



Preparation and Properties of Mg(OH)₂ Whiskers by Ion Exchange Resin Method

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Abstract: Mg(OH)₂ whiskers were successfully prepared under mechanical stirring with D301 ion exchange resin and Mg(NO₃)₂. The products were systematically characterized by testing settling ratio, turbidity, and analyzing SEM, FT-IR. The results showed that Mg(OH)₂ prepared with 0.05 or 0.1 mol/L Mg(NO₃)₂ had excellent dispersion, and the settling ratio was close to 100 % after 360 min; The time extension was extremely helpful to improve the dispersion; The dispersion of product was correspondingly good under pH value of about 9. The products synthesized with different concentrations and reaction time could reach nanoscale, and the product obtained using 0.05 mol/L Mg(NO₃)₂ for 4 h had whisker structure with a length of 10 μm and had a lipophilicity. FT-IR analysis showed the product included some functional groups or fragments from the resin, which had a positive modification effect on the products. The preparation of Mg(OH)₂ whisker with resin as raw material is simple, green, and environmentally friendly, which is worthy of further application.

Keywords: ion-exchange material, chemical synthesis, inorganic, microstructure, scanning electron microscopy

1. Introduction

Mg(OH)₂ has been widely used in chemical industry, environmental protection, medical treatment, and nano material synthesis due to its unique physical, chemical, and mechanical properties [1-6]. In particular, Mg(OH)₂ has the characteristics of high decomposition temperature, good thermal stability, non-toxic, smokeless, and smoke suppression, which can be applied in polymer materials as high-performance inorganic flame retardant [7-10]. At present, the preparation methods of Mg(OH)₂ mainly include processing of natural brucite powder, alkali precipitation of magnesium salt solution, hydration of magnesium oxide, hydrate pyrolysis method and so on [11-13]. For example, Yu Wenqiang et al [14], have prepared Mg(OH)₂ with sheet structure by liquid phase precipitation method using sodium dodecyl sulfate and ethanol as dispersing agents, magnesium chloride hexahydrate and sodium hydroxide as raw materials; Zhu Huabing et al [15] used brine as raw material, and firstly prepared high activity coarse magnesium oxide by direct dynamic pyrolysis, and then prepared flake and strip Mg(OH)₂ by hydration process. In the above methods, the former used ethanol and deionized water to filter and wash the crude product for many times, so as to obtain Mg(OH)₂ with high purity; while the latter pumped and filtered the emulsion containing magnesium quickly, and then adopted distilled water at the corresponding temperature to wash the filter cake for three times, and finally used anhydrous ethanol to wash the filter cake repeatedly, in order to stop the reaction and take away the magnesium nitrate on the solid surface, and finally dried the filter cake to achieve pure Mg(OH)₂ product. It can be seen that these methods usually need a lot of water or organic solvents to wash the product repeatedly in order to improve the purity, which can produce the large amount of wastewater and raise the production cost.

In addition, in the process of continuous application of inorganic particles such as Mg(OH)₂, more and more researchers found that these inorganic particles had high surface energy and poor affinity with organic compounds with low surface energy. In particular, inorganic particles and organic compounds were not appropriately compatible when they were contacted and mixed with each other, which led to more voids on the interface. In order to solve the above problems, it is usually necessary to modify the

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surface of $\text{Mg}(\text{OH})_2$ and other filler particles, and the most common modification method is to add sodium stearate, sodium oleate, sodium dodecyl sulfate and other modifiers [11, 14, 16]. The purpose is to make the surface of filler particles produce new physical, chemical, mechanical properties and new functions, and effectively improve the compatibility of filler particles with organic compounds. For the surface treatment of filler particles, many factors such as selection of modifiers, dosage of modifiers, modification temperature, modification time and so on should be considered systematically. However, this kind of filler surface treatment process can greatly increase the preparation cost of $\text{Mg}(\text{OH})_2$ and other inorganic particles, prolong the preparation time, and make the preparation process complicated.

Furthermore, $\text{Mg}(\text{OH})_2$ is a very interesting inorganic material. By changing the dosage ratio of raw materials, reaction temperature, reaction time, dosage of modifier and other conditions, or using different preparation methods, $\text{Mg}(\text{OH})_2$ with exceptionally diverse micro morphology can be presented, such as flower-like, strip shape, sheet structure, needle-like and honeycomb-like and so on [14, 15, 17, 18].

