Effect of Different Variables on the Size Distribution of Barium Chromate Nanoparticles

Mehdi Simiari^{1,2}, Mehrdad Manteghian¹ & Malihe Ghashamshmi-Iraj.³

¹ Materials Engineering Department, Faculty of Engineering, Tarbiat Modares University, Tehran, Iran

² Department of Physics and Chemistry, Faculty of Science, Emam Ali University, Tehran, Iran

³ Department of Manufacturing Technologies, Malek-Ashtar University of Technology, ShahinShahr, Iran

Correspondence: Mehrdad Manteghian, Materials Engineering Department, Faculty of Engineering, Tarbiat Modares University, Tehran, Iran. E-mail: manteghi@modares.ac.ir

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Abstract

In this study, the synthesis of $BaCrO_4$ nanoparticles is performed by the reaction between $Ba(NO_3)_2$ and $(NH_4)_2CrO_4$ in the presence of surfactant. Different analyses such as X-ray diffraction (XRD), Transmission electron microscopy (TEM), Dynamic light scattering (DLS), and Energy Dispersive X-ray analysis (EDAX) are carried out to identify the features of nanoparticles. The tests are conducted in different concentrations of reactants and surfactants, and at the end, the results are compared. Optimum concentration is also measured and reported to achieve smaller nanoparticles. The results indicate that, in the synthesis of $BaCrO_4$ nanoparticles, the best performance belongs to Sodium Citrate.

Keywords: barium chromate, nanoparticles, surfactant, sodium citrate, size distribution

1. Introduction

Precipitation from a solution is one of the methods of nanoparticle synthesis. In this process, a supersaturated solution is made followed by nucleation and nanoparticle formation ¹⁻³. The shape and size of nanoparticles depend on the conditions pertained during the synthesis, including supersaturation level, temperature, pH and nature of the solvent ^{4, 5}. Stabilizers have been used to prevent the agglomeration of nanoparticles. The nature and concentration of stabilizers are varied to control the size and morphology of nanoparticles ³⁻⁸.

Crystalline barium chromate (BaCrO₄) is known as an oxidant ⁹. Considering the increase of the surface area of nano-sized materials, it is expected that nano-sized materials could increase the oxidation rate. BaCrO₄ nanoparticles are synthesized by two methods: eggshell membrane or precipitation ^{10–12}. In the first method, the outer shell of an eggshell is used to filter and separate the nanoparticles from larger particles. In precipitation method, stabilizers prevent the agglomeration of nanoparticles. Guangjun Zhou et al. synthesized 4-6 nm BaCrO₄ nanoparticles in the presence of an anionic surfactant by reaction between the aqueous solutions of Ba(NO₃)₂ and sodium chromate (Na₂CrO₄)¹².

In this research dodecylbenzene sulfonic acid sodium salt (DBSS), sodium citrate dihydrate (Na₃C₆H₅O₇·2H₂O), and polyvinylpyrolidone (PVP) to choose the best stabilizer were used in synthesizing BaCrO₄ nanoparticles. In order to optimize the parameters of synthesis of nanoparticles including the concentration of (Ba(NO₃)₂ and (NH₄)₂CrO₄, and the type and concentration of stabilizer, the formed nanoparticles' features were identified and analyzed. Finally, the best parameters with the smallest size were reported.

2. Method

Different analyses such as X-Ray Diffraction (XRD), Transmission Electron Microscopy (TEM), Dynamic Light Scattering (DLS), and Energy Dispersive X-Ray (EDX) were carried out to characterize the nanoparticles. BaCrO₄ nanoparticles were synthesized by the reaction between $Ba(NO_3)_2$ and $(NH_4)_2CrO_4$ in the presence of surfactant.

2.1 Experimental

In a typical experiment, 0.9125 g $(NH_4)_2$ CrO₄ and 4.4115 g $Na_3C_6H_5O_7$ ·2H₂O were dissolved in 100 ml distilled water in the main container. The solution was stirred for 2 hr at 500 rpm at room temperature. Then 0.4900 g

 $Ba(NO_3)_2$ was dissolved in 25 ml distilled water, and stirred for 30 min. 20 ml of $Ba(NO_3)_2$ solution was added dropwise to the main container for 20 min under continuous stirring. Then, in order to form $BaCrO_4$ nanoparticles, the solution was stirred for 36 hr. Some yellow precipitates were produced, and the solution was centrifuged for 45 min (at 3 steps, each 15 min) at 10000 rpm. Product separated by centrifugation and then all precipitates along with ethanol and distilled water were washed to remove the impurities, ammonium nitrate and residual surfactants. The washed precipitates were dried in an oven at 150°C for 2 hr. The experiments were conducted at different concentrations of reactants and stabilizers are as shown in Table 1.

