Cd\(^{2+}\) Exchange for Na\(^{+}\) and K\(^{+}\) in the Interlayer of Montmorillonite: Experiment and Molecular Simulation

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Montmorillonite (Mt) has high cation exchange capacity and thus has been studied extensively for its cation exchange interactions with other cations. However, molecular simulations for the forces governing the cation exchange on Mt surfaces or in the interlayer spaces were limited. In this study, Mt with K\(^{+}\) and Na\(^{+}\) in the interlayer spaces was tested for its cation exchange with Cd\(^{2+}\) in solution and the forces driving the cation exchange reaction were simulated by molecular simulations. The experimental results showed that Na\(^{+}\) in Na\(^{+}\)Mt was completely exchanged by Cd\(^{2+}\), while only 50% of K\(^{+}\) in K\(^{+}\)Mt was exchanged by Cd\(^{2+}\). A larger d-value was noticed for Na\(^{+}\)Mt in comparison to K\(^{+}\)Mt, suggesting that the interlayer space is more hydrated with Na\(^{+}\) as the interlayerspacecation. Molecular dynamics simulations revealed a larger energy decrease when Cd\(^{2+}\) substitutes K\(^{+}\). However, the nice fit of the K\(^{+}\) into the 12-coordinated interlayer space sites may restrict its hydration and thus reduce its interlayer space cation exchange capability by Cd\(^{2+}\).

1. Introduction

Cadmium (Cd) has been extensively used in the fields of mining, electroplating, electrolyzing, painting, alloying, plastic generation, and textile processing [1–4]. As Cd is toxic even at very low concentrations, it is classified as a human carcinogen by the US National Toxicology Program. Significant amounts of Cd can be accumulated in food chain and eventually cause health problems in human beings, plants, and animals [1, 2, 5–7]. It is therefore important to develop methods to remove Cd from wastewater before it reaches the natural environments. Various methods developed for the removal and recovery of Cd from industrial wastewater include chemical precipitation, electrolisis, ion exchange, membrane process, and sorption [8–11].

Montmorillonite (Mt) is a 2:1 phyllosilicate mineral and has high cation exchange capacity (CEC) that can absorb and retain metal cations in its interlayer space to achieve electrical neutrality. Interlayer space hydrated cations are mainly stabilized by electrostatic interactions, and they could be exchanged by other cations in solution. The species of interlayer space hydrated cations would affect interlayer space, surface area, adsorption, swelling, dispersion, and crystal stability of Mt [12]. The desorption of adsorbed water and interlayer space water in Mt is a one-step or two-step process according to the species of interlayer space cations. The conductivity of Mt is affected by the electrovalence of interlayer space cations much more than by the ionic radius.

Molecular modeling recently has been recognized as an efficient method for understanding the microstructure of interlayer spaces [13–18] in clay minerals. The advantages of molecular modeling include the following: (1) simultaneously present insight into the microstructure of the interlayer spaces at the atomic level and the thermodynamic information of the system [13–16]; (2) conveniently design different models for systematic investigation of the influence of various factors on the characteristics of clay minerals [14, 17, 18]. Numerous studies have employed molecular modeling for understanding the hydration characteristics of clay minerals [13–17]. Hydration energy of K\(^{+}\), Na\(^{+}\), or Li\(^{+}\)-exchanged Mt was calculated by molecular dynamic (MD) simulations and the results provided thermodynamic explanations for the inhibition of Mt swelling by K\(^{+}\) [14]. In addition, molecular modeling has also been used to study the adsorption...
of contaminants at the interlayer spaces of Mt in several previous reports [19–25]. Moreover, MD simulations can directly predict the adsorption sites for organic contaminants within the interlayer space of organoclays, which include the siloxane surface and the spaces between organic cations [19].

In this study, we investigated cation exchange reaction of Cd\(^{2+}\) for K\(^{+}\) or Na\(^{+}\) in the interlayer space of Mt experimentally in conjunction with XRD analyses and MD simulation, in order to provide the driving force for interactions between the Mt layers and the interlayer space cation as affected by the cation radii and the degrees of hydration.

