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Optimization of Preparation Conditions of Nanometer Magnesium Hydroxide

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Abstract

Nanometer magnesium hydroxide was prepared by positive drop method using magnesium chloride hexahydrate as raw material and sodium hydroxide as precipitant, and its preparation process was optimized. The results of single factor experiments show that the optimized process conditions are as follows: reaction temperature 40˚C, stirring speed 900 rpm, initial concentration of magnesium chloride C = 1 mol/L, concentration ratio of magnesium chloride to sodium hydroxide solution n (Mg^{2+}): n (OH^-) = 1:2, reaction time T = 60 min, drop acceleration of sodium hydroxide solution 2 ml/min.

Subject Areas

Biochemistry

Keywords

Nanometer Magnesium Hydroxide, Magnesium Chloride, Sodium Hydroxide, Positive Drip Method

1. Introduction

Nanomaterials refer to materials with at least one-dimensional nanometer scale (1 - 100 nm) in three-dimensional space, it is a new generation of materials composed of nanoparticles with sizes between atoms, molecules and macroscopic systems [1]. It has attracted wide attention because of its unique surface and interface effects, quantum size effects, small size effects and macroscopic effects. It plays an important role in medicine [2] [3] [4], biodiesel, biogas, biological hydrogen and bioethanol production [5], environmental protection [6] [7], agriculture and so on. As a kind of nano-materials, nano-magnesium hydroxide is a kind of environment-friendly inorganic nano-materials, which has the characteristics of strong adsorption capacity, large specific surface area and low cost. It is widely used in environmental protection [8] [9], flame retardant fillers [10] [11] [12], medicine [13] [14], agriculture, construction [15] [16] [17] and energy storage [18] [19] [20]. At present, there are many methods to prepare nano-magnesium hydroxide, but most of them will be added to control the particle size and other factors, which will increase the impurities in the final magnesium hydroxide [21]. For this reason, this paper uses the positive drop method to synthesize directly (that is, adding sodium hydroxide solution directly to magnesium chloride solution) without adding any catalyst or surfactant. The effects of reaction conditions such as reaction temperature, reaction time, stirring speed, solution ratio, solution concentration and sodium hydroxide solution drop acceleration on the particle size of magnesium hydroxide were investigated [22], and the experimental results were discussed. It is hoped to provide a rough reference for the preparation of nanometer magnesium hydroxide.

2. Experimental Procedure

All the reagents below are analytically pure.

Configure $MgCl_2$ solution of 1 mol/L 75 mL and NaOH solution 75 mL of 2 mol/L, the $MgCl_2$ solution was placed in a 250 ml conical bottle and preheated at 40˚C for half an hour, then the NaOH solution was dripped into the $MgCl_2$ solution, stirred con-tinuously at the speed of 900 rpm during the dripping period, and continued stirring for 4 hours after the dripping was finished. The product was centrifuged at the speed of 10,000 rpm, and the precipitated are washing with distilled water and centrifuged for three times, and dried continuously in an oven at 80˚C for 12 hours. After grinding, the nanometer magnesium hydroxide powder is obtained [23]. The obtained magnesium hydroxide products were configured into 0.005% solution, and the particle size was detected by Malvern laser particle sizer, and the average particle size was measured three times under each condition to get the final result.

In order to optimize the process of preparing nanometer magnesium hydroxide to achieve the minimum particle size by direct precipitation method, six groups of ex-periments were carried out to investigate the effects of reaction temperature, stirring time, stirring speed, magnesium ion concentration, magnesium ion/hydroxide ion ratio and sodium hydroxide drop acceleration on the particle size of magnesium hydroxide materials. Under the condition that all six experiments were completed, four factors significantly related to the particle size of magnesium hydroxide were selected, and the orthogonal experimental system was established to obtain the experimental conditions with the smallest particle size.

