


 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.^{1,2} Therefore, it is widely used as an antibacterial agent in wastewater³⁻⁵ and waste gas, for the treatment of acidic water contaminants, for fillers in the paper industry,⁶ in electronic and optical devices,^{7,8} and as a precursor for producing magnesium oxide⁹ and new generation flame retardants.¹⁰⁻¹⁵ 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.^{16,17} 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.¹⁸ Currently, the use of ultrasound, microwave-assisted technology,¹⁹ and additives,²⁰ 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.²¹ Gong *et al.*²² 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-Villalba²³ *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⁻²⁴ 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⁻²⁵ to control the crystal face orientation and particle size.²⁶ 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 2θ range of 20° to 60° and a step size of 0.02° with a dwell time of 0.05 s was applied during the analyses.

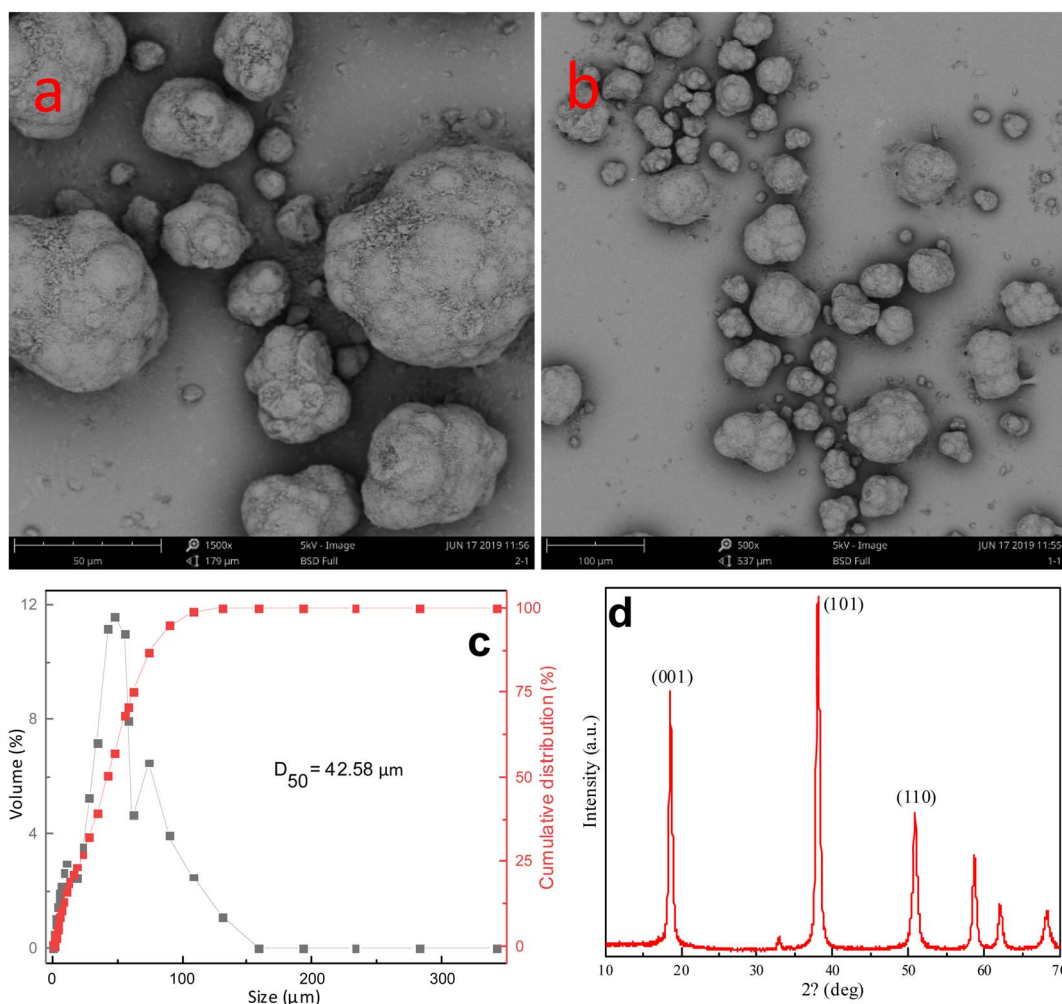


Fig. 1 Morphology, particle size distribution, and crystal phase composition of $Mg(OH)_2$ products produced by the magnesium industry in western Qinghai. ((a and b): SEM images; (c): particle size distribution and cumulative distribution; and (d): XRD pattern).



