

Article

Removal of Chloride Ions from $\text{Mg}(\text{OH})_2$ Using NaOH with Ethanol

Yujie Zhao ¹, Junfeng Cheng ¹, Weiping Liu ^{1,2,*}, Jinhui Li ², Fang Wu ^{2,3}, Wei Sun ¹ and Shafiq Alam ⁴¹ School of Minerals Processing and Bioengineering, Central South University, Changsha 410083, China² Key Laboratory of Ionic Rare Earth Resources and Environment, Ministry of Natural Resources, Ganzhou 341000, China³ School of Material Engineering, Jiangxi College of Applied Technology, Ganzhou 341000, China⁴ Department of Chemical and Biological Engineering, College of Engineering, University of Saskatchewan, Saskatoon, SK S7N 5A9, Canada

* Correspondence: wliu.met@csu.edu.cn

Abstract: China's salt lake-based magnesium industry faces chloride removal challenges, which limit applications and cause pollution. Addressing this enables higher-quality $\text{Mg}(\text{OH})_2$ production. In this regard, chloride ion removal from magnesium hydroxide in a sodium hydroxide solution with ethanol was investigated for the first time. Ethanol and NaOH enhance chloride removal efficiency. Under optimized conditions, the chloride removal efficiency was 68.9%, and the Cl^- content in $\text{Mg}(\text{OH})_2$ was reduced to 0.14%. Kinetic analysis suggests that the removal of chloride ions is primarily controlled by internal diffusion. These results underscore the potential of NaOH and ethanol in enhancing chloride removal efficiency during magnesium hydroxide production, supporting the advancement of cleaner and more versatile magnesium-based materials.

Keywords: chloride ions; ethanol; magnesium hydroxide; kinetics; sodium hydroxide



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1. Introduction

High-purity magnesium hydroxide is essential for producing various magnesium compounds [1]. It finds extensive applications in industries such as steel [2], nonferrous metallurgy [3], chemicals [4], plastics [5], rubber [6], electronics [7], pharmaceuticals [8], food [9], and environmental protection [10], offering significant market potential [11] (Figure 1). High-purity magnesium hydroxide is primarily produced from magnesite and bischofite. The depletion of magnesite resources in Liaoning, China, reduces competitiveness. This creates an opportunity to produce high-purity magnesium hydroxide from Qinghai salt lake brine [12].

The Zarkhan Salt Lake in Qinghai, China, holds 6.5 billion tons of magnesium, but excessive magnesium chloride brine discharge during potassium extraction has caused salt accumulation [13], reducing potassium concentration [14] and increasing extraction costs [15]. The region's favorable evaporation conditions enable cost-effective production of high-quality bischofite [16,17]. Given the large amount of bischofite byproduct and increasing demand for magnesium products, large-scale production of magnesium hydroxide and downstream products offers a viable solution [18,19]. Research has focused on magnesium hydroxide [20], oxide [21,22], and related products [23].

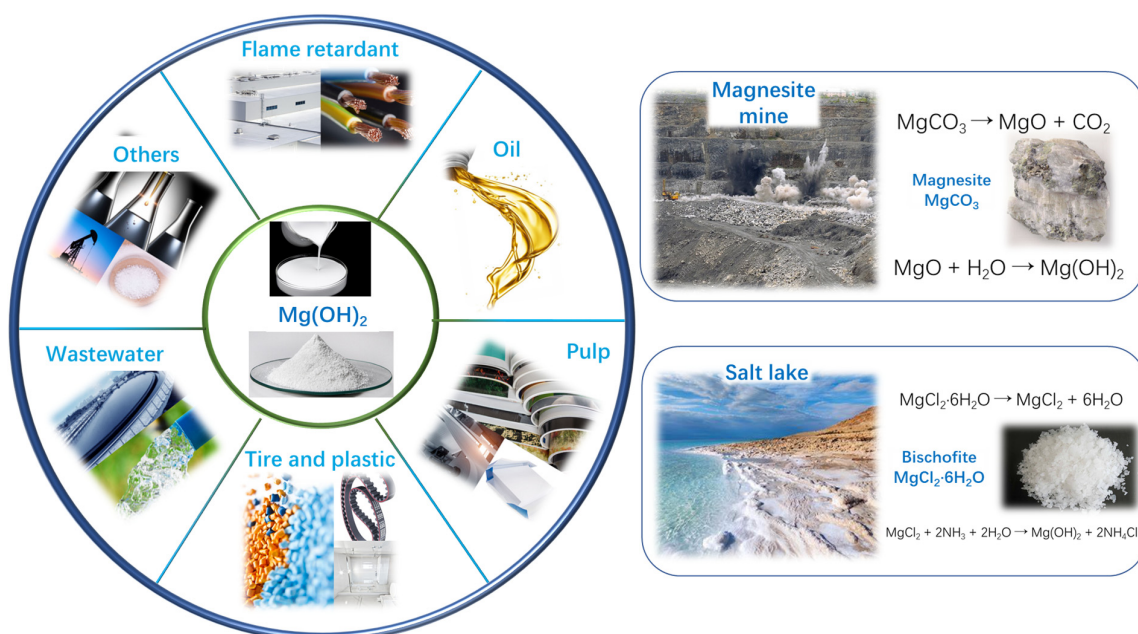


Figure 1. Sources and applications of magnesium hydroxide.

Qinghai Salt Lake enterprises established a 150,000 ton/year high-purity magnesium hydroxide line in Delingha, China, using a Central South University-patented ammonia/lime co-precipitation method [24,25]. However, this process results in a chloride content of 0.3–0.8% [26], mainly due to the ineffective control of initial crystal morphology and the growth rate during the large-scale precipitation process. The resulting magnesium hydroxide forms spherical particles that continuously agglomerate, trapping a large number of chloride ions within the spherical particles. Subsequent filtration and washing processes are unable to reduce the chloride ion content to less than 0.3%. The infiltration of chloride ions not only affects the crystallization, growth, morphology, and particle size of magnesium hydroxide crystals but also impacts the application performance of magnesium hydroxide in flame retardants, neutralizing agents, and pharmaceuticals. For instance, as shown in Figure 2, during the calcination process of magnesium hydroxide, high chloride ion content can generate chlorine gas. The combination of chlorine gas and water molecules produces hydrogen chloride gas, which not only severely corrodes production equipment but also pollutes the environment and affects the health of employees. For example, chloride ions present in magnesium hydroxide are known to facilitate the corrosion of metallic materials [27], including black-chromium-plated plastics [28]. The presence of chlorine ions significantly accelerates the aging processes of plastic substrates [29], which may lead to electrochemical migration and subsequent corrosion failures in printed circuit board components [30]. Consequently, this degradation adversely impacts the overall performance of the materials involved [31]. As shown in Table 1, magnesium hydroxide intended for flame retardant (MC-2-15) necessitates a chloride ion content of less than at least 0.15% [32]. Conversely, magnesium hydroxide designated for industrial applications [33] and nanopowder [34] requires a chloride ion content of less than at least 0.50%. Developing efficient chloride removal methods is critical for the application of Mg(OH)_2 in areas with stringent chloride requirements.

