Sonochemistry

Theory, Reactions, Syntheses, and Applications

Chemical Engineering Methods and Technology

Filip M. Nowak
Editor

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CHEMICAL ENGINEERING METHODS AND TECHNOLOGY

SONOCHEMISTRY: THEORY, REACTIONS, SYNTHESES, AND APPLICATIONS

FILIP M. NOWAK
EDITOR

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PREFACE

The study of sonochemistry is concerned with understanding the effect of sonic waves and wave properties on chemical systems. This book reviews research data in the study of sonochemistry including the application of sonochemistry for the synthesis of various nano-structured materials, ultrasound irradiation in pinacol coupling of carbonyl compounds, ultrasound and hydrophobic interactions in solutions, as well as the use of ultrasound to enhance anticancer agents in sonochemotherapy and the ultrasound-enhanced synthesis and chemical modification of fullerenes.

Chapter 1 - Recently, sonochemistry has been employed extensively in the synthesis of nano-structured materials. Rapid reaction rate, controllable reaction conditions, simplicity and safety of the technique as well as the uniform shape, narrow size distribution, and high purity of prepared nano-sized materials are some of the main advantage of sonochemistry. Sonochemistry uses the ultrasonic irradiation to induce the formation of particles with smaller size and high surface area.

Because of its importance, sonochemistry has experienced a large promotion in various fields concerned with production of new nano-structured materials and improvement of their properties during the recent years. However, it has encountered limitations in the case of production of some nano-materials with specific morphology, size and properties, but the growth of the number of researches and published articles in the field of sonochemistry during the recent years shows a large interest and attempt to apply sonochemistry in nanotechnology. The improvement of shape, size, purity and some other chemical and physical properties of such produced materials has been the scope of the researchers recently.

Sonochemistry uses the powerful ultrasound irradiation (20 kHz to 10 MHz) to induce chemical reaction of molecules. During the ultrasonic irradiation, the acoustic cavitations will occur which consist of the formation, growth and implosive collapse of bubbles in a liquid. The implosive collapse of the bubbles generates a localized hotspot or shock wave formation within the gas phase of the collapsing bubbles (The hot-spot theory).

This chapter is planned to deal with the application of sonochemistry for the synthesis of various nano-structured materials such as metals, metal carbides, metal oxides, chalcogenides and nanocomposites with unique properties. The effect of different ultrasonic parameters on the prepared structures including their size, morphology and properties are investigated. Also, some applications of prepared nano-materials are introduced, e.g. electrochemical energy storage, catalysis, biosensor and electrooxidation.

Chapter 2 - A multitude of useful physical and chemical processes promoted by ultrasonic cavitation have been described in laboratory studies. Industrial-scale

implementation of high-intensity ultrasound has, however, been hindered by several technological limitations, making it difficult to directly scale up ultrasonic systems in order to transfer the results of the laboratory studies to the plant floor. High-capacity flow-through ultrasonic reactor systems required for commercial-scale processing of liquids can only be properly designed if all energy parameters of the cavitation region are correctly evaluated. Conditions which must be fulfilled to ensure effective and continuous operation of an ultrasonic reactor system are provided in this chapter, followed by a detailed description of "shockwave model of acoustic cavitation", which shows how ultrasonic energy is absorbed in the cavitation region, owing to the formation of a spherical micro-shock wave inside each vapor-gas bubble, and makes it possible to explain some newly discovered properties of acoustic cavitation that occur at extremely high intensities of ultrasound. After the theoretical background is laid out, fundamental practical aspects of industrial-scale ultrasonic equipment design are provided, specifically focusing on:

- electromechanical transducer selection principles;
- operation principles and calculation methodology of high-amplitude acoustic horns used for the generation of high-intensity acoustic cavitation in liquids;
- detailed theory of matching acoustic impedances of transducers and cavitating liquids in order to maximize the ultrasonic power transfer efficiency;
- calculation methodology of "barbell horns", which provide the impedance matching and can help achieving the transference of all available acoustic energy from transducers into the liquids. These horns are key to industrial implementation of high-power ultrasound because they permit producing extremely high ultrasonic amplitudes, while the output horn diameters and the resulting liquid processing capacity remain very large;
- optimization of the reactor chamber geometry.

