

# Research Progress on Synthesis of Nano Strontium Titanate for Photocatalytic Applications

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**Abstract.** Environmental pollution and energy shortages pose a significant threat to human life and development in today's industry. Therefore, this essay aims to review the various methods used in the synthesis of nano strontium titanate photocatalysis. These synthesis methods can be tailored to meet different research needs and control the morphology, size, and properties of materials under varying conditions. The methods of synthesis include the solid-phase method, hydrothermal synthesis method, sol-gel method, chemical coprecipitation method, ultrasonic chemical method, and template method. This paper will analyze the development of nano strontium titanate photocatalytic materials and provide valuable support for the control of environmental pollution and energy shortages.

**Keywords:** Nano strontium titanate, environmental pollution, synthesis, photocatalysis, hydrothermal method.

## 1. Introduction

With the development of the times, human beings are facing increasingly serious environmental problems and energy shortages. Environmental problems mainly include water pollution, air pollution and noise pollution and so on. These pollution have caused irreversible harm to human health and the environment. In recent years, the government has begun to encourage the concept of sustainable development and environmental awareness. The development and application of photocatalytic materials provide new ways to solve environmental and energy problems. Since A. Fujishima and K. Honda<sup>[1]</sup> research in 1972 that n-type semiconductor  $\text{TiO}_2$  electrodes can undergo water decomposition under light, the photocatalytic properties of semiconductors have begun to be well known to the public as well as widely studied. Nevertheless, it is important to screen and prepare photocatalysts with efficient performance in photocatalytic reactions.

$\text{SrTiO}_3$  is a new type of photocatalytic material which has perovskite structure with high dielectric constant, low loss and stability at high temperatures<sup>[2]</sup>. It can be used in the field of photoelectrochemistry such as electronic manufacturing, magnetic materials, photoelectric devices<sup>[3]</sup>. Meanwhile,  $\text{SrTiO}_3$  has a special crystal structure with crystal faces (100), (110) and (111) and a large number of defects such as oxygen vacancies. The defects and exposed surfaces promote the dissociation of exciton into free carriers, and  $\text{SrTiO}_3$  has a high carrier mobility, which gives it excellent photocatalytic properties<sup>[4]</sup>. Thus, it has also been widely utilised in the field of photocatalysis, for example, photocatalytic degradation of organic pollutants, water pollution and atmospheric pollution and other environmental problems<sup>[5]</sup>. On the other hand, it is also applied to new energy technologies such as photovoltaic cells, photovoltaic power generation, and photocatalytic water splitting<sup>[6-7]</sup>.

Nonetheless, the progress in the synthesis of  $\text{SrTiO}_3$  suffers from the problems of difficult to control, and high cost. Due to the wide energy band of  $\text{SrTiO}_3$ , it only shows photocatalytic properties under ultraviolet light, which leads to the limitation of its light utilization and photocatalytic performance<sup>[8]</sup>. Therefore, it is important to explore good synthetic methods to achieve the improvement of the full light utilization and photocatalytic performance of  $\text{SrTiO}_3$  for wide applications. With the continuous progress of science and technology, more new synthesis methods have been developed and

investigated to obtain SrTiO<sub>3</sub> with various method. This paper presents a comprehensive analysis and study on the synthesis methods of SrTiO<sub>3</sub> based on photocatalytic applications at both domestic and abroad to provide technical support for the environmental pollution as well as energy shortage.

## 2. Solid-phase method

The solid-phase method involves milling a certain proportion of oxide powder with strontium and titanium atoms until homogeneous, then reacting at a specific temperature and time, and finally milling to obtain SrTiO<sub>3</sub> powder<sup>[9]</sup>. The advantages of this method are simple process, and easy to operate, etc., but there are also some disadvantages, such as low purity and large particle size of the final product leading to difficulties in the production of nanopowders<sup>[10]</sup>.

In Wu<sup>[11]</sup> research, the high temperature solid-phase method to synthesize nano strontium titanate was proposed, SrCO<sub>3</sub> and TiO<sub>2</sub> were used as starting materials, and then mixed evenly and ground. And then, the mixture would be calcined at 1100°C for two hours. Finally, the SrTiO<sub>3</sub> powder with cubic crystalline particle size of about 500 nm was created. Although the SrTiO<sub>3</sub> powder synthesized by high-temperature solid-phase method had a strong absorption in the ultraviolet band, not in the visible region, which can be seen that its photocatalytic activity is weak. However, the researchers further used hydrothermal synthesis method to obtain the composite materials of La-WO<sub>3</sub> and SrTiO<sub>3</sub>, and the photocatalytic effect was significantly improved.

