

Preparation of transparent BaSO₄ nanodispersions by high-gravity reactive precipitation combined with surface modification for transparent X-ray shielding nanocomposite films

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Abstract BaSO₄ nanoparticles as important functional materials have attracted considerable research interests, due to their X-rays barrier and absorption properties. However, most of BaSO₄ nanoparticles prepared by traditional technology are nanopowders with broad size distribution and poor dispersibility, which may greatly limit their applications. To the best of our knowledge, the synthesis of transparent BaSO₄ nanodispersions was rarely reported. Here, we firstly present a novel and efficient method to prepare transparent and stable BaSO₄ nanodispersions with a relatively small particle size around 10 to 17 nm using a precipitation method in a rotating packed bed (RPB), followed by a modification treatment using stearic acid. Compared with the BaSO₄ prepared in a traditional stirred tank, the product prepared using an RPB has much smaller particle size and narrower size distribution. More importantly, by using RPB, the reaction time can be significantly decreased from 20 min to 18 s. Furthermore, the transparent BaSO₄-polyvinyl butyral nanocomposite films with good X-ray shielding performance can be easily fabricated. We believe that the stable BaSO₄ nanodispersions may have a wide range of applications for transparent composite materials and coatings with X-ray shielding performance for future research.

Keywords BaSO₄ nanoparticles, rotating packed bed,

transparent nanodispersions, BaSO₄-PVB films, X-ray shielding

1 Introduction

BaSO₄, as an important inorganic material, has been extensively studied over the latest few decades and widely used in plastics, paints, cosmetics, ceramics, pigments, and paper productions due to their high whiteness, acid and alkali resistance, non-toxicity, low price and X-rays absorption ability [1–5]. Compared with traditional micro-sized BaSO₄ particles, BaSO₄ nanoparticles normally have better gloss and strength properties, because of their high specific surface area, high activity and good dispersion properties [5,6].

There are a number of BaSO₄ nanoparticles preparation methods, such as direct precipitation [7–10], microreactor [11,12], microemulsion [13,14] and template method [15,16]. For example, the direct precipitation is a widely used method, simply adding one precipitating agent to a metal salt solution to get ultrafine BaSO₄ nanoparticles. The existed problem for this method is that the product size is hard to control and size distribution is relatively broad [7,9]. Microreactor method usually involves using a designed microreactor, which contains diverse small channels (equivalent diameter less than 500 μm) to produce even and relatively small BaSO₄ nanoparticles, but with a low production capacity [11]. The BaSO₄ nanoparticles made through microemulsion method are controllable and relatively small, however, the usage of surfactants, co-surfactants and solvents may cause some

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environmental pollutions [13]. The template method means designing templates for preparing special structure BaSO₄ nanoparticles, according to the performance requirements and morphology of synthetic materials, but most of the processes are complicated, high-priced and not suitable for large-scale preparation [15]. Therefore, it is necessary to develop a simple, cost-effective, highly efficient and environmentally friendly manufacturing technology to produce uniform and well dispersed BaSO₄ nanoparticles for various applications.

In addition, most of BaSO₄ nanoparticles prepared by above methods are nanopowders, which may have an uneven particle size and aggregation problems [17–24], and these will significantly affect their application performance. For example, when adding these nanopowders to certain polymers to form X-ray shielding polymer composite materials, such as plastics and rubber, the aggregated nanopowders in the polymers will affect the materials mechanical properties, like rigidity and bending strength, and will also result in an uneven radiation shielding performance. Another example is that when applying BaSO₄ nanopowders to high-end paints or ink products, the poor dispersibility or aggregation problems may affect the smoothness and toughness of the paint products. Compared with conventional filled composite materials, when adding nanodispersions or well-dispersed nanoparticles to another medium for synthesizing compatible and functionalized composite materials, it can lead to enhanced material properties, such as hardness, toughness, heat resistance and ultraviolet weather resistance of these materials [25–28].

BaSO₄ nanoparticles are ideal raw materials or precursors for X-rays absorption. For example, when using fabrics containing BaSO₄, the X-ray transmittance of fabric can be significantly reduced [29]. In addition, cellulose nanocomposite films containing BaSO₄ with a porous transparent structure have a new X-ray shielding properties, and the shielding performance of this material against X-rays can be increased by simply increasing the composite film layers [30]. More importantly, for some special applications, such as glass windows in X-ray examination room, glass window coatings for aerospace vehicles and sandwich filler materials of radiation shielding glass, these products not only require the raw materials having a good X-ray shielding capability, but also need to ensure a relatively good transparency. This is hard to achieve transparent shielding composite materials, when using white BaSO₄ nanopowders. Therefore, there is also need for transparent BaSO₄ nanodispersions, according to the above special applications.

Rotating packed bed (RPB) is an intensification strengthening device, which can generate tens to hundreds of gravitational accelerations (g), and is designed to minimize the equipment scale [31,32]. In a typical RPB, when two reactants enter the inner cavity of rotor from inlets, they are sprayed on the inner edge of rotor through

the nozzle. Then the liquids will reach the packing, and are divided and broken into much smaller liquid droplets to form large and constantly updated surfaces, which can greatly increase the contact area between the reactants, and make the product more uniform and fine [33–35]. RPB is definitely beneficial to form homogeneous nucleation and growth of particles. Until now, RPB has been successfully used for various nanoparticle preparations, including Pd, ZrO₂, SiO₂, CaCO₃, hydroxyapatite, polyaniline, fluorescent dyes and polyurethane [36–44].

