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# Comparative study on ionic dissolution and structural changes of montmorillonite, kaolinite, and muscovite during interfacial reactions with oxalic acid solution

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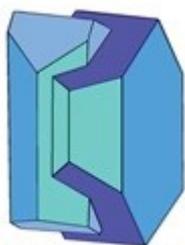
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Abstract: Organic acids are commonly found in soils and sediments, playing an important role in the alteration and weathering of minerals and influencing a series of geochemical processes such as soil fertility, metal cycling, and pollutant migration. In order to better comprehend the reaction mechanisms of different layered silicate minerals with organic acids, three minerals with various structure types, namely montmorillonite, kaolinite, and muscovite, were investigated in this work. In particular, the effects of interfacial reactions with oxalic acid on the crystal structure, chemical composition, morphology, and specific surface area of minerals were compared. The composition and structure of montmorillonite, kaolinite, and muscovite during the interfacial reaction with oxalic acid were characterized via powder X-ray diffraction (XRD), scanning electron microscopy (SEM), and inductively coupled plasma-optical emission spectroscopy (ICP-OES) methods. It was shown that  $\text{Si}^{4+}$  and  $\text{Al}^{3+}$  were gradually dissolved during the interfacial reactions and that the changes in

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the properties of minerals depended on the structural characteristics. After 300 days of the interfacial reactions with oxalic acid, the dissolution percentages of  $\text{Si}^{4+}$  and  $\text{Al}^{3+}$  in montmorillonite, kaolinite, and muscovite were 12.67%, 8.4%, 3.8% and 62.1%, 30.7%, 6.1%, respectively. Moreover, the lamellar morphology of montmorillonite was destroyed upon the interfacial reaction with oxalic acid, and irregular particles with sizes of ~500-100 nm were formed onto the surface. The diameter of kaolinite flake particles decreased from 1500-400 nm to 400-50 nm, and the surface of rod-shaped particles was ruptured. The small particles disappeared from the muscovite surface, and the initially sharp edges became blunted. The specific surface area and the total pore volume of montmorillonite and kaolinite increased after the interfacial reaction with oxalic acid, while the opposite results were obtained for muscovite. The differential dissolution of the minerals during their interfacial reaction with oxalic acid was mainly related to the differences between cation occupancies, structural types, chemical bond strengths, and specific surface areas.

Keywords: Montmorillonite, Kaolinite, Muscovite, Oxalic acid, Ionic dissolution, Structural evolution

## Introduction

Low-molecular-mass organic acids are widespread in the rhizosphere soil, taking their origin from the decomposition of plants and animal remains, plant root secretions, and microbial synthesis (Chen et al., 2023; Yuan et al., 2023). Being involved in various rhizosphere processes, these organic acids influence soil nutrient release and metal ion complexation/chelation (Liu et al., 2017; Onireti et al., 2017; Xue et al., 2018). Organic acids play an important role in the geochemical processes involved in the weathering and evolution of minerals (Huang and Kiang, 1972; Schliemann and Churakov, 2021a). Both free protons and anionic ligands produced by the

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hydrolysis of organic acids can promote mineral weathering (Cama and Ganor, 2006): while protons leading to the decrease in the pH of the fluid surrounding the mineral and shift the balance toward dissolution (Furrer and Stumm, 1986), anionic ligands impact the saturation state of the minerals and the coordination morphology of  $\text{Al}^{3+}$  in the solution (Drever and Stillings, 1997; Kong et al., 2014).

Numerous experimental studies on the dissolution of minerals by organic acids have been reported to date. The protonation of hydroxyl groups on the surface of minerals and the formation of coordination compounds between surface cations and organic ligands weakens the M–O bond energy, thus promoting dissolution (Stumm et al., 1985). When organic acid molecules are adsorbed onto the olivine surface, the cations on the surface of olivine are more inclined to bond with oxygen in the carbonyl group (C=O) of the organic acid molecule, and the H–O bond in the carboxyl group (COOH) is broken, leading to hydroxylation of the mineral surface. In this case, the increase in the number of carboxyl groups favors the dissolution process (Sun et al., 2023). It has been pointed out that oxalic acid can increase the dissolution rate of montmorillonite, and the relevant dissolution reaction proceeds through a ligand-promoted mechanism (Ramos et al., 2011). Various experiments on the dissolution of biotite in nitric acid and oxalic acid have shown that ligands exert a smaller impact on the overall dissolution rate compared to the proton-promoted reaction (Cappelli et al., 2020). In particular, the dissolution rate of  $\text{Si}^{4+}$  in kaolinite is higher in citric acid than that in oxalic acid, whereas the dissolution rate of  $\text{Al}^{3+}$  in oxalic acid will exceed that in citric acid. Moreover, the synergistic effect of oxalic and citric acid accelerates the dissolution of kaolinite, thereby reducing crystallinity and median diameter and enlarging the specific surface area (Lin et al., 2020). Previous studies have mainly focused on the impacts of different acidic solutions on the same mineral and

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the changes in structure and properties of the products. However, the comparative analysis of similarities and differences in the interaction mechanism and the evolution of the products of the acidic solution in minerals with different structural types has rarely been carried out to date.

Herein, the composition and structural changes of montmorillonite, kaolinite, and muscovite during the interfacial reactions with the oxalic acid solution were compared based on their particular polymorphic characteristics. The  $\text{Si}^{4+}$  and  $\text{Al}^{3+}$  ions released upon the interfacial reaction between minerals with different layers and structures and acid water were determined. Besides that, the similarities and differences in dissolution rates of  $\text{SiO}_4$  tetrahedron and  $\text{AlO}_6$  octahedron were established. The mechanisms by which the interfacial reactions with acids would affect the crystal structure, chemical composition, morphology, and specific surface area of montmorillonite, kaolinite, and muscovite were revealed as well. The findings of this work provide a basis for investigating the transformations in the composition and structure of minerals under weathering conditions.

## 2. Experiment

### 2.1. Materials

Montmorillonite (Mnt) was collected in Yanting country, Sichuan province, China; muscovite (Ms) was obtained in Lingshou country, Hebei province, China; kaolinite (Kln) came from Suzhou city, Jiangsu province, China. The minerals were prewashed with deionized water under continuous stirring for 24 h after grinding, filtered and dried in an oven at  $60^\circ\text{C}$ , and ground through a 200-mesh sieve. The chemical compositions of montmorillonite, kaolinite, and muscovite are listed in Table 1, and the relevant chemical formulae are presented in Table 2.

The chemical formulas of montmorillonite, kaolinite and muscovite are calculated using the

following method. By measuring the mass fraction in the chemical composition of the mineral sample, the number of individual atoms was calculated according to the principle of balance between the total amount of negative charges and the total amount of positive charges (i.e., in tetrahedral and octahedral sheets and the interlayer domains) in the general formula of mineral structure, and then allocated to the tetrahedral, octahedral and interlayer domain locations (if any).

