



Leonid Kustov ^{1,2,3,*} and Kseniia Vikanova ^{1,2}

- ¹ N.D. Zelinsky Institute of Organic Chemistry, Russian Academy of Sciences, Leninsky Prospect 47, 119991 Moscow, Russia; ks.vikanova@gmail.com
- ² Chemistry Department, Moscow State University, Leninskie Gory 1, Bldg. 3, 119992 Moscow, Russia
- ³ Institute of Ecology and Engineering, National Science and Technology University MISiS, Leninsky Prospect 4, 119071 Moscow, Russia
- * Correspondence: lmk@ioc.ac.ru

Abstract: Microwave irradiation is widely used to intensify various chemical processes, including in the synthesis of nanomaterials. The purpose of this review is to present recent trends in the application of microwave heating for the preparation of monometallic, bimetallic, and more complicated metal nanoparticles. The advantages and drawbacks behind the use of microwave irradiation in the synthesis of unsupported and supported metal nanoparticles are summarized. Such metals as Ag, Au, Pt, Pd, Rh, Ir, Cu, Ni, Co, etc. are considered. Illustrative examples have been presented of the positive effects of microwave treatments, including the preparation of particles of different shapes and particles of immiscible metals.

Keywords: metal nanoparticles; microwave irradiation; bimetallic nanoparticles; supported nanoparticles; immiscible metals

1. Introduction

The progress in the preparation of nanomaterials becomes possible by using nontraditional approaches, such as nonequilibrium conditions, gradient technologies, and the ex situ or in situ application of electromagnetic activation modes, such as microwave activation, low-temperature plasma, etc. Microwave technologies have been introduced in many areas, including industrial processes [1]. Microwave irradiation is widely explored in the processes of synthesis of diverse materials.

The microwaves are electromagnetic waves with a frequency ranging from 200 MHz to 300 GHz. The 915 MHz frequency (33.3 cm) is most frequently used in industrial devices, whereas the frequency 2.45 GHz (12.2 cm) is used in microwave laboratory setups.

The important advantage of microwave heating is the fast transfer of energy through radiation rather than heat transfer or convection. This ensures fast penetration of energy into the volume of materials transparent to microwave radiation, i.e., instantaneous heating.

The most important effects related to the appearance of temperature gradients and nonequilibrium conditions are observed when a reaction medium or material consists of several phases with different microwave radiation absorption coefficients.

Microwave treatment allows one to decrease the reaction duration, increase yields and selectivities, and decrease energy and reagent consumption. The use of microwave-energy pulses provides an additional reduction in the energy consumption.

One of the possible mechanisms of microwave action, which is widely realized in the case of metal nanoparticle synthesis due to the presence of water, alcohols, and other polar, mostly oxygen-containing compounds in the reaction medium, is dielectric polarization. The second mechanism involves currents of free charges excited in solids and contributing to heating because of Ohmic loss; this mechanism is characteristic of materials possessing a substantial conductivity (metals and semiconductors) and particles supported onto



Citation: Kustov, L.; Vikanova, K. Synthesis of Metal Nanoparticles under Microwave Irradiation: Get Much with Less Energy. *Metals* **2023**, *13*, 1714. https://doi.org/10.3390/ met13101714

Academic Editor: Frank Czerwinski

Received: 4 September 2023 Revised: 26 September 2023 Accepted: 27 September 2023 Published: 8 October 2023



Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). (semi)conductive carriers. The third mechanism that should also be taken into account is caused by the loss of vortex currents excited by magnetic fields.

Certainly, microwave fields cannot initiate chemical reactions by exciting and, especially, breaking chemical bonds, as in photochemical processes under the action of the electromagnetic radiation of the UV-visible range. The energy of microwave photons is just ~1 J/mol, which is significantly lower than the activation energies of chemical processes. So, in the case of metal nanoparticle preparation, all three mechanisms can be realized if the system contains polar molecules and solid materials that are capable of absorbing microwave energy. In the case of metal nanoparticles supported onto a solid carrier, attention should be given to microwave absorption by the materials (carbon materials, polymers, and oxides) used as the components of carriers. Whereas carbon and transition metal oxides reach high temperatures under microwave heating, titanium, and zirconium oxides are heated to moderate temperatures, while aluminum, magnesium, and silicon oxides cannot be heated above 100–150 °C. A dispersed system consisting of conducting particles is a nonuniform material whose dielectric properties exhibit pronounced frequency and phase-composition dependences. The dielectric loss in such materials is related to the aggregation of charges at interphase boundaries and is known as the Maxwell–Wagner effect. It is obvious that in the case of metal nanoparticle synthesis, both the dipolar polarization mechanism and conduction mechanism, as well as the Maxwell–Wagner effect (microwave absorption at the interface of several phases), can be responsible for microwave heating.

Numerous data show that, under microwave-heating conditions, higher reaction rates are observed than in thermally activated processes, and the yields of final products increase. These advantages are often ascribed to so-called "microwave effects" without a detailed analysis of the reaction conditions. It is important to separate thermal and nonthermal (electromagnetic) microwave effects.

There are serious grounds for believing that processes occur under the action of microwave fields in a different way than under conventional thermal activation with convective or conductive heating. The necessary condition for the efficient action of a microwave field on a process is the strong interaction of solid or polar liquid components with this field.

The development of additional criteria (except for temperature-conversion dependences) for revealing specific effects that appear under the action of microwave fields on reacting systems is an important problem. This is related, in particular, to the changes in the kinetic characteristics and dependences (reaction orders with respect to components and activation energies) that are less sensitive to errors in temperature measurements than a comparison of activities or transformation rates. The changes in the process selectivity, with respect to certain products at equal conversions and different methods of reacting system activation, are most interesting and informative.

It is also necessary to track changes in the state of a nanomaterial prepared under the action of microwave fields. In certain instances, the appearance of metastable structures that are not typically formed in the absence of such action can be expected. If they have catalytic properties and/or reactivity different from those observed usually, the appearance of the corresponding nontrivial effects is possible.

Microwave radiation creating temperature gradients in a heterogeneous system whose components differently interact with electromagnetic fields can then cause substantial changes in the reaction rates of the entire process, the rates of different stages, and, what is especially important, may change the distribution of reaction products.

Metal nanoparticles are widely used in catalysis, environment protection, sensors, optoelectronic applications, biocides, and biomedicine. Each of these applications requires metal nanoparticles with a very narrow particle-size distribution in order to provide the optimized performance following the size effects. Also, nanoparticles of definite size should be used to ensure the best activity/selectivity/sensitivity in various applications. As an example, some catalytic reactions require the presence of noble nanoparticles with a very narrow distribution and small size (2 nm), such as Au in CO oxidation or structure-sensitive

dehydrogenation reactions, while other catalytic reactions proceed more efficiently when larger metal particles are present (some hydrogenation and other structure-insensitive reactions). Therefore, the frontiers in the preparation of metal nanoparticles with desirable size and distribution are dependent on the elaboration of the unique methodology providing the required product formation. Microwave-assisted synthesis is considered among the efficient candidate methods to achieve these goals. Thus, the importance and application prospects of microwave-assisted synthesis of metal nanoparticles are high.

The advantages and disadvantages of the use of microwave radiation in the synthesis of metal nanoparticles (supported or nonsupported) compared with conventional thermal heating are presented in Table 1.

Table 1. Advantages and disadvantages of the use of microwave radiation in the synthesis of metal nanoparticles compared with conventional thermal heating.

Advantages	Disadvantages		
Economy of energy	Difficulties with measurements and control of temperature		
Mild reaction conditions	Sometimes nonequilibrium processes are difficult to control		
Reduction of time of synthesis	Sometimes the processes are too fast		
Fast heating	Difficulties with scale-up		
Fast change of regimes	Typically, a small-scale preparation		
Fast cooling	Limited pressure range		
Nonequilibrium process	Limited geometry of the reactor		
High rate of nucleation	Possible electric arching or plasma effects		
Accelerated growth of particles (temporally separated from the nucleation stage)	Not all solvents are suitable		
Selective heating	Temperature dependences of loss tangents are rarely known		
No need to use capping agents or surfactants	Difficulties with in situ monitoring of the process		
Possibility to use pulse mode of operation			
Narrow particle-size distribution			
Small size of nanoparticles			
Hot spots/hot surfaces/superheated areas			
Realization of metastable phases			
Elimination of barrier effects caused by inverted temperature gradients			
Reduced temperature gradient			
Reduced wall effects			
No need to heat the walls, gases			

Some of these advantages are general and some of them are specific to the case of the preparation of metal nanoparticles. The important advantage becomes obvious when fast heating or cooling and a pulsed energy supply are required. In the review, we will try to illustrate most of these advantages/drawbacks with examples of the microwave-assisted synthesis of diverse metal nanoparticles. Apart from the search for and synthesis of suitable catalysts, the development of new microwave equipment for applications on pilot and industrial scales is necessary, as well as process upscaling.

The effects observed in the synthesis of metal nanoparticles, especially bimetallic and core–shell nanoparticles, both supported and unsupported, under the action of microwaves, are still not well understood and have been poorly studied, although it is clear that the irradiation of microwave-absorbing materials can cause rapid volume heating.

In practice, mixtures containing substances that weakly and strongly absorb microwave radiation are often used for microwave heating. By varying the composition of such mixtures, it is possible to control the maximum temperature of the mixture heating and the composition of reaction products. A comparison of traditional conditions with the microwave regime is, however, not always possible because of difficulties related to true process temperature measurements in the latter case.

Actually, the first publications related to the synthesis of metal nanoparticles using MW heating appeared in the 1990s. Synthesis of metal nanoparticles and the basic features of such microwave syntheses, including thermal and nonthermal effects as well as the mechanisms of microwave heating, are discussed in a few excellent reviews and research papers, most of them being published more than 10 years ago [2–20].

Principally, microwave irradiation affords uniform internal heating of the reaction mixture resulting in facile nucleation and crystallization. Other benefits include faster temperature ramping (temperatures of 150–250 °C can be achieved in less than 1 min,

compared to over 30 min for conventional heating), a lack of convection processes, and convenience of operation (Table 1) [21].

The absorption of microwave energy by metal nanoparticles, unlike their bulk counterparts, becomes significant because of their developed surface area and particle curvature, as well as large refractive indices [22,23].

In our paper, we will focus on very recent publications of 2010–2023. Let us discuss the most impressive examples of the use of microwave activation in the preparation of metal nanoparticles. The presentation of results will start with the most often synthesized metals, Ag, Au, Pd, and Pt; then, examples of other noble metals, non-noble metals, and bimetallic (or trimetallic) particles will be discussed.

2. Noble Metals and Silver

2.1. Silver Nanoparticles (NPs)

Silver nanoparticles are widely used in materials with antibacterial activity. The particle-size effect has been disclosed in many publications. Therefore, the development of a "green" and energy-saving method of production of silver nanoparticles with a uniform particle distribution and a desirable median size is of utmost importance [24,25]. Microwave radiation during the synthesis is one of the efficient tools to control the properties of Ag NPs [26–37].

Tsuji et al. developed a general procedure for the preparation of metal nanoparticles in a solution. For instance, Ag, Au, Pt, and AuPd nanoparticles have been obtained by the microwave method [9,38]. Both spherical nanoparticles and single crystalline polygonal plates, sheets, rods, wires, tubes, and dendrites were prepared within a few minutes under MW heating. The general scheme of microwave-assisted synthesis of metal nanoparticles of different shapes reprinted from Ref. [9] is presented in Figure 1. In most cases, metal nanoparticles of a smaller size and narrower particle-size distribution, as well as a higher degree of crystallization, were synthesized under MW heating compared to metal nanoparticles obtained by conventional oil-bath heating or solvothermal synthesis.



Figure 1. The general scheme of microwave-assisted synthesis of metal nanoparticles of different shapes. Reprinted with permission from [9]. Copyright 2004 Wiley-VCH.

Gou et al. succeeded in the synthesis of Ag nanowires in ethylene glycol in the presence of polyvinylpyrrolidone [39]. The authors explained the production of nanowires by the polarization of conducting electrons, resulting in local "hot spot" formation at the wire ends, which, in turn, promoted nanowire growth.

Jiang et al. investigated the effect of the MW frequency on the synthesis of silver nanoparticles via reduction using polyols and found improvement of the distribution (a narrow distribution) with increasing the frequency to about 6–7 GHz [29]. In addition to spherical Ag nanoparticles, triangular plates, sheets, rods, wires, tubes, and dendrites were synthesized under MW irradiation [12,40–45] (Figure 2). While low concentrations of silver in the starting solution are beneficial for the formation of spherical nanoparticles stabilized by polyvinylpyrrolidone (PVP), the increase of the concentration of silver nitrate provides conditions for the generation of more complicated shapes, such as triangular particles, which may be further transformed into dendrites and polygonal shapes.



Figure 2. Formation of triangular Ag plates. Reproduced from Ref. [45].

Tsuji et al. studied in depth the microwave-assisted synthesis of silver nanoparticles [9]. Chemical reduction with NaBH₄ afforded dodecanethiol-capped 1.8-3.5 nm Ag nanoparticles.

Yamamoto et al. [43] synthesized spherical Ag nanoparticles by reducing fatty acid Ag salts with alcohols as reducing agents with MW irradiation for 1–5 min at 140–157 °C. The nanoparticles with definite sizes in the range of 4.9–7.4 nm were obtained only by changing the length of the alkyl chain in the fatty acids.

Triangular Ag nanoplates (50–100 nm prisms) were prepared from silver nitrate in DMF as a solvent and simultaneously as a reducing agent by He et al. [41] while the use of solvents with a low boiling point (pyridine and ethanol) resulted in the formation of 8–30 nm spheres. Typically, prisms appear at an increased concentration of the precursor (AgNO₃) and/or at increasing times of microwave treatment.

Ag nanorods, nanowires, nanosheets, and nanotubes were also reported, and an additive of H_2PtCl_6 as Pt seeds assisted in the formation of such nanostructures in the

presence of polyvinylpyrrolidone (PVP) [46]. The diameters and lengths of the Ag nanorods and nanowires were 30–140 nm and 0.3–10 mm, respectively. The PVP/AgNO₃ molar ratio and the AgNO₃ concentration affected the aspect ratio for the formed elongated nanoparticles. The time of synthesis is reduced from 50 min (oil-bath conditions) to a few minutes (MW conditions). Ag nanosheets are formed only at a very short MW treatment, and such nanostructures are not produced at all under the conventional heating mode of operation.

Zhu and Hu [47] described MW-assisted polythiol reduction using 1,2-ethanedithiol as a reducing agent for the synthesis of Ag nanowires from Ag₂O at 80–140 $^{\circ}$ C with no need to use a surfactant. In the case of ethylene glycol used instead of thiol, only spherical particles are produced. Ag dendrites can also be prepared by the reduction of AgNO₃ [42].

Recent studies demonstrated successful Ag nanoparticle synthesis under microwave irradiation using diverse reducing and capping agents, such as formaldehyde [48], polyvinylpyrrolidone [29], ethanol [28], glycerol [49], ethylene glycol [35], and biological extracts, enzymes, bacteria, etc. [50]. The use of carboxymethyl cellulose as a green reducing and stabilizing agent turned out to be efficient in the preparation of uniform silver nanoparticles [51]. Amino acids can also be applied as reducing agents and soluble starch can be used as a protecting agent (irradiation at 150 °C for just 10 s) [26], providing the formation of Ag nanoparticles with a very narrow particle-size distribution (26 nm).

Synthesis of fluorescent Ag nanoclusters in the presence of sodium polymethacrylate taking just 60–90 s was reported [31]. The use of these nanoparticles, with the size of 2.0 nm for Cr^{3+} sensing based on the selective quenching effect of Cr^{3+} on the fluorescence emission of the Ag nanoclusters, was considered. It is noteworthy that, when conventional heating was used instead of microwave treatment, it took over 120 min for Ag nanoclusters with only weak fluorescence to be formed with a nonhomogeneous size and insignificant quantum yield (1%).

Ionic liquids are known as compounds with a high tangent of dielectric losses and one of them—1'-dialkyl-4,4'-bipyridinium(bis-triflimide)—was efficiently used in the synthesis of spherical silver nanoparticles with an average size of 20–30 nm (10–20 min, 500 W) [52]. The nanoparticles–graphite paste nanocomposite electrodes were used for the sensing of hydrogen peroxide.

The morphology of Ag nanoparticles is governed by the conditions of the MW-assisted synthesis. The MW-polyol procedure is an efficient method for the fast synthesis of nanorods, nanowires, polygonal nanoplates, nanosheets, and dendrite silver nanoparticles [9,12,40,46].

Rapid formation of novel Au core–Ag shell nanostructures by a microwave–polyol method was reported. The use of high-boiling solvents (dimethylformamide, bp = 153 °C), N-methylpyrrolidone, bp = 202 °C), resulted in the formation of well-shaped plate-like particles [41], whereas dendrites can also be formed in dimethylformamide under certain experimental conditions [42]. Triangular Ag nanoplates were prepared in aqueous solutions with PVP [45]. Spherical Ag nanoparticles tend to be formed in diluted AgNO₃ solutions, whereas an increase in the nitrate concentration and reaction time leads to the transformation of the spherical particles into prisms. Small Au@SiO₂ particles with a diameter of 17 nm and with a 2–5 nm SiO₂ shell were prepared under MW activation. In the case of using oleylamine as a capping agent, large 50–200 nm Ag@SiO₂ particles with 20–30 nm silica shells were synthesized under microwave irradiation.

In the case of the polyol–PVP-based procedure [53], the shape of Ag particles changes from plates to spherical ones (Figure 3). A very important advantage of MW synthesis is the possibility to quench (terminate) the synthesis by switching off the MW energy so that very fast cooling of the reaction mixture occurs, which prevents the occurrence of undesirable side reactions and recrystallization. The authors succeeded in taking snapshots of nucleation and growth processes during the preparation. At very short reaction times and low temperatures, polyhedral plates are produced with sizes of about 300 nm and a broad particle-size distribution. However, with an increase in the time or the temperature, the size and shape change to yield 10 nm spherical NPs with low polydispersity (Figure 3). Thus, kinetically favored large silver polyhedral plates are further transformed into thermodynamically favored small spherical nanoparticles.



Figure 3. Ag NP mean sizes depending on the synthetic conditions. Reproduced from Ref. [53] Copyright [2020] American Chemical Society.

Other examples of the synthesis of Ag nanoparticles of different shapes can be found in the recent literature [37,54].

The abundant number of publications on the microwave synthesis of Ag nanoparticles is based on the use of plant extracts as "green" reducing agents, though their "greenness" is questionable taking into account the resources to be spent for the extraction, separation, and purification of organic solvents, etc. [55]. Similarly, microwave combined with ultrasound treatment was used for the synthesis of *Olea europaea*-capped silver nanoparticles with the plant extract used as a capping and stabilizing agent [56]. The prepared Ag nanoparticles were 10 nm in diameter with a polydispersity of 0.191 and spherical-to-elliptical morphology. Antioxidant and antimicrobial activity of the thus prepared nanomaterials were studied and supplemented with theoretical calculations.