To the best of our knowledge, the synthesis of transparent and stable BaSO₄ nanodispersions was rarely reported before. In this work, we firstly present an efficient method to prepare transparent BaSO₄ nanodispersions using a precipitation method in RPB, followed by a modification treatment. The BaSO₄ nanoparticles prepared by RPB were smaller and more regular than those made in a conventional stirred tank reactor (STR). Different rotating speeds and feed flow ratios were explored to determine the optimal reaction conditions. Furthermore, the BaSO₄ nanodispersions prepared under the optimal conditions were applied, specifically applied to prepare X-ray shielding transparent BaSO₄-polyvinyl butyral (PVB) composite films, which were easily fabricated through a cost-effective and efficient solution blending method [45–47].

2 Experimental

2.1 Chemicals

Barium chloride dihydrate (BaCl₂·2H₂O), methanol, ethanol, and concentrated H₂SO₄ (98 wt-%) were purchased from Beijing Chemical Reagent Co., Ltd. Stearic acid was purchased from Fuchen Chemical Reagent Co., Ltd. (Tianjin). PVB was obtained from Sinopharm Chemical Reagent Co., Ltd. (China). All the chemical reagents were analytically pure and used without further purification. Deionized water was provided by a water purification system (RO-DI plus, Hitech, PRC).

2.2 Preparation of BaSO₄ nanodispersions

In a typical precipitation process using an RPB, 1.32 g of BaCl₂·2H₂O was dissolved in 90 mL of methanol, and was stored in container 1. Then 0.378 g of stearic acid as a surfactant was added into container 1. On the other hand, 90 mL of methanol solution containing 0.288 mL of H₂SO₄ (98 wt-%) was prepared in container 2. The solution from container 1 and container 2 were pumped at the same time into the RPB using peristaltic pumps, both at flow rates of 300 mL/min, 1500 r/min and 60 °C. After that, the resulting suspension was transferred to a flask for further reaction at 60 °C for 3 h. Subsequently, the suspension was filtered and washed with acetone and

ethanol for several times. Finally, the resulting wet cake was re-dispersed in ethanol to form BaSO_4 dispersions. The process of preparing BaSO_4 dispersions by an RPB is shown in Fig. 1.

As a control experiment, the BaSO_4 dispersion was also prepared in an STR, all reaction conditions including reactant concentrations, modification temperatures, heating duration for an STR experiment were the same as the set for an RPB experiment. The only difference is that the solution in container 2 was added dropwise to container 1 in an STR experiment, which took up to 20 min to finish the addition process. The duration of this process is much longer than 18 s by an RPB, which reckons by time when the two solutions enter the RPB inlets by the peristaltic pump until all the generated BaSO_4 flowing out of the outlet.

In addition, an STR experiment with 500 r/min stirring speed was set up for preparation of BaSO_4 ethanol nanodispersions. In order to test the intensification performance and effect on different rotating speeds of RPB, 500, 1500 and 2500 r/min were chosen, respectively, for three RPB experiments as comparisons.

2.3 Preparation of transparent BaSO_4 -PVB composite films

The transparent BaSO_4 -PVB composite films were prepared using a solution blending method as follows [45]. Polyethylene terephthalate was chosen as the substrate and pre-cleaned by deionized water and ethanol before experiments. Firstly, a certain amount of PVB was dissolved in ethanol with heating at 55 °C to form a PVB ethanol solution. The BaSO_4 ethanol dispersion was slowly added to the PVB ethanol solution and cooled down to room temperature. The resulting mixture was stirred and allowed to stand until a homogeneously clear solution was obtained. Then the above mixture solution was coated onto the surface of the pre-treated a 5 cm × 5 cm polyethylene terephthalate-based self-made mold and baked in an oven at 40 °C for 1 h, and 80 °C for another hour to obtain the BaSO_4 -PVB nanocomposite films. The

films were kept at room temperature for 24 h to ensure the complete evaporation of organic solvent.

2.4 Characterization

The size and morphology of BaSO_4 nanoparticles were examined using a transmission electron microscope (TEM, JEOL, JEM-2010F, Japan). Nano Measurer software was used to perform particle size statistics on TEM images. The internal structure of the nanocomposite film was examined using a scanning electron microscope (SEM, JEOL, JSM-6701, Japan). The crystal structure of BaSO_4 nanoparticles was measured using an X-ray diffractometer (XRD, XRD-6000, Shimadzu, Japan). The transparency of both BaSO_4 nanodispersions and nanocomposite film was characterized by UV-Vis spectrometer (Shimadzu UV-2501, Japan) in the range of 200–800 nm. The Fourier transform infrared (FTIR) spectra were recorded in a range of 4000–400 cm^{-1} using a Nicolet 6700 spectrometer (Nicolet Instrument Co., USA). The thermal behavior was evaluated by thermogravimetric analysis (TGA) in nitrogen at a heating rate of 20 °C/min using a METTLER TOLEDO TGA/DSC1 SF 1100 system.

