment though Cl^- concentration is low

(Fig. 3(c)). Incorporating amendment into bauxite residue can lead to the reduction of Cl^- concentration, which is in agreement with the result reported by LI et al [24]. Figure 3(d) shows that PG treatment elevates the SO_4^{2-} concentration of bauxite residue leachates. The main difference is that SO_4^{2-} is introduced due to the dissolution of phosphogypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$).

3.1.3 Cation concentrations

With increasing the leaching time, Na^+ concentration of bauxite residue leachates was reduced (Fig. 4(a)). Also, Na^+ concentration following PG treatment increased as compared to CK treatment. As shown in Fig. 4(b), with the increase of leaching time, Ca^{2+} concentration of bauxite residue leachates decreased. Moreover, compared with CK treatment, Ca^{2+} concentration of bauxite residue leachates after PG treatment decreased. This is because Ca^{2+} releases from the phosphogypsum addition, which can preferentially exchange Na^+ [25], further releasing into bauxite residue leachates. In addition, phosphogypsum provides Ca^{2+} source which is easy to form CaCO_3 precipitate with basic anion (i.e., CO_3^{2-}) in bauxite residue [8], further resulting in the reduction of Ca^{2+} concentration of bauxite residue leachates. In this study, the variation of Ca^{2+} concentration is in consistent with CO_3^{2-} concentration of bauxite residue leachates (see Fig. 3(a)). As shown in Figs. 4(c, d), with increasing the leaching time, the concentrations of both Mg^{2+} and K^+ are reduced although the Mg^{2+} concentration of bauxite residue

leachates is lower. Similar cases occurred in previous research [23]. The above results indicated that amendment application has a positive effect on the mitigation of salinization of bauxite residue.

3.2 Saline-alkali characteristics of bauxite residue

3.2.1 pH and EC

The pH values for all treatments are presented in Fig. 5(a). Compared with CK treatment, pH value of the amended residues decreased at depths of 0–25, 25–45, and 45–65 cm. In the 0–25 cm layer, pH value following PG treatment decreased by 12.62%. Residue pH was mainly controlled by HCO_3^- , CO_3^{2-} , and exchangeable Na^+ [24]. Phosphogypsum addition provided excessive Ca^{2+} which could replace exchangeable Na^+ in bauxite residue and react with HCO_3^- and CO_3^{2-} , further resulting in a decrease of pH [26]. Moreover, this result agreed with the previous research work reported by ZHAO et al [12], in which the pH value of saline-sodic soils was reduced by means of gypsum application. At 25–45 and 45–65 cm layers, compared with CK treatment, pH value following