In this study, ion exchange resin was employed as raw material, which reacted with a kind of magnesium salt under strong mechanical stirring. By adjusting and controlling the dosage ratio of ion exchange resin and magnesium salt, the product could be directly applied without extra washing. At the same time, depending on the release uniformly and slowly of crystallographic ions from ion exchange resin with the unique internal channel structure, the product $\text{Mg}(\text{OH})_2$ with whisker-like was obtained successfully, and the dispersion of the product was strengthened significantly. Meanwhile, ion exchange resin had a certain modification effect on the as-obtained product, and the product with a certain lipophilic was achieved satisfactorily. This method of preparing $\text{Mg}(\text{OH})_2$ whisker by ion exchange resin has high industrial application value and prospect for synthesizing additive $\text{Mg}(\text{OH})_2$ flame retardant or $\text{Mg}(\text{OH})_2$ filler particles.

2. Materials and methods

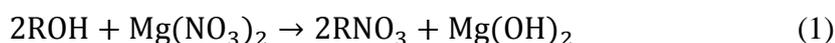
2.1. Preparation of $\text{Mg}(\text{OH})_2$

All chemical reagents, including NaOH , $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, and liquid paraffin, are of analytical grade and purchased from Tianjin (China) Bodi Chemical Co., Ltd. D301 ion exchange resin was provided by Chemical Plant of Nankai University, China, and the main performance indexes are shown in Table 1.

Table 1. Main performance indexes of D301 ion exchange resin

Name	D301 macroporous weakly basic styrene anion exchange resin
Functional group	$-\text{N}(\text{CH}_3)_2$
Moisture content/%	50-60
Mass total exchange capacity /mmol/g	≥ 4.80
Volume total exchange capacity /mmol/mL	≥ 1.45
Wet apparent density /g/ml	0.65-0.72
Wet true density /g/ml	1.03-1.07
Effective particle size /mm	0.50-0.70

D301 ion exchange resin was activated and pretreated by acid and base solution, and finally converted into OH^- form by NaOH solution. 100 mL D301 resins and 100 mL $\text{Mg}(\text{NO}_3)_2$ solution with different concentrations were placed in a conical flask, which was placed in a constant temperature water bath (HH-4, China) with 60°C . A strong electric mixer (ZD-90W, China) with 150 r/min was used to finish the stirring reaction. As the reaction proceeded, the solution became turbid gradually; after a certain time of reaction, the mixture was aged overnight in a water bath at 60°C ; at last the resin and products were easily separated by a proper filter, and the products were directly dried under 105°C without further washing. The reaction equation involved is shown in (1), where R represents the part other than the ion exchange group.



In addition, the *pH* value of the mixture was adjusted by NaOH and HCl solution to evaluate the effect of different *pH* values on the properties of the product.

2.2. Characterization methods

Test method of settling ratio curve: 100 mL of the suspension after complete reaction, that is, the product before standing overnight, was added into a measuring cylinder. The volume (*V*, mL) of the suspension at different times was seriously recorded to calculate the settling ratio according to formula (2), and then the time vs. settling ratio curves were completed correspondingly.

$$\text{Settling ratio} = \frac{V}{100} \times 100\% \quad (2)$$

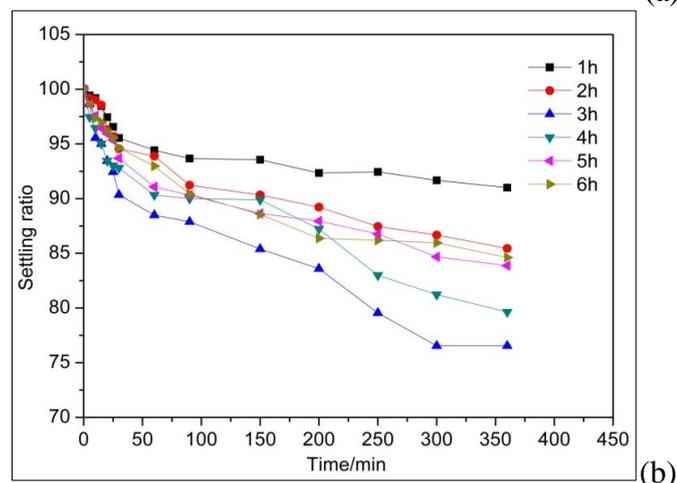
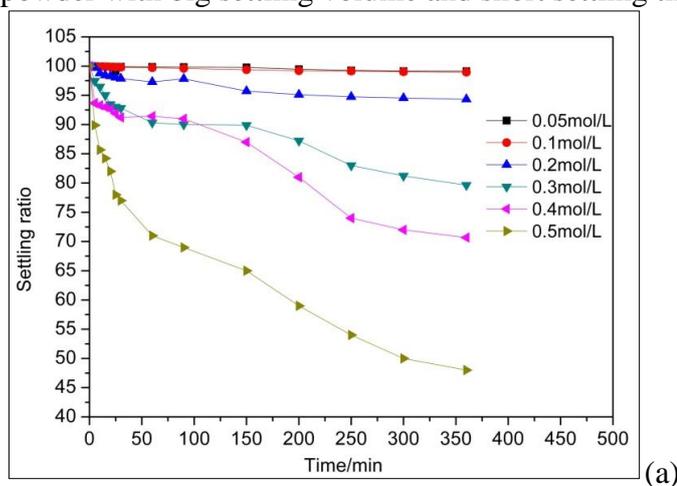
Determination of turbidity: 0.005 g powder product and 10 mL liquid paraffin were efficiently mixed and dispersed for 30 min by ultrasonic vibrator (KQ-100, China), and the mixture was put in turbidity test bottle, finally the turbidity (NTU) of product was measured by turbidity meter (WGZ-B, China).

