Synthesis of copper nanoparticles stabilized with cetylpyridinium chloride micelles

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Abstract

In this paper a methodology of chemical synthesis of copper nanoparticles stabilized with surfactant cetylpyridinium chloride (CPC) micelles has been developed. By varying of the volume of a reduction agent hydrazine hydrate (HH) and value of pH of the reactionary environment the optimum conditions for synthesis of copper nanoparticles with a stable size ranging from 40 to 80 nm were found. The copper nanoparticles obtained by this methodology have a widely perspective for use in such fields as medicine, chemistry, electronics (Langmuir-Blodgett films with conductive properties). The synthesized copper nanoparticles were characterized by scanning electron microscopy (SEM) and UV-visible spectroscopy.
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Keywords: Cu nanoparticles, chemical reduction, synthesis, surfactant.

Introduction

Unique properties of metals in an ultradisperse condition (sizes of particles on the order of nanometers (nm)) open a great opportunity for creation of a new materials. These materials are relevant for the use as catalysts, sensor systems, optical devices, medicines with high biological activity applied in ecology, medical studies and agricultural industry [**1**]. One of the most perspective industries for the use of copper nanoparticles is an electrical sector. Copper is an alternative to noble metals such as gold, silver, platinum nanoparticles etc. It has a high electrical conductivity and a thermal conductivity, being at the same time the more economic material. Copper nanoparticles are used in flexible electronics [**2**, **3**], printing (as a copper ink) [**4-6**], nanocircuits [**7**, **8**] due to their good electrical conductivity, catalytic properties [**9**, **10**] along with high biocompatibility [**11**, **12**] and antibacterial properties [**13**].

Copper nanoparticles have been synthesized through different methods such as a thermal decomposition [**14**, **15**], metal salt reduction [**16-20**], microwave heating [**21**], microemulsion techniques [**22**], laser ablation [**23**], the polyol method [**24**], the solvothermal method [**25**], thermal and sonochemical reduction [**26**]. Such physical methods as a proton radiation, a laser ablation and a vacuum vapor deposition are able to produce a wide range of metal nanoparticles, however they have a low quality compared to chemically synthesized materials. After applying the chemical methods stable nanoparticles can be obtained by a biochemical synthesis in a micelle. This technique consists in a chemical or a radiation-chemical reduction of metal ions from their salts to atoms in conditions favoring the subsequent formation of metal particles compared to the reduction in molecular solution with the stabilizer [**27**].

Chemical methods are widely used methods for synthesis of nanoparticles. They give the highest yield of the final product at the lowest cost, and they are also an environmentally safe, economical and relatively simple. Selection of the type of the reducing agent strongly influences on the size of the resulting nanoparticles. Currently, the stabilization of nanoparticles by surfactants is perspective. They limit the process of the conglomeration of nanoparticles and protect them from oxidation during a certain time interval. The main advantage of the synthesis in micelles is fact that nanoparticles are formed in the core of the micelle, i.e. in a more organized environment. This process is called solubilization. In this case, the shell of the micelle is a certain limit of the growth of these aggregates, allowing to obtain the particles of small sizes [**28**, **29**].

Among different methods the chemical reduction of copper from its salts in aqueous solution is characterized by a high rate of the chemical reaction. As to merits of this method of chemical synthesis of nanoparticles an ability to control the growth of metal nanoparticles and aggregation by optimizing the reaction conditions (e.g., temperature, reactant concentration, micelles sizes, types of surfactants, etc.) [**30**] can be referred, which is very difficult to realize, for example, by laser ablation.

Chemical methods for producing of copper nanoparticles are based on reduction of copper from its salts. Different chemical agents can be reduction agents. Often in synthesis of copper nanoparticles borohydride is used. This synthesis has several disadvantages. The resulting nanoparticles have a low stability, are polluted of boron and adverse products of reduction. There are also methods for synthesis of copper nanoparticles with using as reductions of copper agents are ascorbic acid, hypophosphorous acid, its salts and etc. These methods are characterized by a good reproducibility and small particle size. However, use as stabilizers of different types of stabilizers influences on the physical and chemical properties and stability of nanoparticles.

The goal of these researches were the development of an optimal methodology obtaining copper nanoparticles in micellar solution of cetylpyridinium chloride (CPC) by using standard, often used reduction agent hydrazine hydrate (HH).

