Co-precipitation preparation and burning performance test of delay composition containing barium chromate

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Abstract: In order to optimize the performance of delay composition containing barium chromate, the preparation conditions of barium chromate were optimized, and S/$BaCrO_4$/$KClO_4$ delay composition was prepared by co-precipitation using barium chromate as precipitant. Then, the ignition temperature, delay time and other burning performance were tested. The results show that the ignition temperature of S/$BaCrO_4$/$KClO_4$ delay composition prepared by co-precipitation method is higher than that by traditional mechanical mixing method; the burning rate is faster and the burning time precision is higher because co-precipitation method can make the components mix more evenly. This co-precipitation method with barium chromate can be extended to the preparation of other mixed explosive agents containing barium chromate.

Key words: barium chromate; co-precipitation; delay composition; delay time

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0 Introduction

Barium chromate not only can be used as fuse and pyrotechnic agent, but also can be used to make pigments, ceramics, etc, especially in making the mixed delay composition. Barium chromate has a high decomposition temperature and can absorb heat when it is decomposed, which makes barium chromate multifunctional when used as a delay composition in combination with different combustible agents and acted as an oxidizer or a flame retardant[1]. Therefore, it has a great influence on the burning performance of delay composition. Many investigators have studied the formula, burning performance, and the factors that affect the delay precision[2] of boron[3-6], tungsten[7-8] and silicon type[9] delay composition. Co-precipitation method has greater advantages compared with the traditional mechanical mixing method: First, the co-precipitation method can make the ingredients mix more evenly; Besides, using the traditional mechanical mixing method appears pharmaceutical powder. It is harmful to work in the environment chronically with the great toxicity of barium chromate and other agents. The preparation process of co-precipitation method is basically no dust, which is conductive to industrial hygiene[3].

In this paper, the preparation conditions of barium chromate are optimized firstly. On the basis of this, the S/$BaCrO_4$/$KClO_4$ delay composition is prepared by co-precipitation method and the burning performance is tested.

1 Experiment

1.1 Experimental materials and equipments

Barium chloride, analytical grade, Tianjin Tianli Chemical Reagent Co., Ltd; Sodium dichromate, analytical grade, Tianjin Kaitong Chemical Reagent Co., Ltd; Sodium hydroxide, analytical grade, Tianjin Dalu Chemical Reagent Factory; Sulfur powder, analytical grade, Wenzhou, Zhejiang Dongsheng Chemical Reagent Factory; Potassium perchlorate, analytical grade, Tianjin Sailboat Chemical Reagent Technology Co., Ltd; Methanol, analytical grade, Tianjin Kaitong Chemical Reagent Co., Ltd.

SPY glass thermostatic water bath, Gongyi Yuhua Instrument Co., Ltd; DW-3 digital electric mixer, Gongyi Yuhua Instrument Co., Ltd; HL-2S constant current pump, Shanghai Yue Ming Scientific

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Instrument Co., Ltd; SHZ-D(Ⅲ) Circulating water pump, Hangzhou Ruijia Precision Scientific Instrument Co., Ltd; 1700 Fourier transform infrared spectrometer, the United States Perkin-Elmer company; JSM-6700F scanning electron microscope, Japan Electronics Co., Ltd; FCY-1A Fire point tester, Hebei Xintai Hi-Tech Instruments Manufacturing Co., Ltd.

1.2 Preparation of barium chromate

The reaction formula for the preparation of barium chromate is $2BaCl_2 + Na_2Cr_2O_7 + H_2O \rightarrow 2BaCrO_4\downarrow + 2NaCl + 2HCl$. In the experiment, the flask containing barium chloride solution was placed in a thermostatic water bath, and sodium dichromate solution was added dropwise to the barium chloride solution by a constant current pump. Stired the solution and kept warm for 10 min after adding dropwise. Then removed the flask to be cooled to room temperature and remain for 30 min so that barium chromate can precipitate completely. In order to investigate the effect of different conditions on the yield and morphology of barium chromate, an orthogonal experiment $L_9(4^3)$ was designed.

1.3 Coprecipitation preparation of S/$BaCrO_4$/$KClO_4$ delay composition

The ratio of S, $BaCrO_4$ and $KClO_4$ was 7.5∶83∶8. The weighed sulfur powder and potassium perchlorate together with the $BaCl_2$ solution were placed in a three-necked flask, and then added an appropriate amount of methanol to make the sulfur sink to the bottom in the flask. The three-necked flask was fixed in a thermostatic water bath at 45 ℃ and stirred for 20 min to thoroughly mix the contents in the flask. Subsequently, the $Na_2Cr_2O_7$ solution was dropped into a three-necked flask under the action of a constant current pump, and the mixed solution was naturally cooled to room temperature while dripping, and stir was continued for 10 min after the dropwise addition. Finally, the three-necked flask was removed and kept for 20 min. Suction filtered and washed repeatedly with distilled water. The product was put in an oven and dried.