Chapter 3 - Carbon-carbon bond formation is one of the most important topics in organic synthesis. One of the most powerful methods for constructing a carbon-carbon bond is the reductive coupling of carbonyl compounds giving 1,2-diols. Of these methods, the pinacol coupling, which was described in 1859, is still a useful tool for the synthesis of vicinal diols. 1, 2-Diols obtained in the reaction were very useful synthons for a variety of organic synthesis, and were also used as intermediates for the construction of biologically important natural product skeletons and asymmetric ligands for catalytic asymmetric reaction. In particular, pinacol coupling has been employed as a key step in the construction of HIV-protease inhibitors.

Generally, the reaction is effected by treatment of carbonyl compounds with an appropriate metal reagent and/or metal complex to give rise to the corresponding alcohols and coupled products, The coupling products can have two newly chiral centers formed. Threo, erythro mixtures of diols are usually obtained from reactions. As a consequence, efficient reaction conditions have been required to control the stereochemistry of the 1,2-diols. Recent efforts have focused on the development of new reagents and reaction systems to improve the reactivity of the reagents and diastereoselectivity of the products.

In some of the described methods, anhydrous conditions and long reaction time are required to get satisfactory yields of the reaction products, some of the used reductants are expensive or toxic; excess amounts of metal are needed. Sonication can cause metal in the form of a powder particle rupture, with a consequent decrease in particle size, expose new surface and increase the effective area available for reaction. It was effective in enhancing the reactivity

of metal and favorable for single electron transfer reaction of the aldehydes or ketones with metal to form diols. Some recent applications of ultrasound in pinacol coupling reactions are reviewed. The results are mostly from the author research group.

Chapter 4 - Sonochemistry and solution chemistry have been explicitly brought together by analyzing the effect of ultrasound on kinetics of ester hydrolysis and benzoin condensation, measured by the authors, and similar kinetic data for the solvolysis of tert-butyl chloride, compiled from literature. For the first time the power ultrasound, reaction kinetics and linear free-energy relationships were simultaneously exploited to study ionic reactions in water and aqueous-organic binary solvents and the importance of hydrophobic ground-state stabilization of reagents in aqueous solutions was discussed. This approach has opened novel perspectives for wider understanding of the effect of sonication on chemical reactions in solution, as well as on solvation phenomena in general.

Chapter 5 - Ultrasound generates cavitation, which is "the formation, growth, and implosive collapse of bubbles in a liquid. Cavitation collapse produces intense local heating (~5000 K), high pressures (~1000 atm), and enormous heating and cooling rates (>109 K/sec)" and liquid jet streams (~400 km/h), which can be used as a source of energy for a wide range of chemical processes. This review will concentrate on theory, reactions and synthetic applications of ultrasound in both homogeneous liquids and in liquid-solid systems. Some recent applications of ultrasound in organic synthesis, such as, Suzuki reaction, Sonogashira reaction, Biginelli reaction, Ullmann coupling reaction, Knoevenagel condensation, Claisen-Schmidt condensation, Reformatsky reaction, Bouveault reaction, Baylis-Hillman reaction, Michael addition, Curtius rearrangement, Diels-Alder reaction, Friedal-Craft acylation, Heck reaction, Mannich type reaction, Pechmann condensation and effect of ultrasound on phase transfer catalysis, oxidation-reduction reactions, ionic liquids and photochemistry are reviewed. Ultrasound found to provide an alternative to traditional techniques by means of enhancing the rate, yield and selectivity to the reactions.

