Asian Journal of Chemistry; Vol. 25, No. 8 (2013), 4389-4392

ASIAN JOURNAL OF CHEMISTRY

http://dx.doi.org/10.14233/ajchem.2013.13987

Facile Fabrication of Magnesium Oxide with Different Morphology

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(*Received*: 28 April 2012; *Accepted*: 11 February 2013) AJC-12971

Magnesium oxide nanomaterials have been fabricated using industrial magnesium oxide as raw material *via* precipitation. The XRD result indicated that basic magnesium carbonate [$4MgCO_3{\cdot}xMg(OH)_2{\cdot}yH_2O$, x = 0-1, y = 3-8] was obtained when aqueous magnesium carbonate was heated from 55-95 ºC. According to SEM measurement, different morphologies of basic magnesium carbonate [$4MgCO_3{\cdot}xMg(OH)_2{\cdot}yH_2O$], including rod, cotton stick and petal, could be achieved via varying the reaction temperature. And the morphologies were not destroyed after calcination of $4MgCO_3{\cdot}xMg(OH)_2{\cdot}yH_2O$ to magnesium oxide. Iodine adsorption method was used to test the activity of prepared magnesium oxide. The result showed that high active magnesium oxide was obtained at 85 ºC of reaction temperature, the iodine adsorption value of magnesium oxide was as high as 198 mg/g of MgO.

Key Words: Magnesium oxide, Basic magnesium carbonate, Precursor, Morphology.

INTRODUCTION

Magnesium oxide is a homomorphous compound with the rocksalt structure. Due to its ionic constituents consisting of a relatively small number of electrons, it is likely that MgO exhibits a limited number of reasonably well-defined surface defect structures and has stable oxidation states. Therefore, MgO has been extensively used in catalysis[1], toxic waste remediation[2], antibacterial materials[3], refractory material industries, paints and superconductor products[4]. Recently, many different synthesis methods provide MgO nanomaterials including deposition[5], combustion aerosol synthesis[6], sol-gel[7], hydrothermal[8], laser vaporization[9]. It is well-known that the morphologies of the precursors play an important role in the final morphology of the corresponding MgO materials. Xue *et al*.[10] synthesized different morphologies of MgO *via* precise morphological control of precursors. In previous works[11,12], it has been demonstrated that different morphologies of basic magnesium carbonate, including nanowhiskers and nanorod and spheres, were prepared. The morphology of the precursors will be well maintained in the final samples by controlling the calcination conditions properly. In the present work, magnesium oxide nanomaterials have been fabricated using industrial magnesium oxide as raw material *via* precipitation. The effect of reaction temperature on constituent of basic magnesium carbonate and its morphology was investigated.

EXPERIMENTAL

Magnesium oxide nanomaterials with different morphologies using industrial magnesium oxide as raw material were prepared as follows. 100 g of industrial magnesium oxide was added into 980 mL of 1.43 mol L^{-1} aqueous solution of sulphuric acid. After stirring at room temperature for 2 h, the mixed solution was filtrated and the filtrate was dropped into flask-3-neck, then NH_4HCO_3 solution was added ($MgSO_4$:NH_4HCO_3 = 1:2.5) under stirring at 45 ºC for 2 h. After that, 2 % of surfactant was added into the solution and increased reaction temperature to 55 ºC. After stirring at 55 ºC for 1 h, the mixed solution was filtrated. The obtained precipitation was washed using deionized water and ethanol, finally drying for 8 h. In order to obtained magnesium oxide, the obtained precipitation was calcined in an oven at 560 ºC for 2 h.

XRD patterns were recorded on a D/MAX, using CuK_α radiation, operating at 40 kV, 100 mA. The morphologys of the samples were characterized by scanning electron microscopy (SEM, JSM-6360LV type). The numbers of water comprised in basic magnesium carbonate were obtain from thermogravimetric analysis (TG, EXSTAR X-DSC7000) results and XRD patterms.

