Synthesis and Shape Evolution of Monodisperse Basic Magnesium Carbonate Microspheres

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ABSTRACT: A self-assembled spherical-like basic magnesium carbonate ($Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$) with diameters of 15−17 μm was synthesized in an aqueous solution system without using any organic additives during the precipitation process. Reaction parameters such as reaction time, stirring time, and reaction temperature were found to be important in controlling the final morphology of $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$. By the investigation of the particles from time-dependent experiments, scanning electron microscopy observations and Fourier transform infrared spectra indicate that the formation of these spherical-like $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ is via a complex process, in which the agglomerates built by many fine grains are first produced after the "burst" mixture of K_2CO_3 and $Mg(NO_3)_2$ solutions. Then the agglomerates tend to assemble into needlelike or other similar particles, and the latter, due to their unstable properties from the thermodynamic point of view, will transfer into sheetlike particles, which further assemble into spherical-like particles. During such a process, their corresponding compositions also change from $(MgCO_3)_{0.8}(Mg(OH)_2)_{0.2} \cdot 1.3H_2O$ to $MgCO_3 \cdot xH_2O$, then to $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$. In addition, the stirring time and reaction temperature were found to play a comparative role in determining the final morphology of $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$, and the morphology transition from spherical-like to nest-like, then to layered particles, as well as the decrease of particle size, can be well obtained by carefully adjusting the stirring time (0.5−4 min) of the initial solution and reaction temperature (333−363 K).

1. Introduction

The preparation of monodisperse microspheres with uniform shapes and sizes has been of growing interest due to the utilization for various types of functional application fields, such as chromatographic packing and electronic, optical, and chemical devices.[1] Although there have been a large number of techniques, such as spray drying,[2] oil emulsion drying strategy,[3] and hydrolysis of metal alkoxide,[4] for preparing monodisperse microspheres, the uniformity in shape and size has not been satisfied in most techniques. For example, when these approaches are applied to fabricate monodisperse particles, particular attention should be paid to the emulsification conditions for generating droplet size, to ascertain whether particle classification is demanded after synthesis, or to prepare and handle the costly and hazardous metal−organic precursors. To the best of our knowledge, a precipitation method may be an alternative approach for synthesis of monodisperse microspheres with uniform size based on its various advantages such as a single-step process, simple facility, and facile scale-up. In recent years, many attempts have been successfully made to prepare different spherical particles by precipitation. For example, Boyle et al.[5] have synthesized monodisperse spherical magnesium bis-*o*-cresol (or *o*-methylphenoxide) by the use of seed particles to control nucleation events during precipitation. Bai et al.[6] have prepared highly cross-linked narrow or monodisperse poly-(divinylbenzene) (PDVB) microspheres by distillation−precipitation polymerization in acetonitrile with 2,2′-azobis(2-methyl-propionitrile) (AIBN) as initiator. Soler-Illia and co-workers[7] have demonstrated spherical equally sized mixed copper−zinc basic carbonates and Zn-doped tenorite particles prepared by

precipitation from copper−zinc nitrate solutions. These examples illustrate that a precipitation method may be a promising route for the synthesis of monodisperse particles with narrow size distribution.

$Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$, as an important mineral in geology and planetology, has been widely used in various industrial fields (e.g., pharmaceuticals, cosmetic manufacturing, rubber industry, lithographing inks, and as precursors for other magnesium-based chemicals[8]). To date, a large number of chemical methods including hydrothermal treatment,[9] carbonation,[10] and precipitation,[11] have been developed to generate various morphologies of $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$. However, the report on preparation of monodisperse $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ microspheres with uniform shapes and sizes via a facile route are rare. In a recent study,[12] we have found that various morphologies of magnesium carbonate hydrates (including $MgCO_3 \cdot xH_2O$ and $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$) can be synthesized by carefully adjusting the reaction temperature and pH value of the initial reaction solution in a $MgO−CO_2−H_2O$ system during the precipitation process. Namely, at temperatures below 328 K and lower pH values, magnesium carbonate hydrates are prone to display needlelike $MgCO_3 \cdot xH_2O$, and the regular $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ built of sheetlike crystallites becomes the preferable morphology at reaction temperatures above 333 K and higher pH values. These results demonstrate that spherical $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ with uniform size may be produced at a higher temperature and pH value. Herein, we report on the synthesis of uniform $Mg_5(CO_3)_4$-$(OH)_2 \cdot 4H_2O$ microspheres by the reaction of K_2CO_3 and Mg-$(NO_3)_2$ at temperatures above 333 K and a higher pH value during precipitation. By the investigation of the particles from various reaction times, the shape evolution of these spherical-like particles is discussed in detail. In addition, we also demonstrate that the morphology of $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ is very sensitive to the stirring time of the initial reaction solution and reaction temperature.

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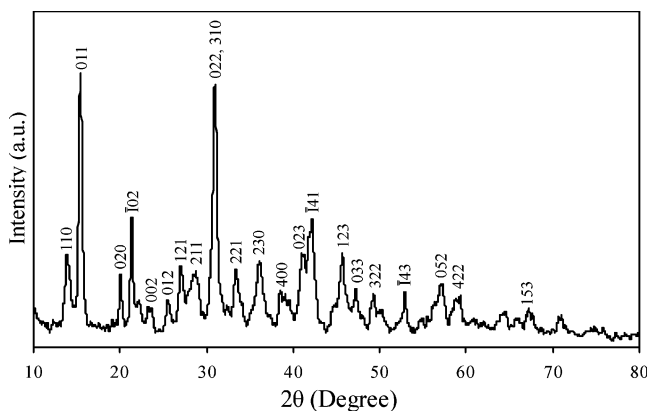**Figure 1.** XRD pattern of the as-synthesized spherical-like $Mg_5(CO_3)_4$-$(OH)_2 \cdot 4H_2O$ prepared at 353 K with a stirring time of 0.5 min.

2. Experimental Section

2.1. Preparation of $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ Microspheres. In a typical procedure, 10.26 g of $Mg(NO_3)_2 \cdot 6H_2O$ was dissolved in 50 mL of double-deionized water, and the pH value was adjusted to 7.5 with dilute KOH solution. Then, the $Mg(NO_3)_2$ solution was transferred to a 250 mL three-necked flask and heated to 353 K in a water bath. Subsequently, 100 mL of 0.4 M K_2CO_3 solution with pH = 11.8 at T = 353 K was rapidly added into the vigorously stirred (ca. 900 rpm) $Mg(NO_3)_2$ solution in 4−5 s. The mixture was further stirred for 0.5 min and then maintained at the temperature for 1 h under static conditions. After that, a white precipitate was collected, filtered off, and washed with double-deionized water and ethanol several times. The MgO sample was prepared by calcination of the spherical-like Mg_5-$(CO_3)_4(OH)_2 \cdot 4H_2O$ in air from room temperature to 573 K with a rate of 2 K/min, then keeping 2 h. After that, the product was calcined from room temperature to 873 K in muffle furnace and then maintained at that temperature for 10 h.

2.2. Characterization. The crystal structures of these products were characterized by X-ray diffraction (XRD) on a D_8 Advance diffractometer using Cu Kα radiation; the operation voltage was 40 kV, and the current was 40 mA. The morphology and size were examined by scanning electron microscopy (SEM), and the images were taken with a JSM-6360LV scanning electron microscope. The Fourier transform infrared (FT−IR) spectra of the obtained samples were recorded with a Perkin-Elmer GS-II FT−IR spectrometer in the range of 4000−400 cm^{-1}; the resolution was 4 cm^{-1}, and 8 scans were signal-averaged in each interferogram. Thermogravimetric analysis (TGA) was performed on a Perkin-Elmer SII Diamond TG-DTA instrument. The operating temperature was raised from 303 to 973 K with the rate of 10 K/min under N_2 atmosphere, and the used sample size was about 4 mg.