According to Liu et al research<sup>[12]</sup>, a lower temperature solid-phase method was used in the experiment, which is more environmentally friendly and cost-effective but has a limited application. Firstly, a certain proportion of TiO<sub>2</sub> and Sr(NO<sub>3</sub>)<sub>2</sub> was ground and calcined at 600°C for 10 hours. After cooling to room temperature, impurities were removed with aqueous HNO<sub>3</sub> solution, and finally dried in air at 80°C to obtain SrTiO<sub>3</sub> with particle size of 30 to 108 nm. The research showed the method was simple and cost effective.

As for Ding et al research<sup>[13]</sup>, a low temperature solid-phase method was also been taken to synthesize SrTiO<sub>3</sub>. By first, TiCl<sub>4</sub> was hydrolyzed into TiOCl<sub>2</sub> aqueous solution. Chloride ions were removed using NH<sub>3</sub>·H<sub>2</sub>O solution to form H<sub>2</sub>TiO<sub>3</sub>. Then, it was mixed with Sr(OH)<sub>2</sub>·8H<sub>2</sub>O before grinding. Lastly, the mixture was dried at 40°C for 5 h to obtain SrTiO<sub>3</sub> with a particle size of 40 nm. The experimental results showed that this method had a short reaction time, did not use solvents so that there was little pollution of the environment and it could effectively avoid the phenomenon of agglomeration.

On top of that, solid-phase method includes high-temperature solid-phase method and low-temperature solid-phase method. Although the low-temperature solid-phase method has little pollution to the environment, it needs to use other experimental method to improve the reactivity of the starting materials. Furthermore, there exists a large amount of energy consumption and difficult to be applied on a large scale industry, so there is less research for it. While the high temperature reaction is short time and easy to operate, but it has high energy consumption, non-uniform particle size, difficult to control the morphology and purity, resulting in poor photocatalytic performance of the synthesised products<sup>[10]</sup>.

## 3. Hydrothermal Synthesis method

In hydrothermal synthesis method, a certain proportion of reactants and solvents would be placed in a tank reactor, and the reaction would be carried out under appropriate hydrothermal conditions<sup>[2]</sup>. The advantage of this method was that nanomaterials with specific morphology, size and crystal structure could be synthesized under relatively mild conditions<sup>[14]</sup>.

According to Kobayashi et al research<sup>[15]</sup>, Sr(OH)<sub>2</sub>·8H<sub>2</sub>O and titanium (IV) bis (ammonium lactato) dihydroxide solution [C<sub>6</sub>H<sub>10</sub>O<sub>8</sub>Ti·2(NH<sub>4</sub>)] were acted as starting materials. Then, they were mixed together and put into tank reactor to hydrothermal heated at 150°C for 72-120h. Finally, SrTiO<sub>3</sub> with

diameter of 20-40 nm was synthesized. The results showed that it could be applied to NO gas degradation. The organic hydrothermal synthesis method resulted in a high specific surface area of SrTiO<sub>3</sub> of about 33.1 m<sup>2</sup>/g compared to conventional methods such as solid-phase reaction, which was only 2.88 m<sup>2</sup>/g. The high specific surface area enhanced the photocatalytic activity, so that NO gas could be degraded effectively.

As for the research by Zhang et al<sup>[16]</sup>, TiO<sub>2</sub> and [Sr(OH)<sub>2</sub>·8H<sub>2</sub>O] were acted as starting material, NaOH solution as medium while oleic acid as additive. The starting materials would be put into the NaOH solution to stir until the mixture became homogeneous. Then, the oleic acid should be added into the mixture and mixed. After that, the mixture would be hydrothermal heated at 200°C for 20-40hour in the tank reactor. Finally, SrTiO<sub>3</sub> particles should be washed and dried. It was shown that the addition of oleic acid could improve the agglomeration and thus adjusted the morphology of the product. When the concentration of oleic acid increased, the number of agglomeration would decrease, and the size distribution and morphology of SrTiO<sub>3</sub> were more uniform. In addition, if the reaction time was reduced or the molar ratio of Sr/Ti decreased, the specific surface area of SrTiO<sub>3</sub> increased and the photocatalytic performance enhanced<sup>[17]</sup>.

