

Research on the influence of thermal decomposition of magnesium chloride hexahydrate on the preparation of magnesium oxide and hydrated magnesium hydroxide

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Abstract: Magnesium chloride hexahydrate is an important intermediate product in magnesite processing. In order to promote the efficient utilization of magnesite resources, based on the pyrolysis interval of magnesium chloride hexahydrate, the relationship between magnesium oxide with different physicochemical properties and the apparent properties of hydrated magnesium hydroxide was studied. The results show that the effect of temperature on magnesium oxide sintering is stronger than that of holding time. With the increase of calcination temperature and the extension of holding time of magnesium chloride hexahydrate, the calcined product magnesium oxide was sintered into large particle size with the characteristic particle size D_{50} of 33.89 μm . The crystal was distorted, the chemical activity deteriorated, and the color development time was up to 407 s. When hexahydrate magnesium chloride was calcined at 480 °C with 2 h, it decomposed almost completely. The product, magnesium oxide, consisted of uniformly distributed small coral rod-like particles with strong chemical reactivity and a color development time of 115 s. The particles were small and evenly distributed, with a characteristic particle size D_{50} of 1.36 μm , and the highest specific surface area reached 7.292 m^2/g . The hydrated magnesium hydroxide particles had well-defined edges and corners, with a characteristic particle size D_{50} of 1.59 μm and a uniform particle size distribution.

Keywords: magnesium chloride hexahydrate, pyrolysis, magnesium oxide, hydration, magnesium hydroxide

1. Introduction

Magnesite is an essential strategic mineral resource and a crucial raw material for the production of magnesian materials (Xu et al., 2022; Cao et al., 2023). It also finds extensive applications in magnesium metal, magnesite refractories, metallurgy, the chemical industry, and other related fields (Zhu et al., 2020; Zhu et al., 2022). At present, the proven reserves of magnesite in the world are about 12.625 billion tons, mainly distributed in China, North Korea, Russia, and other countries and regions. China has proven magnesite reserves of 3.642 billion tons, accounting for 29% of the world's total reserves, ranking first globally (Yang et al., 2022; Tan et al., 2023). Among them, Liaoning has the most abundant magnesite reserves, followed by Shandong. In addition, magnesite resources are also plentiful in Tibet, Xinjiang, Gansu, and other regions (Zhang et al., 2019; Ding et al., 2021). The main characteristics of China's magnesite resources include relatively concentrated reserves, large deposits, and magnesium oxide content generally ranging from 46% to 47% (Lin et al., 2021). However, over the years, extensive resource development has made low value-added magnesium products account for a high proportion, and the effective utilization of magnesite resources, preventing the full realization of the resource advantage as a sustainable industrial benefit (Gong et al., 2024).

Magnesite is a magnesium carbonate mineral with abundant reserves worldwide. During processing, it undergoes extraction and crystallization. Magnesium chloride hexahydrate is a crucial intermediate product in the processing of magnesite (Zan et al., 2023; Xie et al., 2024). Its capacity to be further processed into magnesium oxide, anhydrous magnesium chloride, and other products is essential for industrial production. However, the dehydration behavior of magnesium chloride hexahydrate varies under different pyrolysis conditions. This variability underscores the complexity and importance of magnesium chloride hexahydrate in the processing stages. Xu found that the heating rate was the main factor influencing the difference in dehydration temperature and behavior of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$. The variations in TG-DSC data at different heating rates affected the thermal dehydration characteristics of magnesium chloride hexahydrate (Xu et al., 2021). Zondag conducted a TG test on $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ at a heating rate of $1\text{ }^\circ\text{C}/\text{min}$, determined that the dehydration step from hexahydrate to tetrahydrate occurs at $70\text{ }^\circ\text{C}$ (Zondag et al., 2013). Whiting investigated a TG test on $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ at a heating rate of $2\text{ }^\circ\text{C}/\text{min}$. The initial material might be a mixture of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ and $\text{MgCl}_2 \cdot 4\text{H}_2\text{O}$, and the starting dehydration temperature ranges from 25 to $115\text{ }^\circ\text{C}$ (Whiting et al., 2014). Huang conducted the dehydration process of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ using TG-DSC testing at a heating rate of $5\text{ }^\circ\text{C}/\text{min}$. The results illustrated that $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ decomposed to $\text{MgCl}_2 \cdot 4\text{H}_2\text{O}$ at $69\text{ }^\circ\text{C}$ (Huang et al., 2011).

The properties of magnesium chloride hexahydrate prepared from magnesite determine the physicochemical properties of magnesium oxide. The thermal decomposition of magnesium chloride hexahydrate was controlled by adjusting the technological parameters of the pyrolysis conditions. In this paper, the heating rate of the magnesite intermediate product magnesium chloride hexahydrate was set at $10\text{ }^\circ\text{C}/\text{min}$. The study examines the impact of calcination temperature and time on the physicochemical properties of the resulting magnesium oxide product. It also delves into the mechanism by which the thermal decomposition process of magnesium chloride hexahydrate influences the physicochemical properties of magnesium oxide, as well as the characterization of hydrated magnesium hydroxide, found the preparation method for magnesium oxide with appropriate physical and chemical properties. These findings are of significant importance for improving the quality of magnesium oxide products and advancing technical development.

2. Experimental

2.1. Materials

Using magnesite from Liaoning calcined at $650\text{ }^\circ\text{C}$ and maintained for 4 h, followed by acid leaching, filtration, and drying at $105\text{ }^\circ\text{C}$ to prepare magnesium chloride hexahydrate as the raw material in Fig2. It can be seen from the multi-element analysis in Table 1 that the main component is Mg, with a content of 11.0386%. It also contains small amounts of Ca, K, and Na, as well as other trace elements. Hydrochloric acid was purchased from Tianjin Yongda Chemical Reagent Co., Ltd. Anhydrous ethanol and citric acid were also obtained from Tianjin Yongda Chemical Reagent Co., Ltd.

Table 1. Multielement analysis of magnesium chloride hexahydrate.

Composition	Mg	K	Na	Cl	SO4	Ca	Al	Fe
Wt/ %	11.0386	0.0025	0.0029	30.634	0.423	0.2854	<0.0005	<0.0005

The XRD patterns analysis is shown in Fig. 1

Fig. 1 is the XRD pattern of magnesium chloride hexahydrate. The characteristic diffraction peak is basically consistent with the $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ diffraction pattern on the standard card, with high intensity, which indicates that the magnesium chloride hexahydrate crystal has high purity and crystal integrity.