3. Results and Discussion

Powder XRD and SEM Observation. The crystal structures of the as-synthesized products were confirmed by XRD. As shown in Figure 1, all diffraction peaks in the XRD pattern can be indexed as monoclinic structure of $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ with unit cell parameters of a = 10.11, b = 8.94, and c = 8.38 Å and β = 114.58°, which are in good agreement with the reported data (JCPDS Card 25-513). The shapes of the diffraction peaks illustrate that the $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ sample is well crystallized.

The morphology and size of these $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ particles and their corresponding calcined sample were visualized by SEM, and the representative images are presented in Figure 2. As can be observed, the $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ particles exclusively exhibit spherical-like morphology (Figure 2A), and the spheres have size typically in the range of 15−17 μm. By the magnification image of an individual sphere in Figure 2B, it is interesting that the spherical-like $Mg_5(CO_3)_4$-$(OH)_2 \cdot 4H_2O$ looks like self-assembly by both hemispheres, and

there is a clear suture in the middle of the particle. Additionally, these particles are mainly composed of nanosheet-like structures (Figure 2C), and the surface structure is very similar to that given in the previous reports,[9,12,13] indicating that $Mg_5(CO_3)_4$-$(OH)_2 \cdot 4H_2O$ crystal nuclei tend to assemble into a sheetlike structure in the $MgO-CO_2-H_2O$ system. After calcination of the $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ microspheres at 873 K for hours, the general morphology of the product obtained, which can be indexed as the cubic phase MgO (see Supporting Information SI-1), is displayed in Figure 2D. By comparison of these MgO microspheres with $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$, it can be found that both products, as a whole, possess similar morphology. However, careful observation can be found that there are a few exploding spheres in these particles (Figure 2D). From the detailed view of an individual exploding particle as shown in Figure 2E, the slit just lies in the suture of $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ as discussed above, and there are two empty hemicores similar to the form of butterfly in the center of these exploding particles, which is clear in Figure 2F. To our knowledge, the empty hemicores may come from the incomplete self-assembly of sheetlike structures in the formation of the spherical-like Mg_5-$(CO_3)_4(OH)_2 \cdot 4H_2O$, which leaves a hollow section in the center of these particles. During the calcination, the pressure from the remaining H_2O in the hollow section and the release of CO_2 and H_2O from the decomposition of $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$, will gradually increase with the increase of temperature. Consequently, the spherical-like particles are split into two parts from the suture in the middle of these particles, and two symmetrically empty hemicores are produced.