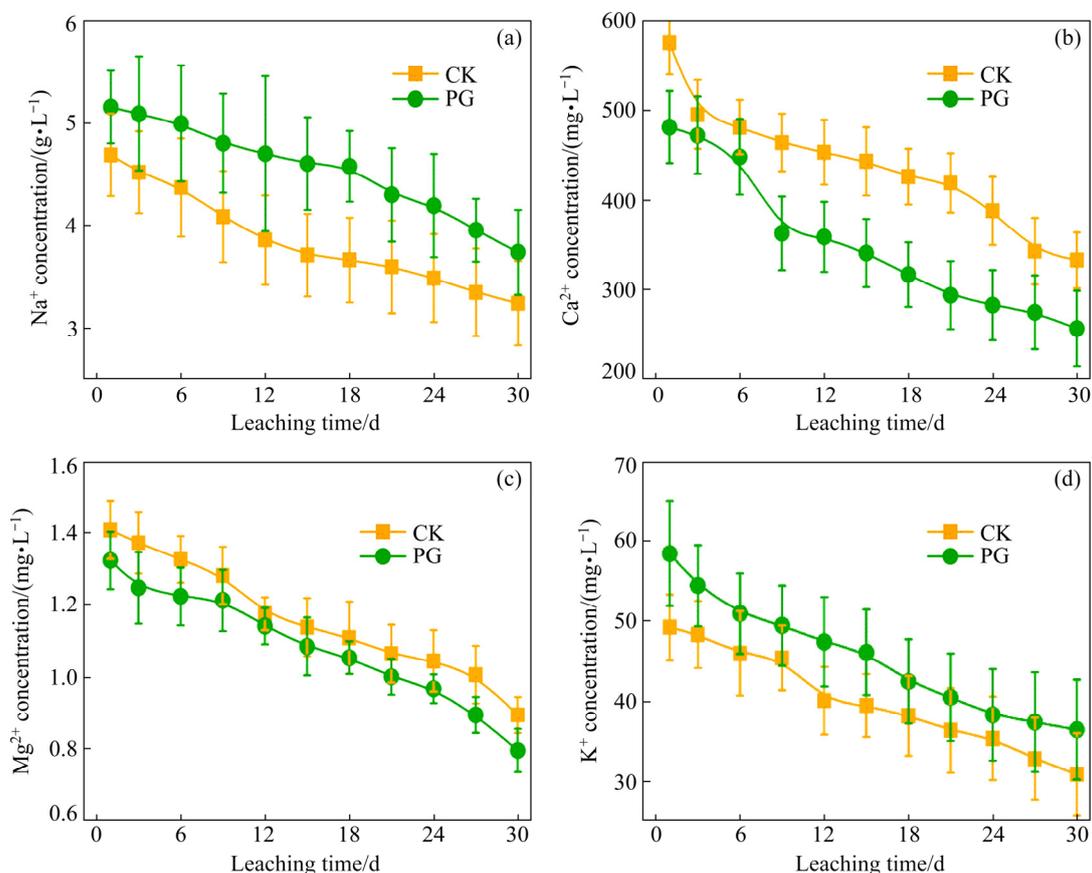


Fig. 4 Effects of different treatments on Na^+ (a), Ca^{2+} (b), Mg^{2+} (c), and K^+ (d) concentrations of bauxite residue leachates

PG treatment exhibited reduction.

Figure 5(b) shows the effect of amendments on the EC value of bauxite residue. EC value of bauxite residue following PG treatment obviously decreased as compared to CK treatment, at column depths of 0–25, 25–45, and 45–65 cm. In the 0–25 cm layer, compared with CK treatment, EC value decreased by 72.22% following PG treatment. This is probably because phosphogypsum caused reduction in Na content of bauxite residue, which is

positively correlated with EC value [27]. However, amendment addition changed EC values of bauxite residue at 25–45 and 45–65 cm layers, which suggested that the amendment effect was mainly accumulated at 0–25 cm layer of bauxite residue.

3.2.2 Exchangeable cation contents

Amendment addition changed the contents of exchangeable cations (Na^+ , Ca^{2+} , Mg^{2+} , and K^+) in bauxite residue at column depths of 0–25, 25–45, and 45–65 cm (Fig. 6). As presented in Fig. 6(a),

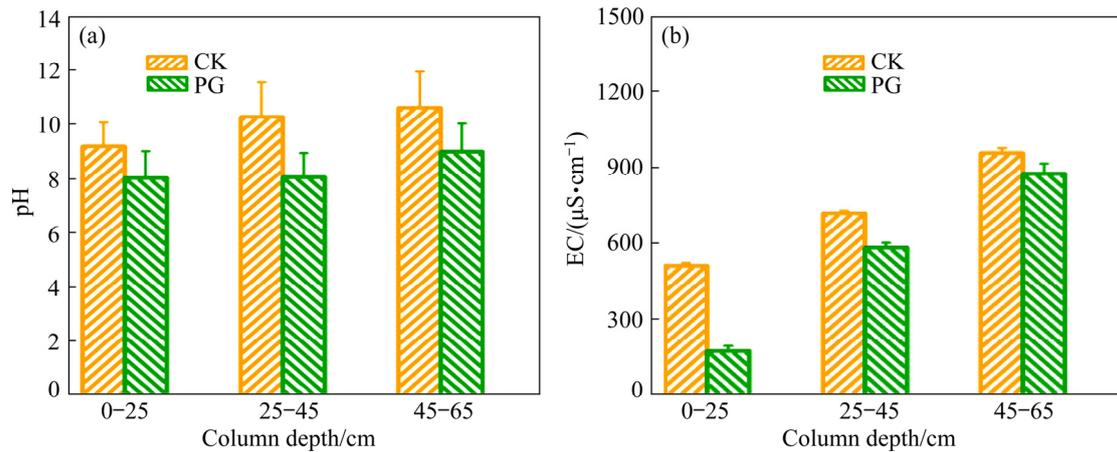


Fig. 5 Variation of pH (a) and EC (b) of bauxite residue at column depths of 0–25, 25–45, and 45–65 cm