2.2. Preparation of magnesium oxide and hydrated magnesium hydroxide

The mass of raw material used is measured in grams, and through acid leaching with light burnt powder (g): hydrochloric acid (ml) = 0.32:1, stirring and drying obtained magnesium chloride hexahydrate crystals. By examining the endothermic and exothermic characteristics of magnesium chloride hexahydrate, the calcination temperature range was chosen to be between $480\text{ }^\circ\text{C}$ and $580\text{ }^\circ\text{C}$, and varying the duration of heat preservation produces the product magnesium oxide. Then, the suspension

was prepared using 4 g of magnesium oxide and anhydrous ethanol in a solid-to-liquid volume ratio of 1:15. The suspension was then pumped peristaltically into a beaker containing 400 mL of distilled water and placed in an ultrasonic device equipped with a heating function. An ultrasonic-assisted action of 300 W was applied at 80°C for 2 h to facilitate the hydration process and prepare magnesium hydroxide.

The experimental process is shown in Fig. 2.

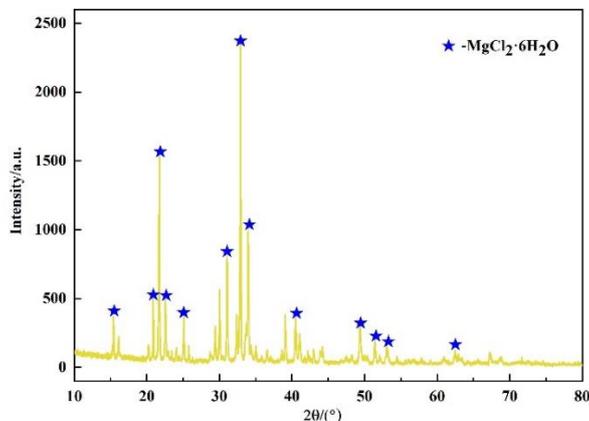


Fig. 1. XRD pattern of magnesium chloride hexahydrate

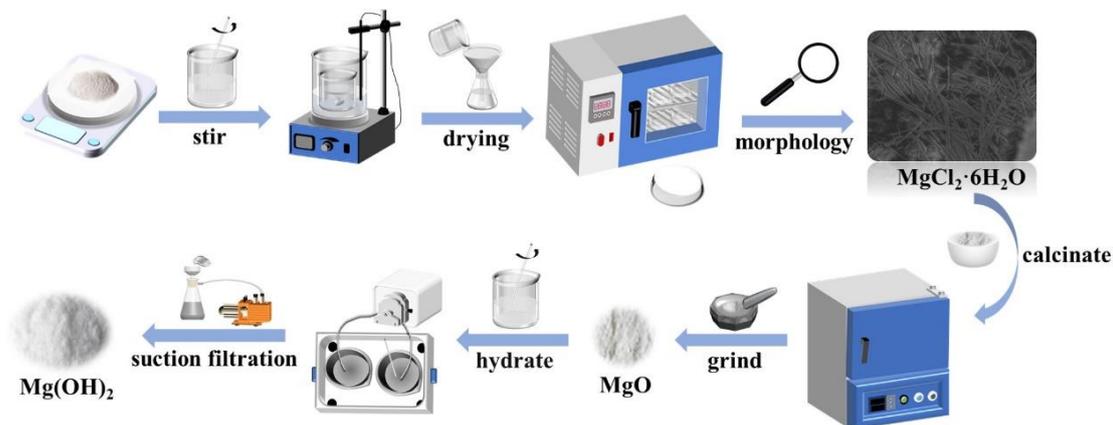


Fig. 2. Schematic diagram of experimental flow

2.3. Characterization of samples

Thermal stability and component analysis were conducted using a simultaneous thermal analyzer (model: STA449F3-DSC200F3) from Netzsch, Germany, with a temperature range of 25-800 °C. Morphological analysis was performed using a scanning electron microscope (model: Scios) manufactured by FEI, Czech Republic, operating at an acceleration voltage of 10 kV and magnifications ranging from 10,000 to 50,000. The lattice fringes and SAED were analyzed using a transmission electron microscope (Model: JEM2000EX) manufactured by Japan Electro-Optics Corporation. The specific surface area was measured using a fully automatic specific surface area tester (Model: JWGB-BK222). Particle size was determined using a fully automatic laser particle size analyzer, NKT6100-D, from HaixinRui Technology Co., Ltd. X-ray diffractometer (model: D/MAX2500PC) produced by RIGAKU Corporation, Japan, was used for phase analysis. The instrument utilized Cu K α radiation ($\lambda=1.54056\text{\AA}$) as the light source, with a scanning speed of 10°/min and an angular scanning range of 10°~80°.

3. Results and discussion

3.1. Thermal decomposition of magnesium chloride hexahydrate

Magnesium chloride hexahydrate consists of six H₂O molecules and two Cl⁻ ions surrounding the Mg atom, forming a centrally symmetric octahedron with a coordination number of six. Among the six water molecules, four have longer bond lengths and are more easily detachable, whereas the remaining

two water molecules are more firmly bound (Liu et al., 2005; Huang et al., 2010; Keishiro et al., 2019). Fig. 3 shows the thermogravimetric analysis curve of magnesium chloride hexahydrate crystals, with a total weight loss of 78.5%, which is basically consistent with the theoretical weight loss of 80.3% when magnesium chloride hexahydrate is fully pyrolyzed to magnesium oxide.

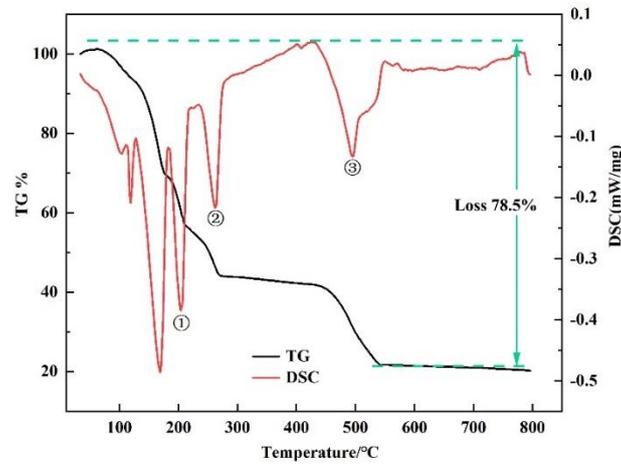


Fig. 3. TG-DSC curves of magnesium chloride hexahydrate

The preparation of magnesium oxide by the calcination of magnesium chloride hexahydrate involves three stages: dehydration, hydrolysis, and pyrolysis (Rammelberg et al., 2012; Zhao et al., 2022), as shown in Fig. 4. Combined with the thermogravimetric curve in Fig. 3, the main endothermic decomposition and mass loss processes of magnesium chloride hexahydrate crystals occur at around 200~400 °C and 470 °C. Beyond 470 °C, the thermal decomposition reaction stage aligns with the heat released during the full pyrolysis of Mg(OH)Cl into MgO. At 570 °C, the sample's mass stabilizes, and the decomposition ends.