2. Materials and Methods

2.1. Materials. The montmorillonite used was SWy-2 obtained from the Clay Mineral Repositories in Purdue University (West Lafayette, IN) and was used without further purification. It has a chemical formula of (Ca\(_{0.12}\) Na\(_{0.32}\) K\(_{0.05}\))[Al\(_{3.01}\) Fe\(_{0.94}\)]\(_{4.01}\) M\(_{0.54}\)\([\text{Si}_{3.98}\text{Al}_{0.02}]\)O\(_{10}\)(OH)\(_4\), a CEC of 85 ± 3 mmol/100 g [26], a layer charge of 0.32 eq/mol per (Si, Al)\(_{0.01}\)O\(_{10}\) [27], an external specific surface area (SSA) of 23 m\(^2\)/g [28], and a mean particle size of 3.2 \(\mu\)m with a \(d_{0.5}\) to \(d_{15}\) in the range of 3–10 \(\mu\)m.

2.2. Methods. The initial Cd\(^{2+}\) concentrations varied from 0.2 to 30 mmol/L for the adsorption isotherm study and were fixed at 2 mmol/L for the kinetic study and pH dependency study. The mass of SWy-2 used was 0.2 g while the volume of solution used was 10 mL for all studies except the kinetic study, in which 20 mL of solution was used. The solid and solution were combined in each 50 mL centrifuge tube and shaken for 2 h at 150 rpm and room temperature for all studies except the kinetic study, in which the shaking time was 1, 3, 5, 30, 60, 120, 600, and 1200 min. After the mixtures were centrifuged at 10000 rpm for 20 min, the supernatants were filtered through 0.22 \(\mu\)m syringe filters before being analyzed for equilibrium Cd\(^{2+}\) concentrations.

Free swelling was determined using 1 g of Mt and 100 mL of water in 100 mL graduated cylinder; the mixture was allowed to fully swell with periodic agitation and eventually to set out. The increase of volume after swelling was determined from the difference between the final swelling volume and the initial dry volume of the solid and expressed as mL/g.

Powder XRD analyses were performed on a Rigaku D/max-IIIa diffractometer (Tokyo, Japan) with a Ni-filtered CuK\(_{α}\) radiation at 30 kV and 20 mA. Orientated samples were scanned from 4\(^{∘}\) to 10\(^{∘}\) at 2\(^{∘}\)/min with a scanning step of 0.01\(^{∘}\). Powder samples were packed in horizontally held trays. The changes in the XRD reflection positions reflect the hydrated size of the metal cations in the interlayer space of Mt.

X-ray fluorescence (XRF) measurements were carried out using a portable XRF spectrometer (Oxford Instruments) with a molybdenum anode, at 25 kV and 0.1 mA. A Si-PIN detector from AMPTEK was employed and characterized by an energy resolution of about 200 eV at 5.9 keV.

FTIR spectra of samples were collected on a Nicolet-560 spectrometer (Thermal Nicolet Co., USA) from 400 to 4000 cm\(^{-1}\) with a nominal resolution of 4 cm\(^{-1}\). For each spectrum 16 runs were collected and averaged. The SWy-2 specimens were prepared by adding approximately 1% of the sample powder to dry KBr powder.

Thermogravimetric (TG) analyses were carried out on TGA Q-500 (TA Instruments, New Castle, USA) from room temperature to 800\(^{∘}\)C, at a heating rate of 10\(^{∘}\)/min under a nitrogen flow of 60 mL/min. TG curves were used to determine the percentage of mass loss. Differential scanning calorimetry (DSC) was performed using a differential scanning calorimeter (TA Instruments Q100) fitted with a cooling system using liquid nitrogen. It was calibrated with an indium standard. Samples of 6 mg of Mt were accurately weighed into aluminum pans, sealed, and then heated from 30 to 800\(^{∘}\)C at 10\(^{∘}\)/min under a nitrogen flow of 60 mL/min.