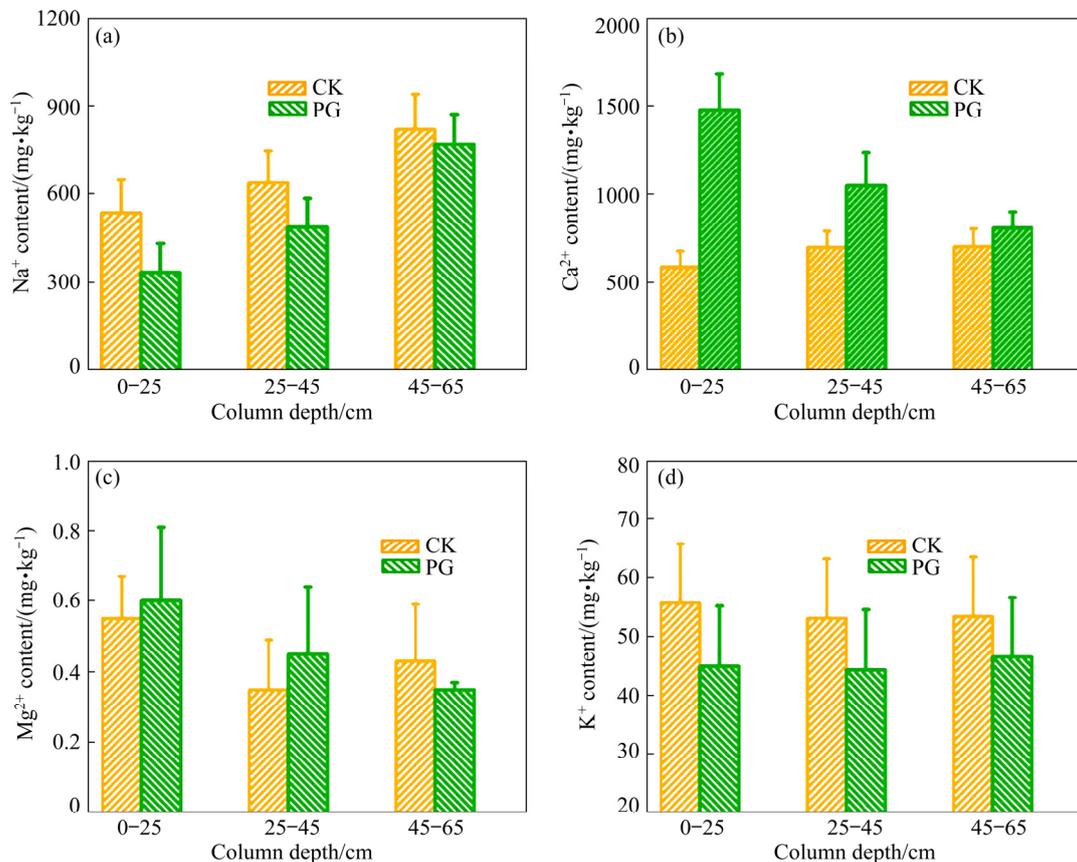


Fig. 6 Effects of different treatments on exchangeable Na^+ (a), Ca^{2+} (b), Mg^{2+} (c), and K^+ (d) contents of bauxite residue at column depths of 0–25, 25–45, and 45–65 cm

for the same treatment, the exchangeable Na^+ content increased with increasing column depth. As compared with CK treatment, exchangeable Na^+ content following PG treatment decreased by 37.39% at the 0–25 cm layer, 23.51% at the 25–45 cm layer, and 6.24% at the 45–65 cm layer. As shown in Fig. 6(b), compared with CK treatment, PG treatment increased the content of exchangeable Ca^{2+} in bauxite residue. This phenomenon can be ascribed to the release of Ca^{2+} which has displacement with Na^+ from the exchange complex system [26,28]. This result was also demonstrated by the EDS result in Fig. 7. In comparison with CK treatment, Na element distribution was sparse and dispersed, while Ca element exhibited a dense distribution following PG

treatment. According to the EDS analysis, the amendment addition could increase content of exchangeable Ca^{2+} and reduce exchangeable Na^+ content of bauxite residue. In comparison with CK treatment, at column depths of 0–25 and 25–45 cm, exchangeable Mg^{2+} content following PG treatment increased, but the similar trend was not obvious at 45–65 cm layer, as shown in Fig. 6(c). PG treatment decreased the content of exchangeable K^+ in bauxite residue as compared to CK treatment, as shown in Fig. 6(d).