The morphology of products was observed and analyzed by SEM (Zeiss Supra55VP, German). FT-IR (PerkinElmer, USA) was adopted to assess the functional group of product by pressing KBr slice, and scanning range was set from 400 to 4000 cm^{-1} .

Results and discussions

3.1. Effect of different concentrations of $\text{Mg}(\text{NO}_3)_2$ solution on the dispersion of $\text{Mg}(\text{OH})_2$

Settling ratio is often used to evaluate the stability of inorganic powder in dispersion system. Normally, the dispersion of powder with small settling volume and long settling time is better; on the contrary, the dispersion of powder with big settling volume and short settling time is worse [19].



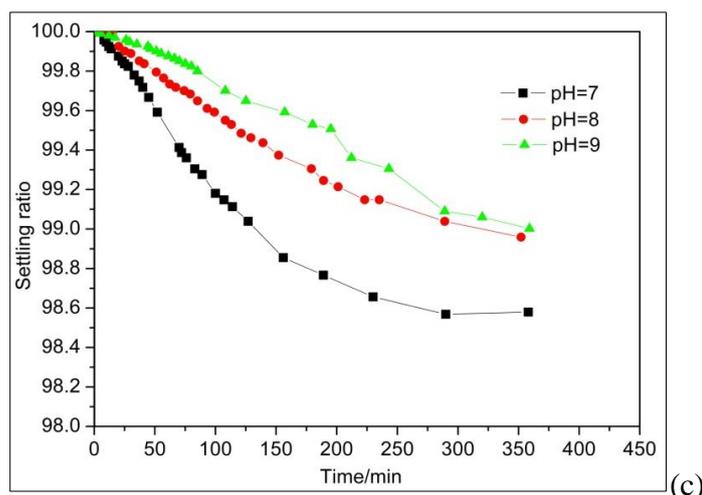


Figure 1. Effect of different reaction conditions on the dispersion of $\text{Mg}(\text{OH})_2$
 (a) $\text{Mg}(\text{NO}_3)_2$ with different concentrations, (b) different reactive time, (c) different pH values

Figure 1a shows the settling ratio curves of $\text{Mg}(\text{OH})_2$ prepared at different concentrations of $\text{Mg}(\text{NO}_3)_2$, in which the reaction conditions includes 4h of reaction time, 60°C of bath temperature, 7 of pH value. It can be observed from the figure that with the increase of $\text{Mg}(\text{NO}_3)_2$ concentration, the settling ratio of $\text{Mg}(\text{OH})_2$ declines obviously. When the concentration of $\text{Mg}(\text{NO}_3)_2$ is 0.05 or 0.1 mol/L, the settling ratio curve is almost a straight line with the extension of time, which is very close to 100 %, and it is very clear that the dispersion of the product is very excellent. When the concentration of $\text{Mg}(\text{NO}_3)_2$ increases to 0.5 mol/L, in the first 60 min the precipitation rate of the product is faster and the settling ratio decreases sharply; while the test time is 60 min, the settling ratio can reach about 70 %, and then the ratio reduces steadily with the increase of time, achieving approximately 50 % at 300 min.

The reason is that when the concentration of $\text{Mg}(\text{NO}_3)_2$ was low, the amount of available Mg^{2+} was limited relatively, the synthesis speed of $\text{Mg}(\text{OH})_2$ was slow and the crystal nucleus grew slowly, which urged the particle size of the product more uniform and the dispersity better, thus the agglomeration performance was weakened, and it was not easy to precipitate, so the settling ratio changed very slowly. With the enhancement of $\text{Mg}(\text{NO}_3)_2$ concentration, the amount of Mg^{2+} reacting with the exchangeable ion OH^- on the ion exchange resin increased obviously, which could cause more nuclei easily to agglomerate, and then more $\text{Mg}(\text{OH})_2$ was produced rapidly, resulting in the increase of particle size and the poor dispersion.