Chapter 6 - Sonochemotherpy is the use of ultrasound to enhance anticancer agents. Preclinical trials have manifested this modality is effective against cancers including chemoresistant lesions. Sonochemotherapy is a target therapy, in which cavitation plays the leading role. Making the occurrence and level of cavitation under control improves the safety and therapeutic efficacy. Sonosensitizers and microbubbles enhance cavitation, being a measure to adjust the level of cavitation. Free radicals due to cavitation have the potentials of restructuring a molecule and changing the conformation; thus the molecular structure and anticancer potency of a cytotoxic agent must be investigated, especially when sonosensitizer and microbubble are employed. A potential clinical model for investigating sonochemotherapy is the residual cancer tissues when performing palliative high intensity focused ultrasound treatment.

Chapter 7 - Ultrasound (US) is a sound wave of a frequency greater than the superior audibility threshold of the human hearing. Sonochemistry is the application of ultrasound in chemistry. It became an exciting new field of research over the past decade. Some applications date back to the 1920s. The 1950s and 1960s subsequently represented the first extensive sonochemical research years and significant progresses were made throughout them. Then it was realized that ultrasound power has a great potential for uses in a wide variety of processes in the chemical and allied industries. In these early years, experiments were often performed without any real knowledge of the fundamental physical background about the US action. The situation changed in the 1980s when a new surge of activity started

and the use of US as a real tool in chemistry began. It was in 1986 that the first ever international symposium on Sonochemistry was held at Warwick University U.K.

Chapter 8 - In this chapter, the use of ultrasounds on fullerenes (C_{60} and C_{70}) and fullerene derivatives is described. The focus is on the articles reporting the ultrasound-promoted treatment of these nanoparticles written in English. The ultrasound-enhanced synthesis and chemical modification of fullerenes are detailed. The improvement obtained by sonicating the reaction mixtures while carrying out traditional organic reactions is discussed. This includes many types of reactions, such as oxidation, cycloaddition, reduction and amination. Also the ultrasound-enhanced crystallization of fullerenes, producing fullerites, and the formation of colloids when the fullerenes are sonicated in various solvent mixtures are detailed, providing the role of ultrasound in these processes.

Chapter 9 - In this chapter, the use of ultrasounds on carbon based nanotubes is reviewed with a focus on the English written articles. The synthesis of carbon nanotubes and their surface modification such as oxidation and covalent functionalization under ultrasounds are reported. The synthesis of hybrid nanocomposite materials where carbon nanotubes are added as a reinforcement agent via ultrasound-induced assembly is not described in this chapter. A detailed survey of the literature concerning the purification and separation of carbon nanotubes under ultrasounds is provided. The effect of sonication on carbon nanotubes suspensions which covers aqueous and organic solutions in the presence of surfactants is discussed with an emphasis being placed on the effect that ultrasounds have on non-covalent interactions between the carbon nanotubes and the components of the suspensions. The effect of ultrasounds on the physical properties of the carbon nanotubes, especially the introduction of wall defects is analyzed. Finally the advantages and shortcomings of sonochemistry described in this chapter are summarized, showing a possible trend in the direction of future research in this field.

CONTENTS

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Chapter 1

SONOCHEMISTRY: A SUITABLE METHOD FOR SYNTHESIS OF NANO-STRUCTURED MATERIALS

M. F. Mousavi[1] and S. Ghasemi
[1] Department of Chemistry, Tarbiat Modares University, Tehran, Iran
[2] Department of Chemistry, The University of Qom, Qom, Iran

ABSTRACT

Recently, sonochemistry has been employed extensively in the synthesis of nano-structured materials. Rapid reaction rate, controllable reaction conditions, simplicity and safety of the technique as well as the uniform shape, narrow size distribution, and high purity of prepared nano-sized materials are some of the main advantage of sonochemistry. Sonochemistry uses the ultrasonic irradiation to induce the formation of particles with smaller size and high surface area [1].