3.2.3 Alkaline characteristics

The variation of ESP of bauxite residue with different treatments is shown in Fig. 8(a). For the 0–25 cm layer, in comparison with CK treatment, the value of ESP with PG treatment decreased, and

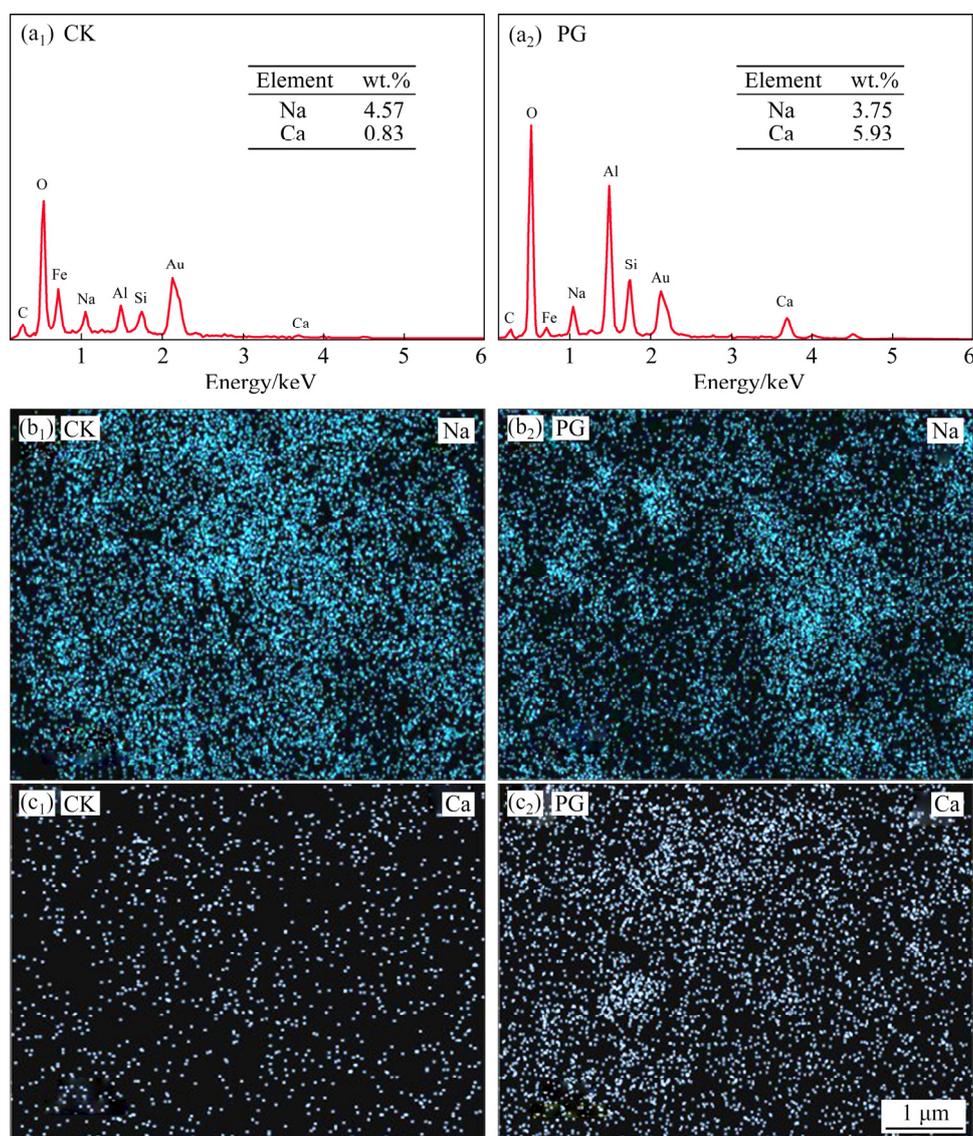


Fig. 7 EDS results showing effects of different treatments on contents and distributions of Na and Ca elements in bauxite residue