**Table 1.** Chemical composition of montmorillonite, kaolinite, and muscovite

sample	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	MgO	CaO	Na <sub>2</sub> O	TiO <sub>2</sub>	Others	LOI	Total
Mnt	56.53	18.31	2.48	0.60	5.26	2.74	0.14	0.18	0.21	13.55	100.00
Kln	43.31	35.55	0.90	0.86	0.18	0.12	0.06	0.19	2.08	16.75	100.00
Ms	50.45	30.04	4.37	8.61	0.55	0.21	0.67	0.73	0.23	4.14	100.00

**Table 2.** Chemical formula of montmorillonite, kaolinite, and muscovite

sample	Chemical formula
Mnt	$(Ca_{0.20}Mg_{0.06}K_{0.05}Na_{0.02})_{0.33}(H_2O)_n\{(Al_{1.38}Mg_{0.48}Fe_{0.13}Ti_{0.01})_{2.00}[(Si_{3.89}Al_{0.11})_{4.00}O_{10}(OH)_2]\}$
Kln	$(Al_{1.94}Fe_{0.03}Ti_{0.01}Mg_{0.01})_{1.99}[Si_2O_5](OH)_4$
Ms	$(K_{0.87}Na_{0.10}Ca_{0.02})_{0.99}\{(Al_{1.67}Fe_{0.25}Mg_{0.05}Ti_{0.04})_{2.01}[Al_{1.11}Si_{2.89}O_{10}](OH)_2\}$

## 2.2. Experimental (considering montmorillonite as an example)

First, 1 g of montmorillonite was put into a 250 mL conical flask, and 100 mL of 0.1 mol·L<sup>-1</sup> oxalic acid and 1 mL of 0.001 mol·L<sup>-1</sup> sodium azide (NaN<sub>3</sub>) were then added to inhibit microbial growth. The flask was shaken to ensure the full contact between montmorillonite and the organic acids. The flask was afterward placed in incubator at 25°C and stirred for 2 min every day. After a certain reaction period (30, 60, 90, 120, 150, 180, 210, 240, 270 and 300 days), the conical flask was removed from the incubator, and the supernatant and filter residue were collected. After that, 5 mL of the reactant supernatant was taken and filtered through a 0.45 µm microporous filter for further analysis. The remaining reactants in the flask were filtered to separate the solids from the

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liquids and rinsed with deionized water until the pH level of 7 was achieved. The filtrate residue was dried at 60°C in an oven for 8 h and then ground and prepared for use. The reactions of muscovite and kaolinite with oxalic acid were conducted conforming to the above steps. The samples after the reaction of montmorillonite, kaolinite and muscovite with oxalic acid solution were named Mnt-t, Kln-t and Ms-t, where t denotes reaction time (d).

### 2.3. Characterization

The phase composition of the samples was studied via X-ray diffraction (XRD, Ultima IV, Rigaku, Japan) with CuK $\alpha$  radiation ( $\lambda = 1.5406$  nm) over a  $2\theta$  angular range of 3–50° at the step size of 0.02° and the scan rate of 2 s/step in continuous scanning mode using a X'celerator detector. The full elemental chemical composition of the samples was analyzed by X-ray fluorescence (XRF) spectroscopy (ARL Perform X, Thermo Fisher, USA) by melting at 1050 °C using Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub> as the melting agent. The element detection limit was 100 mg·L<sup>-1</sup>. The dissolution concentrations of Si<sup>4+</sup> and Al<sup>3+</sup> were analyzed by inductively coupled plasma-optical emission spectroscopy (ICP-OES, iCAP6500, Thermo Fisher, USA) with power of 1150 W, pump speed of 50 rpm, auxiliary gas flow rate of 0.5 L·min<sup>-1</sup>, and nebulizer gas flow rate of 0.55 L·min<sup>-1</sup>. The detection limits of Si<sup>4+</sup> and Al<sup>3+</sup> were 0.02mg·L<sup>-1</sup> and 0.009 mg·L<sup>-1</sup>, respectively. The morphology of each composite was examined by field emission-scanning electron microscopy (SEM, Ultra 55, Zeiss, Germany) with a working voltage of 15 kV, for which the samples were spread onto aluminum sheets and coated with a thin layer of gold using a sputtering machine. The infrared absorption characteristics of the samples were investigated by Fourier transform IR spectroscopy (FTIR, Nicolet-5700, Nicolet, USA). The tablet was pressed after mixing and grinding the sample with KBr powder. Nitrogen adsorption and desorption experiments were conducted with a specific surface area and porosity tester (Autosorb

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IQ, Anton Paar Quanta, USA) under degassing conditions at 100°C for 7 h and the nitrogen temperature of -196°C. The loss on ignition of mineral samples was measured using a programmed controlled muffle furnace (KSD-6-1300, Jinguan Electromechanical Equipment, China). During the experiments, 1.000 g of sample powder pre-dried at 105°C was burned in a muffle furnace at 950°C for 2 h, and the loss on ignition of the sample was then determined by calculating the weight loss percentage.

#### 2.4. Calculation of dissolution amount and dissolution rate

The amount of dissolution  $S_{i,t}$  ( $\text{mmol}\cdot\text{g}^{-1}$ ) and the rate of dissolution  $R_{i,t}$  ( $\text{mmol}\cdot\text{g}^{-1}\cdot\text{d}^{-1}$ ) of the ion  $i$  in the mineral after reaction time  $t$  are calculated by using equations (1) and (2), respectively:

$$S_{i,t} = \frac{(C_{i,t} - C_{i,t_0}) \cdot V}{m_0 \cdot M_i} \quad (1)$$

$$R_{i,t} = \frac{S_{i,t}}{t - t_0} \quad (2)$$

where  $C_{i,t}$  and  $C_{i,t_0}$  are the dissolved concentration (measured by ICP-OES) of ion  $i$  at reaction time  $t$  and  $t_0$ , respectively,  $\text{mg}\cdot\text{L}^{-1}$ ;  $V$  is the volume of the reaction solution, L;  $m_0$  is the initial mineral mass, g;  $M_i$  is the molar mass of ion  $i$ ,  $\text{g}\cdot\text{mol}^{-1}$ ; and  $t$  is the reaction time, d.