The step ① is a series of dehydration reactions that occur within a temperature range of 50~220 °C, and $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ loses five H_2O molecules to form $\text{MgCl}_2 \cdot \text{H}_2\text{O}$. Step ② is a hydrolysis reaction, when the hydrolysis reaction occurs at the endothermic peak corresponding to the interval ①-② at 220~470 °C, $\text{MgCl}_2 \cdot \text{H}_2\text{O}$ is hydrolyzed to Mg(OH)Cl at this step. Simultaneously, it can be observed from the TG curve that the mass loss rate of the sample slows down after experiencing a sharp decline. This indicates that at this moment, there is not only water vapor loss but also some mass loss caused by HCl gas overflow. Step ③ is a decomposition reaction (releasing of HCl and MgO), it occurs at a temperature of 470 °C, which corresponds to the heat released by the pyrolysis of Mg(OH)Cl into MgO (Nejad et al., 2006). For the thermal decomposition process of magnesium chloride hexahydrate crystals, calcination was carried out at 480 °C and 580 °C, respectively. The study investigated the effects of calcination temperature and holding time on the decomposition of magnesium chloride hexahydrate and the physical and chemical properties of magnesium oxide.

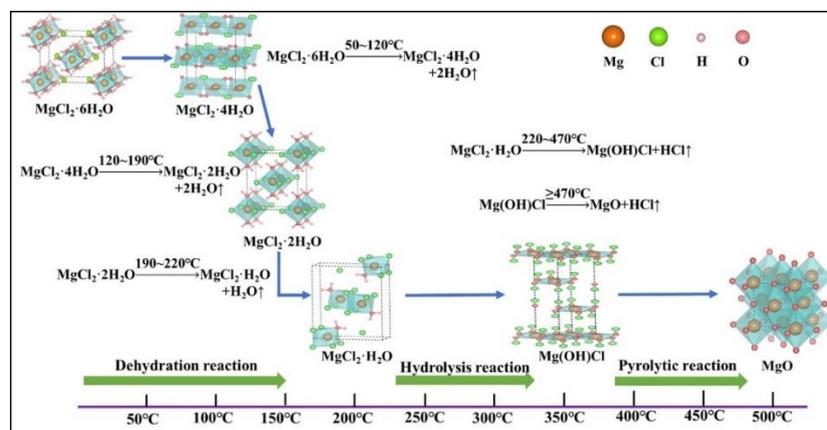
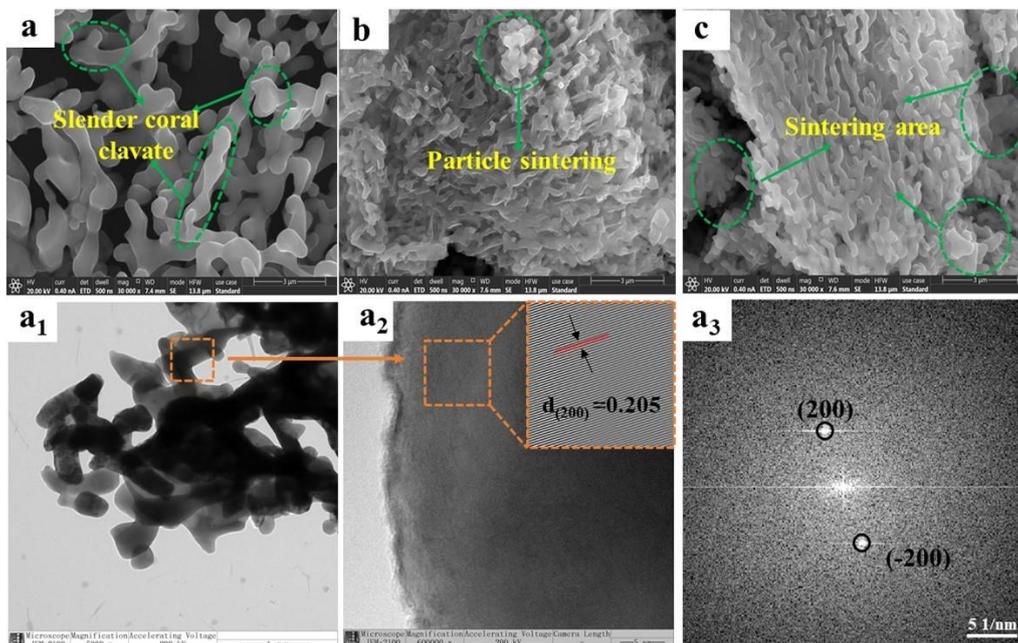


Fig. 4. Thermal decomposition process of magnesium chloride hexahydrate

3.2. Influence of calcination conditions of magnesium chloride hexahydrate on physicochemical properties of magnesium oxide

The morphology of the calcined products of magnesium chloride hexahydrate at 480 °C for different holding times are illustrated in Fig. 5. When held for 2 h (Fig. 5a), magnesium oxide fused at high temperatures into elongated coral-rod-like crystals with distinct gaps in the rod-like structure. After being heat preserved for 4 h, although magnesium oxide still maintained the coral-like structure, the crystal particle size increased, and the gaps between the rod-like crystals decreased (Fig. 5b). After being held at high temperatures for 6 h, the gaps between the magnesium oxide particles disappeared, gradually fusing into larger particles (Fig. 5c).