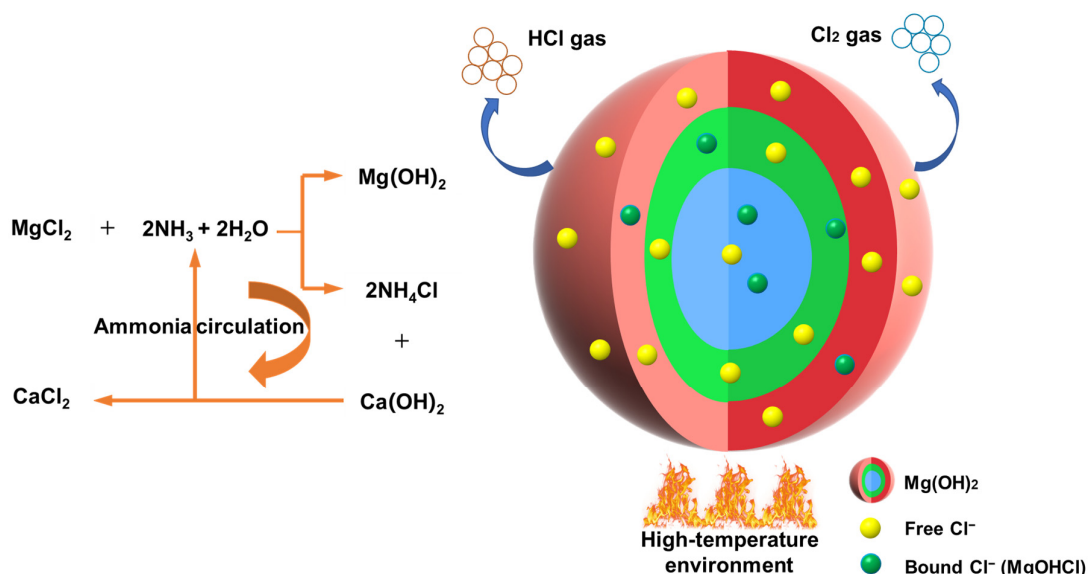


Figure 2. Ammonia/lime combination method and the hazards of chloride ions in $\text{Mg}(\text{OH})_2$.

Table 1. Chloride content requirements for magnesium hydroxide.

Application Area	Product Model	Cl^- %	Standard
Flame retardant	MC-1-2	0.08%	HG/T 4531-2013 [32]
	MC-2-15	0.15%	
Industrial use	I	0.10%	HG/T 3607-2007 [33]
	II/III-1	0.40%	
	II/III-2	0.50%	
Nano-powder	I	0.40%	HG/T 3821-2006 [34]
	II	0.50%	

Extensive research has been conducted to explore the removal of chloride ions from magnesium hydroxide. Liu et al. [35] demonstrated that ball milling effectively disrupts the crystal lattice of magnesium hydroxide, while the addition of strong alkalis under hydrothermal conditions promotes its recrystallization, leading to the release and reduction of chloride ions. Hydrothermal reactions and strong alkalis improve the efficiency of converting basic magnesium chloride to magnesium hydroxide. However, chloride ions are embedded within the magnesium hydroxide particles in successive layers, requiring significant energy to remove them. Wang et al. [36] showed that high-chlorine magnesium hydroxide, when subjected to single-stage or multistage stirring and washing with ammonia, reduced its chloride content from 0.8% to below 0.2%. This method is simpler and less energy-intensive than hydrothermal treatments. The integration of ammonia washing with current processes and the management of ammonia-containing wastewater require further investigation. Song et al. [26] achieved a modest reduction in chloride content, from 0.34% to 0.33% in coarse magnesium hydroxide particles and from 0.42% to 0.34% in fine particles, through water washing. However, the challenges associated with this method include the need for water recycling and limitations in chloride removal efficiency. Wu et al. [37] employed flocculant dosage and temperature control to manage the settling rate of magnesium hydroxide and utilized a sedimentation/separation method to remove adsorbed chloride ions. This approach still requires consideration of chloride ion removal beyond the adsorbed form. Feng et al. [38] developed a novel method using electrodeposition at the cathode to produce magnesium hydroxide from magnesium chloride. Jiang et al. [39]

reported that the magnesium hydroxide obtained by electrodeposition achieved a chloride content of 0.03%. Further industrial-scale verification is needed, and future research should explore the application of electrochemical methods for directly processing high-chlorine magnesium hydroxide from salt lakes. As summarized in Table 2, current methods either require significant energy input and high alkali consumption or face limitations in their applicability to the treatment of magnesium hydroxide slurry containing chloride ions. Therefore, there remains an urgent need for the development of more efficient and innovative chloride ion removal methods.

Table 2. Chloride control practices in magnesium hydroxide.

Author	Method	Initial Cl [−] %	Final Cl [−] %	Chloride Removal Efficiency, %	Advantage	Disadvantage
Liu et al. [35]	Ball milling and strong alkalis under hydrothermal conditions	0.5%	0.01%	98	Chloride removal efficiency high	High energy input and strong alkalis
Wang et al. [36]	Washing with ammonia	0.8%	0.2%	75	Short flowsheet with less energy consumption	Ammonia-containing wastewater treatment
Song et al. [26]	Water washing	0.34%	0.33%	3	Short flowsheet with less energy consumption	Water recycling and low removal efficiency
		0.42%	0.3%	29		
Wu et al. [37]	Flocculant	-	4.5%	-	Short flowsheet	Chlorine content high
Jiang et al. [39]	Electrodeposition	-	0.03%	-	Chlorine level low	Needs to be applied in magnesium hydroxide slurry

In mixed water/ethanol solutions, ethanol significantly affects the morphology and phase stability of crystals [40]. Jiang et al. [41] employed hexahydrate magnesium chloride ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) and sodium hydroxide (NaOH) as primary materials, along with urea and ethanol as additives, to synthesize hexagonal plate-like magnesium hydroxide ($\text{Mg}(\text{OH})_2$) nanoparticles through a surfactant-free precipitation method. The introduction of ethanol into the reaction system led to the formation of predominantly hexagonal plate-like nanoparticles, highlighting ethanol's crucial role in shaping the morphology of magnesium hydroxide nanoparticles. $\text{Mg}(\text{OH})_2$ nano-whiskers were produced using rapid anodization in chloride containing ethanol/water mixtures in several seconds; the high ethanol content caused a decrease in the nano-whiskers' surface coverage [42]. Furthermore, mesoporous spherical magnesium hydroxide was synthesized using a static self-assembly method, incorporating magnesium sulfate, an ammonia solution, ethanol, and sodium hydroxide as precursors [43]. Ethanol was also utilized to extract hexahydrate magnesium chloride from magnesium salt minerals [44]. Although ethanol is widely used, research on its role in chloride ion separation and magnesium hydroxide purification in sodium hydroxide solutions remains scarce, warranting further investigation.

Chloride ions limit the application of magnesium hydroxide in the premium magnesium salt market. A new approach was needed to remove chloride ions from salt lake-based magnesium hydroxide, and thus, the ethanol/sodium hydroxide method was systematically investigated for the first time. Several kinetic models were proposed or employed to analyze the chloride removal process. The characteristics of magnesium hydroxide before and after ethanol/sodium hydroxide treatment were examined to elucidate the potential mechanism of chloride removal. The objective was to enhance the understanding of the

ethanol-assisted sodium hydroxide chloride removal process and provide valuable insights into the application potential of this technique.

2. Experimental Details

2.1. Materials and Sample Preparation

Magnesium hydroxide powder was obtained from a company located in Qinghai province. The physical and chemical properties of magnesium hydroxide are shown in Table 3. The chloride ion content was less than 0.6%. Sodium hydroxide and ethanol of analytical grade were used without further purification. All chemicals and deionized distilled water (conductivity $< 0.1 \mu\text{S}\cdot\text{cm}^{-1}$) were used in the experiments.

Table 3. Main physical and chemical properties of magnesium hydroxide.

	Contents	HCl Insoluble	Water	Cl [−]	CaO	Fe ₂ O ₃	Loss on Ignition
Quality Index	≥99%	≤0.05%	≤0.5%	≤0.6%	≤0.02%	≤0.01%	≥30%

2.2. Experimental Methods

The specific volumes of distilled water and ethanol were accurately measured, and then the distilled water and ethanol were placed in three-necked flasks. The predetermined amount of NaOH was added and the flasks were immersed in a constant temperature water bath. Once the solution temperature in the flasks matched that of the water bath, Mg(OH)₂ particles were introduced and mixed using a stirrer. After a designated reaction time, the suspension was filtered, and the obtained Mg(OH)₂ solid particles were transferred to a drying oven at 100 °C for 8 h. The experimental setup is shown in Figure 3.

