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

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.

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1. Introduction

Mg(OH)₂ is nontoxic and nonpolluting and exhibits hightemperature 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 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), 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 lowpolarity 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.

Material and methods

2.1. Materials

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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 °C for 6 h, and the obtained Mg(OH)2 product was sealed for subsequent characterization and analysis.

Characterization analysis test

Several analytical techniques were used to characterize the obtaine powders.

X-ray powder Diffraction. The resulting powder crystalline phases were carried out using X-ray diffraction (XRD, DX-2700BH). A Cu K α 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.

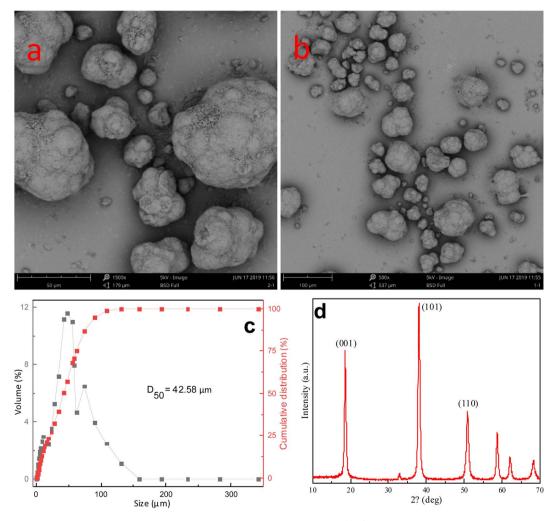


Fig. 1 Morphology, particle size distribution, and crystal phase composition of Mg(OH)₂ products produced by the magnesium industry in western Qinghai. ((a and b): SEM images; (c): particle size distribution and cumulative distribution; and (d): XRD pattern).

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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 Mg(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 $Mg(OH)_2$. Before the test, the samples are degassed overnight at 180 $^{\circ}$ C.

3. Results and discussion

The crystal phases and crystallinity of Mg(OH)2 were determined via X-ray diffraction. Fig. 2 shows the X-ray diffraction (XRD) pattern of Mg(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 Mg(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 Mg(OH)₂, respectively. The diffraction peak appears at 2θ value 58.66, corresponding to the Mg(OH)₂ crystal phase plane (110). The presence of Na⁺ has an impact on the strength of the obtained Mg(OH)2 crystal face. With the increasing content of Na⁺, the strength of the (001) crystal face of the obtained Mg(OH)2 product first increases and then decreases.

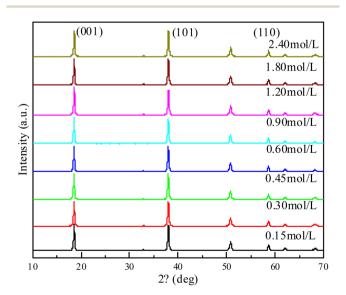


Fig. 2 $Mg(OH)_2$ products prepared using different Na⁺ concentrations (reaction time: 6 h; reaction temperature: 160 °C).

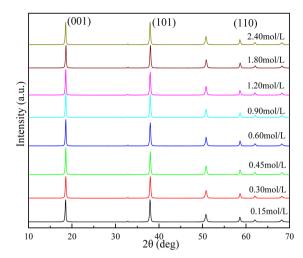


Fig. 3 $Mg(OH)_2$ products with different concentrations of K⁺ (reaction time: 6 h; reaction temperature: 160 °C).

The Mg(OH)2 crystal phases and crystallinity were determined by XRD. Fig. 3 shows the XRD pattern of Mg(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 Mg(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 Mg(OH)2, respectively. The diffraction peak appears at 2θ value 58.66, corresponding to the $Mg(OH)_2$ crystal phase plane (110). The presence of K^+ has an impact on the strength of the obtained Mg(OH)2 crystal face. With the increase in K⁺ content, the crystal face of the obtained Mg(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 Mg(OH)2, which is not conducive to the dissolution-crystallization of Mg²⁺ on the surface of Mg(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 Mg(OH)2 and positive charge on the surface of Mg(OH)2, Cl introduced by NaCl and KCl combines with Mg(OH)₂ more rapidly, further accelerating the dissolution of Mg(OH)2.28 Simultaneously, there is a higher number of impurity ions (Cl⁻) in the solution, which affects the ordered crystallization of Mg²⁺ on the crystal surface of Mg(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.

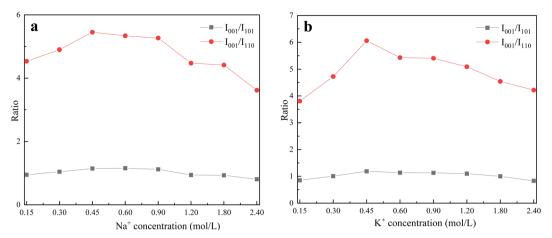


Fig. 4 XRD characteristic peak ratio of Mg(OH)₂ products at different Na⁺ and K⁺ concentrations ((a): Na⁺; (b): K⁺).