According to TEM analysis of the morphology of magnesium oxide in Fig. 5a, the main growth surface during the decomposition of magnesium chloride hexahydrate into magnesium oxide is the (200) surface. The calculated crystal surface spacing of lattice fringe corresponds to the (200) crystal surface of magnesium oxide, as depicted in Figures 5a₁-5a₃. This suggests that magnesium chloride hexahydrate has essentially decomposed after 2 h of heat preservation, and the obtained magnesium oxide has fully developed.



a - magnesium oxide morphology of held 2 h; b - magnesium oxide morphology after held for 4 h; c - magnesium oxide morphology after held for 6 h; a₁ - TEM morphology of held 2 h; a₂ - lattice fringe of held 2 h; a₃ - SAED of held 2 h

Fig. 5. Morphology characterization of magnesium chloride hexahydrate products at 480°C for different holding times

In order to determine whether the holding times affect the size and particle size distribution of magnesium oxide particles, laser particle size analysis is presented in Fig. 6. As the holding time increases, the particle size distribution curve of magnesium oxide shifts to the right overall. At a calcination temperature of 480 °C and held for 2 h, the particle size distribution of magnesium oxide is predominantly normal, ranging from 0.5 to 4 μm. At a particle size of 1.36 μm, the maximum volume content is 12.98%, indicating a fine particle classification. After extending the holding time to 4 h, the particle size distribution ranges from 0.6 to 20 μm, with an average particle size of 2.53 μm and a maximum volume content of 9.06%. When the heat was held for 6 h, the particle size distribution of magnesium oxide changed to two ranges, indicating that the coarse particle size of magnesium oxide particles began to occupy a certain volume distribution at this time, with the particle size range being 0.8 to 47.35 μm. As the characteristic particle size of D₅₀ gradually increases, it suggests that under the same calcination temperature and with the extension of holding time, the small magnesia particles start to fuse with each other into larger particles due to the high temperature.

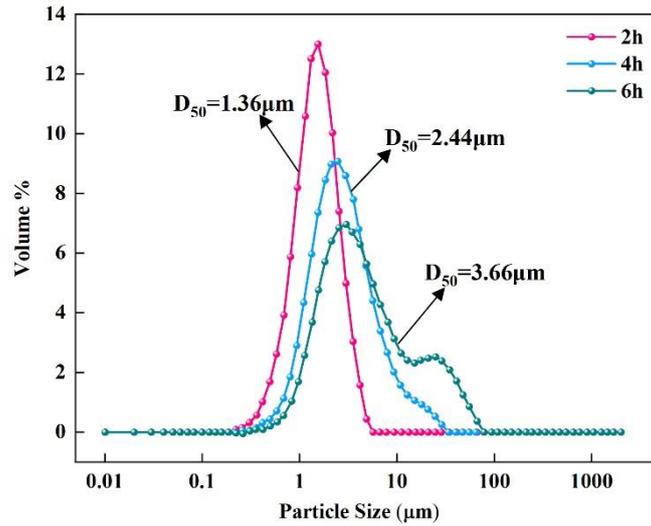


Fig. 6. Effect of calcination at 480°C for different holding times on particle size distribution of magnesium oxide

Fig. 7 shows the XRD patterns of the products of magnesium chloride hydrate for different holding times at a calcination temperature of 480°C. When held for 2 h, magnesium chloride hexahydrate has basically decomposed into magnesium oxide, with only a few magnesium chloride diffraction peaks remaining in the diffraction pattern. After 2 h, the diffraction peak of magnesia becomes increasingly intense as the holding times extended. As can be seen from Table 2, with the extension of holding time, the diffraction peak of the magnesium oxide (200) crystal surface becomes sharper, the half-peak width gradually decreases, the grain size of the sample also increases, and the crystallinity of the magnesium oxide crystal becomes more completed.

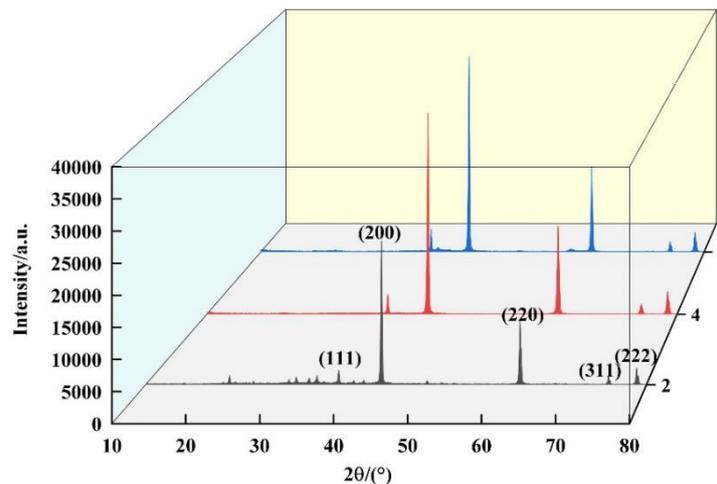
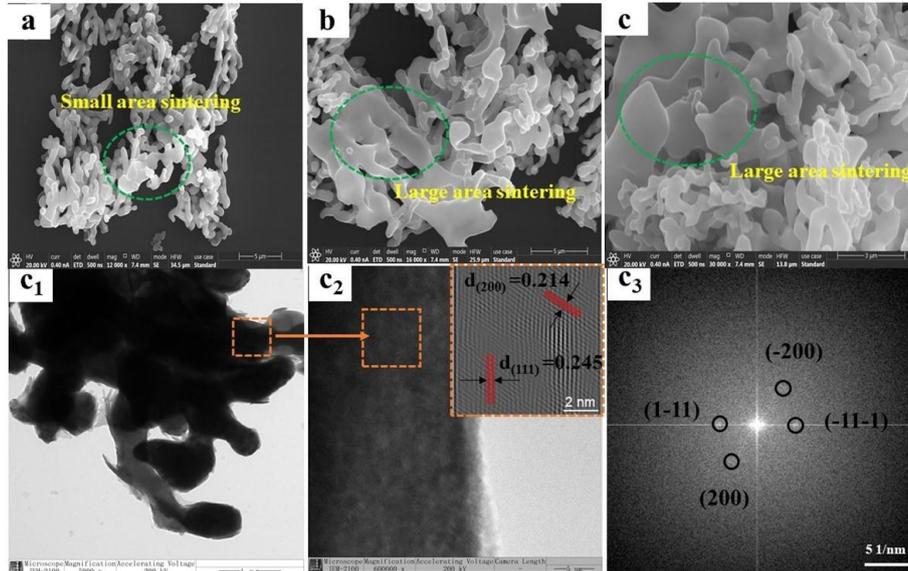


Fig. 7. XRD pattern of magnesium chloride hexahydrate at calcination temperature 480 °C for different holding times

