Hydrothermal Preparation of Nanosized Sm_2O_3 Assisted by Carbon Microspheres as Templates

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Abstract—In this experiment, Sm $(NO_3)_3 \cdot 6H_2O$, hexamethylene tetramine (HMT) and glucose as the main raw material, nanosized Sm_2O_3 was prepared by hydrothermal method. Sm_2O_3 samples were characterized by XRD, SEM, FT-IR, TG-DTA and DRS respectively. It found that plus C elements could increase the crystallinity of the sample by exothermic reaction, but carbon dioxide lead to the destruction of Sm_2O_3 nanospheres. In photocatalytic test, nano Sm_2O_3 was applied in degradation of methyl orange by adjusting catalyst quality and illumination time to optimize parameters. The catalytic time and mass of the catalyst can increase the degradation rate of methyl orange, but excessive catalyst will discourage the degradation of methyl orange.

Keywords- nanosized Sm_2O_3; hydrothermal method; carbon microspheres; photocatalytic; templates

I. INTRODUCTION

Sm_2O_3 is white powder with a little bit of yellowish, have a body- centered cubic or monoclinic [1-3], belong to a rare earth metal oxides. Samarium oxide material can also be used to increase the performance of polypropylene plastics [4].There are researchers who use the samarium oxide material as a function particles, prepared the radiation performance of samarium oxide/epoxy samples [5] is also greatly improved. Due to ceria solid electrolyte doped the samarium oxide and other rare earth metal oxide nitride oxide, which leads to the degree of lattice distortion smaller, powder becomes high degree of crystallization, makes electrical conductivity also increases [6-7].And because samarium oxide itself as a catalyst [8] use small amount, have well in the application prospect, is a new and environmentally friendly catalysts. With the rapid development of nanotechnology and technology, the use of carbon microspheres are more and more, in terms of photonic crystals, chemical sensors, pollution prevention, drug delivery, etc.[9],all use of carbon microspheres can be found. This paper will use glucose as raw material, and carbon microspheres can be prepared by hydrothermal method, as a template for preparing spherical nano Sm_2O_3 and studied its photocatalytic properties of nano Sm_2O_3.

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II. PREPARATION AND CHARACTERIZATION

First, take 10 g of glucose in a beaker, added 50 mL of distilled water to make a glucose solution. A brown solution of carbon microspheres was heated at a temperature of 180 ^{O}C in muffle furnace for 4 hours. Some of absolute ethanol was added in the carbon microspheres obtained in solution, the carbon nanoparticles may be completely dissolved. Then adding 1 : 1 samarium nitrate and hexamethylene tetramine solid and a number of distilled water, and stirring sufficiently such that drug dissolution .Then heating at a temperature of 75 ^{o}C for two hours by hydrothermal method. The obtained sample was dried give a dark brown gum, placed gum in a muffle furnace at a temperature of 900 ^{o}C calcined for two hours to give a white solid .The resulting solid was pulverized into a powder, and characterized by Beijing 's spectral analysis of general Instrument Co., Ltd. XD-60 ray diffraction spectroscopy (XRD),Hitachi SN3400 scanning electron microscope (SEM),Thermo Fisser Scientific Inc UV - visible spectrometer (5A2PO54001) measuring

diffuse reflectance spectroscopy (DRS),Beijing permanent Scientific Instrument Factory differential thermal analyzer (HCT-2) and differential thermal analysis (DTA).Catalytic properties under UV light by measuring the degradation rate of the degradation of methyl orange solution to characterize. In controlled trial, in addition to without add carbon microspheres, the rest are unchanged. These involve chemical reagents were analytical grade.