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). N_2 adsorption-desorption isotherms are recorded on a Micro Active for ASAP 2460 Version 2.02 at 77.300 K. 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 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 2θ 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 2θ value 58.66, corresponding to the $\text{Mg}(\text{OH})_2$ crystal phase plane (110). The presence of Na^+ has an impact on the strength of the obtained $\text{Mg}(\text{OH})_2$ crystal face. With the increasing content of Na^+ , the strength of the (001) crystal face of the obtained $\text{Mg}(\text{OH})_2$ product first increases and then decreases.

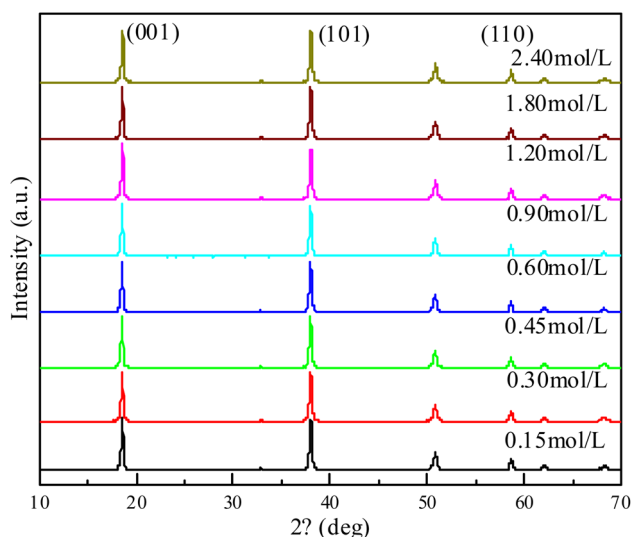


Fig. 2 $\text{Mg}(\text{OH})_2$ products prepared using different Na^+ concentrations (reaction time: 6 h; reaction temperature: 160 °C).

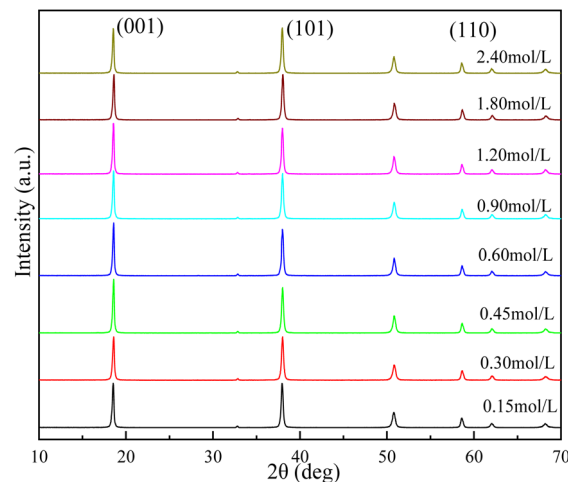


Fig. 3 $\text{Mg}(\text{OH})_2$ products with different concentrations of K^+ (reaction time: 6 h; reaction temperature: 160 °C).

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 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 2θ 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 2θ value 58.66, corresponding to the $\text{Mg}(\text{OH})_2$ crystal phase plane (110). The presence of K^+ has an impact on the strength of the obtained $\text{Mg}(\text{OH})_2$ crystal face. With the increase in 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 K^+ and 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 K^+ and Na^+ is 0.45 mol L^{-1} . This is attributed to less H^+ generated by hydrolysis when K^+ and Na^+ concentration is low, leading to a slower dissolution of $\text{Mg}(\text{OH})_2$, which is not conducive to the dissolution-crystallization of Mg^{2+} on the surface of $\text{Mg}(\text{OH})_2$, which hinders crystal growth. At high concentrations of K^+ and Na^+ , hydrolysis releases more H^+ and the pH of the system is maintained at a low level, which is also not conducive to crystal growth.²⁷ Furthermore, due to the charge neutralization effect of anionic flocculants on $\text{Mg}(\text{OH})_2$ and positive charge on the surface of $\text{Mg}(\text{OH})_2$, Cl^- introduced by NaCl and KCl combines with $\text{Mg}(\text{OH})_2$ more rapidly, further accelerating the dissolution of $\text{Mg}(\text{OH})_2$.²⁸ Simultaneously, there is a higher number of impurity ions (Cl^-) in the solution, which affects the ordered crystallization of 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.



