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Editorial

Hydrothermal Synthesis of Nanomaterials

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Hydrothermal synthesis is one of the most commonly used methods for preparation of nanomaterials. It is basically a solution reaction-based approach. In hydrothermal synthesis, the formation of nanomaterials can happen in a wide temperature range from room temperature to very high temperatures. To control the morphology of the materials to be prepared, either low-pressure or high-pressure conditions can be used depending on the vapor pressure of the main composition in the reaction. Many types of nanomaterials have been successfully synthesized by the use of this approach. There are significant advantages of hydrothermal synthesis method over others. Hydrothermal synthesis can generate nanomaterials which are not stable at elevated temperatures. Nanomaterials with high vapor pressures can be produced by the hydrothermal method with minimum loss of materials. The compositions of nanomaterials to be synthesized can be well controlled in hydrothermal synthesis through liquid phase or multiphase chemical reactions. This special issue serves as a forum presenting the recent research results of hydrothermal synthesis of nanomaterials. Several papers on hydrothermal synthesis of nanoparticles, nanorods, nanotubes, hollow nanospheres, and graphene nanosheets have been published in this special issue. New synthesis methods, for example, microwave-assisted hydrothermal synthesis and template-free self-assembling catalytic synthesis, are reported in this special issue. Research work on optimization of the synthesis conditions is included as well. Nanomaterials for applications such as energy harvesting and biosensing are also studied in the papers published in this special issue. In addition, hydrothermal synthesis using waste materials to achieve environment protection was studied in one of the papers. A brief summary of all the eleven accepted papers is presented as follows.

The paper by Z. Rák and D. W. Brenner presents the fundamental work on the formation of nickel ferrite ($NiFe_2O_4$) nanoparticles under hydrothermal conditions. A model was established via a method that combines results of first-principle calculations, elements of aqueous thermochemistry, and experimental free energies of formation. Based on calculations using the model, negative formation energies for the (111) surfaces and positive free energies for the formation of bulk nickel ferrite were predicted. The combination of the negative surface and positive bulk energies yields thermodynamically stable nickel ferrite nanoparticles with sizes between 30 and 150 nm in the temperature range of 300 to 400 K under alkaline conditions. The effect of processing condition on the stability of the nickel ferrite nanoparticle was discussed.

The work by M. L. M. Napi et al. deals with hydrothermal synthesis condition parameter optimization. Design

of Experiment (DOE) was used to determine the processing parameters for hydrothermal growth of one-dimensional fluorine-doped zinc oxide (1D-FZO) using Au nanoparticles as the catalyst. The DOE includes three design points on each of the parameter. The selected parameters are the gold sputtering time (10 s, 15 s, and 20 s), hydrothermal reaction time (3 hours, 6.5 hours, and 10 hours), and hydrothermal temperature (50°C, 75°C, and 100°C). The effects of these parameters on the quality of 1D-FZO produced are analyzed statistically. It is found that the sputtering time of the Au nanoparticles has significant effect on the morphology and electrical property of the 1D-FZO. The lowest resistance value of 22.57 Ω was achieved for the 1D-FZO grown with the longest Au sputtering time. The hydrothermal growth temperature below 100°C was suggested.

The work performed by T. H. P. Nguyen et al. investigated the stability of electrochemical and biosensing properties of ZnO nanorod-based platinum screen-printed electrodes (SPEs) applied for detection of bacterial pathogens. The ZnO nanorods (NRs) were grown on the platinum working electrode using the hydrothermal method. The standard photolithography and lift-off process were used to fabricate the patterned platinum SPEs on a silicon wafer. For sensor property characterization, Salmonella polyclonal antibodies were immobilized at the ZnO NR surface through crosslinking. Morphological and structural analysis of the ZnO NRs by scanning electron microscopy (SEM), transmission electron microscopy (TEM), and X-ray diffraction (XRD) shows that the ZnO NRs were grown vertically on platinum electrodes with a diameter around 20-200 nm and a length of 5-7 μm. These modified electrodes were applied for detecting Salmonella enteritidis at a concentration of 103 cfu/mL. Electrochemical measurements including cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) reveal that the ZnO NR-modified platinum electrodes could detect Salmonella bacteria well with signal to noise ratio much higher than 3 : 1. This indicates that the ZnO NR-modified platinum SPEs have potential for the development of biochips for electrochemical detection of bacterial pathogens.