III. RESULTS AND DISCUSSION

A. *The impact of the sample's crystal form by adding carbon microspheres*

As shown in Fig. 1, samples' X-ray diffraction patterns are Sm_2O_3-I (carbon-free microspheres, at room temperature), Sm_2O_3-II (carbon-free microspheres, at 300 ^oC), Sm_2O_3-III (carbon-free microspheres, at 900 ^oC), Sm_2O_3-IV (plus carbon microspheres, at 900 ^oC), respectively. As can be seen from the figure, Sm_2O_3-I (carbon-free microspheres, room temperature) samples can see Sm_2O_3 characteristic diffraction peaks, but not obvious, indicates a low degree of crystallinity; Sm_2O_3-II (carbon-free microspheres, at 300 ^oC) sample basically do not see Sm_2O_3 characteristic diffraction peaks.Sm_2O_3-III (carbon-free microspheres, at 900 ^oC) and Sm_2O_3-IV (plus carbon microspheres, 900 ^oC) has significantly Sm_2O_3 characteristic diffraction peaks, indicate high crystallinity Sm_2O_3 sample was obtained .Contrasting standard card JCPDS (43-1029) is know, Sm_2O_3 is cubic crystal. Therefore, in present experiment, carbon microspheres as a template hydrothermal method (900 ^oC) to prepare nano-Sm_2O_3 microspheres.

B. *The impact of the sample's morphology by adding carbon microspheres.*

In obtained carbon microspheres solution, after repeated centrifugation obtaining little solid carbon microspheres, causing 80 ^oC ovens dried to give a coffee -colored powder. Fig. 2 is the scanning electron micrographs of carbon microspheres .As shown in Figure, carbon microspheres particles is obviously and diameter about 2 μm, dispersion more uniform .Due to these phenomena of carbon microspheres have a spherical fine, small specific surface area, bulk density, permeability and surface stability advantages, and not prone entangled than carbon nanotubes. The scanning electron micrographs of Sm_2O_3-III sample is shown in Fig. 3. As shown in Figure, particle diameter of the Sm_2O_3-III sample around 100 nm is block. Sample after calcination, because of thermal stress generated dislocations, leading to transformation is from orderly to disorderly and form aggregates.

The scanning electron micrographs of Sm_2O_3-V is shown Fig. 4. The Sm_2O_3-I particles gathered together, not obvious, around 40 nm, become the nanoscale grade. From the figure also can be seen, the nanospheres have been destroyed, showing a flaky crust .This explanation show that the preparation process of experimental techniques need to be improved.

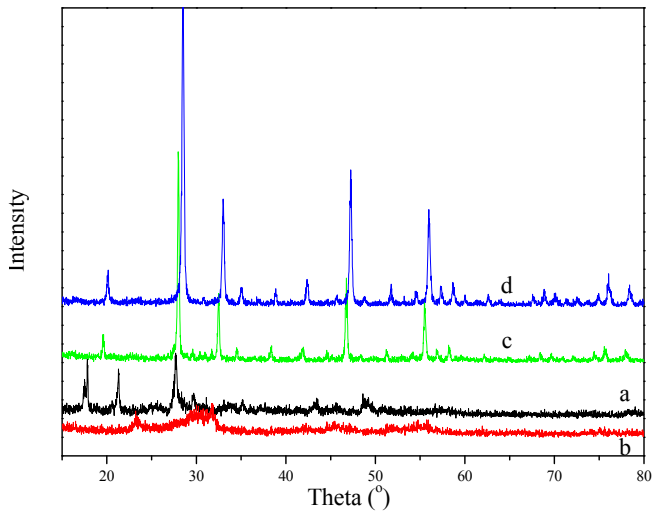

Figure 1. XRD pattern of Sm_2O_3 samples a:Sm_2O_3-I, b: Sm_2O_3-II, c: Sm_2O_3-III, d: Sm_2O_3-IV.