Table 2. Influence of different calcination temperature and holding times on magnesium oxide (200) crystal surface

Temperature/°C	Holding time/h	FWMH	Crystallite Size/nm
480	2	0.242	34.93
	4	0.211	40.07
	6	0.199	42.49
580	1	0.204	41.44
	2	0.183	46.20
	4	0.164	51.55

Since magnesium chloride hexahydrate remained in the calcined products at the initial stage of 480 °C insulation, the calcination temperature was increased to 580 °C, and the insulation time was different. As depicted in Fig. 8, large-scale sintering and fusion into large pieces of magnesium oxide occurred at each holding time at the calcination temperature of 580 °C. With the extension of holding time, the elongated rod-like structure of magnesium oxide particles transformed into a large area of rock-like particles, and the degree of sintering was more severe than that at 480 °C.



a - magnesium oxide morphology of held 1 h; b - magnesium oxide morphology after held for 2 h; c - magnesium oxide morphology after held for 4 h; c₁ - TEM morphology of held 4 h; c₂ - lattice fringe of held 4 h; c₃ - SAED of held 4 h

Fig. 8. Morphology characterization of magnesium chloride hexahydrate products at 480°C for different holding times

According to the laser particle size analysis in Fig. 9, the particle size distribution curve of the magnesium oxide product produced at each holding time at 580 °C exhibited two distinct particle size ranges: fine particle grade and coarse particle grade. With the extension of holding time, the entire curve shifted to the right, transitioning the distribution from fine to coarse particle sizes, and the characteristic particle size D_{50} gradually increased. In comparison to the calcination temperature of 480 °C, at a holding temperature of 580 °C for 1h, the curve began to exhibit two ranges, with the volume proportion of coarse particles increasing as the holding time extended. This suggests that some magnesium oxide particles start to merge into larger particles under high temperatures, indicating that temperature has a more pronounced effect on the sintering of magnesium oxide than the holding time.

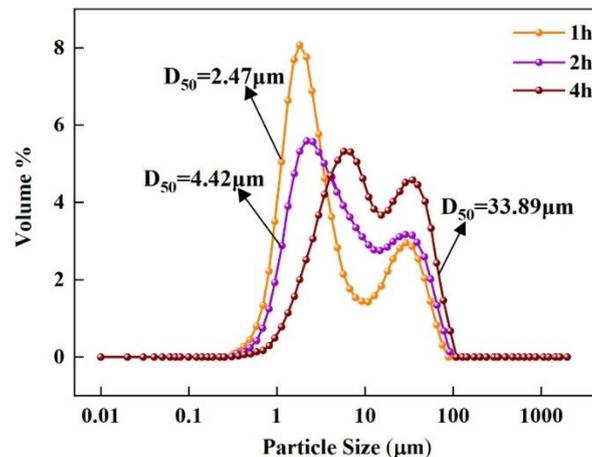


Fig. 9. Effect of calcination at 580 °C for different holding times on particle size distribution of magnesium oxide

The XRD patterns of magnesium chloride hexahydrate at a calcination temperature of 580 °C for different holding times are illustrated in Fig. 10, with the resulting products being magnesium oxide. Table 2 demonstrates that as the holding time increases, the diffraction peak of magnesium oxide becomes sharper, the grain size increases, and the crystallinity improves.

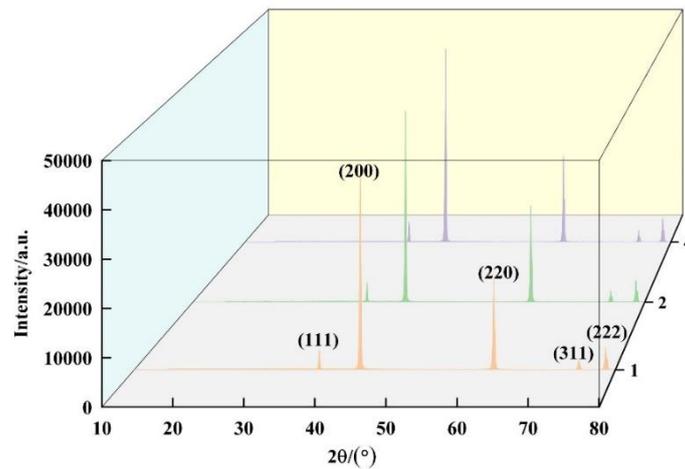


Fig. 10. XRD pattern of magnesium chloride hexahydrate at calcination temperature 580 °C for different holding time

The color development of magnesium oxide at different calcination temperatures and holding times are illustrated in Figure 11. When calcined and held at 480 °C for 2 h, the shortest color development time was 115 s. With the extension of holding time, the color development time gradually lengthened, reaching 242 s after 6 h. The findings suggest that prolonging the holding time results in increased magnesium oxide particle size, grain size, and complete crystallinity, leading to worsened distortion and ultimately decreased magnesium oxide activity. At 580 °C, the overall activity of calcined magnesium oxide decreased compared to 480 °C, indicating that the impact of calcination temperature on magnesium oxide decomposition is significantly more pronounced than that of holding time.

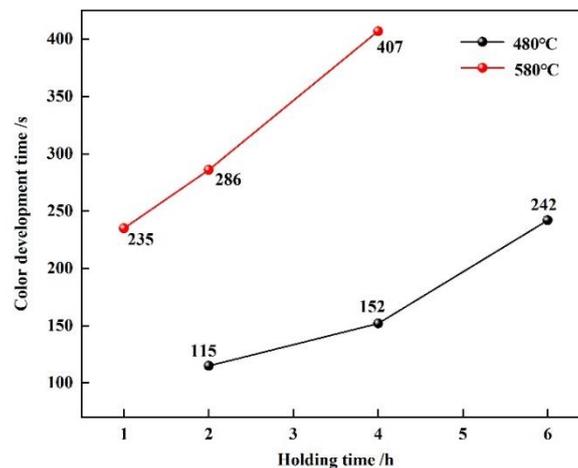


Fig. 11. Color development time of magnesium oxide produced by magnesium chloride hexahydrate at different calcination temperature and holding times

In addition, the variations in the specific surface area of magnesium oxide products at different calcination temperatures and holding times are presented in Table 3. At a calcination temperature of 480 °C, the specific surface area of magnesium oxide decreases as the holding time extends, from 7.292 m²/g for 2 h to 2.974 m²/g for 6 h. The decrease in specific surface area indicates a gradual reduction in the pores between particles. This is further supported by the change in magnesium oxide morphology shown in Fig. 5, where the gap distance between small magnesium oxide particles decreases as the holding time increases due to the influence of high temperature, leading to the gradual sintering of bulk

magnesium oxide particles. At a calcination temperature of 580 °C, the specific surface area of magnesium oxide also decreases with longer holding times, but the decline is less pronounced compared to that at 480 °C.