2.3. Computation Details. Molecular simulation was performed under the module “CASTEP” of Materials Studio 6.0 software to investigate the sorption sites of K\(^{+}\), Na\(^{+}\), and Cd\(^{2+}\) on SWy-2. The primitive unit cell of SWy-2 was optimized with the generalized gradient approximation (GGA) for the exchange-correlation potential (PW91) that is appropriate for the relatively weak interactions present in the models studied. The resulting primitive unit cell was characterized by the parameters \(a = 15.540\ \text{Å}, b = 17.940\ \text{Å}, c = 12.56\ \text{Å}\), and \(α = γ = 90^{\circ}, β = 99^{\circ}\). Based on the primitive unit cell, a series of \((3 \times 2 \times 1)\) supercells were built with the \(d\)-value of layers set to 10.55, 12.74, and 14.81 Å for K + Mt, Na + Mt, and Cd + Mt, respectively.

3. Results and Discussion

3.1. Preparation of K + Mt. The cation exchangeability of Mt is affected by interaction force of interlayer space cation and Mt layer and force of binding water. To investigate the impact of species of interlayer space cation on cation exchangeability, the experiment was designed first to compare properties of K + Mt and Na + Mt, as Na\(^{+}\) and K\(^{+}\) belong to the same main group and have the same electrovalence, but different ionic radii. To keep all conditions the same, except the type of interlayer cations, the Na + Mt was first exchanged with K\(^{+}\) to prepared K + Mt. After cation exchange by K\(^{+}\), the Na + Mt was completely converted to K + Mt (Table 1).

The thickness of dehydrated Mt is 9.8 Å, and the interlayer space will be expanded when Na\(^{+}\) and K\(^{+}\) hydrated cations enter the interlayer space. As hydration ability of different cations varies, interlayer spaces of Mts with different cations in the interlayer space differ apparently. K\(^{+}\) concentrations of varying multiples of CEC in solution will result in different amounts of K\(^{+}\) uptake in Mt, which will result in a change in \(d_{001}\) spacing (Figure 1). The \(d_{001}\) of original Na + Mt was 12.74 Å. As the amount of K\(^{+}\) substitution increased, the interlayer space decreased. At an initial input of K\(^{+}\) at 10 times the CEC of Mt, the \(d_{001}\) of Mt reduced to 10.55 Å (Figure 1). However, this \(d\)-value is still greater than that of illite or mica, suggesting still presence of limited interlayer space water.

Hydration of cations on interlayer space and outer surface leads to volume expansion of Mt, called free swell. The free swelling of Mt decreased as the amount of K\(^{+}\) uptake increased (Table 2). Pure Na + Mt had a free swelling of 41.5 mL/g. It decreased to 11.5 mL/g after Na\(^{+}\) was fully
Table 1: Chemical composition of Mts and their chemical formula.

<table>
<thead>
<tr>
<th>Chemical formula</th>
<th>Na + Mt</th>
<th>Na + Mt after Cd$^{2+}$ exchange</th>
<th>K + Mt</th>
<th>K + Mt after Cd$^{2+}$ exchange</th>
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<tr>
<td>SiO$_2$</td>
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<td>62.56</td>
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<tr>
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<td>14.60</td>
<td>15.16</td>
<td>15.23</td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
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<td>6.75</td>
<td>6.62</td>
<td>6.55</td>
</tr>
<tr>
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<td>2.86</td>
<td>3.51</td>
<td>2.87</td>
</tr>
<tr>
<td>CaO</td>
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<td>2.95</td>
<td>3.35</td>
<td>2.12</td>
</tr>
<tr>
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<td>0.00</td>
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<td>0.00</td>
</tr>
<tr>
<td>K$_2$O</td>
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<td>0.00</td>
<td>4.73</td>
<td>2.04</td>
</tr>
<tr>
<td>CdO</td>
<td>0.00</td>
<td>9.48</td>
<td>0.00</td>
<td>3.66</td>
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</table>

Table 2: Free swelling (mL/g) of raw Na + Mt and Mt after being in contact with KCl of varying amounts for 120 min.