### 3. Results and discussion

#### 3.1. Phase and structural characteristics

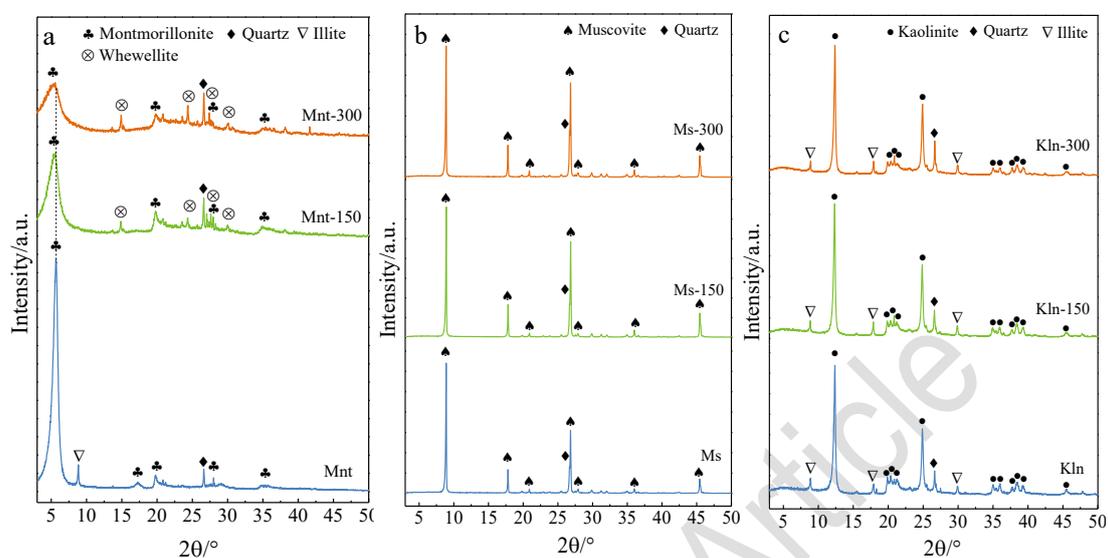
Figure 1 depicts the XRD patterns of the original montmorillonite, kaolinite, and muscovite as well as the products obtained after the interfacial reaction with oxalic acid. Figure 1a shows that the main mineral phase of the montmorillonite was calcium-based montmorillonite, with small amounts of illite and quartz. With the increase in the reaction time between the montmorillonite and the oxalic acid, the (001) peak of montmorillonite has gradually broadened and shifted toward the lower angles.

Table 3 lists the  $d$  values, the intensity ratios of the strongest diffraction peaks to the (101) peak of

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quartz, and full width at half maximum (FWHM) values of montmorillonite, kaolinite, and muscovite as well as those of the products obtained after the reaction with oxalic acid. Table 3 presents the data acquired after the reaction with oxalic acid for 150 and 300 days, the  $2\theta$  value of the FWHM of the montmorillonite (001) peaks increased from  $0.839^\circ$  to  $2.021^\circ$  and  $2.413^\circ$ , respectively, and the corresponding  $d_{001}$  value increased from  $15.5864 \text{ \AA}$  to  $16.1793 \text{ \AA}$  and  $16.6069 \text{ \AA}$ , respectively. The increase in the  $d_{001}$  value and FWHM of montmorillonite was due to the replacement of the smaller interlayer  $[\text{Ca}(\text{H}_2\text{O})_6]^{2+}$  (effective diameter=600 pm) and  $[\text{Mg}(\text{H}_2\text{O})_6]^{2+}$  (effective diameter=800 pm) ions in the interlayer domain with  $[\text{H}(\text{H}_2\text{O})_n]^+$  (effective diameter=900 pm) from the solution, which augmented the interlayer spacing in montmorillonite (Kielland, 1937; Wang et al., 2024). In addition, calcium oxalate appeared in the physical phase, which was due to the dissolution of  $\text{Ca}^{2+}$  in the interlayer and their combination with oxalic acid. Figures 1b and 1c illustrate that the main mineral phase of the kaolinite was kaolinite, containing a small amount of illite and quartz. The main mineral phase in the muscovite was muscovite, with small amounts of quartz. With the increase in interfacial reaction time with oxalic acid, the change in the  $d_{001}$  value of kaolinite,  $d_{002}$  value of muscovite and FWHMs of both minerals were minimal, indicating that their crystallinity degree remained high. According to Table 3, the ratios of the strongest peaks of montmorillonite, kaolinite, and muscovite to the (101) peaks of quartz decreased gradually, among which the  $I_{\text{Mnt}001}/I_{\text{Qz}101}$  ratio demonstrated the most pronounced decrease. This indicates that oxalic acid has affected montmorillonite, kaolinite, and muscovite to a greater extent than quartz. Moreover, the results indicated that the crystal structure of montmorillonite underwent the maximum changes, which was related to the structure of the expandable crystal layer of montmorillonite (Robin et al., 2017), where  $\text{H}^+$  were more likely to enter into the interlayer and exchange with the cations during

the reaction (Laszlo, 1987). As a result,  $H^+$  were more likely to attack hydroxyl in the structure, promoting the dissolution of montmorillonite and breakdown of its structure.



**Fig. 1.** XRD patterns of montmorillonite (a), kaolinite (b), and muscovite (c) after 150 and 300 days of the interfacial reactions with oxalic acid

**Table 3.** The d values, peak strength, and FWHM of montmorillonite, kaolinite, and muscovite after reaction with oxalic acid

Sample	Characteristics	Reaction time (d)		
		0	150	300
Mnt	$d_{Mnt001}/\text{\AA}$	15.5864	16.1793	16.6069
	$I_{Mnt001}/I_{Qz101}$	10.398	2.041	1.129
	FWHM/ $^{\circ}$	0.839	2.021	2.413
Kln	$d_{Kln001}/\text{\AA}$	7.1514	7.1683	7.1458
	$I_{Kln001}/I_{Qz101}$	5.242	4.748	3.450
	FWHM/ $^{\circ}$	0.295	0.289	0.248
Ms	$d_{Ms002}/\text{\AA}$	9.9260	9.9293	9.9229
	$I_{Ms002}/I_{Qz101}$	4.858	3.857	2.793
	FWHM/ $^{\circ}$	0.108	0.113	0.123

### 3.2. Ionic dissolution

Figure 2 depicts the variation curves of the dissolution amounts of  $Si^{4+}$  and  $Al^{3+}$  with time during the reactions of montmorillonite, kaolinite, and muscovite with oxalic acid. Figure 2