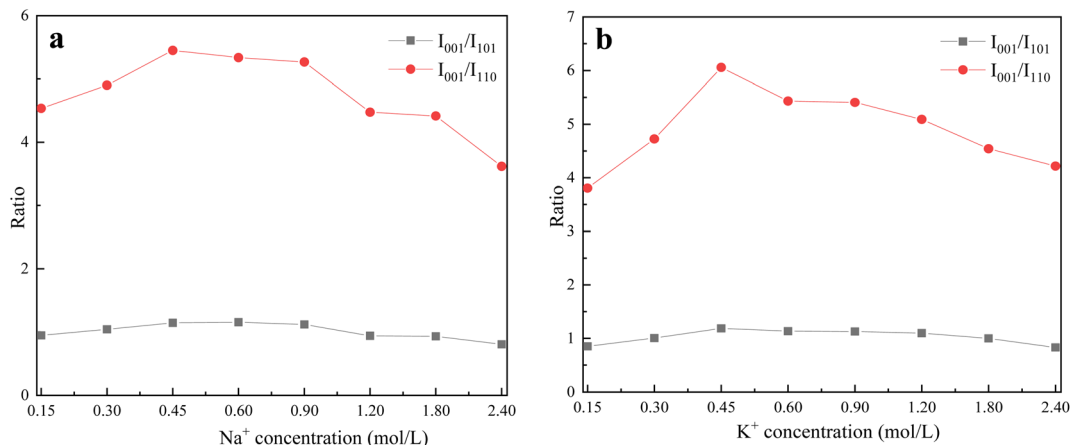


Fig. 4 XRD characteristic peak ratio of Mg(OH)₂ products at different Na⁺ and K⁺ concentrations ((a): Na⁺; (b): K⁺).

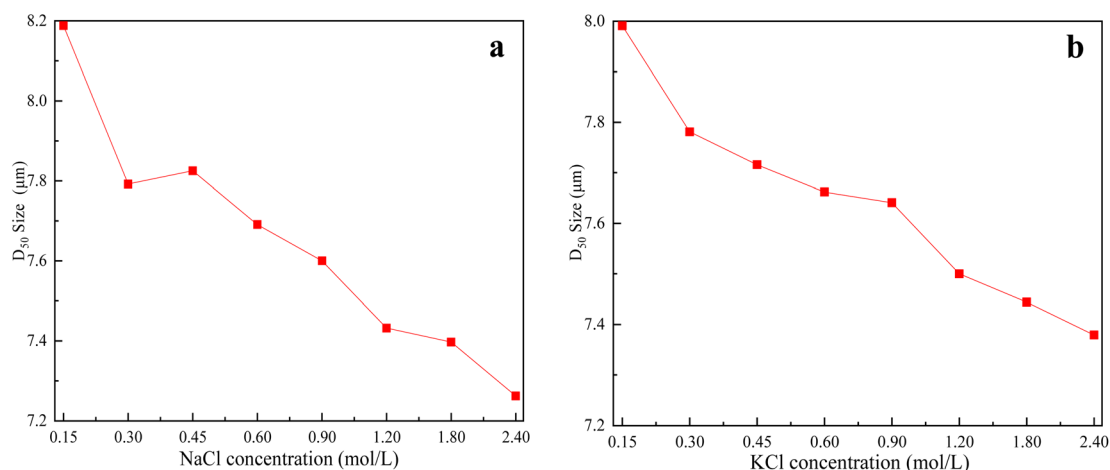


Fig. 5 D_{50} particle size variations of Mg(OH)₂ products at different Na⁺ and K⁺ concentrations ((a): Na⁺; (b): K⁺).