3.2. Effect of different reactive time on the dispersion of $\text{Mg}(\text{OH})_2$

Figure 1b presents the settling ratio curves of $\text{Mg}(\text{OH})_2$ prepared under different reaction time when the concentration of $\text{Mg}(\text{NO}_3)_2$ is 0.5 mol/L, and the pH value of the mixture is about 7 in a water bath at 60°C . It can be seen that no matter how long the reaction time is, the settling ratio of the product drops sharply in the first 50 min, and then decreases slowly. When the settling time reaches 360 min, the settling ratio is almost above 92% under the reaction time of 1 h; when the reaction time reaches 3 h, the settling ratio is reduced to about 75%, and the descending slope of the whole curve is larger relatively; when the reaction time is more than 4 h, the curve of settling ratio decreases slowly, which is alleviated compared with that when the reaction time is 3 h.

When the reaction time was 1 h, $\text{Mg}(\text{NO}_3)_2$ and ion exchange resin did not react completely; with the extension of reaction time, NO_3^- and OH^- on the resin could fully contact with each other, and when the reaction time was 3 h, the reaction was basically finished; when the reaction time was extended again, the ion exchange resin acted as a small “stirrer”, and under the effect of mechanical stirring, the resin particles and the product $\text{Mg}(\text{OH})_2$ were in full contact and friction with each other, so the extension of reaction time was extremely helpful to refine the product and improve the dispersion.

3.3. Effect of different pH values on the dispersion of Mg(OH)₂

Figure 1c describes settling ratio curves of Mg(OH)₂ prepared by 0.1 mol/L Mg(NO₃)₂ at different pH values, in which the reaction time is 4 h and the temperature of water bath is set to 60°C. It shows from this figure that with the increase of pH value, the settling ratio curve gradually moves upward; when the pH value of the mixture reaches 9, the settling ratio is still as high as 99% for 350 min testing, which illustrates that the increasing of pH value is conducive to enhance the dispersion of the product. This is because the pH range for the complete precipitation of Mg(OH)₂ is 9.4-12.4 [20]. Therefore, when the pH value is close to 9, it is helpful for the generation and growth of Mg(OH)₂ crystal nucleus.

3.4. Characterization of SEM

Figure 2 exhibits Scanning Electron Microscope (abbreviated as SEM) images enlarged 10000 times of the product obtained at 60°C water bath, by mechanical stirring for 0.05 mol/L Mg(NO₃)₂ reacting with ion exchange resin for 1, 4 and 6 h, respectively. It shows that no matter how long the reaction time is, the products present whisker shape. When the reaction time is 1 h, the whisker length is about 1 μm and the width is around 100 nm, and the phenomenon of partial agglomeration occurs. When the reaction time continues to extend to 4 h, the product presents clearer whisker morphology, the width is basically unchanged, but the length is up to 10 μm, and the dispersion is slightly improved compared with the former. Under the reaction time of 6 h, the width of the product increases slightly, but the length decreases to 4-6 μm. Hence, the reaction time has a certain influence on the homogeneity and tendency of the products. The reason is that the extension of reaction time is conducive to the gradual growth of the product, but a more intense mechanical stirring mode for too long time will destroy the formed whiskers, and make the whiskers fold into several sections, and meanwhile the products could gradually grow along the transverse direction of the whiskers, so as to increase the width of the whiskers.

