


Cite this: *RSC Adv.*, 2024, 14, 14640

# Influence of alkali metal ions ( $K^+$ and $Na^+$ ) on the preparation of magnesium hydroxide hexagonal flakes

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Magnesium hydroxide ( $Mg(OH)_2$ ), as a green halogen-free flame retardant, has attracted significant attention in the field of flame retardant composite materials. In addition to conventional indicators such as purity and whiteness,  $Mg(OH)_2$  is required to take the form of regular hexagonal sheets to ensure the dispersion of composite materials. We use irregular large particles of  $Mg(OH)_2$  prepared by the magnesium factory in western Qinghai as raw materials to study the influence of alkali metal ions  $K^+$  and  $Na^+$  mainly present in salt lakes on the physicochemical properties of  $Mg(OH)_2$ . The products were characterized via X-ray diffraction, scanning electron microscopy, automatic nitrogen physical adsorption apparatus, and other modern characterization techniques. Results show that alkali metal ions  $K^+$  and  $Na^+$  considerably influence the crystal surface polarity, particle size, and morphology of the prepared  $Mg(OH)_2$ . The mechanism analysis shows that the presence of  $K^+$  and  $Na^+$  alters the dissolution, recrystallization, and growth characteristics of  $Mg(OH)_2$ . This study provides theoretical support for the realization of high-performance  $Mg(OH)_2$  using salt lake resources and demonstrates the value for promoting the large-scale industrial application of the salt lake industry.

Received 12th January 2024

Accepted 22nd April 2024

DOI: 10.1039/d4ra00305e

rsc.li/rsc-advances

## 1. Introduction

$Mg(OH)_2$  is nontoxic and nonpolluting and exhibits high-temperature resistance, corrosion resistance, and other excellent physical and chemical properties.<sup>1,2</sup> Therefore, it is widely used as an antibacterial agent in wastewater<sup>3–5</sup> and waste gas, for the treatment of acidic water contaminants, for fillers in the paper industry,<sup>6</sup> in electronic and optical devices,<sup>7,8</sup> and as a precursor for producing magnesium oxide<sup>9</sup> and new generation flame retardants.<sup>10–15</sup> The physical and chemical properties of  $Mg(OH)_2$  products, such as the morphology, particle size, particle size distribution, dispersion, specific surface area, whiteness, and purity, play a key role in their applications. The preparation of  $Mg(OH)_2$  products not only requires their high purity but also necessitates the regulation of their physical and chemical properties, such as the particle size and morphology.<sup>16,17</sup> Despite significant research in this area, numerous challenges remain in the actual production and preparing high-performance  $Mg(OH)_2$  is still a topic of interest at present.

The hydrothermal method is commonly employed to control nucleation growth of the  $Mg(OH)_2$  crystal surface by regulating its dissolution and recrystallization.<sup>18</sup> Currently, the use of ultrasound, microwave-assisted technology,<sup>19</sup> and additives,<sup>20</sup> among other, to regulate  $Mg(OH)_2$  formation during hydrothermal reactions is a hot topic. Further, the most commonly employed method involves regulating the nucleation, crystallization, and other growth processes of  $Mg(OH)_2$  using various additives.<sup>21</sup> Gong *et al.*<sup>22</sup> showed that the presence of magnesium lactate can transfer hydroxyl groups from brucite to  $Mg(OH)_2$  nanoplates, thereby acting as a conveyor belt. Gómez-Villalba<sup>23</sup> *et al.* showed that using hydrazine hydrate as a surfactant significantly affects the physical and chemical properties, especially the morphology, of the obtained  $Mg(OH)_2$  products. According to literature, inorganic alkaline crystal surface regulators, such as NaOH, can provide sufficient  $OH^-$ <sup>24</sup> for  $Mg(OH)_2$  recrystallization and thus rearrange its structure by taking advantage of the differences in the action of different polar crystal faces and  $OH^-$ <sup>25</sup> to control the crystal face orientation and particle size.<sup>26</sup> Although there have been some studies on the use of sodium hydroxide and potassium hydroxide as crystal regulators to regulate  $Mg(OH)_2$  growth, no studies exist on alkali metal ions  $K^+$  and  $Na^+$  as additives. Hence, herein, we conducted a study on the effects of  $K^+$  and  $Na^+$  on the hydrothermal synthesis of  $Mg(OH)_2$  with large and irregular particles. The study provides a great reference value for the preparation of high-performance  $Mg(OH)_2$ .

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The influence of  $K^+$  and  $Na^+$  on the physical and chemical properties of prepared  $Mg(OH)_2$  was studied using NaCl and KCl as crystal surface regulators and  $Mg(OH)_2$  with large particles produced by the magnesium industry in western Qinghai as the raw material. Alkali metal ions  $K^+$  and  $Na^+$  were used to regulate the dissolution and recrystallization of  $Mg(OH)_2$ , related crystal growth mechanism was discussed, and low-polarity  $Mg(OH)_2$  hexagonal flakes were prepared. This study has important theoretical and practical significance for the preparation of flame retardants using high-performance  $Mg(OH)_2$  hexagonal flakes.