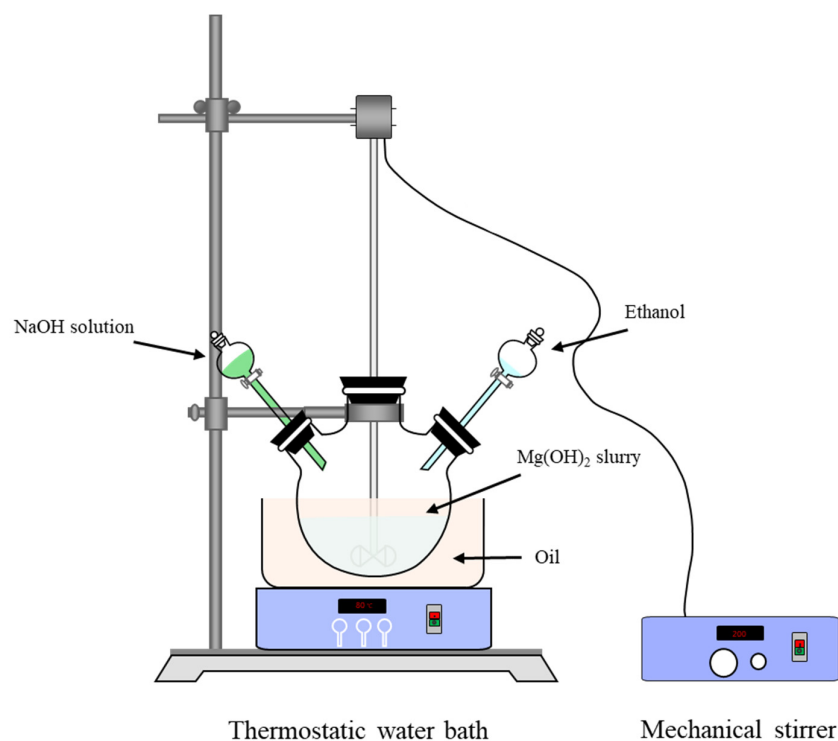


Figure 3. Layout of the experimental apparatus.

2.3. Chloride Measurement

Approximately 0.5 g of magnesium hydroxide was dissolved using 8 mol/L nitric acid (HNO₃). The pH was then adjusted to a range of 7–8. The solution was transferred to a

50 mL volumetric flask and diluted to the mark with deionized water. Finally, chloride ion concentration was determined by titration with silver nitrate (AgNO_3). The titration was conducted with a ZDJ-4A automatic potentiometric titrator. The concentration of released chloride ions was calculated using the following formula.

$$C_m = \frac{35.45 \times C_{\text{AgNO}_3} \times V_1 \times V_2}{G \times V_3} \quad (1)$$

where C_m represents the concentration of chloride ions in the sample (mg/g), equivalent to the released chloride ions in this section; C_{AgNO_3} is the concentration of the silver nitrate solution (0.01 mol/L in this experiment); V_1 is the volume of silver nitrate used at the equivalence point (mL); V_2 is the immersion sample volume (mL); V_3 is the volume of the sample (mL); and G is the weight of the sample (g).

Experiments were performed under carefully controlled conditions, with key parameters like temperature, time, and concentration closely monitored. The relative standard deviations of triplicates fell within certified laboratory limits; mean values with error bars are reported in the tables and figures, and mean values excluding error bars are reported in the text.

2.4. Characterization

The morphology of the products was analyzed using a Hitachi S-3500N scanning electron microscope (SEM). Particle size distribution was measured using a Brookhaven 90 Plus ultrasonic particle size analyzer.

3. Results and Discussion

This study conducted a thermodynamic analysis to identify potential chemical reactions involved in the process. Single-factor experiments were performed to examine the effects of liquid-to-solid ratio, ethanol content, NaOH content, reaction time, and reaction temperature on the chloride removal rate, ultimately determining the optimal experimental conditions. Additionally, a kinetic analysis was carried out to understand the rate-limiting step, and the underlying mechanisms governing chloride removal were examined.

3.1. The Cl^- Ion in $\text{Mg}(\text{OH})_2$ and Thermodynamic Analysis

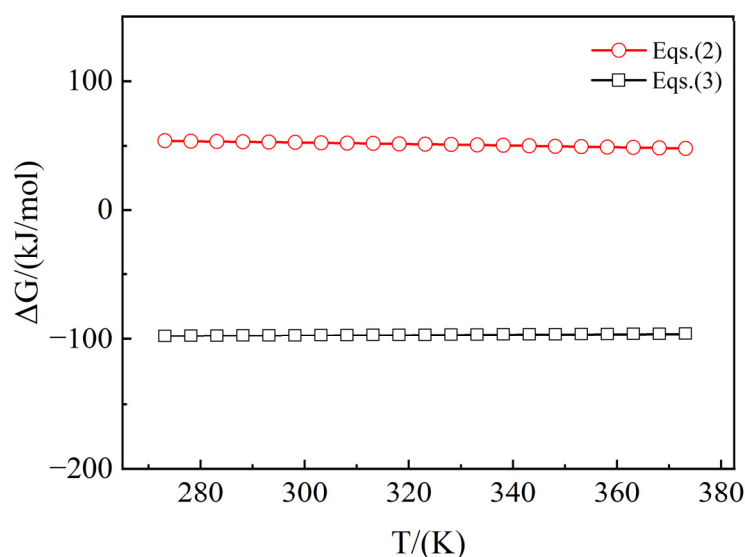
The removal of trace chloride ions from magnesium hydroxide remains a critical focus in industry, with efforts directed toward simpler and more efficient methods [45]. Liu et al. [46] identified two forms of chloride ions in magnesium hydroxide: adsorbed and crystalline. While adsorbed chloride ions can be removed by washing or calcination, crystalline chloride ions are more resistant to removal under typical conditions [47]. Studies show that adsorbed chloride ions generally comprise a larger proportion, although crystalline chloride ions are the primary contributors to impurities in the final product [48]. The presence of adsorbed chloride ions and the formation of basic magnesium chloride (MgClOH) complicate chloride removal during industrial-scale production using the ammonia method [49]. Therefore, the removal of chloride ions from magnesium hydroxide is crucial and urgently needed.

To better understand the key changes in the chloride removal process, the thermodynamic behavior of the main components in the NaOH-based cleaning of $\text{Mg}(\text{OH})_2$ particles was investigated. The primary reactions occurring in the $\text{Mg}(\text{OH})_2$ - $\text{Mg}(\text{OH})\text{Cl}$ -NaOH system with the corresponding ΔG_T° -T relationships are summarized in Table 4.

Table 4. Main reactions and ΔG_T^θ -T relationships in the $\text{Mg}(\text{OH})_2$ - $\text{Mg}(\text{OH})\text{Cl}$ - NaOH system.

Reaction Equations	ΔG_T^θ -T(kJ/mol)	Eqs.
$2\text{Mg}(\text{OH})\text{Cl}(\text{s}) = \text{Mg}(\text{OH})_2(\text{s}) + \text{MgCl}_2(\text{l})$	$\Delta G_T^\theta = -0.05748T + 69.65166$	(2)
$\text{Mg}(\text{OH})\text{Cl}(\text{s}) + \text{NaOH}(\text{l}) = \text{Mg}(\text{OH})_2(\text{s}) + \text{NaCl}(\text{l})$	$\Delta G_T^\theta = 0.01675T - 102.15172$	(3)

Thermodynamic feasibility, as indicated by Gibbs energy, remains constant across temperature ranges, as illustrated in Figure 4. The ΔG_T^θ curve for Equation (2) remains positive, indicating that the reaction is not thermodynamically favorable, and chloride ions in the form of $\text{Mg}(\text{OH})\text{Cl}$ cannot hydrolyze into the liquid phase. In contrast, ΔG_T^θ for Equation (3) is consistently negative, indicating thermodynamic feasibility. The addition of NaOH in Equation (3) lowers ΔG_T^θ , thereby facilitating the removal of chloride ions.

**Figure 4.** The main reactions and ΔG_T^θ -T curves during the chloride removal process from $\text{Mg}(\text{OH})_2$ solid particles.