The X-ray shielding performance of the nanocomposite film was evaluated using an X-ray tube (10–160 kV, YXLON, Germany) from the Institute of Ionization Radiation of National Institute of Metrology, China. The process of X-ray shielding performance test is shown in Fig. 2. The X-rays emitted by the X-ray tube pass through a beam-limiting diaphragm first, and then pass through an additional filter. At this time, the X-rays can be viewed as a narrow beam. There will also be a beam-limiting diaphragm where the shielding material (composite film) is measured to ensure that the X-rays pass through the film as a narrow beam. The shielding film is measured 100 cm away from the focal point of the X-ray tube, and the detector is located 200 cm away. The radiation field can be blocked by the diaphragm at 100 cm, and will not directly irradiate the detector. The X-rays emitted from the X-ray machine can be tested by the detector after passing through

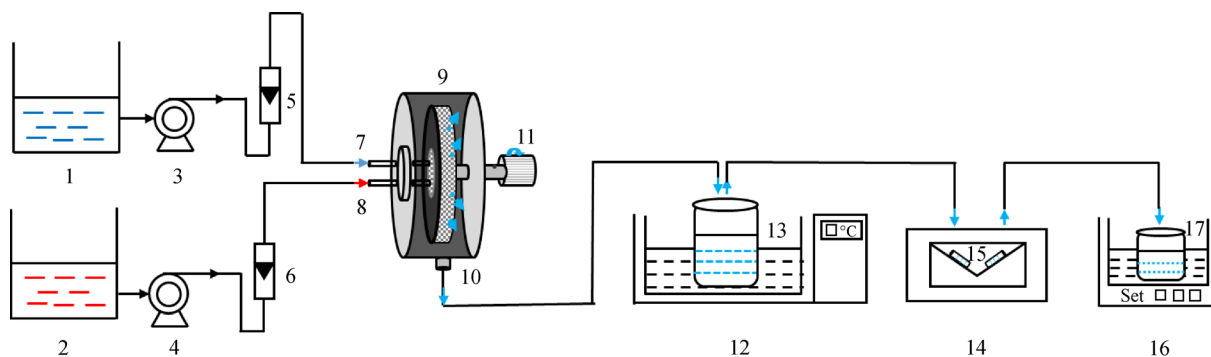


Fig. 1 Schematic diagram of the preparation process of BaSO_4 nanoparticles in an RPB: (1) BaCl_2 and stearic acid methanol solution container, (2) H_2SO_4 methanol solution container, (3/4) pump, (5/6) flowmeter, (7/8) inlet, (9) RPB, (10) outlet, (11) motor, (12) water bath, (13) flask, (14) centrifuge, (15) centrifuge tube, (16) sonicator and (17) BaSO_4 dispersions.

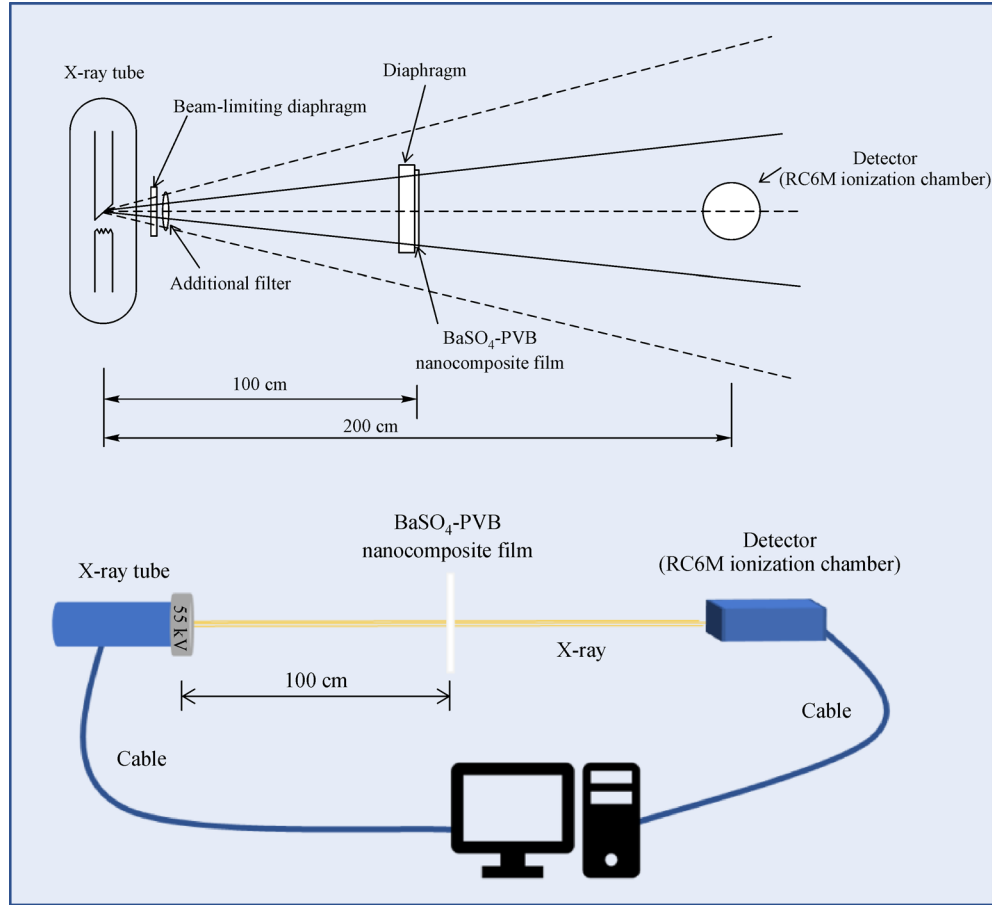


Fig. 2 Schematic diagram of X-ray shielding performance test.

the shielding film, and the detector converts the optical signal into an electrical signal, which will transmit to the computer. The shielding properties of as-prepared films can be characterized by half-valued layer (HVL) and tenth-valued layer (TVL). The half-value layer means the thickness of the material layer required to weaken the incident X or gamma-ray photon number to 50%. Relation of HVL to linear attenuation coefficient (μ) is defined by the following equation [48]:

$$I = I_0 \left(\frac{1}{2} \right)^n, \text{ where } n = 1, I = I_0 \exp(-\mu x),$$

$$\ln 2 = \mu x_{1/2},$$

$$x_{1/2} = \frac{\ln 2}{\mu}, \text{ where } x_{1/2} = \text{HVL.} \quad (1)$$

Similarly, the tenth-valued layer means the thickness of the material layer that reduces the incident X or gamma-ray photon number to 10%. Relation of TVL to linear attenuation coefficient (μ) is defined by the following equation [48]:

$$I = I_0 \left(\frac{1}{10} \right)^n, \text{ where } n = 1,$$

$$\ln 10 = \mu x_{1/10},$$

$$x_{1/10} = \frac{\ln 10}{\mu}, \text{ where } x_{1/10} = \text{TVL.} \quad (2)$$

And μ can be calculated according to the following formula:

$$\mu = \frac{\ln \left(\frac{I_0}{I} \right)}{d}, \quad (3)$$

where I is the intensity of attenuation beam, and I_0 is the incident intensity.

