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Preparation and Characterization of Nano-polymer Porous MgO

Xue Liang ZHAI*, Ai Dong ZHAO, Mi Ying JIA

Department of Chemistry, Hebei Normal Universty, Shijiazhuang 050016

Absract: Porous carrier MgO which was aggregated by nano-particles has been firstly prepared by using a normal technology route. The MgO was rod-shaped and had large surface area. The factors which affect grain size and microstructure of MgO were explored.

Keywords: Nanometer, porous MgO, bitten, sodium carbonate.

With the development of nano-materials, a new type of high functional fine inorganic material, known as nano-MgO is produced. Porous MgO has spacious structure, which can carry a great deal of effective catalytic compositions to form a carrier compound catalyst. Porous MgO displays high activity and adsorption, which is similar to that of nano-MgO.

Based on the techology for the preparation of nano-MgO, we have developed a new techcology to produce nano-polymer porous MgO under the simple conditions.

Experimental

$MgCl_2$ and Na_2CO_3 were dissolved in water with a final concentration of 0.5 mol/L for both solutions. A small amount of cetyltrimethylammonium bromide was added to the above two solutions. Under different temperatures, the Na_2CO_3 solution was slowly added to the $MgCl_2$ solution with stirring to obtain rob-shaped $MgCO_3{\cdot}3H_2O$. The products were aged for 3 to 96 h, and then translated at the 333 ~ 373 K for a certain time until magnesium carbonate (basic) was formed. After filtration, washing, drying, the products were calcined for 0.5 ~ 4 h at 873 ～ 1273 K to get MgO. Microstructure of MgO was observed by SEM and surface area was measured by BET method.

Results and Discussion

$MgCl_2$ solution reacts with Na_2CO_3 solution to produce $MgCO_3{\cdot}3H_2O$，if the reaction temperature is high, it would favor to form a large number of the cores in short time. It makes the cores do not have enough time to grow up. So $MgCO_3{\cdot}3H_2O$ in larger size could not be obtained, this is unfavorable to the further reaction to get porous MgO.

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* E-mail: xlzhai253@hebtu.edu.cn

The aging time also affects the grain size of $MgCO_3·3H_2O$. The axis and length of $MgCO_3·3H_2O$ were increased as the aging time was prolonged. However the axis increase rate was slightly higher than that of length (**Table 1**). These results showed that at room temperature and aging time for 48 h, acicular-shaped $MgCO_3·3H_2O$ with the axis of 3μm and length of 15μm coule be obtained.

Table 1 The relationship between grain size of $MgCl_2 · 3H_2O$ and reaction temperature（aging for 48 h）

Temperature (K)	283	288	293	298	303	308
Average aperture（μm）	4.0	3.7	3.4	3.1	2.65	2.5
Average length（μm）	12.0	11.1	10.3	9.5	8.4	7.5

To benefit the production of porous MgO after calcining, $4MgCO_3·Mg(OH)_2·4H_2O$ should be kept rob-shaped form. Thus we can use relatively low temperature and longer time for that $MgCO_3·3H_2O$ would transform into a structurally intensed magnesium carbonate (basic). In general, when the temperature is controlled above 333K, we could obtain the ideal intermediate products (**Figure 1a**). In addition, during the transformation stirring should be stopped to avoid the distraction of rob-shaped and shortening the length of the product. Otherwise scattered or flat-shaped polymer products will be produced **(Figure 1b, 1c**).