The paper by X. Wu et al. reported the findings on synthesis of graphene sheets with high quality by calcinating composites intercalated with montmorillonite (MMT) and asphaltene. The graphene sample obtained is continuous and as long as 10.79 μm. The surface is smooth with little defects. By analyzing the XRD patterns of graphene/MMT composites at different conditions, the graphene formation mechanism is proposed and the optimal calcination conditions are suggested. This work provides important insights into how to make low-cost graphene using asphaltene extracted from heavy crude oil.

In the paper by J. Zhu et al., carbon quantum dots (CQDs) with high quantum yield and good stability were synthesized from waste tea leaves and peanut shells by a one-step hydrothermal method. This work introduced the concept of waste utilization and environmental protection while making useful nanomaterials from sustainable sources. The synthesis conditions, structures, and optical properties of CQDs were researched. Their unique characteristics of emitting strong and steady blue fluorescence under excitation of ultraviolet light were found due to the existence of plenty of hydrophilic groups at the surface of the CQDs. It is concluded that the CQDs have potentials for analytical detection and for application as biomarkers.

The paper by X. Yan et al. shows that the continuous-flow hydrothermal processes have advantages for synthesizing VO_2 particles over the traditional batch reaction systems. Specifically, the role of mixers in continuous-flow synthesis of thermochromic VO_2 particles via rapid one-step hydrothermal reaction was studied. In this work, a Center T-Mixer and a Collision Cross-Mixer are developed and implemented in a hot water fluidized suspension reaction (HWFSR) system. The influence of the resident time on the particle phase and size was examined, and properties of particles derived from systems equipped with differing mixers were compared. The resulting particles were characterized using techniques of X-ray powder diffraction (XRD) analysis, scanning electron microscopy (SEM), and differential scanning calorimetry (DSC). The results confirm the crucial role of mixers in particle fabrication in continuous-flow systems. When compared with the Center T-Mixer, the Collision Cross-Mixer has better control regarding the morphology and size distribution of resulting particles while improving the transition temperatures of the as-synthesized materials. HWFSR systems containing novel mixer designs are capable of producing pure M-phase VO_2 particles in a single step contrary to the current reactor design that uses a second post heat treatment step, and they are capable of synthesizing many other nanoparticle species, especially those requiring high temperature and pressure reaction conditions. The synthesized VO_2 particles are promising materials for thermochromic smart windows that reduce building energy loss.

The paper by P. T. Lien et al. reported synthesis of $GdPO_4{\cdot}nH_2O$ and Tb^{3+}-doped $GdPO_4{\cdot}nH_2O$ nanorods@silica-NH_2 conjugated with IgG antibody by hydrothermal, sol-gel, and coprecipitation methods. The effect of Tb^{3+}/Gd^{3+} molar ratio on the size, morphology, and luminescence of the synthesized samples was investigated. It is found that under optimized conditions, the $GdPO_4{\cdot}nH_2O{:}Tb^{3+}$ as uniform nanorods sizing from 10 to 30 nm in diameter and from 200 to 300 nm in length shows the strongest luminescence in green color with narrow bands under the UV excitation (325 nm). After being coated with silica-NH_2 and conjugated with IgG antibody, all luminescence characteristic peaks of $GdPO_4{\cdot}nH_2O{:}Tb^{3+}$ corresponding to the process of energy transfer from Gd^{3+} to Tb^{3+} and then the emission from $^5D_4 \rightarrow {}^7F_J$ ($J = 6, 5, 4, 3$) of Tb^{3+} can still be observed. The application of the Tb^{3+}-doped $GdPO_4{\cdot}nH_2O$ nanorods@silica-NH_2 conjugated with IgG antibody for rapid selective detection of Naja atra cobra venom was also shown.