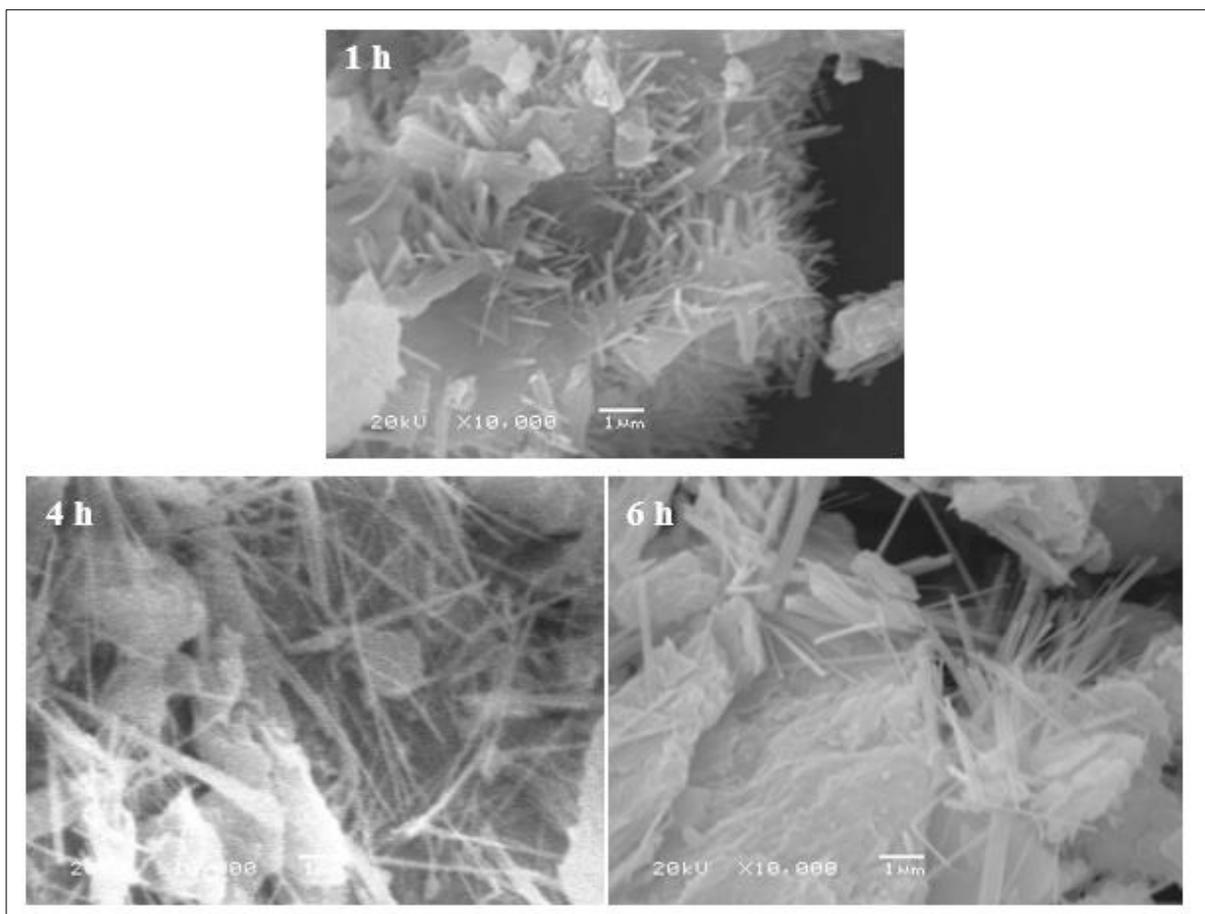


Figure 2. SEM images of the products prepared at different reaction time with 0.05mol/L Mg(NO₃)₂

In order to compare the effect of different concentrations of $\text{Mg}(\text{NO}_3)_2$ on the morphology of $\text{Mg}(\text{OH})_2$, the $\text{Mg}(\text{OH})_2$ prepared by 0.3 mol/L $\text{Mg}(\text{NO}_3)_2$ was characterized by SEM, as shown in Figure 3. It can be seen that after increasing the concentration of $\text{Mg}(\text{NO}_3)_2$, the $\text{Mg}(\text{OH})_2$ obtained at different reaction time is still of whisker shape with a length of 2-4 μm and a width of 300-800 nm, and the width is slightly bigger than that of the product shown in Figure 2. Generally speaking, its dispersion is better than that shown in Figure 2. This is due to the increase of $\text{Mg}(\text{NO}_3)_2$ concentration is good to sufficiently provide more Mg^{2+} and produce more $\text{Mg}(\text{OH})_2$, thus further increasing the width of whisker. Furthermore, the frequent collision and friction between more $\text{Mg}(\text{OH})_2$ and resin particles and between $\text{Mg}(\text{OH})_2$ crystalline grains are fairly beneficial to the refinement, dispersion, and homogeneity of products.