## 2. Material and methods

### 2.1. Materials

KCl and NaCl were procured from Xilong Science Co, LTD. Purified water was prepared in the laboratory.  $Mg(OH)_2$  with large particles was produced by the magnesium industry in western Qinghai was used as the raw material; its morphology, particle size distribution, and crystal phase composition are shown in Fig. 1.

### 2.2. Experimental methods

A total of 40 g of  $Mg(OH)_2$  (0.69 mol) and a certain amount of NaCl and KCl were weighed and added into a high-temperature, high-pressure reactor along with 160 mL purified water. Hydrothermal heat treatment was then performed at 500  $r\ min^{-1}$  with varying additive concentration (namely KCl and NaCl concentration), reaction temperature, and reaction time. After the reaction was completed, the  $Mg(OH)_2$  cake was washed using an ethanol solution to neutralize it, dried at 105  $^{\circ}C$  for 6 h, and the obtained  $Mg(OH)_2$  product was sealed for subsequent characterization and analysis.

### 2.3. Characterization analysis test

Several analytical techniques were used to characterize the obtained powders.

**X-ray powder Diffraction.** The resulting powder crystalline phases were carried out using X-ray diffraction (XRD, DX-2700BH). A Cu  $K\alpha$  source was used over a  $2\theta$  range of  $20^{\circ}$  to  $60^{\circ}$  and a step size of  $0.02^{\circ}$  with a dwell time of 0.05 s was applied during the analyses.

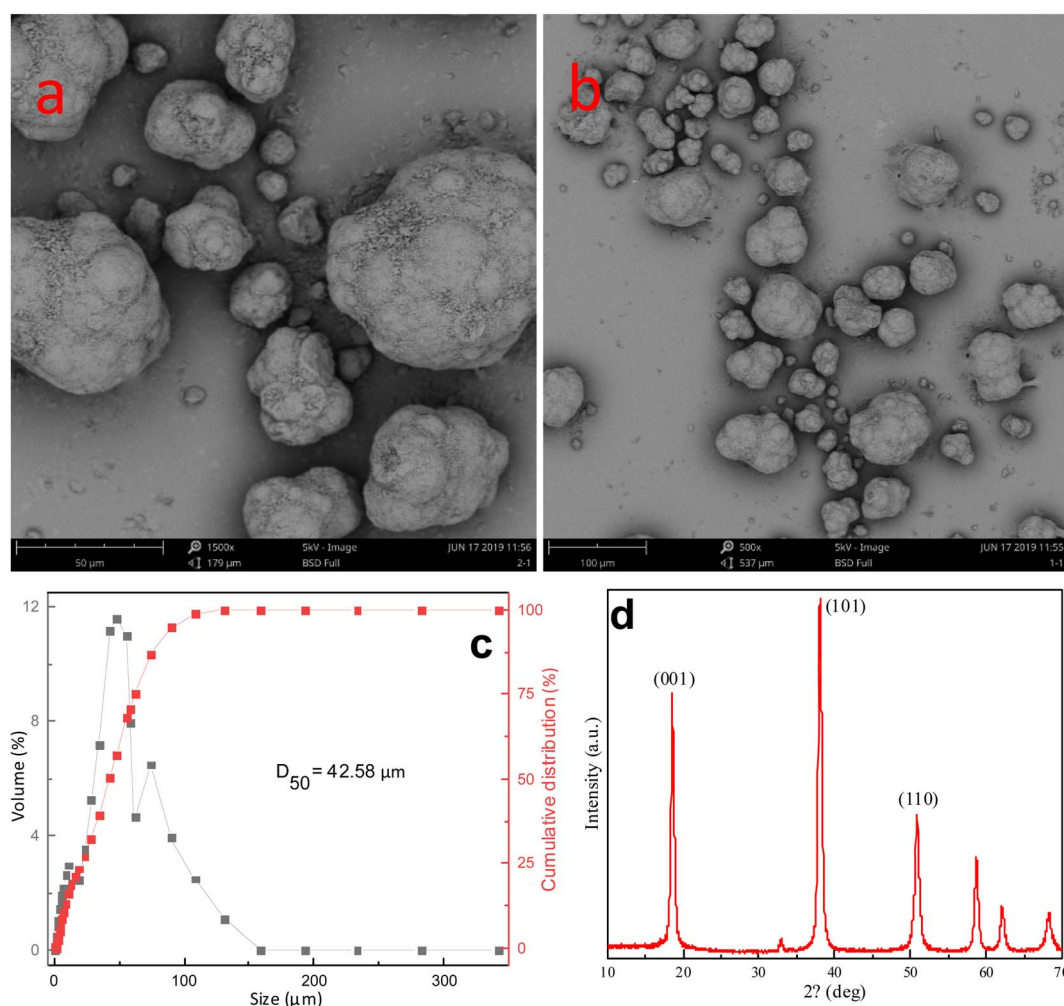


Fig. 1 Morphology, particle size distribution, and crystal phase composition of  $Mg(OH)_2$  products produced by the magnesium industry in western Qinghai. ((a and b): SEM images; (c): particle size distribution and cumulative distribution; and (d): XRD pattern).