3. Results

3.1. Effect of Stirring Speed on Particle Size of Materials

The stirring speed determines the mixing uniformity of sodium hydroxide and magnesium chloride during the reaction and the degree of ion collision in the reaction, which has a great influence on the nucleation and agglomeration of magnesium hydroxide crystals. Under the same other conditions, the stirring speed was changed to 300, 600, 900, 1200 and 1500 rpm respectively to explore the effect of stirring speed on the product of magnesium hydroxide, and the optimal condition was selected with particle size as the screening condition. It can be seen from **Figure 1** that when the stirring speed is in the range of 300 - 900 rpm, the particle size of magnesium hydroxide decreases with the increase of stirring speed, and the particle size changes very fast. This is because the stirring speed is too slow, it is easy to make the magnesium hydroxide close to each other to adhere to each other, thus forming a larger magnesium hydroxide, and with the increase of the stirring speed, the movement between magnesium hydroxide powders is accelerated and the occurrence of agglomeration is effectively reduced. When the stirring speed is in the range of 900 - 1500 rpm, the particle size of magnesium hydroxide increases gradually with the increase of stirring speed, but the increase is slow. This is because the rapid stirring speed accelerates the collision between magnesium hydroxide powders, which leads to the possibility of magnesium hydroxide agglomeration. It is known from the results that the average particle size is the smallest when the stirring speed is 900 rpm.

3.2. Effect of Reaction Temperature on Particle Size of Materials

The reaction temperature will affect the movement rate and supersaturation of the particles in the reaction, as well as the crystal precipitation in the whole reaction system, and have a certain influence on the production of magnesium hydroxide. Under the same other conditions and changing the stirring condition to 900 rpm (the optimal stirring condition), the reaction temperature was changed to room temperature (20˚C), 40˚C, 60˚C, 80˚C and 100˚C respectively. The

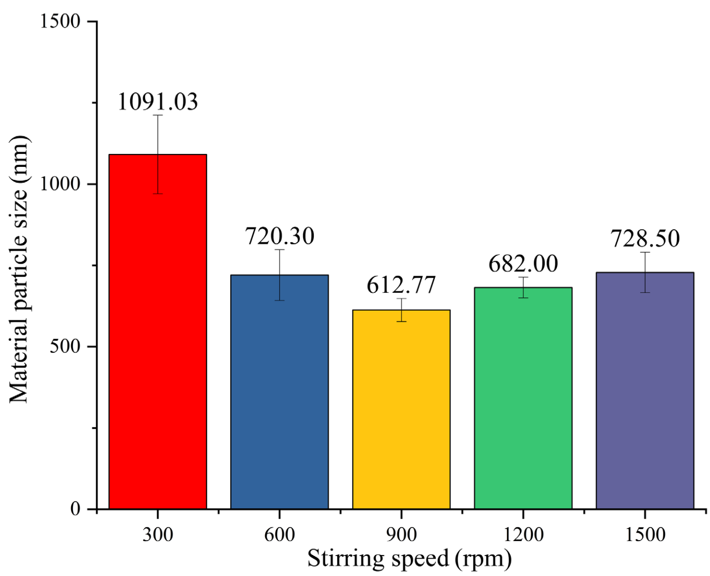

Figure 1. Effect of stirring speed on magnesium hydroxide.

effects of reaction temperature on the particle size, density and yield of magnesium hydroxide were investigated, and the optimal conditions were selected with particle size as the screening condition. It can be seen from **Figure 2** that the particle size at room temperature is larger than that at 40˚C. This is because when the temperature is low, the particle movement rate is smaller and the diffusion rate is slower. And the crystal nucleation rate is small, so the number of core crystals is small, the product particle size is large, with the increase of temperature, the particle movement rate is faster, and nucleation is easier. After 40˚C, the particles move too fast, which increases the collision probability, so that magnesium hydroxide is adsorbed by the crystal core to form individuals with larger particle size. According to the experimental results of reaction temperature, the particle size of magnesium hydroxide obtained at 40˚C is the smallest, so the reaction temperature at 40˚C is chosen in the follow-up experiment.