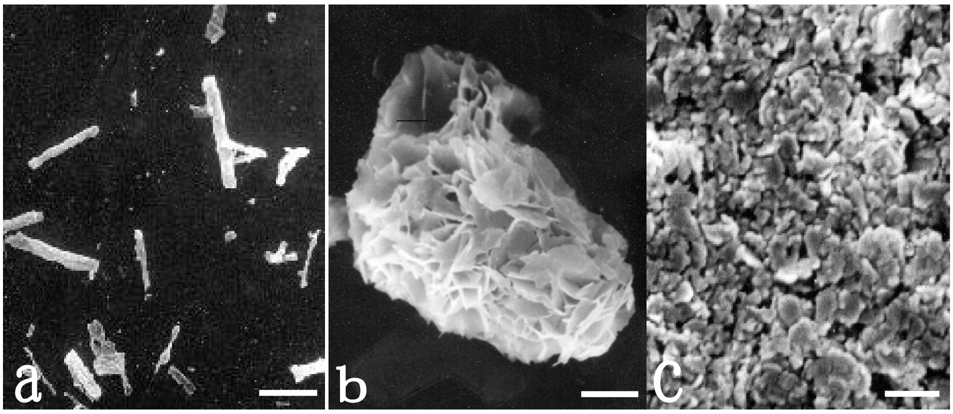
Table 2 The relationship between the surface area of MgO and calcination temperatures

Calnation temperatures（K）	873	923	973	1023	1073	1123	1173	1273
Sureface area（m^2/g）	102	112	121	113	108	97	94	81

The experimental results indicated that after drying and calcining, the rob-shaped magnesia alba was transformed into porous MgO. This type of MgO had special structure, resembling the appearance of $MgCO_3·3H_2O$ (**Figure 2a**) and with relatively larger surface area（**Table 2**). The SEM micrographs of higher multiple showed that this type of MgO was consisted of polymerized nano-MgO closely. Therefore, the new polymer with special appearance possibly would have characteristics of nanometer and porous materials, such as high adsorption, adsorbing selection, chemical activity and so on. The related research is still undergoing.

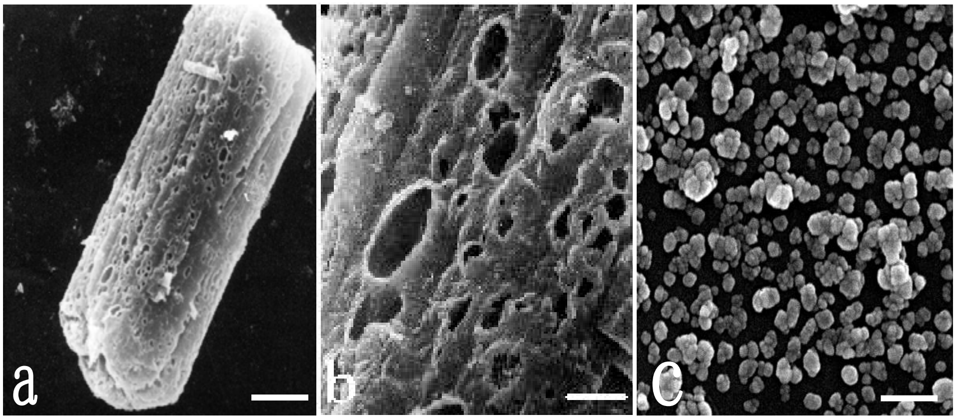
Magnesia alba contained a large amounts of water due to it formed in water. Thus the factors of drying and calcination temperatures can affect the characteristics of the products. If the drying and calcination temperatures were too high or the calcination temperature was increased too quickly, a large amount of water would be evaporated rapidly and then the appearance of the products would be destroyed. As a result, we can only get the nano-MgO, no porous MgO could be obtained (**Figure 2c**). If the calcination temperature was too high, the products would be easily sintered and the surface area and activity of MgO would be declined. Only by controlling the optimal aging time, drying and calcination temperatures, the ideal porous MgO (**Figure 2a**) could be obtained.

Figure 1 SEM micrograhps of magnesia alba



a. Bar = 10μm b. Bar = 5μm c. Bar = 5μm

Figure 2 SEM micrographs of MgO



a: porous MgO(bar = 3 μm) b: porous MgO of high times(bar = 1 μm)
c: scattered nano-MgO(bar =400nm)

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