1.4 Burning performance test of delay composition

According to GJB772A-97 method 606.1, the ignition temperature of 5 s delay time was tested. The burning time of delay composition was tested by photoelectric method[10].

2 Results and discussion

2.1 Optimization of preparation of barium chromate

The $L_9(4^3)$ orthogonal experiment for preparing barium chromate is shown in Table 1.

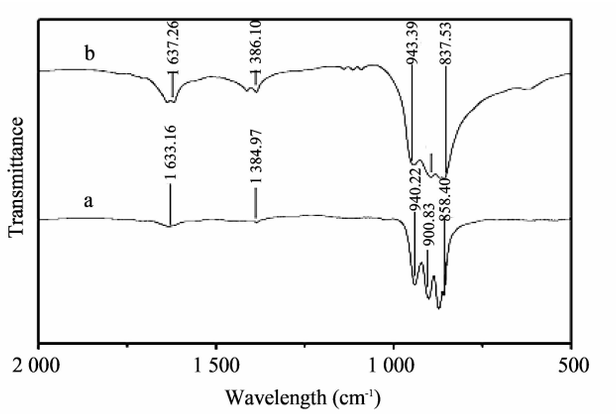
Table 1 Orthogonal test of preparation of barium chromate

Test number	Combination	A Dripping speed v (ml/min)	B Solution concentration c (mol/L)	C Temperature T (℃)	D pH value	Yield (%)
1	$A_1B_1C_1D_1$	0.71	0.6	25	3	81.7
2	$A_1B_2C_2D_2$	0.71	0.8	45	5	87.6
3	$A_1B_3C_3D_3$	0.71	1.0	65	7	84.9
4	$A_2B_1C_2D_3$	1.17	0.6	45	7	88.6
5	$A_2B_2C_3D_1$	1.17	0.8	65	3	82.9
6	$A_2B_3C_1D_2$	1.17	1.0	25	5	86.9
7	$A_3B_1C_3D_2$	2.17	0.6	65	5	86.4
8	$A_3B_2C_1D_3$	2.17	0.8	25	7	88.1
9	$A_3B_3C_2D_1$	2.17	1.0	45	3	81.4

According to the orthogonal table, set yield rate as an indicator of range analysis, the order of the four factors is D>B>A>C, and the optimal combination is $A_2B_2C_2D_3$. Namely, the conditions for obtaining the highest yield of barium chromate are as follows: the dripping speed is 1.17 ml/min, the concentration is 0.8 mol/L, the temperature is 45 ℃ and pH is 7.

The 1 700 Fourier transform infrared spectrometer is used to analyze the infrared spectra of barium chromate prepared by the optimal conditions. The FT-IR of the barium chromate is shown in Fig. 1.

The infrared absorption peak at 940.22, 900.83 and 858.40 cm^{-1} of the standard spectrum in Fig. 1 can be attributed to the characteristic absorption peak of CrO_4^{2-}. The infrared spectrum of prepared barium chromate is basically the same as that of the standard spectrum, and no other absorption peak of the prepared barium chromate is observed, indicating that the purity of the prepared barium chromate is high.

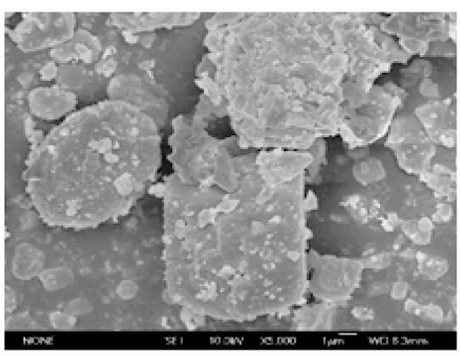


a: Standard spectrum of barium chromate; b: Spectrum of prepared barium chromate

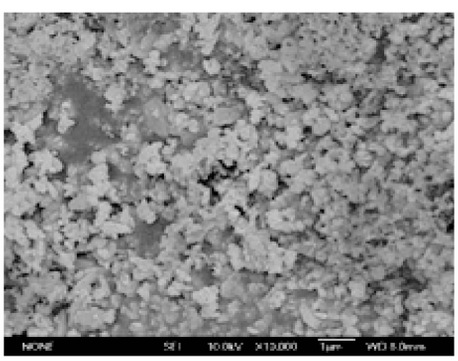
Fig. 1 Infrared spectrum of purity analysis of barium chromate

2.2 SEM analysis of $S/BaCrO_4/KClO_4$ co-precipitation delay composition

The morphologie of $S/BaCrO_4/KClO_4$ delay composition prepared by co-precipitation method is analyzed by JSM-6700F scanning electron microscope. The SEM photographs of $S/BaCrO_4/KClO_4$ delay composition and the prepared barium chromate are shown in Fig. 2.