RESULTS AND DISCUSSION

Fig. 1 presents the XRD diagrams of basic magnesium carbonate prepared at reaction temperature between 55 and

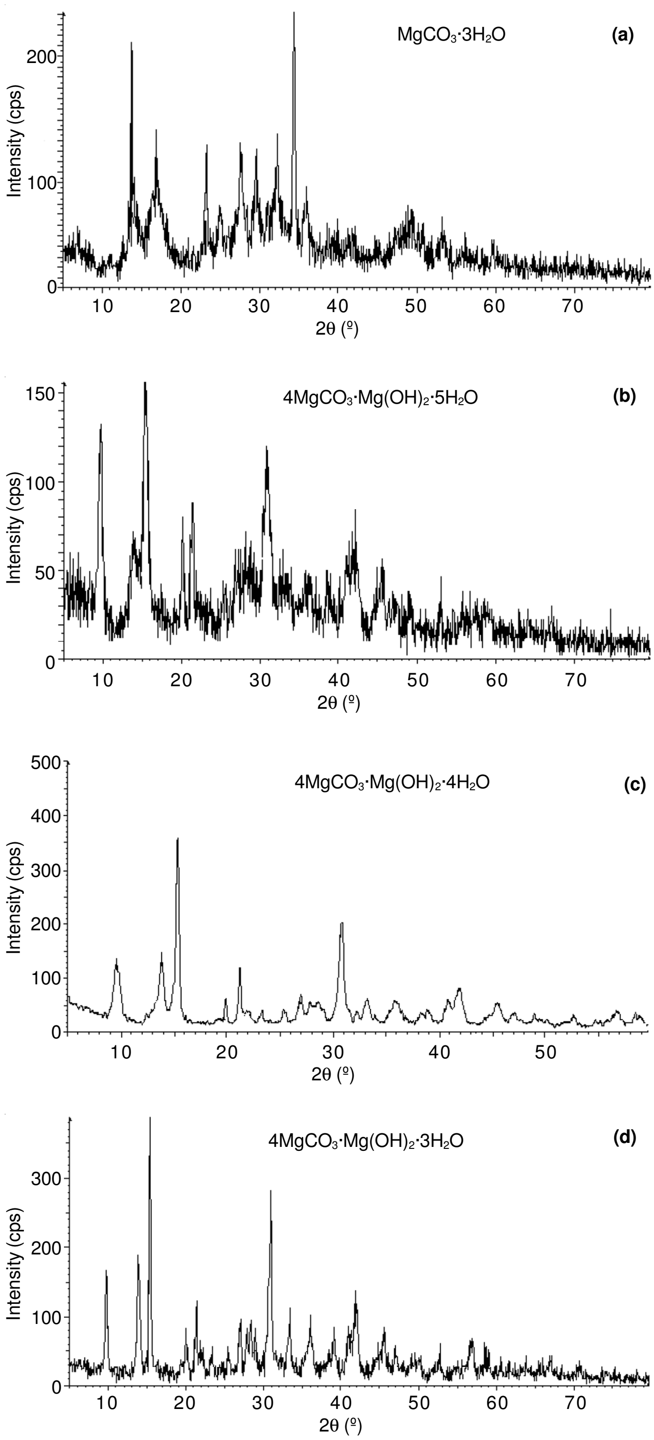


Fig. 1. XRD diagram of basic magnesium carbonate with different heating temperature

100 ºC. The results indicated that $MgCO_3 \cdot 3H_2O$ (Fig. 1a) and $4MgCO_3 \cdot Mg(OH)_2 \cdot 5H_2O$ (Fig. 1b) and $4MgCO_3 \cdot Mg(OH)_2 \cdot 4H_2O$ (Fig. 1c) was obtained at 55, 75 and 95 ºC, respectively. And further increasing reaction temperature to 100 ºC, $4MgCO_3 \cdot Mg(OH)_2 \cdot 3H_2O$ was obtained (Fig. 1d). This result showed that the reaction temperature dramatically affected the components of Mg based products synthesized. Song *et al.*[13], found that the rod-like crystal of $MgCO_3 \cdot 3H_2O$ could be obtained at 20 º, but $MgCO_3 \cdot 3H_2O$ was transferred to microsphere of $4MgCO_3 \cdot Mg(OH)_2 \cdot 4H_2O$ at 80 ºC. This phenomena was similar to that of previous experiment, although the morphology and transformation temperature were different compared with previous results[11,12].