Shape Evolution of Spherical-like $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$. To obtain a better understanding of the formation and evolution of these spherical-like $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$, time-dependent experiments were carried out. Figure 3 shows the typical SEM images of these particles synthesized from various reaction times at 353 K. It is obvious that at the early reaction stage (2 min), the primary nanoparticles tended to form agglomerates built by the fine grains with a homogeneous distribution (Figure 3a,b). With the increase of reaction time up to 5 min, it is interesting that there is a serpentine strip with a length of 550 μm and a width of 20 μm among these agglomerates as shown in Figure 3c. After careful observation, it can be seen that there are sparse nestlike particles on the strip (Figure 3d). With the extension of the reaction time to 10 min, many splitting spherical-like particles with a diameter of ca. 15−17 μm are produced as shown in Figure 3e,f, but the agglomerates are still dominant in these particles. When the reaction time is up to 20 min, these particles are all transferred into uniform spherical-like particles with diameters of 15−17 μm (Figure 3g), and there is a minute slit in the middle of these particles. Upon further increase of the reaction time up to 30 min, the slit almost disappears as shown in Figure 3h. From these results, it can be learned that the formation of these spherical-like particles is mainly between the reaction time of 5−10 min, and the crystal seeds of the spherical-like particles are probably hatched from the strip as discussed above or other similar structures. More importantly, if the particles obtained from a stirring time of 2−10 min are dried at 323 K under vacuum conditions, the particles will transfer into other morphologies. For example, if the particles obtained by a stirring time of 5 min are dried at 323 K under vacuum conditions for hours, the agglomerates will be transferred into columnar and spherical-like particles (see Supporting Information SI-2). From the detained view of a typical columnar particle, it can be observed that some sheetlike and spherical-like particles are being hatched on the surface of the columnar
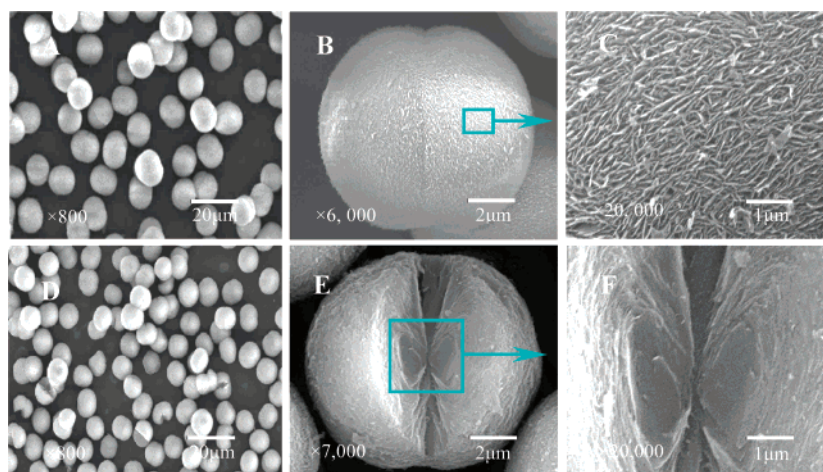
Figure 2. Representative SEM images of $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ synthesized at 353 K with a stirring time of 0.5 min (A−C) and MgO (D−F): (A, D) at low magnification; (B, E) individual sphere; (C, F) close-up image of an individual sphere.
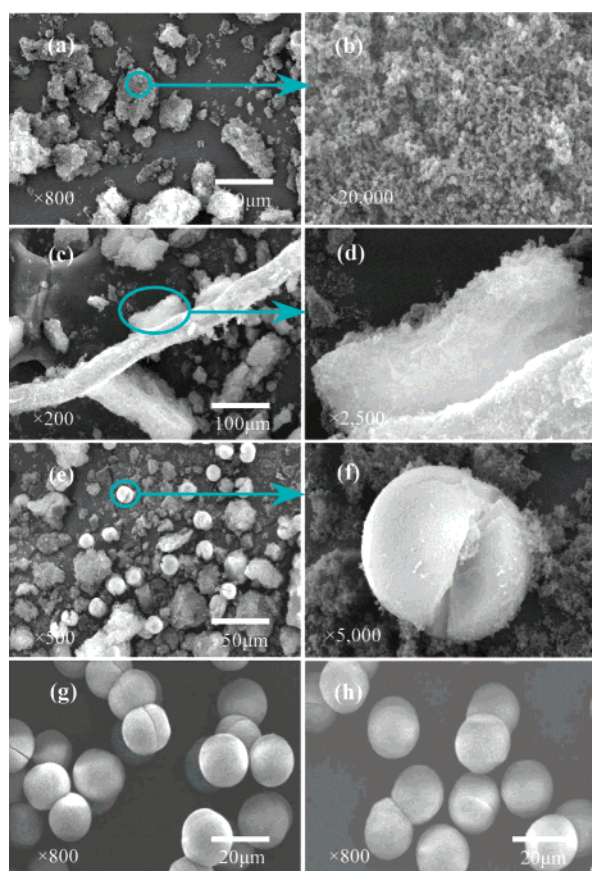