3.3. Effect of Stirring Time on Particle Size of Materials

When the stirring speed and reaction temperature are constant, the collision possibility of the particles is equal, and the stirring time affects the sufficient degree of the reaction. Under the same other conditions and selecting the above two optimal conditions, the stirring time was changed to 1, 2, 4, 8, 16 and 24 hours respectively, in order to explore the effect of stirring time on magnesium hydroxide, and the optimal conditions were selected with particle size as the screening condition. When the stirring time is 1 h, the reaction is incomplete and it is also because the stirring time is short and the crystal is not reunited during nucleation, so the reaction is stopped, resulting in a smaller particle size. At 2 and 4 hours, with the extension of stirring time, the reaction between sodium hydroxide and magnesium chloride is gradually complete, the extension of stirring time leads to more likely collision and agglomeration after crystal

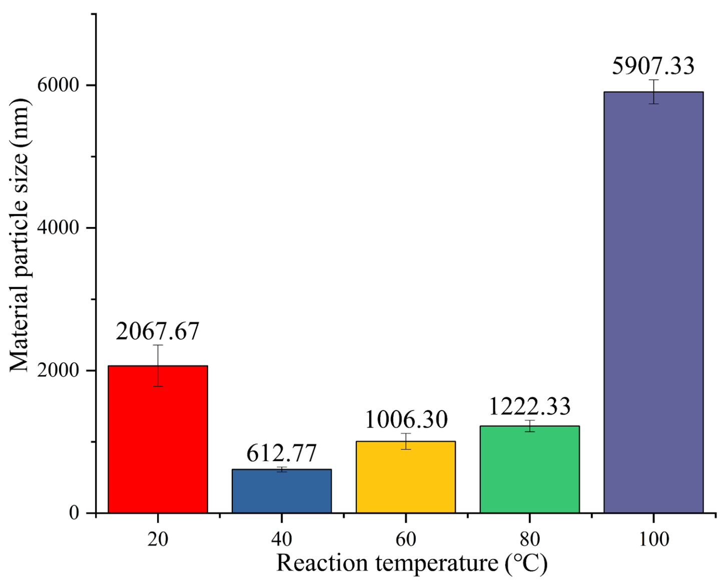

Figure 2. Effect of reaction temperature on magnesium hydroxide.

nucleation, which increases the particle size of magnesium hydroxide powder. After 4 hours, the reaction between sodium hydroxide and magnesium chloride was complete, and with the extension of stirring time, the particle size of magnesium hydroxide decreased, and the average difference of particle size at 8, 16 and 24 hours was very small. It is inferred that the number of crystals attached to each crystal nucleus decreases with the increase of the number of magnesium hydroxide nucleation after 4 hours. It is known from **Figure 3** that the stirring time of the smallest particle size is 1 h, although the particle size still has a trend of decreasing after 8 h, but the trend is not obvious, and the further increase of stirring time is not in line with the principle of energy saving, so the final stirring time is 1 h.

3.4. Effect of the Ratio of Magnesium Ion to Hydroxide Ion on the Particle Size of Materials

The difference of ion ratio will affect the pH of the reaction system, the collision probability of particles in the system and the reaction end point. In the chemical re-action, the ratio of magnesium ion to hydroxide ion is 1:2. If n (Mg^{2+}): n (OH^-) > 1:2, it is recorded as sodium hydroxide hyperstoichiometry reaction, otherwise, it is recorded as sodium hydroxide deficiency. Under the same other conditions and the optimal conditions of stirring time, stirring speed and reaction speed, the ion ratio is changed to make it to be 1:1, 1:1.5, 1:2, 1:2.5, 1:3. And take the particle size and ion utilization rate as the screening conditions to select the optimal conditions for follow-up experimental operation. According to the different ratio of particles, when the volume and ion concentration of magnesium chloride solution remain the same, there are two cases in which the ion ratio of magnesium ion and hydroxide ion can be changed to meet the required requirements. The first is to change the concentration of sodium hydroxide