Because of its importance, sonochemistry has experienced a large promotion in various fields concerned with production of new nano-structured materials and improvement of their properties during the recent years. However, it has encountered limitations in the case of production of some nano-materials with specific morphology, size and properties, but the growth of the number of researches and published articles in the field of sonochemistry during the recent years shows a large interest and attempt to apply sonochemistry in nanotechnology. The improvement of shape, size, purity and some other chemical and physical properties of such produced materials has been the scope of the researchers recently [2].

Sonochemistry uses the powerful ultrasound irradiation (20 kHz to 10 MHz) to induce chemical reaction of molecules. During the ultrasonic irradiation, the acoustic cavitations will occur which consist of the formation, growth and implosive collapse of bubbles in a liquid. The implosive collapse of the bubbles generates a localized hotspot or

[1] Corresponding author. M.F. Mousavi, Department of Chemistry, Tarbiat Modares University, P.O. Box 14115-175, Tehran, Iran Tel.: +98 21 82883474/9; fax: +98 21 82883455. E-mail addresses: mousavim@modares.ac.ir, mfmousavi@yahoo.com (M.F. Mousavi).

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shock wave formation within the gas phase of the collapsing bubbles (The hot-spot theory) [3].

This chapter is planned to deal with the application of sonochemistry for the synthesis of various nano-structured materials such as metals, metal carbides, metal oxides, chalcogenides and nanocomposites with unique properties. The effect of different ultrasonic parameters on the prepared structures including their size, morphology and properties are investigated. Also, some applications of prepared nano-materials are introduced, e.g. electrochemical energy storage, catalysis, biosensor and electrooxidation.

1. INTRODUCTION

When ultrasound radiations interact with molecules, chemical reactions can be initiated. Sonochemistry is an interesting research area deal with the processes occurs during the application of powerful ultrasound (20 KHz–10 MHz). Sonochemistry arises from acoustic cavitations. Bubbles undergo the formation, growth, and implosive collapse in a liquid under ultrasonic irradiation. Bubble growth occurs through the diffusion of solute vapor into the bubble. A bubble can be included evaporated water molecules and dissolved gas molecules. When the bubble size reaches to a radius down to several μm, the bubbles collapse provides extreme conditions of transient high temperature(as high as 5000K) and high pressure (up to ~1800 atm) within the collapsing bubbles, shock wave generation, and radical formation. The collapsing bubbles provide reaction sites, named hot spots. At this sites, sonolysis of water molecules to hydrogen radicals (H•) and hydroxyl radicals (OH•) is occurred which is responsible to sonochemical reaction. Also, organic molecules in solution can form organic radicals with a reducing ability. The size of a bubble depends on ultrasonic frequency and intensity. Bubbles collapse occurs in very short time (nanosecond) and cooling rate of 1011 K/s is obtained. The fast kinetics of such process can hinders the growth of nuclei produced during the collapse of bubbles. This may be the reason of formation of nanostructured materials.

Sonochemical synthesis of different types of nanostructured materials consisted of metals and their oxides, alloy, semiconductors, carbon carbonic and polymeric materials and their nanocomposite have received much attention in recent years.

A number of factors can influence on cavitation efficiency and the properties of the products. The dissolved gas, ultrasonic power and frequency, temperature of the bulk solution, and type of solvent are all important factors that control the yield and properties of the synthesized materials.

In the field of sonochemistry, a number of book chapter and reviews have been published 4. Y. Mastai and A. Gedanken reviewed articles in the field of sonochemistry published before 2004 in a chapter of book entitled "Sonochemistry and Other Novel Methods Developed for the Synthesis of Nanoparticles" [2]. Also a review articles was published by Gedanken in 2004 entitled "Using sonochemistry for the fabrication of nanomaterials" focused on the typical shape of products obtained in sonochemistry [1]. Another review articles also published dealt with insertion of nanoparticles into mesoporous materials [5] and the sonochemical doping of various nanoparticles into ceramics and polymers [6].