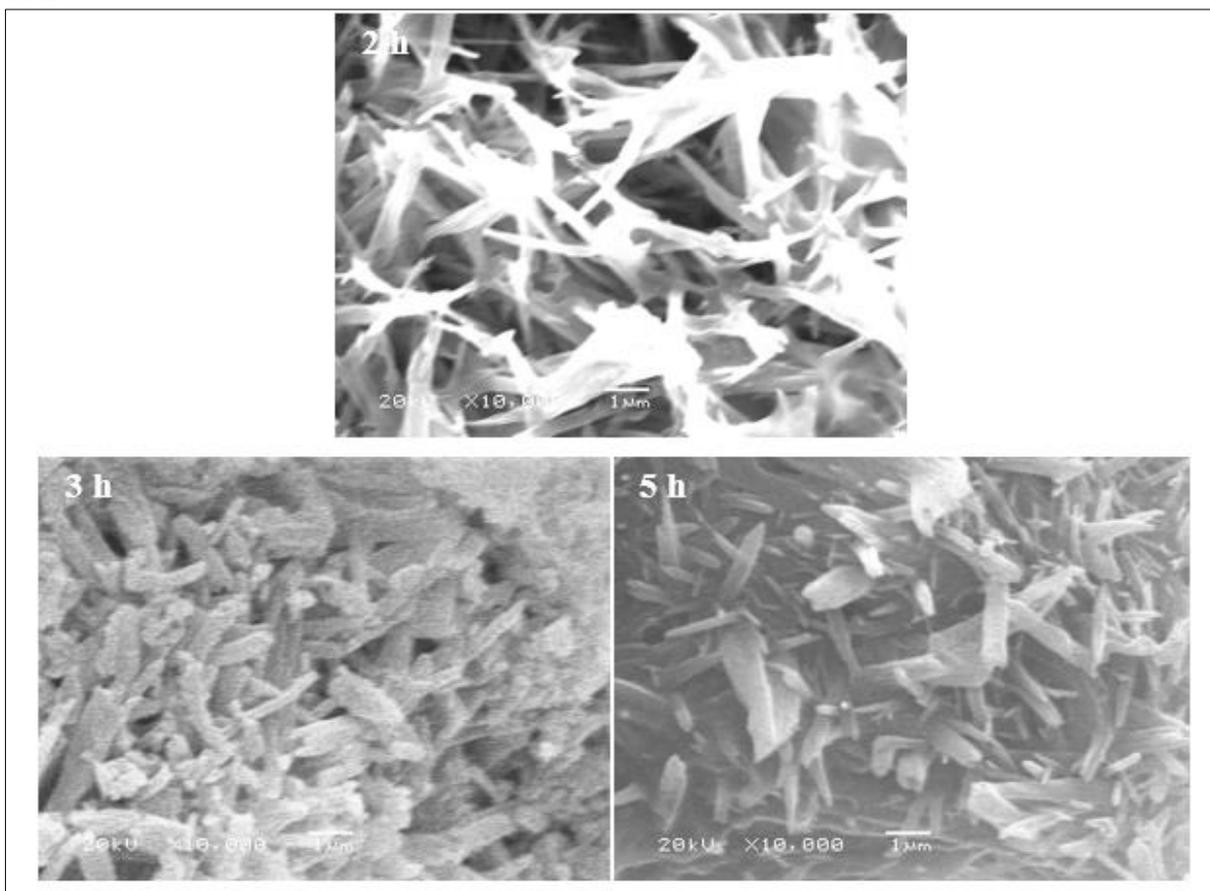


Figure 3. SEM images of the products prepared at different reaction time with 0.3mol/L $\text{Mg}(\text{NO}_3)_2$

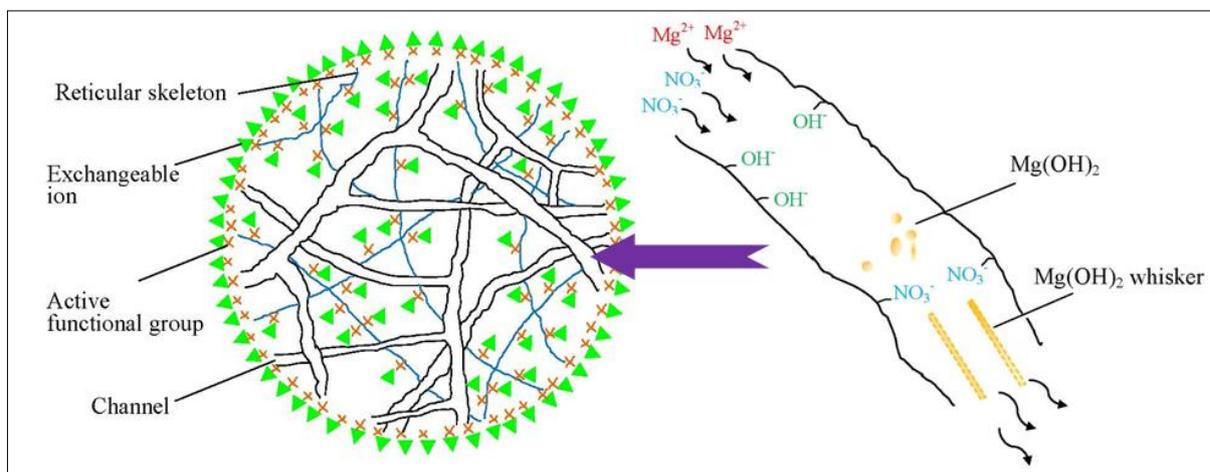


Figure 4. Structure of ion exchange resin and generation process of $\text{Mg}(\text{OH})_2$