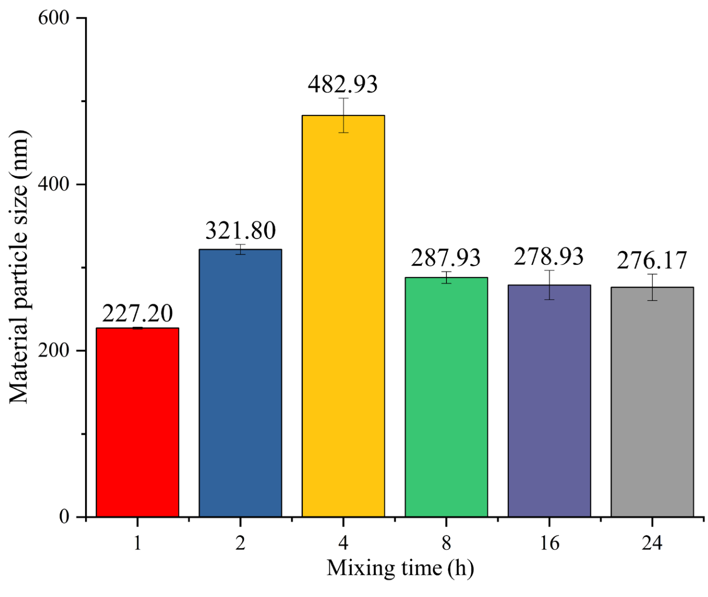

Figure 3. Effect of stirring time on magnesium hydroxide.

without changing its volume. In this case (**Figure 4**), the particle size of magnesium hydroxide powder first decreases and then increases with the increase of hydroxide ion concentration, and the particle size is the smallest when n (Mg^{2+}): n (OH^-) = 1:2. This is due to the different concentration of sodium hydroxide and magnesium chloride, which leads to the change of pH of the solution at the end of the reaction, thus affecting the particle size.

The second is to change the volume of sodium hydroxide solution without changing the concentration, so as to achieve the purpose of controlling the ion ratio (**Figure 5**). In this case, when n (Mg^{2+}): n (OH^-) = 1:1.5, the particle size of

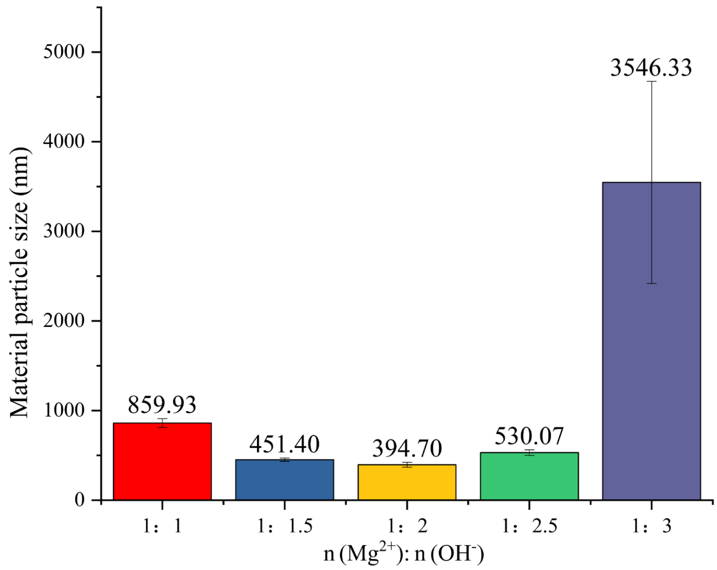

Figure 4. Effect of n (Mg^{2+}): n (OH^-) on magnesium hydroxide without changing the volume of sodium hydroxide solution.