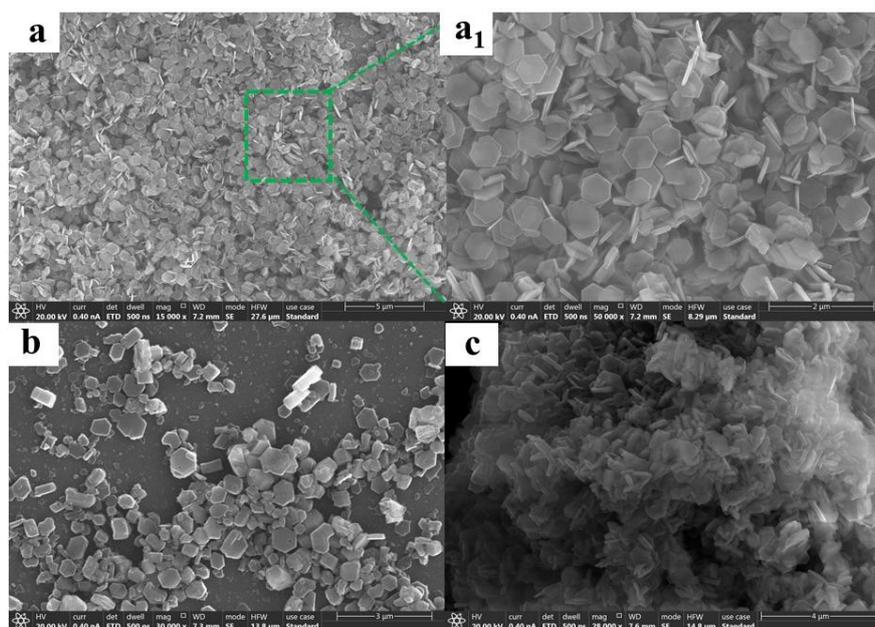
Table 3. Specific surface area of magnesium oxide produced by magnesium chloride hexahydrate at different temperatures and holding times

Temperature/°C	Holding time/h	Specific surface area/m ² ·g ⁻¹
480	2	7.292
	4	4.542
	6	2.974
	1	3.857
580	2	2.622
	4	1.778

When subjected to two different calcination temperatures, the specific surface area and morphology of magnesium oxide are positively correlated. The specific surface area of the sample is larger when the morphology consists of uniformly distributed coral-like particles. Conversely, the specific surface area of large sintered magnesium oxide is relatively smaller. Therefore, when the difference in morphology is not significant, the variation in the specific surface area of magnesium oxide is relatively small.

3.3. Influence of calcination conditions of magnesium chloride hexahydrate on hydration products of magnesium oxide

The morphology of magnesium hydroxide, prepared by hydration after producing magnesium oxide from magnesium chloride hexahydrate at 480 °C, is shown in Fig. 12. The morphology of magnesium hydroxide prepared by hydration of the magnesium oxide product at 480 °C for 2 h were mostly exhibited regular hexagonal flake structures with angular edges, uniform particle size, and good dispersion (Fig. 12a-a₁). When the magnesium oxide product was held for 4 h, the thickness of the hydration product layer increased significantly, resulting in an extremely uneven distribution and partial agglomeration phenomenon (Fig. 12b). The particle size distribution of the hydrated magnesium hydroxide obtained after being held for 6h was also uneven (Fig. 12c).



a -magnesium hydroxide morphology of held 2 h; a₁ - Partial magnification of magnesium hydroxide; b - magnesium hydroxide morphology after held for 4 h; c - magnesium hydroxide morphology after held for 6 h

Fig. 12. The morphology of magnesium hydroxide prepared by the hydration of the product magnesium oxide at 480 °C

Fig. 13 shows the particle size distribution curve of hydrated magnesium hydroxide, shifting to the right as a whole. With the extension of holding time, the particle size of the hydration product increases overall. When held for 2 h, the particle size distribution curve of magnesium hydroxide formed a narrow normal distribution curve, with the characteristic particle size $D_{50}=1.59\ \mu\text{m}$. The proportion of particles with a size $<2\ \mu\text{m}$ is 79.42%. The particle size distribution was uniform, and the dispersion was good. The characteristic particle size D_{50} of magnesium hydroxide increased to $2.08\ \mu\text{m}$ after being held for 4h, which was caused by the thickening of hexagonal layers and partial agglomeration of particles. Additionally, the proportion of particles smaller than $2\ \mu\text{m}$ decreased to 43.65%. In the size distribution of magnesium hydroxide held for 6 h, the coarse particles ($>3\ \mu\text{m}$) accounted for 60.32%, while fine particles ($<2\ \mu\text{m}$) only made up 19.54%, suggesting an uneven particle size distribution. According to the index requirements of magnesium hydroxide HG/T 4531-2013 for flame retardants ($D_{50} \leq 5.00\ \mu\text{m}$) (Zeng et al., 2024). The variations in particle size of the prepared magnesium hydroxide meet the requirements for flame retardants, demonstrating its suitability for these applications.

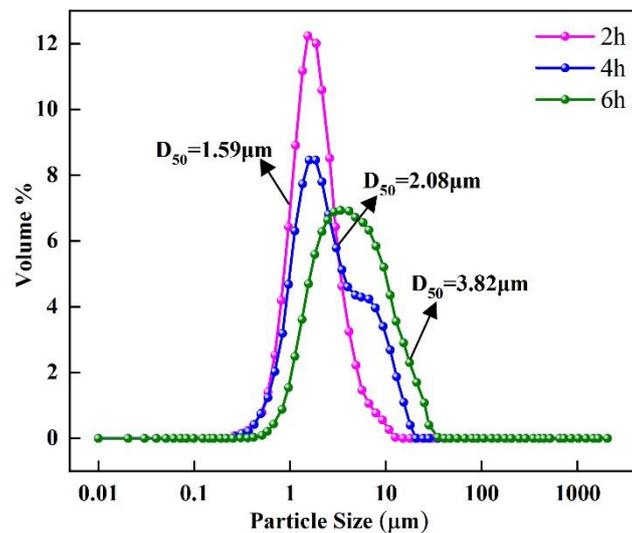


Fig. 13. Particle size distribution of magnesium hydroxide prepared from magnesium oxide by hydration of magnesium chloride hexahydrate at $480\ \text{°C}$