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

crystal surface. However, when K⁺ and Na⁺ concentration is low, the dissolution efficiency of Mg(OH)₂ decreases and the growth rate of the (001) crystal plane is lower than those of the (101) and

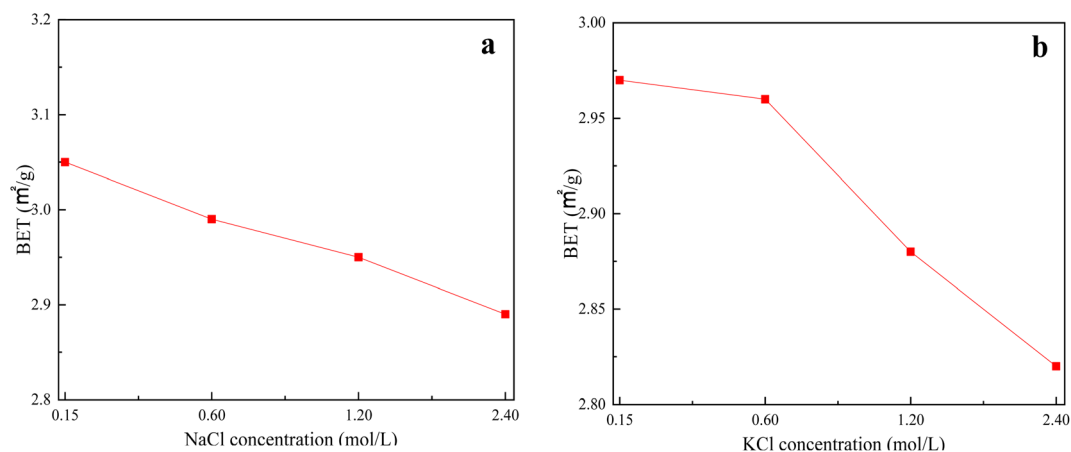


Fig. 6 BET changes of Mg(OH)₂ products at different Na⁺ and K⁺ concentrations ((a): Na⁺; (b): K⁺).



(110) crystal planes. When the crystal-surface-regulator concentration is high, excess Cl^- is introduced, which has a significant influence on the growth of the (001) crystal surface. We conclude that the concentrations of K^+ and 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 OH^- is smaller than that of the Cl^- in the precipitation system,²⁹ OH^- is more easily adsorbed onto the basic crystal plane of microcrystals and promotes the growth of particle edges. Thus, K^+ and Na^+ promote the growth of $\text{Mg}(\text{OH})_2$ edges by providing Cl^- and improve the growth of the (001) crystal surface.³⁰

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 Na^+ concentration in the reaction system increases from 0.15 to 2.40 mol L⁻¹, the particle size of the obtained $\text{Mg}(\text{OH})_2$ products increases from 8.18 μm dropped to 7.26 μm . Similarly, as shown in Fig. 5b, when the concentration of K^+ in the reaction system increases from 0.15 to 2.40 mol L⁻¹, the particle size of the obtained $\text{Mg}(\text{OH})_2$ products increases from 7.99 μm dropped to 7.38 μm . We conclude that the increasing concentration of K^+ and Na^+ leads to a decrease in the particle size of the obtained $\text{Mg}(\text{OH})_2$ products. Thus, the presence of K^+ and 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.