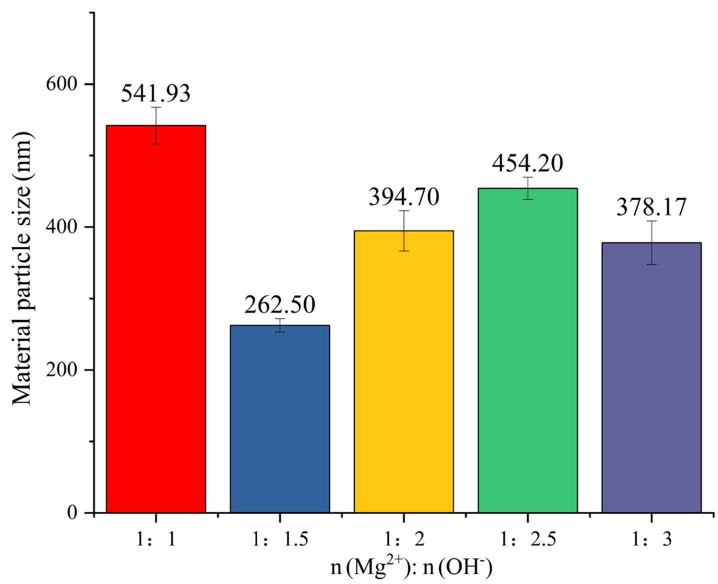

Figure 5. Effect of n (Mg^{2+}): n (OH^-) on magnesium hydroxide when changing the volume of sodium hydroxide solution.

magnesium hydroxide powder is the smallest, but the content of magnesium chloride in the waste after centrifugation is redundant, which is not in line with the concept of environment and resource conservation, and there is too much OH^- or Mg^{2+} in the solution, which leading to large ion product, high saturation and poor dispersibility of the solution.

3.5. Effect of Magnesium Ion Concentration on Particle Size of Materials

The effect of Mg^{2+} concentration on the particle size of the obtained powder was investigated without changing the volume of n (Mg^{2+}): n (OH^-) and magnesium chloride solution. After exploring the previous conditions, continue to explore the effect of magnesium ion concentration on magnesium hydroxide, configure the magnesium chloride solution of 1.5 mol/L, and dilute it into 5 parts to 0.5, 0.75, 1, 1.25 and 1.5 mol/L respectively, at the same time, configure the corresponding sodium hydroxide concentration according to the optimal ion ratio, and carry on the reaction, select the optimal condition with the particle size as the screening condition, and carry on the follow-up operation. **Figure 6** shows that with the increase of Mg^{2+} concentration, the particle size of magnesium hydroxide powder increases at first, then decreases and then increases. It is speculated that when Mg^{2+} concentration is 0.5 mol/L, the collision probability between particles is small, and the reaction is not complete, so the yield is small, it is not easy to form new nuclei, and the particle size is small. In the range of 0.5 - 0.75 mol/L, the reaction tends to be complete with the increase of Mg^{2+} concentration, but the nucleation rate is still low. Therefore, the particle size of the product increases, and in the range of 0.75 - 1 mol/L, the crystal is easier to form new nuclei, and the concentration of the crystal in the solution is suitable, so the particle size is smaller. After the increase of Mg^{2+} concentration to 1 mol/L, the