Dun	oncentration of Ba(NO ₃) ₂ Concentration of (NH ₄) ₂ CrO ₄ _{True of surfactor}			ncentration of surfactant
Kun	(M)	(M)	Type of surfactant	(M)
1	0.075	0.12	Na ₃ C ₆ H ₅ O ₇ ·2H ₂ O	0.3
2	0.3	0.1	Na ₃ C ₆ H ₅ O ₇ ·2H ₂ O	0.01
3	0.1875	0.03	Na ₃ C ₆ H ₅ O ₇ ·2H ₂ O	0.1
4	0.1875	0.12	PVP	0.01
5	0.075	0.1	PVP	0.1
6	0.3	0.03	PVP	0.3
7	0.1875	0.1	DDBS	0.3
8	0.075	0.03	DDBS	0.01
9	0.3	0.12	DDBS	0.1

Table 1. Conditions of experiments

Different experiments were conducted to identify and analyze the features of the formed nanoparticles: X-Ray Diffraction (XRD) was conducted to perform phase analysis. Transmission Electron Microscopy (TEM) was used to study the shape and size of the formed particles. Particle size distribution was studied by Dynamic Light Scattering (DLS), spectrophotometry and Energy Dispersive X-Ray (EDX) analysis were performed.

3. Results and Discussion

The reaction of Ba(NO₃)₂ and (NH₄)₂CrO₄ proceed as follows:

$$Ba(NO_3)_{2(aq)} + (NH_4)_2 CrO_{4(aq)} \rightarrow BaCrO_{4(s)} + 2NH_4NO_{3(aq)}$$

The results of XRD analysis (Fig. 1) showed that the solid phase product is $BaCrO_4$ as nanoparticles in the space group Pnma (JCPDS file number 35-0642)[11]. EDX indicated that the produced particles are made of barium, chromium and oxygen (Fig. 2).



Figure 1. XRD spectrum of BaCrO₄ nanoparticles



Figure 2. EDX spectrum of the product

UV-vis spectroscopy was performed in the wavelength range of 190-700 nm. The results revealed that the absorption peak occurred in the wavelength of 375 nm, complying with the reported peak of previous researches for BaCrO₄ nanoparticles $\frac{100}{100}$ (Fig. 3).



Figure 3. Absorption peak of BaCrO₄ in UV-vis analysis

TEM represents the shape of nanoparticles. The result shows that uniform nanoparticles have been formed in this study (Fig. 4). With regard to the effect of reactants' and surfactants' concentration on the nanoparticles' size, the experiments were conducted at different concentrations of reactants and stabilizers to optimize the experimental conditions. For this purpose, an experimental design procedure was followed. Variables are shown in Table 1. All samples were tested by DLS analysis to determine the size of formed particles (Fig. 5). As Table 2 depicts, increasing the $(NH_4)_2$ CrO₄ concentration leads to the reduction of the formed particles. The smaller size of the particles obtained at higher concentrations of $(NH_4)_2$ CrO₄ may be attributed to the high initial supersaturation of BaCrO₄ in the solution that, in turn, increases the nucleation rate. In fact, the high level of supersaturation causes fast nucleation, and newborn particles do not grow. On the other hand, smaller nanoparticles are formed at lower concentrations of Ba(NO₃)₂. Drop-wise addition of Ba(NO₃)₂ causes a fast reaction; as a result, clusters do not have enough time to reach a critical size, and thus previously formed particles grow and the size of particles increases. Comparison of the presence of DBSS, Na₃C₆H₅O₇·2H₂O and PVP as the stabilizers with equal concentrations shows that smaller particles are formed in the presence of $Na_3C_6H_3O_7$, $2H_2O_7$. This can be explained by better dissolving of $Na_3C_6H_5O_7$ $2H_2O$ in water, allowing more additive be added to the system, and better coverage of nanoparticles by the surfactant. Na₃C₆H₅O₇·2H₂O will prevent nanoparticles from agglomeration by its good distribution in the solution and forming a covering film on the BaCrO₄ nanoparticles. Furthermore, it was observed that the nanoparticles obtained when larger amounts of surfactant were used have a smaller size. It is concluded that at high concentrations, more surfactant molecules surround the nanoparticles, and the surfactant will stabilize the nanoparticles much more efficiently.



Figure 4. TEM image of BaCrO₄ nanoparticles





Figure 5. DLS analysis curves of the BaCrO₄ nanoparticles obtained from different experiments: (a) Run 1, (b) Run 3, and (c) Run 2

Table 2. Effect of concentrations on the size of nanoparticles

Runpl	H of solution	Size of particle (nm)	es Mean size of particles (nm)
1	7	12-35	25
2	8	25-500	290
3	8	30-170	80
4	7	50-265	111
5	7	14-50	38
6	7	34-170	79
7	11	39-180	95
8	10	80-470	217
9	11	52-270	150

The tests were conducted with different concentrations of reactants and surfactants, and the results were compared. It was revealed that, in the synthesis of $BaCrO_4$ nanoparticles, sodium citrate acts as a more effective nanoparticle stabilizer.

4. Conclusion

In this paper, $BaCrO_4$ nanoparticles were synthesized at different concentrations of $Ba(NO_3)_2$ and $(NH_4)_2CrO_4$ and surfactants. The DLS and TEM results showed that, in BaCrO4 nanoparticles synthesis, increasing the concentration of $(NH_4)_2CrO_4$ and surfactant will decrease the nanoparticles' size. Furthermore, smaller particles of BaCrO₄ are formed in the presence of Na₃C₆H₅O₇·2H₂O as a stabilizer. Using $(NH_4)_2CrO_4$ to produce BaCrO₄ nanoparticles has been performed for the first time in this research. The XRD and EDX analyses confirmed that the method yields a pure product.

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