In addition, D301 resin is a kind of macroporous anion exchange resin, and there are so many channels inside the resin particles, as shown in Figure 4. There are exchangeable ions on the inner wall of the channels, and the channel size of the wetting resin is between 100 and 500 nm [21]. The bare ion radius and hydrated radius of Mg^{2+} , OH^- , NO_3^- are listed in Table 2 [22]. It is thus clear that Mg^{2+} , OH^- and NO_3^- could migrate freely in the channel of the resin. When Mg^{2+} and NO_3^- migrated into the channels, OH^- could be released uniformly and slowly inside the ion exchange resin [23] which promoted the replacement reaction of NO_3^- with these OH^- , and NO_3^- was replaced and fixed on the resin, while OH^- and Mg^{2+} combined rapidly to form $Mg(OH)_2$ and then $Mg(OH)_2$ naturally entered into the solution. A schematic diagram of the generation process of $Mg(OH)_2$ is shown in Figure 4. Consequently, the narrow and long channels inside the resin could restrict the further growth of the product $Mg(OH)_2$, which could exactly regulate the morphology of the product.

During the preparation of $Mg(OH)_2$, compared with the amount of $Mg(NO_3)_2$, the amount of ion exchange resin is excessive, which ensures that Mg^{2+} and OH^- can fully react, so that there is no other impurity ions mixed in the solution after the reaction, and the purity of the product is well guaranteed. That is to say, the reaction is a “solid-liquid” reaction, and the transfer of NO_3^- is from liquid to solid. The ion exchange resin can firmly grasp NO_3^- , so that NO_3^- cannot enter the solution to interfere with the final product. Therefore, the product after the reaction is only $Mg(OH)_2$, so that the product can be directly separated from the ion exchange resin by filtration, so as to obtain the desired product with high purity without washing the product repeatedly. This is just one of the characteristics of $Mg(OH)_2$ prepared by ion exchange resin method.

Table 2. Bare ion radius and hydrated radius

Ion	Bare ion radius /nm	Hydrated radius /nm
Mg^{2+}	0.065	0.428
OH^-	0.176	0.300
NO_3^-	0.264	0.335

3.5. Study on the lipophilicity of the product

Turbidity can indirectly characterize the dispersion stability of the product in liquid medium. If the solid-liquid compatibility is good, the powder is evenly dispersed in the liquid medium and it is not easy to settle, which causes that the suspension has high turbidity, and the results show that the product is lipophilic; on the contrary, if the solid-liquid compatibility is poor, it is easy to settle for the powder in the liquid medium and the turbidity is low, which indicates that the dispersion of the powder in this liquid medium is poor comparatively.

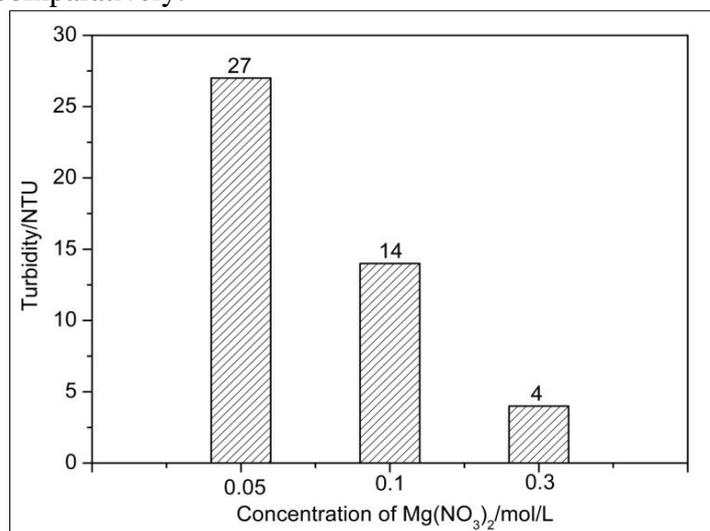


Figure 5. Turbidity measurement results of the product $Mg(OH)_2$ in paraffin

The lipophilicity of the product is studied in liquid paraffin with $\text{Mg}(\text{NO}_3)_2$ of 0.05, 0.1 and 0.3 mol/L, respectively, and the turbidity measurement results are depicted in Figure 5. It can be clearly conveyed that with the decline of the concentration, the turbidity of the product in paraffin rises gradually from 4 to 27 NTU (called Nephelometric Turbidity Unit), namely, the dispersion and lipophilicity of the product in oily medium strengthen gradually. This result can be explained from the fact that some functional groups or fragments on the resin, such as $-\text{N}(\text{CH}_3)_2$, styrene, divinylbenzene or individual benzene ring, had entered into the solution and were doped in the final product within 4 h reaction time. These substances played a vital role in surface modification of the product $\text{Mg}(\text{OH})_2$. When the concentration of $\text{Mg}(\text{NO}_3)_2$ was low, the amount of product as-obtained was less, by comparison, the modification effect was good and obvious; on the contrary, when the concentration of $\text{Mg}(\text{NO}_3)_2$ was high relatively, the amount of product as-obtained was more, the modification effect was poor naturally. It is this modification that increases the lipophilicity of the product. It can be highlighted that the ion exchange resin can not only provide crystallographic ions and control the morphology of the product, but also contribute to the modification of the product.

