April 2008 Volume 3 Issue 2

Trade Science Inc.

Inorganic CHEMISTRY

An Indian Journal

Full Paper

ICAIJ, 3(2), 2008 [139-141]

Preparation of α-Fe_2O_3 nanoparticles by solid-phase method

Yan Jun DU*, Bao An DU, Lei ZHAO, Jian Tong LV

College of Chemistry and Environmental Science, Hebei University, Baoding, 071002, (P.R.CHINA)

E-mail: duyanjun@mail.hbu.edu.cn.

Received: 24th March, 2008 ; Accepted: 29th March, 2008

ABSTRACT

Nanoparticles of α-Fe_2O_3 were prepared by solid-phase method. The products were characterized by XRD and TEM. The results showed that the products were α-Fe_2O_3. The sizes of the particles were around 50 nm and their shapes were peanut-like.

KEYWORDS

Nanosized α-Fe_2O_3;
Preparation;
Characterization;
Solid-phase method.

INTRODUCTION

Nanoparticles of α-Fe_2O_3 were used as functional materials widely, because of its good physical and chemical stability. It can be used to prepare magnetic materials, gas-sensing materials, catalyst materials and so on[1]. Generally nanoparticles of α-Fe_2O_3 were prepared by sol-gel method[2], microemulsion method[3], hydrothermal method[1], microwave method[4,5], solid-phase method[6], et al. In the present paper, nanoparticles of α-Fe_2O_3 were prepared by using $Fe(NO_3)_3 \cdot 9H_2O$ and NaOH as raw materials, solid-phase method was adopted. Compared with other methods, the technology has advantages of low cost of production, simple process and short period of production. So it was suitable for industrial production.

EXPERIMENTAL

Preparation of nanoparticles of α-Fe_2O_3: 10g $Fe(NO_3)_3 \cdot 9H_2O$ and 3g NaOH were put into agate mortar, mixed thoroughly and grinded. The reagents became glue gradually, then, solidified. The solid reagents were grinded to become powders, washed three times with water and ethanol respectively, decompress filtered, hydrated naturally. The precursor was obtained. Then the precursor was grinded, calcined at different conditions. Finally, the products were obtained. The phase compositions of the products were examined by XRD. The sizes and shapes of the products were testified by TEM.

RESULTS AND DISCUSSION

XRD analysis of the nanoparticles of α-Fe_2O_3

In the experiment, the copper target used was: CuKα, λ_{Cu} = 0.154178 nm. Five samples were testified by XRD. The technologies of calcining samples were showed in TABLE 1. The XRD patterns of samples were showed in figure 1.

According to figure 1, the sample Sa was α-Fe_2O_3 and belongs to hexagonally, the shapes of peaks were consistent with the other references[7,8]. Besides some γ-Fe_2O_3 was included in sample Sa and the 3.66 characteristic peak was absent in the XRD pattern of sample

TABLE 1: Technologies of calcining the samples

Samples	Sa	Sb	Sc	Sd	Se
Temperature and period	673K 2h	773K 2h	873K 2h	673K 2h , 873K 2h	573K 2h , 973K 2h

Full Paper

TABLE 2: Average primary Diameter of samples by calculation according to XRD data

Samples	Sa	Sb	Sc	Sd	Se
D (nm)	20.004	25.786	23.339	26.922	25.110

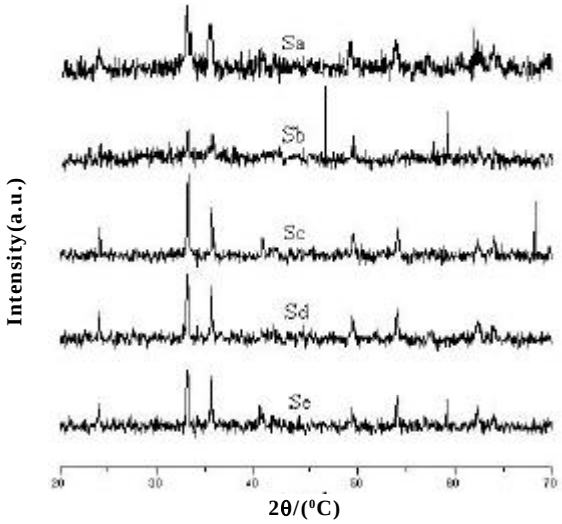

Figure 1 : XRD patterns of samples

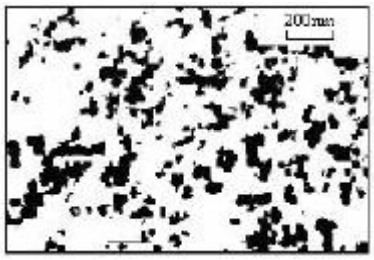

Figure 2 : TEM photograph of Sc

Sa. So we can conclude: the main product of calcining the precursor for 2h at 673K was α-Fe_2O_3; on the other hand, the reagents were not changed into α-Fe_2O_3 completely because of a little lower temperature and shorter period, some impurities and the residual precursor still exist[6]. Whereas the sample Sb was better than the sample Sa, some impurities were still contained in the sample Sb. According to figure 1, the sample Sc, Sd and Se were pure α-Fe_2O_3.