Two experimental units for continuous synthesis of Ag nanoparticles using microwave heating and conventional heating have been compared [32] and the data demonstrate a different temperature profile along the reactor, with the case of microwave heating being more favorable; the nanoparticles were obtained with a greater yield of 54% and a narrower particle-size distribution (19 ± 4.3 nm). On top of that, the microwave procedure allowed fast cooling of the product stream, thereby preventing the agglomeration of nanoparticles.

Ag@SiO₂ core–shell materials containing spherical Ag nanoparticles with an average diameter of 90 nm covered with a 2.5–3 nm thick silica shell were prepared for SERS application [57].

The synthesis of Ag nanoparticles supported onto a mesoporous silicate SBA-15 has been accomplished under 200 W MW heating for 70, 35, and 17 s under intensive stirring [58]. The materials were used as catalysts for the activation of alkynes showing excellent activity for the formation of C–O, C–N, and C–C bonds. Being compared with homogeneous Ag salts, Ag NPs demonstrated enhanced catalytic activity toward the cyclization of substrates, while minimizing the metal loading and allowing the separation, as well as the recyclization, of the catalyst in a cyclic mode.

The sensibility to visible light for titanate nanotubes as a photocatalyst for rhodamine B dye degradation was achieved by adding Ag nanoparticles prepared by microwave treatment (100 W, 5 min) using sodium borohydride as a reducing agent [59]. Ethylene glycol was chosen as the best solvent.

Ag nanoparticles were also applied to encapsulate PbS nanoparticles, and the materials prepared were used in optoelectronic applications [60]. Lead nitrate, sodium sulfide, cetyltrimethylammonium bromide, and silver nitrate were used as precursors; the microwave treatment was performed only at the final stage of the preparation when the preliminarily formed PbS nanoparticles were encapsulated in Ag nanoparticles (10 min at 700 W). Unfortunately, no comparison was made with the fully conventional preparation method. The resulting particles had a size of 15–40 nm. The band gap of Ag:PbS NPs was 1.95–2.51 eV and increased with increasing Ag loading, as well as the intensity of the photoluminescence quenching. The materials of this type can find application as visible-light photodetectors. The higher the Ag loading, the more significant enhancement of the main electrical parameters was found, including dark current, photocurrent, surface resistivity, responsivity, quantum efficiency, and specific detectivity.

ZnO is widely used as a support in the synthesis of Ag nanoparticles. Microwaveassisted solution synthesis of Ag nanoparticles supported on flower-like ZnO particles and the evaluation of their photocatalytic activity in photodegradation of methylene blue under ultraviolet light was studied [61]. The enhanced photocatalytic activity was ascribed to the formation of a Schottky barrier at the metal–semiconductor interface in Ag/ZnO nanocomposites. Unfortunately, no comparison with thermally prepared Ag/ZnO nanocomposites was made, as in many other publications.

A nanostructured flower-like Ag hierarchical composite based on Ag nanoparticles supported on ZnO was prepared by microwave heating, but the Ag nanoparticles were prepared by a conventional reduction procedure [62], whereas the microwave treatment was used only at the final stage of the composite formation. Again, no comparison was made with the conventional preparation method. The material synthesized was studied in the SERS application and the enhancement of the sensitivity (the lowest detectable concentration) being 5–8 orders of magnitude was demonstrated for Rhodamine B. The high sensitivity was also shown using glucose and paracetamol as analytes.

Materials of similar composition (Ag/ZnO) were synthesized under MW conditions by using a plant extract (*Averrhoa carambola*) as a reducing agent aiming at the target application of the cotton-coated materials containing Ag/ZnO particles as bactericides and UV-light protecting materials [63].

Starch-stabilized silver nanoparticles with a size of below 10 nm were also prepared using microwave heating [64]. Similarly, chitosans have been used in the microwave-assisted synthesis of Ag nanoparticles [65].

2.2. Gold Particles

Gold nanoparticles invoke interest because of their unique optical (plasmon resonance etc.) and catalytic properties. Gold nanoparticles were among the first reported examples of metal nanoparticles prepared under microwave irradiation [9,66]. Typically, spherical particles with a size of about 10 nm or smaller are formed within a few minutes under these conditions in organic solvents with PVP added to stabilize the nanostructures and ethylene glycol as a reducing agent, while longer treatments result in the production of different shapes of crystals, including triangular, truncated triangular, square, pentagonal, or hexagonal nanoplates (30–90 nm). It has been shown that anisotropic growth takes place at uniform heating under MW irradiation, yielding the most stable {111} face on the top surfaces of the particles. It was suggested that nonthermal effects play a significant role in the formation and growth of metal nanoparticles, as evidenced by different shape and size distributions in the case of oil-bath heating and microwave heating (either continuous or pulse mode). Acceleration of the nucleation rate due to rapid and homogeneous dielectric heating, which is not achievable under oil-bath heating, was the main reason behind the observed differences. Also, temperature gradients at the particle surface due to polarization of the stabilizing molecules may contribute to this difference. The addition of specific

surfactants that induce anisotropic growth leads to the formation of Au nanorods and nanowires under MW heating. Spherical 13-nm particles were also reported by Liu after the MW processing for 20–30 min [67]. Kundu et al. used 2,7-dihydroxynaphthalene for the reduction of gold ions and for the synthesis of gold nanoparticles of different shapes [68]. Spheres, rods, or triangles were produced by changing the ratio of metal ions to cetyltrimethylammonium bromide and the concentration of the reducing agent.

Ionic liquids demonstrate unique properties as reducing and templating agents in the synthesis of metal nanoparticles. Ren et al. used the advantage of the microwave-absorbing properties of ionic liquids and their templating effect for the production of gold nanostructures [69]. The microwave-assisted ionic liquid method has been proven to be suitable for the rapid synthesis of a variety of elemental and compound nanostructures, including Au [70]. The authors synthesized Au nanoparticles with a size of 1–2 nm by microwave-assisted decomposition/reduction of Au(CO)Cl or KAuCl₄ in the ionic liquids [BMIm]⁺[BF₄]⁻, [BMIm]⁺[OTf]⁻, [BtMA]⁺[NTf₂]⁻ (where [BMIm]⁺ stands for n-butylmethylimidazolium cation, [BtMA]⁺ is n-butyltrimethylammonium ion, [OTf]⁻ = $-O_3$ SCF₃, [NTf₂]⁻ = $-N(O_2$ SCF₃)₂). The particle diameter increased with the increasing volume of the anion.

Au nanosheets were derived from $HAuCl_4$ in $[BMIM][BF_4]$ under microwave heating [71]. The ionic liquid served as a template for the genesis of Au nanosheets. Ren et al. [69,72] prepared Au polyhedral crystals, single-crystalline nanoplates, hollow trapeziform crystals, holey polyhedra, and dendrites from $HAuCl_4$ in imidazolium and pyridinium ionic liquids without any capping agent or reducing agent under the microwave mode of operation. The nature of anions and cations affected the morphology of the Au nanoparticles.

Quite a number of publications considered the use of bioavailable molecules for the preparation and reduction of gold from precursors under microwave heating [73–75]. Nguyen et al. studied the microwave-assisted synthesis of Au nanoparticles using a *Ganoderma lucidum* extract [76]. In comparison with conventional heating, the microwave procedure produced smaller AuNPs with a size of 22 nm in a short synthesis time period (10 min at 400 W). Microwave-assisted synthesis promoted nucleation. Therefore, MW-AuNPs with high crystallinity and smaller and uniform particles can be produced. The naked Au nanoparticles manifested high activity in 4-nitrophenol to 4-aminophenol reduction with NaBH₄. One more example of the use of plant extracts for the microwave-assisted (3 min at 600 W) reduction to produce 10-nm Au nanoparticles is presented by Kumari et al. [77]. The nanoparticles were efficient in the reduction of p-nitrophenol, Congo Red, and Methylene Blue with NaBH₄.

Gold nanoparticles prepared by microwave heating with a plant extract had about the same size (about 17 nm) as conventionally prepared Au NPs (reduction with the same extract) [78]. Dextran was also used for the microwave reduction of gold with a very short treatment time (40 s) [79].

Of particular interest are the Au particles with a size of 5 nm and below. In this concern, the one-step solid-state microwave synthesis (1200 W, 40 s) of Au nanoparticles of different sizes and shapes ranging from 3-nm nanoparticles to polyhedra of about 200 nm by using a paste of glucose powder with an aqueous solution of HAuCl₄ may provide some guidelines [80]. It is noteworthy that, the reaction mixture underwent a sudden explosion (with rapid volume expansion) with the instantaneous formation of a brownish solid mass. This is the first example of the nucleation and growth of Au NPs occurring in the solid state.

Au nanoparticles (average size 22.34 nm) with a uniform distribution on the carbon support were prepared using solid-state microwave heating of the supported Au precursor (contrary to the liquid-phase syntheses of metal nanoparticles) [81]. Magnetic gold nanotriangular nanoparticles were also prepared by microwave-assisted polyol synthesis [82].

Quite large (200–500 nm) gold nanoparticles with hexagonal plate morphology with (111) planes as basal surfaces were prepared by the microwave method (800 W, 60 s) in an

aqueous media by using, for the first time, (3-mercaptopropyl)trimethoxysilane (MPTMS) as a reducing agent [83]. Au NPs demonstrated a high activity in the reduction of 4nitrophenol to 4-aminophenol. Partial separation of the stages of nucleation and growth results in the formation of first small and then large gold nanoparticles, but of the same hexagonal morphology (Figure 4).



Figure 4. MW synthesis procedure of hexagonal gold nanoparticles (**a**) and schematics of Au NPs growth and formation (**b**). Reprinted with permission from [83].

So-called "self-supported superstructures" formed by smaller Au nanoparticles have been designed by using microwave-assisted synthesis [84].

Supported Au nanoparticles usually demonstrate good stability at a small particle size. Au NPs on diverse transition metal oxides (Ti, Fe, etc.) are known from the works by Morad et al. to show high activity in CO oxidation under mild conditions. Au NPs were supported onto Mn_3O_4 under microwave heating with hydrazine hydrate as a reducing agent [85].

The use of ZnO as a support for gold nanoparticles, with a size of about 12 nm synthesized under microwave heating (300 W, 20 min), and the efficient performance in photocatalytic degradation of methylene blue, were also demonstrated [86].

Au nanoparticles on nitrogen-doped graphene were prepared using microwave irradiation with sodium citrate as a reducing agent [87]. Outstanding electrochemical properties of these materials were demonstrated.

Other examples of the use of microwave irradiation in the preparation of Au nanoparticles of diverse shapes (hexagon nanoplates, spherical triangles) are available in the literature [67,80,82,88–94].

2.3. Palladium Particles

Palladium nanoparticles prepared by the glycol-assisted reduction of a Pd precursor under MW radiation turned out to be a little smaller than the particles synthesized by conventional heating (3.74 nm vs. 4.22 nm) [95].

Continuous (flow conditions) synthesis of large quantities of very small polymerstabilized Pd particles with a size of about 1.5 nm under microwave irradiation at contact times of 15–30 s in ethylene glycol has been reported [11].

A comparison of conventional water-bath heating and microwave heating for the preparation of Pd nanoparticles in the presence of cellulose with further testing in the catalytic application of methylene blue degradation was performed [96]. The MW-synthesized sample turned out to be about three times more active than the thermally prepared one [97]. MW-assisted preparation of Pd nanoparticles on a carbon support by the polyol procedure was performed in a continuous mode using a flow-type single-mode microwave reactor [98].

Pd nanoparticles (average size 19.77 nm) with a uniform distribution on the carbon support were prepared using solid-state microwave heating of the supported Pd precursor (contrary to the liquid-phase syntheses of Pd nanoparticles) [81].

Nitrogen-doped and oxygen-functionalized carbon nano-onions were chosen to support palladium nanoparticles by the microwave method, which were further applied as anodic electrocatalysts for direct alkaline ethanol fuel cells [99]. Both nitrogen and oxygen species of the support are responsible for the stabilization of Pd nanoparticles (with a size of 5–7 nm), thereby improving the electrochemical performance of the anode materials. Microwave synthesis of Pd nanoparticles over N-doped reduced graphene oxide was reported (300 W, 220 °C, 1 h) [100], and the materials were used efficiently for the ethanol oxidation reaction in alkaline media, with the Pd/N-rGO catalyst demonstrating enhanced catalytic activity compared to the Pd/rGO catalyst. Li et al. prepared Pd/rGO under microwave irradiation and ethylene glycol was used as a reducing agent [101]. The size of palladium nanoparticles ranged within 10–15 nm.

Similarly, microwave-assisted reduction of the precursor with ascorbic acid as a green reducing agent (irradiation for 4 min at 70 °C) to produce Pd nanoparticles (the size of about 28–29 nm) on reduced graphene oxide was described [102]. It is noteworthy that this method is used for the simultaneous reduction of both palladium and graphene oxide.

The microwave irradiation method (850 W, 3 min) was used for the synthesis of palladium nanoparticles of the spherical shape with a particle-size distribution of 13–33 nm using ascorbic acid (as a reducing agent) and sodium alginate (as a stabilizer) [103]. The antioxidant properties and cytotoxic effects of the Pd nanoparticles were assessed.

Transition metal oxides were also used as supports for Pd nanoparticles prepared by the microwave method, for instance, CuO [104]. The reduction conditions were as follows: palladium nitrate as a precursor, MW input power 250 W, temperature 95 °C, and duration 20 s. The Suzuki cross-coupling reaction on these catalysts was also carried out under microwave heating. The same authors prepared Pd nanoparticles on different transition metal oxides—FeO, CoO, and Ni(OH)₂—and used these materials for CO oxidation [105].

The microwave irradiation synthesis of a Pd/TiO₂ nanocomposite at 150 °C and 11 bar taking 3 min using titanium (IV) isopropoxide and palladium(II) chloride as precursors was reported [106]. The materials turned out to be active in photocatalytic nitrate reduction and ammonia production. It is interesting to note that TiO₂ itself is also prepared by the microwave method.

Pd nanoparticles on titania were also prepared by a nonaqueous sol–gel microwave method (15 min at 80 °C) by different authors [107], the Pd/TiO₂ systems were tested to reveal their visible-light photocatalytic activity, for instance, in photocatalytic H₂ production from methanol.

There are some examples of the microwave-assisted synthesis of more complicated materials, for instance, Pd nanoparticle-modified Bi_2MoO_6 nanoplates (microwave heating at 300 W, 10 min) [108]. The nanocomposites were used as visible-light-driven photocatalysts for rhodamine B degradation.

2.4. Platinum Particles

Spherical Pt nanoparticles were the first metal nanoparticles prepared by microwave dielectric heating of a poly(N-vinyl-2-pyrrolidone)/ $H_2PtCl_6/NaOH/H_2O$ /ethylene gly-col mixture [13]. Synthesis of Pt nanoparticles under microwave heating of a solution containing H_2PtCl_6 and 3-thiophenemalonic acid in water was presented by Luo [109].

Continuous synthesis of large quantities of very small polymer-stabilized Pt particles with a size of about 1.5 nm under microwave irradiation with contact times of 15–30 s in ethylene glycol has been reported [11].

Microwave-assisted synthesis in toluene using $Pt(acac)_2$, oleyl amine as a shapecontrolling additive, and ascorbic acid as a reducing agent resulted in cubic Pt NPs with a size of 8 nm (1200 W, 135 °C, 30 min) [110]. The use of the maximum power of 1200 W is caused by the choice of the solvent, toluene, which is a poor MW-absorbing substance.

Pt nanoparticles supported on carbon black, carbon nanotubes, and herringbone-type carbon nanofibers were prepared by an MW-assisted polyol process [111]. Pt nanoparticles (average size 2.51 nm) with a uniform distribution over the carbon support were prepared using solid-state microwave heating of the supported Pt precursor (contrary to the liquid-phase synthesis of Pt nanoparticles) [81].

Ionic liquids turned out to demonstrate improved performances in microwave heating in the synthesis of Pt nanoparticles supported onto carbon nanotubes due to their high tangent loss and their specific dependence on the temperature [112].

Very interesting results were found when studying the MW synthesis of Pt nanoparticles under high pressures and high temperatures [113]; i.e., under conditions of the near-supercritical liquid, the experiments were carried out in water, which changes its polar properties when approaching the supercritical state. The reactor enables the operation in the pressure range from 1 to 50 bar, the maximum temperature is 300 °C. The advantage of the high pressures and high temperatures in such a synthesis is observed when the ionic strength of the system is low. This reduces the electrostatic stabilization of the Pt particles. Furthermore, the morphology of Pt particles changes under high pressures and temperatures. The authors succeeded in preparing elongated nanoparticles of Pt using sodium citrate under MW conditions.

Platinum nanoparticles are most often used in fuel-cell electrodes and as the catalytically active phase of hydrogenation reactions. Unlike rhodium and palladium nanoparticles prepared by MW-assisted and conventional reduction with a glycol method, the MW-synthesized platinum particles were somewhat smaller in diameter compared to the conventionally prepared Pt particles (3.09 nm vs. 4.14 nm) [95]. One more research group reported the microwave synthesis of Pt-Cu nanoparticles by the microwave method (1000 W, 200 °C, 30 bar, 5 min) [114].

Yttria-stabilized Pt nanoparticles synthesized by the microwave method from platinum(II) acetylacetonate and yttrium nitrate hexahydrate demonstrate enhanced activity in the oxygen-reduction reaction in comparison with a conventional Pt/C catalyst with a low content of the precious metal in the electrocatalyst [115].

Tsuji et al. demonstrated that a 30 min reduction via the microwave polyol method produced Pt-YOx/C materials with the Pt particle size of 3 nm [116]. A comparison with conventional thermal preparation showed that the microwave procedure led to lower particle aggregation.

Carbon materials are most often used as supports for Pt nanoparticles due to the abovementioned fuel-cell applications; therefore, many research papers are focused on the use of microwave heating in the preparation of such modified Pt/C electrocatalysts [117–121]. Pt/C systems were synthesized via the polyol route using (NH₄)₂PtCl₆ as a Pt precursor under microwave irradiation and their electrochemical behavior was evaluated [122]. The mixture of water and ethylene glycol (30:70 *v*/*v*) was found to be most optimal for producing Pt nanoparticles with a particle size of ~2 nm that are most suitable for electrocatalytic application. The optimal duration of the MW-assisted reduction was found to be 400 s. The MW treatment also affected the structure and durability of the carbon support [123].

Carbon nanofibers were found to be a suitable support for Pt nanoparticles that were prepared from $PtCl_2$ by the microwave method with the time of the treatment of 15–30 s [124]. The potential application of such materials is in the area of polymer electrolyte membrane fuel cells. The average particle size was 4.25 nm. The particles prepared by chemical reduction with 15 s microwave irradiation followed by carbonization at 800 °C had an average size of 0.86 nm and showed high electrochemical performance.