3 Results and discussion

Figure 3 shows the TEM images of BaSO₄ nanoparticles prepared by an STR or RPB, and their corresponding

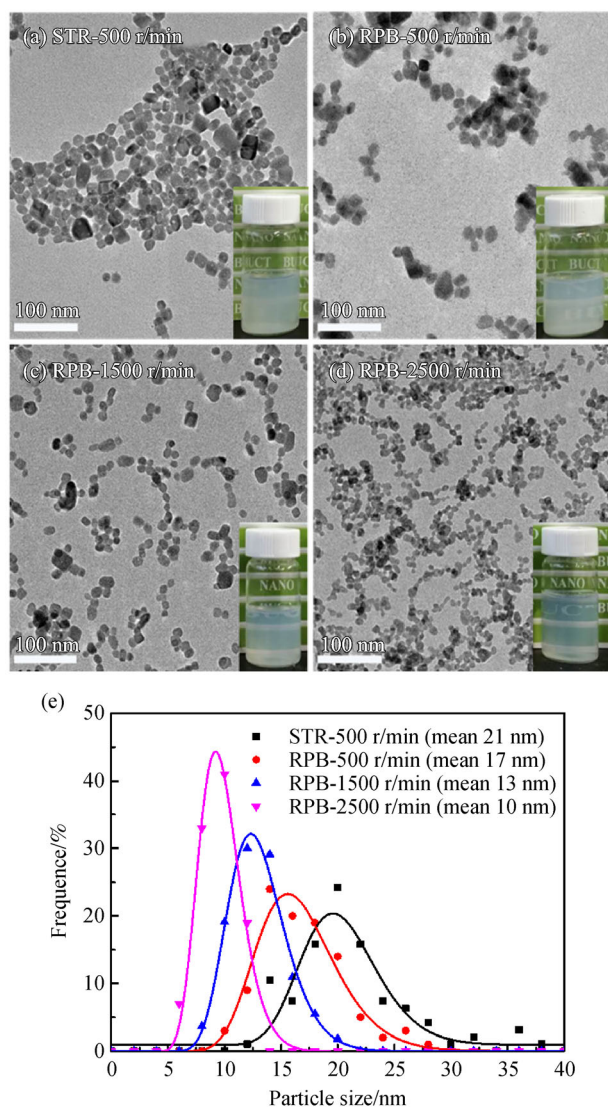


Fig. 3 TEM images of BaSO_4 nanoparticles prepared at different rotating speeds: (a) using STR at 500 r/min, (b) using RPB at 500 r/min, (c) using RPB at 1500 r/min, (d) RPB at 2500 r/min, and (e) the corresponding particle size distributions.

digital photographs of the BaSO_4 nanodispersions (inset). The solid content of ethanol BaSO_4 nanodispersions in the digital photographs are all about 1 wt-%. The BaSO_4 ethanol nanodispersions are transparent and stable, which can be stored for a few months without any sediments. It can be seen from Figs. 3(a)–3(d) that the well dispersed BaSO_4 nanoparticles with irregular shapes can be prepared for all samples under these experimental conditions. The reaction time using an RPB is 18 s, which is much shorter than 20 min using an STR. More importantly, it can be seen from Fig. 3(e) that the particle size of the BaSO_4 nanoparticles prepared using RPB at 500 r/min is smaller, and the size distribution (10–25 nm) is narrower than that using STR at the same stirring speed. According to the statistics results, the average particle size of BaSO_4 nanoparticles prepared by STR is around 21 nm, and the ones prepared by RPB decreases with increasing the RPB speed from 500, 1500 and 2500 r/min, to around 17, 13 and 10 nm.

RPB can create a high-gravity environment, which greatly strengthens the micro-mixing effect for particle preparation [35,36,38,39,42]. The two reactants BaCl_2 methanol and H_2SO_4 methanol solutions are sheared and torn into small liquid droplets by the packing inside RPB, which can increase the reaction contact areas and shorten the reaction time. This is the reason why the BaSO_4 nanoparticles prepared in RPB have shorter reaction time and smaller size than those prepared by the STR under the same speed. With increasing the rotating speed of RPB, the intensity of micro-mixing are also enhanced, therefore, the average particle size will gradually decrease with increasing the rotating speed. When the rotating speed is increased to a certain value, the cutting and mixing effects will finally reach the maximum.