Experimental

Materials

Copper chloride (II) [$CuCl_2 \cdot 2H_2O$] (Mr = 171 g/mol), cetylpyridinium chloride (CPC) is a cationogenic surfactant [$C_{21}H_{40}ClNO$] (Mr = 357.5 g/mol) with a critical micelle concentration (CMC) = 10^{-3} M, hydrazine hydrate (HH) [$N_2H_4 \cdot H_2O$] (Mr = 50 g/mol) (Vekton), distilled water [H_2O] and aqueous ammonia [NH_4OH] UHP 25-5 (Vekton).

Material synthesis

The copper nanoparticles were produced by means of a chemical reaction of reduction. In a separate flask, an aqueous solution of CPC $C_{21}H_{40}ClNO$ (0.01 M) was prepared. Resulting aqueous solution of $C_{21}H_{40}ClNO$ was divided in two and poured into two conical flasks of 100 ml volume. In the first flask with 0.01 M $C_{21}H_{40}ClNO$ 0.0428 g of $CuCl_2 \cdot 2H_2O$ salt with concentration of 0.01 M was added. The light blue solution was obtained. Then 5 ml of an aqueous ammonia (NH_4OH) was added in order to achieve pH = 11. Solution became intensely blue and transparent to the light (**Fig. 1**(**a**)). In the second flask with 0.01 M $C_{21}H_{40}ClNO$ hydrazine hydrate $N_2H_4 \cdot H_2O$ was added in different ratios (1 ml; 5 ml; 15 ml; 25 ml), and changed values of pH of the reactionary environment (9 and 11) for detecting optimal synthesis. In a water solution with CPC the HH dissolved with generate the heat. In the second flask the color of a solution became light yellow (**Fig. 1**(**b**)). Solutions of the first and second flasks solutions were merged being constantly stirred. Solution became light yellow (**Fig. 1**(**c**)).

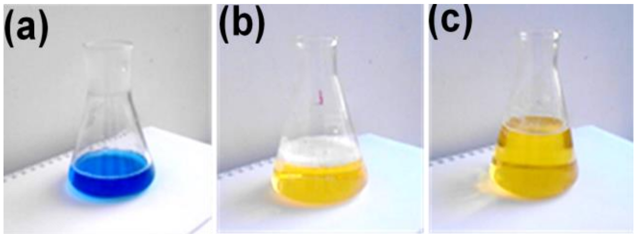

Fig. 1. Images of original solutions: (a) flask 1; (b) flask 2; (c) after mixing of solutions 1 and 2.

Then the resulting solution was stirred for 2 hours using thermal magnetic stirrer at 500 r/min and at a temperature 30 °C. Every 10 minutes the record of the aqueous solution color was made. This color changed from light yellow to brown (**Fig. 2**).

For identification of nanoparticles in the product of synthesis absorption spectrum was obtained in the wavelength range from 500 to 750 nm. Reduction of copper ions is manifested in the absorption spectrums of the system by advent of maximum average intensity in the wavelength range from 580 to 600 nm [**31**].

Reduction of copper by means of HH proceeds according to the chemical reaction (**1**),

$$Cu^{2+} + N_2H_4 + 4OH^- = 2Cu + N_2 + 4H_2O \tag{1}$$

In analyzing of the synthesis products by means of SEM, it was found, that the visualization of copper nanoparticles was hampered existence of large quantities of surfactants on the substrate. For obtaining a quality images of the surface morphology and the elemental (chemical) composition of copper nanoparticles by means of SEM was conducted a sample preparation of the final solution according to the following scheme. Sample of solution of the synthesis product was selected in quantities of 1 ml and placed in epindorf tube. Then 0.5 ml of distilled water was added. It was centrifuged for 5 minutes at 5000 r/min. Further, decantation of the solution in a volume of 0.5 ml was performed with followed addition of distilled water of the same volume. Centrifuged again. This operation was repeated 6 times. The resulting solution containing copper nanoparticles was applied to a silicon substrate and was heated at a temperature 100 °C for 5 and 30 minutes.