Nitrogen-doped graphene with supported Pt nanoparticles (1.5 nm) prepared by the microwave method (130 °C, 800 W) was used in electrochemical hydrogen production, and

a synergy between Pt nanoparticles and nitrogen-doped graphene was found [125]. Similarly, Pt/rGO materials were prepared under the microwave treatment at 120 °C, 15 min, and 800 W [126]. A higher stability during the accelerated degradation test was revealed in comparison with a commercial Pt/C catalyst. The existence of Pt ions accelerates the reduction of graphite oxide to graphene under microwave irradiation, and, simultaneously, the existence of defect sites on the graphene surface improves the nucleation of Pt during the reduction process [127].

Carbon nanotubes modified by polybenzimidazole were used to support 2–5 nm Pt nanoparticles via reduction with polyol under microwave treatment (60 s at 600 W) for application in proton exchange membrane fuel cells [128]. The developed materials are characterized by the uniform dispersion of Pt nanoparticles and are more efficient in hydrogen oxidation as electrocatalysts in comparison with a commercial Pt/C system.

The authors [129] studied the dynamics of the formation and growth of Pt nanoparticles depending on the microwave parameters. The core state of Pt nanoparticles is polycrystalline with twinned crystal planes if fast microwave heating is used; under these conditions, the stage of nucleation is faster than the stage of crystal growth. But, single crystalline Pt NPs are formed if slow microwave heating is chosen for the synthesis when the rate of crystal growth is higher than the rate of nucleation (Figure 5). While the obtained Pt NPs are supported on hydroxyapatite, a high-performance catalyst for the elimination of volatile organic compounds can be derived.



Fast MW heating:





Silica can also be considered a good high-porosity carrier for metal nanoparticles; so, Pt nanoparticles were supported by reducing Pt precursors under microwave irradiation [130]. Microwave synthesis was also used to produce platinum nanoparticles on mesoporous silica to be used further as dual-modality contrast agents in computer tomography and near-infrared optical imaging [131]. The mesoporous silica particles with an average particle diameter of ~50 nm were covered with ~3 nm diameter naked Pt nanoparticles to enhance the contrast.

More impressive examples of the microwave synthesis of Pt nanoparticles and their application are presented elsewhere [117,132–143].

2.5. Other Noble Metal Nanoparticles

Comparison of the conventional and microwave heating methods in the synthesis of Rh nanoparticles via reduction of RhCl₃ with ethyleneglycol has shown that under the MW regime, larger Rh particles are formed (4.5–12.1 nm vs. 3.8–10.9 nm for the conventional mode of operation) [95]. The materials were tested in the hydrogenation of cyclohexene, which is known to be a structure-sensitive reaction. Therefore, the activity of the MW-assisted material was about two times higher compared to the conventional materials as a result of the formation of specific crystal planes. A comparison of Rh nanoparticles prepared by solvent reduction using microwave and conventional heating has shown that the microwave-processed catalysts exhibited a two-fold increase in activity for cyclohexene hydrogenation, compared to those prepared by conventional heating. It was found that microwave-prepared catalysts contained predominantly tetrahedral particles of 5–7 nm diameter, while the conventionally prepared catalysts displayed particles of varying morphology and size.

Pradhan et al. [144] produced by the microwave method 1D Rh nanostructures with CTAB as a reductant and a template. On the contrary, the conventional hydrothermal process gave spherical Rh(0) nanoparticles.

As in the case of Pt nanoparticles, the use of ionic liquids demonstrated some benefits compared to organic solvents because of the excellent ability to absorb microwave energy in the synthesis of Rh nanoparticles supported on carbon nanotubes [112].

Ru/graphene and Rh/graphene nanomaterials were synthesized by Marquardt et al. using ionic liquids as media for the reduction of metal precursors [145].

Fine Ru nanoparticles (2.57 nm) with a uniform distribution in the carbon matrix were prepared using solid-state microwave heating of the supported RuCl₃ precursor (contrary to the liquid-phase synthesis of Ru nanoparticles). Significant improvement in the electrocatalytic performance as the cathode in a Li–O₂ battery was demonstrated [81]. The temperature under microwave activation was raised to 1270 K within 6 s.

Other examples of the MW-assisted synthesis of Rh and Ru nanoparticles by using the polyol method are reported [49,146–148]. Rh, Ir, and Ru nanoparticles were also prepared under microwave heating by other authors [120,149–151].

3. Non-Noble Metal Nanoparticles

Nanoparticles of non-noble metals usually are much less stable to coalescence, aggregation, and effect of oxygen than nanoparticles of noble metals, and, thus, they require better stabilization. Spherical copper nanoparticles with an average size of 40–50 nm were synthesized under microwave radiation (20 min, 500 W) [52]. The Cu/graphite paste nanocomposite electrodes were used for the sensing of hydrogen peroxide.

Valodkar et al. synthesized Cu nanoparticles by a microwave-assisted process in water with ascorbic acid as a reductant; starch was used to stabilize the nanoparticles [152].

Cu nanowires with a diameter of about 50 nm and lengths over 10 μ m were prepared under microwave operation from CuCl₂, ascorbic acid, and hexadecyl amine in water (120 °C, 2 h) [153]. The aspect ratio was about 300. Other examples of the MW syntheses of copper nanostructures without using any protecting and stabilizing agents are reported by other authors [154,155]. Many more papers have been published on the microwave-assisted polyol method of the synthesis of Cu [155–158], Fe [159], Co [160], and Ni [161–167].

Ni nanoparticles with a size ranging from 30 to 100 nm were prepared in ethylene glycol with polyvinylpyrrolidone and dodecylamine as a protecting agent, with the metal particles forming chain-like structures [163].

Except for Cu and Ni, Co, Sb, Bi, Te, and Si have also been prepared, though the two latter do not fall within the scope of our review (they are not metals) and will not be discussed in detail [168,169]. Sb nanostructures with the morphology of dendrites formed by nanoflakes of 20–40 nm were made in water from antimony sodium tartrate and Zn powder (as a reducing agent) by microwave heating at 280 W for 30 min [170]. Te nanowires or nanotubes or 1D structures were synthesized from Na₂TeO₃ or H₂TeO₃ in water by the microwave procedure (150 °C, 15 min) [171–173].

It is noteworthy that the use of ionic liquids as solvents or templates in the microwaveassisted synthesis of metal nanoparticles provided certain advantages (smaller particle size and more uniform distribution). Thus, Co, Mn, Cr, Mo, W, Re, Ru, Os, Rh, and Ir nanoparticles were synthesized under microwave heating [174] by decomposing metal carbonyls in the functionalized ionic liquid 1-methyl-3-(3-carboxyethyl)-imidazolium tetrafluoroborate [EmimCO₂H][BF₄]. For instance, the sizes of the Co and Mn nanoparticles were 1.6 and 4.3 nm, which remained stable for over 6 months. Volmer et al. reported the short (3 min) and energy-saving (10 W) microwave synthesis [175]. A comparison of the microwave method with UV irradiation (1000 W, 15 min) or thermal decomposition (180–250 °C, 6–12 h) of metal carbonyls in the ionic liquid showed that the nanoparticles produced in the microwave regime were smaller (<5 nm) and more uniform than those prepared by the reference methods.

Ionic liquids have also been successfully used in the synthesis of supported non-noble metal nanoparticles, for instance, on a carbon carrier: Cu/C, Ni/C [176].

4. Bi- and Trimetallic Nanoparticles

Mono- and bimetallic particles containing Ag and Rh (5 wt.%, Ag-Rh ratios 1:1, 1:4, and 4:1) were prepared using a microwave-assisted polyol method [177]. AgNO₃ and RhCl₃ were used as precursors, and poly(N-vinylpyrrolidone) served as a stabilizing agent. The duration of the microwave treatment was 1 min. Homogeneous nucleation occurred under the MW radiation with a reduction of the crystallization time. So, a narrow distribution of nanoparticles was obtained. Ag and Rh nanoparticles were supported onto activated carbon or Y_2O_3 . The particle sizes of Ag, Rh, and Ag-Rh nanoparticles were 20–40 nm, 15–20 nm, and <10 nm, respectively. The materials were tested in H₂ evolution using liquid organic hydrogen carriers.

Another example of Ag-containing bimetallic Ag-Ir nanoparticles is described by Guo et al. [178]. Ag-Ir alloys are unknown; therefore, the formation of such alloys in the supported systems presents interest. AgNO₃ and IrCl₃ are used as precursors. The MW-assisted synthesis was performed in ethylene glycol-containing poly(vinylpyrrolidone) at 197 °C (30 min). The Ag-Ir particle size is small (2.5–5.5 nm) and the distribution is narrow. The strength of H-binding on the Ag-Ir alloy particles is much weaker than that on pure Ir particles, which provides activity in hydrogenation processes. The material's activity in cyclohexene hydrogenation increases with increasing Ir percentage in the mixture of the metals. Particles rich in Ag supported on amorphous Co₃O₄ exhibit higher activity in crotonaldehyde hydrogenation with the formation of crotyl alcohol.

Similarly, it became possible to make classically immiscible rhodium–silver and rhodium–gold alloy nanoparticles, which turned out to be very efficient as hydrogenation catalysts [179].

Gold–silver alloy particles were prepared using microwave irradiation (1800 W, 120 °C, 30 s) by the deposition of silver onto gold nanoclusters as seeds [180]. The size of Ag-Au NPs increased with increasing the Ag content in the alloy. The nanoparticles were reduced

and stabilized by a glucan (polysaccharide) and exhibited good antifungal activity and low cytotoxicity.

Enhanced electrocatalytic activity for the oxidation of formic acid was also reported for microwave-synthesized Pt-Au nanoparticles [181]. Similarly, Pd@Pt core–shell nanostructures with the controllable composition synthesized by a microwave method demonstrated enhanced electrocatalytic activity toward oxygen reduction and methanol oxidation [182]. Song et al. reported on the fast synthesis of carbon-supported platinum-copper nanoparticles with enhanced electrocatalytic activity via microwave-assisted heating [183].

Valodkar et al. succeeded in preparing Ag-Cu nanoparticles by a microwave-assisted process in an aqueous medium with ascorbic acid as a reductant, with starch being used to stabilize the nanoparticles [152].

Although the further two examples may be included in the section related to silver nanoparticles synthesis, we believe it would be better considered as a bimetallic system; a successive attempt at the use of Pt seeds for the microwave preparation of Ag nanoparticles was made by the polyol method using PVP [30]. Silver nanostructures of different shapes were prepared by the reduction of AgNO₃ with ethylene glycol: one-dimensional (1D) nanorods and nanowires, 2D nanosheets and nanoplates, and 3D spherical and cubic nanoparticles. The synthesis takes just a few minutes, while the morphology and size of silver nanoparticles were governed by the concentrations of Pt seeds and the duration of the microwave treatment. The 1D nanorods and nanowires and the 2D nanosheets are formed when a medium Pt concentration and low PVP concentrations are used, while a low Pt concentration or a high PVP concentration of nanorods, nanowires, and cubic nanoparticles with the synthesis time over 3 min shows that adsorption of PVP on the {100} facets of silver governs the whole process. The formation of nanosheets at a short heating time pointed also to the PVP adsorption on {111} facets.

Similarly, Liu et al. detected the formation of Ag nanorods in aqueous solutions in the presence of Au seeds while using sodium citrate as a reducing agent [184]. By varying the MW-heating time, the authors succeeded in changing the aspect ratio of such Au core–Ag shell nanorods, denoted as Au@Ag.

Pd3Ag nanoparticles were prepared on a carbon support by the microwave method (240 W, 6 min) using extracted polysaccharide (*Lilium brownie*) and applied as a sensing material for glucose detection [185].

One of the first reported syntheses of bimetallic core–shell particles by Harpness and Gedanken [186] was focused on gold/palladium nanoparticles prepared under microwave conditions by the simultaneous reduction of Au^{3+} and Pd^{2+} ions in ethylene glycol. Although the reduction potentials for these two metals are very close, the authors succeeded in separating the stages of gold reduction and palladium reduction, though the overall MW-synthesis time was just a few minutes; first, Au was reduced to produce the core of about 8 nm, and, then, Pd reduction took place to form the shell with Au^0 acting as a nucleation center for the growth of Pd to produce the ~20 nm bimetallic nanoparticle.

Bimetallic core–shell particles with palladium included both in the core and in the shell were prepared under microwave irradiation (15 min) [187]. The core–shell nanoparticles with a size of 10–20 nm demonstrated good activity in the direct synthesis of hydrogen peroxide from hydrogen and oxygen; a synergistic effect was found upon the addition of Au to Pd. The activity of the MW samples in hydrogen peroxide synthesis was about twice as high compared to the conventional samples prepared by sol immobilization. It should be noted that the sol-immobilization method usually provides nanoparticles of a smaller size (2–4 nm), but capping agents must be used in this method, unlike the microwave technique.

MW-assisted preparation of 3–4 nm Pd-Pt nanoparticles representing a Pd core with further coating with a Pt shell on a carbon support by the polyol procedure was performed in a continuous mode using a flow-type single-mode microwave reactor [98].

Pd@Pt core–shell nanoparticles were synthesized under microwave heating in water using K₂PtCl₄, PdCl₂, and CTAB as precursors at pH 9 (200 W, 3 min), with the morphology of the core–shell particles being dependent on the molar ratio of Pt to Pd precursors [182].

Mixed Pt/Pd and Pt/Rh bimetallic nanoparticles were derived by microwave-assisted synthesis in polymer (PVP) solutions by the polyol reduction method [188]. H₂PtCl₆, PdCl₂, and RhCl₃ were used as precursors. Both multimode and single-mode microwave irradiation were tested under batch-wise and continuous-flow conditions with irradiation for about 2–3 min. The highest dispersion of colloidal bimetallic nanoparticles with sizes of 6–9 nm for Pt/Pd and about 3–5 nm for Pt/Rh nanoparticles was achieved when single-mode microwave irradiation and continuous-flow conditions were used. A "cluster-in-cluster" structure of the bimetallic nanoparticles was revealed by EXAFS; i.e., some segregation of the two metals takes place (Figure 6). Both single atoms of one metal in another metal or specific cluster shapes (hexagonal or more complicated) are formed under the effect of microwave heating.





Patel et al. [189] prepared Pt/Ag and Pd/Ag bimetallic nanoparticles by the MW-polyol method in the presence of PVP and found a narrow particle-size distribution.

Bimetallic Pt–Ru nanoparticles placed onto carbon nanotubes were successfully prepared by the microwave-assisted reduction of precursors in the appropriate ionic liquid that is characterized by the strong absorption of microwave radiation [190].

Other bimetallic compositions (Pd-Au and Pt-Au) of nanoparticles and their MW synthesis and characterization have been described in other publications [191,192].

Bensebaa et al. [193] applied MW treatment to synthesize Pt/Ru bimetallic nanoparticles in ethylene glycol with PVP and revealed that the variation of the parameters of the microwave irradiation is capable of changing the surface composition and structure of these particular nanoparticles. MW synthesis of Pt-Ru nanoparticles with sizes in the range of 2–6 nm on carbon black and carbon nanotubes was performed. The obtained PtRu/C electrocatalysts were much more efficient in methanol oxidation than the conventionally prepared Pt/C systems. Other carbon-supported bimetallic nanoparticles PtRu/C were reported [120].

Li et al. prepared Pd-Ni bimetallic nanoparticles (10–15 nm) onto reduced graphene oxide under microwave irradiation with ethylene glycol used as a reducing agent [101].

Carbon-supported platinum–copper nanoparticles were prepared by microwave reduction using the polyol procedure [183]. Pt-Ni nanoparticles supported on a carbon carrier were produced under microwave conditions. They were also efficient in the electrochemical methanol oxidation reaction, where they were applied as electrocatalysts [194,195]. The microwave parameters used were as follows: temperature = 200 °C, power = 750 W, and time = 30 min. The 50:50 Pt:Ni composition demonstrated the best performance in this process. The behavior of the electrocatalysts was explained by the incorporation of Ni atoms into the Pt lattice and, as a result, by the increase of the electrocatalytically active

iclos domonstrated enhanced electrocatalytic activity and

Pt surface area. The Pt-Ni particles demonstrated enhanced electrocatalytic activity and stability in ethanol oxidation reaction and methanol oxidation reaction compared to Pt/C.

One more research group reported the microwave synthesis of 13 nm Pt-Cu nanoparticles, which were then supported on SiO₂, with ethylene glycol as a solvent and polyvinylpyrrolidone as a capping agent (1000 W, 200 °C, 30 bar, 13 min) [114]. PtCu NPs were synthesized using a conventional microwave oven within 2 min but with poor size distribution [196]. The synthesis of 5 nm particles required a 12 h premixing of the reaction mixture before the short microwave treatment [197]. Also, supported PtCu alloy nanoparticles of 3.7 nm were prepared in 10 min by the microwave-assisted polyol method [198].

Pt-Ni nanoparticles on carbon supports are among the most carefully studied systems because of their high efficiency in fuel-cell applications as electrocatalysts. Carbon-supported Pt-Ni alloy polyhedral (octahedral and tetrahedral) nanoparticles with a size of 7–9 nm were synthesized under microwave conditions [199]. They were applied to produce electrodes for the oxygen-reduction reaction in proton-exchange membrane fuel cells.

Microwave-enhanced surface diffusion during solvothermal synthesis of Pt–Ni octahedral nanoparticles was used to govern the process of segregation of the two metals in the particles [200]. The Pt-Ni nanoparticles were prepared by the microwave method at 120 to 200 °C. Microwave irradiation was found to significantly promote the atomic diffusion, especially surface diffusion, in the Pt-Ni octahedra, which resulted in Pt segregation on the {111} facets leading to the Pt-rich surface and Ni-rich bulk with preservation of the octahedral morphology of the nanoparticles, which is not the case of the comparative solvothermal method (Figure 7). The enrichment of the nanoparticle surface in platinum is clearly seen in this figure. The segregated particles turned out to outperform nonsegregated ones as catalysts in the electrochemical oxygen-reduction reaction. The reduction of the reaction time from 42 to 6 h was reported for the microwave-assisted solvothermal process compared to the purely solvothermal process. The results obtained demonstrate that the surface diffusion process, which usually under a purely thermal mode of operation occurred above 300–400 °C under conditions of vacuum/gas atmosphere thermal annealing, can also be achieved in the microwave-assisted liquid-phase "annealing" and at a much lower temperature (190 $^{\circ}$ C). The authors believe that microwave energy can be directly absorbed by surface atoms, and this can provide a driving force to overcome the diffusion energy barriers.

The carbon support coated with amorphous nickel via electroless deposition was used as a carrier for 4–5 nm Pt nanoparticles supported via reduction with ethylene glycol after depositing the nickel coating under microwave irradiation that was further applied to catalyze the methanol oxidation reaction in direct methanol fuel cells [201]. Although no bimetallic particles are formed in this case, there is a direct-contact interaction between Pt and Ni. The comparison with a sample of carbon nanotubes with supported Pt showed the higher efficiency of the Ni-modified catalyst in methanol oxidation, which was attributed to a more uniform distribution of Pt nanoparticles and the synergistic effect between Pt and Ni.