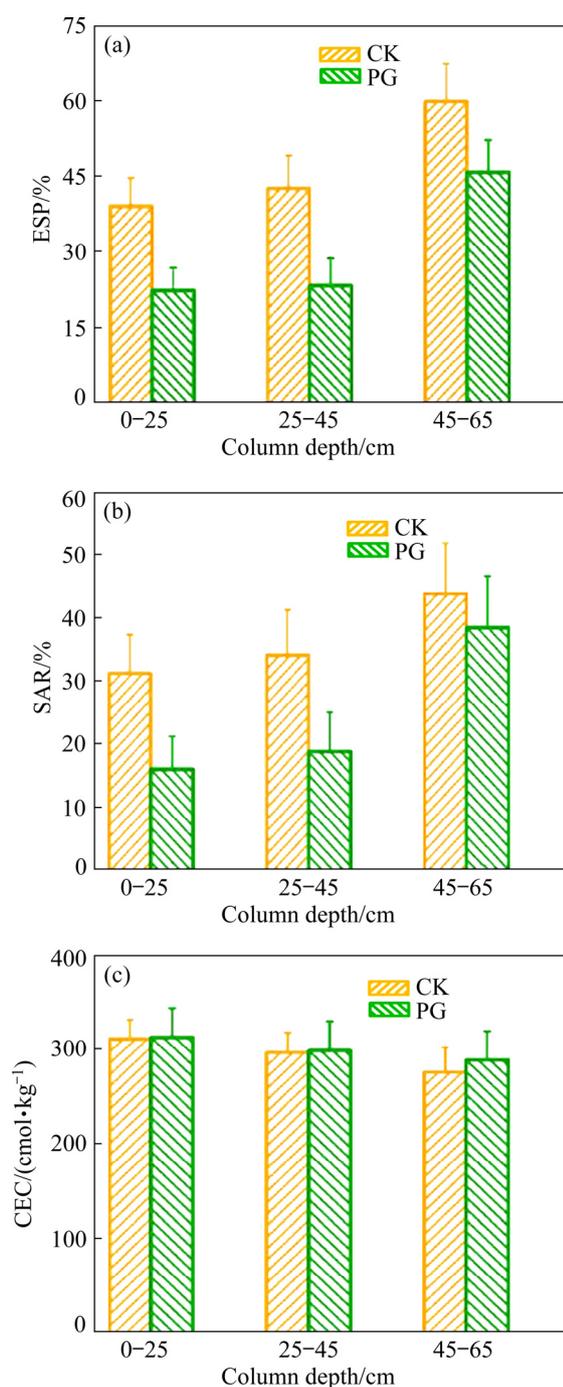


Fig. 8 Changes in ESP (a), SAR (b), and CEC (c) of bauxite residue at column depths of 0–25, 25–45 and 45–65 cm

similar results were observed at 25–45 and 45–65 cm layers. This may be because the increase of ESP value of bauxite residue can reflect the high exchangeable Na⁺ content [29].

SAR is one of the key parameters to inspect soil salinization [7,12]. The higher the SAR value is, the more toxic the soil will be [7]. The effect of amendment application on the SAR of bauxite

residue is presented in Fig. 8(b). As compared to CK treatment, SAR decreased by 48.70%, 44.83%, and 12.15% after PG treatment at 0–25, 25–45, and 45–65 cm layers, respectively. Similar result was reported by CHAGANTI et al [7]. In their research, SAR value exhibited obvious reduction after leaching saline–sodic soils with the application of gypsum. In similarity with ESP, SAR reduction of bauxite residue may be induced by the decrease of Na⁺ content of bauxite residue, due to the exchange reaction between Na⁺ and Ca²⁺ generated from phosphogypsum [29]. The amendment effect on the CEC of bauxite residue is depicted in Fig. 8(c). For the 0–25 cm layer, in comparison with CK treatment, PG treatment enhanced CEC value of bauxite residue. However, amendment addition has insignificant influence on the CEC value of bauxite residue layers at column depths of 25–45 and 45–65 cm.