Scanning Electron Spectroscopy (SEM). The particle shape and size were analyzed by scanning electron microscopy (SEM, SU8010). The powder samples for SEM were shown and observed at a working distance of 3.5 mm and a stimulation voltage of 0.7 kV.

Dynamic light scattering (DLS). Using the dynamic light scattering method, the obtained  $\text{Mg}(\text{OH})_2$  particle, D50 size, and particle size distribution were researched by laser particle size analysis (OMCC, SCF-106A).

Brunauer–Emmett–Teller (BET).  $\text{N}_2$  adsorption–desorption isotherms are recorded on a Micro Active for ASAP 2460 Version 2.02 at 77.300 K.  $\text{N}_2$  adsorption–desorption isotherm (ASAP2460) is employed to analyze pore structure and surface area of  $\text{Mg}(\text{OH})_2$ . Before the test, the samples are degassed overnight at 180 °C.

### 3. Results and discussion

The crystal phases and crystallinity of  $\text{Mg}(\text{OH})_2$  were determined *via* X-ray diffraction. Fig. 2 shows the X-ray diffraction (XRD) pattern of  $\text{Mg}(\text{OH})_2$  samples prepared at 160 °C in 6 h using different  $\text{Na}^+$  concentrations. These XRD patterns exhibit typical diffraction peaks, which were assigned to (001), (100), (101), (102), and (110) planes of the structure of  $\text{Mg}(\text{OH})_2$ . No additional XRD peaks arising from impurities were detected. As shown in Fig. 2, the strongest (*hkl*) peaks are located at  $2\theta$  values of 18.60°, 38.02°, and 50.84°, and the corresponding (*hkl*) index is as follows: (001), (101), and (102) are the main peaks of (001), (101) and (102) of  $\text{Mg}(\text{OH})_2$ , respectively. The diffraction peak appears at  $2\theta$  value 58.66, corresponding to the  $\text{Mg}(\text{OH})_2$  crystal phase plane (110). The presence of  $\text{Na}^+$  has an impact on the strength of the obtained  $\text{Mg}(\text{OH})_2$  crystal face. With the increasing content of  $\text{Na}^+$ , the strength of the (001) crystal face of the obtained  $\text{Mg}(\text{OH})_2$  product first increases and then decreases.

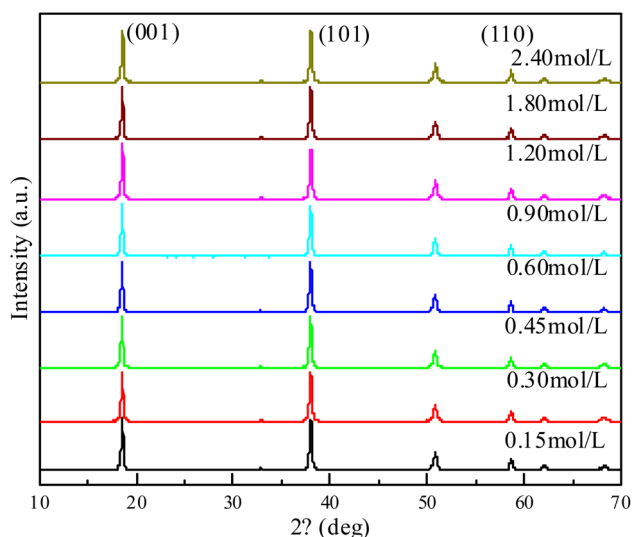


Fig. 2  $\text{Mg}(\text{OH})_2$  products prepared using different  $\text{Na}^+$  concentrations (reaction time: 6 h; reaction temperature: 160 °C).

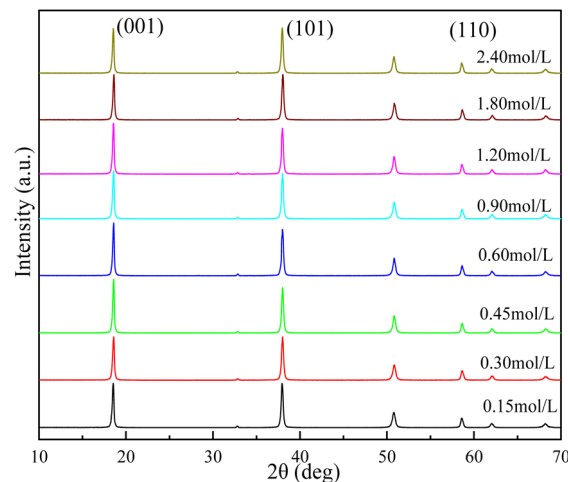


Fig. 3  $\text{Mg}(\text{OH})_2$  products with different concentrations of  $\text{K}^+$  (reaction time: 6 h; reaction temperature: 160 °C).

