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SYNTHESIS OF CUPRUM OXIDE NANOPARTICLES IN WATER SUSPENSIONS

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Abstract

In this study, plasma discharge in a liquid at intensive ultrasonic field above the cavitation threshold has been proven to be of great interest for synthesis of cupric oxide nanoparticles with narrow particle size distribution. Further exploration of synthesized nanoparticles has demonstrated that the factor of ultrasonic cavitation during the synthesis substantially affects optical characteristics of nanoparticles.

Keywords

Plasma – Ultrasound - Cupric oxide

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Introduction

In recent years, approaches to synthesis of nanomaterials and nanopowders in particular have been of increasing interest and have attracted a great deal of attention of many groups of researchers¹. A range of techniques, including sol-gel method, high-energy dispersing, nanoscale biosynthesis has been being applied for several decades already that enabled to obtain nanoparticles of titania, silica, mica, carbon black and other materials². Along with traditional chemical methods of synthesis, some physical methods, such as electric discharges and ultrasound cavitation became increasingly important in view of creation of novel functional and composite materials.

One of the new promising ways to obtain nanoscale materials, including metal and metal oxide powders is the combined effect of the elastic oscillations of high intensity ultrasound and pulsed or steady electric fields in a liquid medium³. This type of plasma, being of great interest as a new object of physical study, has several advantages as a method for the synthesis of nanomaterials – relatively narrow particle size distribution of the synthesized nanopowder, specific composition and properties of nanomaterials⁴.

More specifically, the traditional arc discharge in aqueous electrolytes, which is widely used in engineering, is the most well-known form of stationary plasma discharge in liquid media⁵. For many years, such discharge is being used in physical and chemical studies and in the synthesis of various materials. The specific feature of arc discharge in liquid media is the localization of plasma region near the electrode ends and “falling” form of volt-ampere characteristics⁶.

However, when an ultrasonic cavitation is applied to a liquid, phase composition and physical properties of the liquid change abruptly, and this effect can lead to some specific features of the formation of electric discharges in the liquid⁷. In the region of intensive cavitation, the fraction of gas-vapor component in the liquid has a significant value, therefore it can be assumed that the conditions of electric breakdown in the cavitation region should become easier, which can result in the initiation of discharges of

¹ Yu. P. Aleksandrova; N. S. Budanova; A. A. Farmakovskaya; N. S. Okorokova; G. N. Ustyuzhaninova; N. P. Zharova y V. Kohlert, “New approaches to stabilize aqueous soot suspensions in the field of ultrasound”. *Revista Inclusiones*, Vol: 7 num 4 (2020).

² Yu. P. Aleksandrova; N. S. Budanova; A. A. Farmakovskaya; N. S. Okorokova; G. N. Ustyuzhaninova; N. P. Zharova y V. Kohlert, “Modification of the surface of carbon black with vinyl ether copolymers under ultrasonic treatment”. *Revista Inclusiones*, Vol: 7 num 4 (2020).

³ Yu. P. Aleksandrova; N. S. Budanova; A. A. Farmakovskaya; N. S. Okorokova; G. N. Ustyuzhaninova; N. P. Zharova y V. Kohlert, “The effect of surface mechanical activation of inorganic pigments on the stability of their aqueous dispersions in the presence of ethyl hydroxyethyl cellulose”. *Revista Inclusiones*, Vol: 7 num 4 (2020).

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⁵ A. V. Rudnev; N. G. Vanifatova; T. G. Dzherayan; E. V. Lazareva y N. A. Bulychev, “Study of stability and dispersion composition of calcium hydroxyapatite in aqueous suspensions by capillary zone electrophoresis”, *Russian Journal of Analytical Chemistry*, Vol: 68 num 8 (2013): 700.

⁶ V. N. Nikiforov; N. A. Bulychev y V. V. Rzhetskii, “Elastic properties of HTSC ceramics”, *Bulletin of the Lebedev Physical Institute*, Vol: 43 num 2 (2016): 74-79.

⁷ Yu. V. Ioni; S. V. Tkachev; N. A. Bulychev y S. P. Gubin, “Preparation of Finely Dispersed Nanographite”, *Inorganic Materials*, Vol: 47 num 6, (2011): 597-602.

different forms⁸. Varying the parameters of ultrasonic field, it is apparently possible to exert an influence on the processes of plasma glow in a cavitating liquid⁹. Furthermore, in this configuration, ultrasonic cavitation will interact with micro- and nanoparticles obtained in plasma and affect their physical properties and surface characteristics¹⁰.