Figure 3. Representative SEM images of the particles from various reaction times at 353 K with a stirring time of 0.5 min: (a) 2 min; (b) the detailed view of panel a; (c) 5 min; (d) the large magnification of a particle in panel c; (e) 10 min; (f) the large magnification of a particle in panel e; (g) 20 min; (h) 30 min (note: samples a−f were dried by freeze-dried technique around 235 K, and the other samples were dried at 373 K in air).

particles, suggesting that the crystal seeds of the spherical-like $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ particles are from the columnar particles. From the results, we can speculate that the formation of the spherical-like particles may undergo a complex process, in which the agglomerates are first assembled into the columnar particles or other similar structures, but the latter is not stable in such a reaction system. With the further progress of the reaction, these structures transform into the more stable sheetlike structures, which will assemble into spherical-like particles.

As discussed above, the crystal seeds of these spherical-like particles are produced by the strip, columnar, or other structures, but not by the agglomerates. From the morphology evolution of magnesium carbonate hydrates (including $MgCO_3 \cdot xH_2O$ and $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$) with the variation of reaction temperature and pH value of the initial reaction solution,[12] only the needlelike, platelike, and other particles with sheetlike structures are captured in the present experimental conditions. In our opinion, the needlelike particles are very likely to be the crystal seeds of these spherical-like particles, and the captured columnar structures in Figure SI-2 may come from the needlelike particles, which can be partly confirmed by the report of Mitsuhashi et al. on the synthesis of $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ microtubes with a surface of "house of cards" structure via needlelike $MgCO_3 \cdot xH_2O$ particles.[10] The reason for difficultly capturing them can be attributed to the unstable properties of these needlelike particles in a higher reaction temperature.