 Pt_3Co alloy nanoparticles (3.4 nm) on a carbon support were successfully prepared by the microwave method and used in the electrochemical oxygen-reduction reaction [202]. The samples demonstrated a significant improvement in the electrochemical parameters compared to the comparative Pt/C electrocatalyst.

A solvothermal microwave process was explored for the production of Pt–Co nanowires [203] and nanoparticles [204,205] as active electrocatalysts.

Ruthenium is not quite often used in combination with other metals because, first, it has not found such a wide application as platinum, palladium, rhodium, and iridium, and, second, it is hardly miscible with other metals to form bimetallic alloys. Therefore, the study by Du et al. [206] evokes interest as an example of the synthesis of immiscible alloy nanoparticles based on ruthenium and iron. Moreover, such nanoparticles revealed improved activity in the water–gas shift reaction to produce hydrogen. Similarly, immiscible metal alloy particles were produced from iron and platinum or palladium [207–209].





Köhler et al. [210] prepared Bi–Rh pseudohexagonal nanoplates using bismuth acetate and rhodium acetate dimer in EG by a microwave-assisted polyol process at 240 °C for 1 h. FeNi₃ nanochains were prepared by reducing iron (III) acetylacetonate and nickel (II) acetylacetonate with hydrazine in EG under microwave irradiation at 160 °C for 5 min [211]. $Co_{0.8}Ni_{0.2}$ nanostructures were prepared in 1,2-propanediol by a microwave-assisted polyol process, and it was found that rapid heating led to the formation of nanowires, while slow heating resulted in nanourchins; the aspect ratio of $Co_{0.8}Ni_{0.2}$ nanowires decreased with increasing temperature [212].

Two metals of this group (Fe and Ni) have been assembled under microwave activation into nanochains that demonstrated enhanced magnetic susceptibility [211]. It is noteworthy that no template was used to produce such nanochain shapes.

Finally, to close the iron triad, a series of bimetallic particles containing iron in combination with platinum and palladium was prepared and their genesis under variable microwave conditions was studied [207].

One more example is related to the production of Ni-Co bimetallic composites on a carbon matrix [213]. The particles demonstrated a tremella-like shape and were found to exhibit high efficiency as microwave-absorbing materials in a very wide frequency range, from 2 to 40 GHz. Similar compositions of bimetallic nanoparticles have been produced by the microwave-assisted polyol synthetic approach [212].

A very strange composition was chosen in the preparation of Bi-Rh nanoplates that turned out to exhibit activity in the selective hydrogenation of the triple bond in acetylenic molecules [210].

Trimetallic Au-Pt-Cu nanoparticles were prepared from bimetallic nanoislands and their activity as biocidal agents was compared to that of monometallic Au and bimetallic Au-Pt nanoparticles [214]. The cyclic mode was chosen for the synthesis (20 s on, 10 s off, for a total of 3 min at 360 W). The increase in the number of components results in a smaller particle size and the structure contains more defects. So, the trimetallic particles turn out to be more effective against both the gram-negative and gram-positive bacteria.

The microwave method was also used for the preparation of carbon-supported Pt-Sn-Rh alloy nanoparticles used in electrocatalysts in fuel cells [215]. The microwave-heating mode was as follows: pulses of 10 s on/off at 800 W were used to heat the reactant mixture

for 200 s until the temperature reached 408 K, and the mixture was kept at this temperature for about 480 s. H_2PtCl_6 , $SnCl_2$ and $RhCl_3$ were used as precursors to be reduced with ethylene glycol. The nanoparticles were extremely small—2 nm. They were tested in methanol and ethanol oxidation reactions and demonstrated a 3–6 times higher activity in the title reactions and higher stability in comparison to a commercial Pt/C electrocatalyst.

Core-shell nanoparticles composed of two different metals or one metal and one alloy or even two different alloy compositions are expected to exhibit unique properties in diverse applications, including catalysis, sensing, etc. However, thermal methods of preparation of bimetallic particles of this kind suffer from the instability of the core-shell structures that may be easily transformed into a different segregated morphology or into a more homogeneous particle. Therefore, the application of microwave techniques attracted attention as a means (1) to realize milder conditions of synthesis of core–shell structures, and (2) to provide conditions for the stabilization of core-shell nanoparticles because of the fast cooling of the entire system, which prevents undesirable thermodynamic equilibria and migration/transformation of the just formed nanoparticles that occur in the reactor with thermal operation after the heating is switched off. Microwave-assisted synthesis not only allows the formation of diverse core-shell nanoparticles but also makes it possible to control the growth of nanoparticles of different geometries/shapes/morphologies [216]. The authors focused on Au-Ag nanoparticles by using a strategy of creating a bilayer organic barrier around the core using citrate and ascorbic acid as capping agents providing a sharp boundary between the core and the shell. Spherical and triangular seed cores were used. First, gold nanoparticles were prepared at the microwave power of 100 W for 60 s. At a higher MW power (200 W), gold nanoprisms are formed. After that, a silver shell was deposited at 300 W and 60 s. With a shorter time and at a lower microwave power, the silver precursor was not reduced. The use of ethanol as a solvent in the synthesis of the gold seed solutions rapidly increases the temperature of the reaction because of the higher tangent of dielectric losses for ethanol (0.941) compared to water (0.123).

One more important type of core–shell bimetallic nanoparticles is reported. Its importance is related to the non-noble nature of the constituting metals, the nanoparticles contain a Cu core and a Ni shell, the uniform particle-size distribution with a maximum at 12 nm was found upon MW irradiation of nickel(II), and copper(II) formate complexes dissolved in 1-octanol at 190 °C for 10 min. Since the reduction potentials are different for nickel(II) and copper(II) formate complexes, and exhibit different temperature dependences, first the Cu core seeds were formed at 160 °C; then, the Ni shell was deposited at 190 °C. The fast kinetics prevents the conversion of the Cu@Ni core–shell particles into the CuNi alloy [217].

It is very difficult, if possible at all, to prepare a single-phase alloy containing two, three, or more metals in its composition. Such compositions provide unique advantages, like economy of critical and noble metals, possibility to tune the electronic properties of each metal in the composition, and sometimes enhance (in a nonpredictable way) catalytic activity or other performance characteristics. In this concern, the paper by Wu et al. [218] seems very attractive because the results obtained demonstrate the efficiency of the unique procedure based on the microwave heating/cooling of multimetal alloy particles in a MW-nonabsorbing medium, like oil. So far, attempts to prepare uniformly mixed multiple metals to form alloys with single crystalline phases failed. The reason is the thermodynamic immiscibility of different metals resulting most often in segregation of the metals. By using the developed rationale, the authors succeeded in the synthesis of alloy particles of immiscible metals—Au-Pt-Pd-Cu.

There are more illustrative examples of the synthesis of bimetallic nanoparticles, including immiscible metals [179,191,196,198,219–223].

One more recent example of the microwave-assisted synthesis of polymetallic nanoparticles is quite impressive since the authors succeeded in preparing nanoparticles containing five (!) metals—Ir, Fe, Co, Ni, and Cu, with none of the four latter being miscible with iridium [224]. Such nanoparticles are interesting as potential catalysts for electrochemical energy storage processes, including oxygen evolution reactions. The IrFeCoNiCu alloy nanoparticles on carbon paper were prepared by microwave-assisted shock synthesis. Such high-entropy alloy nanoparticles demonstrate superior activity with an overpotential of \sim 302 mV at 10 mA cm⁻² and enhanced stability for 12 h in comparison with monometallic Ir. The active Ir-rich shell was revealed on the surface of IrFeCoNiCu alloy nanoparticles right after electrochemical activation due to the dissolution of a part of the 3d metals. The core of the particles preserved the homogeneous alloy structure without phase separation or metal segregation.

5. Composites Based on Metal Nanoparticles

Diverse composites find applications in energy harvesting and storage, sensors, catalysis, biomedicine, etc. The border between nanomaterials and nanocomposites is not sharp, and quite a few, if not many, examples given above may be considered nanocomposites. Anyway, we decided to include a separate section devoted to the microwave-assisted synthesis of nanocomposites containing metal nanoparticles.

Wang et al. prepared silver nanoparticles on reduced graphene oxide (AgNP/rGO) composites via the combination of phytosynthesis, continuous flow conditions, and microwave irradiation [225]. The authors used oriental persimmon (*Diospyros kaki Thunb.*) extracts as reducing and capping agents. The conditions of the synthesis were optimized, and the morphologies of the prepared materials were determined. Predominantly spherical AgNPs were formed with a uniform particle distribution on rGO sheets. The reduction of p-nitrophenol with NaBH₄ on AgNP/rGO catalysts was studied. The complete degradation was found to take just 12 min, with the composite demonstrating a much better activity than the naked AgNPs and rGO. Comparison with the conventional chemical method of preparation shows significant benefits of the developed microwave method, which is facile, fast, cost-efficient, and environmentally friendly.

Microwave-assisted preparation of rod-like and hexagonal plate-like AgNP@Ni-BTC composites was proposed [226]. The important application of such composites is related to sensing glucose. A new composite material, silver nanoparticle@Ni-BTC (AgNP@Ni-BTC), based on a metal–organic framework containing benzenetricarboxylate linkers was designed by using a one-pot methodology under microwave conditions at 130 °C for 1 h at 200 W. The 4%AgNP@Ni-BTC on a carbon paste electrode demonstrated better sensor sensitivity, reproducibility, and stability compared to Ni-BTC, the detection limit of 14.73 μ M was achieved, and the sensitivity of 6584.89 μ A mM⁻¹ cm⁻² with a linear range of concentrations 10–1250 μ M was found.

Layered composite materials based on molybdenum chalcogenides have attracted attention recently as carriers for metal nanoparticles and their application in electrochemical processes. Ag/MoS₂ nanosheets were prepared under microwave conditions and used for efficient electrocatalytic oxygen reduction [227]. The composite material prepared is characterized by the increased specific surface area; as a result, the Ag/MoS₂ nanohybrid demonstrates enhanced activity in the oxygen reduction reaction and about the same stability when compared with MoS₂ and Pt/C. Due to the heterojunction between Ag and MoS₂, the Ag/MoS₂ nanohybrid composite shows very good stability after 2000 cycles.

A composite material with a quite complicated composition based on a Pd-decorated rGO-MoSe₂ 2D hybrid network was developed for application in direct methanol fuel cells [228]. The new composite is capable of decreasing the deactivation of electrocatalysts due to CO adsorption in the course of methanol oxidation. A green microwave-assisted technique was applied for the preparation of hybrid Pd-MoSe₂-rGO and Pd-rGO nanocomposites. The materials were studied by XRD, FESEM, and TEM methods to reveal their morphology, XPS to determine the state of the elements present in the composites, and Raman spectroscopy. The electrocatalytic activity was determined by cyclic voltammetry, chronoamperometry, and CO stripping methods. The value of the electrocative surface area of the synthesized electrocatalyst Pd-MoSe₂-rGO (51.81 m² g⁻¹ Pd) was about 3.4 times higher than that of Pd-rGO (15.30 m² g⁻¹ Pd). The new catalyst with a 3D cross-linked hybrid network had a better distribution of metal nanoparticles and manifested four times

higher electrocatalytic activity (1935 mA mg⁻¹ Pd) in methanol oxidation compared to Pd-rGO (546 mA mg⁻¹ Pd). Using a fixed potential value, it has been shown that the electrocatalytic activity of Pd-MoSe₂-rGO exceeded 50 times that of Pd-rGO (1 h). The introduction of MoSe₂ in the composition of the electrocatalyst increases the tolerance towards CO poisoning.

Another composite that found application in electrochemical energy-storage devices was based on bismuth nanoparticles on a carbon nanotube [229]. This composite prepared by microwave synthesis demonstrated high capacity, long cycle life, and fast kinetics as a material of anodes in sodium-ion capacitors. The microwave irradiation method was successfully used for the in situ growth of nanosized Bi uniformly grafted on the surface of carbon nanotubes. The electrode film prepared from this composite effectively retards the pulverization of Bi nanoparticles, providing fast sodium-storage kinetics, high-power performance (278.1 mAh g⁻¹–30 A g⁻¹), and high capacity (94%) for 3500 cycles. The coin-cell morphology of the anode afforded an enhanced energy density of 128.5 Wh kg⁻¹ and power density of 12.3 kW kg⁻¹. On top of that, the system possessed a high volumetric energy density of 21 mWh cm⁻³ with excellent cycling stability (90%) in 1500 cycles.

Microwave arc discharge was used for the fast synthesis of carbon-coated metal nanoparticles (M@CNPs) [230]. The influence of the arc-discharge intensity on the morphology of the composite was studied while varying the microwave power. The nature of the organic groups (i.e., cyclopentadienyl or acetylacetonate) and the metal catalyst affects the morphology of the composite particles. Ferrocene, iron acetylacetonate, nickelocene, and nickel acetylacetonate were used as precursors. It has been shown that moderate microwave power cannot destroy the precursor to form a carbon shell. Cyclopentadienyl groups were found to encapsulate the metal core to form graphitized carbon layers due to the strong coordination bond between cyclopentadienyl and metal. In the case of acetylacetonate groups, the open-loop structure of the organic groups is formed with Ni being more active in the formation of carbon nanotubes compared to Fe.

6. Comparison of the Microwave Method of Metal Nanoparticles Synthesis with Conventional Heating

In spite of a great deal of data available in the literature on the preparation of metal nanoparticles under microwave heating, there are almost no publications focused on rigorous analysis and comparison of the energy expenses for the synthesis of such nanomaterials. There are many papers where the authors claim energy savings for the microwave mode of operation and, qualitatively, the results are supportive of these claims; for instance, if we compare the duration of the synthesis: usually the time required for the microwave synthesis extends from 10 s to 20 min, although there are examples of the long synthesis (up to 4 h), while the conventional heating procedure typically takes a few hours. Comparison of the applied power is also a problem. Indeed, in the case of microwave heating, the efficiency of the conversion of electric energy into microwaves is about 50%. Then, the real efficiency of the microwave units that are used for the synthesis (whether these are home appliances or specially designed MW setups like Discover or similar units) varies from about 20% (home appliances) to ~70–90% (special monomode or resonator units). Also, resonator units with an efficiency close to 100% are available. On the other hand, the efficiency of conventional heating (hot plate, oil bath, or others) is also quite low taking into account heating of the walls, environment, etc., and ranges from 20 to 50%. So, if we accept the same efficiency of both methods of heating (roughly it is true), then we can compare only the duration of the treatment performed to reach about the same result (size and shape of the nanoparticles). We included a few examples from the literature, especially the papers where such data as duration, temperature, and power are available for both methods, in Table 2. It should be noted again that the last column representing an estimate of the gain in energy expenses for the microwave method vs. conventional heating is our approximate calculation result. Anyway, even if we accept a 50% error, the energy gain is obvious for the use of the microwave technique.

Table of Nanoparticles	Microwave (MW) Heating Conditions		Conventional Heating (CH) Conditions		Nister	Relative Gain in the Energy Consumption
	Power/Temperature	Synthesis Time	Temperature	Synthesis Time	- Notes	of MW vs. CH (Rough Estimate)
Ag nanoclusters [31]	200 W	60–90 s	No information	120 min	The quantum yield for MW-synthesized NPs is 6%, while the quantum yield for NPs prepared by thermal heating is 1%	80–120
Ag nanorods [46]	-	1–3 min	-	50 min	Ag nanosheets are formed only at a very short MW treatment, and they are not produced at all under conventional heating	15–50
Au nanoparticles [76]	400 W	10 min	85 °C	6 h	The size of MW synthesized NPs was 22 nm, while the size of CH NPs was 33 nm	36
Pd nanoparticles [96]	300 W, 60 °C	4 h	60 °C	8 h	Twice faster process under MW conditions	2
Pt nanoparticles [129]	160 °C	200 s	160 °C	2000 s	The size of MW synthesized NPs was 3.6 nm, while the size of CH (oil-bath) NPs was 10.1 nm	10
Ir nanoparticles on antimony tin oxide [151]	170 °C	30 s	170 °C	2.5 h	Obtained NPs characterized by a similar size, surface area, and morphology	300
Cu nanofluids [156]	350 W	5 min	120 °C	1 h	The size of MW-synthesized NPs was <20 nm, while the size of CH NPs was 30–80 nm. MW-obtained nanofluids were stable for 3 weeks, while CH-synthesized ones were only for 1 week	12
Metal nanoparticles [175]	Just 10 W	3 min	UV irradiation—1000 W, CH—180–250 °C	UV irradiation—15 min, CH—6–12 h	NPs produced in the microwave regime were smaller (<5 nm) and more uniform than those prepared by reference methods.	A gain of 500 compared to UV irradiation and 360–720 compared to CH
Pt-Ni nanoparticles [195]	750 W 200 °C	30 min	200 °C	4 h	Electrocatalytic activity of MW synthesized NPs is higher than CH-synthesized NPs	8
Fe-Pt nanoparticles [209]	800 W 250 °C	1–2.5 h	290 °C	3 h	Coercivity of NPs obtained by the MW method was almost 10 times higher than that of CH-obtained NPs	<3

Table 2. Comparison of the microwave method of synthesis of metal nanoparticles with conventional (oil bath or UV irradiation) methods.

7. Future Perspectives

The future perspective of the use of microwave irradiation in the synthesis of metal nanoparticles, in the authors' opinion, looks bright and may be related to the development in the following directions:

- Continuous mode of operation (flow-type reactors for the synthesis of metal nanoparticles under microwave heating, suitable for unsupported nanoparticles only);
- In-depth exploration of specific or nonthermal effects in the course of metal nanoparticle preparation;
- Optimization of the frequency range beyond 2.45 GHz; there are some grounds to believe that higher frequencies are much more efficient in the microwave-assisted syntheses of nanomaterials, but this is a subject for a separate review;
- More inventive use of solvents with a strong dependence on the tangent of losses on temperature, like ionic liquids and deep eutectics solvents;
- More attention should be paid to the early stages of the synthesis (seconds to a minute); in many publications, it looks like the authors missed the very early stages and the consecutive transformations of the shapes from dots (spheres) to more complicated morphologies;
- Synthesis of metal carbides (underexplored so far);
- More inventive synthesis of hybrid nanomaterials;
- Synthesis of quasicrystals;
- Synthesis of immiscible phases (like immiscible metals);
- Heuristic syntheses of metastable phases (novel catalytically active materials);
- Original procedures of preparation of defect materials with control of the defect nature and concentration;
- Development of new MW-absorbing materials and compounds, including stimuliresponsive materials, in particular, materials and compounds with high sensitivity to MW frequency;
- Use of greener solvent mixtures;
- MW syntheses under elevated pressures (up to the supercritical region);
- Wide use of seeding in the synthesis of core-shell and other nanomaterials,
- Combination of the microwave and plasma-assisted preparation,
- MW syntheses combined with other unique methodologies (electrochemical, sonochemical, mechanochemical, photochemical, radiation-induced, and solid-state synthesis).