In this chapter, we will present a literature survey on the various inorganic, organic/inorganic and inorganic/inorganic systems more recently have been synthesized by using ultrasonic method from January 2004 to January 2010s.

2. SYNTHESIS OF NANOMETALS

Intensive works on metal nanostructures such as noble metals (Au, Pt, Pd) with various size and morphology have been achieved due to their potential applications in the fabrication of electronic, optical, optoelectronic, and magnetic devices. They can be obtained form sonication of solution containing related metal ion in the absence and presence of capping agents. With controlling size, shape, and crystallinity of nanometals, it can be possible to tune the intrinsic properties of a metal nanostructure.

2.1. Gold

Gold and other noble metal nanoparticles have been extensively considered in recent years because of their potential applications in optics, electronics, and catalysis, etc. Okitsu et al reported the synthesis of Au nanoparticles and investigate the dependence of sonochemical reduction rate of Au(III) to Au nanoparticles in aqueous solutions containing 1-propanol as accelerator and their particle size to the ultrasound frequency so that the highest reduction rate was at 213 kHz in the range of 20 to 1062 kHz [7]. The average size of Au particles was 15.5 nm in 20 mM 1-propanol.

This group also synthesized Gold nanorods by using sonochemical reduction (frequency, 200 kHz; power, 200 W) of gold ions in aqueous solution (60 mL) containing of HAuCl4 and CTAB including 1.2 mL of AgNO3 (4.0 mM) and 240 μL of ascorbic acid (0.050 M) with pH 3.5 [8]. During the reaction, Au (III) is immediately reduced to Au (I) by reaction with the ascorbic acid. CTAB and AgNO3 act as effective capping agents for the shape controlled growth of gold seeds. The solution was purged with argon for 15 min and then sonicated in a water bath (at 27 °C) by a water circulation system. In the presence of ultrasonic, the following reactions are proposed:

$$H_2O \xrightarrow{)))} H^{\bullet} + OH^{\bullet} \quad (1)$$

$$CTAB + OH^{\bullet}(H^{\bullet}) \rightarrow H_2O(H_2) + \text{reducing species} \quad (2)$$

$$CTAB + H_2O \rightarrow \text{pyrolysis radicals and unstable products} \quad (3)$$

$$Au^{+} + M \rightarrow Au^{0} + H^{+} + M' \quad (4)$$

$$nAu^{0} \rightarrow (Au^{0})_n \quad (5)$$

$$Au^{0} + (Au^{0})_n \rightarrow (Au^{0})_{n+1} \quad (6)$$

Where M corresponds to various reducing species, pyrolysis radicals and unstable products. In reaction 3, pyrolysis radicals and unstable products are formed via pyrolysis of

CTAB and water. The size of the sonochemically formed gold nanorods was less than 50 nm, and their average aspect ratio decreased with increasing pH of the solution.

At pH 7.7, irregular shaped gold nanoparticles were formed. At pH 9.8, most of the particles formed had a spherical shape with a smaller particle size than those formed in the lower pH solutions. Based on the obtained results, it was clear that the size and shape of the sonochemically formed gold nanoparticles are dramatically dependent on the pH value of the solution (Figure 1).

From the obtained results, it was demonstrated that longer gold nanorods would be obtained if the synthesis was performed in solution with acidic pH.

Li et al. reported the synthesis of single-crystal Au nanoprisms with triangular or hexagonal shape, 30-40 nm planar dimensions, and 6-10 nm thickness from solution of HAuCl4 and PVP in ethylene glycol solution [9]. Ethylene glycol, the surfactant poly(vinylpyrrolidone), and ultrasonic irradiation play important roles in the formation of Au nanoprisms.

Single-crystalline gold nanobelts have been prepared sonochemically from aqueous solution of HAuCl4 in the presence of α-D-glucose, a biological directing agent, under ambient conditions (Figure 2).