The  $\text{Mg}(\text{OH})_2$  crystal phases and crystallinity were determined by XRD. Fig. 3 shows the XRD pattern of  $\text{Mg}(\text{OH})_2$  samples prepared at a synthesis temperature of 160 °C and a synthesis time of 6 h in the presence of different concentrations of  $\text{K}^+$ . These XRD patterns exhibit typical diffraction peaks assigned to (001), (100), (101), (102), and (110) planes of the structure of  $\text{Mg}(\text{OH})_2$ . No additional XRD peaks arising from impurities were detected. The strongest (*hkl*) peak detected is located at  $2\theta$  values of 18.60, 38.02, and 50.84, and the corresponding (*hkl*) index is as follows: (001), (101), and (102) are the main peaks of (001), (101) and (102) of  $\text{Mg}(\text{OH})_2$ , respectively. The diffraction peak appears at  $2\theta$  value 58.66, corresponding to the  $\text{Mg}(\text{OH})_2$  crystal phase plane (110). The presence of  $\text{K}^+$  has an impact on the strength of the obtained  $\text{Mg}(\text{OH})_2$  crystal face. With the increase in  $\text{K}^+$  content, the crystal face of the obtained  $\text{Mg}(\text{OH})_2$  product first increases and then decreases.

Fig. 2 and 3 show that the increase in the concentration of  $\text{K}^+$  and  $\text{Na}^+$  leads to an increase and subsequent decrease of the intensity of (001), (101), and (110) diffraction peaks, with the intensity being the highest when the concentration of  $\text{K}^+$  and  $\text{Na}^+$  is 0.45 mol  $\text{L}^{-1}$ . This is attributed to less  $\text{H}^+$  generated by hydrolysis when  $\text{K}^+$  and  $\text{Na}^+$  concentration is low, leading to a slower dissolution of  $\text{Mg}(\text{OH})_2$ , which is not conducive to the dissolution-crystallization of  $\text{Mg}^{2+}$  on the surface of  $\text{Mg}(\text{OH})_2$ , which hinders crystal growth. At high concentrations of  $\text{K}^+$  and  $\text{Na}^+$ , hydrolysis releases more  $\text{H}^+$  and the pH of the system is maintained at a low level, which is also not conducive to crystal growth.<sup>27</sup> Furthermore, due to the charge neutralization effect of anionic flocculants on  $\text{Mg}(\text{OH})_2$  and positive charge on the surface of  $\text{Mg}(\text{OH})_2$ ,  $\text{Cl}^-$  introduced by NaCl and KCl combines with  $\text{Mg}(\text{OH})_2$  more rapidly, further accelerating the dissolution of  $\text{Mg}(\text{OH})_2$ .<sup>28</sup> Simultaneously, there is a higher number of impurity ions ( $\text{Cl}^-$ ) in the solution, which affects the ordered crystallization of  $\text{Mg}^{2+}$  on the crystal surface of  $\text{Mg}(\text{OH})_2$  and reduces the integrity of crystal growth.

Fig. 4a shows that  $I_{001}/I_{101}$ , and  $I_{001}/I_{110}$  first increase and then decrease with increasing NaCl and KCl concentrations.



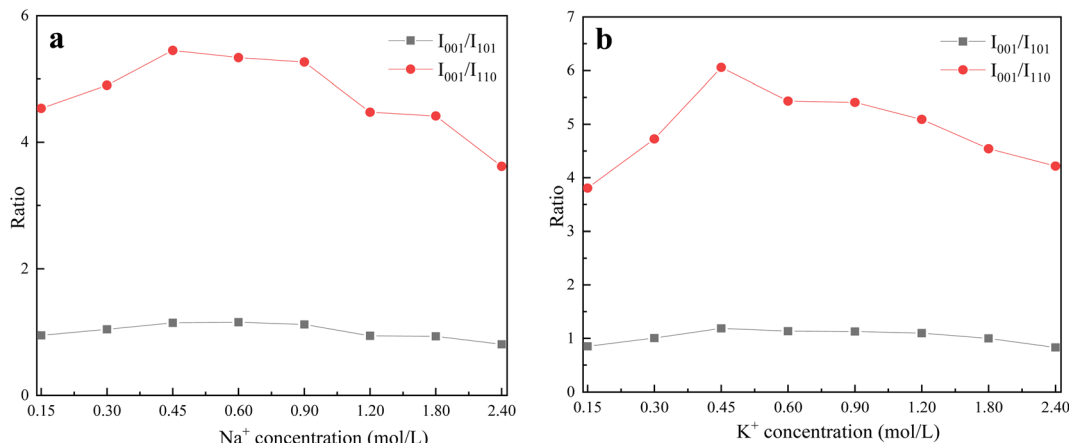


Fig. 4 XRD characteristic peak ratio of  $\text{Mg}(\text{OH})_2$  products at different  $\text{Na}^+$  and  $\text{K}^+$  concentrations ((a):  $\text{Na}^+$ ; (b):  $\text{K}^+$ ).