(a) $S/BaCrO_4/KClO_4$ delay composition



(b) Barium chromate

Fig. 2 SEM photographs of $S/BaCrO_4/KClO_4$ delay composition and barium chromate

From Fig. 2(a), it is observed that there are some spherical particles on the surface of the irregular cakes. The size of the particles is under 1 μm, which is consistent with that of barium chromate in Fig. 2(b). The sulfur powder used in the experiment is an irregular shape with a particle size of about 35 μm, and the potassium perchlorate is square with a particle size of about 15 μm. It is speculated that the larger irregularities in Fig. 2(a) are that sulfur and the potassium perchlorate adheres to the outside of spherical particles. It shows that the innermost layer of the delay composition prepared by the co-precipitation method is sulfur powder, barium chromate absorbs on the surface of sulfur and potassium perchlorate adheres to barium chromate.

2.3 Results of burning performance of delay composition

The ignition temperature of 5 s delay time of $S/BaCrO_4/KClO_4$ delay composition prepared by co-precipitation method and traditional mechanical mixing method are 392.6 ℃ and 347.6 ℃ respectively. The igniting temperature mainly depends on the ignition point of the combustible agent in the explosive agent. The ignition point of the sulfur powder in the delay composition is 230 ℃, while the barium chromate has a higher ignition point. It is speculated that the delay composition prepared by co-precipitation has a higher ignition point, which is due to barium chromate is wrapped on the surface of sulfur powder.

Press the delay composition into a delay tube with inner diameter of 6.9 mm and wall thickness of 0.8 mm in three times. Ignite the delay composition with 5 W laser light. The results of burning time and burning rate of $S/BaCrO_4/KClO_4$ delay composition prepared by co-precipitation method and traditional mechanical mixing method are shown in Table 2.

As seen from Table 2, the standard deviation of burning time prepared by the co-precipitation method is 0.75, the average burning rate is 2.22 mm/s; while the standard deviation of the burning time mixed by the traditional mechanical mixing method is 1.96, the average burning rate is 2.04 mm/s. The delay time of the former is more uniform and the burning speed is faster, indicating that the three components in the delay composition prepared by co-precipitation method are mixed more uniform and the burning time is more accurate.

Table 2 Burning rate of S/$BaCrO_4$/$KClO_4$ delay composion

Preparation method	Burning time t (s)	Average burning time t (s)	Standard deviation of burning time	Burning rate (mm/s)	Average burning rate (mm/s)	Standard deviation of burning rate
Co-precipitation	13.1	12.86	0.75	2.14	2.22	0.13
	13.1			2.18		
	11.8			2.41		
	13.8			2.09		
	12.5			2.28		
Hand-mixed	15.6	14.3	1.96	1.85	2.04	0.26
	12.4			2.33		
	16.9			1.70		
	14.1			2.04		
	12.5			2.30		

3 Conclusion

1) In this paper, the preparation process of barium chromate is optimized by $L_9(4^3)$. The optimized preparation conditions are as follows: the drpping speed is 1.17 mL/min, the concentration is 0.8 mol/L, the temperature is 45 ℃ and the pH is 7.

2) Based on the optimized conditions of barium chromate, S/$BaCrO_4$/$KClO_4$ delay composition is prepared with barium chromate as the precipitator. The SEM photographs show that barium chromate is on the surface of sulfur powder and potassium perchlorate is adhered on barium chromate.

3) Comparing the burning properties of delay composition prepared by coprecipitation method with traditional mechanical mixing method, it is concluded that the S/$BaCrO_4$/$KClO_4$ delay composition prepared by co-precipitation method has higher ignition temperature, faster burning rate and higher burning time precision.

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含铬酸钡的共沉淀延期药制备及燃烧性能测试

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摘　要： 为了优化含铬酸钡延期药的性能，对铬酸钡的制备工艺条件进行了优化，并以铬酸钡为沉淀剂用共沉淀法制备出了 S/$BaCrO_4$/$KClO_4$ 延期药，对其发火点温度、延期时间等燃烧性能进行了测试。结果表明：与传统机械混合法相比，共沉淀法制备的 S/$BaCrO_4$/$KClO_4$ 延期药的发火点温度更高；燃速更快，燃烧时间精度更高，是因为共沉淀法可以使延期药中的各组分混合的更加均匀。这种含铬酸钡的共沉淀制备方法可推广到其它含铬酸钡的混合火工药剂的制备。

关键词： 铬酸钡；共沉淀法；延期药；延期时间

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