3.2. Effect of Liquid/Solid Ratio

Reducing the liquid/solid ratio and increasing the solution viscosity can decrease the effective collisions between hydroxyl ions and basic magnesium chloride molecules, leading to a decline in Cl^- removal efficiency. Figure 5 shows that as the liquid-to-solid ratio decreases, with an increase in $\text{Mg}(\text{OH})_2$ concentration, the chloride removal rate significantly decreases. However, the addition of a certain amount of ethanol to the solution effectively enhances chloride removal. As the liquid-to-solid ratio decreases, the aggregation of particles in the solution increases, reducing the contact between chloride ions and solid particles. This leads to a decline in chloride removal efficiency. Ethanol enhances chloride removal efficiency by adsorbing onto the surface of solid particles, thereby reducing the solution's polarity. This results in an optimal chloride removal rate of 62.2%, achieved at a liquid-to-solid ratio of 80:1.

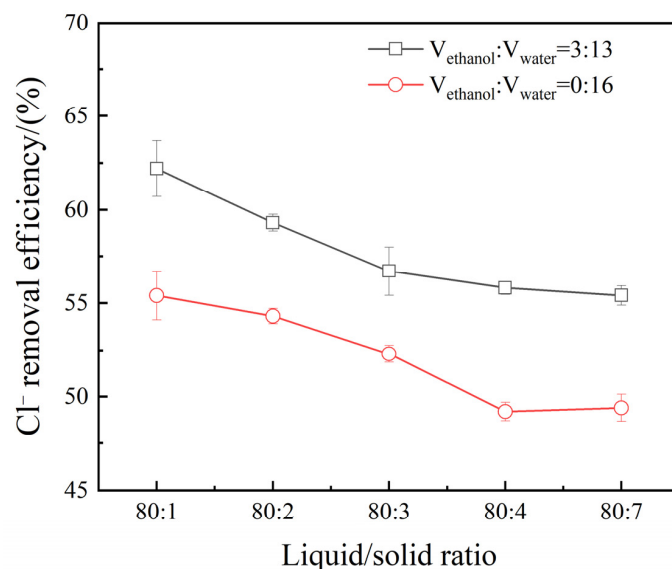


Figure 5. Effect of liquid/solid ratio on chloride removal efficiency (temperature 75 °C, time 1 h, NaOH 0.08 mol/L).

3.3. Effect of Ethanol Concentration

Basic magnesium chloride readily converts to $\text{Mg}(\text{OH})_2$ in the presence of NaOH, which also promotes the transformation of Mg^{2+} in the solution into $\text{Mg}(\text{OH})_2$. The effectiveness of ethanol in removing Cl^- ions is significantly influenced by its concentration. Ethanol adsorbs onto the surface of solid particles, reducing surface energy and system polarity, which aids in Cl^- removal. However, excessive ethanol can create steric hindrance, reducing collision rates [50] and subsequently lowering Cl^- removal efficiency.

Figure 6 illustrates that, in a NaOH-containing solvent, the addition of ethanol enhanced chloride ion removal efficiency, reaching a maximum of 56.0%. This was achieved at an ethanol-to-water volume ratio of 3:13. NaOH facilitates the replacement of Cl^- with OH^- , converting it into $\text{Mg}(\text{OH})_2$. Increasing the NaOH concentration shifts the reaction equilibrium to the right, thereby improving Cl^- removal. Conversely, without NaOH, the Cl^- removal rate decreases as ethanol concentration increases, reaching its lowest in pure ethanol.

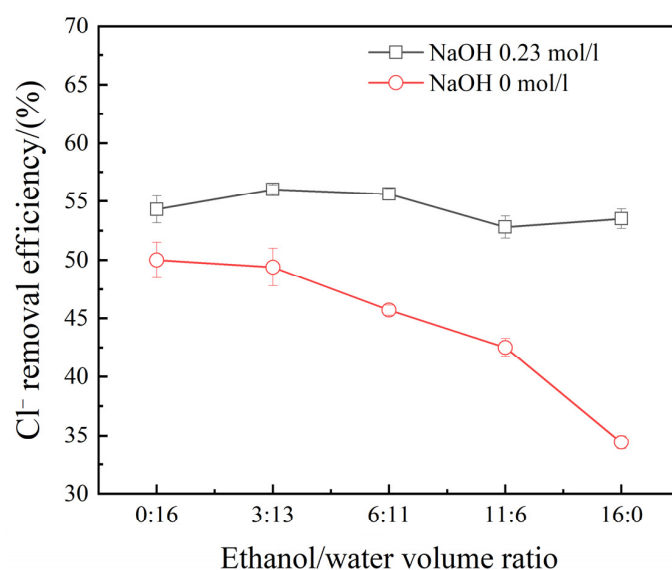


Figure 6. Effect of ethanol on chloride removal efficiency (temperature 75 °C, time 1 h, liquid/solid ratio 80:1).

3.4. Effect of NaOH Concentration

Chloride ions exist in two primary forms: some are physically adsorbed on the solid surface or trapped within the crystalline structure of magnesium hydroxide particles, while others may be present as basic magnesium chloride. The Cl^- ions adsorbed on the solid surface can be removed by solvent washing. NaOH effectively converts basic magnesium chloride into $\text{Mg}(\text{OH})_2$, and with increasing NaOH concentration, the reaction equilibrium shifts to favor this conversion, thereby enhancing Cl^- removal efficiency. The excess hydroxide ions (OH^-) in the solution facilitate the substitution of chloride ions (Cl^-) on the surface of solid particles. Data from Figure 7 indicate that increasing the amount of NaOH markedly improves the Cl^- removal rate, which stabilizes once the NaOH reaches a certain threshold of NaOH concentration, 0.08 mol/L.

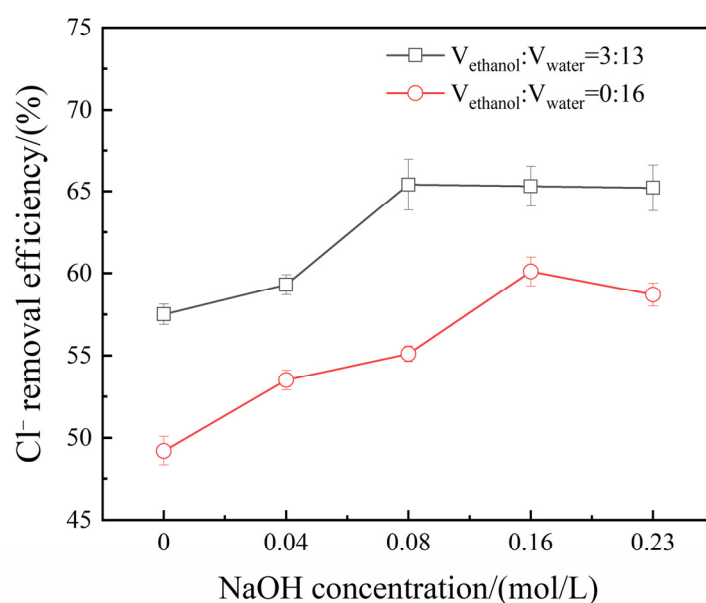


Figure 7. Effect of NaOH/ $\text{Mg}(\text{OH})_2$ mass ratio on chloride removal efficiency (temperature 75 °C, time 1 h, liquid/solid ratio 80).

The addition of ethanol to the solvent enhances chloride ion removal, achieving a maximum removal rate of 65.4% at a NaOH concentration of 0.08 mol/L. Ethanol adsorbs onto the particle surface, reducing surface energy and the polarity of the system, which aids in Cl^- removal. Baranek et al. demonstrated that in ethanol-containing media, basic magnesium salts with crystalline water more readily convert to magnesium hydroxide [49]. At a NaOH concentration of 0 mol/L, the chloride removal efficiency increased by 8.3% as the ethanol/water volume ratio was adjusted from 0 to 3:13. When the NaOH concentration was subsequently increased to 0.08 mol/L, the chloride removal efficiency further improved to 10.3%. The additional increase of 2% can be directly attributed to the influence of NaOH. Therefore, it can be concluded that the effect of ethanol on chloride removal is more significant than that of NaOH, as evidenced by the comparison of their respective contributions to the removal of chloride ions. Overall, optimizing the use of NaOH and ethanol in the solvent enhances the efficiency of chloride ion removal from magnesium hydroxide, making the process more effective and environmentally friendly.