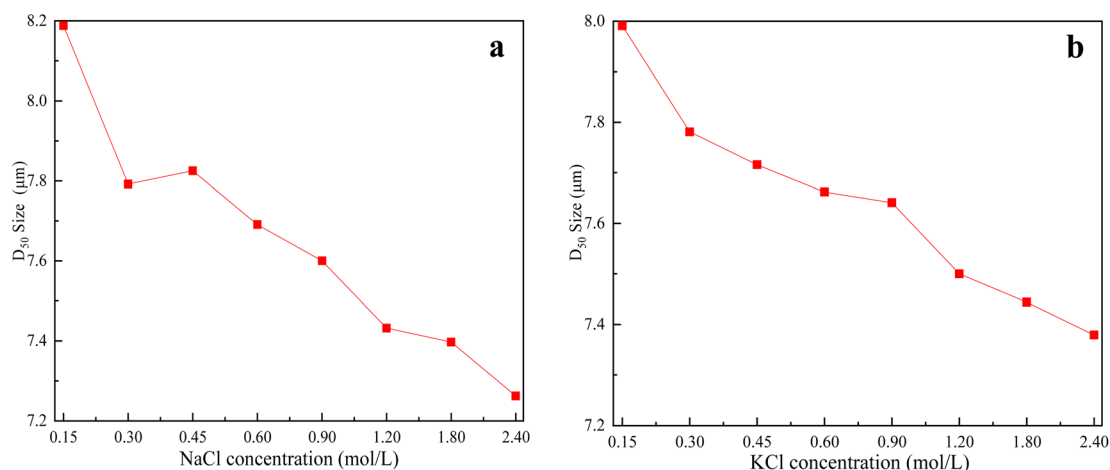


Fig. 5  $D_{50}$  particle size variations of  $\text{Mg}(\text{OH})_2$  products at different  $\text{Na}^+$  and  $\text{K}^+$  concentrations ((a):  $\text{Na}^+$ ; (b):  $\text{K}^+$ ).

When  $\text{K}^+$  and  $\text{Na}^+$  concentration is  $0.45 \text{ mol L}^{-1}$ ,  $I_{001}/I_{101}$  reaches its maximum. This shows that  $\text{K}^+$  and  $\text{Na}^+$ , as crystal surface regulators, promote the selective growth of the (001)

crystal surface. However, when  $\text{K}^+$  and  $\text{Na}^+$  concentration is low, the dissolution efficiency of  $\text{Mg}(\text{OH})_2$  decreases and the growth rate of the (001) crystal plane is lower than those of the (101) and

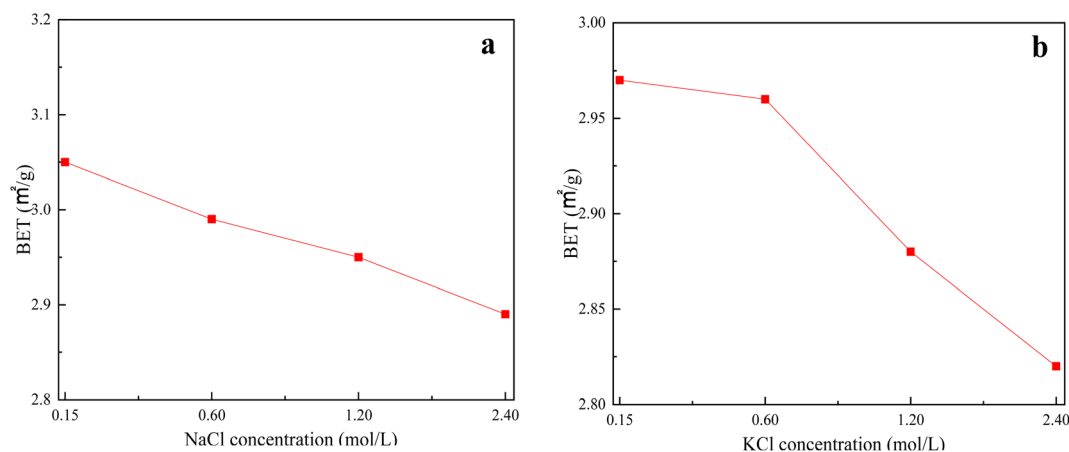


Fig. 6 BET changes of  $\text{Mg}(\text{OH})_2$  products at different  $\text{Na}^+$  and  $\text{K}^+$  concentrations ((a):  $\text{Na}^+$ ; (b):  $\text{K}^+$ ).