The objective of the present work is to check this hypothesis and, moreover, to analyze the properties of nanoparticles synthesized in such acoustoplasma discharge and possible variations in optical characteristics of nanoparticles synthesized under intensive ultrasonic cavitation.

Synthesis of nanoparticles by acoustoplasma

Acoustoplasma technique involves the initiating of an electric discharge in a liquid that goes along with ultrasonic power assisted cavitation. In general, the specially designed acoustoplasma reactor includes a reservoir with two immersed electrodes and ultrasonic cavitator¹¹. Cavitation bubbles in the liquid phase provide the outstanding characteristics of a discharge and these characteristics have been proven to be governed by the regimes of ultrasonification¹². Furthermore, ultrasonic power prevents the secondary agglomeration of nanoparticles being synthesized from metal electrodes in electric discharge¹³. The scheme of experimental setup is shown in the figure 1¹⁴.

The setup consists of a reaction chamber, where discharge electrodes and an ultrasonic irradiator are introduced, optionally a generator of high-voltage pulses for discharge initiation, a power supply of discharge in liquid, an ultrasonic generator and blocks of control of electric and acoustic characteristics¹⁵.

⁸ N. A. Bulychev; A. I. Erokhin y M. A. Kazaryan, "A Comparative Study of Anti-Stokes Shift under Stimulated Rayleigh-Mie Scattering in Suspensions of Ag Nanoparticles Obtained in Plasma Discharge in Liquid under Ultrasonic Cavitation", Proceedings of SPIE, Vol: 11322 (2019): article 113222G.

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¹² Yu. O. Kirilina; I. V. Bakeeva; N. A. Bulychev y V. P. Zubov, "Organic-inorganic hybrid hydrogels based on linear poly(N-vinylpyrrolidone) and products of hydrolytic polycondensation of tetramethoxysilane", Polymer Science Series B, Vol: 51 num 3-4 (2009): 135.

¹³ N. A. Bulychev; M. A. Kazaryan; E. S. Gridneva; E. N. Murav'ev; V. F. Solinov; K. K. Koshelev; O. K. Kosheleva; V. I. Sachkov y S. G. Chen, "Plasma discharge with bulk glow in the liquid phase exposed to ultrasound", Bulletin of the Lebedev Physical Institute, Vol: 39 num 7 (2012): 214-220.

¹⁴ Yu. P. Aleksandrova; N. S. Budanova; A. A. Farmakovskaya; N. S. Okorokova; G. N. Ustyuzhaninova; N. P. Zharova y V. Kohlert, "Organic pigments surface modification by isobutyl vinyl ether copolymers under the action of ultrasonic", Revista Inclusiones, Vol: 7, num Especial (2020): 11-21.

¹⁵ I. S. Burkhanov; L. L. Chaikov; N. A. Bulychev; M. A. Kazaryan y V. I. Krasovskii, "Nanoscale metal oxide particles produced in the plasma discharge in the liquid phase upon exposure to

The chamber is provided with quartz windows for the observation of dynamic processes and registration of optical spectra of visible discharge glow¹⁶.

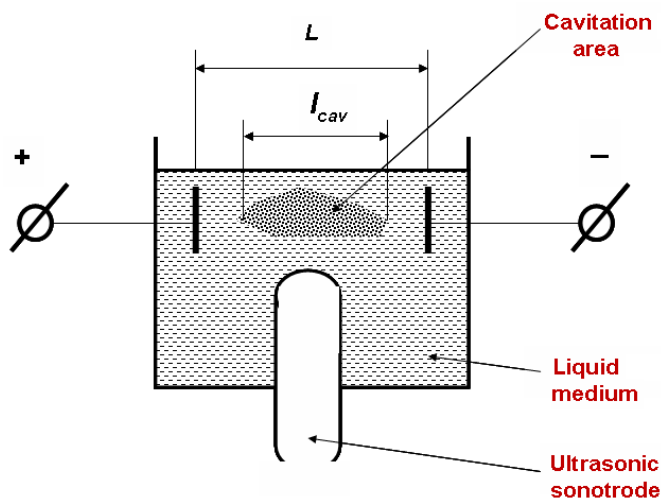


Figure 1

Scheme of experiment. L means the distance between the electrodes, L_{cav} means the size of cavitation zone.

The ultrasonic generator with a piezoceramic transducer provides the output acoustic power up to 2 kW in the frequency range 27-44 kHz. The parameters of acoustic equipment allow one to implement the intensity of an ultrasonic field in the volume of liquid up to 10 W/cm² and to vary cavitation regime in a wide range¹⁷.