Figure 4 displays the representative XRD patterns and FTIR spectra of BaSO_4 nanoparticles prepared in an STR and RPB, respectively. It can be seen from Fig. 4(a) that all of the peaks of the three samples are in good agreement with the reported values (PDF#24-1035). Here, pure BaSO_4 particles were made using the same method in

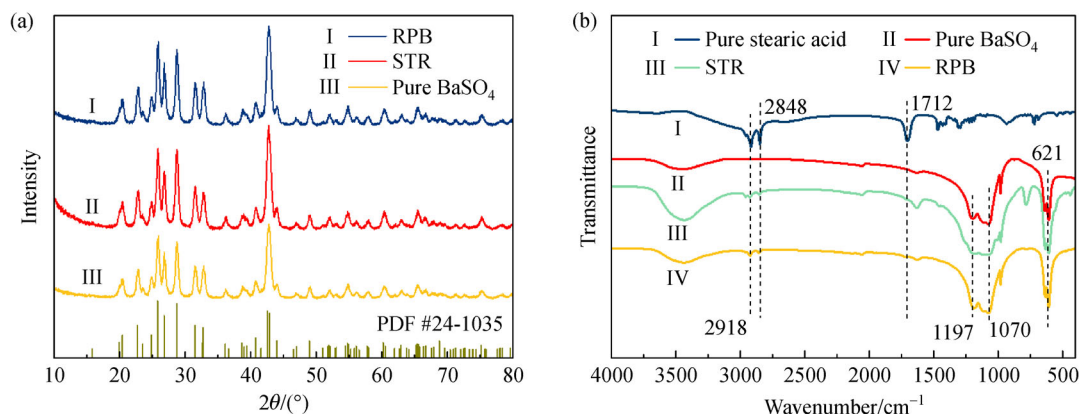


Fig. 4 (a) XRD patterns and (b) FTIR spectra of BaSO_4 nanoparticles prepared in RPB and STR, and the pure micro-sized BaSO_4 .

STR without adding the surfactant stearic acid. The characteristic peaks of three samples are relatively strong, and there are no impurity characteristic peaks. This demonstrates that the BaSO₄ nanoparticles with a high purity can be successfully prepared by using this precipitation method. In addition, BaSO₄ products prepared in STR and RPB have the slightly higher diffraction peaks compared to pure BaSO₄ particles, which suggests a higher crystallinity.

Figure 4(b) shows the FTIR spectra of BaSO₄ nanoparticles prepared in RPB and STR, and pure BaSO₄. The FTIR absorption peaks at 2918 and 2848 cm⁻¹ are ascribed to the antisymmetric stretching vibration and the out-of-plane bending vibration of -CH₃ and -CH₂- of stearic acid, which can also be seen for BaSO₄ samples prepared in RPB and STR, indicating that the surfactant was indeed adsorbed on the surface of BaSO₄ nanoparticles. The absorption peak at 1712 cm⁻¹ is attributed to the -C=O- vibration, which cannot be seen

for samples prepared by STR and RPB. This indicates that the -COOH group of stearic acid may have already reacted with BaSO₄ nanoparticles. The absorption peak appears at about 3440 cm⁻¹ should be due to stretching vibration of O-H in water. The other peaks at 1917, 1070 and 621 cm⁻¹ are assigned to the stretching and bending vibration of BaSO₄. All the above evidences suggest that BaSO₄ nanoparticles with high purity have been successfully prepared in both STR and RPB, and stearic acid has successfully covered on the surface of BaSO₄.

RPB feed flow ratio as an important factor for the BaSO₄ nanoparticles preparation was also explored to determine the optimal reaction conditions. Figures 5(a–c) present the TEM images of dispersions prepared at different RPB feed flow ratios. The three different feed flow rates were set to 3:1, 1:1 and 1:3. For example, the feed flow rate of first group was set to 300 mL/min of BaCl₂ methanol solution and 100 mL/min of H₂SO₄ methanol solution, and the second group was set to 300 mL/min of BaCl₂ methanol solution and 300 mL/min of H₂SO₄ methanol solution. The third group was set to 100 mL/min of BaCl₂ methanol solution and 300 mL/min of H₂SO₄ methanol solution. The average particle size and corresponding particle size distribution are shown in Fig. 5(d).

According to Fig. 5, it can be intuitively seen that the obtained nanoparticles are smaller and the morphology is more regular when the feed flow ratio is 1:1. The average particle size of these three samples are around 16, 13, and 17 nm. The particle size distribution of 1:1 feed flow rate is also narrower than the other two groups. This should be attributed to the synergistic effect of both mixing intensity and supersaturation [40]. In addition, the theoretical reaction ratio is 1:1 for the reaction of H₂SO₄ and BaCl₂, so the uniform 1:1 feed may completely react to the reactants and promote rapid nucleation, which is suitable for generating nanoparticles with smaller particle size. Therefore, the 1:1 feed flow ratio was selected as an optimal condition.

Figure 6 shows the possible formation process illustration of BaSO₄ nanodispersions. Moreover, TGA was also performed to further verify the specific content of stearic acid coated on the surface of BaSO₄ for this modification reaction in Fig. 7. From the weight loss curves, when the

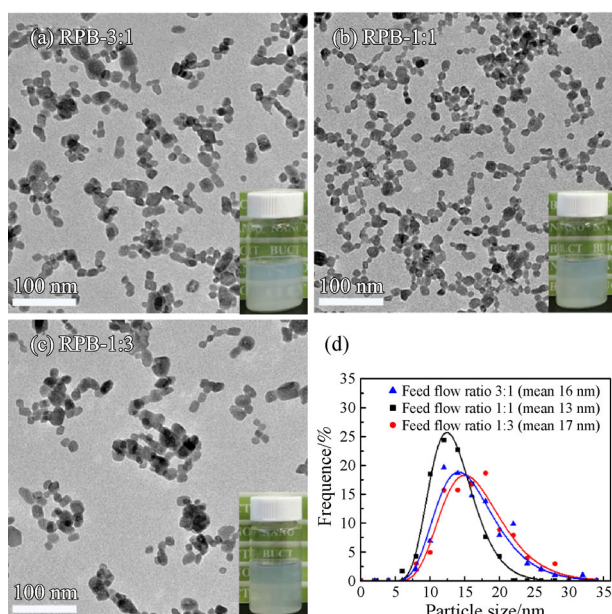


Fig. 5 TEM images of BaSO₄ nanoparticles prepared at different RPB feed flow ratios (BaCl₂:H₂SO₄): (a) 3:1, (b) 1:1, (c) 1:3, and (d) the corresponding particle size distributions.