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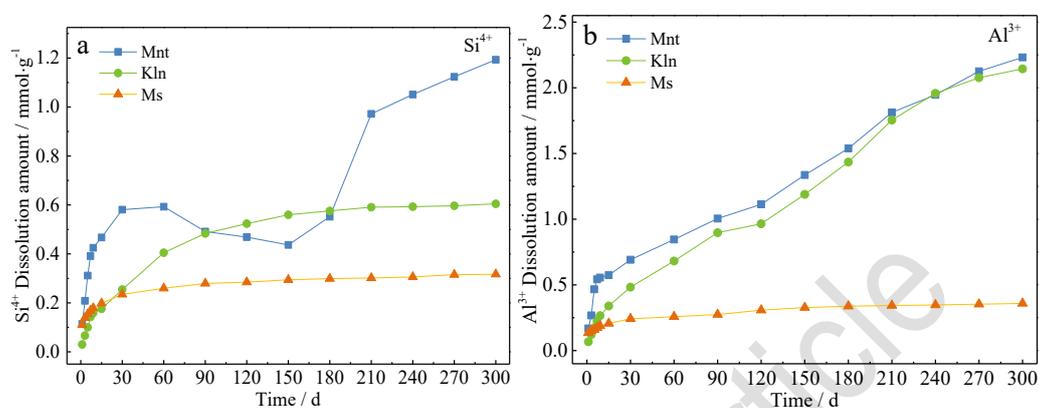
demonstrates that the reactions of the minerals with oxalic acid led to the dissolution of the cations in their structure, among which the dissolved amounts of  $\text{Si}^{4+}$  and  $\text{Al}^{3+}$  of kaolinite and muscovite increased gradually with the lengthening of the reaction time, while the dissolved amount of  $\text{Si}^{4+}$  in montmorillonite showed an alternate tendency. This was attributed to the formation of acid insoluble amorphous silica upon the reaction, which interfered with the measured  $\text{Si}^{4+}$  concentration. After the interfacial reaction with oxalic acid, montmorillonite exhibited the higher solubility of  $\text{Si}^{4+}$  and similar solubility of  $\text{Al}^{3+}$  relative to kaolinite, while muscovite had the lower solubility of  $\text{Si}^{4+}$  and  $\text{Al}^{3+}$ . Since the reaction of montmorillonite with oxalic acid induced the formation of amorphous silica, thereby affecting the dissolution process of  $\text{Si}^{4+}$ , the amount of  $\text{Si}^{4+}$  and  $\text{Al}^{3+}$  dissolved in oxalic acid in the total content of the mineral, that is, the dissolution percentage, was further calculated, and the results are shown in Table 4. After 300 days of the reaction with oxalic acid, the dissolution percentages of  $\text{Si}^{4+}$  and  $\text{Al}^{3+}$  in montmorillonite, kaolinite, and muscovite were 12.7%, 8.4%, 3.8% and 62.1%, 30.7%, 6.1%, respectively. It was evident that oxalic acid had a greater influence on the dissolution of cations in montmorillonite, followed by kaolinite and muscovite. Figure 3 shows the variation curves of the dissolution rates of  $\text{Si}^{4+}$  and  $\text{Al}^{3+}$  with time during the reactions of montmorillonite, kaolinite, and muscovite with oxalic acid. The figure illustrates that at the initial stage of the reaction, minerals possessed the fast dissolution of  $\text{Si}^{4+}$  and  $\text{Al}^{3+}$ , which was attributed to the dissolution of fine particles and the defect sites within the large grains (Kalinowski and Schweda, 1996; Huertas et al., 1999). On the first day of the reaction, montmorillonite had the largest dissolution rates of  $\text{Si}^{4+}$  and  $\text{Al}^{3+}$ , followed by muscovite and kaolinite. The dissolution rates of  $\text{Si}^{4+}$  and  $\text{Al}^{3+}$  in montmorillonite still remained greatest even after 3 days of the reaction, while the lowest dissolution rates in muscovite were consistently observed

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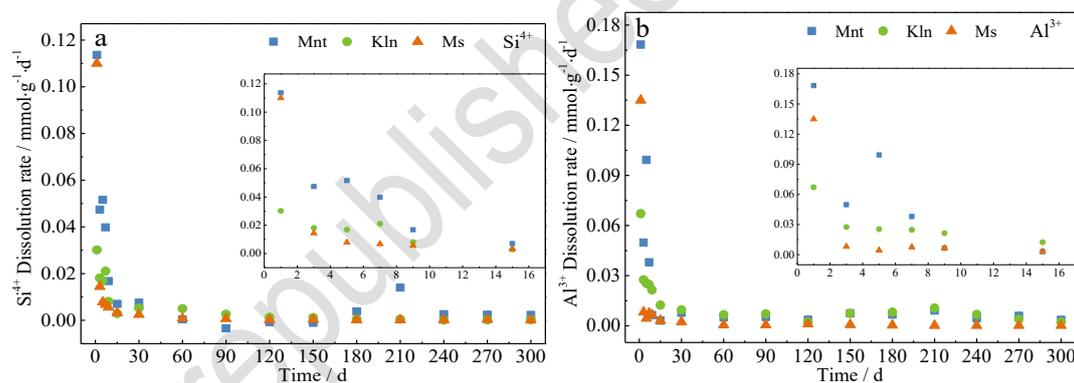
over the remaining 297 days. With the progress of the reaction, montmorillonite and kaolinite exhibited similar dissolution rates of  $\text{Al}^{3+}$ , being an order of magnitude higher than those in muscovite.

The changes in ion dissolution rates in the minerals after the interfacial reactions with oxalic acid were related to a variety of factors, such as particle size, specific surface area, and properties of tetrahedral and octahedral cations. Montmorillonite, kaolinite and muscovite belong to dioctahedral phyllosilicate minerals whose basic units are  $\text{SiO}_4$  tetrahedral and  $\text{AlO}_6$  octahedral sheets. According to the different combinations of basic units, the above minerals can be divided into 1:1 (tetrahedral -octahedral) and 2:1 (tetrahedral -octahedral -tetrahedral) type phyllosilicate minerals. In particular, montmorillonite has a 2:1 layer structure with exchangeable cations ( $\text{Ca}^{2+}$ ,  $\text{Na}^+$ ,  $\text{K}^+$ ) in the interlayer. When it reacts with oxalic acid, the interlayer cations are preferentially replaced by  $\text{H}^+$  and dissolved, and then  $\text{Al}^{3+}$  and  $\text{Si}^{4+}$  are gradually dissolved along the edge structure. In this process,  $\text{H}^+$  continuously penetrates the structure from the interlayer, and the expansion characteristic of montmorillonite also enlarges the effective area between montmorillonite and acid, thereby accelerating the dissolution of  $\text{Al}^{3+}$  and  $\text{Si}^{4+}$ . In turn, kaolinite has a 1:1 layer structure, wherein the theoretical ion dissolution rate increases after the interfacial reaction with oxalic acid. However, it lacks exchangeable cations in the interlayer, making it difficult the penetration of  $\text{H}^+$  therein, and it can only destroy the structural layer from the edge. As a result, the dissolution rates of  $\text{Al}^{3+}$  and  $\text{Si}^{4+}$  are lower than those in montmorillonite under the same conditions. Finally, muscovite has a 2:1 layer structure with  $\text{K}^+$  in the interlayer, but its specific surface area is an order of magnitude smaller than that of montmorillonite. Moreover, the morphology of the muscovite structure is presented by a large layer, whereby the edge of the surface area is negligible compared

to the substrate, which reduces the ion dissolution rate. Therefore, the higher ion dissolution rate of muscovite in the early stage of the reaction could be caused by the fracture of the lamellar structure and the exposure of more active edge sites during the grinding and preparation process.