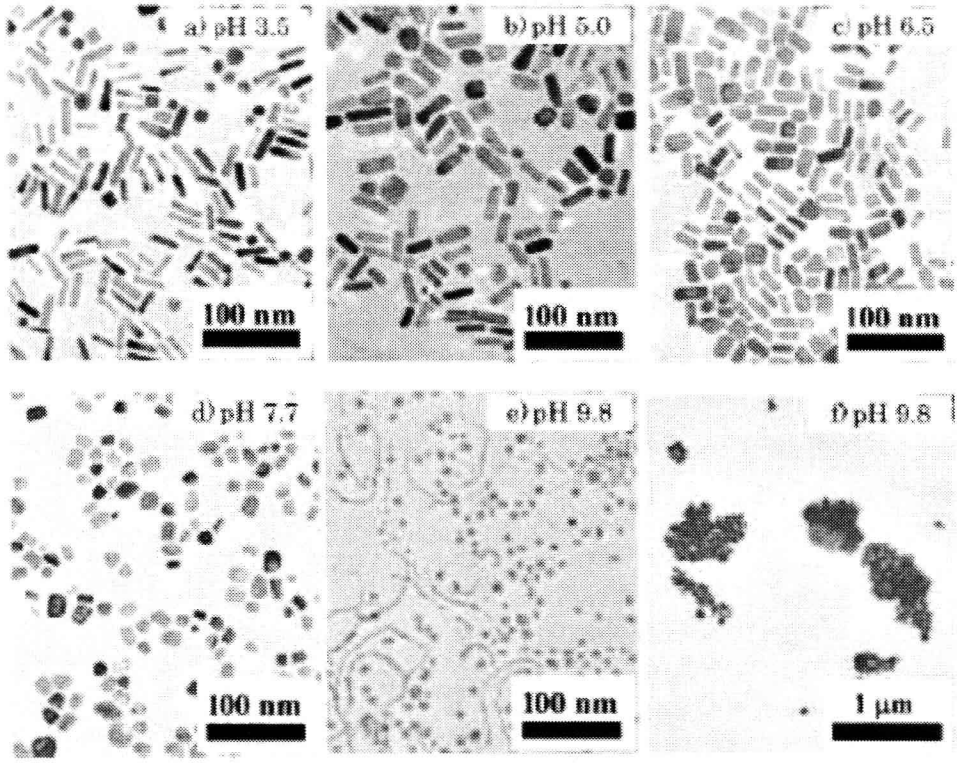

Figure 1. TEM images of gold nanorods and nanoparticles formed in different pH solutions of (a) pH 3.5, (b) pH 5.0, (c) pH 6.5, (d) pH 7.7, and (e) pH 9.8 after 180 min irradiation under argon. (f) TEM image of gold nanoparticles formed in pH 9.8 without ultrasonic irradiation.