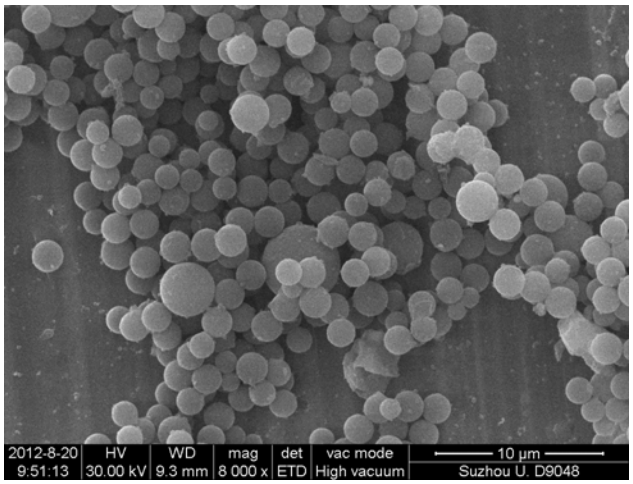

Figure 2. SEM micrographs of carbon microspheres.

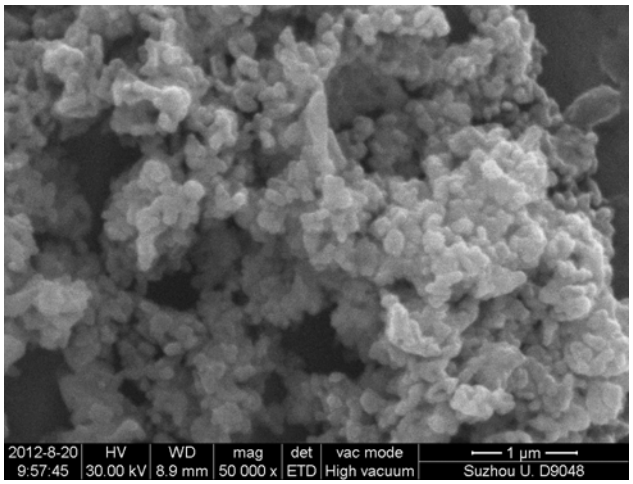

Figure 3. SEM micrographs of Sm_2O_3-III sample.

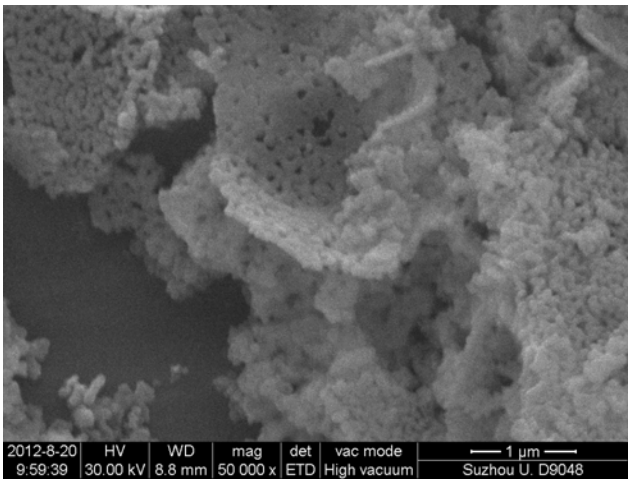

Figure 4. SEM micrographs of Sm_2O_3-V samples.

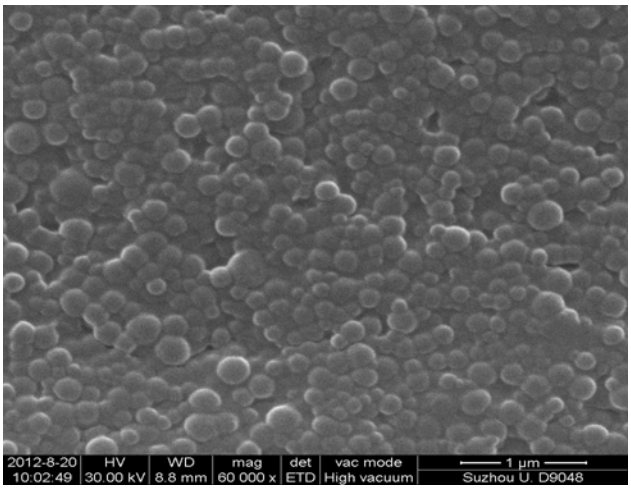

Figure 5. micrographs of Sm_2O_3-IV sample.