3.3 Micromorphology and structure of bauxite residue

The micro-aggregate characteristics of bauxite residue with different treatments were analyzed by scanning electron microscopy (SEM) observation, as displayed in Fig. 9. Most fine particles existed following CK treatment (Fig. 9(a₁)). Following PG treatment, aggregate size increased in Fig. 9(a₂), which indicated that PG treatment could effectively increase aggregate size, which suggested the improvement of aggregate structure and physical conditions of bauxite residue. The particle sizes from Figs. 9(b₁, b₂) were obtained in terms of the Gauss distribution fitting [30], as shown in Figs. 9(c₁, c₂). From the histogram, the average particle sizes of samples after CK and PG treatments were found to be 155 and 193 nm, respectively. To intuitively analyze the aggregate structure of bauxite residue following amendment application, the synchrotron irradiation technique was used to characterize the two-dimensional (2D) and three-dimensional (3D) structures of microaggregates, as shown in Fig. 10. Compared with CK treatment, bauxite residue following PG treatment had larger aggregate and pores, which is beneficial to plants growth in bauxite residue disposal areas [17]. ZHU et al [31] confirmed that larger aggregate size could improve physical structure and enhance aggregate stability of bauxite residue.

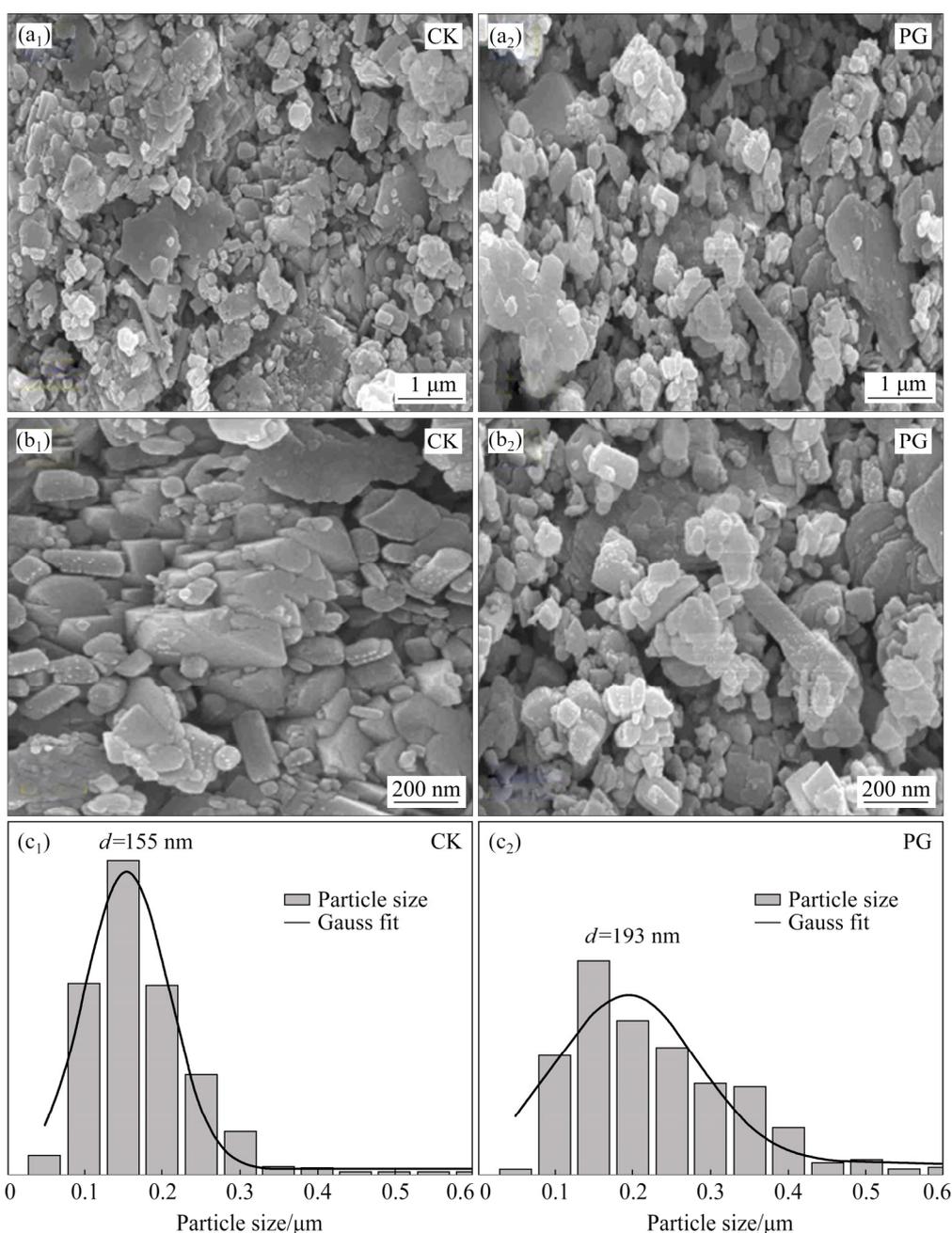


Fig. 9 Effects of different amendments on microstructures (a₁, b₁, a₂, b₂) and aggregate sizes (c₁, c₂) of bauxite residue

3.4 Potential mechanism for remediation of disposal areas

The above results and discussion suggested that phosphogypsum addition decreased the values of pH, EC, ESP, SAR, and exchangeable Na⁺ content of bauxite residue. By comparing the salinity, alkalinity, and aggregate stability of bauxite residue with different treatments, PG treatment exhibited superiority in reducing saline-alkali properties and enhancing aggregate stability. Figure 11 shows the effect of amendment (PG) on

the saline-alkali properties of bauxite residue and the evolution of bauxite residue aggregate. On one hand, phosphogypsum acts as an industrial waste, and its main component is CaSO₄·2H₂O which contains lots of Ca²⁺. Therefore, partial precipitates (e.g., CaCO₃) appear in bauxite residue when Ca²⁺ of phosphogypsum suffers from soluble alkalinity (i.e., HCO₃⁻ or CO₃²⁻) of bauxite residue [24], which induced pH reduction of bauxite residue. On the other hand, phosphogypsum can release large amount of Ca²⁺, which would displace the weak