(110) crystal planes. When the crystal-surface-regulator concentration is high, excess  $\text{Cl}^-$  is introduced, which has a significant influence on the growth of the (001) crystal surface. We conclude that the concentrations of  $\text{K}^+$  and  $\text{Na}^+$ , the crystal surface regulators, have a significant influence on the selective growth of the  $\text{Mg}(\text{OH})_2$  crystal surface. The increase in  $I_{001}/I_{101}$  and  $I_{001}/I_{110}$  may be attributed to the positive charge on the surface of the  $\text{Mg}(\text{OH})_2$  particles, which easily adsorb anions. As the radius of  $\text{OH}^-$  is smaller than that of the  $\text{Cl}^-$  in the precipitation system,<sup>29</sup>  $\text{OH}^-$  is more easily adsorbed onto the basic crystal plane of microcrystals and promotes the growth of particle edges. Thus,  $\text{K}^+$  and  $\text{Na}^+$  promote the growth of  $\text{Mg}(\text{OH})_2$  edges by providing  $\text{Cl}^-$  and improve the growth of the (001) crystal surface.<sup>30</sup>

Fig. 5 shows the variation in the particle size  $D_{50}$  of  $\text{Mg}(\text{OH})_2$  products prepared using different concentrations of NaCl and KCl. As shown in Fig. 5a, when the  $\text{Na}^+$  concentration in the reaction system increases from 0.15 to 2.40  $\text{mol L}^{-1}$ , the particle size of the obtained  $\text{Mg}(\text{OH})_2$  products increases from 8.18  $\mu\text{m}$  dropped to 7.26  $\mu\text{m}$ . Similarly, as shown in Fig. 5b, when the concentration of  $\text{K}^+$  in the reaction system increases from 0.15 to 2.40  $\text{mol L}^{-1}$ , the particle size of the obtained  $\text{Mg}(\text{OH})_2$  products increases from 7.99  $\mu\text{m}$  dropped to 7.38  $\mu\text{m}$ . We conclude that the increasing concentration of  $\text{K}^+$  and  $\text{Na}^+$  leads to a decrease in the particle size of the obtained  $\text{Mg}(\text{OH})_2$  products. Thus, the presence of  $\text{K}^+$  and  $\text{Na}^+$  has a significant effect on the dissolution and recrystallization of  $\text{Mg}(\text{OH})_2$ , resulting in a continuous decrease in the particle size of the obtained products.