The scanning electron micrographs of Sm_2O_3-IV are shown Fig. 5. The sample distribution is very uniform, and particle size can be well-proportioned, basically around 150nm. Sm_2O_3-IV sample particles significantly more perfect than Sm_2O_3-V and Sm_2O_3-III Samarium oxide particles was able to grow on the carbon microspheres, is because it has the electrostatic interaction between the particles. This effect can make carbon microspheres surface of positive and negative charges particles combine together. After calcination, loss of carbon microspheres template have benefit to form a hollow nano-samarium oxide materials.

C. *Diffuse reflectance spectroscopy characterization*

The diffuse reflectance absorption light spectrums of Sm_2O_3-III and Sm_2O_3-IV sample are shown in Fig. 6. From the figure we can know, a good absorbency is exhibited between in 230 nm-260 nm of Sample Sm_2O_3-III, and then it is very slow, almost zero, there is no absorption .After drawing a tangent, can get their absorption edges are λ_1 and λ_2 respectively, λ_1 = 264 nm, λ_2 = 326 nm.Eg_1 = 4.70 eV, Eg_2 = 3.80 eV by using Eg = 1240 /λ (eV) can be calculated .The wavelength of Sm_2O_3-III samples tend to be more long-wave, the

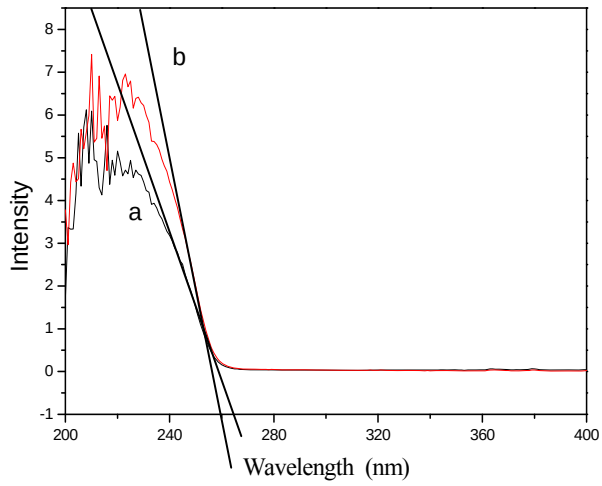

Figure 6. The diffuse reflectance absorption light spectrum of Sm_2O_3-III and Sm_2O_3-IV sample. a: Sm_2O_3-III, b:Sm_2O_3-IV.

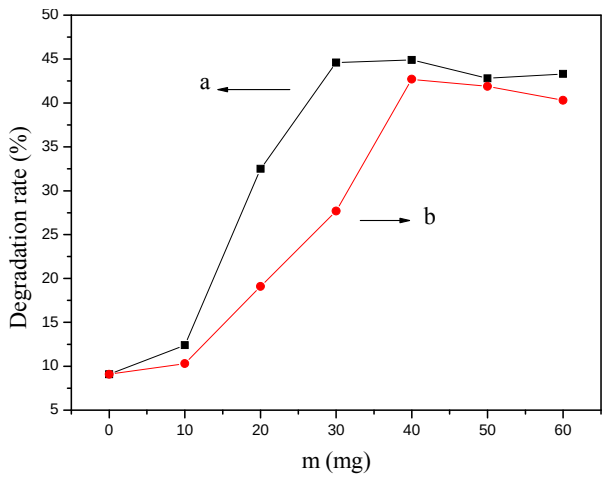

Figure 7. Different quality of Sm_2O_3 to degradation rate of methyl orange solution curve. a: Sm_2O_3-III, b:Sm_2O_3-IV.

excitation energy lower. It can be concluded, comparing Sm_2O_3-III samples to Sm_2O_3-IV samples, Sm_2O_3-III is easier to be excited, so it can be speculated that the photocatalytic efficiency of the former will be better than the latter. Therefore, we will conduct the photocatalytic performance test to verify this.