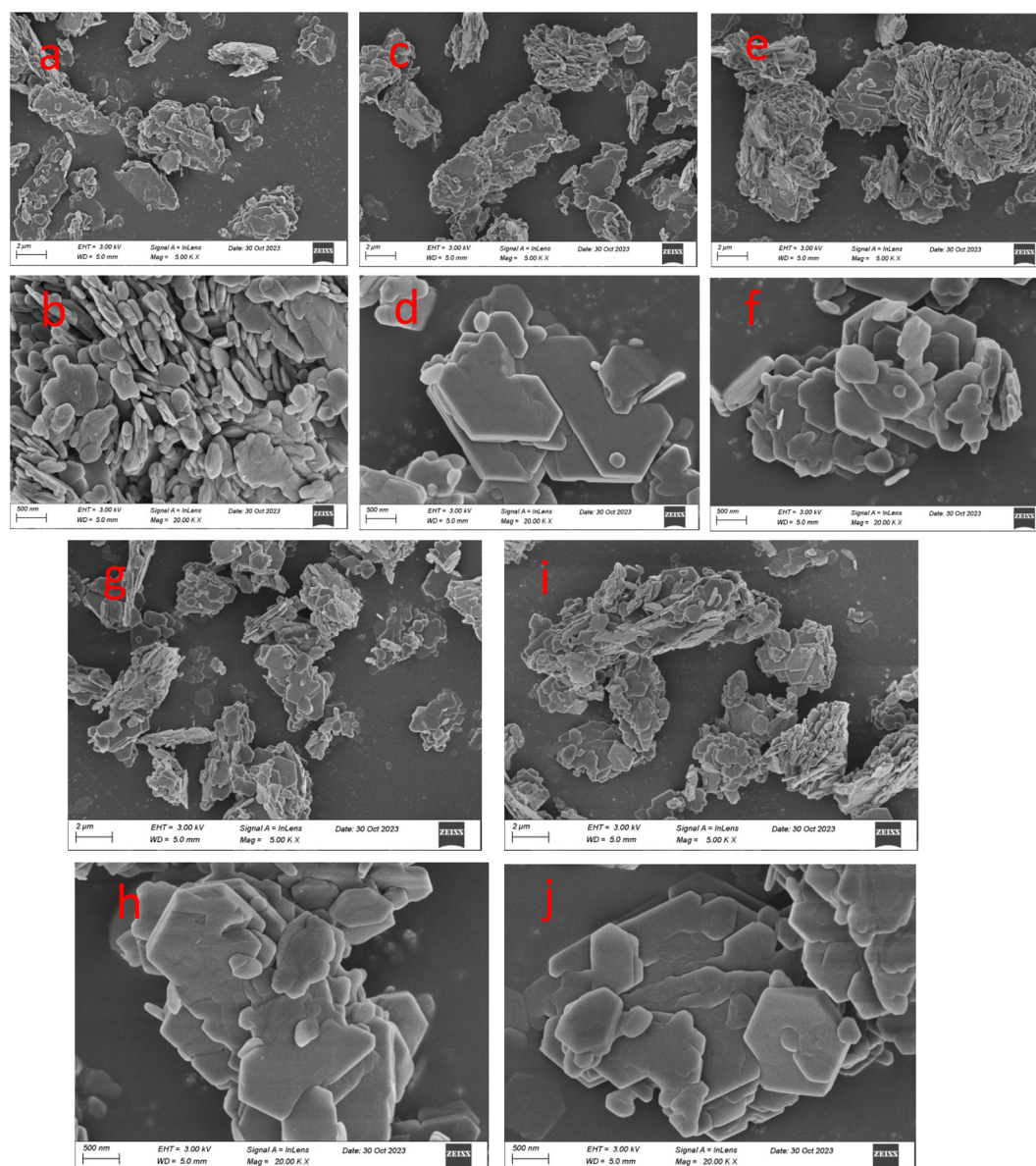


Fig. 7 Morphology of $\text{Mg}(\text{OH})_2$ products prepared using different concentrations of Na^+ (a and b): 0.15 mol L⁻¹; (c and d): 0.30 mol L⁻¹; (e and f): 0.60 mol L⁻¹; (g and h): 1.20 mol L⁻¹; (i, j and k): 2.40 mol L⁻¹.