In magnesium hydroxide crystals, the surface energy of the (001) and (101) crystal surfaces are relatively low, resulting in low growth rates. These surfaces are stable and are identified as the main growth crystal surfaces of magnesium hydroxide (Wang et al., 2024). According to the characteristic analysis of the hexagonal crystal system of magnesium hydroxide, the surface energy is low, and the polarity is weak because the (001) crystal surface is exposed to the face of the ligand. And the (101) crystal surface is exposed by the vertex of the ligand, an increase in the upper surface of the (101) crystal surface and resulting in higher polarity. The (001) crystal surface is a non-polar plane, while the (101) crystal surface is a polar plane. The surface polarity of magnesium hydroxide can be characterized by the intensity ratio of the I_{001} and I_{101} diffraction peaks (Tang et al., 2020; Bhatt et al., 2021; Li et al., 2022).

The XRD pattern of magnesium hydroxide, obtained by hydration magnesium oxide prepared from magnesium chloride hexahydrate at $480\ \text{°C}$, is depicted in Fig. 14. The ratio of the weak polar crystal surface (001) to the strong polar crystal surface (101) of magnesium hydroxide first increases and then decreases. The maximum value was 0.983 at $480\ \text{°C}$ for 2 h and decreased to 0.777 at 6 h. In addition, the XRD of the hydrated product magnesium hydroxide for 4 h and 6 h revealed an incomplete hydrated magnesium oxide hybrid peak, and the I_{001}/I_{101} ratio gradually decreased. This suggests that the growth of the strongly polar crystal surface (101) is more prominent, the layer of hexagonal sheets becomes thicker, the overall polarity of magnesium hydroxide increases, and the adsorption force between particles is strengthened.

Fig. 15 shows the morphological characterization of magnesium hydroxide prepared through the hydration of magnesium oxide produced from magnesium chloride hexahydrate at $580\ \text{°C}$ for different holding times. The morphology of magnesium hydroxide prepared by hydration of the magnesium

oxide product at 580 °C for 1 h were irregular hexagonal flake structures, the particle size distribution is not uniform, and some of them are agglomerated into large particle aggregates (Fig. 15a). The magnesium hydroxide, prepared by hydration of the magnesia produced by holding at 580 °C for 2 h became thicker, and the agglomeration phenomenon became more serious (Fig. 15b). Large particles appear in magnesium hydroxide prepared by hydration of the magnesium oxide product at 580 °C for 4 h. And there are small particles of magnesium hydroxide flake particle aggregates on the surface (Fig. 15c).

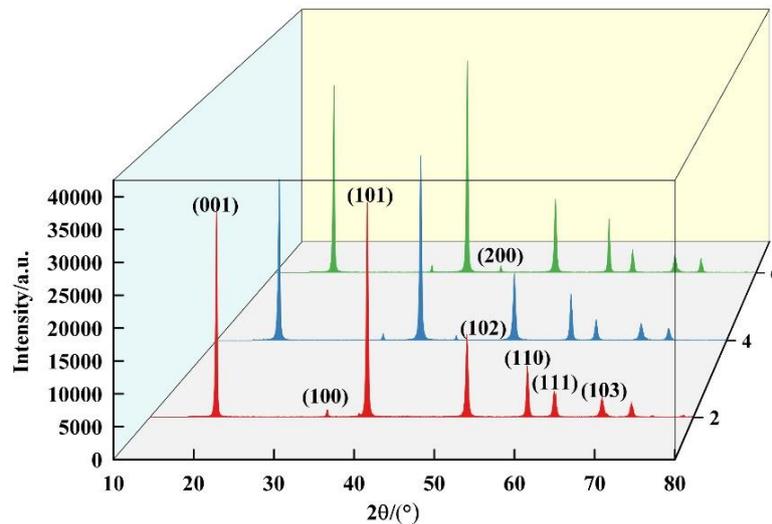
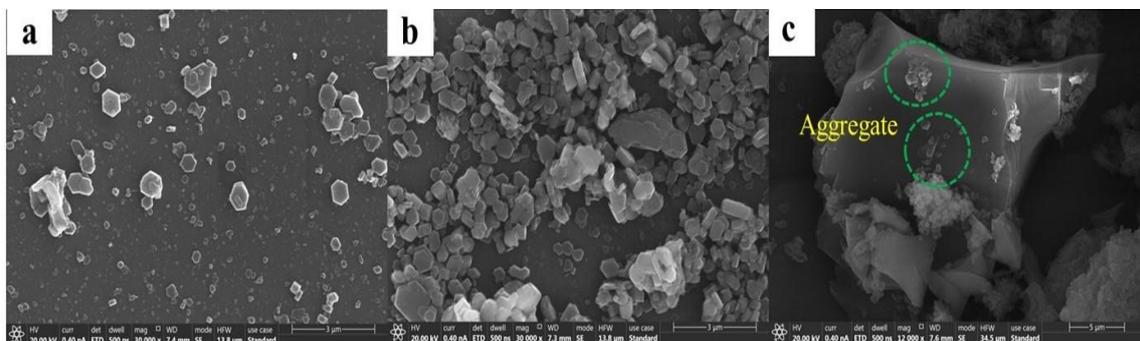


Fig. 14. XRD pattern of magnesium hydroxide prepared by hydration of magnesium oxide at 480 °C with magnesium chloride hexahydrate



a -magnesium hydroxide morphology of held 1 h; b - magnesium hydroxide morphology after held for 2 h; c - magnesium hydroxide morphology after held for 4 h

Fig. 15. The morphology of magnesium hydroxide prepared by the hydration of the product magnesium oxide at 580 °C

The particle size distribution of magnesium hydroxide prepared through hydration under different holding times at 580 °C is shown in Fig. 16. As the holding time is extended, the particle size distribution curve obtained through hydration typically shifts to the right. When held for 1h, the characteristic particle size of magnesium hydroxide was $D_{50}=2.93 \mu\text{m}$, and two distribution peaks appeared in the curve, ranging from 1-10 μm and 10-40 μm . The distribution of particles with a size $<2 \mu\text{m}$ was 21.15%, while the proportion of larger particles with a size $>5 \mu\text{m}$ was 33.69%, indicating an uneven particle size distribution. When held for 2 h, the characteristic particle size of magnesium hydroxide was $D_{50}=3.66 \mu\text{m}$, with dominant particle size distribution ranges of 1-15 μm and 15-45 μm . The proportion of particles smaller than 2 μm was 15.38%, while particles larger than 5 μm accounted for 38.83%. The distribution was more uneven. The characteristic particle size D_{50} of magnesium hydroxide increased to 5.39 μm after 4 h of incubation. The proportion of particles $<2 \mu\text{m}$ was 3.37%, the proportion of particles $>5 \mu\text{m}$ was 63.68%, and the distribution of super-large particles $>20 \mu\text{m}$ was 4.45%.