<table>
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<th>Na + Mt</th>
<th>0.25</th>
<th>0.33</th>
<th>0.5</th>
<th>0.7</th>
<th>1</th>
<th>2</th>
<th>5</th>
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<tr>
<td>41.5</td>
<td>36</td>
<td>30</td>
<td>27</td>
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<td>18</td>
<td>14</td>
<td>12</td>
<td>11.5</td>
</tr>
</tbody>
</table>

Figure 1: X-ray diffraction patterns of K-MMT. The d-spacing is in Å.

Table 3: Chemical composition of Mts and their chemical formula.

3.2. Cd$^{2+}$ Uptake from K + Mt and Na + Mt. Interaction forces between Mt layer and interlayer space are electrostatic attraction, intermolecular forces, van der Waals forces, and so forth. Owing to combination of these interactions, metal cations stay stable in the interlayer space place. The experiment used Cd$^{2+}$ to exchange interlayer space cations of Na$^+$ or K$^+$ from Na + Mt or K + Mt. By discussing influence of time and concentration of Cd$^{2+}$ on exchanging Na$^+$ or K$^+$ from Na + Mt or K + Mt, exchanging rate and volume of two cationic Mts can also be calculated. The difficulty of exchanging cation in Mt interlayer space is relevant with interaction force of interlayer cation and layer, which means stronger interaction force leading to more difficulty in exchange [30].

The cation exchange process of Cd$^{2+}$ for Na$^+$ or K$^+$ in the interlayer space of Mt is relatively fast (Figure 2), which can reach 60% of total exchange capacity in 30 min and complete exchange in 2 h, especially for K + Mt, reaching the exchanging balance in 30 min. However, the amount of Cd$^{2+}$ uptake on K$^+$-Mt was lower than that on Na$^+$-Mt. The maximal exchange capacity reached 0.43 mmol/g or 0.86 mmol/g. As the CEC of the original Mt was 0.85 mmol/g, it can be inferred that Na$^+$ in Na + Mt was completely exchanged while K$^+$ in K + Mt was only partially exchanged. The Cd$^{2+}$ isotherm data agreed well with the chemical analysis data (Table 1 and Figure 2). This indicated that Mts with different interlayer cations lead to different exchange rate and capacity for Cd$^{2+}$, which is directly related to the type of interlayer cation [31].

After linear fit of the experimental results, it was found that the exchange Cd$^{2+}$ for Na$^+$ or K$^+$ followed the first-order kinetics. The rate constants for Ca$^{2+}$ to exchange K$^+$ or Na$^+$ in K + Mt or Na + Mt were 9.8 g/mmol and 3.9 g/mmol, respectively.

The results of XRD analyses indicated the progressive completion of Cd$^{2+}$ exchange for Na in Na + Mt and partial exchange of Cd$^{2+}$ for K$^+$ in K + Mt. This could be used to determine element composition and content of exchanged Na + Mt and K + Mt and then to calculate the amount of cations exchanged. XRF results showed that the content of Na$^+$ for Na + Mt after being exchanged by Cd$^{2+}$ is 0 (Table 1), which means interlayer cations are almost comprised of Cd$^{2+}$ and exchange rate is 100%. On the other hand, more than 40% of the original K$^+$ was still remaining (Table 1).
indicating that K⁺ cations were only partially exchanged, even though the input amount of Cd²⁺ was equivalent to 3 times the CEC of the Mt. These results showed that K⁺ as the interlayer cation is more stable, as in the case of illite and mica.

The effect of initial solution concentration on exchange capacity had a similar result; that is, the interlayer cation Na⁺ in Na + Mt can be completely exchanged by Cd²⁺ ions, the amount of exchange increases as Cd²⁺ concentration increased, and exchange balance was achieved when the input Cd²⁺ was 3 times the CEC. The discrepancy in exchange amounts of Mts with different interlayer space cations is due to diverse interaction forces of layers and interlayer cations [32, 33]. The result of exchange amounts means interaction force between Mt layer and K⁺ was greater than that between Mt layer and Na⁺. This larger interaction force may prevent complete Cd²⁺ exchange for K⁺ in the interlayer space of Mt.