**Fig. 2.** Dissolution amount curves of  $\text{Si}^{4+}$  (a) and  $\text{Al}^{3+}$  (b) with time for montmorillonite, kaolinite, and muscovite samples with oxalic acid



**Fig. 3.** Dissolution rate curves of  $\text{Si}^{4+}$  (a) and  $\text{Al}^{3+}$  (b) with time for montmorillonite, kaolinite, and muscovite with oxalic acid

**Table 4.** Dissolution percentages of  $\text{Si}^{4+}$  and  $\text{Al}^{3+}$  in montmorillonite, kaolinite, and muscovite after reactions with oxalic acid (%)

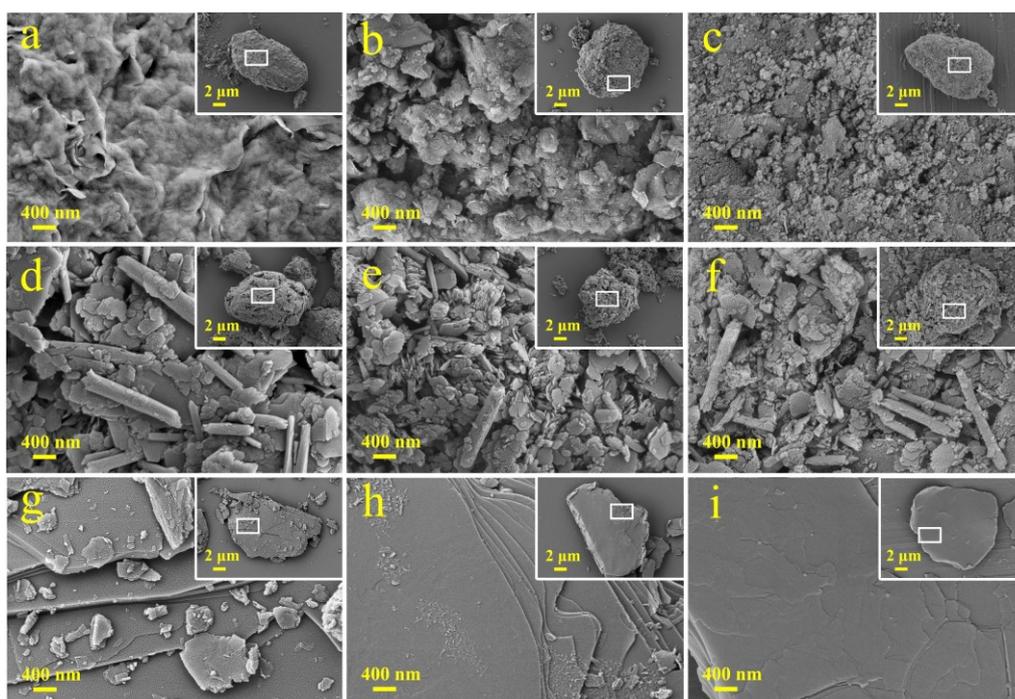
Samples	Mnt			Kln			Ms		
	60 d	150 d	300 d	60 d	150 d	300 d	60 d	150 d	300 d
$\text{Si}^{4+}$	6.3	4.6	12.7	5.6	7.8	8.4	3.1	2.4	3.8
$\text{Al}^{3+}$	23.5	37.2	62.1	9.8	17.0	30.7	3.4	5.6	6.1

### 3.3. Morphological changes

Figure 4 shows the SEM images of montmorillonite, kaolinite, and muscovite as well as the

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products after 150 and 300 days of the reaction with oxalic acid. Figures. 4a-4c illustrate that the montmorillonite aggregated from the lamellar morphology, exhibiting the obvious curling of thin edges. After the reaction with oxalic acid for 150 days, the lamellar morphology was destroyed, and irregular particles with sizes of about 500-100 nm were formed onto the surface. After 300 days of the reaction, these irregular particles became smaller (about 50nm), making the structure loose and porous. Figures 4d-4f show that kaolinite was made by pseudo-hexagonal lamellae and rod-like particles with a diameter of about 200 nm. After the reaction with oxalic acid for 150 days, the edges of the lamellae were obviously blunted, the particles with diameters of 1500-400 nm were decomposed into those with sizes of 400-50 nm. The outer surface of the rod-shaped particles ruptured with many fine lamellar particles was attached to them. The above phenomenon became more obvious after the reaction for 300 days. Figures 4g-4i exhibit that the original morphology of muscovite was presented by lamellae with the average size of about 30  $\mu\text{m}$  and fine particles attached to their surface. After 150 days of the reaction with oxalic acid, the amount of fine particles attached to the surface of muscovite decreased, making its smooth, and the initially sharp lamellar edges became blunted. After 300 days of the reaction, obvious etching pits appeared on the surface of muscovite, which might be related to the preferential dissolution of defect sites on the surface of the mineral.



**Fig. 4.** SEM images of montmorillonite, kaolinite, and muscovite as well as the products after the reaction with oxalic acid for 150 and 300 days a. Mnt, b. Mnt-150, c. Mnt-300, d. Kln, e. Kln-150, f. Kln-300, g. Ms, h. Ms-150, i. Ms-300.

