Advanced Nanomaterial Synthesis Procedures

Experiment 1: Titanium Dioxide Nanoparticle Synthesis

Procedure

Titanium dioxide nanoparticles were synthesized using a sol-gel method. First, 10 mL of titanium isopropoxide was dissolved in 50 mL of ethanol at room temperature. The solution was stirred continuously for 30 minutes using a magnetic stirrer at 300 rpm. Then, 2 mL of acetic acid was added dropwise as a stabilizing agent to prevent rapid hydrolysis. The mixture was heated to 80°C and maintained at this temperature for 3 hours under constant stirring. After cooling to room temperature, the precipitate was collected by centrifugation at 5000 rpm for 15 minutes. The product was washed three times with deionized water and ethanol alternately. Finally, the material was dried in an oven at 100°C for 12 hours and calcined at 450°C for 4 hours in air to obtain crystalline anatase phase titanium dioxide nanoparticles.

Results

The synthesized titanium dioxide nanoparticles exhibited an average particle size of 25 nm as determined by transmission electron microscopy. X-ray diffraction analysis confirmed the anatase crystal structure with characteristic peaks at 25.3°, 37.8°, and 48.0°.

Experiment 2: Gold Nanoparticle Synthesis via Turkevich Method

Synthesis Protocol

Gold nanoparticles were prepared by the classical Turkevich method. A solution of 100 mL containing 0.01% gold(III) chloride trihydrate was brought to a vigorous boil using a heating mantle. Once boiling commenced, 10 mL of 1% trisodium citrate solution was added rapidly while maintaining vigorous stirring. The color of the solution changed from pale yellow to deep purple and finally to ruby red within 5 minutes, indicating nanoparticle formation. The heating was continued for 15 minutes to ensure complete reduction. The solution was then cooled naturally to room temperature while stirring was maintained at 200 rpm for 1 hour.

Characterization

UV-visible spectroscopy showed a characteristic surface plasmon resonance peak at 520 nm. Dynamic light scattering measurements indicated a hydrodynamic diameter of 15 nm with a narrow size distribution.

Experiment 3: Zinc Oxide Nanowire Array Fabrication

Growth Method

Zinc oxide nanowire arrays were grown on silicon substrates using a hydrothermal method. First, silicon wafers were cleaned with acetone, isopropanol, and deionized water sequentially. A seed layer was deposited by spin-coating a 5 mM zinc acetate solution in ethanol at 3000 rpm for 30 seconds. The substrates were then heated at 350°C for 20 minutes to decompose the zinc acetate. For nanowire growth, a solution containing 25 mM zinc nitrate hexahydrate and 25 mM hexamethylenetetramine in 200 mL of deionized water was prepared. The seeded substrates were placed face-down in the growth solution and heated at 90°C for 6 hours in a sealed container. After growth, the samples were removed, rinsed thoroughly with deionized water, and dried at 60°C for 2 hours.

Analysis

Scanning electron microscopy revealed vertically aligned nanowires with an average diameter of 80 nm and length of 2 micrometers. The aspect ratio was approximately 25:1.

Experiment 4: Silver Nanoparticle Synthesis by Chemical Reduction

Experimental Details

Silver nanoparticles were synthesized through chemical reduction of silver nitrate. In a typical synthesis, 90 mL of deionized water was heated to 60°C in a round-bottom flask. Then, 5 mL of 1 mM silver nitrate solution was added under magnetic stirring at 400 rpm. After 2 minutes, 5 mL of freshly prepared 1% sodium borohydride solution was added dropwise over 3 minutes while maintaining the temperature at 60°C. The solution immediately turned yellow, indicating nanoparticle formation. Stirring was continued for 30 minutes. To stabilize the nanoparticles, 1 mL of 1% polyvinylpyrrolidone solution was added and stirring continued for another 15 minutes. The final product was stored at 4°C in a dark container.

Properties

The synthesized silver nanoparticles showed excellent stability for over 6 months. Transmission electron microscopy indicated spherical particles with an average size

of 10 nm. Energy-dispersive X-ray spectroscopy confirmed the presence of metallic silver.

Experiment 5: Graphene Oxide Synthesis via Modified Hummers Method

Preparation

Graphene oxide was synthesized using a modified Hummers method. Initially, 2 grams of graphite powder was mixed with 1 gram of sodium nitrate in a 500 mL flask. The flask was cooled in an ice bath to 0°C, and 46 mL of concentrated sulfuric acid was added slowly while stirring. After 30 minutes, 6 grams of potassium permanganate was added gradually over 1 hour, keeping the temperature below 5°C. The ice bath was then removed, and the mixture was heated to 35°C for 2 hours, forming a thick paste. Next, 92 mL of deionized water was added slowly, causing a violent exothermic reaction and raising the temperature to 98°C. The mixture was maintained at this temperature for 15 minutes. Then, 280 mL of warm deionized water was added, followed by 20 mL of 30% hydrogen peroxide solution, which turned the mixture bright yellow. The product was washed by repeated centrifugation at 4000 rpm with deionized water and 5% hydrochloric acid until the pH reached 6. Finally, the graphene oxide was dried under vacuum at 50°C for 24 hours.

Confirmation

Fourier-transform infrared spectroscopy confirmed the presence of oxygen-containing functional groups including hydroxyl, epoxide, and carboxyl groups. Raman spectroscopy showed characteristic D and G bands at 1350 and 1590 cm^{-1} respectively.