3.6. Characterization of FT-IR

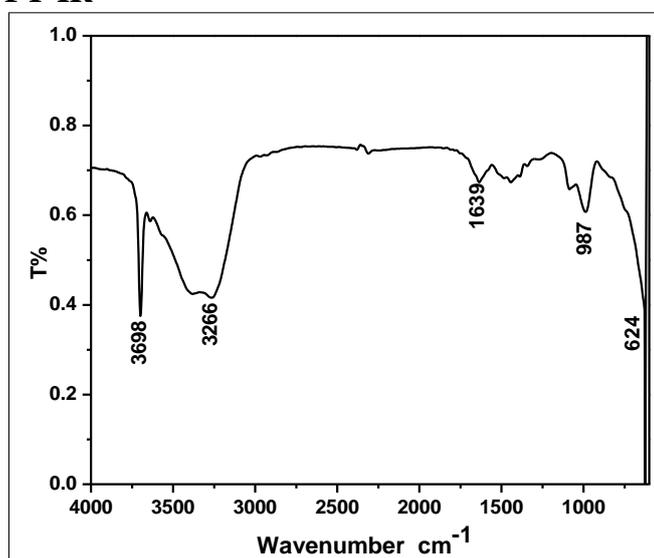


Figure 6. FT-IR spectrum of the product $\text{Mg}(\text{OH})_2$

At present, infrared spectroscopy is often used in the characterization of inorganic materials. The detailed structure information of the tested material can be obtained by the number, position, shape and intensity of infrared bands.

In this study, in order to further analyze the composition of the product, the product prepared with 0.05 mol/L $\text{Mg}(\text{NO}_3)_2$ and 4h reaction time was analyzed by Fourier Transform Infrared Spectrometer (abbreviated as FT-IR), and the spectrum result is displayed in Figure 6. The peaks at 3698 and 3266 cm^{-1} correspond to the characteristic stretching vibration ones of $-\text{OH}$ on the surface of $\text{Mg}(\text{OH})_2$ [24]; the absorption peaks near 1639 cm^{-1} belong to the vibration ones of benzene ring skeleton; the absorption peak at around 987 cm^{-1} attribute the characteristic peak of tertiary amino $-\text{N}(\text{CH}_3)_2$; the absorption peaks below 800 cm^{-1} show the lattice vibration ones of O-Mg-O .

Through the analysis of the above absorption peak position, it can be confirmed that there are a certain amount of benzene ring and tertiary amine group in the product, and the benzene ring and tertiary amine group are exactly the skeleton component and active functional group of D301 ion exchange resin, respectively. Consequently, this result can fully display that in the reaction process, after collision and friction of ion exchange resins, some functional groups or fragments on the resin would enter into the final product, which plays an effective modification for the product $\text{Mg}(\text{OH})_2$. In addition, this result



also explains the lipophilicity of the obtained products. Generally, this preparation method has a potential significance for the synthesis of additive $\text{Mg}(\text{OH})_2$ flame retardant.

4. Conclusions

In summary, $\text{Mg}(\text{OH})_2$ whiskers were successfully synthesized from D301 ion exchange resin and $\text{Mg}(\text{NO}_3)_2$ by mechanical stirring. Through the measurement and comparison of settling ratio, the results show that the $\text{Mg}(\text{OH})_2$ prepared with 0.05 or 0.1 mol/L $\text{Mg}(\text{NO}_3)_2$, reaction time more than 4 h and pH value of about 9 has excellent dispersion; SEM characterization showed that all the products prepared with different concentration and reaction time could reach nanometer size, and the products using 0.05 mol/L $\text{Mg}(\text{NO}_3)_2$ and reaction time of 4 h had whisker structure with length of 10 μm , and they had good dispersion in liquid paraffin; FT-IR analysis presented that the product contained some functional groups or fragments on the resin, which played a crucial role in modification the product. Therefore, the ion exchange resin can not only provide crystallographic ions and control the morphology of the products, but also contribute to the modification of the products. In addition, by controlling the dosage ratio of ion exchange resin and $\text{Mg}(\text{NO}_3)_2$, the as-prepared product can be used directly without washing. This study expands the application of ion exchange resin in the preparation of inorganic nano materials, and provides a simple green preparation method of inorganic materials, which is worthy of further research and promotion.

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