The most important future trends in the application of microwave irradiation in the synthesis of metal nanoparticles should be focused on scaling up and the development of a continuous process. The success in these two directions will make it possible to launch a mass production of metal nanoparticles for the realization of their wide applications in biomedicine, as bactericides, in catalysis, and other areas of the highest priority. The green syntheses of metal nanoparticles remain to be a focus of future research, and, here, diverse natural compounds and extracts are expected to serve as both reducing and complexing agents.

From the point of view of fundamental knowledge, special interest should be in the development of microwave-assisted methods of synthesis of quasicrystals, hybrid materials, composites, alloys of immiscible metals, and metastable phases. Such materials may find, in the future, more interesting and challenging applications compared to traditional systems.

Author Contributions: L.K.: conceptualization, supervision, project administration, funding acquisition, writing—original draft preparation, writing—review and editing; K.V.: validation, formal analysis, data curation, writing—original draft preparation, writing—review and editing. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by Russian Science Foundation, grant number 23-73-30007. L. M. Kustov thanks the «Priority-2030» academic leadership selectivity program, project number K7-2022-062.

Conflicts of Interest: The authors declare no conflict of interest.

References

- Ishii, T.K. Industrial Applications of Microwaves. In *Handbook of Microwave Technology*; Academic Press: San Diego, CA, USA, 1995; pp. 277–307.
- de la Hoz, A.; Díaz-Ortiz, À.; Moreno, A. Microwaves in Organic Synthesis. Thermal and Non-Thermal Microwave Effects. *Chem. Soc. Rev.* 2005, 34, 164–178. [CrossRef] [PubMed]
- Rao, K.J.; Vaidhyanathan, B.; Ganguli, M.; Ramakrishnan, P.A. Synthesis of Inorganic Solids Using Microwaves. *Chem. Mater.* 1999, 11, 882–895. [CrossRef]
- Baghbanzadeh, M.; Carbone, L.; Cozzoli, P.D.; Kappe, C.O. Microwave-Assisted Synthesis of Colloidal Inorganic Nanocrystals. Angew. Chem.-Int. Ed. 2011, 50, 11312–11359.
- Tsukahara, Y.; Higashi, A.; Yamauchi, T.; Nakamura, T.; Yasuda, M.; Baba, A.; Wada, Y. In Situ Observation of Nonequilibrium Local Heating as an Origin of Special Effect of Microwave on Chemistry. J. Phys. Chem. C 2010, 114, 8965–8970. [CrossRef]
- Pathak, D.D.; Grover, V. Mechanochemistry: Synthesis That Uses Force. In *Handbook on Synthesis Strategies for Advanced Materials*; Tyagi, A.K., Ningthoujam, R.S., Eds.; Indian Institute of Metals Series; Springer: Singapore, 2021; pp. 657–682.
- Bilecka, I.; Niederberger, M. Microwave Chemistry for Inorganic Nanomaterials Synthesis. Nanoscale 2010, 2, 1358–1374. [CrossRef] [PubMed]
- 8. Zhu, Y.J.; Chen, F. Microwave-Assisted Preparation of Inorganic Nanostructures in Liquid Phase. Chem. Rev. 2014, 114, 6462–6555.
- Tsuji, M.; Hashimoto, M.; Nishizawa, Y.; Kubokawa, M.; Tsuji, T. Microwave-Assisted Synthesis of Metallic Nanostructures in Solution. *Chem.-A Eur. J.* 2005, 11, 440–452. [CrossRef]
- 10. Pentsak, E.O.; Cherepanova, V.A.; Ananikov, V.P. Dynamic Behavior of Metal Nanoparticles in Pd/C and Pt/C Catalytic Systems under Microwave and Conventional Heating. *ACS Appl. Mater. Interfaces* **2017**, *9*, 36723–36732. [CrossRef]
- Tu, W.; Liu, H. Continuous Synthesis of Colloidal Metal Nanoclusters by Microwave Irradiation. *Chem. Mater.* 2000, 12, 564–567. [CrossRef]
- Komarneni, S.; Li, D.; Newalkar, B.; Katsuki, H.; Bhalla, A.S. Microwave–Polyol Process for Pt and Ag Nanoparticles. *Langmuir* 2002, 18, 5959–5962. [CrossRef]
- 13. Yu, W.; Tu, W.; Liu, H. Synthesis of Nanoscale Platinum Colloids by Microwave Dielectric Heating. *Langmuir* **1999**, *15*, 6–9. [CrossRef]
- Anumol, E.A.; Kundu, P.; Deshpande, P.A.; Madras, G.; Ravishankar, N. New Insights into Selective Heterogeneous Nucleation of Metal Nanoparticles on Oxides by Microwave-Assisted Reduction: Rapid Synthesis of High-Activity Supported Catalysts. ACS Nano 2011, 5, 8049–8061. [CrossRef] [PubMed]
- 15. Tu, W.; Liu, H. Rapid Synthesis of Nanoscale Colloidal Metal Clusters by Microwave Irradiation. J. Mater. Chem. 2000, 10, 2207–2211. [CrossRef]
- 16. Kou, J.; Bennett-Stamper, C.; Varma, R.S. Green Synthesis of Noble Nanometals (Au, Pt, Pd) Using Glycerol under Microwave Irradiation Conditions. *ACS Sustain. Chem. Eng.* **2013**, *1*, 810–816. [CrossRef]
- 17. Mallikarjuna, N.N.; Varma, R.S. Microwave-Assisted Shape-Controlled Bulk Synthesis of Noble Nanocrystals and Their Catalytic Properties. *Cryst. Growth Des.* 2007, 7, 686–690. [CrossRef]
- 18. Zhu, J.F.; Zhu, Y.J. Microwave-Assisted One-Step Synthesis of Polyacrylamide-Metal (M = Ag, Pt, Cu) Nanocomposites in Ethylene Glycol. *J. Phys. Chem. B* 2006, *110*, 8593–8597. [CrossRef]
- 19. Harada, M.; Cong, C. Microwave-Assisted Polyol Synthesis of Polymer-Protected Monometallic Nanoparticles Prepared in Batch and Continuous-Flow Processing. *Ind. Eng. Chem. Res.* **2016**, *55*, 5634–5643. [CrossRef]
- 20. Sumi, T.; Dillert, R.; Horikoshi, S. Utilization of the Microwave Electric or Magnetic Field in the Synthesis of Monometallic and Bimetallic Nanoparticles. *RSC Adv.* **2015**, *5*, 14637–14645. [CrossRef]
- Xu, S.; Zhong, G.; Chen, C.; Zhou, M.; Kline, D.J.; Jacob, R.J.; Xie, H.; He, S.; Huang, Z.; Dai, J.; et al. Uniform, Scalable, High-Temperature Microwave Shock for Nanoparticle Synthesis through Defect Engineering. *Matter* 2019, 1, 759–769. [CrossRef]
- Zhao, X.; Zhang, Z.; Wang, L.; Xi, K.; Cao, Q.; Wang, D.; Yang, Y.; Du, Y. Excellent Microwave Absorption Property of Graphene-Coated Fe Nanocomposites. *Sci. Rep.* 2013, *3*, 3421. [CrossRef]
- Qi, X.; Hu, Q.; Xu, J.; Xie, R.; Bai, Z.; Jiang, Y.; Qin, S.; Zhong, W.; Du, Y. Enhanced Microwave Absorption Properties and Mechanism of Core/Shell Structured Magnetic Nanoparticles/Carbon-Based Nanohybrids. *Mater. Sci. Eng. B Solid. State Mater. Adv. Technol.* 2016, 211, 3421. [CrossRef]
- Sharma, V.K.; Yngard, R.A.; Lin, Y. Silver Nanoparticles: Green Synthesis and Their Antimicrobial Activities. Adv. Colloid. Interface Sci. 2009, 145, 83–96. [PubMed]
- Saloga, P.E.J.; Kästner, C.; Thünemann, A.F. High-Speed but Not Magic: Microwave-Assisted Synthesis of Ultra-Small Silver Nanoparticles. *Langmuir* 2018, 34, 147–153. [CrossRef] [PubMed]
- Hu, B.; Wang, S.B.; Wang, K.; Zhang, M.; Yu, S.H. Microwave-Assisted Rapid Facile "Green" Synthesis of Uniform Silver Nanoparticles: Self-Assembly into Multilayered Films and Their Optical Properties. J. Phys. Chem. C 2008, 112, 11169–11174. [CrossRef]
- 27. Singh, A.K.; Raykar, V.S. Microwave Synthesis of Silver Nanofluids with Polyvinylpyrrolidone (PVP) and Their Transport Properties. *Colloid. Polym. Sci.* 2008, 286, 1667–1673. [CrossRef]

- Pal, A.; Shah, S.; Devi, S. Microwave-Assisted Synthesis of Silver Nanoparticles Using Ethanol as a Reducing Agent. *Mater. Chem.* Phys. 2009, 114, 530–532. [CrossRef]
- Jiang, H.; Moon, K.S.; Zhang, Z.; Pothukuchi, S.; Wong, C.P. Variable Frequency Microwave Synthesis of Silver Nanoparticles. J. Nanoparticle Res. 2006, 8, 117–124. [CrossRef]
- Tsuji, M.; Nishizawa, Y.; Matsumoto, K.; Miyamae, N.; Tsuji, T.; Zhang, X. Rapid Synthesis of Silver Nanostructures by Using Microwave-Polyol Method with the Assistance of Pt Seeds and Polyvinylpyrrolidone. *Colloids Surf. A Physicochem. Eng. Asp.* 2007, 293, 185–194. [CrossRef]
- Liu, S.; Lu, F.; Zhu, J.J. Highly Fluorescent Ag Nanoclusters: Microwave-Assisted Green Synthesis and Cr3+ Sensing. *Chem. Commun.* 2011, 47, 2661–2663. [CrossRef]
- Manno, R.; Sebastian, V.; Mallada, R.; Santamaria, J. 110th Anniversary: Nucleation of Ag Nanoparticles in Helical Microfluidic Reactor. Comparison between Microwave and Conventional Heating. *Ind. Eng. Chem. Res.* 2019, 58, 12702–12711. [CrossRef]
- Horikoshi, S.; Sumi, T.; Serpone, N. A Hybrid Microreactor/Microwave High-Pressure Flow System of a Novel Concept Design and Its Application to the Synthesis of Silver Nanoparticles. *Chem. Eng. Process. Process Intensif.* 2013, 73, 59–66. [CrossRef]
- Horikoshi, S.; Abe, H.; Torigoe, K.; Abe, M.; Serpone, N. Access to Small Size Distributions of Nanoparticles by Microwave-Assisted Synthesis. Formation of Ag Nanoparticles in Aqueous Carboxymethylcellulose Solutions in Batch and Continuous-Flow Reactors. *Nanoscale* 2010, 2, 1441–1447. [CrossRef] [PubMed]
- Nishioka, M.; Miyakawa, M.; Kataoka, H.; Koda, H.; Sato, K.; Suzuki, T.M. Continuous Synthesis of Monodispersed Silver Nanoparticles Using a Homogeneous Heating Microwave Reactor System. *Nanoscale* 2011, 3, 2621–2626. [CrossRef] [PubMed]
- 36. Özkar, S.; Finke, R.G. Silver Nanoparticles Synthesized by Microwave Heating: A Kinetic and Mechanistic Re-Analysis and Re-Interpretation. *J. Phys. Chem. C* 2017, 121, 27643–27654. [CrossRef]
- Dzido, G.; Markowski, P.; Małachowska-Jutsz, A.; Prusik, K.; Jarzębski, A.B. Rapid Continuous Microwave-Assisted Synthesis of Silver Nanoparticles to Achieve Very High Productivity and Full Yield: From Mechanistic Study to Optimal Fabrication Strategy. J. Nanoparticle Res. 2015, 17, 27. [CrossRef]
- Tsuji, M.; Matsumoto, K.; Jiang, P.; Matsuo, R.; Tang, X.L.; Kamarudin, K.S.N. Roles of Pt Seeds and Chloride Anions in the Preparation of Silver Nanorods and Nanowires by Microwave-Polyol Method. *Colloids Surf. A Physicochem. Eng. Asp.* 2008, 316, 266–277. [CrossRef]
- 39. Gou, L.; Chipara, M.; Zaleski, J.M. Convenient, Rapid Synthesis of Ag Nanowires. Chem. Mater. 2007, 19, 1755–1760. [CrossRef]
- 40. Pastoriza-Santos, I.; Liz-Marzán, L.M. Synthesis of Silver Nanoprisms in DMF. Nano Lett. 2002, 2, 903–905. [CrossRef]
- He, R.; Qian, X.; Yin, J.; Zhu, Z. Preparation of Polychrome Silver Nanoparticles in Different Solvents. J. Mater. Chem. 2002, 12, 3783–3786. [CrossRef]
- He, R.; Qian, X.; Yin, J.; Zhu, Z. Formation of Silver Dendrites under Microwave Irradiation. *Chem. Phys. Lett.* 2003, 369, 454–458. [CrossRef]
- Yamamoto, T.; Wada, Y.; Sakata, T.; Mori, H.; Goto, M.; Hibino, S.; Yanagida, S. Microwave-Assisted Preparation of Silver Nanoparticles. *Chem. Lett.* 2004, 33, 158–159. [CrossRef]
- 44. Tsuji, M.; Hashimoto, M.; Nishizawa, Y.; Tsuji, T. Synthesis of Gold Nanorods and Nanowires by a Microwave-Polyol Method. *Mater. Lett.* **2004**, *58*, 2326–2330. [CrossRef]
- 45. Yamamoto, T.; Yin, H.; Wada, Y.; Kitamura, T.; Sakata, T.; Mori, H.; Yanagida, S. Morphology-Control in Microwave-Assisted Synthesis of Silver Particles in Aqueous Solutions. *Bull. Chem. Soc. Jpn.* **2004**, *77*, 757–761. [CrossRef]
- 46. Tsuji, M.; Nishizawa, Y.; Hashimoto, M.; Tsuji, T. Syntheses of Silver Nanofilms, Nanorods, and Nanowires by a Microwave-Polyol Method in the Presence of Pt Seeds and Polyvinylpyrrolidone. *Chem. Lett.* **2004**, *33*, 370–371. [CrossRef]
- Zhu, Y.-J.; Hu, X.-L. Microwave-Assisted Polythiol Reduction Method: A New Solid–Liquid Route to Fast Preparation of Silver Nanowires. *Mater. Lett.* 2004, 58, 1517–1519. [CrossRef]
- 48. Yin, H.; Yamamoto, T.; Wada, Y.; Yanagida, S. Large-Scale and Size-Controlled Synthesis of Silver Nanoparticles under Microwave Irradiation. *Mater. Chem. Phys.* 2004, *83*, 66–70. [CrossRef]
- 49. Nirmala Grace, A.; Pandian, K. One Pot Synthesis of Polymer Protected Pt, Pd, Ag and Ru Nanoparticles and Nanoprisms under Reflux and Microwave Mode of Heating in Glycerol-A Comparative Study. *Mater. Chem. Phys.* **2007**, *104*, 191–198. [CrossRef]
- 50. Hebbalalu, D.; Lalley, J.; Nadagouda, M.N.; Varma, R.S. Greener Techniques for the Synthesis of Silver Nanoparticles Using Plant Extracts, Enzymes, Bacteria, Biodegradable Polymers, and Microwaves. *ACS Sustain. Chem. Eng.* **2013**, *1*, 703–712. [CrossRef]
- 51. Chen, J.; Wang, J.; Zhang, X.; Jin, Y. Microwave-Assisted Green Synthesis of Silver Nanoparticles by Carboxymethyl Cellulose Sodium and Silver Nitrate. *Mater. Chem. Phys.* 2008, 108, 421–424. [CrossRef]
- Tohidi, M.; Ghanbari, A.; Honarasa, F. Synthesis of Copper and Silver Nanoparticles by Using Microwave-Assisted Ionic Liquid Crystal Method and Their Application for Nonenzymatic Hydrogen Peroxide Determination. *Electrocatalysis* 2021, 12, 350–361. [CrossRef]
- 53. Torras, M.; Roig, A. From Silver Plates to Spherical Nanoparticles: Snapshots of Microwave-Assisted Polyol Synthesis. *ACS Omega* **2020**, *5*, 5731–5738. [CrossRef] [PubMed]
- Darmanin, T.; Nativo, P.; Gilliland, D.; Ceccone, G.; Pascual, C.; De Berardis, B.; Guittard, F.; Rossi, F. Microwave-Assisted Synthesis of Silver Nanoprisms/Nanoplates Using a "Modified Polyol Process". *Colloids Surf. A Physicochem. Eng. Asp.* 2012, 395, 145–151. [CrossRef]