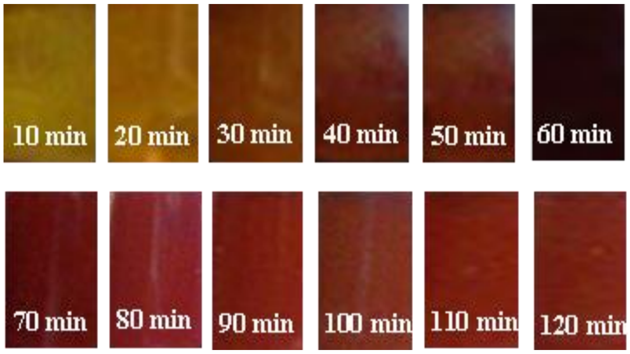

Fig. 2. The change of color of the obtained solution during a two-hour mixing.

Characterizations

Solution mixing during the process of copper nanoparticles synthesis and heating the substrate with copper nanoparticles on silicon substrate was performed using ICA RCT basic magnetic stir bar. Centrifugation of copper nanoparticles was performed using Elmi CM-70M centrifuge. The morphology of a surface and the chemical composition were determined by means of the scanning electronic microscope (SEM) (Tescan Mira II LMU) in the mode of detecting of secondary electrons (SE) (at 30 kV). UV-visible absorption spectra were registered on the spectrophotometer (SHIMADZU UV-2550) in the range of lengths of waves from 500 to 750 nm. All measurements were made at usual conditions.

Results and discussion

For the choice of an optimum methodology of synthesis of copper nanoparticles the studies were conducted at various volume of 1; 5; 15; 25 ml HH and value of pH = 11 of the reactionary environment being constantly stirred for 2 hours at temperature 30 °C.

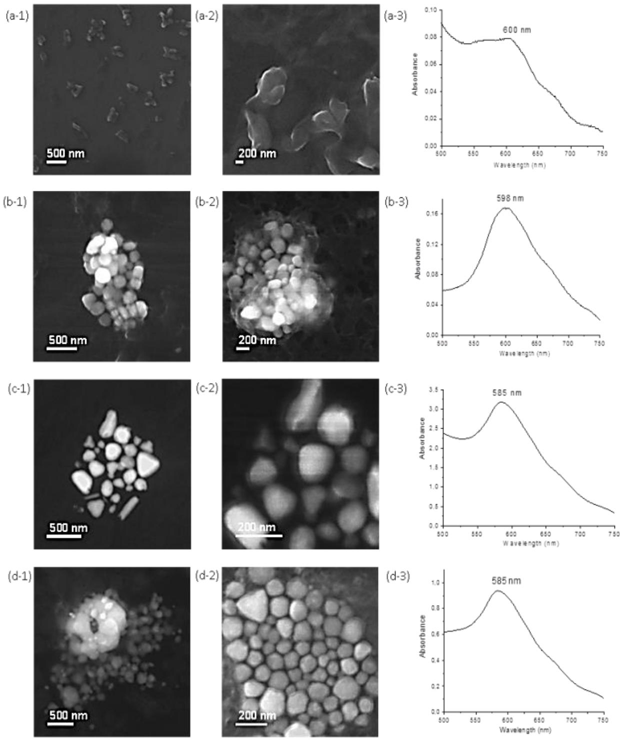

Fig. 3. (a-1) and (a-2) SEM images, (a-3) UV-visible spectrum of copper nanoparticles synthesized with 1 ml $N_2H_4 \cdot H_2O$. (b-1) and (b-2) SEM images, (b-3) UV- visible spectrum of copper nanoparticles synthesized with 5 ml $N_2H_4 \cdot H_2O$. (c-1) and (c-2) SEM images, (c-3) UV- visible spectrum of copper nanoparticles synthesized with 15 ml $N_2H_4 \cdot H_2O$. (d-1) and (d-2) SEM images, (d-3) UV- visible spectrum of copper nanoparticles synthesized with 25 ml $N_2H_4 \cdot H_2O$.