[8,10] For further confirmation of this assumption, the initial product of the needlelike particles prepared from a stirring time of 4 min at 313 K,[12] as well as the product by the heat treatment of the needlelike particles, is investigated in detail, which is presented in Figure 4. Figure 4a shows the initial product of the needlelike particles, which displays a similar morphology as the particles from a stirring time of 0.5 min at 353 K for 2 min (Figure 3a). When these particles are treated at 313 K for 20 min, the uniform needlelike particles with a surface structure of hexagonal prism (Figure 4b) were obtained.[12] For the investigation of their heat stability and corresponding heat treatment product, the needlelike particles are put into 100 mL of double-deionized water with a temperature of 353 K for minutes, and the typical SEM images of the obtained product are given in Figure 4c−f. It is observed that the needlelike particles are partly converted into columnar particles when the heat treatment time is 15 min (Figure 4c), and more interesting is that a few nestlike particles are being hatched on the surface of the needlelike and columnar particle. In addition, there are sparse large spherical-like particles on the surface of the columnar particles (the large magnification part of Figure 4c), which can be attributed that with the transition from the needlelike particles to the columnar ones, the trend of self-assembly of the sheetlike particles into the spherical-like ones also increases. When the heat treatment time is increased up to 25 min, these needlelike and columnar particles are all converted into sheetlike and spherical-like particles (Figure
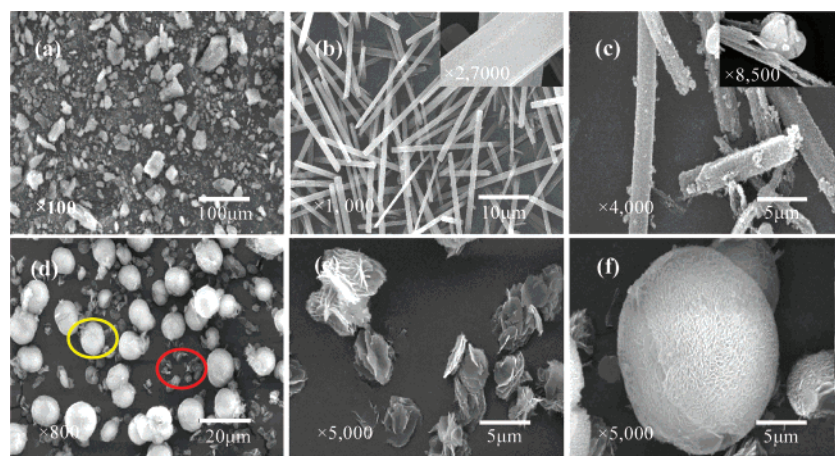
Figure 4. Representative SEM images of the particles from various reaction conditions: (a) the particles from a stirring time of 2 min at 313 K for 0 min; (b) the particles from a stirring time of 4 min at 313 K for 20 min; (c) the particles by adding the ones of panel b into 100 mL of double-deionized water with a temperature of 353 K for 15 min and (d) 25 min; (e) the large magnification of the red ellipse in panel d; (f) the large magnification of the yellow ellipse in panel d (note: sample a was dried by freeze-dried technique around 235K, and the other samples were dried at 373 K in air for hours).
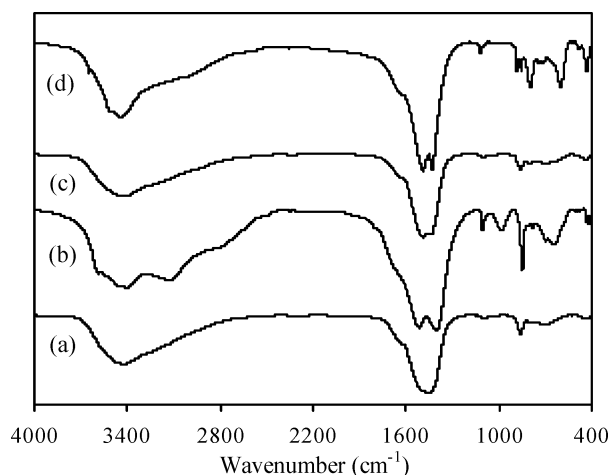