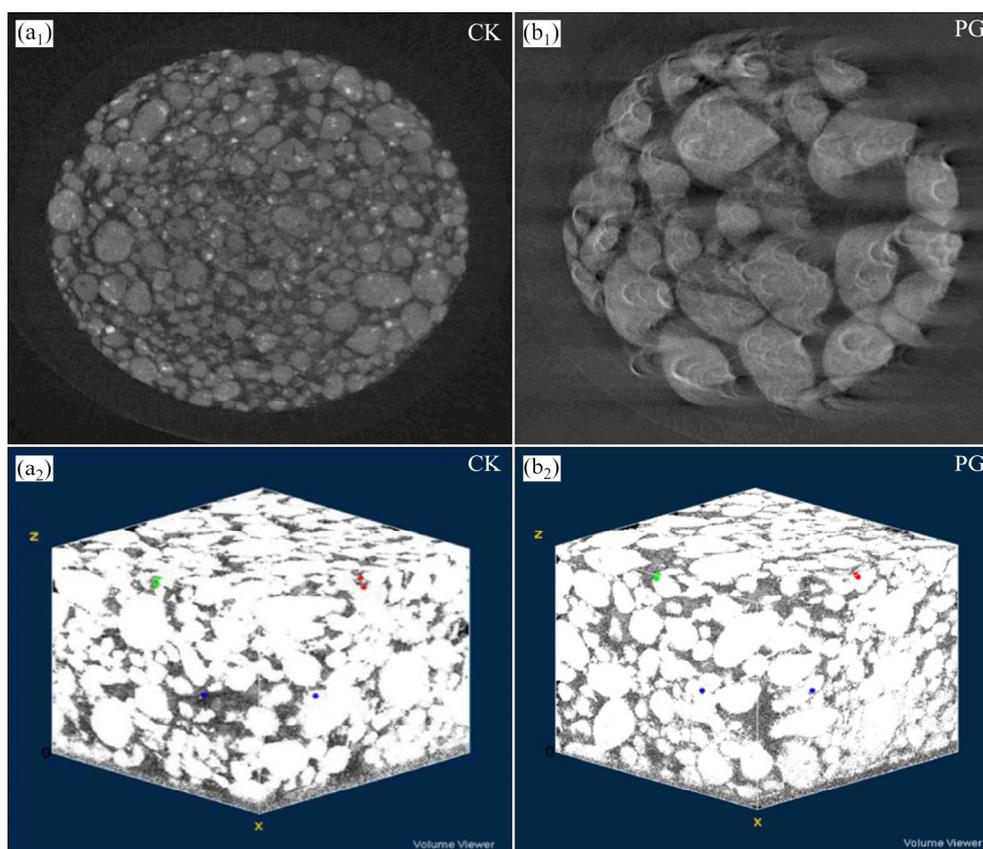


Fig. 10 Representative 2D slices (a_1 , b_1) and 3D pore structures (a_2 , b_2) of bauxite residue aggregates after different treatments

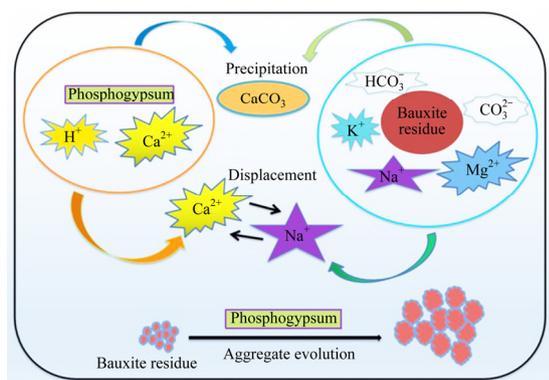


Fig. 11 Schematic diagram showing remediation mechanism of bauxite residue following phosphogypsum application

monovalent Na^+ of bauxite residue from cation exchange sites [32,33]. Meanwhile, Na^+ content of bauxite residue was positively correlated with EC [10,27] and SAR values [7]. Therefore, the decrease of Na^+ content resulted in reduction of saline-alkalinity of bauxite residue. In addition, the bauxite residue aggregates following PG treatment acquired larger particle size and pore size from

SEM and micro-CT, which enhanced aggregate stability of bauxite residue [20]. Our results demonstrated that PG treatment is a promising, controllable, and environment-friendly method for reducing the saline-alkali properties and enhancing aggregate stability of bauxite residue, which will be beneficial to vegetation reconstruction applications in bauxite residue disposal areas.

4 Conclusions

(1) With increasing leaching time, the parameters of saline-alkalinity in bauxite residue leachate were reduced except for SO_4^{2-} concentration.