- 55. Korkmaz, N.; Karadağ, A. Microwave Assisted Green Synthesis of Ag, Ag₂O, and Ag₂O₃ Nanoparticles. *J. Turk. Chem. Soc. Sect. A: Chem.* **2021**, *8*, 585–592. [CrossRef]
- 56. Ragunathan, V.; Chithra, K. Sequential Microwave-Ultrasound-Assisted Silver Nanoparticles Synthesis: A Swift Approach, Their Antioxidant, Antimicrobial, and in-Silico Studies. J. Mol. Liq. 2022, 347, 117954. [CrossRef]
- 57. Daublytė, E.; Zdaniauskienė, A.; Talaikis, M.; Drabavičius, A.; Charkova, T. A Facile Microwave-Assisted Synthesis of Ag@SiO₂ Nanoparticles for Raman Spectroscopy. *New J. Chem.* **2021**, *45*, 10952–10958. [CrossRef]
- Manno, R.; Ranjan, P.; Sebastian, V.; Mallada, R.; Irusta, S.; Sharma, U.K.; Van der Eycken, E.V.; Santamaria, J. Continuous Microwave-Assisted Synthesis of Silver Nanoclusters Confined in Mesoporous SBA-15: Application in Alkyne Cyclizations. *Chem. Mater.* 2020, 32, 2874–2883. [CrossRef]
- Souza, H.T.S.; Oliveira, S.A.A.; Souza, J.S. Modulating the Photocatalytic Activity of Ag Nanoparticles-Titanate Nanotubes Heterojunctions through Control of Microwave-Assisted Synthesis Conditions. J. Photochem. Photobiol. A Chem. 2020, 390, 112264. [CrossRef]
- 60. Shkir, M.; Khan, M.T.; Ashraf, I.M.; AlFaify, S.; El-Toni, A.M.; Aldalbahi, A.; Ghaithan, H.; Khan, A. Rapid Microwave-Assisted Synthesis of Ag-Doped PbS Nanoparticles for Optoelectronic Applications. *Ceram. Int.* **2019**, *45*, 21975–21985. [CrossRef]
- 61. Phuruangrat, A.; Wongwiwat, N.; Thongtem, T.; Thongtem, S. Microwave-Assisted Solution Synthesis and Photocatalytic Activity of Ag Nanoparticles Supported on ZnO Nanostructure Flowers. *Res. Chem. Intermed.* **2018**, 44, 7427–7436. [CrossRef]
- 62. Ajay Rakkesh, R.; Durgalakshmi, D.; Karthe, P.; Balakumar, S. Anisotropic Growth and Strain-Induced Tunable Optical Properties of Ag–ZnO Hierarchical Nanostructures by a Microwave Synthesis Method. *Mater. Chem. Phys.* 2020, 244, 122720. [CrossRef]
- 63. Porrawatkul, P.; Pimsen, R.; Kuyyogsuy, A.; Teppaya, N.; Noypha, A.; Chanthai, S.; Nuengmatcha, P. Microwave-Assisted Synthesis of Ag/ZnO Nanoparticles Using Averrhoa Carambola Fruit Extract as the Reducing Agent and Their Application in Cotton Fabrics with Antibacterial and UV-Protection Properties. *RSC Adv.* **2022**, *12*, 15008–15019. [CrossRef]
- 64. Kumar, S.V.; Bafana, A.P.; Pawar, P.; Rahman, A.; Dahoumane, S.A.; Jeffryes, C.S. High Conversion Synthesis of <10 Nm Starch-Stabilized Silver Nanoparticles Using Microwave Technology. *Sci. Rep.* **2018**, *8*, 5106. [CrossRef] [PubMed]
- 65. Wang, B.; Zhuang, X.; Deng, W.; Cheng, B. Microwave-Assisted Synthesis of Silver Nanoparticles in Alkalic Carboxymethyl Chitosan Solution. *Engineering* **2010**, *2*, 387–390. [CrossRef]
- 66. Tsuji, M.; Hashimoto, M.; Nishizawa, Y.; Tsuji, T. Preparation of Gold Nanoplates by a Microwave-Polyol Method. *Chem. Lett.* **2003**, *32*, 1114–1115. [CrossRef]
- 67. Liu, F.-K.; Ker, C.-J.; Chang, Y.-C.; Ko, F.-H.; Chu, T.-C.; Dai, B.-T. Microwave Heating for the Preparation of Nanometer Gold Particles. *Jpn. J. Appl. Phys.* 2003, 42, 4152–4158. [CrossRef]
- 68. Kundu, S.; Peng, L.; Liang, H. A New Route to Obtain High-Yield Multiple-Shaped Gold Nanoparticles in Aqueous Solution Using Microwave Irradiation. *Inorg. Chem.* **2008**, *47*, 6344–6352. [CrossRef]
- 69. Ren, L.; Meng, L.; Lu, Q.; Fei, Z.; Dyson, P.J. Fabrication of Gold Nano- and Microstructures in Ionic Liquids—A Remarkable Anion Effect. J. Colloid. Interface Sci. 2008, 323, 260–266. [CrossRef] [PubMed]
- Redel, E.; Walter, M.; Thomann, R.; Vollmer, C.; Hussein, L.; Scherer, H.; Krüger, M.; Janiak, C. Synthesis, Stabilization, Functionalization and, DFT Calculations of Gold Nanoparticles in Fluorous Phases (PTFE and Ionic Liquids). *Chem.-A Eur. J.* 2009, 15, 10047–10059. [CrossRef]
- Li, Z.; Liu, Z.; Zhang, J.; Han, B.; Du, J.; Gao, Y.; Jiang, T. Synthesis of Single-Crystal Gold Nanosheets of Large Size in Ionic Liquids. J. Phys. Chem. B 2005, 109, 14445–14448. [CrossRef]
- 72. Ren, L.; Meng, L.; Lu, Q. Fabrication of Octahedral Gold Nanostructures Using an Alcoholic Ionic Liquid. *Chem. Lett.* **2008**, 37, 106–107. [CrossRef]
- 73. Joseph, S.; Mathew, B. Microwave Assisted Facile Green Synthesis of Silver and Gold Nanocatalysts Using the Leaf Extract of Aerva Lanata. *Spectrochim. Acta A Mol. Biomol. Spectrosc.* 2015, 136, 1371–1379. [CrossRef] [PubMed]
- Gangapuram, B.R.; Bandi, R.; Alle, M.; Dadigala, R.; Kotu, G.M.; Guttena, V. Microwave Assisted Rapid Green Synthesis of Gold Nanoparticles Using Annona Squamosa L Peel Extract for the Efficient Catalytic Reduction of Organic Pollutants. *J. Mol. Struct.* 2018, 1167, 305–315. [CrossRef]
- 75. Thanayutsiri, T.; Patrojanasophon, P.; Opanasopit, P.; Ngawhirunpat, T.; Laiwattanapaisal, W.; Rojanarata, T. Rapid and Efficient Microwave-Assisted Extraction of Caesalpinia Sappan Linn. Heartwood and Subsequent Synthesis of Gold Nanoparticles. *Green.* Process. Synth. 2023, 12, 20228109. [CrossRef]
- Nguyen, V.P.; Le Trung, H.; Nguyen, T.H.; Hoang, D.; Tran, T.H. Advancement of Microwave-Assisted Biosynthesis for Preparing Au Nanoparticles Using Ganoderma Lucidum Extract and Evaluation of Their Catalytic Reduction of 4-Nitrophenol. ACS Omega 2021, 6, 32198–32207. [CrossRef] [PubMed]
- 77. Kumari, K.A.; Reddy, G.B.; Mittapalli, V. Microwave Assisted Synthesis of Gold Nanoparticles with Phyla Nodiflora (L.) Greene Leaves Extract and Its Studies of Catalytic Reduction of Organic Pollutants. *Mater. Today Proc.* 2020, 27, 1449–1454. [CrossRef]
- 78. Putri, S.E.; Pratiwi, D.E.; Side, S. The Effect Of Microwave Irradiation on Synthesis of Gold Nanoparticles Using Ethanol Extract of White Bol Guava Leaves. *J. Phys. Conf. Ser.* **2021**, *1752*, 012058. [CrossRef]
- Hussein, J.; El-Naggar, M.E.; Fouda, M.M.G.; Othman, S.I.; Allam, A.A.; Nadwa, E.H.; Rashwan, E.K.; Hendawy, O.M. Eco-Friendly Microwave Synthesis of Gold Nanoparticles for Attenuation of Brain Dysfunction in Diabetic Rats. *J. Clust. Sci.* 2021, 32, 423–435. [CrossRef]

- Madhavan, V.; Gangadharan, P.K.; Ajayan, A.; Chandran, S.; Raveendran, P. Microwave-Assisted Solid-State Synthesis of Au Nanoparticles, Size-Selective Speciation, and Their Self-Assembly into 2D-Superlattice. *Nano-Struct. Nano-Objects* 2019, 17, 218–222. [CrossRef]
- Zhong, G.; Xu, S.; Cui, M.; Dong, Q.; Wang, X.; Xia, Q.; Gao, J.; Pei, Y.; Qiao, Y.; Pastel, G.; et al. Rapid, High-Temperature, In Situ Microwave Synthesis of Bulk Nanocatalysts. *Small* 2019, *15*, 1904881. [CrossRef]
- 82. Yu, S.; Hachtel, J.A.; Chisholm, M.F.; Pantelides, S.T.; Laromaine, A.; Roig, A. Magnetic Gold Nanotriangles by Microwave-Assisted Polyol Synthesis. *Nanoscale* 2015, *7*, 14039–14046. [CrossRef]
- Shah, K.W.; Zheng, L. Microwave-Assisted Synthesis of Hexagonal Gold Nanoparticles Reduced by Organosilane (3-Mercaptopropyl)Trimethoxysilane. *Materials* 2019, 12, 1680. [CrossRef] [PubMed]
- 84. Gutiérrez-Wing, C.; Esparza, R.; Vargas-Hernández, C.; Fernández García, M.E.; José-Yacamán, M. Microwave-Assisted Synthesis of Gold Nanoparticles Self-Assembled into Self-Supported Superstructures. *Nanoscale* **2012**, *4*, 2281. [CrossRef] [PubMed]
- 85. Morad, M.; Karim, M.A.; Altass, H.M.; Khder, A.E.R.S. Microwave-Assisted Synthesis of Gold Nanoparticles Supported on Mn₃O₄ Catalyst for Low Temperature CO Oxidation. *Environ. Technol.* **2021**, *42*, 2680–2689. [CrossRef]
- Phuruangrat, A.; Prapassornwattana, P.; Thongtem, S.; Thongtem, T. Synthesis of Heterostructure Au/ZnO Nanocomposites by Microwave-Assisted Deposition Method and Their Photocatalytic Activity in Methylene Blue Degradation. *Russ. J. Phys. Chem. A* 2020, 94, 1464–1470. [CrossRef]
- 87. Zhang, K.; Zhang, J.; Qi, S. Synthesis of Nitrogen-Doped, Graphene-Supported Gold Nanoparticles via a Microwave Irradiation Method and Their Electrochemical Properties. *Res. Chem. Intermed.* **2020**, *46*, 2017–2024. [CrossRef]
- Wang, J.; Wang, Z. Rapid Synthesis of Hexagon-Shaped Gold Nanoplates by Microwave Assistant Method. *Mater. Lett.* 2007, 61, 4149–4151. [CrossRef]
- 89. Horikoshi, S.; Abe, H.; Sumi, T.; Torigoe, K.; Sakai, H.; Serpone, N.; Abe, M. Microwave Frequency Effect in the Formation of Au Nanocolloids in Polar and Non-Polar Solvents. *Nanoscale* **2011**, *3*, 1697. [CrossRef]
- 90. Mohamed, M.B.; AbouZeid, K.M.; Abdelsayed, V.; Aljarash, A.A.; El-Shall, M.S. Growth Mechanism of Anisotropic Gold Nanocrystals via Microwave Synthesis: Formation of Dioleamide by Gold Nanocatalysis. *ACS Nano* **2010**, *4*, 2766–2772. [CrossRef]
- Tsuji, M.; Miyamae, N.; Hashimoto, M.; Nishio, M.; Hikino, S.; Ishigami, N.; Tanaka, I. Shape and Size Controlled Synthesis of Gold Nanocrystals Using Oxidative Etching by AuCl4– and Cl– Anions in Microwave-Polyol Process. *Colloids Surf. A Physicochem. Eng. Asp.* 2007, 302, 587–598. [CrossRef]
- Shang, L.; Yang, L.; Stockmar, F.; Popescu, R.; Trouillet, V.; Bruns, M.; Gerthsen, D.; Nienhaus, G.U. Microwave-Assisted Rapid Synthesis of Luminescent Gold Nanoclusters for Sensing Hg²⁺ in Living Cells Using Fluorescence Imaging. *Nanoscale* 2012, 4, 4155. [CrossRef] [PubMed]
- May-Masnou, A.; Soler, L.; Torras, M.; Salles, P.; Llorca, J.; Roig, A. Fast and Simple Microwave Synthesis of TiO₂/Au Nanoparticles for Gas-Phase Photocatalytic Hydrogen Generation. *Front. Chem.* 2018, *6*, 110. [CrossRef] [PubMed]
- 94. Arshi, N.; Ahmed, F.; Kumar, S.; Anwar, M.S.; Lu, J.; Koo, B.H.; Lee, C.G. Microwave Assisted Synthesis of Gold Nanoparticles and Their Antibacterial Activity against *Escherichia coli* (*E. coli*). *Curr. Appl. Phys.* **2011**, *11*, S360–S363. [CrossRef]
- Dahal, N.; García, S.; Zhou, J.; Humphrey, S.M. Beneficial Effects of Microwave-Assisted Heating versus Conventional Heating in Noble Metal Nanoparticle Synthesis. ACS Nano 2012, 6, 9433–9446. [CrossRef] [PubMed]
- 96. Zhang, D.-Y.; Zhang, X.-Q.; Yao, X.-H.; Wan, Y.; Song, P.; Liu, Z.-Y.; Fu, Y.-J. Microwave-Assisted Synthesis of PdNPs by Cellulose Solution to Prepare 3D Porous Microspheres Applied on Dyes Discoloration. *Carbohydr. Polym.* **2020**, 247, 116569. [CrossRef]
- Heinrich, F.; Keßler, M.T.; Dohmen, S.; Singh, M.; Prechtl, M.H.G.; Mathur, S. Molecular Palladium Precursors for Pd0 Nanoparticle Preparation by Microwave Irradiation: Synthesis, Structural Characterization and Catalytic Activity. *Eur. J. Inorg. Chem.* 2012, 2012, 6027–6033. [CrossRef]
- Miyakawa, M.; Hiyoshi, N.; Koda, H.; Watanabe, K.; Kunigami, H.; Kunigami, H.; Miyazawa, A.; Nishioka, M. Continuous Syntheses of Carbon-Supported Pd and Pd@Pt Core–Shell Nanoparticles Using a Flow-Type Single-Mode Microwave Reactor. RSC Adv. 2020, 10, 6571–6575. [CrossRef] [PubMed]
- Sikeyi, L.L.; Ntuli, T.D.; Mongwe, T.H.; Maxakato, N.W.; Carleschi, E.; Doyle, B.P.; Coville, N.J.; Maubane-Nkadimeng, M.S. Microwave Assisted Synthesis of Nitrogen Doped and Oxygen Functionalized Carbon Nano Onions Supported Palladium Nanoparticles as Hybrid Anodic Electrocatalysts for Direct Alkaline Ethanol Fuel Cells. *Int. J. Hydrog. Energy* 2021, 46, 10862–10875. [CrossRef]
- 100. Kumar, R.; da Silva, E.T.S.G.; Singh, R.K.; Savu, R.; Alaferdov, A.V.; Fonseca, L.C.; Carossi, L.C.; Singh, A.; Khandka, S.; Kar, K.K.; et al. Microwave-Assisted Synthesis of Palladium Nanoparticles Intercalated Nitrogen Doped Reduced Graphene Oxide and Their Electrocatalytic Activity for Direct-Ethanol Fuel Cells. J. Colloid. Interface Sci. 2018, 515, 160–171. [CrossRef] [PubMed]
- Li, M.; Liu, R.; Han, G.; Tian, Y.; Chang, Y.; Xiao, Y. Facile Synthesis of Pd-Ni Nanoparticles on Reduced Graphene Oxide under Microwave Irradiation for Formic Acid Oxidation. *Chin. J. Chem.* 2017, 35, 1405–1410. [CrossRef]
- 102. Fatmawati, D.A.; Triyono, T.; Trisunaryanti, W.; Chasanah, U. Microwave-Assisted Chemical Co-Reduction of Pd Nanoparticles Anchored on Reduced Graphene Oxide with Different Loading Amounts. *Indones. J. Chem.* **2022**, *22*, 1282. [CrossRef]
- 103. Ameri, A.; Shakibaie, M.; Rahimi, H.-R.; Adeli-Sardou, M.; Raeisi, M.; Najafi, A.; Forootanfar, H. Rapid and Facile Microwave-Assisted Synthesis of Palladium Nanoparticles and Evaluation of Their Antioxidant Properties and Cytotoxic Effects Against Fibroblast-Like (HSkMC) and Human Lung Carcinoma (A549) Cell Lines. *Biol. Trace Elem. Res.* 2020, 197, 132–140. [CrossRef] [PubMed]