In terms of Xu et al research<sup>[18]</sup>, Sr(NO<sub>3</sub>)<sub>2</sub> and TiCl<sub>4</sub> could be the starting materials, KOH as medium, and DBS as surfactant. The starting materials were mixed and then KOH and DBS was added into the mixture respectively. Until they stirred well, it would be put into tank reactor for a hydrothermal reaction, heated at 200°C for 8 hour. Finally, the DBS-coated SrTiO<sub>3</sub> was created. The research showed that the shape of this SrTiO<sub>3</sub> was relatively uniform, the particle size distribution of the powder was narrow, and there were fewer agglomerates. And also, the addition of DBS could change the polarity of SrTiO<sub>3</sub>, from a pure SrTiO<sub>3</sub> polar powder to a non-polar material after wrapping.

Hydrothermal synthesis method was an effective method for the synthesis of SrTiO<sub>3</sub>, which could be carried out at lower temperatures, with high product quality and low production cost, but with a long reaction time<sup>[9,10,17]</sup>.

#### 4. Sol-Gel method

Sol-Gel method was a method of synthesis of metal inorganic compounds or organic compounds as starting materials at low temperature<sup>[2]</sup>. The starting materials were went through three stages, solution, sol and gel. The starting materials were dissolved in water or solvent to form a solution. The solution underwent hydrolysis to form a sol, then, dried it to form a gel, and then heat treated to obtain the product. The advantage was that very pure composites could be produced<sup>[10,19]</sup>.

According to Zhang et al research<sup>[20]</sup>, Sr(NO<sub>3</sub>)<sub>2</sub> and Ti(C<sub>4</sub>H<sub>9</sub>)<sub>4</sub> were been used as starting materials. Firstly ,ethanol absolute was added into Ti(C<sub>4</sub>H<sub>9</sub>)<sub>4</sub> solution, and then added into Sr(NO<sub>3</sub>)<sub>2</sub> solution to mix with CH<sub>3</sub>COOH and stirred well. Meanwhile, glycerin was added into it and continue stirred the mixture until the formation of a sol. The sol was dried at 60 °C to form a gel. Finally, the gel was calcined to obtain SrTiO<sub>3</sub>. According to the experimental results, the medium, solvent, pH and other factors would had an effect on SrTiO<sub>3</sub>. When the temperature increased, the time of gel formation would be shortened, and hydrolysis or polymerization would occur, thus affecting the quality of the gel.

Regarding to Jiang et al research<sup>[21]</sup>, SrTiO<sub>3</sub> was synthesized by starting material of SrCO<sub>3</sub>, C<sub>4</sub>H<sub>6</sub>O<sub>6</sub> and C<sub>16</sub>H<sub>36</sub>O<sub>4</sub>Ti and used the product for the degradation of methylene blue. At first, SrCO<sub>3</sub> and C<sub>4</sub>H<sub>6</sub>O<sub>6</sub> was stirred well at 70 °C, and then the appropriate amount of C<sub>16</sub>H<sub>36</sub>O<sub>4</sub>Ti was added and stirred until formed a sol. Finally, the sol was calcined at 80 °C for 10 h to gel, and the gel was dried to form SrTiO<sub>3</sub>. It was shown that the optimum conditions for the synthesis of SrTiO<sub>3</sub> using the sol-gel method were a ratio of SrCO<sub>3</sub> and C<sub>4</sub>H<sub>6</sub>O<sub>6</sub> of 1:4, and the calcination temperature should 700°C for 2 h. The removal of 60.7% of the methylene blue could be achieved by irradiation with a 250 W mercury lamp for about 10 h.

Although the reaction conditions were mild and the product purity was high, the starting material was organic material with high cost, the synthesis period was long, and the product was easy to be agglomerated and the yield was small. The use of a large amount of organic material in the synthesis process of sol-gel method might lead to the increase of cost and environmental pollution problems [10,22].

## 5. Chemical coprecipitation method

Chemical coprecipitation method usually involved the addition of coprecipitant or adjusting the pH value of two or more aqueous solutions containing metal ions to produce precipitation of the substances in the solution, and then filtration, washing, drying, and calcination to obtain the target product [2,10]. Chemical coprecipitation methods included oxalate coprecipitation, carbonate coprecipitation and homogeneous coprecipitation, etc. The advantages of these methods were that the products obtained were high purity and had uniform particle size distribution, but they required high reaction conditions and prone to environmental pollution [23].