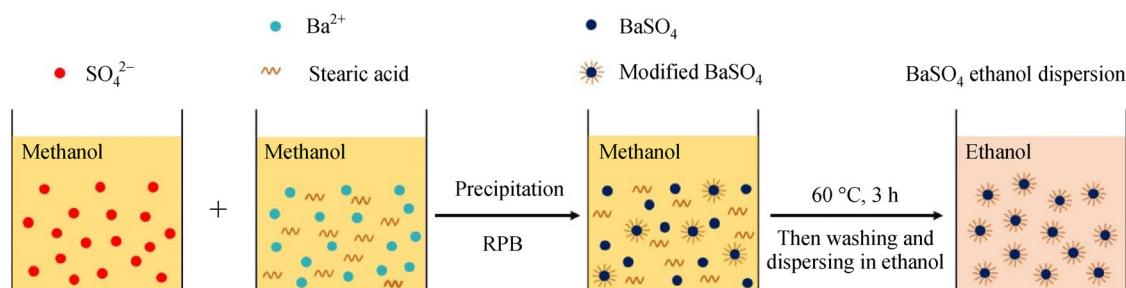


Fig. 6 Possible formation process illustration of BaSO₄ nanodispersions.

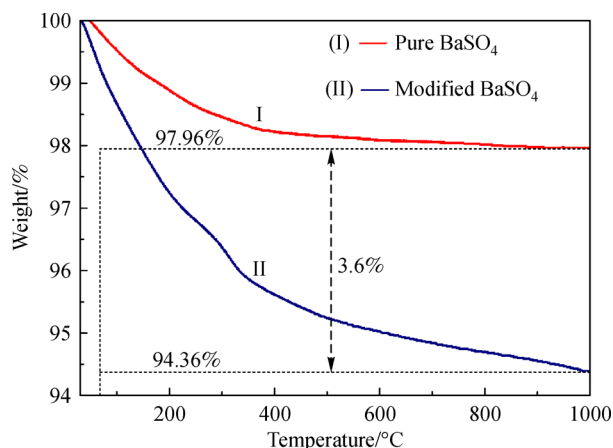


Fig. 7 TGA curves of pure and modified BaSO_4 nanoparticles prepared in RPB.

temperature was changed from room temperature to 1000 °C, the quality of pure BaSO_4 was only reduced by about 2 wt-%, which may be caused by the interlayer water and a small amount of surface adsorbed water. The modified BaSO_4 samples have a total weight loss of 5.6 wt-%. Except for the interlayer water and surface adsorbed water, the weight loss should be due to the loss of stearic acid on the surface of the BaSO_4 , when the temperature was increased from 220 to 1000 °C. Compared with the TG data of pure BaSO_4 , it can be calculated that the quantity of stearic acid combined on the surface of the BaSO_4 should be about 3.6 wt-%. Based on the same preparation process, the modified BaSO_4 nanoparticles can also be dispersed in ethylene glycol and water to form transparent and stable BaSO_4 ethylene glycol and aqueous dispersions, as shown in Fig. 8.

The as-prepared transparent BaSO_4 ethanol nanodispersions were further used for fabricating BaSO_4 -PVB nanocomposite films. Figure 9 displays the digital photograph and transmission of the BaSO_4 -PVB films with different BaSO_4 content. The thickness of BaSO_4 -PVB films is all around 0.4 mm. As shown in Fig. 9(a), when the amount of BaSO_4 is increased from 0 to 50 wt-%,

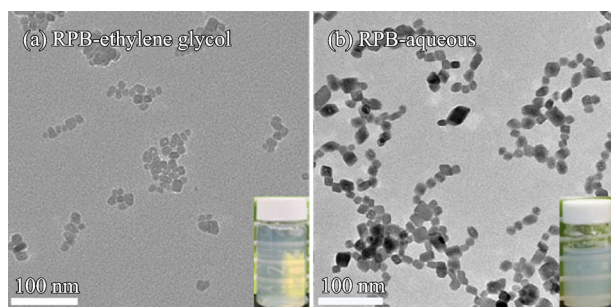


Fig. 8 TEM images and digital photographs of (a) ethylene glycol and (b) aqueous BaSO_4 nanodispersions.

the characters of the background become more and more unclear through the film. When the BaSO_4 content reaches 30 wt-%, BaSO_4 -PVB films start to have a little milky white color. It is worth mentioning that the film is still transparent, and we can see the words on the background board, even when the content of BaSO_4 accounts for 50 wt-% of the PVB. It can be seen from Fig. 9(b) that the visible light transmittance of all nanocomposite films at the visible light range exceeds 40%, and the visible light transmittance at 555 nm, which is the most sensitive of the human eye, are 87.9%, 85.1%, 75.1%, 61.2% and 59.1% for nanocomposite films A to F. The decline of the visible light transmittance value might be mainly because the light scattering on the BaSO_4 nanoparticles [30]. The high visible light transmittance of the films should be ascribed to the small particle size and good dispersity in the composite films.

The SEM images of cross-section of BaSO_4 -PVB films with different BaSO_4 content are shown in Fig. 10. The cross-sectional SEM images show that the BaSO_4 nanoparticles are nearly uniformly dispersed in PVB medium. However, with increasing the BaSO_4 content from 0 to 50 wt-%, it can be seen that there exists a slight agglomeration of BaSO_4 nanoparticles in PVB.