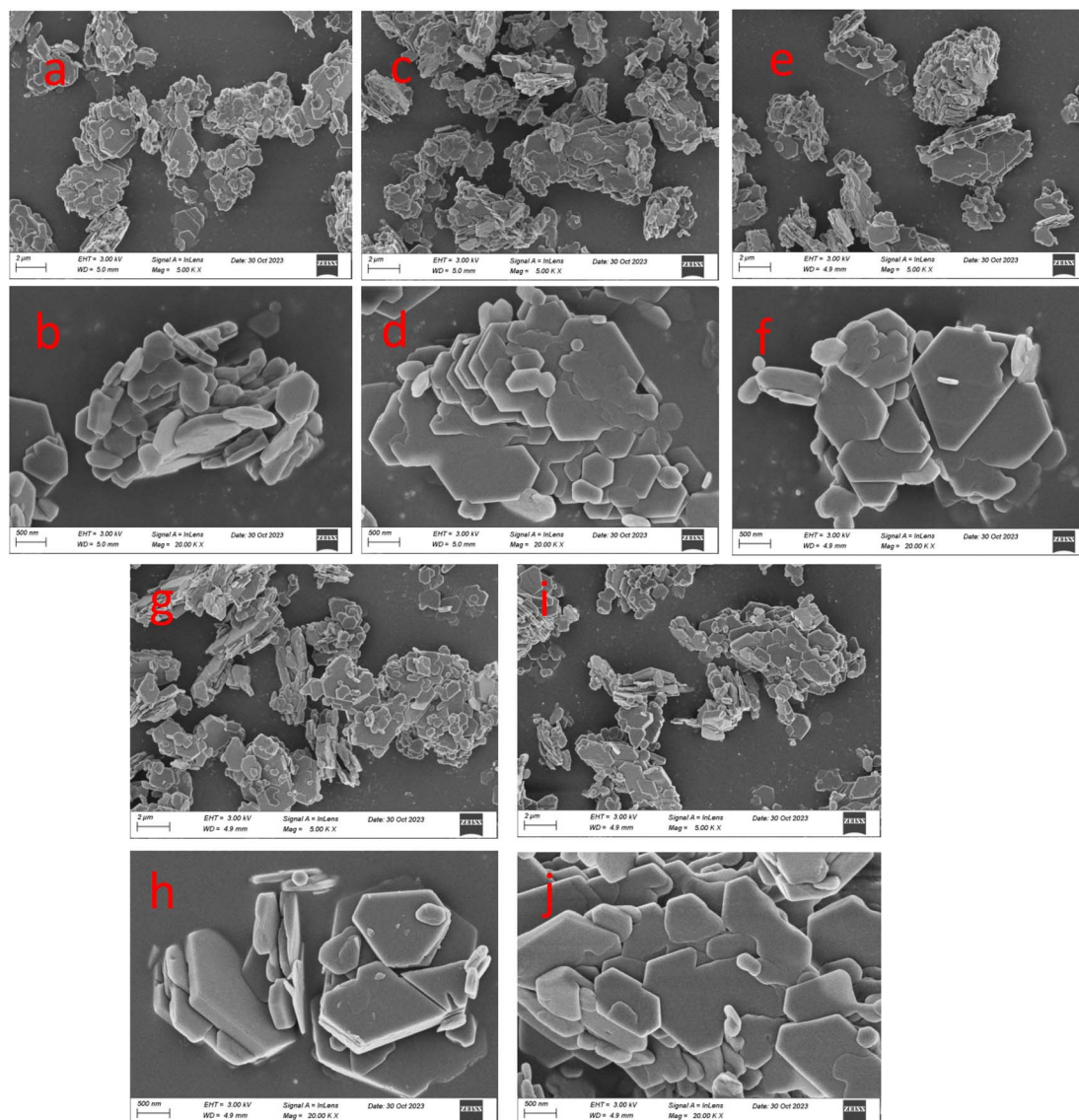


Fig. 8 Morphology of $\text{Mg}(\text{OH})_2$ products prepared using different K^+ concentrations ((a and b): 0.15 mol L^{-1} ; (c and d): 0.30 mol L^{-1} ; (e and f): 0.60 mol L^{-1} ; (g and h): 1.20 mol L^{-1} ; (i, j and k): 2.40 mol L^{-1}).

Fig. 6 shows the BET changes of $\text{Mg}(\text{OH})_2$ products obtained using different K^+ and Na^+ concentrations. With an increase in Na^+ concentration from 0.15 to 2.40 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 K^+ concentration from 0.15 to 2.40 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 K^+ and 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 K^+ and 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

K^+ and 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 K^+ and Na^+ play an important role 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 K^+ and Na^+ as crystal surface regulators on the hydrothermal dissolution and recrystallization of $\text{Mg}(\text{OH})_2$. The results show that the presence of K^+ and Na^+ has a significant effect on the crystal

structure, polarity, and particle size of the Mg(OH)₂ products. Under specific hydrothermal conditions, K⁺ and Na⁺ play a major role in controlling the selective growth of low-polarity crystal planes of Mg(OH)₂. The polarity ratio of the Mg(OH)₂ products prepared using Na⁺ decreased from the highest value of 1.19 to 0.83. When the concentration of NaCl in the reaction system increases from 0.15 to 2.40 mol L⁻¹, the particle size of the obtained Mg(OH)₂ products increases from 8.18 μm dropped to 7.26 μm. Similarly, an increase in the concentration of K⁺ in the reaction system from 0.15 to 2.40 mol L⁻¹ leads to an increase in the particle size of the obtained Mg(OH)₂ products from 7.99 μm dropped to 7.38 μm. K⁺ and Na⁺ promote the transformation of irregular large particles of Mg(OH)₂ products into regular hexagonal flakes under hydrothermal conditions. This study provides theoretical support for the preparation of hexagonal flakes of high-performance Mg(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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