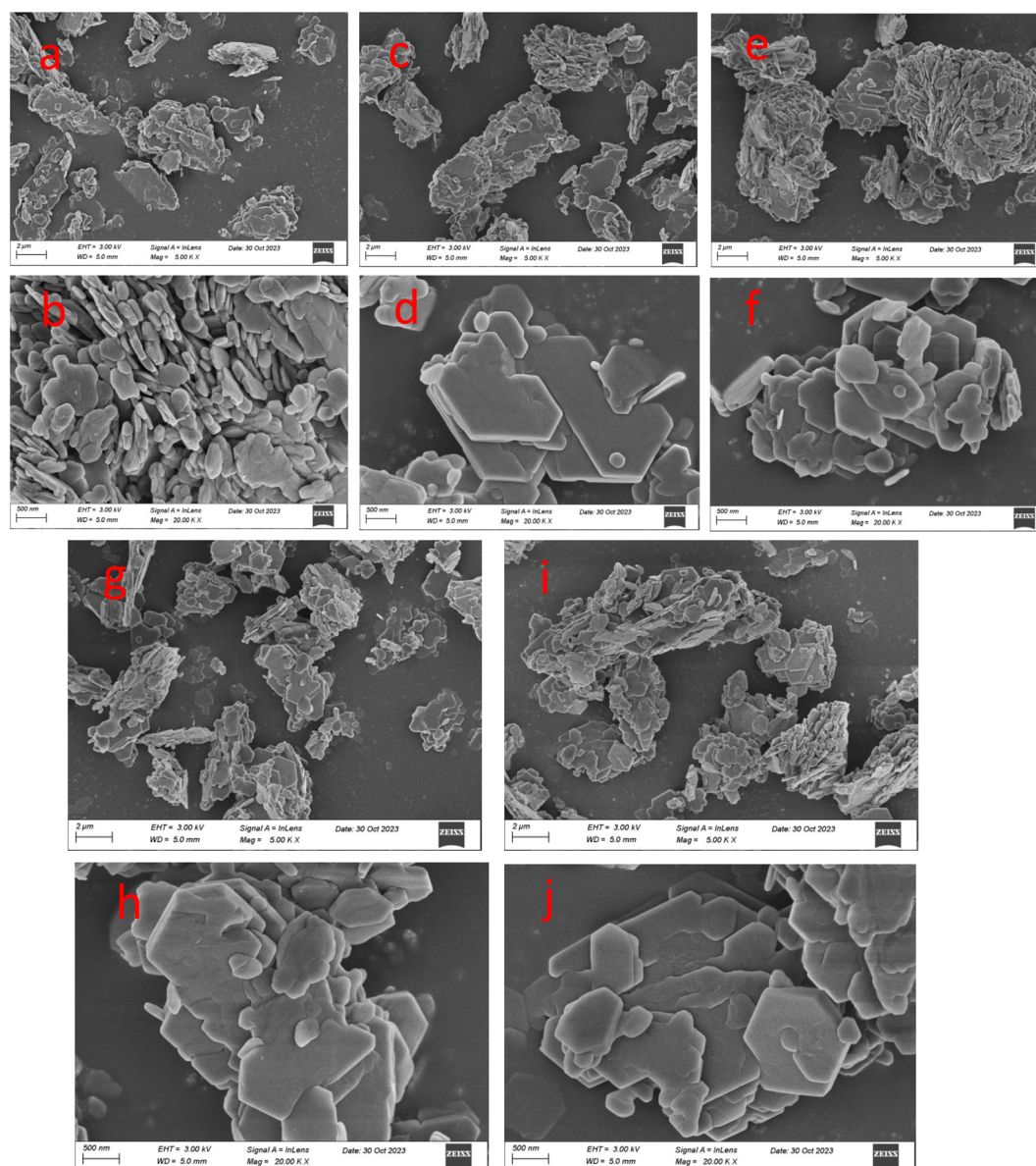


Fig. 7 Morphology of  $\text{Mg}(\text{OH})_2$  products prepared using different concentrations of  $\text{Na}^+$  ((a and b): 0.15  $\text{mol L}^{-1}$ ; (c and d): 0.30  $\text{mol L}^{-1}$ ; (e and f): 0.60  $\text{mol L}^{-1}$ ; (g and h): 1.20  $\text{mol L}^{-1}$ ; (i, j and k): 2.40  $\text{mol L}^{-1}$ ).



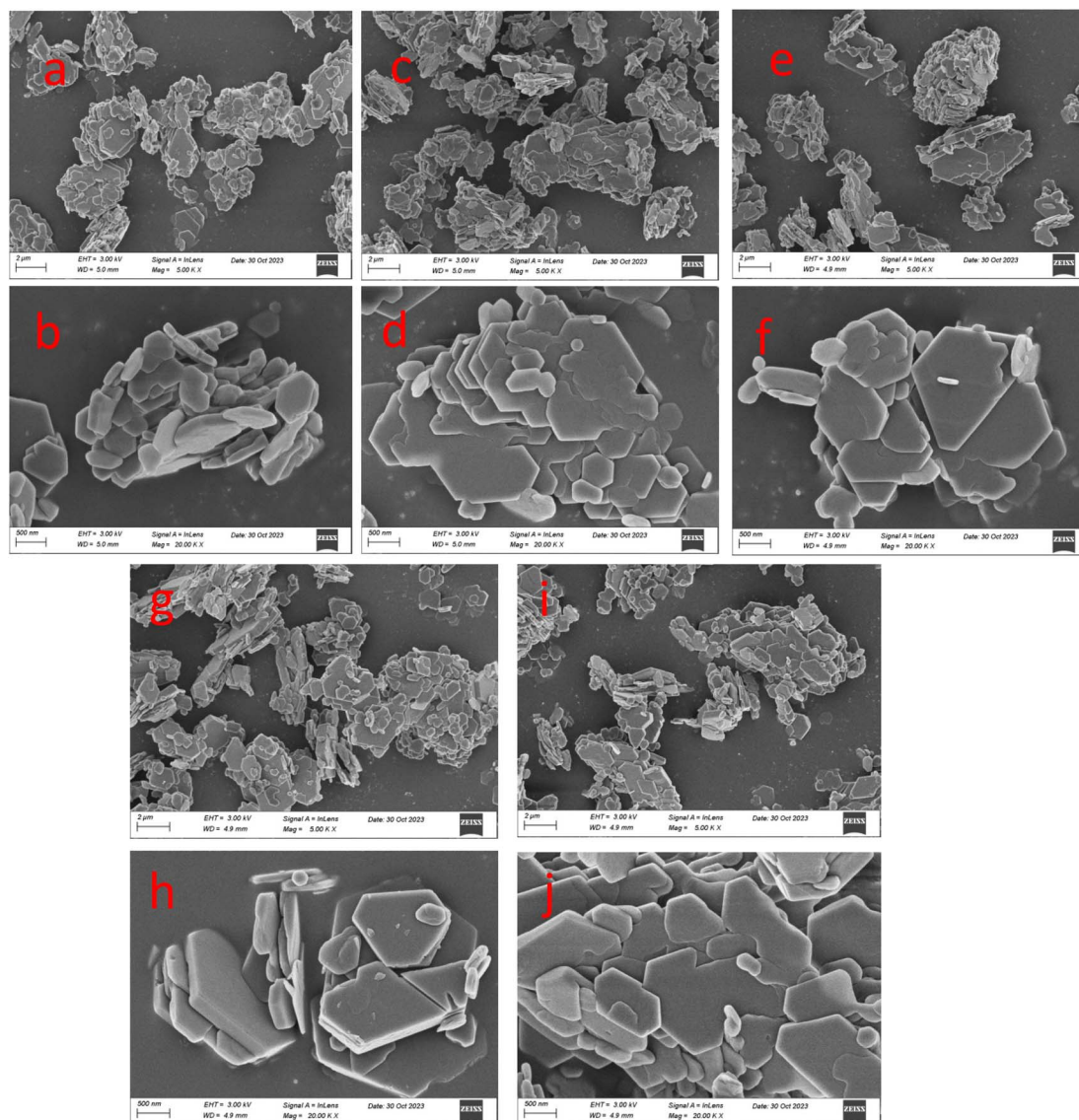


Fig. 8 Morphology of  $\text{Mg}(\text{OH})_2$  products prepared using different  $\text{K}^+$  concentrations ((a and b):  $0.15 \text{ mol L}^{-1}$ ; (c and d):  $0.30 \text{ mol L}^{-1}$ ; (e and f):  $0.60 \text{ mol L}^{-1}$ ; (g and h):  $1.20 \text{ mol L}^{-1}$ ; (i, j and k):  $2.40 \text{ mol L}^{-1}$ ).