3.5. Effect of Reaction Time

Chloride ion migration requires movement from the interior of solid particles to the reaction surface. At the reaction interface, OH^- ions replace Cl^- , which then continues to migrate into the liquid phase, reaching equilibrium over time. Figure 8 shows that when

the reaction time extends from 1 to 10 h, the Cl^- removal rate in a pure water system fluctuates within a range of 54.2% to 55.0%. In contrast, when ethanol is present in the system, the removal rate varies within a smaller range, from 68.3% to 68.8%. These data indicate that while the presence of ethanol improves Cl^- removal efficiency, the reaction time has a minimal impact. This is likely because the reaction reaches equilibrium within the given time frame of 1 h, resulting in no further significant increase in Cl^- removal rate.

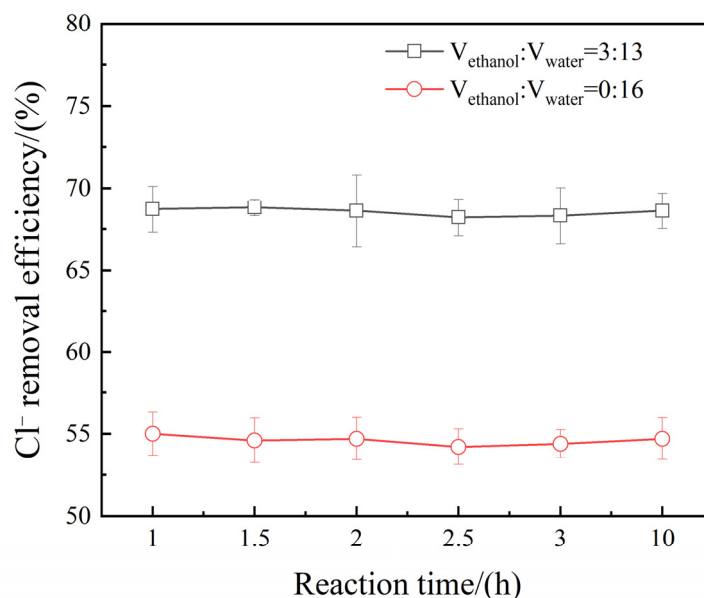


Figure 8. Effect of reaction time on chloride removal efficiency (temperature 75 °C, liquid/solid ratio 80:1; NaOH 0.08 mol/L).

3.6. Effect of Reaction Temperature

The reaction temperature significantly influences both the reactivity of substances and the reaction equilibrium. Generally, increasing the temperature shifts the equilibrium of an endothermic reaction to the right and enhances ion mobility. As demonstrated in Figure 9, the chloride ion (Cl^-) removal rate increases with rising reaction temperatures. Notably, Cl^- ions that are physically adsorbed on the surface of crystals or solid particles are more likely to desorb into the solution at higher temperatures. Moreover, elevated temperatures facilitate the migration of Cl^- , allowing a greater amount to transfer into the solution within the same time frame. Ethanol further aids Cl^- removal by adsorbing onto the surface of magnesium hydroxide, reducing surface energy and decreasing the system's polarity, thereby enhancing chloride ion extraction. As demonstrated in Figure 9, at a NaOH concentration of 0.08 mol/L and a reaction temperature of 90 °C, the chloride ion removal rate increased by 12.7% as the ethanol-to-water volume ratio was raised from 0 to 3:13. This result highlights the substantial enhancement in removal efficiency attributable to the presence of ethanol. However, at 90 °C, substantial liquid evaporation and energy consumption occur. Therefore, a temperature of 75 °C, yielding a chloride removal efficiency of 68.8% with the optimal ethanol-to-water volume ratio, offers a balance between energy consumption and chloride removal efficiency. Consequently, this temperature was chosen for subsequent experiments.

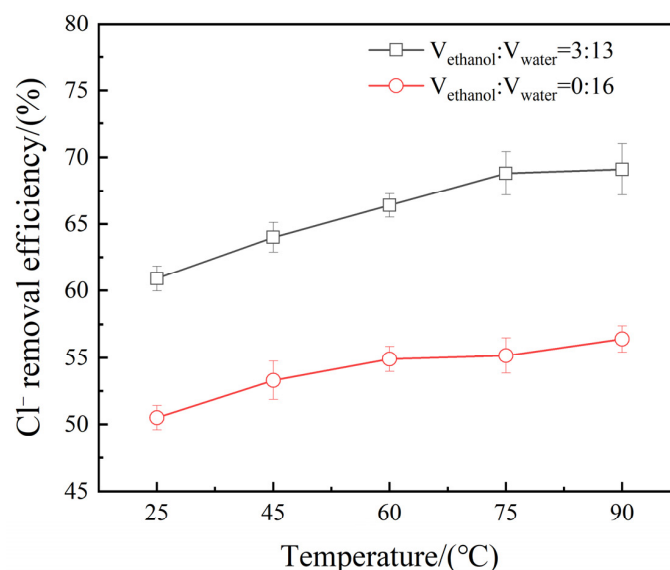


Figure 9. Effect of temperature on chloride removal efficiency (time 1 h, liquid/solid ratio 80:1; NaOH concentration 0.08 mol/L).

3.7. Kinetic Analysis

The removal of chloride ions in a liquid/solid reaction system involves three main steps: first, OH^- ions replace Cl^- ions, which then diffuse from the interior of solid particles or grains to the surface; second, the Cl^- ions cross the solid/liquid interface; and finally, the Cl^- ions enter the liquid phase.

As shown in Figure 10, at both 25 °C and 75 °C, the Cl^- removal reaches equilibrium after 10 min of reaction time. At 45 °C, the removal rate achieves dynamic equilibrium within the same time frame. Temperature significantly influences the Cl^- removal rate, with higher temperatures generally enhancing the process. This is because increased temperature promotes the disordered diffusion of Cl^- , allowing more ions to be removed from the particles. However, higher temperatures also increase the likelihood of collisions between chloride ions and particles, creating a bidirectional acceleration effect within a certain temperature range. This dual effect explains the fluctuations in Cl^- removal rates observed at 45 °C [51].

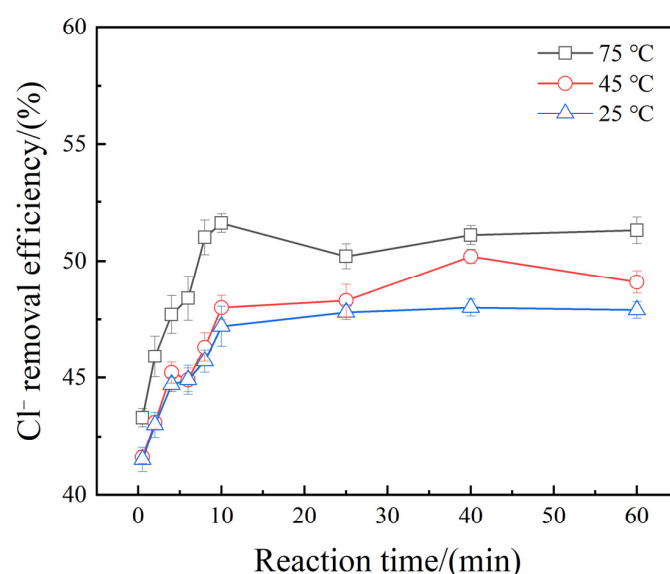


Figure 10. Effect of reaction time and temperature on chloride removal efficiency (liquid/solid ratio 80:7, ethanol/water ratio 3:13; NaOH 0.08 mol/L).

The reaction system is a solid/liquid phase reaction, with magnesium hydroxide as the raw material, which has been airflow-crushed into particles with various shapes, including spherical, flaky, and blocky forms, as shown in Figure 11. The kinetic model used in this study is detailed in Table 5.