### 3.4. Molecular vibration and bond energy change

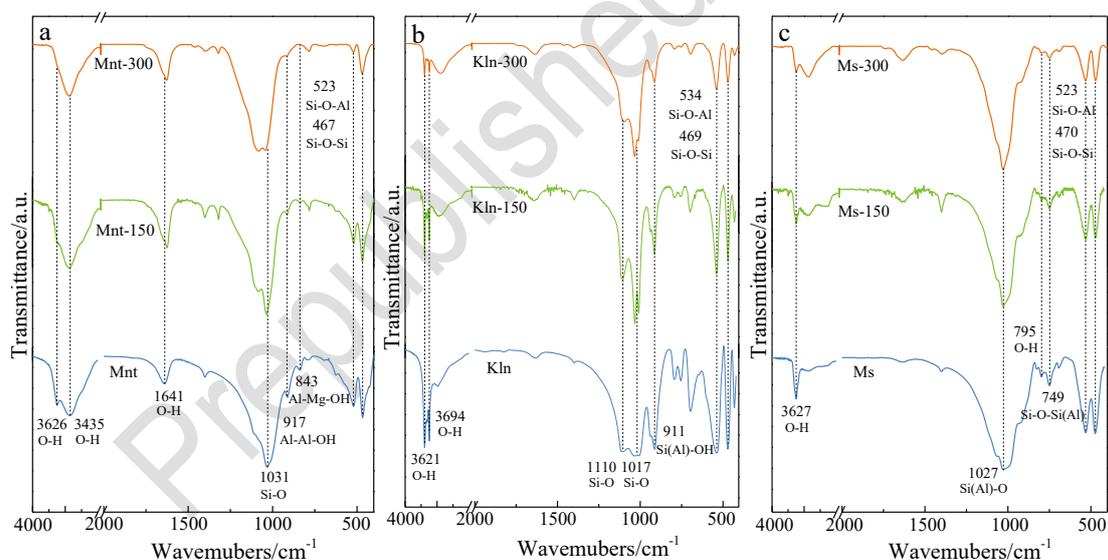
Figure 5 shows the FT-IR spectra of montmorillonite, kaolinite, and muscovite as well as their products after 150 and 300 days of the reaction with oxalic acid. In Figure 5a, the peaks at 3435 and 1641  $\text{cm}^{-1}$  in the spectrum of montmorillonite were caused by the  $\text{H}_2\text{O}$  stretching and bending vibrations in adsorbed and interlayer water in the sample (Deepika and Sethuraman, 2023). Moreover, there was only one O–H stretching vibration peak (at 3626  $\text{cm}^{-1}$ ) as a result of the combination of hydroxyl group with the  $\text{Al}^{3+}$  cation (Farmer and Russell, 1964), indicating that the octahedral Al content in the montmorillonite was high. The peaks at 917  $\text{cm}^{-1}$  (Al–Al–OH) and 843  $\text{cm}^{-1}$  (Al–Mg–OH) confirmed the substitution of octahedral layers (Zhao et al., 2015). The peak at 1031  $\text{cm}^{-1}$  was attributed to a Si–O stretching vibration, and the peaks at 523 and 467  $\text{cm}^{-1}$  were ascribed to the tetrahedral Si–O–Al and Si–O–Si bending vibrations, respectively (Farmer et al.,

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1979; Hassanien et al., 2010; Godek et al., 2022). The intensities of the above vibrational peaks gradually decreased after the interfacial reaction with oxalic acid, and the strengths of the Si–O–Al peaks decreased to a greater extent compared with that of Si–O–Si bonding. This result showed that the interfacial reaction with oxalic acid led to the replacement of the ions by protons in the octahedra and tetrahedra of the montmorillonite, and that tetrahedral  $\text{Si}^{4+}$  were more stable than octahedral  $\text{Al}^{3+}$ .

According to Figure 5b, the peak at  $3621\text{ cm}^{-1}$  in the spectrum of kaolinite was induced by the O–H stretching vibration of the inner hydroxyl group between tetrahedral and octahedral sheets, and the band at  $3694\text{ cm}^{-1}$  was caused by the O–H stretching vibration of the edge hydroxyl group (Farmer and Russell, 1964; Farmer, 1968; Madejová, 2003). The vibrational peaks of the inner and edge hydroxyl groups both decreased with the lengthening of the interfacial reaction with oxalic acid, indicating that protons entered into the structural layer and attacked the hydroxyl groups, leading to dehydroxylation which caused the continuous leaching of  $\text{Al}^{3+}$  from the octahedral layer (Panda et al., 2010). The peaks at  $1110$  and  $1017\text{ cm}^{-1}$  were assigned to the Si–O stretching vibrations (Wang et al., 2016). With the lengthening of the reaction, the peak at  $1017\text{ cm}^{-1}$  was split into the features at  $1028$  and  $1009\text{ cm}^{-1}$ . The peak at  $911\text{ cm}^{-1}$  was caused by the Si(Al)–OH bending vibration (Ehsan et al., 2023), and those at  $469$  and  $534\text{ cm}^{-1}$  corresponded to the Si–O–Si and Si–O–Al bending vibrations, respectively, and the intensities of the above-mentioned vibration peaks decreased with the lengthening of the reaction. The changes in the peak intensities at  $911$ ,  $1009$ ,  $1028$  and  $1110\text{ cm}^{-1}$  indicated that the reaction affected the bonding strength of O–H groups in Si(Al)–O–Al–OH and Si–O in Al(Si)–O–Si–O chains, and the ions at the corresponding positions were replaced by protons (Madejová et al., 1998; Nguetnkam et al., 2011).

Figure 5c illustrates that the muscovite exhibited the O–H stretching vibrational peak at 3627  $\text{cm}^{-1}$ , and the peak at 795  $\text{cm}^{-1}$  was attributed to the O–H out-of-plane oscillating absorption (Fan et al., 2023). The peak intensity gradually decreased after the reaction with oxalic acid, indicating that the OH structure was destroyed in the process. The peaks at 1027 and 749  $\text{cm}^{-1}$  corresponded to the Si(Al)–O and Si–O–Si(Al) stretching vibrations (Luo et al., 2021), and the features at 523 and 470  $\text{cm}^{-1}$  were attributed to the bending vibrations of tetrahedral Si–O–Al and Si–O–Si, respectively. With the lengthening of the reaction, the intensities of the above vibrational peaks decreased gradually. In general, the contents of functional groups in the montmorillonite, kaolinite, and muscovite changed to different degrees after the reactions with oxalic acid, which proved the existence of the interaction between  $\text{H}^+$  and the mineral structure.



**Fig. 5.** FTIR spectra of montmorillonite (a), kaolinite (b), and muscovite (c) as well as the products after the reaction with oxalic acid for 150 and 300 days

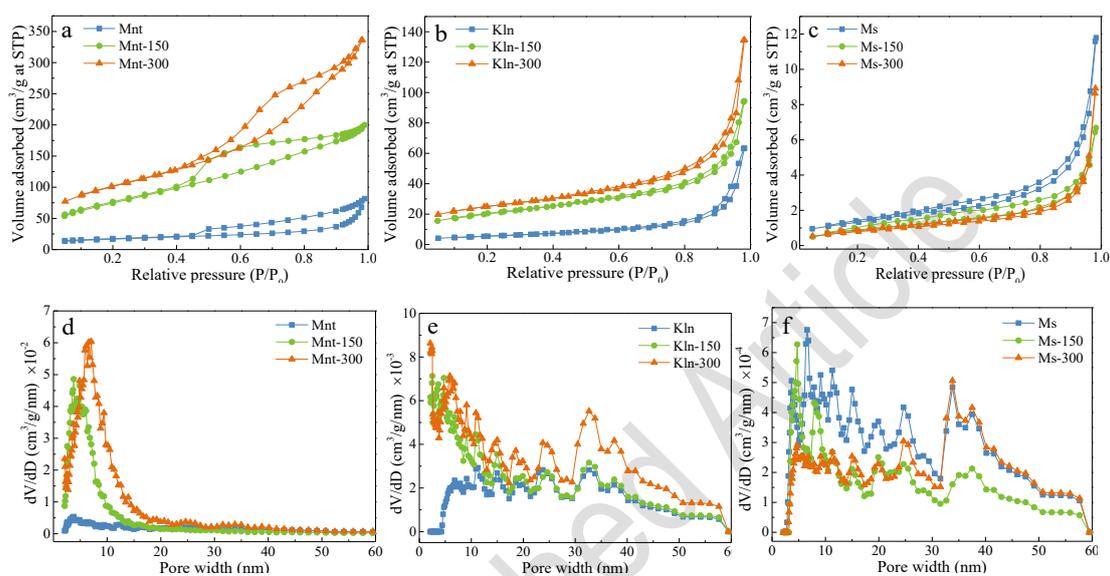
### 3.5. $\text{N}_2$ adsorption-desorption isotherm