L. Ma et al. published their work on hydrothermal synthesis of various Co-doped $Zn_{1-x}Co_xMn_2O$ nanocrystals with a spinel structure. The nanocrystals form hollow nanospheres. The influence of Co doping concentration on the structure, morphology, elemental composition, and optical and photocatalytic properties of the samples was studied. It is found that Co^{2+} ions replaced some of the lattice sites of

Zn^{2+} in the $ZnMn_2O_4$ nanocrystals. The crystalline size decreased with the increase of Co doping. The band gap of $Zn_{1-x}Co_xMn_2O$ is smaller than that of pure $ZnMn_2O_4$ and red shifted was found for the doped sample. It is also found that the photocatalytic activity of the Co-doped sample is higher than that of the undoped sample during the photodegradation of methyl orange (MO) under visible light irradiation. The results indicate that Co-doped spinel $ZnMn_2O_4$ nanocrystals are effective in photocatalytic degradation of the pollutants.

Y. Wang and Y. Li reported their research on hydrothermal synthesizing titanium dioxide hollow nanospheres for photoluminescence and catalysis. As known, there exists challenge in preparing high-purity brookite TiO_2 with some unique structure. In this work, high-purity brookite TiO_2 hollow spheres were hydrothermally synthesized by employing titanium sulfate as the titanium source and chloroacetic acid and sodium hydroxide as the pH regulator. The structure, morphology, and optical properties were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS), and UV-Vis diffuse reflectance spectroscopy (UV-Vis DRS). The results showed that the as-prepared brookite TiO_2 hollow spheres in a size of about 1.0 micrometer had a direct band gap of 3.13 eV. Thermal analysis in combination with infrared spectroscopy showed that the as-prepared brookite TiO_2 was surface capped with water and organic molecules. The photocatalytic and photoluminescence properties of brookite TiO_2 were shown.

The paper by S. B. S. Gusmão et al. focuses on the microwave-assisted hydrothermal reaction method. One-pot synthesis of titanate nanotubes decorated with anatase nanoparticles was demonstrated for the first time. This nanoheterostructure of titanate nanotubes decorated with anatase nanoparticles (TiNT@AnNP) was characterized by various methods including X-ray diffraction, Raman spectroscopy, scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDXS), high-resolution transmission electron microscopy (HRTEM), selected-area electron diffraction, and X-ray photoelectron spectroscopy (XPS). The results showed that the TiNT@AnNP nanomaterial is highly crystalline and in nanometer size. The synthesized TiNT@AnNP degraded an anionic dye (Remazol blue) more efficiently under UV-visible light (380–780 nm) than a commercial anatase-TiO_2 precursor. This increased efficiency of photodegradation is due to the large surface area and the effective separation of the photon induced electron-hole pairs. The benefit of microwave-assisted hydrothermal synthesis in the production of TiNT@AnNP for environmental applications was discussed as well.

In the paper by M. C. Uribe López et al., synthesis of ZnO-ZrO_2 nanocomposites was introduced. Characterization of the nanomaterials for photocatalytic degradation and mineralization of phenol was also presented. Zinc (II) acetylacetonate was used as the source materials for generating different ZnO contents (13, 25, 50, and 75% mol) in the nanocomposites. The synthesized ZnO-ZrO_2 nanomaterials showed both the tetragonal crystalline structure of zirconia and the hexagonal one of ZnO. The morphology was observed, and the size of the nanomaterials was analyzed by electron microscopy. The formation of ZnO nanorods with the size ranging from 50 nm to 300 nm and zirconia particles with the size smaller than 50 nm was revealed. The advantage of using the nanocomposites for photocatalytic degradation of phenol was demonstrated. The mineralization degree of the 75ZnO-25ZrO_2 nanocomposite has higher mineralization degree than pure ZnO. The nanocomposite can also inhibit the generation of undesirable intermediates.

Conflicts of Interest

The editors declare that they have no conflicts of interest regarding the publication of this special issue.

Acknowledgments

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