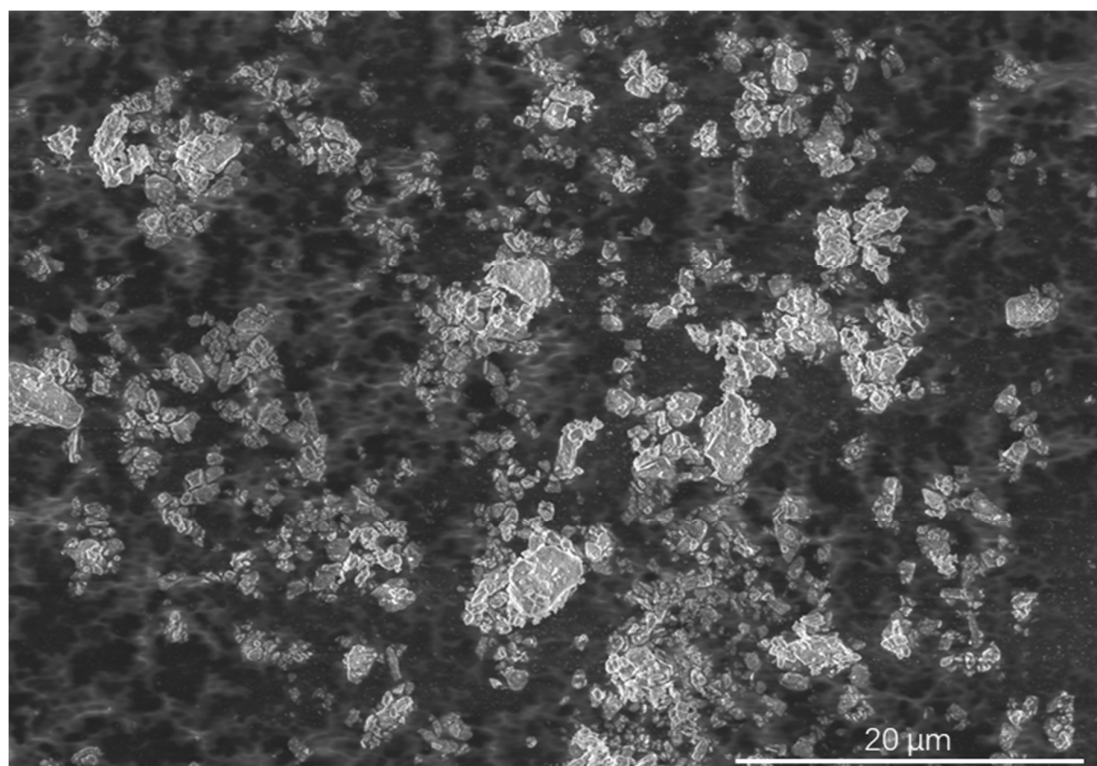


Figure 11. Morphology of raw materials.

Table 5. Kinetic models and control equations.

Kinetic Control Steps	Spherical Unreacted Shrinkage Nuclei Model	Plate-Like Unreacted Shrinkage Nuclei Model
External diffusion control	$x = k_1 t$	$x = k_1 t$
Surface chemical reaction control	$1 - (1 - x)^{\frac{1}{3}} = k_2 t$	$x = k_4 t$
Solid film diffusion control	$1 - \frac{2}{3}x - (1 - x)^{\frac{2}{3}} = k_3 t$	$x^2 = k_5 t$

x is the conversion fraction (leaching rate) of Cl^- , %; k is the reaction rate constant; t is the leaching times, min.

By utilizing the chloride removal models outlined in Table 5 and combining them with the chloride removal rates depicted in Figure 12, experimental data were incorporated into the kinetics equations provided in Table 5 to calculate the relationship between equations and practical outcomes. The fitting correlation coefficient results are presented in Table 6.

Table 6 indicates that the spherical and plate-like unreacted core models for diffusion-controlled kinetics equations exhibit better fitting results for chloride (Cl^-) data. Comparatively, the spherical particle model demonstrates slightly higher fitting confidence, suggesting that the kinetics of magnesium hydroxide removing trace amounts of chloride ions can be described using the spherical unreacted core model, controlled by internal diffusion. Employing this model to fit the kinetics equation for chloride yields the results depicted in Figure 12. It is evident that the reaction rate constant, k , increases with temperature, indicating that higher temperatures facilitate diffusion and mass transfer of Cl^- , promoting the forward reaction [52].

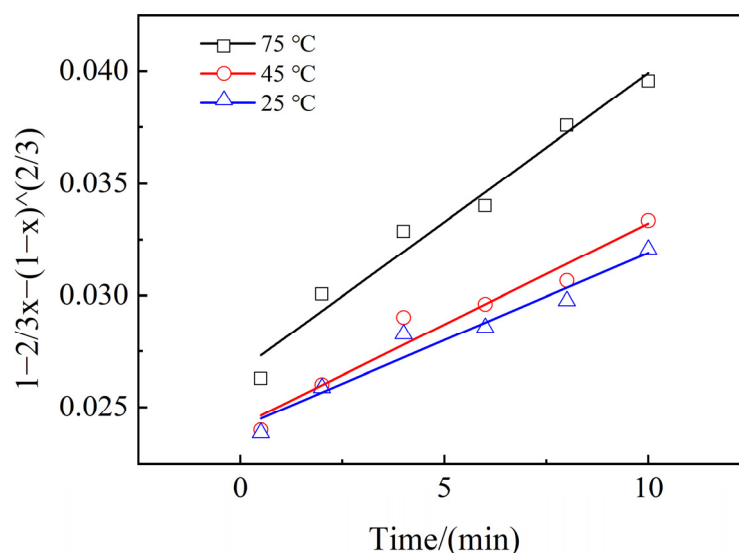


Figure 12. The relationship between $1 - \frac{2}{3}x - (1-x)^{\frac{2}{3}}$ and reaction time t .

Table 6. Fitting parameters of different equations for chloride (Cl^-) removal.

Models	Control Equation	T/°C	k/ 10^{-3}	R ²
Spherical	$1 - (1-x)^{\frac{1}{3}} = k_1 t$	75	4.35	0.95296
		45	3.04	0.92542
		25	2.69	0.93143
	$1 - \frac{2}{3}x - (1-x)^{\frac{2}{3}} = k_2 t$	75	1.37	0.95908
		45	0.89	0.92738
		25	0.78	0.93596
Plate-like	$x = k_1 t$ $x = k_4 t$	75	8.45	0.94825
		45	6.14	0.92380
		25	5.45	0.92853
	$x^2 = k_5 t$	75	8.08	0.95576
		45	5.50	0.92655
		25	4.84	0.93381

3.8. Calculation of Apparent Activation Energy

The Arrhenius equation can be used to compute the apparent activation energy of a reaction, establishing a relationship between the apparent rate constant k and temperature T . The formula is as follows:

$$k = Ae^{\frac{-E_a}{RT}} \quad (4)$$

where k —rate constant, min^{-1} ; A —pre-exponential factor, min^{-1} ; E_a —activation energy of the reaction, kJ/mol ; R —ideal gas constant, $8.314 \text{ J/(mol}\cdot\text{K)}$; T —thermodynamic temperature, K .

Using Equation (4), the following formula can be derived:

$$\ln k = \frac{-E_a}{RT} + A \quad (5)$$

The apparent activation energy is calculated from the slope and intercept in Figure 13 as 9.768 kJ/mol , falling within the range of 8 to 30 kJ/mol [53], consistent with characteristics of internal diffusion control. This further elucidates that the removal of Cl^- is primarily governed by internal diffusion.

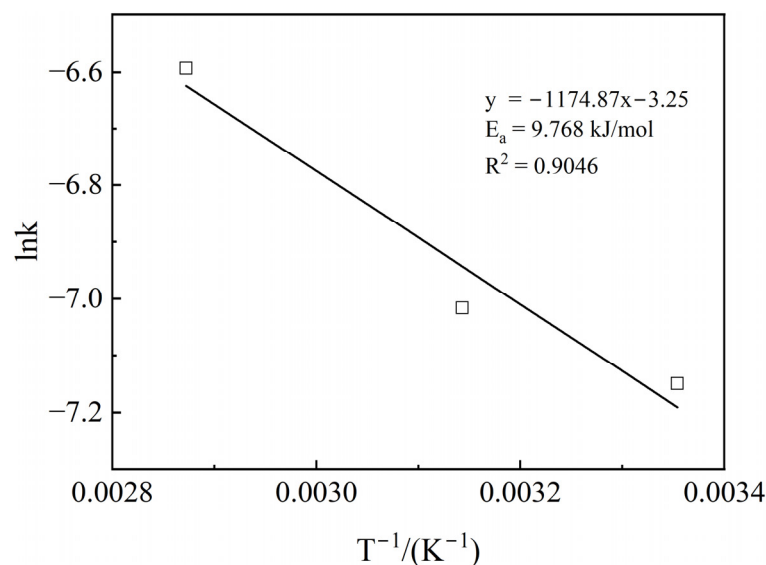


Figure 13. Arrhenius equation fitting curve.