Figures 6a-6c show the  $\text{N}_2$  adsorption-desorption isotherms of montmorillonite, kaolinite, and muscovite as well as their products obtained after the reaction with oxalic acid. The isotherms of

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the montmorillonite were similar to the type IV isotherm of the IUPAC classification system, indicating a typical mesoporous structure (Zaghouane-Boudiaf and Boutahala, 2011) with an obvious H3-type hysteresis loop (Vrbková et al., 2023). The isotherms of the kaolinite and muscovite corresponded to those of type-II (Gao et al., 2016), which are generally attributed to the nonporous or macroporous materials. The hysteresis loop can reflect the information about pore distribution and morphology within the specimen (Phadungbut et al., 2016; Toncón-Leal et al., 2021). After the montmorillonite reacted with oxalic acid, the slope of hysteresis loop increased, indicating the pore morphology changed from slit pores between flaky particles to irregularly internal voids (Sing, 1982). Moreover, no obvious change was observed in the adsorption isotherms of the kaolinite and muscovite after the reaction. Based on the obtained adsorption isotherms, Brunauer-Emmett-Teller method (BET) and Nonlocal-Density-Functional-Theory (NLDFT), the specific surface areas and total pore volumes were calculated, and the results are shown in Table 5 and Figures 6d-6f. Figures 6d-6f demonstrate that the pore size distributions of the montmorillonite and kaolinite after the reactions with oxalic acid became wider and the number of pores increased significantly in the range of 2–15 nm. In turn, the pore size distribution in muscovite remained almost unchanged but the porosity decreased significantly. According to Table 5, the specific surface areas of montmorillonite and kaolinite increased by 4.65 and 6.11 times and by 3.59 and 4.41 times after 150 and 300 days of the reaction with oxalic acid, respectively, and the total pore volume also increased to different degrees. The formation of mesopores and micropores occurred due to the partial leaching of  $\text{Al}^{3+}$  cations from the octahedral sheets (Krupskaya et al., 2019). Furthermore, the dissolution of cations made the particles negatively charged, and the charge repulsions existing between the particles caused the original agglomerates to disintegrate, thus increasing the specific

surface area. Although pits and pores were formed during the dissolution of muscovite, the changes were minor compared to those after extensive removal of smaller impurities. The contribution from smaller impurities to the specific surface area was greater than that from pits, resulting in the decrease in the specific surface area and total pore volume of muscovite.



**Fig. 6.** N<sub>2</sub> adsorption-desorption isotherms (a-c) and pore size distribution curves (d-f) of montmorillonite, kaolinite, and muscovite as well as the products after the reaction with oxalic acid for 150 and 300 days

**Table 5.** Total pore volume and specific surface area of montmorillonite, kaolinite, and muscovite as well as the products after the reaction with oxalic acid

Samples	Mnt			Kln			Ms		
	0 d	150 d	300 d	0 d	150 d	300 d	0 d	150 d	300 d
Specific surface area(m <sup>2</sup> ·g <sup>-1</sup> )	58.81	273.31	359.46	19.80	71.16	87.36	4.95	3.29	2.96
Total pore volume(cm <sup>3</sup> ·g <sup>-1</sup> )	0.0208	0.0836	0.1198	0.0064	0.0241	0.0306	0.0015	0.0008	0.0008

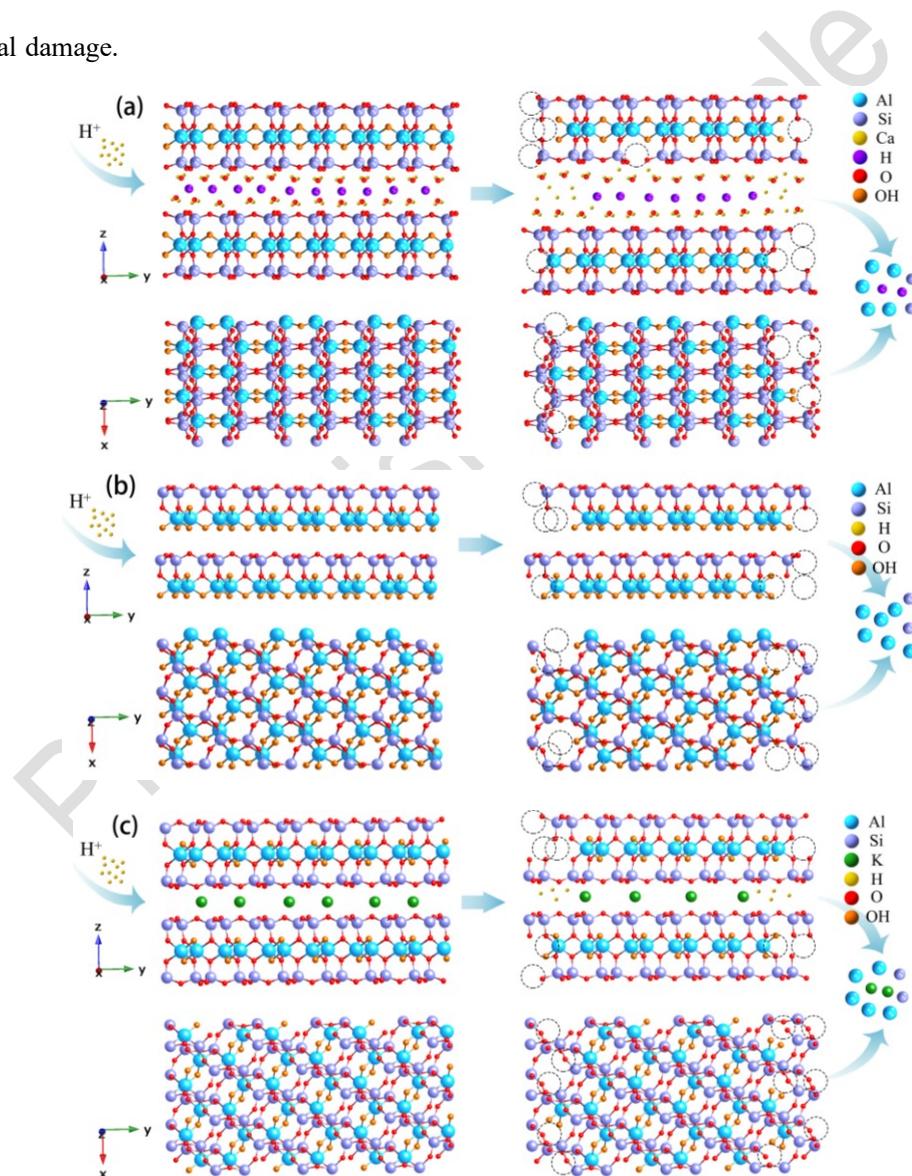
### 3.6. Differential dissolution

Figure 7 displays a schematic diagram of the reaction process. During the reactions between minerals and oxalic acid, H<sup>+</sup> ions first enter into the interlayer and exchange with interlayer cations