In this work, the capability of plasma combined with ultrasonic cavitation for synthesis of novel nanoparticles was exemplarily investigated on cupric oxide nanoparticles¹⁸. For this purpose, electric discharge in liquid medium was initiated using cupric electrodes in water medium¹⁹. The discharge was initiated by direct current of 4A and the voltage 40 V²⁰.

ultrasonic cavitation. 2. Sizes and stability. Dynamic light scattering study”, *Bulletin of the Lebedev Physical Institute*, Vol:41 num 10 (2014): 297-304.

¹⁶ N. A. Bulychev; M. A. Kazaryan; A. Ethiraj y L. L. Chaikov, “Plasma Discharge in Liquid Phase Media under Ultrasonic Cavitation as a Technique for Synthesizing Gaseous Hydrogen”, *Bulletin of the Lebedev Physical Institute*, Vol: 45 num 9 (2018): 263-266.

¹⁷ M. N. Kirichenko; L. L. Chaikov; I. S. Burkhanov; N. A. Bulychev y M. A. Kazaryan, “Interaction of aluminum oxide nanoparticles with human blood plasma thrombin (according to light scattering)”, *Proceedings of SPIE*, Vol: 11322 (2019): article 113221Y.

¹⁸ Yu. P. Aleksandrova; N. S. Budanova; A. A. Farmakovskaya; N. S. Okorokova; G. N. Ustyuzhaninova; N. P. Zharova y V. Kohlert, “Ultrasonic treatment impact on the stability of aqueous dispersions of inorganic and organic pigments in the presence of surfactants”, *Revista Inclusiones*, Vol: 7, num Especial (2020): 387-397.

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²⁰ A. V. Ivanov; V. N. Nikiforov; S. V. Shevchenko; V. Yu. Timoshenko; V. V. Pryadun; N. A. Bulychev; A. B. Bychenko y M. A. Kazaryan, “Properties of Metal Oxide Nanoparticles Prepared by Plasma Discharge in Water with Ultrasonic Cavitation”, *International Journal of Nanotechnology*, Vol: 14 num 7/8 (2017): 618-626.

Cupric oxide nanoparticles were characterized by particle size measurements. The results are presented in the figure 1.

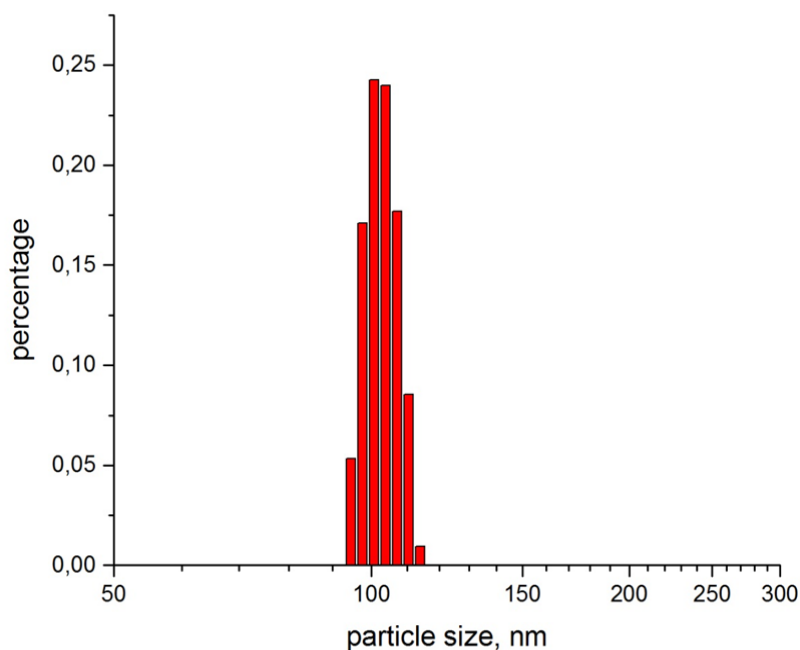


Figure 2
Size distribution of Cu oxide nanoparticles

Two perspective avenues of further application of these nanopowders can be performed: nanoparticles could first be deposited on a flat substrate and so create a surface modifying coating and the second option is an entrainment of the nanoparticles into the monomer matrix which can be polymerized afterwards yielding a polymer with immobilized nanoparticles²¹.

Conclusions

Acoustoplasma technique based on combination of a discharge in liquid with acoustic cavitation treatment provides an effective route for synthesis of solid nanoparticles of metals, oxides and semiconductors. Important feature is that such nanoparticles have relatively low particle size distribution that might be advantageous in comparison to other methods. This was demonstrated on the cupric oxide nanoparticles.

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