It can be indicated from interlayer space of Mt (Figure 3) that increasing concentration of solution prompts resulted in sharp and enhanced reflection of (001). The $d_{001}$ value of Cd + Mt increased as the amount of Cd²⁺ adsorption increased and reflected at 14.8 Å. This value is larger than 13.15 Å for
Figure 4: X-ray diffraction patterns of K-MMT cations exchanged with Cd²⁺ (d/Å). Effect of time/min (a) and Cd²⁺ input in CEC (b).

Figure 5: FTIR analyses of K-MMT, Na-MMT, and Cd-MMT.

Pb-exchanged Mt and smaller than 15.90 Å for Zn-exchanged Mt [34]. In contrast to Na + Mt, the (001) reflection of K + Mt after being exchanged with different amounts of Cd²⁺ was no longer present, suggesting delamination of Mt (Figure 4).

The FTIR spectra of Mt in contact with different amounts of Cd²⁺ were illustrated in Figure 5. The wide absorbing band observed at 3625 cm⁻¹ was assigned to Al-O-H stretching vibration of Mt structural water, and wide adsorbing band at 3436 cm⁻¹ could be assigned to stretching of hydroxyl in adsorbed and interlayer space water [35]. The shoulder within 1630–1640 cm⁻¹ was due to hydroxyl bending vibration of adsorbed water, while another shoulder observed at about
1035 cm$^{-1}$ could be attributed to stretching of Si-O-Si in Mt lattice. The weak adsorb band observed at 914 cm$^{-1}$ was owing to bending vibration of octahedral hydroxyl in Mt lattice, and the sharp band at 523 cm$^{-1}$ is attributed to bending vibration of Si-O-Si [36].

For K + Mt, as the amount of Cd$^{2+}$ uptake increased, the band within 1630–1640 cm$^{-1}$ increased gradually, while band at 1447 cm$^{-1}$ disappeared. Bands at these two positions are attributed to bending vibration of hydroxy in adsorbed water, which means changes in interaction force between Mt layer and interlayer cation would lead to variation in position and intensity of these bands. However, the band at 1035 cm$^{-1}$ representing bending vibration of Si-O-Si in Mt lattice and band at 3625 cm$^{-1}$ representing bending of Al-O-H in Mt structural water both decrease as intercalation amount of Cd$^{2+}$ increases, meaning interaction force of K$^+$ and Mt layer was stronger, leading to a minor change in Mt structure that affected force in layer. For Na + Mt, as Cd$^{2+}$ exchange preceded, except for disappearance of absorption band at 1447 cm$^{-1}$ and increase in transmittance at 1648 cm$^{-1}$, bands at other positions showed no apparent changes. It means that Na + Mt had a weaker interaction force between the Mt layer and interlayer cations in comparison to K + Mt. This would lead to an easy exchange of Cd$^{2+}$ for Na$^+$. The mass change within 25–175°C was due to removal of adsorbed surface and interlayer space water of Mt [37]. For K + Mt, as Cd$^{2+}$ uptake increased, mass loss within 25–175°C increased markedly, from initial 4.1% to 12.9% (Figure 6(a)). The result indicated less amount of interlayer space water in K + Mt in comparison to Cd + Mt, suggesting weaker interaction force between K$^+$ cation and water molecule than that between Cd$^{2+}$ and water molecules; that is, K$^+$ was less hydrated than Cd$^{2+}$. The differential thermal analysis (DTA) curves showed a similar trend within 25–175°C. (Figure 6(b)). The endothermic valley was due to removal of interlayer space and adsorbed water. As the amount of Cd$^{2+}$ uptake increased, endothermic valley moves to a slightly higher temperature, indicating that interaction force between Cd$^{2+}$ and water molecule was greater than that between K$^+$ and water. Mass loss of Na + Mt in 25–175°C was 10.8%, 6.7% more than that of K + Mt (Figure 6(c)), illustrating that Na$^+$ in Na + Mt was more hydrated than K$^+$ in K + Mt. As less interlayer space water resulted in smaller interlayer space, a stronger electrostatic attraction of interlayer cation and layers and difficult exchangeability would be anticipated. Therefore, amount of interlayer space water played an important role in easiness of cation exchange for Mt. The mass loss of Na + Mt in 25–175°C increased from initial 10.8% to 17% as the amount of Cd$^{2+}$ uptake increased (Figure 6(c)), while the maximum mass loss after Cd$^{2+}$ exchanging K$^+$ in K + Mt was only 12.9%. It indicated better cation exchangeability of Na + Mt than K + Mt by Cd$^{2+}$ once again. And in DTA curve (Figure 6(d)), within 25–175°C, endothermic valley remained at the same position as the amount of Cd$^{2+}$ uptake on Na + Mt increased, showing similar interaction force of Na$^+$ with Cd$^{2+}$ to water molecule, while the amount of combined water of Cd$^{2+}$ was more than Na$^+$. The interlayer space of K + Mt, Na + Mt, and Cd + Mt was 10.55, 12.5, and 14.81 Å, respectively. MD simulation showed that minimum distances from K$^+$, Na$^+$, and Cd$^{2+}$ to oxygen in Mt silicate-oxygen tetrahedron would be 2.303, 2.443, and 4.648 Å, respectively (Figure 7). From top view, it can be seen that K$^+$, Na$^+$, and Cd$^{2+}$ are all located on oxygen hexagonal ring, which is above Mg substituting position of alumina in octahedron; maximum angles between the three ions to the nearest oxygen and diagonal oxygen were 102.58°, 103.36°, and 54.28°. From the relations of distance and angle of interlayer cation and oxygen in Mt, silica-oxygen tetrahedron, interaction force of K$^+$ and Mt layer is the strongest, the distance is less than 2.5 Å, and the angle is obtuse; thus it is possible to form chemical bonds. While interaction force of Na$^+$ and Mt layer is weaker than that of K$^+$, so the exchange capacity of Na$^+$ is higher than that of K$^+$. The interlayer cations are all hydrated, where minimal distance of K$^+$, Na$^+$, or Cd$^{2+}$ to oxygen in water molecular was 2.965, 2.274, or 3.83 Å, respectively. Only Na$^+$-O distance is less than 2.5 Å, suggesting forming a weak bond.