- 104. Elazab, H.A.; Sadek, M.A.; El-Idreesy, T.T. Microwave-Assisted Synthesis of Palladium Nanoparticles Supported on Copper Oxide in Aqueous Medium as an Efficient Catalyst for Suzuki Cross-Coupling Reaction. Adsorpt. Sci. Technol. 2018, 36, 1352–1365. [CrossRef]
- 105. Elazab, H.A.; Moussa, S.; Gupton, B.F.; El-Shall, M.S. Microwave-Assisted Synthesis of Pd Nanoparticles Supported on Fe₃O₄, Co₃O₄, and Ni(OH)₂ Nanoplates and Catalysis Application for CO Oxidation. *J. Nanoparticle Res.* 2014, 16, 2477. [CrossRef]
- 106. Walls, J.M.; Sagu, J.S.; Upul Wijayantha, K.G. Microwave Synthesised Pd–TiO₂ for Photocatalytic Ammonia Production. RSC Adv. 2019, 9, 6387–6394. [CrossRef]
- 107. Kwon, J.; Choi, K.; Tervoort, E.; Niederberger, M. One-Pot Microwave Synthesis of Pd Modified Titanium Dioxide Nanocrystals for 3D Aerogel Monoliths with Efficient Visible-Light Photocatalytic Activity in a Heated Gas Flow Reactor. J. Mater. Chem. A Mater. 2022, 10, 18383–18395. [CrossRef]
- Phuruangrat, A.; Klangnoi, T.; Patiphatpanya, P.; Dumrongrojthanath, P.; Thongtem, S.; Thongtem, T. Synthesis of Pd Nanoparticles Modified Bi2MoO6 Nanoplates by Microwave-Assisted Deposition with Their Enhanced Visible-Light-Driven Photocatalyst. *Optik* 2020, 212, 164674. [CrossRef]
- Luo, Y. A Simple Microwave-Based Route for Size-Controlled Preparation of Colloidal Pt Nanoparticles. *Mater. Lett.* 2007, 61, 1873–1875. [CrossRef]
- Chin, C.D.-W.; Treadwell, L.J.; Wiley, J.B. Microwave Synthetic Routes for Shape-Controlled Catalyst Nanoparticles and Nanocomposites. *Molecules* 2021, 26, 3647. [CrossRef] [PubMed]
- 111. Liu, Z.; Ling, X.Y.; Lee, J.Y.; Su, X.; Gan, L.M. Nanosized Pt and PtRu Colloids as Precursors for Direct Methanol Fuel Cell Catalysts. *J. Mater. Chem.* **2003**, *13*, 3049. [CrossRef]
- Liu, Z.; Sun, Z.; Han, B.; Zhang, J.; Huang, J.; Du, J.; Miao, S. Microwave-Assisted Synthesis of Pt Nanocrystals and Deposition on Carbon Nanotubes in Ionic Liquids. *J. Nanosci. Nanotechnol.* 2006, *6*, 175–179. [CrossRef]
- 113. Wojnicki, M.; Luty-Błocho, M.; Kwolek, P.; Gajewska, M.; Socha, R.P.; Pędzich, Z.; Csapó, E.; Hessel, V. The Influence of Dielectric Permittivity of Water on the Shape of PtNPs Synthesized in High-Pressure High-Temperature Microwave Reactor. *Sci. Rep.* 2021, 11, 4851. [CrossRef] [PubMed]
- 114. Kalyva, M.; Sunding, M.F.; Gunnæs, A.E.; Diplas, S.; Redekop, E.A. Correlation between Surface Chemistry and Morphology of PtCu and Pt Nanoparticles during Oxidation-Reduction Cycle. *Appl. Surf. Sci.* **2020**, *532*, 147369. [CrossRef]
- 115. Sandström, R.; Gracia-Espino, E.; Hu, G.; Shchukarev, A.; Ma, J.; Wågberg, T. Yttria Stabilized and Surface Activated Platinum (PtxYOy) Nanoparticles through Rapid Microwave Assisted Synthesis for Oxygen Reduction Reaction. *Nano Energy* 2018, 46, 141–149. [CrossRef]
- 116. Tsuji, M.; Uto, K.; Nagami, T.; Muto, A.; Fukushima, H.; Hayashi, J. Synthesis of Carbon-Supported Pt-YOx and PtY Nanoparticles with High Catalytic Activity for the Oxygen Reduction Reaction Using a Microwave-Based Polyol Method. *ChemCatChem* 2017, 9, 962–970. [CrossRef]
- Song, S.; Wang, Y.; Shen, P.K. Pulse-Microwave Assisted Polyol Synthesis of Highly Dispersed High Loading Pt/C Electrocatalyst for Oxygen Reduction Reaction. J. Power Sources 2007, 170, 46–49. [CrossRef]
- Zhang, W.; Chen, J.; Swiegers, G.F.; Ma, Z.-F.; Wallace, G.G. Microwave-Assisted Synthesis of Pt/CNT Nanocomposite Electrocatalysts for PEM Fuel Cells. *Nanoscale* 2010, 2, 282–286. [CrossRef]
- Bharti, A.; Cheruvally, G.; Muliankeezhu, S. Microwave Assisted, Facile Synthesis of Pt/CNT Catalyst for Proton Exchange Membrane Fuel Cell Application. *Int. J. Hydrog. Energy* 2017, 42, 11622–11631. [CrossRef]
- 120. Harish, S.; Baranton, S.; Coutanceau, C.; Joseph, J. Microwave Assisted Polyol Method for the Preparation of Pt/C, Ru/C and PtRu/C Nanoparticles and Its Application in Electrooxidation of Methanol. *J. Power Sources* **2012**, 214, 33–39. [CrossRef]
- 121. Sharma, R.; Wang, Y.; Li, F.; Chamier, J.; Andersen, S.M. Synthesis of a Pt/C Electrocatalyst from a User-Friendly Pt Precursor (Ammonium Hexachloroplatinate) through Microwave-Assisted Polyol Synthesis. ACS Appl. Energy Mater. 2019, 2, 6875–6882. [CrossRef]
- 122. Sharma, R.; Gyergyek, S.; Andersen, S.M. Microwave-Assisted Scalable Synthesis of Pt/C: Impact of the Microwave Irradiation and Carrier Solution Polarity on Nanoparticle Formation and Aging of the Support Carbon. *ACS Appl. Energy Mater.* 2022, *5*, 705–716. [CrossRef]
- 123. Sharma, R.; Gyergyek, S.; Chamier, J.; Morgen, P.; Andersen, S.M. Pt/C Electrocatalyst Durability Enhancement by Inhibition of Pt Nanoparticle Growth Through Microwave Pretreatment of Carbon Support. *ChemElectroChem* **2021**, *8*, 1183–1195. [CrossRef]
- Shakoorioskooie, M.; Menceloglu, Y.Z.; Unal, S.; Hayat Soytas, S. Rapid Microwave-Assisted Synthesis of Platinum Nanoparticles Immobilized in Electrospun Carbon Nanofibers for Electrochemical Catalysis. ACS Appl. Nano Mater. 2018, 1, 6236–6246. [CrossRef]
- 125. Ma, T.; Zhang, M.; Liu, H.; Wang, Y.; Pan, D. Synthesis of Novel Three-Dimensional Mesoporous Nitrogen Doped Graphene Supported Pt Nanoparticles as Superior Catalyst for Hydrogen Generation. Int. J. Hydrog. Energy 2018, 43, 19327–19335. [CrossRef]
- 126. Marinoiu, A.; Carcadea, E.; Sacca, A.; Carbone, A.; Sisu, C.; Dogaru, A.; Raceanu, M.; Varlam, M. One-Step Synthesis of Graphene Supported Platinum Nanoparticles as Electrocatalyst for PEM Fuel Cells. *Int. J. Hydrog. Energy* **2021**, *46*, 12242–12253. [CrossRef]
- 127. Sridhar, V.; Jeon, J.-H.; Oh, I.-K. Synthesis of Graphene Nano-Sheets Using Eco-Friendly Chemicals and Microwave Radiation. *Carbon* 2010, 48, 2953–2957. [CrossRef]

- Eren, E.O.; Özkan, N.; Devrim, Y. Polybenzimidazole-Modified Carbon Nanotubes as a Support Material for Platinum-Based High-Temperature Proton Exchange Membrane Fuel Cell Electrocatalysts. *Int. J. Hydrog. Energy* 2021, 46, 29556–29567. [CrossRef]
- Xin, Y.; Nagata, T.; Kato, K.; Shirai, T. Microwave-Assisted Synthesis of Pt Nanoparticles via Liquid-Phase Polyol Reaction for Catalytic Volatile Organic Compound Elimination. ACS Appl. Nano Mater. 2022, 5, 4305–4315. [CrossRef]
- Tsai, C.-H.; Yang, F.-L.; Chang, C.-H.; Chen-Yang, Y.W. Microwave-Assisted Synthesis of Silica Aerogel Supported Pt Nanoparticles for Self-Humidifying Proton Exchange Membrane Fuel Cell. *Int. J. Hydrog. Energy* 2012, *37*, 7669–7676. [CrossRef]
- Chu, C.-H.; Cheng, S.-H.; Chen, N.-T.; Liao, W.-N.; Lo, L.-W. Microwave-Synthesized Platinum-Embedded Mesoporous Silica Nanoparticles as Dual-Modality Contrast Agents: Computed Tomography and Optical Imaging. *Int. J. Mol. Sci.* 2019, 20, 1560. [CrossRef]
- 132. Ano, T.; Maitani, M.M.; Sato, Y.; Tsubaki, S.; Wada, Y. Drastic Microwave Heating of Percolated Pt Metal Nanoparticles Supported on Al₂O₃ Substrate. *Processes* **2020**, *8*, 72. [CrossRef]
- Lebègue, E.; Baranton, S.; Coutanceau, C. Polyol Synthesis of Nanosized Pt/C Electrocatalysts Assisted by Pulse Microwave Activation. J. Power Sources 2011, 196, 920–927. [CrossRef]
- Wang, H.-W.; Dong, R.-X.; Chang, H.-Y.; Liu, C.-L.; Chen-Yang, Y.-W. Preparation and Catalytic Activity of Pt/C Materials via Microwave Irradiation. *Mater. Lett.* 2007, 61, 830–833. [CrossRef]
- Kim, T.H.; Yoo, J.H.; Yi, S.C. Graphene Supported Platinum for Oxygen Reduction Reaction Electrocatalyst through a Facile Microwave-Assisted Polyol Synthesis. J. Ceram. Process. Res. 2017, 18, 261–264.
- 136. Zhang, F.; Wang, Z.; Zhang, Y.; Zheng, Z.; Wang, C.; Du, Y.; Ye, W. Microwave-Assisted Synthesis of Pt/Graphene Nanocomposites for Nonenzymatic Hydrogen Peroxide Sensor. *Int. J. Electrochem. Sci.* 2012, *7*, 1968–1977. [CrossRef]
- Kundu, P.; Nethravathi, C.; Deshpande, P.A.; Rajamathi, M.; Madras, G.; Ravishankar, N. Ultrafast Microwave-Assisted Route to Surfactant-Free Ultrafine Pt Nanoparticles on Graphene: Synergistic Co-Reduction Mechanism and High Catalytic Activity. *Chem. Mater.* 2011, 23, 2772–2780. [CrossRef]
- Sakthivel, M.; Schlange, A.; Kunz, U.; Turek, T. Microwave Assisted Synthesis of Surfactant Stabilized Platinum/Carbon Nanotube Electrocatalysts for Direct Methanol Fuel Cell Applications. J. Power Sources 2010, 195, 7083–7089. [CrossRef]
- 139. Liu, S.-J.; Huang, C.-H.; Huang, C.-K.; Hwang, W.-S. Chelating Agent Assisted Microwave Synthesis of Carbon Supported Pt Nanoparticles for Low Temperature Polymer Electrolyte Fuel Cells. *Electrochem. Commun.* **2009**, *11*, 1792–1795. [CrossRef]
- 140. Chu, Y.-Y.; Wang, Z.-B.; Gu, D.-M.; Yin, G.-P. Performance of Pt/C Catalysts Prepared by Microwave-Assisted Polyol Process for Methanol Electrooxidation. *J. Power Sources* **2010**, *195*, 1799–1804. [CrossRef]
- 141. Song, S.; Liu, J.; Shi, J.; Liu, H.; Maragou, V.; Wang, Y.; Tsiakaras, P. The Effect of Microwave Operation Parameters on the Electrochemical Performance of Pt/C Catalysts. *Appl. Catal. B* **2011**, *103*, 287–293. [CrossRef]
- 142. Wang, M.; Wang, Z.; Wei, L.; Li, J.; Zhao, X. Catalytic Performance and Synthesis of a Pt/Graphene-TiO₂ Catalyst Using an Environmentally Friendly Microwave-Assisted Solvothermal Method. *Chin. J. Catal.* **2017**, *38*, 1680–1687. [CrossRef]
- 143. Ling, X.Y.; Liu, Z.; Lee, J.Y. Microwave-Assisted Synthesis of Platinum Nanoparticles. J. Metastable Nanocrystalline Mater. 2005, 23, 199–202.
- 144. Pradhan, M.; Sarkar, S.; Sinha, A.K.; Basu, M.; Pal, T. High-Yield Synthesis of 1D Rh Nanostructures from Surfactant Mediated Reductive Pathway and Their Shape Transformation. J. Phys. Chem. C 2010, 114, 16129–16142. [CrossRef]
- 145. Marquardt, D.; Vollmer, C.; Thomann, R.; Steurer, P.; Mülhaupt, R.; Redel, E.; Janiak, C. The Use of Microwave Irradiation for the
- Easy Synthesis of Graphene-Supported Transition Metal Nanoparticles in Ionic Liquids. *Carbon* **2011**, *49*, 1326–1332. [CrossRef] 146. He, B.; Chen, Y.; Liu, H.; Liu, Y. Synthesis of Solvent-Stabilized Colloidal Nanoparticles of Platinum, Rhodium, and Rutheniumby
- Microwave-Polyol Process. J. Nanosci. Nanotechnol. 2005, 5, 266–270. [CrossRef] [PubMed]
 147. Ma, X.; Lin, R.; Beuerle, C.; Jackson, J.E.; Obare, S.O.; Ofoli, R.Y. Effects of Surface Activation on the Structural and Catalytic Properties of Ruthenium Nanoparticles Supported on Mesoporous Silica. Nanotechnology 2014, 25, 045701. [CrossRef] [PubMed]
- Raspolli Galletti, A.M.; Antonetti, C.; Longo, I.; Capannelli, G.; Venezia, A.M. A Novel Microwave Assisted Process for the Synthesis of Nanostructured Ruthenium Catalysts Active in the Hydrogenation of Phenol to Cyclohexanone^A. *Appl. Catal. A Gen.* 2008, 350, 46–52. [CrossRef]
- 149. Ugalde, M.; Chavira, E.; Figueroa, I.A.; Quintanar, C.; Espinosa-Magaña, F.; Zaragoza-Contreras, E.A.; Ochoa-Lara, M.T. Preparation of Rhodium Nano-Particles Using Microwaves. J. Solgel Sci. Technol. 2013, 65, 311–317. [CrossRef]
- Suryawanshi, Y.R.; Chakraborty, M.; Jauhari, S.; Mukhopadhyay, S.; Shenoy, K.T.; Shridharkrishna, R. Microwave Irradiation Solvothermal Technique: An Optimized Protocol for Size-Control Synthesis of Ru Nanoparticles. *Cryst. Res. Technol.* 2013, 48, 69–74. [CrossRef]
- 151. Karimi, F.; Peppley, B.A. Comparison of Conventional versus Microwave Heating for Polyol Synthesis of Supported Iridium Based Electrocatalyst for Polymer Electrolyte Membrane Water Electrolysis. *Int. J. Hydrog. Energy* **2017**, *42*, 5083–5094. [CrossRef]
- 152. Valodkar, M.; Modi, S.; Pal, A.; Thakore, S. Synthesis and Anti-Bacterial Activity of Cu, Ag and Cu–Ag Alloy Nanoparticles: A Green Approach. *Mater. Res. Bull.* 2011, 46, 384–389. [CrossRef]
- 153. Liu, Y.-Q.; Zhang, M.; Wang, F.-X.; Pan, G.-B. Facile Microwave-Assisted Synthesis of Uniform Single-Crystal Copper Nanowires with Excellent Electrical Conductivity. *RSC Adv.* **2012**, *2*, 11235. [CrossRef]
- 154. Zhu, H.; Zhang, C.; Yin, Y. Rapid Synthesis of Copper Nanoparticles by Sodium Hypophosphite Reduction in Ethylene Glycol under Microwave Irradiation. *J. Cryst. Growth* **2004**, 270, 722–728. [CrossRef]

- Kawasaki, H.; Kosaka, Y.; Myoujin, Y.; Narushima, T.; Yonezawa, T.; Arakawa, R. Microwave-Assisted Polyol Synthesis of Copper Nanocrystals without Using Additional Protective Agents. *Chem. Commun.* 2011, 47, 7740. [CrossRef]
- 156. Zhu, H.; Lin, Y.; Yin, Y. A Novel One-Step Chemical Method for Preparation of Copper Nanofluids. J. Colloid. Interface Sci. 2004, 277, 100–103. [CrossRef]
- 157. Zhu, H.; Zhang, C.; Yin, Y. Novel Synthesis of Copper Nanoparticles: Influence of the Synthesis Conditions on the Particle Size. *Nanotechnology* **2005**, *16*, 3079–3083. [CrossRef]
- 158. Blosi, M.; Albonetti, S.; Dondi, M.; Martelli, C.; Baldi, G. Microwave-Assisted Polyol Synthesis of Cu Nanoparticles. *J. Nanoparticle Res.* 2011, *13*, 127–138. [CrossRef]
- 159. Jung, Y.; Son, Y.-H.; Lee, J.-K. 3-D Self-Assembly of Flower-like Particles via Microwave Irradiation for Water Treatment. *RSC Adv.* **2012**, *2*, 5877. [CrossRef]
- Cheng, W.-T.; Cheng, H.W. Synthesis and Characterization of Cobalt Nano-Particles through Microwave Polyol Process. *AIChE J.* 2009, 55, 1383–1389. [CrossRef]
- 161. Wada, Y.; Kuramoto, H.; Sakata, T.; Mori, H.; Sumida, T.; Kitamura, T.; Yanagida, S. Preparation of Nano-Sized Nickel Metal Particles by Microwave Irradiation. *Chem. Lett.* **1999**, *28*, 607–608. [CrossRef]
- Eluri, R.; Paul, B. Microwave Assisted Greener Synthesis of Nickel Nanoparticles Using Sodium Hypophosphite. *Mater. Lett.* 2012, 76, 36–39. [CrossRef]
- Li, D.; Komarneni, S. Microwave-Assisted Polyol Process for Synthesis of Ni Nanoparticles. J. Am. Ceram. Soc. 2006, 89, 1510–1517. [CrossRef]
- Liu, X.; Meridor, U.; Zhao, P.; Song, G.; Frydman, A.; Gedanken, A. The Synthesis and Magnetic Properties of Monodispersed Single-Domain Nickel Nanospheres and Highly Globular Nanostructures of NicoreNiOshell. J. Magn. Magn. Mater. 2006, 301, 13–21. [CrossRef]
- 165. Xu, W.; Liew, K.Y.; Liu, H.; Huang, T.; Sun, C.; Zhao, Y. Microwave-Assisted Synthesis of Nickel Nanoparticles. *Mater. Lett.* 2008, 62, 2571–2573. [CrossRef]
- Donegan, K.P.; Godsell, J.F.; Tobin, J.M.; O'Byrne, J.P.; Otway, D.J.; Morris, M.A.; Roy, S.; Holmes, J.D. Microwave-Assisted Synthesis of Icosahedral Nickel Nanocrystals. *CrystEngComm* 2011, 13, 2023. [CrossRef]
- Hu, X.; Yu, J.C. High-Yield Synthesis of Nickel and Nickel Phosphide Nanowires via Microwave-Assisted Processes. *Chem. Mater.* 2008, 20, 6743–6749. [CrossRef]
- Zhou, B.; Ren, T.; Zhu, J.-J. A Rapid Preparation of Bismuth Nanowires Via A Microwave-Assisted Polyol Method. Int. J. Mod. Phys. B 2005, 19, 2829–2834. [CrossRef]
- Fouad, O.A.; El-Shall, M.S. Microwave Irradiation Assisted Growth of Cu, Ni, Co Metals and/or Oxides Nanoclusters and Their Catalytic Performance. *Nano* 2012, 07, 1250034. [CrossRef]
- Zhou, B.; Hong, J.-M.; Zhu, J.-J. Microwave-Assisted Rapid Synthesis of Antimony Dendrites. *Mater. Lett.* 2005, 59, 3081–3084.
 [CrossRef]
- 171. Liu, J.-W.; Chen, F.; Zhang, M.; Qi, H.; Zhang, C.-L.; Yu, S.-H. Rapid Microwave-Assisted Synthesis of Uniform Ultralong Te Nanowires, Optical Property, and Chemical Stability. *Langmuir* 2010, *26*, 11372–11377. [CrossRef] [PubMed]
- 172. Liu, T.; Zhang, G.; Su, X.; Chen, X.; Wang, D.; Qin, J. Tellurium Nanotubes Synthesized with Microwave-Assisted Monosaccharide Reduction Method. J. Nanosci. Nanotechnol. 2007, 7, 2500–2505. [CrossRef]
- 173. Gao, F.; Lu, Q.; Meng, X.; Komarneni, S. Synthesis of Nanorods and Nanowires Using Biomolecules under Conventional- and Microwave-Hydrothermal Conditions. *J. Mater. Sci.* 2008, 43, 2377–2386. [CrossRef]
- 174. Marquardt, D.; Xie, Z.; Taubert, A.; Thomann, R.; Janiak, C. Microwave Synthesis and Inherent Stabilization of Metal Nanoparticles in 1-Methyl-3-(3-Carboxyethyl)-Imidazolium Tetrafluoroborate. *Dalton Trans.* 2011, 40, 8290. [CrossRef] [PubMed]
- 175. Vollmer, C.; Redel, E.; Abu-Shandi, K.; Thomann, R.; Manyar, H.; Hardacre, C.; Janiak, C. Microwave Irradiation for the Facile Synthesis of Transition-Metal Nanoparticles (NPs) in Ionic Liquids (ILs) from Metal-Carbonyl Precursors and Ru-, Rh-, and Ir-NP/IL Dispersions as Biphasic Liquid-Liquid Hydrogenation Nanocatalysts for Cyclohexene. *Chem.-A Eur. J.* 2010, 16, 3849–3858. [CrossRef]
- 176. Jacob, D.S.; Genish, I.; Klein, L.; Gedanken, A. Carbon-Coated Core Shell Structured Copper and Nickel Nanoparticles Synthesized in an Ionic Liquid. *J. Phys. Chem. B* **2006**, *110*, 17711–17714. [CrossRef]
- 177. Pande, J.V.; Bindwal, A.B.; Pakade, Y.B.; Biniwale, R.B. Application of Microwave Synthesized Ag-Rh Nanoparticles in Cyclohexane Dehydrogenation for Enhanced H2 Delivery. *Int. J. Hydrog. Energy* **2018**, *43*, 7411–7423. [CrossRef]
- 178. Guo, H.; Li, H.; Jarvis, K.; Wan, H.; Kunal, P.; Dunning, S.G.; Liu, Y.; Henkelman, G.; Humphrey, S.M. Microwave-Assisted Synthesis of Classically Immiscible Ag–Ir Alloy Nanoparticle Catalysts. *ACS Catal.* **2018**, *8*, 11386–11397. [CrossRef]
- 179. García, S.; Zhang, L.; Piburn, G.W.; Henkelman, G.; Humphrey, S.M. Microwave Synthesis of Classically Immiscible Rhodium– Silver and Rhodium–Gold Alloy Nanoparticles: Highly Active Hydrogenation Catalysts. ACS Nano 2014, 8, 11512–11521. [CrossRef]
- Jia, X.; Yao, Y.; Yu, G.; Qu, L.; Li, T.; Li, Z.; Xu, C. Synthesis of Gold-Silver Nanoalloys under Microwave-Assisted Irradiation by Deposition of Silver on Gold Nanoclusters/Triple Helix Glucan and Antifungal Activity. *Carbohydr. Polym.* 2020, 238, 116169. [CrossRef]

