Synthesis and Characterization of Ultrafine Aluminum Phosphate Powder in CTAB System

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Abstract. The phosphoric acid and aluminum hydroxide and CTAB reagents are used as raw materials. The ultrafine aluminum phosphate is prepared by liquid phase method. The sample composition and scanning electron microscope are used to observe the sample. Morphological characteristics were used to analyze the effects of CTAB and temperature of calcination on the morphology of aluminum phosphate powder.

1. Introduction

Aluminum phosphate has the characteristics of high melting point, good fire resistance, good wear resistance and high strength. Aluminum phosphate belongs to polar substance, which shows hydrophilicity, hydrophobicity and poor compatibility. Interfacial separation can be observed in the polymerization reaction with organic polymers [1]. Widely used in fireproof coating, paper industry, textile industry, antifouling agent, flux and other aspects [2]. In the past, there are many methods to prepare aluminum phosphate [3-5], but it is difficult to prepare pure aluminum phosphate, because there are many by-products of direct reaction between aluminum hydroxide and phosphoric acid, a small amount of phosphoric acid reacts with aluminum hydroxide: H₃PO₄ + Al(OH)₃ = AlPO₄ + 3H₂O; a proper amount of phosphoric acid reacts with aluminum hydroxide: 3H₃PO₄ + 2Al(OH)₃ = Al₂(HPO₄)₃ + 6H₂O.

Excessive phosphoric acid reacts with aluminum hydroxide: 3H₃PO₄ + Al(OH)₃ = Al(H₂PO₄)₃ + 3H₂O; therefore, it is very important to choose the preparation method of partial transportation. Sixteen alkyl three methyl ammonium bromide (CTAB) is a quaternary ammonium cationic surfactant. It can be dissolved in water and produce large amount of foam when it oscillate. It has good compatibility with anionic, nonionic and amphoteric surfactants [6]. It has excellent permeability, flexibility, emulsification, antistatic, biodegradability and bactericidal properties [7]. It is often used in the preparation of nano materials to control the size and morphology of nano particles. CTAB system is also an excellent detergent. In this experiment, CTAB system is used to prepare aluminum phosphate ultrafine powder, which can not only adjust particle size and morphology [8-10], but also increase the anti fouling ability of aluminum phosphate. At the same time, the synthetic route has the advantages of small pollution, high efficiency and low cost, which is a good method worth popularizing.

2. Experimental Part

2.1. Reagents and Instruments
Phosphoric acid (85%), Al(OH)₃, and NH₃•H₂O were purchased from Tianjin comio Chemical Reagent Co., Ltd. Cetyltrimethylammonium bromide (CTAB) was purchased from Beijing Yili fine...
chemical Limited company. They were all analytical reagents. SU-5000 Field emission scanning electron microscope (Hitachi, Japan), Panako multifunctional powder X-ray diffractometer (test voltage of 40 kV, current of 20 mA, scanning speed of 0.03°/s, continuous scan, 10° to 80°, Cu Ka (λ = 0.15418 nm)), Instruments include electronic balance (JA5003N, Shanghai Precision Scientific Instrument Co., Ltd.), high-speed centrifuge (TG16K-II, Dongwang Instrument Co., Ltd.), electric heating blast drying oven (HG101-2A, Nanjing Yingxin Experimental Instrument Co., Ltd.).

2.2. Synthesis of Products
After a series of optimization experiments, the concentration and calcination time of CTAB were changed, and the optimized process conditions were obtained. The typical experimental steps are as follows: prepare 40ml of 60% phosphoric acid, transfer it into two 500ml beakers respectively, label a and B, and add 5ml of CTAB with concentration of 0.001mol/l to the solution of beaker B. Weigh two 3.9G aluminum hydroxide, stir it continuously, add A and B beaker respectively, get thick gel, then add 200ml deionized water, add ammonia water to adjust pH value to 5, then put two beaker in 75 degree water bath to keep 2H. After the heat preservation, it can be seen that there are obvious layers in both groups A and B beakers, the upper layer is clear and transparent, and the lower layer is white precipitate. Filter separately and collect white precipitate. The precipitate was placed in a vacuum drying oven and dried at 100 °C for 12h. After drying, the white massive solid ball is grind into powder shape, and then A and B samples are divided into four parts: A1, A2, B1 and B2, and A1 and B1 are tested. A2 and B2 were put into a box resistance furnace and calcined at 800 °C for 3h to obtain the product.