Fig. 3(**a-1**) and (**a-2**) shows SEM images of copper particle size from 55 nm to several micrometers obtained at 1 ml $N_2H_4 \cdot H_2O$. **Fig. 3**(**a-3**) represents the UV-visible absorption spectrum without the pronounced peak. At this volume of HH the peak was not detected, which is related to the adsorption of copper plaque on the flask walls while stirring solution on magnetic stirrer. **Fig. 3**(**b-1**) and (**b-2**) shows SEM images of copper particle size from 100 nm to several micrometers obtained at 5 ml $N_2H_4 \cdot H_2O$. These particles are uniformly arranged on a substrate and have the form of irregular polyhedrons with different shapes. **Fig. 3**(**b-3**) represents the UV-visible absorption spectrum with pronounced peak at 598 nm, which confirms the presence of copper particles in the solution. **Fig. 3**(**c-1**) and (**c-2**) shows SEM images of copper particles produced at 15 ml $N_2H_4 \cdot H_2O$. **Fig. 3**(**c-3**) represents the UV-visible absorption spectrum with pronounced peak at 585 nm, which confirms the presence of copper nanoparticles in the solution. **Fig. 3**(**d-1**) and (**d-2**) shows SEM images of copper particles having an average size from 40 to 80 nm obtained with 25 ml $N_2H_4 \cdot H_2O$. These particles have mostly spherical shape and they can be found on the entire substrate. **Fig. 3**(**d-3**) represents the UV-visible absorption spectrum with a pronounced peak at 585 nm, which confirms the presence of copper nanoparticles in the solution.

In experimental studies the optimum synthesis condition was detected at volume of 25 ml HH, at the same time the final solution is characterized by a number of features. Surfactant form aggregates in aqueous solution called micelles. They consist of tens, hundreds, thousands of monomeric molecules (ions). In the synthesis process the surfactant was taken at a concentration of slightly more than CMC, which contributes to spontaneous micelling of CPC molecules in aqueous solution. Cohesion between the polar water molecules is greater than that between the hydrocarbon chains and water. Therefore, any processes associated with the transition of the hydrocarbon radicals from the water to closest in polarity phases are energetically favorable. The hydrophobic chains are displaced from the water in the core of micelles. The core of the micelles is hydrophobic but may contain some water. It is a kind of nanoreactors for the centers of nucleation of copper nanoparticles. Reducing copper ranked in this nanoreactor is surrounded by a hydrophobic part of the micelle which protects it from oxidation. The size and shape of the copper nanoparticles is regulated by surfactant.

At varying value of pH of the reactionary environment we used the optimum volume of 25 ml HH, and in the first case the NH_4OH was added in order to achieve pH = 9, in the second case the same substance we added in order to achieve pH = 11. It is important to specify that the final color of solution was not red-brown in case of pH = 9, characteristic of metal copper. After the 30th minute mixing of solution on a magnetic stirrer the solution acquired black color and kept it within the remained 1.5 hours of mixing (**Fig. 4**(**a-2**)). During the spectral analysis at pH = 9 was not observe the accurate peak of absorption in a range of wavelength near to 585 nm (**Fig. 4**(**a-1**)). This fact testifies about overriding availability of compounds of copper: oxides, hydroxides and salts of copper.

At pH = 11 there is a characteristic peak of the absorption spectrum at a wavelength of 585 nm (**Fig. 4** (**b-1**)). As a result of chemical reaction in case of pH = 11 the deposit of red-brown color (**Fig. 4**(**b-2**)) characteristic of metal copper was formed (that was proven by means of spectrophotometry). The deviation of pH to lower area significantly changes the reaction products, resulting in not detecting the copper particles.

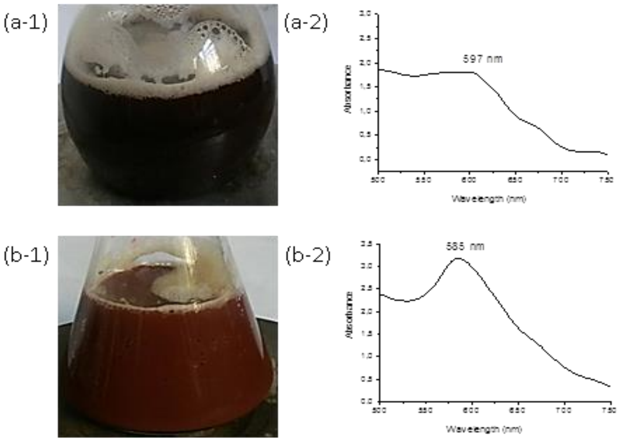

Fig. 4. (a-1) the end solution and (a-2) UV-visible spectra of copper nanoparticles synthesized at pH = 9, 25 ml $N_2H_4{\cdot}H_2O$; (b-1) the end solution and (b-2) UV-visible spectra of copper nanoparticles synthesized at pH = 11, 25 ml $N_2H_4{\cdot}H_2O$.