According to figure1, with the calcining temperature ascending and the period extending, the X-ray diffraction peaks become higher and narrower. The average sizes of particles were calculated by the Scherrer formula that was $D = k\cdot\lambda/\beta\cdot COS\theta$(with D: the primary diameter of the product/nm; k: 1.075(spherical crystal); λ_{Cu}: 0.154178/nm; β: integral peak width/arc; θ: angle of diffraction/degree). Many X-ray diffraction peaks of low angular degree(generally $2\theta \leq 50°$) were selected for calculation, and the average value was obtained. The results were showed in TABLE 2. According to TABLE 2, a conclusion can be obtained: the primary diameter of particles was between 20-30 nm.

TEM analysis of nanosized particles of α-Fe_2O_3

Figure 2 was the TEM photograph of sample Sc. According to figure 2 the sizes of the particles were 50 nm and their shapes were peanut-like. The TEM photographs of the other samples were similar to figure 2, so the photographs were omitted. Commonly, the calculated primary diameter based on XRD was less than the primary diameter based on TEM, our result was accordant with this phenomenon.

Mechanism of preparation of nanosized particles by solid-phase method

The mechanism can be divided into the follow two steps:

$$Fe(NO_3)_3\cdot 9H_2O + 3\,NaOH \rightarrow Fe(OH)_3 + 3NaNO_3 + 9H_2O$$

$$2Fe(OH)_3 \rightarrow \alpha\text{-}Fe_2O_3 + 3H_2O$$

In the process of solid-phase reaction, the contact area of reagents becomes larger by grind; meanwhile the temperature of some area ascends, which can initiate reaction of the reagents. Crystal water was included by $Fe(NO_3)_3\cdot 9H_2O$ and the melting point of $Fe(NO_3)_3\cdot 9H_2O$ was low, therefore the trace crystal water can be used as space of accelerating the reaction. The particles collide with each other and nucleuses were formed rapidly, but particles were very difficult to cross every phase, therefore the nucleuses could not grow rapidly. According to the theories of crystal science, when the speed of forming nucleuses far exceeded the speed of growing of nucleuses, small particles can be formed easily. Therefore solid-phase method was a good way for preparation of nanosized particles.

CONCLUSION

The temperature and period of calcining the reagents were important elements of preparation of nanosized α-Fe_2O_3 particles. Pure and uniform $Fe(OH)_3$ began to decompose at 575.1K, α-Fe_2O_3 emerges at 573-673K, with the temperature ascending constantly,

$Fe(OH)_3$ decomposes completely at 873K 2h, thenα-Fe_2O_3 was obtained. With the temperature ascending and the period extending, the sizes of α-Fe_2O_3 particles become larger and larger. The technology of preparation of nanosized α-Fe_2O_3 particles by solid-phase method had advantages of low cost of production, simple process and short period of production. So it was suitable for industrial production.

REFERENCES

[1] D.H.Wu, Z.X.Zhang, L.F.Xu, et al; Chinese Journal of Inorganic Chemistry, **20(1)**, 31-36 **(2004)**.

[2] Y.S.Wang, J.Li; Journal of Inorganic Materials, **13(5)**, 745-750 **(1998)**.

[3] L.W.Chen, L.H.Gan, T.Y.Yue, et al; Acta Physico-Chimica Sinica, **10(8)**, 750-754 **(1994)**.

[4] Z.B.Jia, Y.Wei, H.M.Wang; Journal of Inorganic Materials, **15(5)**, 926-928 **(2000)**.

[5] Z.Fu, X.S.Hao, Z.C.Wu, et al; Chinese Journal of Inorganic Chemistry, **12(2)**, 219-222 **(1999)**.

[6] C.X.Qiu, J.S.Jiang, Z.J.Zhao, et al; Journal of Inorganic Materials, **16(5)**, 957-960 **(2001)**.

[7] D.H.Wu, Z.X.Zhang, L.F.Xu, et al; Chinese Journal of Inorganic Chemistry, **20(1)**, 31-36 **(2004)**.

[8] Y.Wei, X.L.Liu, X.Z.Zheng; Acta Physico-Chimica Sinica, **12(6)**, 551-553 **(1996)**.