Experiment 6: Copper Oxide Nanorod Synthesis

Synthesis Procedure

Copper oxide nanorods were prepared through a precipitation method followed by thermal treatment. A solution of 0.1 M copper(II) sulfate pentahydrate in 100 mL deionized water was prepared. Separately, a 0.5 M sodium hydroxide solution was prepared in 50 mL deionized water. The sodium hydroxide solution was added dropwise to the copper sulfate solution under vigorous stirring at 400 rpm over 20 minutes. A blue precipitate of copper hydroxide formed immediately. The mixture was aged at room temperature for 1 hour with continuous stirring. The precipitate was collected by filtration and washed with deionized water until the filtrate pH was neutral. The washed precipitate was dried at 80°C for 6 hours. The dried powder was then calcined in a tube furnace at 400°C for 3 hours under air atmosphere with a heating rate of 5°C per minute.

Morphology

X-ray diffraction patterns confirmed the monoclinic CuO phase. Scanning electron microscopy showed rod-like structures with diameters ranging from 50 to 100 nm and lengths of 500 nm to 1 micrometer.

Experiment 7: Silica Nanoparticle Synthesis by Stöber Method

Synthesis

Monodisperse silica nanoparticles were synthesized using the Stöber method. In a typical procedure, 40 mL of absolute ethanol, 8 mL of deionized water, and 3 mL of ammonia solution (28-30%) were mixed in a round-bottom flask at 25°C under magnetic stirring at 250 rpm. After 10 minutes of equilibration, 2 mL of tetraethyl orthosilicate was added dropwise over 5 minutes. The reaction mixture became turbid immediately, indicating nanoparticle formation. Stirring was continued for 6 hours at room temperature. The silica nanoparticles were collected by centrifugation at 10000 rpm for 20 minutes, washed three times with ethanol, and dried at 60°C overnight.

Size Control

The particle size could be controlled between 50 and 500 nm by adjusting the ammonia concentration and water-to-ethanol ratio. At the conditions used, the average particle size was 200 nm with a coefficient of variation less than 5%.

Experiment 8: Iron Oxide Magnetic Nanoparticle Synthesis

Co-precipitation Method

Magnetic iron oxide nanoparticles were synthesized via co-precipitation. Solutions of 0.1 M iron(II) chloride tetrahydrate and 0.2 M iron(III) chloride hexahydrate were prepared in 50 mL of deoxygenated water under nitrogen atmosphere. The solutions were mixed in a three-neck flask equipped with a mechanical stirrer. The mixture was heated to 80°C, and then 10 mL of 5 M sodium hydroxide solution was added rapidly while stirring vigorously at 600 rpm. A black precipitate formed immediately. The reaction was maintained at 80°C for 30 minutes under nitrogen flow. The nanoparticles were separated magnetically using a neodymium magnet, washed five times with deionized water and twice with ethanol. The product was dried under vacuum at 70°C for 12 hours.

Magnetic Properties

Vibrating sample magnetometry showed superparamagnetic behavior at room temperature with a saturation magnetization of 65 emu/g. The blocking temperature was determined to be approximately 150 K.

Experiment 9: Cadmium Sulfide Quantum Dots

Synthesis in Aqueous Phase

Cadmium sulfide quantum dots were synthesized in aqueous solution using mercaptopropionic acid as a stabilizer. First, 0.5 mmol of cadmium chloride was dissolved in 50 mL of deionized water. Then, 1.2 mmol of mercaptopropionic acid was added, and the pH was adjusted to 11 using 1 M sodium hydroxide solution. The solution was degassed by bubbling nitrogen for 30 minutes. In a separate flask, 0.5 mmol of sodium sulfide was dissolved in 10 mL of deoxygenated water. This solution was injected rapidly into the cadmium solution while stirring at 300 rpm. The mixture immediately turned yellow. The reaction was continued at room temperature for 2 hours. The quantum dots were precipitated by adding ethanol, collected by centrifugation at 8000 rpm for 10 minutes, and redispersed in water.

Optical Properties

Photoluminescence spectroscopy showed emission at 520 nm under 400 nm excitation. The quantum yield was estimated to be 15%. The absorption onset was at 480 nm, corresponding to a particle size of approximately 3 nm.

Experiment 10: Zinc Sulfide Nanoparticles via Solvothermal Method

Solvothermal Synthesis

Zinc sulfide nanoparticles were prepared using a solvothermal method. In a typical synthesis, 1 mmol of zinc acetate dihydrate and 2 mmol of thiourea were dissolved in 40 mL of ethylene glycol. The solution was transferred to a 100 mL Teflon-lined stainless steel autoclave. The autoclave was sealed and heated at 180°C for 12 hours. After cooling naturally to room temperature, the white precipitate was collected by centrifugation at 6000 rpm for 15 minutes, washed alternately with ethanol and water three times each, and dried at 80°C for 8 hours under vacuum.

Characterization Results

X-ray diffraction showed a cubic zinc blende structure. Photoluminescence spectra exhibited a strong blue emission at 440 nm. Transmission electron microscopy revealed nearly spherical nanoparticles with an average diameter of 5 nm.

Safety Considerations

All experiments were conducted in well-ventilated fume hoods. Appropriate personal protective equipment including lab coats, safety goggles, and nitrile gloves were worn at all times. Concentrated acids and bases were handled with extreme caution. Chemical waste was disposed of according to institutional guidelines.

Conclusion

These synthesis procedures demonstrate various approaches to nanomaterial fabrication including sol-gel, hydrothermal, solvothermal, chemical reduction, and co-precipitation methods. Each method offers distinct advantages in terms of particle size control, morphology, and scalability.