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(if any). At the same time,  $H^+$  attacks the Al-O(OH) bonds at the edges of octahedra, allowing  $Al^{3+}$  to enter the solution with the hydroxyl groups. In this process, the dissolution of  $Al^{3+}$  makes the Si-O tetrahedron at the edge unstable, causing  $Si^{4+}$  ions to enter into the solution in the form of  $H_2SiO_3$ , which destroys the mineral structure. The differences between the dissolution rates of minerals are related to the variations in particle size, surface charge, mineral structure and specific surface area, which can arise owing to the following reasons. First, compared with the complexity of the interlayer cations in montmorillonite, kaolinite and muscovite exhibit limited homogeneous and heterogeneous ionic substitutions in their crystal lattices, as well as very few cations other than  $Si^{4+}$  and  $Al^{3+}$ . During the reaction with the acid, the cations in the interlayers in montmorillonite, such as  $Mg^{2+}$  and  $Ca^{2+}$  are first attacked by  $H^+$ , and the continuous penetration of  $H^+$  into the structure accelerates the leaching of  $Al^{3+}$ . The leaching of octahedral cations leads to a decrease in crystallinity and the weakening of the interlayer forces, which accelerates the dissolution process. Second, the dissolution rate of the mineral shows a high correlation with the specific surface area. Assuming that the accessibility of  $N_2$  to the minerals is equal to that of oxalic acid, it appears that the specific surface area determined from the  $N_2$  adsorption-desorption isotherm is equal to the contact area between the mineral and oxalic acid. Therefore, the cation dissolution rates of kaolinite and muscovite with relatively small specific surface areas will be much smaller than those of montmorillonite. During the dissolution process, the Al-O(OH) bonds suffer from the impact of oxalic acid only at the edges of the mineral particles, which means that the dissolution starts from the edge hydroxyl groups (Schliemann and Churakov, 2021b), and the size of the edge specific surface area is more important. The morphological analysis indicates that montmorillonite and kaolinite contain more edge structures, while the edge surface area in the lamellar structure of

muscovite is negligible in relation to that of the basal plane, which reduces its dissolution rate. Third, the specific expandability of montmorillonite not only makes it easy for  $H^+$  to enter into the interlayer and get exchanged with interlayer cations, but also increases the effective interaction area between montmorillonite and the acid, allowing  $H^+$  to attack the tetrahedral Si from the inside and thus accelerating the dissolution. At the same time, the exchanged  $Ca^{2+}$  and oxalate ions form calcium oxalate precipitates, which affect the ion balance and eventually cause more serious structural damage.



**Fig. 7.** Schematic showing the reaction process between minerals and oxalic acid a. montmorillonite, b. kaolinite, c. muscovite.

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## 4. Conclusion

Based on the findings of this study, the main conclusions can be drawn as follows.

(1) The reactions of the minerals with oxalic acid led to the dissolution of ions such as  $\text{Si}^{4+}$  and  $\text{Al}^{3+}$ , and the dissolved amount of ions in kaolinite and muscovite increased gradually with the lengthening of the reaction. The reaction of montmorillonite with oxalic acid produced amorphous silica which is insoluble in the acid. The dissolution rate of  $\text{Al}^{3+}$  was used to assess the effect of oxalic acid on the minerals, which descended in the following order: montmorillonite, kaolinite, and muscovite.

(2) After the interfacial reaction with oxalic acid, the flaky morphology on the surface of montmorillonite was destroyed, and irregular particles with sizes of about 500-100 nm were formed onto the surface. The flake size in kaolinite decreased from 1500-400 nm to 400-50 nm, and the surface of rod-shaped particles was ruptured. The fine particles present on the surface of muscovite disappeared, and the initially sharp lamellar edges became blunted.

(3) The specific surface areas of the products of montmorillonite, kaolinite, and muscovite after 300 days of the reactions with oxalic acid were 6.11, 4.41 and 0.60 times that of the original, respectively. In montmorillonite, the slit-like pores between lamellar particles were transformed into irregularly internal voids. The lamellar particles in kaolinite became smaller and the rod particles were ruptured, which led to the increase in the number of mesopores. The reaction between oxalic acid and muscovite enabled to remove the surface impurities and exerted a little effect on the morphology.

(4) The differential dissolution of the minerals during the interfacial reaction with oxalic acid was related to the complexity of the interlayer cations, the specific surface area, the number of

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edge structures, and the differences in the properties of the minerals. Compared with kaolinite and muscovite, montmorillonite has abundant interlayer cations and edge structures along with the large interlayer spacing and specific surface area, which are conducive to the increase in ion dissolution rate.

It is worth noting that since this work focuses on the chemical weathering of minerals under acidic conditions, microbial inhibitors (sodium azide,  $\text{NaN}_3$ ) have been added to avoid the influence of microorganisms on the mineral-aqueous interface. In fact, the life activities of microorganisms have a great influence on the dissolution of mineral components. Bacteria and their exometabolites can change the element morphology and saturation state of minerals in solutions, and then promote the dissolution of cations in mineral structures (Bundeleva et al., 2014; Lamérand et al., 2020). Fungi affect the weathering of minerals mainly through their selective adhesion to the mineral surface containing growth nutrients, thereby reducing the pH in the environment through the production of organic acids and  $\text{CO}_2$  via respiration and thus accelerating the dissolution of minerals (Lamérand et al., 2022; Li et al., 2022). The presence of fungal microorganisms also impacts the mineral formation and transformation, facilitating the conversion of montmorillonite to illite and chlorite to vermiculite (Ross and Kodama, 1976; Biswas et al., 2017). Therefore, the influence of microorganisms on mineral weathering is very complex. Based on the study of the mineral-aqueous interface reactions, the influence of microorganisms can be further explored, which is of significance for in-depth understanding of the mineral weathering in the surface environment.

#### **Declaration of competing interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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