Fig. 2 gives XRD pattern of Mg-based products calcined at 550 ºC. It can be seen that only magnesium oxide peaks were observed when Mg-based precusors were calcined at 550 ºC. This result indicated that Mg-based precusors were converted to MgO after calcination at 550 ºC.

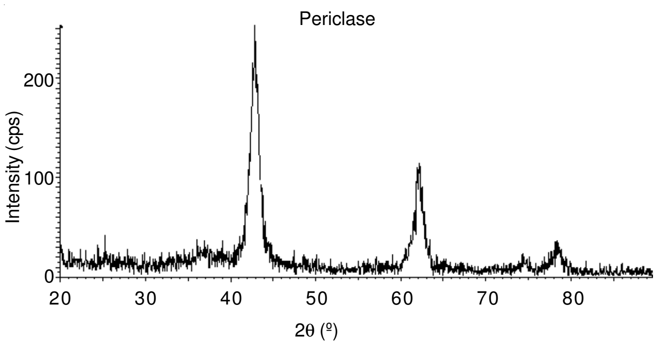
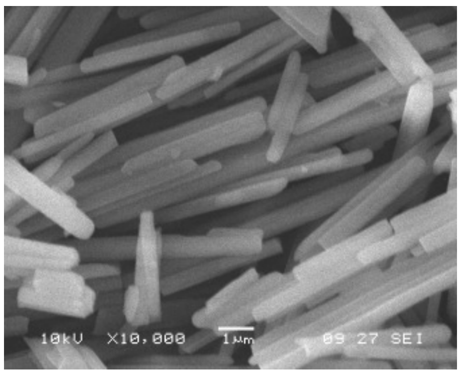
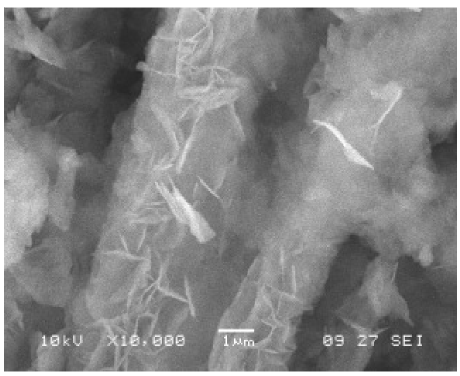


Fig. 2. XRD diagram of magnesium oxide

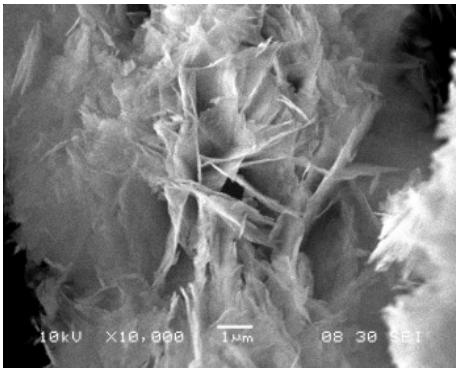
Fig. 3 gives SEM patterns of Mg-based precusors prepared at different reaction temperature. It can be seen that nanorods of basic magnesium carbonate were achieved at 55 ºC of reaction temperature (Fig. 3a). Further increasing reaction temperature, cotton stick was observed at 75 ºC of reaction temperature (Fig. 3b) and petaliform was obtained at 95 ºC of reaction temperature (Fig. 3c). However, the structure of basic magnesium carbonate was destroyed when the reaction temperature increased to 100 ºC (not shown here). This result showed that the morphology of basic magnesium carbonate could be tailored *via* varying reaction temperature.