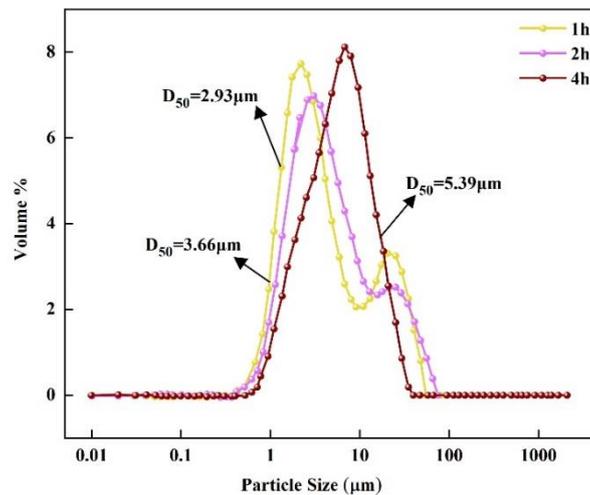


Fig. 16. Particle size distribution of magnesium hydroxide prepared from magnesium oxide by hydration of magnesium chloride hexahydrate at 580 °C

The XRD pattern of magnesium hydroxide, obtained by hydration magnesium oxide prepared from magnesium chloride hexahydrate at 580 °C, is depicted in Fig. 17. The diffraction peak of magnesium oxide appeared at different times during holding at 580 °C, and the prepared magnesium oxide could not be completely hydrated and converted into magnesium hydroxide. The (200) crystal surface diffraction peak of magnesium oxide appears for the first time in the XRD peak obtained by hydration of magnesium oxide prepared at 580 °C for 1 h. With the increase in holding time, the diffraction peak intensity of the unconverted magnesium oxide (200) crystal surface in the XRD pattern of the hydrated product magnesium hydroxide becomes more pronounced. Additionally, the (311) and (222) crystal surfaces of the unconverted magnesium oxide emerge after a 2 h holding period.

The peak intensity of the miscellaneous peak (200) crystal surface of magnesium oxide hydrate obtained at 580 °C for 4 h was the highest. The physicochemical properties of magnesium oxide deteriorated with prolonged holding time, preventing the complete conversion of magnesium oxide into magnesium hydroxide.

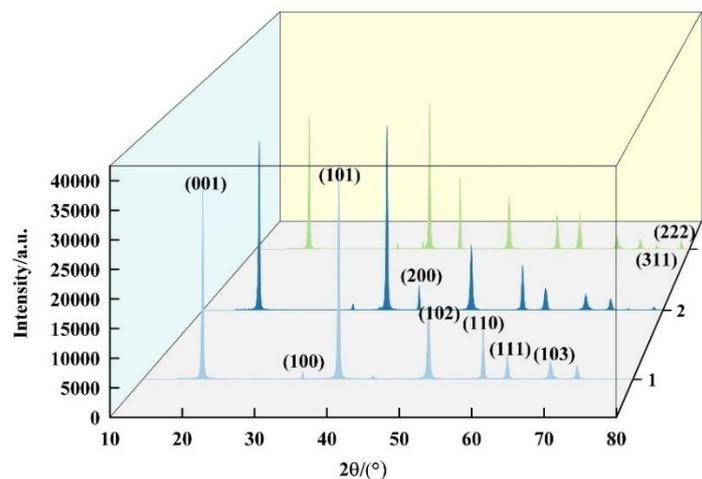


Fig. 17. XRD pattern of magnesium hydroxide prepared by hydration of product magnesium oxide with magnesium chloride hexahydrate at 580 °C

4. Conclusions

The pyrolysis interval based on magnesium chloride hexahydrate significantly affects the physicochemical properties of magnesium oxide. Magnesium chloride hexahydrate prolong the holding time at higher calcination temperature, which makes the decomposition process significantly rapid, and

the calcination product is completely decomposed into magnesium oxide. When the calcination temperature reaches 580°C and the holding time is extended, the thermal decomposition of magnesium chloride hexahydrate is accelerated, resulting in its complete conversion into magnesium oxide. Prolonging the holding time makes the grain size of magnesium oxide larger, the crystallinity of the crystal more complete, the degree of distortion worse, small particles sintered into larger pieces of magnesium oxide, the particle size increases and the distribution is uneven, the specific area of the sample becomes smaller, and the chemical activity of magnesium oxide becomes worse. When magnesium chloride hexahydrate was maintained at 480 °C for 2 h, it underwent complete decomposition into coral-rod shapes. The color development time was 115 s, and the chemical activity was strong. The characteristic particle size D_{50} was 1.36 μm , and the maximum specific surface area was 7.292 m^2/g .

The calcined product, magnesium oxide, mainly affects the relative growth habits of hydrated magnesium hydroxide crystals (001) and (101) through their physical and chemical properties, which helps in directing the growth of magnesium hydroxide crystals. The morphology and crystal structure of hydrated magnesium hydroxide differ. Magnesium oxide with excellent physical and chemical properties can notably boost crystal growth on the (001) crystal surface during hydration, making the hexagonal flakes of crystal particles, a more stable crystal structure, and improved particle size distribution and dispersion. Future studies could further investigate the crystal growth habits to more comprehensively understand the role and mechanism of magnesium oxide in promoting the growth of hydrated magnesium hydroxide crystals. Additionally, the promoting effect of magnesium hydroxide on the growth of these crystals can be improved by regulating microstructure and surface properties. This approach aims to achieve finer and more regular crystal morphology and higher crystallinity, thereby expanding potential applications in fields such as catalysis, adsorption, and as fillers. Ultimately, this research seeks to provide innovative materials for industrial production and environmental protection.

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