Figure 5. FT−IR spectra of the particles from various reaction conditions: (a) the particles from a stirring time of 2 min at 313 K for 0 min; (b) the particles from a stirring time of 4 min at 313 K for 20 min; (c) the particles by adding the ones of panel b into 100 mL of double-deionized water with a temperature of 353 K for 15 min and (d) 25 min.

4d−f). A more interesting result is that the spherical-like particle has an almost similar structure as the ones obtained from a stirring time of 0.5 min at 353 K for 60 min (Figure 2), and these results, to some extent, well confirm that the crystal seeds of the spherical-like particles are hatched from the needlelike particles.

In general, the particle shapes and their properties are closely related, and different forms of particles sometimes give different particle properties even for the same substances.[10] Figure 5 shows the corresponding IR spectra of these particles from various conditions at 313 and 353 K. It can be seen that the IR spectra of the initial product of the needlelike particles (Figure 5a) are different, according to our recent study,[12] from those of needlelike $MgCO_3 \cdot xH_2O$. Namely, there is no trace of the broad band at 3130 cm^{-1} in the present IR spectra, which may originate from different numbers of water of crystallization.[12] In addition, the split band of the CO_3^{2-} ν_3 antisymmetric stretching vibration exists as a single absorption band, which is associated, according to the report of Botha and Strydom,[14] with the amorphous phase. For further confirmation of the composition of these particles, the TG analysis was performed. After a calculation from the results of TG analyses (see Supporting Information SI-3A), these particles have a simple formula of $(MgCO_3)_{0.8}((Mg(OH)_2)_{0.2} \cdot 1.3H_2O$. When these particles are converted into the needlelike particles, the IR spectra can be assigned to the characteristic absorption of $MgCO_3 \cdot xH_2O$ (Figure 5b), which has been discussed in detail in our recent study.[12] After a heat treatment of these needlelike particles at 353 K for 15 min, a great change takes place in the IR spectra of the product (Figure 5c). For example, the bands at 3130 and 985 cm^{-1} have disappeared, and the bands at 1105 and 854 cm^{-1} become very weak. However, these spectra are similar to those of the particles from a stirring time of 2 min at 313 K for 0 min (Figure 5a). From these results and the corresponding morphological characterization (Figure 4c), their particles may be a mixture of $MgCO_3 \cdot xH_2O$ and $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$. Further increasing the heat treatment time up to 25 min, the product based on the IR spectra (Figure 5d) can be assigned to $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$,[8b,12,15] which can be further confirmed by the results of TG analysis (see Supporting Information SI-3B). From these results, it can be learned that the formation of these spherical-like particles at 353 K is via such a procedure as the following: the agglomerates built by the fine grains with a homogeneous distribution are first produced by the "burst" mixture of K_2CO_3 and $Mg(NO_3)_2$ solutions, and these particles tend to assemble into needlelike or other similar structures. However, these structures are not stable from the thermodynamic point of view,[8,10] and their structures undergo further rearrangement or self-assembly into the more stable spherical-like particles. During the process, their corresponding compositions also change from $(MgCO_3)_{0.8}((Mg(OH)_2)_{0.2} \cdot 1.3H_2O$ to $MgCO_3 \cdot xH_2O$ then to the more stable $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$. By the way, it should be noted that the IR spectra of the particles from various reaction times at 353 K with a stirring time of 0.5 min have a similar change pattern (see Supporting Information SI-4), suggesting that the formation process of the $Mg_5(CO_3)_4$-$(OH)_2 \cdot 4H_2O$ microspheres as discussed above is reasonable. Additionally, as reported in the literature,[10] the change from amorphous $xMgCO_3 \cdot Mg(OH)_2$ to $MgCO_3 \cdot xH_2O$ is said to pass through a process in which the amorphous particles dissolve and the needlelike hydrate redeposits. With the increase of pH value or temperature of the reaction solution,[8b,10] $MgCO_3 \cdot xH_2O$
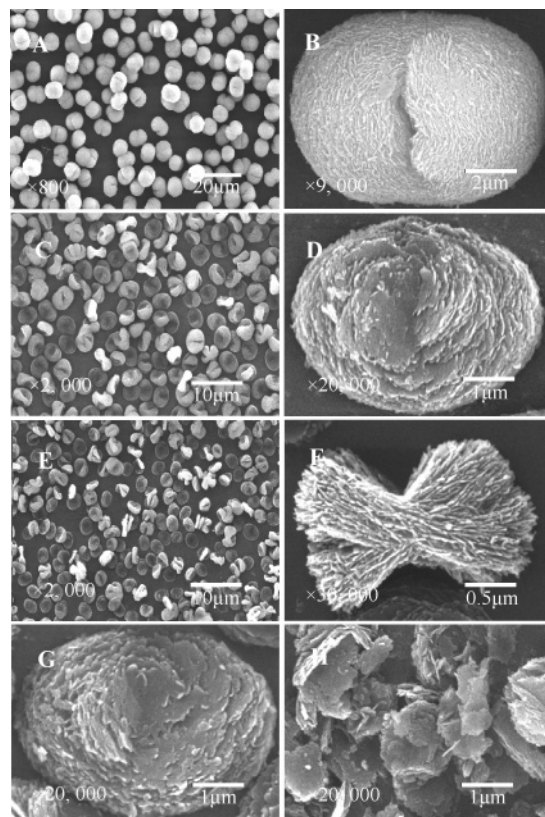
Figure 6. Representative SEM images of the particles from various stirring times at 353 K by different magnifications: (A, B) 1 min; (C, D) 1.5 min; (E−G) 2 min; (H) 4.0 min.

will transform into $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$, which further confirms the results discussed above.