(a)



(b)



(c)

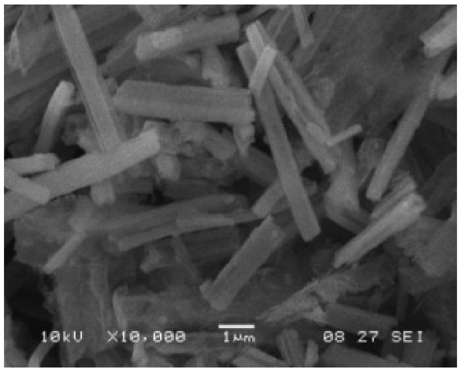
Fig. 3. SEM diagram of basic magnesium carbonate with different heating temperature

Table-1 showed the effect of reaction temperature on the crystal morphology and the numbers of water comprised in basic magnesium carbonate. It can be seen that the numbers of water comprised in basic magnesium carbonate increased when the reaction temperature increased from 55 to 80 ºC. Further increasing reaction temperature, the numbers of water in basic magnesium carbonate decreased with the increase of reaction temperature. It also can be seen that, rod-like $MgCO_3·3H_2O$ was obtained between 55-60 ºC, further increasing reaction temperature from 60-80 ºC, rod-like $MgCO_3·3H_2O$ was gradually converted to Cotton stick of $MgCO_3·Mg(OH)_2·5H_2O$ or $MgCO_3·Mg(OH)_2·8\ H_2O$. While the water comprised in basic magnesium carbonate decreased when the reaction temperature increased from 85 to 100 ºC. This result indicated that the high reaction temperature caused the loss of crystal water molecule.

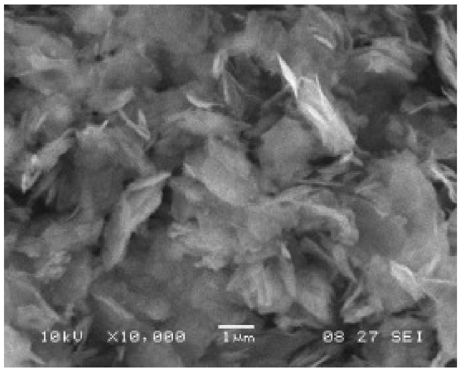
TABLE-1
EFFECT OF REACTION TEMPERATURE ON CRYSTAL MORPHOLOGY AND THE NUMBERS OF WATER COMPRISED IN BASIC MAGNESIUM CARBONATE

No.	T (ºC)	Chemical formula	Number of crystal water	Crystal morphology
1	55	$4MgCO_3·3H_2O$	3	Rod
2	60	$4MgCO_3·3H_2O$	3	Rod
3	65	$4MgCO_3·Mg(OH)_2·5H_2O$	5	Translation
4	70	$4MgCO_3·Mg(OH)_2·5H_2O$	5	Translation
5	75	$4MgCO_3·Mg(OH)_2·5H_2O$	5	Cotton stick
6	80	$4MgCO_3·Mg(OH)_2·8H_2O$	8	Cotton stick
7	85	$4MgCO_3·Mg(OH)_2·6H_2O$	6-7	Translation
8	90	$4MgCO_3·Mg(OH)_2·5H_2O$	5	Petal
9	95	$4MgCO_3·Mg(OH)_2·4H_2O$	4	Petal
10	100	$4MgCO_3·Mg(OH)_2·3H_2O$	3	Breaking

