The Effect of the Acid Precipitate Conditions on the Size Distribution of Molybdenum Trioxide Particle

Tang Jun-Li

Technical Center of jin dui cheng Molybdenum Mining Corp, Xi'an 710068, China

Abstract

The effect of the preparation factors, such as the feeding mode and rate of raw materials, the reaction temperature and the surfactant on the size distribution of molybdenum trioxide particle were investigated by orthogonal test. The optimum conditions for the preparation of MoO_3 precursors are as following; opposite feeding fast, reaction temperature of 60 ℃ and adding dispersant.

Keywords：molybdenum trioxide, ultrafine powder, orthogonal test, size distribution

1 Introduction

MoO_3 powder has a wide range of applications in the field of electric materials due to its photochromic, electrochoromic properties and inflaming retarding and smoke suppression function. The quality of MoO_3 not only depends on chemical purity, but also depends on the particle size. Processing for the synthesis of MoO_3 often have imporant effenct on the properties of the materials. Ultrafine particles exhibit the materials that differ from those of the bulk solid many important industrial application.

In order to make high-quality and ultrafine powders,various synthesis processing have been used for preparing MoO_3 powders[1]. However, these processes require expensive equipment and high emergy consumption.

In this paper, the precursors of MoO_3 were synthesized by chemical precipitation from the aqueous solution. MoO_3 powder characteristics are strongly influenced by the precursors synthesis condition.[2] The effect of the feeding order and rate of raw materials, the reaction temperature and the surfactant on the size distribution of molybdenum trioxide particle were investigated by orthogonal test.

2 Experimental Procedures

the raw materials ammonia molybdate and HAc used are reagent grade. Powder morphology was observed with SEM, powder particle size was measured with Malvern particle size analyzer. During the preparation, the precursors were firstly obtained, then they were decomposed and separated by heating. As a result, 1μm MoO_3 powders were obtained[2].

The orthogonal design test was L_6 （$3^1\times2^3$） ,the orthogonal standards was given in table 1, the orthogonal design test and MoO_3 powder particle size were given in table 2. the feeding mode normal feeding: dropping 50% HAc into 100 mL ammonia molybdate at the rate of 100 mL/min or 10 mL/min. opposite feeding: dropping 100 mL ammonia molybdate into 50% HAc at the rate of 100 mL/min or 10 mL/min. parallel feeding: dropping HAc and ammonia molybdate at the same time dispersant: adding alcohol

Table 1. the orthogonal standards

factor	feeding mode	temperature/℃	the feeding rate	dispersant
Ⅰ	normal feeding	25	fast	YES
Ⅱ	opposite feeding	60	slow	NO
Ⅲ	parallel feeding			

Table 2. the orthogonal design test and MoO_3 powder particle size

No.	feeding mode	temperature/℃	the feeding rate	Adding dispersant	$D_{0.5}$/ μ m
1	normal	25	slow	NO	2.006
2	normal	60	fast	YES	1.911
3	opposite	25	slow	NO	1.435
4	opposite	60	fast	YES	1.060
5	parallel	25	slow	YES	2.690
6	parallel	60	fast	NO	2.867
Ⅰ	3.917	6.131	5.838	5.661	
Ⅱ	2.495	5.838	6.131	6.308	
Ⅲ	5.557				
R	3.062	0.293	0.293	0.647	

3. Results and Discussion

The test results show that the size of No. 4 is smallest and its size distribution (fig 1 and fig 2) is narrow. So MoO_3 powder characteristics are strongly influenced by the feeding mode and dispersant. The optimum conditions for the preparation of the functional material from molybdenum trioxide are as following; opposite feeding fast, reaction temperature of 60 ℃ and adding dispersant.

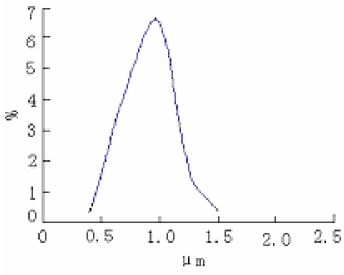

Fig. 1. the size distribution of No. 4.