(2) Compared with CK treatment, pH value and the concentrations of CO_3^{2-} , HCO_3^- , Cl^- , and Ca^{2+} decreased, while EC value and the concentrations of SO_4^{2-} and Na^+ of bauxite residue leachates increased after amendment applications.

(3) In comparison with CK treatment, the values of pH, EC, ESP, SAR, and exchangeable Na^+ content of bauxite residue were reduced, whilst

aggregate size and pore structure of bauxite residue were improved following PG treatment.

(4) This study provided a low-cost effective strategy to control potential ecological risk in the disposal areas by amendment application, and demonstrated that PG treatment is conducive to pedogenesis process of bauxite residue.

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磷石膏对赤泥盐碱性及团聚体稳定性的影响

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摘要: 通过土柱实验研究磷石膏改良剂对赤泥盐碱性和团聚体稳定性的影响。结果显示: 随着淋溶时间的延长, 赤泥渗滤液中盐碱性离子浓度降低, 而 SO_4^{2-} 浓度增加; 与对照组相比, 添加磷石膏可显著降低赤泥中 pH、电导率、可交换钠百分比、钠吸收比和交换态 Na^+ 含量; 对照组和磷石膏改良赤泥团聚体的平均颗粒尺寸分别为 155 和 193 nm。同时, 同步辐射微 CT 扫描结果也证明经磷石膏改良的赤泥获得更大的团聚体尺寸和孔径。以上结果表明, 磷石膏改良可以显著调控赤泥的盐碱性, 同时改善赤泥团聚体的稳定性, 为赤泥堆场的修复提供一种经济有效的途径。

关键词: 赤泥; 磷石膏; 盐碱性; 团聚体稳定性

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