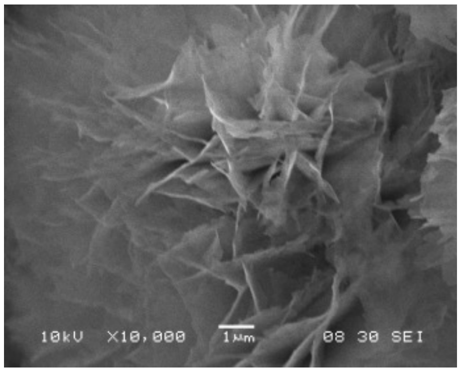
Fig. 4 presents the SEM patterns of magnesium oxide through calcining basic magnesium carbonate at 550 ºC. The results indicated that the rods, cotton sticks and petaliforms of magnesium oxide were obtained through calcining relative morphologies of basic magnesium carbonate. The rods of magnesium oxide (Fig. 4a) were obtained through calcination of rods of basic magnesium carbonate (Fig. 3a). And the petaliforms of magnesium oxide (Fig. 4c) could be obtained *via* calcination of the petaliforms of basic magnesium carbonate (Fig. 3c). This means that morphology of magnesium oxide can be easily fabricated by calcining the relative morphology of basic magnesium carbonate.



(a)



(b)



(c)

Fig. 4. SEM diagram of MgO derived from calcination of basic magnesium carbonate prepared at different reaction temperature

MgO can be achieved *via* calcining basic magnesium carbonate and iodine adsorption method is generally used to test the activity of magnesium oxide. Fig. 5 only gives iodine adsorption value of magnesium oxide prepared at the reaction temperature from 80 to 100 ºC, because small amount of iodine can be adsorpted as for magnesium oxide synthesized below 80 ºC. It can be seen that, with the increase of reaction temperature from 80 to 100 ºC, iodine adsorption value of magnesium oxide presents a single peak form and the highest active magnesium oxide was obtained at 85 ºC of reaction temperature, the iodine adsorption value of magnesium oxide was as high as 198 mg/g of MgO. This means that high active magnesium oxide can be facilly achieved *via* varying reaction temperature.

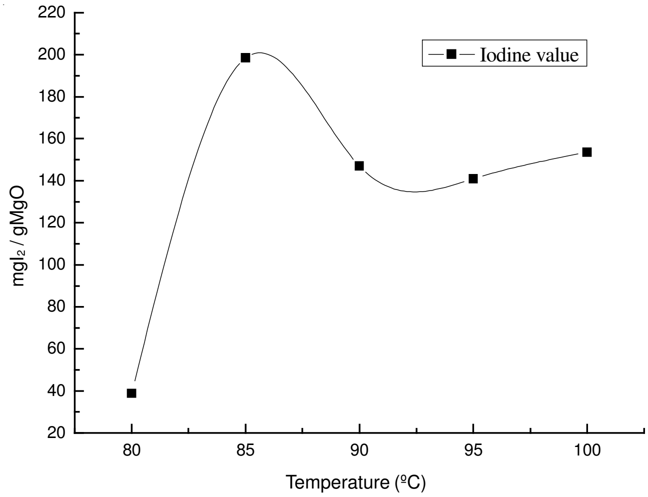


Fig. 5. Effect of reaction temperature on the activity of magnesium oxide

Conclusion

The rod, cotton wool and petal shape of basic magnesium carbonate were easily prepared *via* varying the reaction temperature using industrial magnesium oxide as raw material by precipitation. The morphology of Mg-based precusors can be tailored *via* varying reaction temperature. The rod-like crystal of $MgCO_3 \cdot 3H_2O$ could be achieved at 55-65 ºC, further increasing reaction temperature from 65-100 ºC, cotton stick (below 80 ºC) and petal-like basic magnesium carbonate crystals were obtained respectively. It is worth noting that the numbers of crystal waters presented a single peak form with the increase of reaction temperature and basic magnesium carbonate with eight crystal waters [$4MgCO_3 \cdot Mg(OH)_2 \cdot 8H_2O$] was obtained at 85 ºC. In addition, the high active magnesium oxide was obtained at 85 ºC of reaction temperature, the iodine adsorption value of magnesium oxide was as high as 198 mg/g of MgO.

ACKNOWLEDGEMENTS

This work supported by Liaoning Provincial Natural Science Foundation of China (No. 201202175).

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