D. *Photocatalytic properties*

The main is research different catalysts quality effect on the catalytic properties of Sm_2O_3 samples and different illumination time effect on the catalytic properties of the Sm_2O_3 sample in Fig. 7. Taking Sm_2O_3-III and Sm_2O_3-IV sample several number (m = 10 mg, 20 mg, 30 mg, 40 mg, 50 mg, 60 mg), place under the same light for photocatalytic, one hour later removed, after twice centrifugation use 752 UV-Vis spectrophotometer measure the absorbance at 464 nm. According to the absorbance can calculate the degradation rate, and using origin7.0 to draw the degradation rate graphs. From Fig. 8 can be seen, the degradation rate of Sm_2O_3-III sample quality between 10-30 mg increase the fastest, the degradation rate is almost unchanged between 30-60 mg. But the degradation

rate of Sm_2O_3-IV sample quality between 10-30 mg increase the fastest, up to 44.9%. The degradation rate is almost unchanged between 30-60 mg. The photocatalytic effect of Sm_2O_3-III sample is better than the latter samples, results of diffuse reflectance absorption spectrum accord with Fig. 6. Also can be observed, whether Sm_2O_3-III or Sm_2O_3-IV sample, finally catalytic rates are no longer rising, even slightly trend to decline. This may be because of a significant increase the solid powder in solution, resulting in scattering is increased, thus preventing the degradation of methyl orange. The excessive catalyst does not make the catalytic efficiency increase.

From Fig. 8 can be seen ,the degradation rate of Sm_2O_3-III sample between 0.5-2.0 h increase the fastest, up to 73.1 %.But the degradation rate is almost unchanged after 2.0 h. Between 0.5-2.5 h, the degradation rate of Sm_2O_3-IV sample continued to rise, after it degradation rate increase slowly, almost unchanged .The photocatalytic efficiency of Sm_2O_3-IV sample is up to 71.2% .But the photocatalytic of Sm_2O_3-III sample effect slightly better .Consistent with the results obtained by Diffuse reflectance spectroscopy characterization .That Sm_2O_3-III sample easier than Sm_2O_3-IV samples to catalytic degradation of methyl orange. Whether for change Catalyst quality or change the time, the catalytic properties of Sm_2O_3-III is better than Sm_2O_3-IV .The literature shows that most likely due to the presence of carbon element.

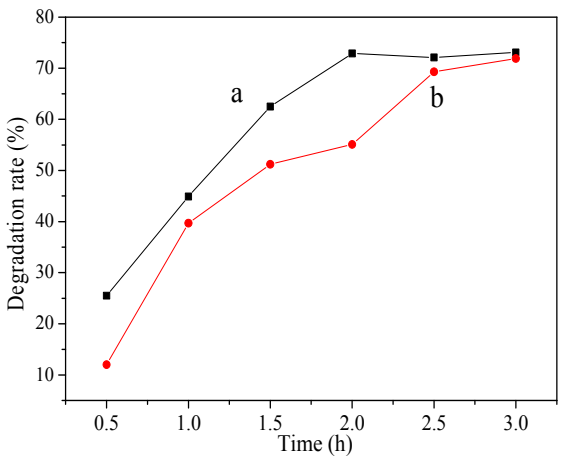

Figure 8. Different illumination time of Sm_2O_3 to degradation rate of methyl orange solution curve. a: Sm_2O_3-III, b:Sm_2O_3-IV.

IV. CONCLUSION

The main conclusions in this paper are: 1) successfully synthesized by hydrothermal method the average of diameter is about 150 nm, cubic crystal of ball nano-Sm_2O_3 sample; and optimum calcination temperature of the sample was 900 ^oC; 2) Due to the impact of carbon, the wavelength of Sm_2O_3-III is slightly longer than Sm_2O_3-IV, led to the photocatalytic properties of the latter is worse than the former.

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