4. Mechanism of Chloride Removal Process with NaOH and Ethanol

To further understand the mechanism of chloride removal by water, ethanol, and NaOH, particle size distribution and zeta potential measurement were conducted on products obtained under different conditions. These analyses revealed that each of these three components—water, ethanol, and sodium hydroxide—affects the dispersion of the solid particles. These effects ultimately influence the chloride removal efficiency.

4.1. Particle Size Distribution Analysis

The distribution of particle sizes impacts chloride removal. When excessive particle aggregation occurs, the specific surface area decreases, preventing adequate interaction between chloride ions and the solid particles. In contrast, better particle dispersion increases the specific surface area, allowing more effective contact between chloride ions and the solution, thereby improving chloride removal. Water, ethanol, and sodium hydroxide each affect the particle distribution, as shown in the particle distribution curves in Figure 14.

Water, ethanol, and sodium hydroxide in the solution all help to mitigate the aggregation of solid magnesium hydroxide ($Mg(OH)_2$) particles. In an aqueous solution, $Mg(OH)_2$ undergoes aging, which improves particle crystallinity. Ethanol reduces the system's polarity, making the particle surfaces smoother. Sodium hydroxide releases OH^- ions that can replace chloride ions (Cl^-), and in the aging process, a controlled amount of sodium hydroxide reduces surface defects in the particles, improving their crystallinity and reducing aggregation [54].

The $Mg(OH)_2$ used in this study underwent crushing, leading to small particles adsorbing onto larger ones and aggregating. The particle size distribution showed three distinct peaks, indicative of small particles clustering together. During the chloride removal process in water, the solid particles were effectively dispersed, with water cleaning Cl^- from the particle surfaces. The addition of ethanol lowered the system's polarity, aiding in the desorption of Cl^- . The mean particle size of Curve 1 was $4.43\ \mu m$, while Curve 2, which underwent water washing, showed a smaller mean particle size of $3.17\ \mu m$, indicating reduced aggregation of the solid particles. However, adding ethanol to pure water (Curve 2 vs. Curve 3) had little effect on the particle size.

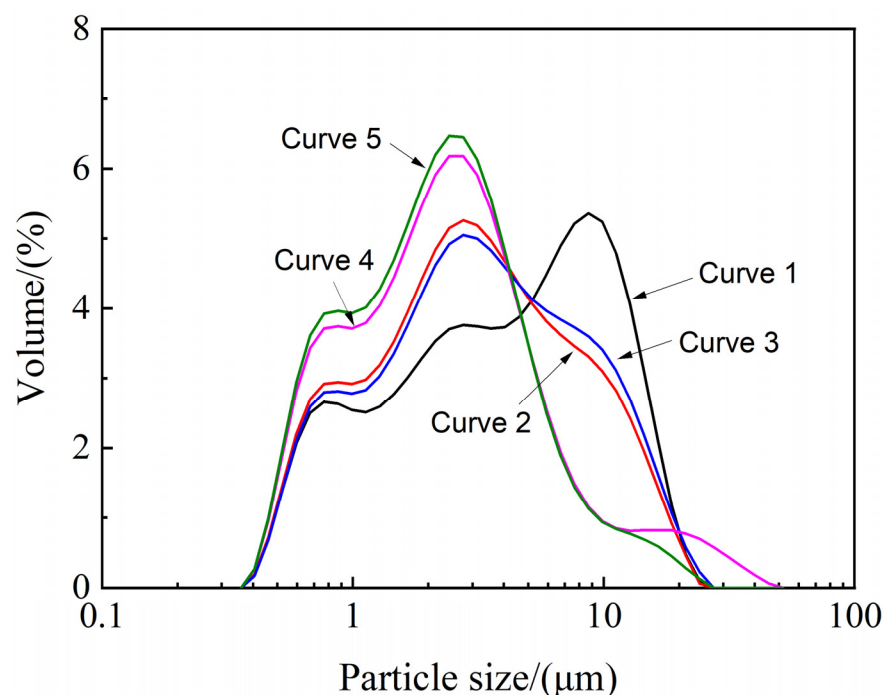


Figure 14. Particle size distribution diagram (Curve 1. Raw $\text{Mg}(\text{OH})_2$; Curve 2. Water: 75 °C, pure water, $t = 1$ h, liquid/solid ratio 80; Curve 3. Ethanol: 75 °C, ethanol/water ratio 3:13, $t = 1$ h, liquid/solid ratio 80; Curve 4. NaOH: 75 °C, pure water, $t = 1$ h, liquid/solid ratio 80, NaOH 0.23 mol/L; Curve 5. Ethanol/NaOH: 75 °C, ethanol/water ratio 3:13, $t = 1$ h, liquid/solid ratio 80, NaOH 0.23 mol/L).

The data from Curves 4 and 2 in Figure 14 indicate that sodium hydroxide addition reduces particle aggregation. When sodium hydroxide is present, adding ethanol further enhances the dispersion of particles, as shown by comparing Curves 3, 4, and 5. According to Table 7, sodium hydroxide increases the specific surface area of the solid particles, and ethanol addition further enhances this surface area. The mean specific surface areas for Curves 2, 3, 4, and 5 were 2832 m^2/kg , 2731 m^2/kg , 3364 m^2/kg , and 3510 m^2/kg , respectively, showing that ethanol effectively reduces aggregation when sodium hydroxide is present. However, in systems containing only water and ethanol, ethanol has minimal effect on particle aggregation.

Table 7. Median particle size and specific surface area corresponding to each curve.

	Curve 1. Raw $\text{Mg}(\text{OH})_2$	Curve 2. Water	Curve 3. Ethanol	Curve 4. NaOH	Curve 5. Ethanol/NaOH
Median particle diameter (μm)	4.43 ± 0.02	3.17 ± 0.03	3.37 ± 0.02	2.47 ± 0.01	2.34 ± 0.03
Specific surface area (m^2/kg)	2502 ± 2	2832 ± 4	2731 ± 3	3364 ± 6	3510 ± 5

Description: 1. Primary product; 2. Temperature 75 °C, pure water, $t = 1$ h, liquid/solid ratio 80; 3. Temperature 75 °C, ethanol/water ratio 3:13, $t = 1$ h, liquid/solid ratio 80; 4. Temperature 75 °C, pure water, $t = 1$ h, liquid/solid ratio 80, NaOH 0.23 mol/L; 5. Temperature 75 °C, ethanol/water ratio 3:13, $t = 1$ h, liquid/solid ratio 80, NaOH 0.23 mol/L.

NaOH, by providing OH^- ions, rapidly displaces Cl^- and induces the nucleation of $\text{Mg}(\text{OH})_2$, resulting in the formation of small particles. However, secondary agglomeration of these particles reduces the effective reactive surface area, thereby trapping Cl^- ions. Consequently, NaOH alone demonstrates relatively limited efficiency in chloride removal. Conversely, ethanol acts as a dispersing agent, exposing more internal Cl^- by dispersing the particles and shortening the diffusion path. Moreover, the synergistic effect between ethanol's dispersing properties and NaOH's chemical substitution not only facilitates the

release of Cl^- but also prevents its re-adsorption. Therefore, the combined use of ethanol and NaOH significantly enhances chloride removal efficiency.

4.2. Electrochemical Property of $\text{Mg}(\text{OH})_2$

Zeta potential measurements are shown in Table 8. The data indicate that adding ethanol to the system increases the zeta potential, while the addition of NaOH lowers it. When ethanol and NaOH are combined, the zeta potential drops sharply. This suggests that ethanol enhances the adsorption of OH^- on the particle surface, facilitating the expulsion of Cl^- and improving the removal efficiency. These findings highlight that the addition of ethanol and NaOH effectively improves the morphology of $\text{Mg}(\text{OH})_2$ particles, reduces aggregation, and enhances chloride removal.