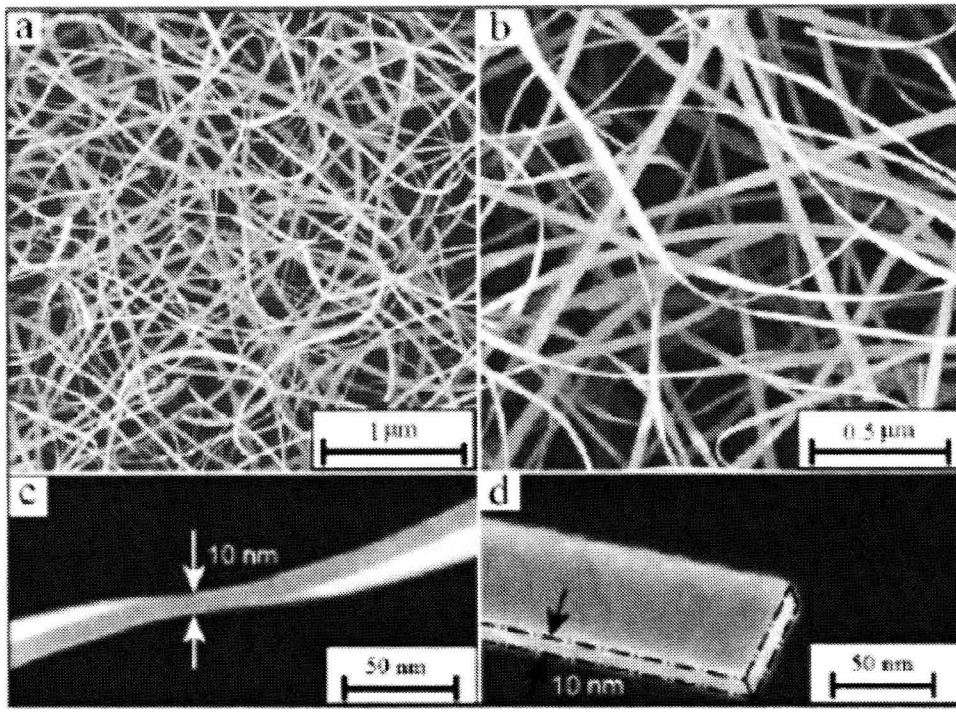

Figure 2. a,b) SEM images and c,d) high-magnification SEM images of as-synthesized gold nanobelts; $[HAuCl_4]$=50 $mgmL^{-1}$, [α-D-glucose]= 0.2 m, ultrasound time=1 h.

The formation of gold nanobelts depends on the concentration of α-D-glucose. When its concentration was as low as 0.05 M, only gold particles with a size of approximately 40 nm were obtained [10]. In the dilute solution, the glucose can not provide effective coverage or passivation of gold facets. The gold nanobelts have a width of 30–50 nm and a length of several micrometers with highly flexibility. Nanobelts have thickness of approximately 10 nm. Authors also showed that only spherical particles with a diameter of approximately 30 nm were obtained in the presence of β-cyclodextrin. It was mentioned that ultrasound irradiation can enhance the entanglement and rearrangement of the α-D-glucose molecules on gold crystals.

Park et al. showed the effects of concentration of stabilizer (sodium dodecylsulfate: SDS) and ultrasonic irradiation power on the formation of gold nanoparticles (Au-NPs) [11]. The multiple shapes and size distribution of Au-NPs are observed by different ratio of Au (III) ion/SDS and ultrasonic irradiation power.

A sonochemical method in preparation of gold nanoparticles capped by thiol-functionalized ionic liquid (TFIL) in the presence of hydrogen peroxide as a reducing agent reported by Jin et al. [12]. It was demonstrated that the molar ratio of gold atom in chloroauric acid to thiol group in TFIL (Au/S) has great effects on the particles size and distribution of gold nanoparticles. Small gold nanoparticles size of 2.7±0.3 nm can be synthesized when ultrasound irradiation applied to a solution with the molar ratio of Au/S = 1:2 for 12 h.

2.2. Palladium

Nemamcha et al reported the sonochemical synthesis of stable palladium nanoparticles by ultrasonic irradiation of palladium (II) nitrate solution in ethylene glycol and in the presence of poly(vinylpyrrolidone) (PVP) for 180 min [13]. During the ultrasonic irradiation of the palladium (II) nitrate mixture, the color of the solutions turned from the initial pale yellow to a dark brown. The following mechanism was proposed:

$$H_2O \xrightarrow{))))} OH^{\bullet} + H^{\bullet} \tag{7}$$

$$HOCH_2CH_2OH + OH^{\bullet}(OH^{\bullet}) \rightarrow HOCH_2C^{\bullet}HOH + H_2O(H_2) \tag{8}$$

$$nPd(II) + 2nHOCH_2C^{\bullet}HOH \rightarrow nPd(0) + 2nHOCH_2CHO + nH^{+} \tag{9}$$

The coordination of the PVP carbonyl group to the palladium atoms causes to the stabilization of the Pd nanoparticles in ethylene glycol. It has been shown by TEM that the increase of the Pd (II)/PVP molar ratio from 0.13 ×10-3 to 0.53 ×10-3 decreases the number of palladium nanoparticles with a slight increase in particle size. For the highest Pd (II)/PVP value, 0.53 × 10-3, the reduction reaction leads to the unexpected smallest aggregated nanoparticles.