3. Results and Discussion

3.1. XRD Analysis

![XRD Analysis](image)

Figure 1. The XRD pattern of the products obtained from the reaction of phosphoric acid and aluminum hydroxide without CTAB (a: aluminum phosphate of PDF standard 50-0303; b: diagram of sample A1; c: diagram of sample A2)

Fig. 1 is the XRD pattern of the product obtained from the reaction of phosphoric acid and aluminum hydroxide, FIG. 1a is the diffraction peak diagram of aluminum phosphate PDF standard card 50-0303, FIG. 1b is the XRD diagram of aluminum phosphate produced by the reaction of 60% phosphoric acid and aluminum hydroxide, FIG. 1c is 60% phosphoric acid and aluminum hydroxide reacted at 800 °C and baked at high temperature. Compared with FIG. 1a, the diffracted peaks of Fig. 1b and Fig. 1C are close to the diffracted peaks of aluminum phosphate PDF standard card 50-0303, which shows that the main components of sample A2 and A1 are aluminum phosphate, but they all have hetero peaks. Compared with FIG. 1b and Fig. 1C, the hetero peaks are reduced after 800 °C high temperature
roasting, which proves that high-temperature roasting can remove some impurities.

![Figure 2. XRD pattern of the products obtained from the reaction of phosphoric acid and aluminum hydroxide after adding CTAB (a: aluminum phosphate of PDF standard 76-0234; b: diagram of sample B1; c: diagram of sample B2)](image)

Fig. 2 is the XRD diagram of the product obtained from the reaction of phosphoric acid and aluminum hydroxide after adding CTAB, FIG. 2a is the diffraction peak diagram of aluminum phosphate PDF standard card 76-0234, FIG. 2b is the XRD diagram of aluminum phosphate produced by adding CTAB in reaction of 60% concentration phosphoric acid and aluminum hydroxide, FIG. 2C is the XRD diagram of aluminum phosphate produced by adding CTAB in reaction of 60% concentration phosphoric acid and aluminum hydroxide and calcining at 800 °C. The diffraction peaks of Fig. 2B and C correspond to the diffraction peaks of No. 76-0234 of aluminum phosphate PDF standard card, but there are miscellaneous peaks, indicating that the main products of sample B2 and B1 are aluminum phosphate, containing impurities. The comparative analysis of Fig. 2B and Fig. 2C shows that the corresponding miscellaneous peaks decrease after high temperature roasting at 800 °C, which is basically the same as that of No. 76-0234 of aluminum phosphate PDF standard card, further proving that high temperature roasting can remove impurities.

3.2. SEM Analysis

Fig. 3 is the SEM image of the product from the reaction of 60% phosphoric acid with aluminum hydroxide. It can be observed that the size of aluminum phosphate particles in the sample image is large, the average size is about 40 microns, most of them are irregular bodies, and the distribution is relatively disordered.

![Figure 3. SEM image of the product from the reaction of 60% phosphoric acid with aluminum hydroxide](image)
Fig. 4 is the SEM image of the product obtained after the reaction of 60% phosphoric acid with aluminum hydroxide and calcined at 800 °C. Compared with Fig. 3, it can be seen that the particle size of the product after calcined at 800 °C decreases, the average size is about 25 μm, and there is a trend of forming regular shape particles. The results show that calcination at high temperature will make aluminum phosphate particles smaller and obtain regular morphology.

Fig. 5 is the SEM image of the product obtained by adding CTAB in the reaction of 60% phosphoric acid and aluminum hydroxide. Compared with Fig. 3, it can be seen that after adding CTAB, the aluminum phosphate particles become thinner, the average particle size is only about 15 μm, and the morphology also changes, forming a few regular ellipsoid or square block crystals, which is related to CTAB’s ability to adjust the surface morphology of particles.
Fig. 6 is the SEM image of the product obtained from the reaction of 60% phosphoric acid and aluminum hydroxide, adding CTAB, and calcining at 800 °C, compared with Fig. 3, aluminum phosphate particles become smaller, more uniform in size, better distribution, and more regular shape particles. Compared with FIG. 5, the particle size is also smaller, with some regular shapes. However, from Fig. 6, it can be seen that the average particle size after calcination at 800 °C is only 1-3 μm, and some aluminum phosphate particles reach nanometer size.

4. Conclusion
The results show that the aluminum phosphate powder prepared by the reaction of 60% phosphoric acid and aluminum hydroxide in 0.001 CTAB system has the smallest particle size; the high temperature calcination at 800 °C can make the aluminum phosphate particles smaller and promote the formation of regular morphology, and the high temperature calcination can effectively remove impurities.

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