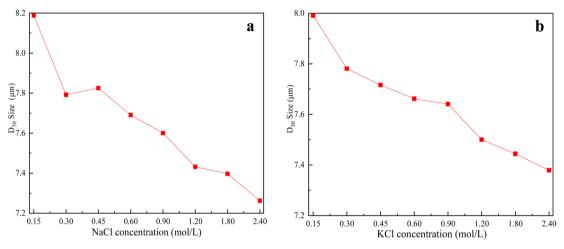


Fig. 5 D_{50} particle size variations of Mg(OH)₂ products at different Na⁺ and K⁺ concentrations ((a): Na⁺; (b): K⁺).

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

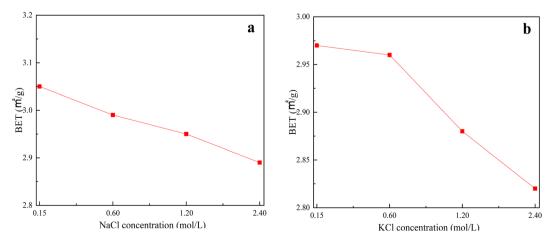


Fig. 6 BET changes of Mg(OH)₂ products at different Na⁺ and K⁺ concentrations ((a): Na⁺; (b): K⁺).

(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 $\mathrm{Mg}(\mathrm{OH})_2$ 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 $\mathrm{Mg}(\mathrm{OH})_2$ 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 $\mathrm{Mg}(\mathrm{OH})_2$ 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 Mg(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 Mg(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 Mg(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 Mg(OH)₂ products. Thus, the presence of K⁺ and Na⁺ has a significant effect on the dissolution and recrystallization of Mg(OH)₂, resulting in a continuous decrease in the particle size of the obtained products.

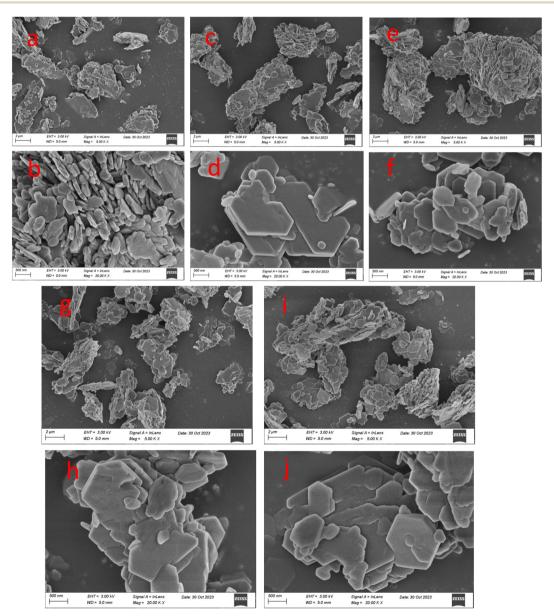


Fig. 7 Morphology of $Mg(OH)_2$ products prepared using different concentrations of Na^+ ((a and b): 0.15 mol L^{-1} ; (c and d): 0.30 mol L^{-1} ; (e and f): 0.60 mol L^{-1} ; (g and h): 1.20 mol L^{-1} ; (i, j and k): 2.40 mol L^{-1}).

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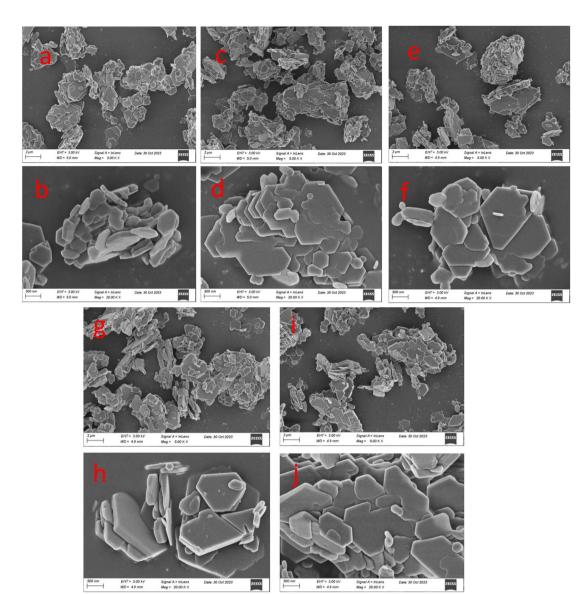


Fig. 8 Morphology of Mg(OH)₂ products prepared using different K⁺ concentrations ((a and b): 0.15 mol L⁻¹; (c and d): 0.30 mol L⁻¹; (e and f): 0.60 mol L⁻¹; (g and h): 1.20 mol L⁻¹; (i, j and k): 2.40 mol L⁻¹).

Fig. 6 shows the BET changes of $Mg(OH)_2$ 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 $Mg(OH)_2$ product decreases from 3.05 to 2.89 m² 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 $Mg(OH)_2$ product decreases from 2.97 to 2.82 m² g⁻¹ (Fig. 6b). The continuous increase of alkali metal ions K^+ and Na^+ causes the specific surface area of the obtained $Mg(OH)_2$ product to decreases continuously, albeit the decrease is small. This may be attributed to the small polarity of the obtained $Mg(OH)_2$ 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 $Mg(OH)_2$.

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

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

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