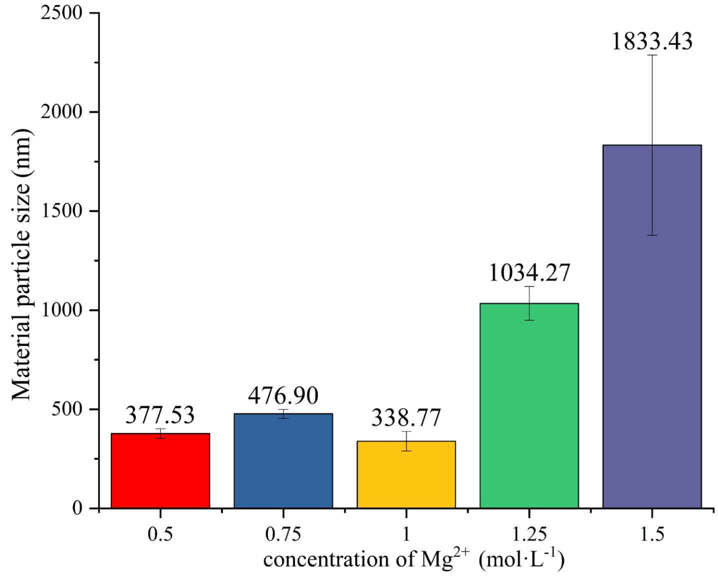

Figure 6. Effect of Mg^{2+} concentration on magnesium hydroxide.

particle size of the obtained powder increases obviously, which is inferred that the collision probability increases and agglomeration occurs because the product concentration is too high. The final concentration of Mg^{2+} was selected as 1 mol/L according to the final particle size detection results.

3.6. Effect of Sodium Hydroxide Drop Acceleration on Particle Size of Materials

The drop acceleration of NaOH solution affects the supersaturation of the reaction system, thus affecting the performance of magnesium hydroxide [10]. Under the condition that the optimal experiments were selected in the above five experiments, the dripping rate of sodium hydroxide was changed to 0.5, 1, 1.5, 2 and 2.5 ml/min respectively to explore the effect of sodium hydroxide dripping speed on the properties of magnesium hydroxide. As can be seen from **Figure 7**, the relationship between the particle size of magnesium hydroxide and the drop acceleration of sodium hydroxide solution is that with the increase of the drop acceleration of sodium hydroxide, the particle size of the powder increases at first, then decreases and then increases, and when the drop acceleration is 2 $ml \cdot min^{-1}$, the particle size of the powder is the smallest.

3.7. Orthogonal Test

According to the six experimental results, four factors including reaction temperature, stirring speed, magnesium ion concentration and NaOH solution dripping speed were selected for orthogonal experiment. Under each factor, three levels were selected, the reaction temperature was 20˚C, 40˚C, 60˚C, the stirring speed was 600, 900 and 1200 rpm, and the magnesium concentration was 0.75, 1, 1.25 $mol \cdot L^{-1}$. The three levels of drop acceleration of NaOH solution are 1.5, 2 and 2.5 $ml \cdot min^{-1}$ respectively, and the particle size is taken as the experimental evaluation index. The results are shown in the following table (**Table 1**).

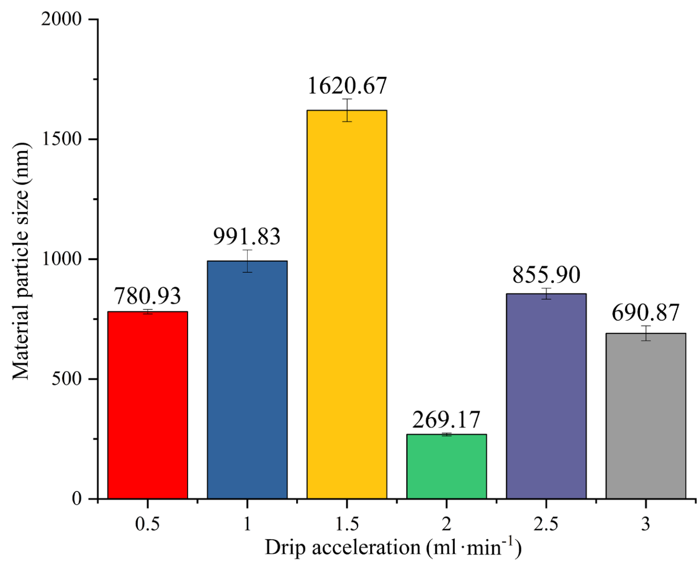

Figure 7. Effect of dripping rate of NaOH solution on magnesium hydroxide.

Table 1. Results of orthogonal experiment.