Fig. 6 shows the BET changes of  $\text{Mg}(\text{OH})_2$  products obtained using different  $\text{K}^+$  and  $\text{Na}^+$  concentrations. With an increase in  $\text{Na}^+$  concentration from  $0.15$  to  $2.40 \text{ mol L}^{-1}$ , the specific surface area of the obtained  $\text{Mg}(\text{OH})_2$  product decreases from  $3.05$  to  $2.89 \text{ m}^2 \text{ g}^{-1}$  (Fig. 6a). With an increase in  $\text{K}^+$  concentration from  $0.15$  to  $2.40 \text{ mol L}^{-1}$ , the specific surface area of the obtained  $\text{Mg}(\text{OH})_2$  product decreases from  $2.97$  to  $2.82 \text{ m}^2 \text{ g}^{-1}$  (Fig. 6b). The continuous increase of alkali metal ions  $\text{K}^+$  and  $\text{Na}^+$  causes the specific surface area of the obtained  $\text{Mg}(\text{OH})_2$  product to decrease continuously, albeit the decrease is small. This may be attributed to the small polarity of the obtained  $\text{Mg}(\text{OH})_2$  product under these conditions, such that the presence of alkali metal ions  $\text{K}^+$  and  $\text{Na}^+$  has a relatively small impact on the specific surface area of  $\text{Mg}(\text{OH})_2$ .

Fig. 7 and 8 show SEM images of  $\text{Mg}(\text{OH})_2$  products obtained in the presence of different concentrations of alkali metal ions

$\text{K}^+$  and  $\text{Na}^+$ . The products obtained under these conditions are all hexagonal  $\text{Mg}(\text{OH})_2$  products. Compared with the morphology of the products obtained in Fig. 1, the morphology was subjected a significant change from irregular large particle  $\text{Mg}(\text{OH})_2$  to regular hexagonal flake  $\text{Mg}(\text{OH})_2$ . This indicates that different concentrations of alkali metal ions  $\text{K}^+$  and  $\text{Na}^+$  play an important in the dissolution and recrystallization process of  $\text{Mg}(\text{OH})_2$ , influencing the morphology of the obtained products.

## 4. Conclusions

Herein, we studied the influence of alkali metal ions  $\text{K}^+$  and  $\text{Na}^+$  as crystal surface regulators on the hydrothermal dissolution and recrystallization of  $\text{Mg}(\text{OH})_2$ . The results show that the presence of  $\text{K}^+$  and  $\text{Na}^+$  has a significant effect on the crystal





structure, polarity, and particle size of the  $\text{Mg}(\text{OH})_2$  products. Under specific hydrothermal conditions,  $\text{K}^+$  and  $\text{Na}^+$  play a major role in controlling the selective growth of low-polarity crystal planes of  $\text{Mg}(\text{OH})_2$ . The polarity ratio of the  $\text{Mg}(\text{OH})_2$  products prepared using  $\text{Na}^+$  decreased from the highest value of 1.19 to 0.83. When the concentration of  $\text{NaCl}$  in the reaction system increases from 0.15 to 2.40 mol  $\text{L}^{-1}$ , the particle size of the obtained  $\text{Mg}(\text{OH})_2$  products increases from 8.18  $\mu\text{m}$  dropped to 7.26  $\mu\text{m}$ . Similarly, an increase in the concentration of  $\text{K}^+$  in the reaction system from 0.15 to 2.40 mol  $\text{L}^{-1}$  leads to an increase in the particle size of the obtained  $\text{Mg}(\text{OH})_2$  products from 7.99  $\mu\text{m}$  dropped to 7.38  $\mu\text{m}$ .  $\text{K}^+$  and  $\text{Na}^+$  promote the transformation of irregular large particles of  $\text{Mg}(\text{OH})_2$  products into regular hexagonal flakes under hydrothermal conditions. This study provides theoretical support for the preparation of hexagonal flakes of high-performance  $\text{Mg}(\text{OH})_2$ .

## Conflicts of interest

There are no conflicts to declare.

## Acknowledgements

This work was partially supported by the Open Project of Salt Lake Chemical Engineering Research Complex, Qinghai University (2023-DXSSKF-01), the National Key Research and Development Program of China (2022YFC2904305) and Major Science and Technology Projects of Qinghai Province (2020-GX-A1).

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