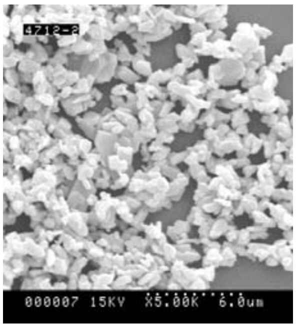

Fig. 2. SEM photograph of No. 4.

the feeding mode

According to Zjemg Ruli,.Wang Enbo[3], main species of the acidification of MoO_4^{2-} was given in table 3. It shows that pH 3~4 is suitable for the preparation of ultrafine powders. Molybdate is an amphiprotic compound.The precipitate were dissolved if the pH ≤2. So opposite feeding can create directly acid condition good for precipitate Molybdate.

Table 3. the acidification of MoO_4^{2-}

[Mo(VI)] mol /L	pH	Main species
All	> 6	$[MoO_4]^{2-}$
10-8	> 5	$[MoO_4]^{2-}$(ca 100%)
10-8	4	$[MoO_4]^{2-}$ (30%), $[HMoO_4]^-$ $[MoO(OH)_5]^-$ (10%) H_2MoO_4 $Mo(OH)_6$ (60%)
10-8	2 - 3	$H_2MoO_4Mo(OH)_6$ (ca 100%)
10-8	1	H_2MoO_4 $Mo(OH)_6$ (80%) $[H_3MoO_4]$+ $[Mo(OH)_5(H_2O)]^+$ (20%)
< 10-3	> 1	Monomeric species only
> 10-3	5-6	$[Mo_7O_{24}]^{6-}$, $[HMo_7O_{24}]^{5-}$, $[H_2Mo_7O_{24}]^{4-}$
> 10-3	4 – 5	$[Mo_8O_{26}]^{4-}$

Dispersant

The formation of precursors particles comes from aggregation of MoO_4^{2-} in aqueous. In this process, the surfactant molecules coat the precursors particles to prevent growth of the single particles and adhesion of particles. In addition, ultrafine particles are metastable owing to their large surface area. And dispersant can reduce the surface tension and prevent agglomeration.

Temperature and feeding rate

At the beginning of the reaction, only a few crystal can be produced in solution. And then slowly grow into large particles. But when temperature is high and feeding rate is rapid, a large number of nuclei were produced in solution. According to the Lamer Chart of formation of ultrafine particle[4], and the growth theory of crystal[5], competition between the nucleation and growth of crystal exists in the solution. The more rapid the nucleating process is, the more the number of nuclei before release of supersaturation is. As a result, the average precursors particle size decreases when temperature is high and feeding rate is rapid, the increase in nucleation rate result in small particle size.

3. Conclusions

The effect of the preparation factors, such as the feeding order and rate of raw materials, the reaction temperature and the surfactant on the size distribution of molybdenum trioxide particle is investigated by orthogonal test. The optimum conditions for the preparation of the functional material from molybdenum trioxide are as following ; opposite feeding fast, reaction temperature of 60 ℃ and adding the dispersant.

4. References

1 Tang Junli, Preparation and Research of Uitrafine Molybdenum Trioxide[J]. China Molybdenum Industry. 2004.28（4）: 32.
2 Tang Junli, Wang Xianqin, Zhao Baohua, Exeprimental Investigation on Preparation of Smoke Suppression Filling/Flame Retardant- α - MoO_3 China Molybdenum Industry. 2004.28（1）: 36-38
3 Zjemg Ruli,.Wang Enbo, Polyacids. Chemstry of Molybdenum and Tungsten[J]. Chemstry . 1984（9）12-18.
4 V.K. Lamer, R. H. Dinegar, J. Am. Chem. Soc., 1950, 72, 4847.
5 A. W. Adamson, Physical Chemistry of Surfaces, 3rd ed., Science Press, Beijing, 1985, 394.