One Na$^+$ cation is surrounded by two water molecules, while distances from the central cation to the two water molecules were different. Distances from K$^+$ or Cd$^{2+}$ to oxygen in water molecules were all longer than 2.5 Å, impossible to form a chemical bond. In addition, the position of Cd$^{2+}$ to water differs from that of Na$^+$ and K$^+$, as Na$^+$ and K$^+$ are surrounded by two water molecules, monolayer arranged, while Cd$^{2+}$ is surrounded by four water molecules and bilayer arranged. This would lead to expansion of the interlayer space to 14.81 Å. The interaction force of K$^+$ and Mt layer is the strongest, and that of K$^+$ and
water molecular is the weakest; interaction force of Na⁺ and water is the weakest, resulting in a higher free swelling of Na⁺ Mt.

4. Conclusions

Cd²⁺ was tested to exchange with different interlayer cations in Mts. By comparing exchange capacity, interlayer space, and energy, we discovered great diversity in degrees of cation exchange of different Mts. Na⁺ Mt can be thoroughly exchanged by Cd²⁺. Conversely, only 60% of K in Mt can be exchanged by Cd²⁺ at an input amount equivalent to 3 times the CEC. The result of XRD indicated that exchange of Na⁺ by Cd²⁺ in Mt was uniform and complete, and molecule dynamics simulation proved that electrostatic force of K⁺ is larger than that of Na⁺, showing an easier cation exchange of Cd²⁺ for Na⁺. A better swelling was found for Na⁺ Mt compared to K⁺ Mt, suggesting a better hydration of Na⁺ in the interlayer space of Mt. The result of molecule dynamic simulation showed energies of three Mts following Cd⁺ Mt < Na⁺ Mt < K⁺ Mt. Interaction force with Mt layer followed K⁺ > Na⁺ > Cd²⁺. Therefore, Mts with diverse interaction cations have different properties and should be used in different fields.

Figure 6: TG and DTA curves of K-MMT (a, b), Na-MMT (c, d), and Cd-MMT.
**Conflict of Interests**

The authors declare that there is no conflict of interests regarding the publication of this paper.

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**References**


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