	Drop acceleration of NaOH solution/ml·min^{-1}	Stirring speed/rpm	Reaction temperature/˚C	Magnesium ion concentration/mol·L^{-1}	Average particle size/nm
	2.5	1200	20	1	4122.67
	2	900	20	1.25	5760.33
	1.5	600	20	0.75	496.7
	2.5	600	40	1.25	537.57
	2	1200	40	0.75	3222.33
	1.5	900	40	1	328.77
	2.5	900	60	0.75	510.27
	2	600	60	1	302.57
	1.5	1200	60	1.25	1019.433
K1	1844.9	1336.83	10379.7	4229.3	
K2	9285.23	6599.37	4088.67	4754	
K3	5170.5	8364.43	1832.27	7317.33	
k1	614.97	445.61	3459.9	1409.77	
k2	3095.078	2199.789	1362.889	1584.67	
k3	1723.5	2788.144	610.7556	2439.11	
R	2480.111	2342.533	2849.144	1029.34	

From the experimental results, it is known that the influence degree of the four in-fluencing factors on the experimental results is temperature > dripping speed > ro-tational speed > ion concentration. Because the smaller the particle size is, the better the particle size is, so the levels of the four influencing factors selected in this experiment are temperature 60˚C, dripping rate 1.5 mL·min^{-1}, rotational speed 600 rpm, magnesium ion concentration 0.75 mol·L^{-1}.

However, through the verification experiment, it is found that the particle size of the material not only does not decrease, but increases, so the experimental data are re-adjusted, and two experimental schemes are tried, one is the minimum particle size condition in the orthogonal experiment, that is, temperature 60˚C, drip acceleration 2 mL·min^{-1}, rotational speed 600 rpm, magnesium ion concentration 1 mol·L^{-1}, the other is temperature 60˚C, drip acceleration 1.5 mL·min^{-1}, rotational speed 600 rpm, the concentration of magnesium ion is 1 mol·L^{-1}. Results the nanometer magnesium hydroxide with an average particle size of 235 nm was successfully prepared under the conditions of temperature 60˚C, dripping speed 2 ml·min^{-1}, rotational speed 600 rpm and magnesium ion concentration 1 moL·L^{-1}. The optimum experimental conditions were determined as follows: temperature 60˚C, dripping speed 2 mL·min^{-1}, rotational speed 600 rpm, magnesium ion concentration 1 mol·L^{-1}.

3.8. Definition of Nanomaterials

In this paper, the particle size of the sample is mainly measured by the laser particle size analyzer, and the results are of reference significance, but the size measured by the Malvern laser particle size analyzer does not directly reflect the sample particle size, resulting in the minimum particle size is not within the 100 nm. In order to ensure that the samples are nanomaterials, the samples of single factor experiments with stirring speed of 900 and 1200 rpm are randomly selected for scanning electron microscope observation (**Figure 8**). The test magnification is 50,000 times. The test results are as follows. As can be seen from the picture, the prepared magnesium hydroxide is hexagonal flake, and one-dimensional size in three-dimensional space is smaller than 100 nm, which accords with the characteristics of nanomaterials, so the prepared magnesium hydroxide can be called nanomaterials.