Figure 11 shows the HVL and TVL of BaSO_4 -PVB nanocomposite films under different tube voltages. As we can see from Fig. 11, the value of both HVL and TVL are smaller when using a lower tube voltage under the same BaSO_4 content, which indicates the X-ray shielding performance of the film is more obvious at 55 kV. With

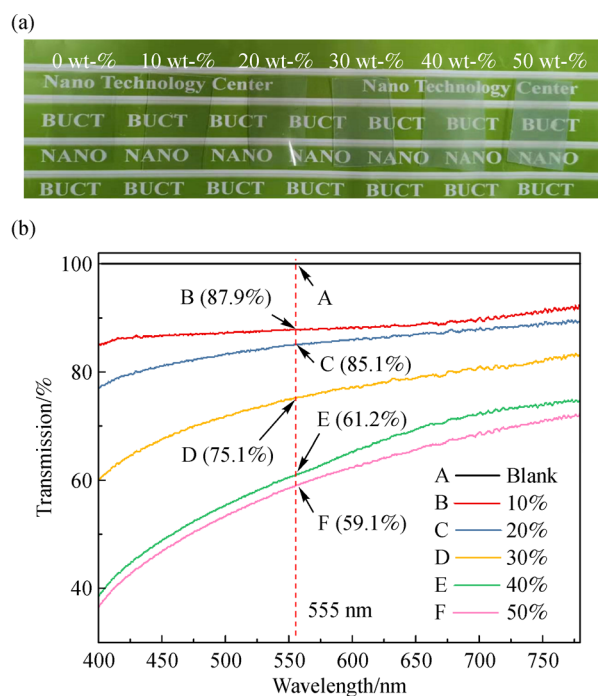


Fig. 9 (a) Digital photograph and (b) transmission of BaSO_4 -PVB films with different BaSO_4 content.

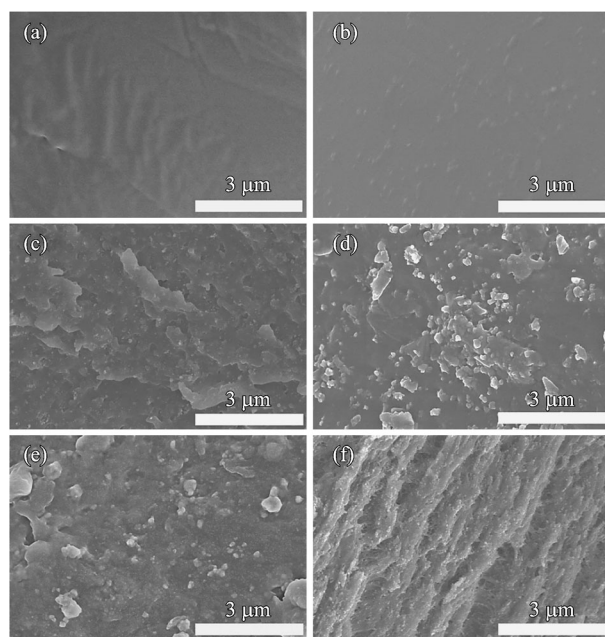


Fig. 10 SEM images of cross-section of BaSO₄-PVB nanocomposite films with different BaSO₄ content: (a) 0 wt-%, (b) 10 wt-%, (c) 20 wt-%, (d) 30 wt-%, (e) 40 wt-% and (f) 50 wt-%.

increasing the BaSO₄ content under the same tube voltage, the value of both HVL and TVL have a similar descending trend, which indicates that the shielding performance against X-rays can be easily enhanced with increasing the BaSO₄ content. Therefore, it can be concluded that the BaSO₄-PVB nanocomposite films prepared by the solution blending method have an obvious shielding property against X-rays and are possible to be used for transparent composite material preparation with X-ray shielding performance for future research. Considering both transparency and X-ray shielding performance, the content of 30 wt-% BaSO₄ can be selected as the optimal concentration for the films, which have a relatively high transpar-

ency of 75.1%, HVL of 0.11 cm and TVL of 0.38 cm under 55 kV.

Barium is a heavy metal element, and mass of its inner core is large. The X-rays are difficult to penetrate from heavy metal cores, therefore, the energy of X-rays is easily absorbed by the heavy metals during the X-rays collision. The schematic diagram of the preparation of the BaSO₄-PVB nanocomposite film and its X-ray shielding mechanism is shown in Fig. 12. BaSO₄ nanoparticles are evenly dispersed in the film and provide X-ray shielding abilities. There were two routes to contribute the X-ray attenuation, including reflection loss and absorption loss. When X-rays enter the shielding layers, part of X-rays is reflected by the surface of the composite film, which is called the reflection loss. Another part is absorbed by the BaSO₄ nanoparticles inside the composite films when passing through the shielding layers, which is called the absorption loss. The remaining part is not reflected or absorbed, which penetrates the shielding layer, as called transmitted X-rays [30]. If the content of BaSO₄ nanoparticles in the composite film is higher, the absorption loss of the film will be higher, the transmitted X-rays from the composite film will be less, and the shielding performance of composite film should be better.