Influence of Stirring Time and Reaction Temperature. The stirring time and reaction temperature were also found to play important roles in determining the final morphology and size of $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$. The typical SEM images of $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ synthesized from various stirring times at 353 K are displayed in Figures 2 and 6. The precipitate, after a stirring time of 0.5 min, is found to consist of uniform spherical-like particles with diameters of $15-17 \ \mu m$, and more interesting is a clear suture in the middle of these particles (Figure 2A,B). When the stirring time is increased to 1.0 min, as shown in Figure 6A, the particle shape is very similar to the sample obtained from 0.5 min. However, the size of the spherical-like particles decreases, and it becomes $10-11 \ \mu m$ in diameter. More importantly, the suture in the middle of the particles becomes a slight slit, which is obvious in Figure 6B. When the stirring time is increased up to 1.5 min, the SEM images in Figure 6C,D show multilayered nestlike $Mg_5(CO_3)_4$-$(OH)_2 \cdot 4H_2O$ with rather rough surfaces. There exists a majority of nestlike crystals and only a few dumbbell-like crystals in the product, and the longitudinal and transverse axes for the nestlike crystals are ca. 5 and 4 μm, respectively. It is remarkable that the middle section of the crystals is sunken and the thickness of each layer of the nestlike $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ is about 30 nm (Figure 6D), which has a similar surface structure as reported by Yan and Xue via a hydrothermal approach.[9a] Upon further increase of the stirring time up to 2 min, the nestlike crystals become very few, and the cakelike and dumbbell-like $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ are dominant, which is clear in Figure 6E. Each also has a multilayered structure similar to that shown in Figure 6C,D, but the surfaces of the cakelike and dumbbell-like crystals are rougher than those of the nestlike crystals

(Figure 6F,G). If the stirring time is continued to extend 4 min, the sheetlike particles are produced. As shown in Figure 6H, the sheetlike particles are obviously built by nanosheets with layer-by-layer. At the same time, by examining the influence of reaction temperatures ranged from 333 to 363 K with a stirring time of 1 min on the morphology of $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$, the particles also change from spherical-like to nestlike morphology, as well as showing the decrease of the diameter in the range of $15-6 \ \mu m$, and their corresponding SEM images are given in the Supporting Information SI-5. Therefore, for better governing the morphology of MgO precursors, the growth stage in the precipitation should be precisely controlled by carefully adjusting the stirring time of the initial reaction solution and reaction temperature.

4. Conclusions

In summary, we demonstrate a facile route for preparation of monodisperse $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ microspheres, which were obtained by the "burst" mixture of $Mg(NO_3)_2$ and K_2CO_3 during precipitation. To better understand the formation and shape evolution of these $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ microspheres, syntheses were carried out under various conditions. Reaction time, stirring time of the initial reaction solution, and reaction temperature were found to play important roles in determining the final morphology of $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$. By the time-dependent experiments, the results illustrate that the formation of $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ microspheres is via a complex process, in which the shapes of the products change from agglomerates to needlelike or other similar particles, then to the stable spherical-like particles, and their corresponding compositions also go through $(MgCO_3)_{0.8}((Mg(OH)_2)_{0.2} \cdot 1.3H_2O$ to $MgCO_3 \cdot xH_2O$ then to $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$. We believe that the knowledge not only greatly promotes our better understanding of the process of the primary nanoparticles into spherical-like $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$, but also provides an alternative way to prepare uniform MgO microspheres by calcination of $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$, which may find important applications as a packing material for high performance liquid chromatography.

Supporting Information Available: The XRD pattern of the particles by calcination of spherical-like $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$ prepared at 353 K with a stirring time of 0.5 min; the SEM images of the particles prepared from a reaction time of 5 min at 353 K with a stirring time of 0.5 min and dried under the vacuum condition at 323 K for hours; the TG and DTG profiles of the particles (A) from a stirring time of 2 min at 313 K for 0 min, and (B) the particles by adding the needlelike particles into 100 mL of double-deionized water with a temperature of 353 K for 25 min; the FT−IR spectra of the particles from various reaction times at 353 K with a stirring time of 0.5 min; and the representative SEM images of the particles from various reaction temperatures with a stirring time of 1 min. This material is available free of charge via the Internet at http://pubs.acs.org.

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