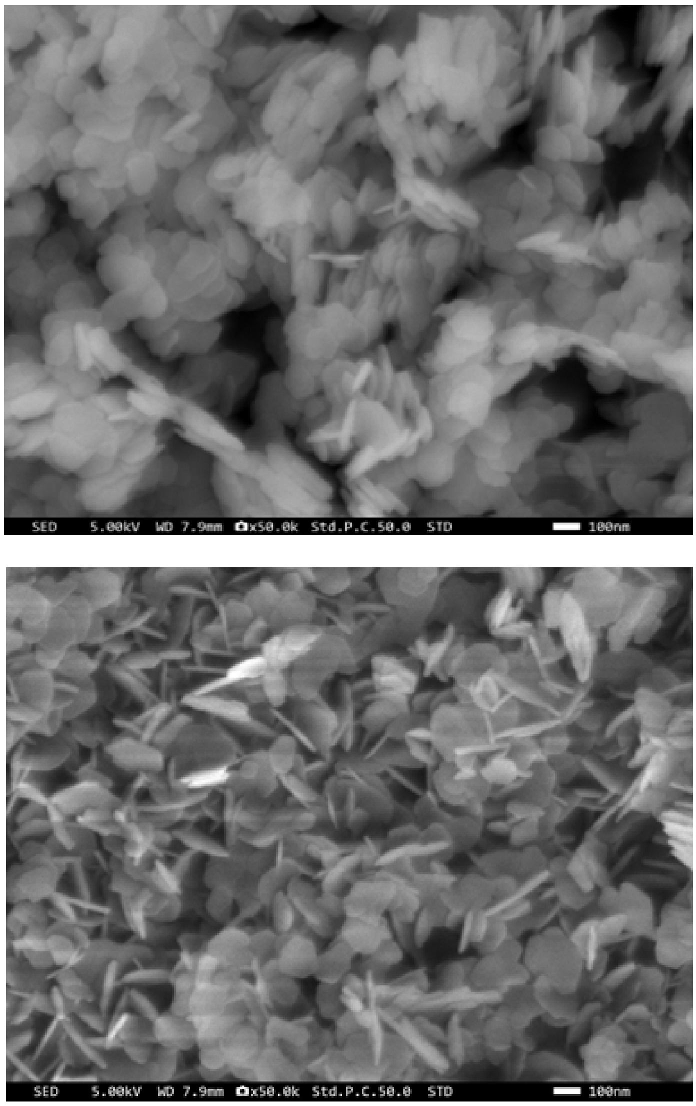

Figure 8. Comparison of scanning electron microscope pictures of sodium hydroxide. Left is stirring speed 900 rpm; right is stirring speed 1200 rpm.

4. Discussion and Conclusion

In this paper, magnesium hydroxide is prepared from sodium hydroxide and magnesium chloride solution by the positive drop method of direct precipitation method, there are no catalyst and surfactant are added in the preparation process. And there are many materials for the preparation of magnesium hydroxide, for example, we can add ammonia or ammonia water to the solution containing magnesium ion [24], or we can do it by mixing magnesium sulfate and sodium hydroxide solution. There are also many preparation methods of magnesium hydroxide, such as hydrothermal method [21] [24] [25], lime method, etc., but the control conditions are roughly the same, including stirring time, reaction temperature, stirring speed, material ratio, ion concentration, material addition rate and so on. Some methods for the preparation of catalysts and surfactants are also controlled by the conditions of the amount of catalysts or surfactants. Improving the production process of magnesium hydroxide and strengthening the research on its process will help to achieve accurate control of the particle size and morphology of the product in the preparation method and process, and prepare high-standard nano-materials through cheap magnesium compound resources and strengthen the application of its products in water treatment, flame retardant filling, medicine, agriculture and other fields, which have a great impact on the economy, society and environment.

Based on the above results and charts, we can draw a conclusion that the single factor optimization conditions of dropping sodium hydroxide into magnesium chloride by direct precipitation method are as follows: reaction temperature 40˚C, stirring speed 900 rpm, initial concentration of magnesium chloride C = 1 mol·L^{-1}, concentration ratio of magnesium chloride solution to sodium hydroxide solution n (Mg^{2+}): n (OH^-) = 1:2, reaction time T = 60 min, drop acceleration of so-dium hydroxide solution 2 ml·min^{-1}. The optimum conditions of orthogonal ex-periment were as follows: temperature 60˚C, dripping rate 2 ml·min^{-1}, rotational speed 600 rpm, concentration of magnesium ion 1 mol·L^{-1}, ratio of magnesium chloride solution to sodium hydroxide solution n (Mg^{2+}): n (OH^-) = 1:2, reaction time T = 60 min.

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Conflicts of Interest

The authors declare no conflicts of interest.

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