Table 8. Zeta potential of $\text{Mg}(\text{OH})_2$.

	Sample b: Water	Sample c: Ethanol	Sample d: NaOH	Sample e: Ethanol/NaOH
Zeta potential	9.14 ± 0.03	17.30 ± 0.05	-3.39 ± 0.07	-13.00 ± 0.08
pH	7.55 ± 0.04	8.19 ± 0.03	11.2 ± 0.02	11.34 ± 0.02

Notes: Sample b. Temperature 75 °C, water, $t = 1$ h, liquid/solid ratio 80; Sample c. Temperature 75 °C, ethanol/water ratio 3:13, $t = 1$ h, liquid/solid ratio 80; Sample d. Temperature 75 °C, water, $t = 1$ h, liquid/solid ratio 80, NaOH 0.23 mol/L; Sample e. Temperature 75 °C, ethanol/water ratio 3:13, $t = 1$ h, liquid/solid ratio 80, NaOH 0.23 mol/L.

4.3. Chloride Removal Mechanism

Under the optimal conditions of a liquid-to-solid ratio of 80:1, an ethanol-to-water ratio of 3:13, a NaOH concentration of 0.08 mol/L, and a reaction time of 1 h, the chloride removal efficiency achieved 68.9%, as illustrated in Figure 15. This outcome contrasts with the chloride concentrations observed in both the products and raw materials, demonstrating a significant reduction in chloride ion content. Specifically, the chloride concentration decreased from 0.43% to 0.14%. Moreover, the residual chloride ion level in the magnesium hydroxide after treatment meets the requirements of the MC-2-15 flame retardant standard, as well as the industrial use II/III 1/2 and nano-powder I/II product standards.

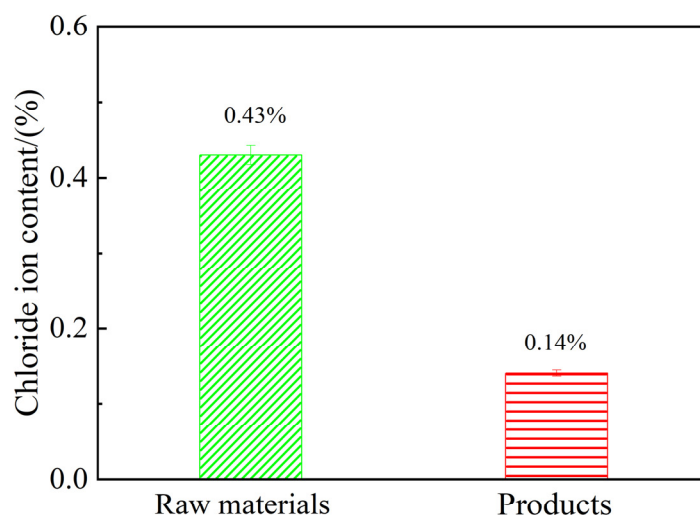


Figure 15. Chloride ion content in $\text{Mg}(\text{OH})_2$ raw materials and products after treatment.

Figure 16 further elucidates the mechanism by which ethanol enhances chloride removal from $\text{Mg}(\text{OH})_2$. During treatment, $\text{Mg}(\text{OH})_2$ undergoes aging, which promotes in situ crystal growth and improves crystallinity [55]. Ethanol ($\text{C}_2\text{H}_5\text{OH}$) reduces the solvent's polarity by lowering the dielectric constant, thereby destabilizing chloride ion solvation

and facilitating the release of chloride ions from the $\text{Mg}(\text{OH})_2$ surface. Additionally, ethanol selectively dehydrates the $\text{Mg}(\text{OH})_2$ surface, weakening the adsorption of chloride ions. Furthermore, the controlled addition of NaOH contributes to chloride removal by converting weakly adsorbed chloride ions, such as those in $\text{Mg}\text{--}\text{Cl}$ complexes, into free ions via ligand displacement. NaOH also reacts with $\text{Mg}(\text{OH})\text{Cl}$ to form $\text{Mg}(\text{OH})_2$ and NaCl , further releasing chloride ions while preserving the integrity of the $\text{Mg}(\text{OH})_2$ crystal structure and improving particle dispersion. Therefore, the synergistic interaction of ethanol and NaOH significantly enhances chloride removal efficiency.

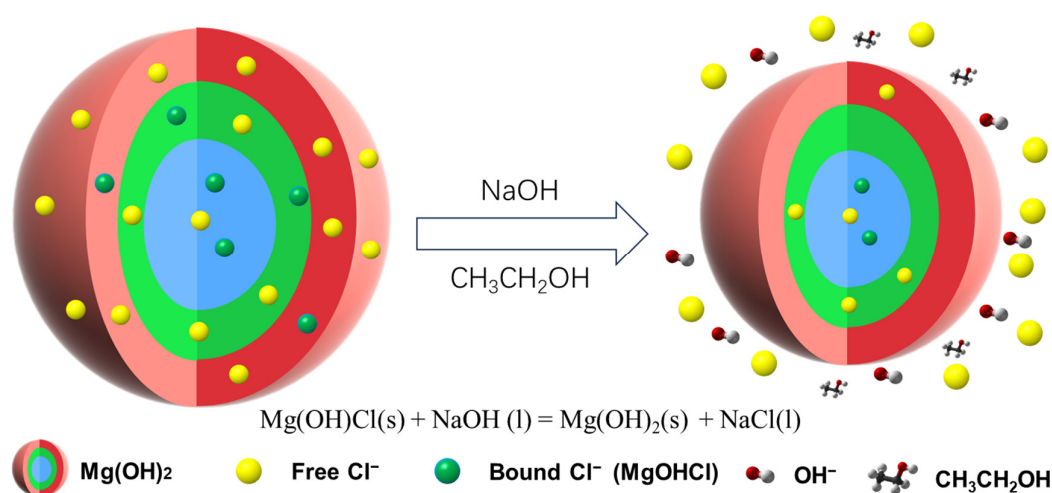


Figure 16. Schematic diagram of the process of removing Cl^- .

5. Summary and Conclusions

China possesses abundant magnesium resources in salt lakes, and the production technology of high-purity magnesium hydroxide is well developed. However, in the process of synthesizing magnesium hydroxide from bischofite using the ammoniation method under high-concentration reaction conditions, the presence of adsorbed chloride ions and the formation of basic magnesium chloride can result in the incorporation of chloride ions into magnesium hydroxide. This leads to excessive trace amounts of chlorine in magnesium hydroxide, which restricts its application and causes significant environmental pollution issues. Therefore, effectively and cost-efficiently removing trace chloride ions from magnesium hydroxide is one of the key challenges that urgently needs to be addressed for the high-quality development of the salt lake magnesium industry. In this regard, the removal of chloride from $\text{Mg}(\text{OH})_2$ using NaOH with ethanol was examined for the first time.

Additional studies are required to elucidate the interaction mechanisms of chloride ions, water molecules, sodium hydroxide, and ethanol in magnesium hydroxide slurry, utilizing solution chemistry, quantum chemistry, high-resolution transmission electron microscopy (HRTEM), atom probe tomography (APT), and molecular dynamics simulations. The comparison of other organic solvents with ethanol should be examined. The impact of chloride ions and water molecules on the chemical states of surface atoms can be assessed through techniques such as infrared and photoelectron spectroscopy, along with other surface characterization methods. Moreover, research should address chloride ion removal and the recycling of filtrates, investigating their potential incorporation into current high-chlorine magnesium hydroxide production processes with a DOE approach. Ongoing investigations will continue, with the following conclusions drawn at this stage:

1. The addition of NaOH to the aqueous solution improves the dispersion of $\text{Mg}(\text{OH})_2$ particles, and the removal of Cl^- by NaOH is primarily governed by internal diffusion.

2. Ethanol adsorbs to the surface of magnesium hydroxide, which decreases surface energy and reduces the system's polarity, thereby facilitating chloride ion removal.
3. The chloride removal rate reached 68.9% and Cl^- content in $\text{Mg}(\text{OH})_2$ decreased to 0.14% under optimized conditions.

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