As for Zhang et al research [23], they prepared SrTiO<sub>3</sub> from H<sub>2</sub>TiO<sub>3</sub>, H<sub>2</sub>O<sub>2</sub>, NH<sub>3</sub>·H<sub>2</sub>O, and Sr(NO<sub>3</sub>)<sub>2</sub>, and oleic acid. The H<sub>2</sub>TiO<sub>3</sub> was purified to reduce the impurities in the target material before used, and the purified H<sub>2</sub>TiO<sub>3</sub> was mixed with H<sub>2</sub>O<sub>2</sub>. Next, NH<sub>3</sub>·H<sub>2</sub>O was put in an ice bath at a certain molar ratio, forming NH<sub>4</sub>TiO<sub>3</sub>·H<sub>2</sub>O<sub>2</sub>. Then the dissolved Sr(NO<sub>3</sub>)<sub>2</sub> was dropped into the NH<sub>4</sub>TiO<sub>3</sub>·H<sub>2</sub>O<sub>2</sub> solution and precipitated, and then the precipitate was washed and dried at 105°C for 3 hours. Finally, SrTiO<sub>3</sub> powder with a particle size of about 60 nm was obtained by calcining at 400 °C, 600 °C and 800 °C for 1 h, respectively. The experimental results showed that the addition of oleic acid could lead to the reduction of agglomeration and uniform particle size distribution. When the starting material molar ratio n(H<sub>2</sub>TiO<sub>3</sub>) : n(H<sub>2</sub>O<sub>2</sub>) : n(NH<sub>3</sub>·H<sub>2</sub>O) is 1.0:6.5:2.5, the reaction rate would become faster and the cost was lower. And also when the calcination temperature was 600 °C better cubic phase SrTiO<sub>3</sub> could be prepared.

Fang et al [24] suggested that ultrafine SrTiO<sub>3</sub> spherical powders could also be prepared from H<sub>2</sub>TiO<sub>3</sub> and SrCl<sub>2</sub>. Firstly, the H<sub>2</sub>TiO<sub>3</sub> was purified, and then it was stirred and mixed to form a suspension. Then, (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> solution was added to stir. At the same time, SrCl<sub>2</sub> solution was added for the reaction to generate precipitation. Finally, the resulting precipitate was filtered, washed, dried, and calcined to obtain high-purity ultrafine SrTiO<sub>3</sub> spherical powder with an average particle size of 0.4 μm and uniform distribution. The experimental results showed that the best conditions for the reaction: (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub>/ SrCl<sub>2</sub> was 1.06-1.10; Sr/Ti was 1.01. The reaction temperature was 25°C for 1.5 h, and the calcination temperature was 950°C for 2.5 h. The chemical coprecipitation method had a low reaction temperature and short time, but it was easy to introduce impurities, required more treatment to treat it and the products were easy to agglomerate [10].

## 6. Ultrasonic Chemical method

Ultrasonic Chemical method was a technique that utilizing the chemical effects of ultrasound to facilitate chemical reactions that would normally require extreme preparation conditions such as high temperatures and pressures. This method was based on the "cavitation" effect that happened when ultrasound waves propagated through a liquid, where gas bubbles in the liquid phase formed, grew and collapsed dramatically in response to the sound waves [25]. Specifically, when bubble collapsed, the collapsed region generated higher temperatures and pressures, while generating excitation waves that broke the chemical bonds to facilitate the reaction. The advantages of this method in the preparation of nanomaterials were controllable particle size, simple equipment, low cost, and most importantly, the process does not require the use of toxic solvents, making it an environmentally friendly [26].

In 2003 Yu et al research [27], 0.03 mol of Sr(OH)<sub>2</sub>·8H<sub>2</sub>O and Ti<sub>4</sub>(OCH<sub>3</sub>)<sub>16</sub> were acted as raw materials. These raw materials were dissolved in ethanol to maintain the molar ratio of Sr/Ti, and an appropriate amount of deionized water was added to promote hydrolysis. All mixtures were put into a 47 kHz

ultrasonic water bath for 2 h at 50 °C, and finally washed and dried to obtain pure cubic phase SrTiO<sub>3</sub> powders with particle sizes of 6.0-28 nm. This method explored that the cavitation effect for the rapid synthesized of SrTiO<sub>3</sub> at atmospheric pressure and low temperature. The experimental results showed that the average particle size of the crystals could be adjusted by controlling the volume ratio of water and ethanol. Moreover, the materials synthesized using ultrasound had pure cubic phase and nano porosity, which is advantageous for photocatalytic applications. It was because nano porosity could be used for surface area and active sites for reactions. In addition, this method was able to synthesize materials at a temperature of 50 °C, which was more energy efficient than conventional methods.