- Cabello, G.; Davoglio, R.A.; Hartl, F.W.; Marco, J.F.; Pereira, E.C.; Biaggio, S.R.; Varela, H.; Cuesta, A. Microwave-Assisted Synthesis of Pt-Au Nanoparticles with Enhanced Electrocatalytic Activity for the Oxidation of Formic Acid. *Electrochim. Acta* 2017, 224, 56–63. [CrossRef]
- Zhang, H.; Yin, Y.; Hu, Y.; Li, C.; Wu, P.; Wei, S.; Cai, C. Pd@Pt Core—Shell Nanostructures with Controllable Composition Synthesized by a Microwave Method and Their Enhanced Electrocatalytic Activity toward Oxygen Reduction and Methanol Oxidation. J. Phys. Chem. C 2010, 114, 11861–11867. [CrossRef]
- 183. Song, P.; Lei, Y.; Hu, X.; Wang, C.; Wang, J.; Tang, Y. Rapid One-Step Synthesis of Carbon-Supported Platinum–Copper Nanoparticles with Enhanced Electrocatalytic Activity via Microwave-Assisted Heating. J. Colloid. Interface Sci. 2020, 574, 421–429. [CrossRef]
- Liu, F.-K.; Huang, P.-W.; Chang, Y.-C.; Ko, C.-J.; Ko, F.-H.; Chu, T.-C. Formation of Silver Nanorods by Microwave Heating in the Presence of Gold Seeds. J. Cryst. Growth 2005, 273, 439–445. [CrossRef]
- 185. Niu, X.; Wang, F.; Wang, W.; Wang, Y.; Huang, Y.; Zhang, J. Microwave-Assisted Synthesis of Pd3Ag Nanocomposite via Nature Polysaccharide Applied to Glucose Detection. *Int. J. Biol. Macromol.* **2018**, *118*, 2065–2070. [CrossRef] [PubMed]
- Harpeness, R.; Gedanken, A. Microwave Synthesis of Core–Shell Gold/Palladium Bimetallic Nanoparticles. *Langmuir* 2004, 20, 3431–3434. [CrossRef] [PubMed]
- 187. Howe, A.G.R.; Miedziak, P.J.; Morgan, D.J.; He, Q.; Strasser, P.; Edwards, J.K. One Pot Microwave Synthesis of Highly Stable AuPd@Pd Supported Core–Shell Nanoparticles. *Faraday Discuss.* **2018**, *208*, 409–425. [CrossRef] [PubMed]
- Cong, C.; Nakayama, S.; Maenosono, S.; Harada, M. Microwave-Assisted Polyol Synthesis of Pt/Pd and Pt/Rh Bimetallic Nanoparticles in Polymer Solutions Prepared by Batch and Continuous-Flow Processing. *Ind. Eng. Chem. Res.* 2018, 57, 179–190. [CrossRef]
- Patel, K.; Kapoor, S.; Dave, D.P.; Mukherjee, T. Synthesis of Pt, Pd, Pt/Ag and Pd/Ag Nanoparticles by Microwave-Polyol Method. J. Chem. Sci. 2005, 117, 311–316. [CrossRef]
- Guo, D.-J. Novel Synthesis of PtRu/Multi-Walled Carbon Nanotube Catalyst via a Microwave-Assisted Imidazolium Ionic Liquid Method for Methanol Oxidation. J. Power Sources 2010, 195, 7234–7237. [CrossRef]
- 191. Kunal, P.; Li, H.; Dewing, B.L.; Zhang, L.; Jarvis, K.; Henkelman, G.; Humphrey, S.M. Microwave-Assisted Synthesis of Pd x Au 100-x Alloy Nanoparticles: A Combined Experimental and Theoretical Assessment of Synthetic and Compositional Effects upon Catalytic Reactivity. ACS Catal. 2016, 6, 4882–4893. [CrossRef]
- 192. Zhang, D.; Chen, C.; Wang, X.; Guo, G.; Sun, Y. Synthesis of PtAu Alloy Nanocrystals in Micelle Nanoreactors Enabled by Flash Heating and Cooling. *Part. Part. Syst. Charact.* **2018**, *35*, 1700413. [CrossRef]
- 193. Bensebaa, F.; Patrito, N.; Le Page, Y.; L'Ecuyer, P.; Wang, D. Tunable Platinum–Ruthenium Nanoparticle Properties Using Microwave Synthesis. J. Mater. Chem. 2004, 14, 3378–3384. [CrossRef]
- Mathe, N.R.; Scriba, M.R.; Rikhotso, R.S.; Coville, N.J. Microwave-Irradiation Polyol Synthesis of PVP-Protected Pt–Ni Electrocatalysts for Methanol Oxidation Reaction. *Electrocatalysis* 2018, 9, 388–399. [CrossRef]
- Mathe, N.R.; Scriba, M.R.; Coville, N.J. Methanol Oxidation Reaction Activity of Microwave-Irradiated and Heat-Treated Pt/Co and Pt/Ni Nano-Electrocatalysts. Int. J. Hydrog. Energy 2014, 39, 18871–18881. [CrossRef]
- 196. Abdelsayed, V.; Aljarash, A.; El-Shall, M.S.; Al Othman, Z.A.; Alghamdi, A.H. Microwave Synthesis of Bimetallic Nanoalloys and CO Oxidation on Ceria-Supported Nanoalloys. *Chem. Mater.* **2009**, *21*, 2825–2834. [CrossRef]
- 197. Peng, X.; Chen, D.; Yang, X.; Wang, D.; Li, M.; Tseng, C.-C.; Panneerselvam, R.; Wang, X.; Hu, W.; Tian, J.; et al. Microwave-Assisted Synthesis of Highly Dispersed PtCu Nanoparticles on Three-Dimensional Nitrogen-Doped Graphene Networks with Remarkably Enhanced Methanol Electrooxidation. ACS Appl. Mater. Interfaces 2016, 8, 33673–33680. [CrossRef]
- 198. El-Deeb, H.; Bron, M. Microwave-Assisted Polyol Synthesis of PtCu/Carbon Nanotube Catalysts for Electrocatalytic Oxygen Reduction. J. Power Sources 2015, 275, 893–900. [CrossRef]
- Lin, R.; Cai, X.; Hao, Z.; Pu, H.; Yan, H. Rapid Microwave-Assisted Solvothermal Synthesis of Shape-Controlled Pt-Ni Alloy Nanoparticles for PEMFC. *Electrochim. Acta* 2018, 283, 764–771. [CrossRef]
- Ma, Y.; Miao, L.; Guo, W.; Yao, X.; Qin, F.; Wang, Z.; Du, H.; Li, J.; Kang, F.; Gan, L. Modulating Surface Composition and Oxygen Reduction Reaction Activities of Pt–Ni Octahedral Nanoparticles by Microwave-Enhanced Surface Diffusion during Solvothermal Synthesis. *Chem. Mater.* 2018, *30*, 4355–4360. [CrossRef]
- Tan, Y.; Zhang, Y.; Wang, X.; Zeng, L.; Luo, F.; Liu, A. Amorphous Nickel Coating on Carbon Nanotubes Supported Pt Nanoparticles as a Highly Durable and Active Electrocatalyst for Methanol Oxidation Reaction. J. Electroanal. Chem. 2020, 856, 113739. [CrossRef]
- 202. Sandström, R.; Ekspong, J.; Gracia-Espino, E.; Wågberg, T. Oxidatively Induced Exposure of Active Surface Area during Microwave Assisted Formation of Pt₃ Co Nanoparticles for Oxygen Reduction Reaction. *RSC Adv.* **2019**, *9*, 17979–17987. [CrossRef]
- Higgins, D.C.; Ye, S.; Knights, S.; Chen, Z. Highly Durable Platinum-Cobalt Nanowires by Microwave Irradiation as Oxygen Reduction Catalyst for PEM Fuel Cell. *Electrochem. Solid-State Lett.* 2012, 15, B83. [CrossRef]
- Kepenienė, V.; Tamašauskaitė-Tamašiūnaitė, L.; Jablonskienė, J.; Vaičiūnienė, J.; Kondrotas, R.; Juškėnas, R.; Norkus, E. Investigation of Graphene Supported Platinum-Cobalt Nanocomposites as Electrocatalysts for Ethanol Oxidation. J. Electrochem. Soc. 2014, 161, F1354–F1359. [CrossRef]
- Zhang, M.; Li, Y.; Yan, Z.; Jing, J.; Xie, J.; Chen, M. Improved Catalytic Activity of Cobalt Core–Platinum Shell Nanoparticles Supported on Surface Functionalized Graphene for Methanol Electro-Oxidation. *Electrochim. Acta* 2015, 158, 81–88. [CrossRef]
- 206. Du, J.-Q.; Zhang, Y.; Tian, T.; Yan, S.-C.; Wang, H.-T. Microwave Irradiation Assisted Rapid Synthesis of Fe–Ru Bimetallic Nanoparticles and Their Catalytic Properties in Water-Gas Shift Reaction. *Mater. Res. Bull.* 2009, 44, 1347–1351. [CrossRef]

- 207. Nguyen, H.L.; Howard, L.E.M.; Giblin, S.R.; Tanner, B.K.; Terry, I.; Hughes, A.K.; Ross, I.M.; Serres, A.; Bürckstümmer, H.; Evans, J.S.O. Synthesis of Monodispersed Fcc and Fct FePt/FePd Nanoparticles by Microwave Irradiation. J. Mater. Chem. 2005, 15, 5136. [CrossRef]
- 208. Harpeness, R.; Gedanken, A. The Microwave-Assisted Polyol Synthesis of Nanosized Hard Magnetic Material, FePt. J. Mater. Chem. 2005, 15, 698. [CrossRef]
- 209. Minami, R.; Kitamoto, Y.; Chikata, T.; Kato, S. Direct Synthesis of L10 Type Fe–Pt Nanoparticles Using Microwave-Polyol Method. *Electrochim. Acta* 2005, *51*, 864–866. [CrossRef]
- Köhler, D.; Heise, M.; Baranov, A.I.; Luo, Y.; Geiger, D.; Ruck, M.; Armbrüster, M. Synthesis of BiRh Nanoplates with Superior Catalytic Performance in the Semihydrogenation of Acetylene. *Chem. Mater.* 2012, 24, 1639–1644. [CrossRef]
- Jia, J.; Yu, J.C.; Wang, Y.-X.J.; Chan, K.M. Magnetic Nanochains of FeNi ₃ Prepared by a Template-Free Microwave-Hydrothermal Method. ACS Appl. Mater. Interfaces 2010, 2, 2579–2584. [CrossRef]
- GUO, X.; LI, Y.; LIU, Q.; SHEN, W. Microwave-Assisted Polyol-Synthesis of CoNi Nanomaterials. *Chin. J. Catal.* 2012, 33, 645–650. [CrossRef]
- Li, C.; Sui, J.; Zhang, Z.; Jiang, X.; Zhang, Z.; Yu, L. Microwave-Assisted Synthesis of Tremella-like NiCo/C Composites for Efficient Broadband Electromagnetic Wave Absorption at 2–40 GHz. *Chem. Eng. J.* 2019, 375, 122017. [CrossRef]
- Yadav, N.; Yadav, R.R.; Dey, K.K. Microwave Assisted Formation of Trimetallic AuPtCu Nanoparticles from Bimetallic Nano-Islands: Why It Is a Superior New Age Biocidal Agent Compared to Monometallic & Bimetallic Nanoparticles. J. Alloys Compd. 2022, 896, 163073. [CrossRef]
- 215. Hu, X.; Song, P.; Yang, X.; Wang, C.; Wang, J.; Tang, Y.; Zhang, J.; Mao, Z. One-Step Microwave-Assisted Synthesis of Carbon-Supported Ternary Pt-Sn-Rh Alloy Nanoparticles for Fuel Cells. J. Taiwan. Inst. Chem. Eng. 2020, 115, 272–278. [CrossRef]
- Womiloju, A.A.; Höppener, C.; Schubert, U.S.; Hoeppener, S. Microwave-Assisted Synthesis of Core–Shell Nanoparticles—Insights into the Growth of Different Geometries. *Part. Part. Syst. Charact.* 2020, 37, 2000019. [CrossRef]
- Yamauchi, T.; Tsukahara, Y.; Sakata, T.; Mori, H.; Yanagida, T.; Kawai, T.; Wada, Y. Magnetic Cu–Ni (Core–Shell) Nanoparticles in a One-Pot Reaction under Microwave Irradiation. *Nanoscale* 2010, 2, 515. [CrossRef]
- Wu, S.; Liu, Y.; Ren, Y.; Wei, Q.; Sun, Y. Microwave Synthesis of Single-Phase Nanoparticles Made of Multi-Principal Element Alloys. Nano Res. 2022, 15, 4886–4892. [CrossRef]
- 219. Kalyva, M.; Wragg, D.S.; Fjellvåg, H.; Sjåstad, A.O. Engineering Functions into Platinum and Platinum-Rhodium Nanoparticles in a One-Step Microwave Irradiation Synthesis. *ChemistryOpen* **2017**, *6*, 273–281. [CrossRef]
- Chen, Z.; Mochizuki, D.; Maitani, M.M.; Wada, Y. Facile Synthesis of Bimetallic Cu–Ag Nanoparticles under Microwave Irradiation and Their Oxidation Resistance. *Nanotechnology* 2013, 24, 265602. [CrossRef] [PubMed]
- 221. Cui, X.; Li, H.; Yu, G.; Yuan, M.; Yang, J.; Xu, D.; Hou, Y.; Dong, Z. Pt Coated Co Nanoparticles Supported on N-Doped Mesoporous Carbon as Highly Efficient, Magnetically Recyclable and Reusable Catalyst for Hydrogen Generation from Ammonia Borane. Int. J. Hydrog. Energy 2017, 42, 27055–27065. [CrossRef]
- Liu, Z.; Guo, B.; Hong, L.; Lim, T.H. Microwave Heated Polyol Synthesis of Carbon-Supported PtSn Nanoparticles for Methanol Electrooxidation. *Electrochem. Commun.* 2006, *8*, 83–90. [CrossRef]
- 223. Sarkar, A.; Vadivel Murugan, A.; Manthiram, A. Rapid Microwave-Assisted Solvothermal Synthesis of Methanol Tolerant Pt-Pd-Co Nanoalloy Electrocatalysts. *Fuel Cells* **2010**, *10*, 375–383. [CrossRef]
- 224. Maulana, A.L.; Chen, P.-C.; Shi, Z.; Yang, Y.; Lizandara-Pueyo, C.; Seeler, F.; Abruña, H.D.; Muller, D.; Schierle-Arndt, K.; Yang, P. Understanding the Structural Evolution of IrFeCoNiCu High-Entropy Alloy Nanoparticles under the Acidic Oxygen Evolution Reaction. *Nano Lett.* 2023, 23, 6637–6644. [CrossRef] [PubMed]
- 225. Wang, H.; Yuan, C.-G.; Liu, C.; Duan, X.; Guo, Q.; Shen, Y.; Liu, J.; Chen, Y. Microwave-Assisted Continuous Flow Phytosynthesis of Silver Nanoparticle/Reduced Graphene Oxide Composites and Related Visible Light Catalytic Performance. *J. Environ. Sci.* 2022, 115, 286–293. [CrossRef] [PubMed]
- 226. Amri, F.; Kasim, W.; Rochliadi, A.; Patah, A. Facile One-Pot Microwave-Assisted Synthesis of Rod-like and Hexagonal Plate-like AgNP@Ni-BTC Composites for a Potential Salivary Glucose Sensor. *Sens. Actuators Rep.* **2023**, *5*, 100141. [CrossRef]
- 227. Prabhakar Vattikuti, S.V.; Nagajyothi, P.C.; Devarayapalli, K.C.; Yoo, K.; Dang Nam, N.; Shim, J. Hybrid Ag/MoS₂ Nanosheets for Efficient Electrocatalytic Oxygen Reduction. *Appl. Surf. Sci.* 2020, 526, 146751. [CrossRef]
- 228. Venishetty, S.K.; Kummari, S.; Karingula, S.; Moru, S.; Gobi, K.V. Design and Synthesis of Pd Decorated RGO-MoSe₂ 2D Hybrid Network as High Performance Electrocatalyst toward Methanol Electrooxidation. *Int. J. Hydrog. Energy* 2023, 48, 21487–21498. [CrossRef]
- 229. Wang, Y.; Ding, Y.; Gao, J.; Zhang, X.; Sun, H.; Wang, G. Microwave-Regulated Bi Nanoparticles on Carbon Nanotube Networks as a Freestanding Electrode for Flexible Sodium-Ion Capacitors. J. Colloid. Interface Sci. 2023, 643, 420–427. [CrossRef]
- Jia, P.; Sun, J.; Wang, W.; Song, Z.; Zhao, X.; Mao, Y. Study on the Underpinning Mechanisms of Microwave-Induced Synthesis of Carbon-Coated Metal Nanoparticles. *Front. Energy Res.* 2023, 10, 1044283. [CrossRef]

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.