The radiation shielding materials have always been studied by other researchers [29,30,46–49]. For instance, Kaewjaeng et al. [48] have prepared the La₂O₃-CaO-B₂O₃-SiO₂ glasses, the thickness of glass is about 3 mm and the HVL of glass with 30 mol-% La₂O₃ content is 0.18 at a 100 kV tube voltage. The films we prepared have higher HVL under the same conditions, and the film thickness is only 0.4 mm. Other reported composite materials, such as Gd₂O₃/epoxy composite films [49], polypyrrole/Pb nanocomposites [50] and a tungsten functional paper [51], indicate a better X-ray shielding performance than our BaSO₄-PVB films, but with an opaque capability. The HVL of commercial window and commercial X-ray shielding window at 120 kV are 0.7 and 0.23, respectively [48]. In view of the experimental conditions, the HVL of

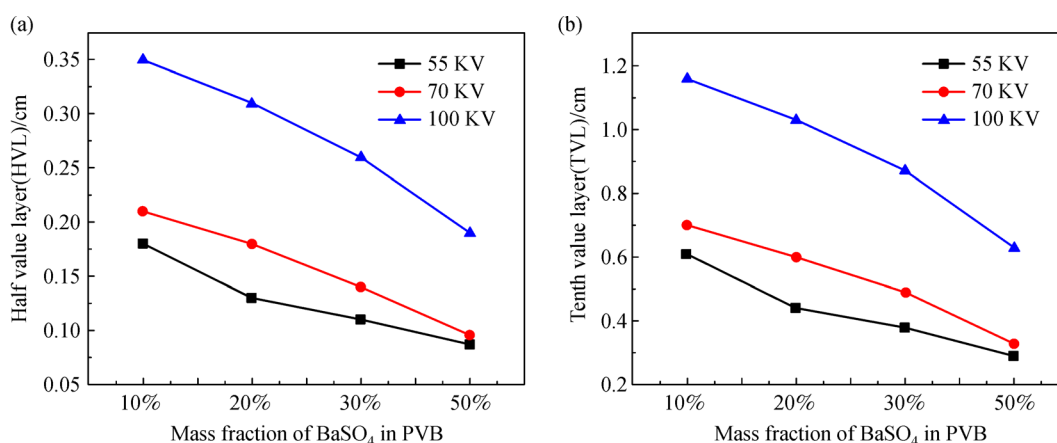


Fig. 11 (a) HVL and (b) TVL of BaSO₄-PVB nanocomposite films under different tube voltages.

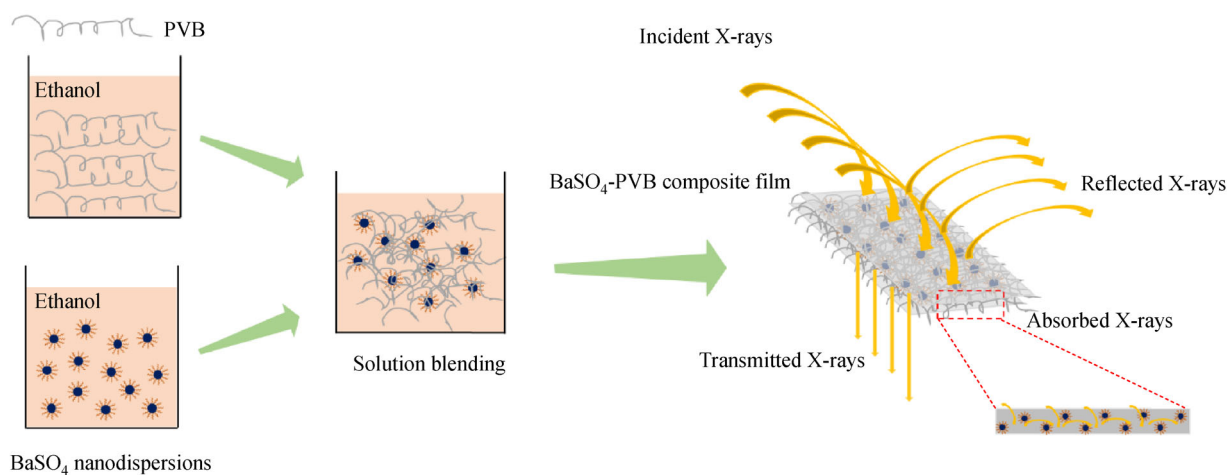


Fig. 12 Schematic of preparation of BaSO₄-PVB nanocomposite film and the X-ray radiation shielding mechanism.

our X-ray shielding film with 30 wt-% BaSO₄ content at a tube voltage of 100 kV is 0.26. The shielding capability is comparable to commercial X-ray shielding windows at a tube voltage of 120 kV. The advantage of our as-prepared composite films is possessing a high transparency even with large addition of BaSO₄ nanodispersions. For the X-ray shielding properties, there is still room for improvement in future study.

4 Conclusions

In this work, we have successfully prepared transparent BaSO₄ ethanol nanodispersions using a precipitation method in RPB, followed by a modification treatment using stearic acid. The optimal conditions were obtained as follows. The rotating speed of the RPB was 2500 r/min, and the feed flow ratio was 1:1. Compared with the conventional STR, the product prepared using RPB has a smaller average particle size and a narrower size distribution. The reaction time can be significantly decreased from 20 min to 18 s. The BaSO₄ ethanol nanodispersions are transparent and stable and can all keep for several months without any precipitations. Except for ethanol dispersions, the modified BaSO₄ nanoparticles can also be dispersed in ethylene glycol and water to form transparent and stable BaSO₄ ethylene glycol and aqueous dispersions. More importantly, based on the transparent BaSO₄ ethanol nanodispersions, the transparent nanocomposite films with X-ray shielding properties have been successfully prepared by a solution blending method. Considering both transparency and X-ray shielding performance, the content of 30 wt-% BaSO₄ is selected as the optimal concentration for the BaSO₄-PVB film. It is foreseeable that such stable and transparent BaSO₄ dispersions may have great application prospects for transparent composite material

perpetration and coatings with X-ray shielding performance for future research.

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