2. 3. Tellurium

Crystalline tellurium nanorods and nanorod branched structures are successfully prepared at room temperature via an ultrasonic-induced process in alkaline aqueous solution containing tellurium nitrate, D-glucose and polyethylene glycol (PEG-400,CP) for 2 h treatment in an ultrasonic bath [14]. A yellow sol was produced and was kept in darkness for 24 h to allow the growth of Te nanocrystals. The as-obtained nanorods are single crystalline with [0 0 1] growth orientation, and have 30–60 nm in diameter with 200–300 nm in length. Some branched architectures, consisting of several nanorods, are also found in the products. The formation of the branched structures is suggested to be the result of multi-nuclei growth in monomer colloid.

2.4. Tin

Metallic tin nanorods were synthesized by a sonochemical method employing the polyol process [15]. In the reaction a solution of SnCl2 in ethylene glycol was exposed to high-intense ultrasound irradiation. The crystallized metallic tin nanorods have diameters of 50–100 nm and lengths of up to 3 μm were synthesized. In the absence of the high-intensity ultrasonic irradiation, no reduction of tin ions occurs even at temperatures as high as 500 °C in a closed cell.

2.5. Ruthenium

Ruthenium nanoparticles have been prepared by sonochemical reduction of a ruthenium chloride solution in 0.1 M perchloric acid containing propanol and SDS for almost 13 h [16]. The effects of different ultrasound frequencies in the range 20–1056 kHz were investigated. The Ru particles have diameters between 10 and 20 nm. The rate of Ru (III) reduction by the sonochemical method is very slow. The sonochemical reduction rate has been found to influence by ultrasound frequency. An optimum reduction rate was determined in the frequency range 213–355 kHz.

2.6. Germanium

Wu et al. reported a method based on ultrasonic solution reduction of GeCl4 by metal hydride (LiAlH4 and NaBH4) or alkaline (N2H4·H2O) in tetrahydrofuran (THF) and in ambient condition [17]. The germanium nanocrystals have narrow size distribution with average grain sizes ranging from 3 to 10 nm. Octanol was used as capping agent. To prevent the formation of GeO2 formed in the presence of water, the anhydrous salt is added to form a transparent ionic solution in THF.

2.7. Selenium

Single crystalline trigonal selenium (t-Se) nanotubes with diameters of less than 200 nm and nanowires with diameters of 20-50 nm have been synthesized by the reduction of H2SeO3 in different solvents with a sonochemical method [18]. The morphology of the products depends on the reaction conditions including ultrasonic parameters (e.g., frequency, power, and time), aging time, and solvent. Hydrazine hydrate was dissolved in ethylene glycol, water, etc. to form solutions. The solution was added dropwise to the corresponding selenious acid solution. At the same time, ultrasound was preceded to the solution, and the ultrasonic time is 30-60 min. Selenium nanotube and nanowire formation involved several stage:

$$H_2SeO_3 + N_2H_4 \xrightarrow{)))} Spherical(\alpha - Se) \xrightarrow{)))} Spherical - like(t - Se) \xrightarrow{)))} Nanowires(t - Se) \quad (10)$$

2.8. Silver

Dendritic silver nanostructures were formed by means of ultrasonic irradiation[19] of an aqueous solution of silver nitrate with isopropanol as reducing agent and PEG400 as disperser for 2 h.

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