According to Xu et al research<sup>[28]</sup>, SrCl<sub>2</sub>·6H<sub>2</sub>O and TiCl<sub>4</sub> were raw materials. The raw materials were placed in 100 mL of deionized water at a certain stoichiometric ratio, and then an equal volume ratio of NaOH solution was quickly added and stirred to ensure that the reaction system was strongly basic. The mixed solution was subjected to ultrasonication (20 kHz) at room temperature and the reaction was terminated when the temperature of the solution rose to 80 °C. Finally, the material was washed and dried to obtain SrTiO<sub>3</sub> powder with particle size of 86-500 nm. The results showed that the size morphology of the product could be controlled by adjusting the reactant concentration, ultrasonic power, and ultrasonic time. For example, increasing the concentration of reactants could reduce the particle size from 300-400 nm to 70-90 nm. Meanwhile, it could also affect the morphology of the product, resulting in a variety of morphologies such as star, sphere, and cube. In addition, it took a long time to obtain uniform spherical SrTiO<sub>3</sub> particles when the ultrasonic wave was lower than 400W, but when the ultrasonic wave was higher than 1000W, it took only 10 minutes to obtain uniform spherical SrTiO<sub>3</sub> particles.

Ultrasonic chemical method played an important role in modern chemical research and industrial applications. By precisely controlling the ultrasonic parameters such as frequency, power and irradiation time, the reaction conditions could be further optimized to improve the reaction efficiency and selectivity. The main features of this method included high efficiency, environmental friendliness, and wide range of use<sup>[22,29]</sup>, but it was difficult to use for industrial production.

## 7. Template method

The template method was used to synthesise nano structure materials by using templates to control the internal structure of the material, including the shape and size of the pores and the properties of the pore walls. This method was suitable for preparing materials with specific pore structures. The template method involved dispersing a template agent in a solution of a precursor and facilitating the conversion of the precursor into the target material by chemical or physical process. At the final stage of the synthesis process, the template was removed by methods such as combustion or dissolution. The advantages of this method were the pore structure could be controlled, the product was homogeneous with high purity<sup>[30,31]</sup>.

As for Wang et al research<sup>[32]</sup>, porous SrTiO<sub>3</sub> were prepared by sol-gel method using PEG4000 as templating agent, while C<sub>16</sub>H<sub>36</sub>O<sub>4</sub>Ti and Sr(NO<sub>3</sub>) were the starting materials. In the experiment, 10 mL of CH<sub>3</sub>COOH was dissolved in deionised water, and then different amounts of PEG4000 were added. Additionally, Sr(NO<sub>3</sub>) was added and stirred to make a mixed solution. Then another 1.7 mL of PEG4000 was dissolved in ethanol absolute and stirred to get a light yellow solution, which was dripped into the mixed solution obtained before. And also, ethylene glycol was added as a stabilizer and stirred rapidly. As the solvent evaporated to form a sol, the sol was aged for 24 h and then dried to form a gel. Finally, the gel was calcined to form a porous SrTiO<sub>3</sub>. It was shown that the addition of template agent could increase the specific surface area of SrTiO<sub>3</sub>, and the grain size would decrease with increasing adding templating agent. The photocatalytic properties of SrTiO<sub>3</sub> is the strongest when the addition of template agent is 1.5 g.

The suitable amount of template agent and calcination temperature were important for product synthesis. The amount of template agent could affect the number and size of pores and thus the specific surface area of the material. However, the calcination temperature would affect the purity of

the material. This method could control the number of pores and pore volume fraction of materials, but its long aging time would extend the experimental period, and it also required high chemical reaction, because the sol-gel method was more sensitive to temperature and pH<sup>[33]</sup>.

## 8. Conclusion

(1) The products prepared by the solid-phase method were uneven in particle size, difficult to control the morphology and purity, resulting in poor photocatalytic performance of the synthesised products. Meanwhile, there were problems such as high energy consumption and difficult to be applied on a large scale, so the exploration for it was less still.

(2) Hydrothermal synthesis method was an effective method to synthesize SrTiO<sub>3</sub>, which could be carried out under lower temperature conditions, high product quality and low production cost, but long reaction time.

(3) Although the sol-gel method had mild reaction conditions and high product purity, the preparation period was long, the product was easy to agglomerate and the yield was small. The use of a large number of organic substances in the preparation process, leading to high costs and environmental pollution.

(4) The chemical coprecipitation method had low reaction temperature and short time, but it was easy to introduce impurities, requires complicated post-treatment and the product particles were easy to agglomerate.

(5) The main features of the ultrasonic chemical method included high efficiency, environmental friendliness and wide range of use, but it was difficult to use the ultrasonic chemical method for industrial production.

(6) The template agent method could effectively control the internal structure of the material and was suitable for the preparation of SrTiO<sub>3</sub> with specific microscopic pore structure. The prepared products were homogeneous, with high purity and large specific surface area, which could effectively improve the photocatalytic activity. However, the method will be still explored.

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