Based on the aforesaid, the most optimum for the synthesis of copper nanoparticles is the reactionary environment at pH = 11. This conclusions is made on the bases of the follow facts as the characteristic for metal copper and the absorption peak is positioned in UV-visible area (with a wavelength of 585 nm).

To conduct the study the solution with optimal conditions of synthesis at volume of 25 ml HH and value of pH = 11 of the reactionary environment was selected (**Fig. 5**).

When heating a sample for 5 minutes at a temperature 100 °C the visibility of the copper nanoparticles was much better, They have not been vague and were well discerned (**Fig. 5**(**a**)). And when heating to the temperature 100 °C for 30 minutes, the long crystals are formed (**Fig. 5**(**b**)). Presumably, it is a copper salts and a residual quantity of surfactants, that could be formed and react due to the longer heating of the substrate. One can see that studied product have acicular, irregular crystalline structure located in a chaotic manner. The diameter of the crystals ranges from 120 to 350 nm.

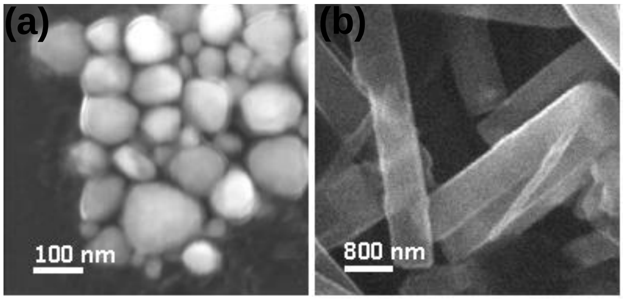

Fig. 5. SEM images of the synthesis products on a silicon substrate after heating at a temperature 100 °C for (a) 5 min and (b) 30 min.

It follows that the most optimum time of heating of a sample for SEM studies at a temperature 100 °C for 5 minutes.

The obtained chemical structure of sample of copper nanoparticles on substrates was studied by means of SEM. The study of the chemical composition of these films was made in various places of her surface (**Fig. 6**). Data have been written down in **Table 1**.

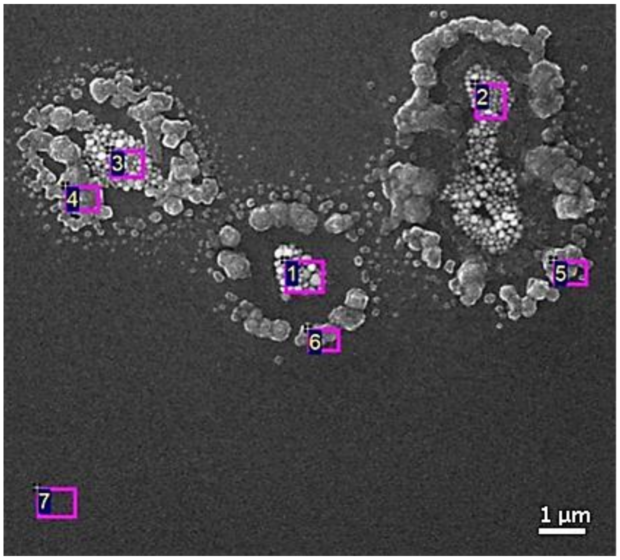

Fig. 6. SEM image of synthesis products on a silicon substrate after heating at a temperature 100 °C for 5 min.

Table 1. The chemical composition of products of copper nanoparticles synthesis on Si substrate (all results in weight %).

Point №	C	Cl	Cu	Result
1	52.73	2.21	45.07	100.00
2	52.26		47.74	100.00
3	55.78	1.69	42.53	100.00
4	93.75	6.25		100.00
5	92.35	7.65		100.00
6	92.78	7.22		100.00
7	100.00			100.00

The hydrazine hydrate is a kinetic active reducer allowing to carry out synthesis of copper nanoparticles under normal conditions. Absence of the stabilizing agent leads to formation of large units, therefore in this work we applied the stabilizing cationogenic surfactant CPC.

In this paper we applied a complexing ligand – ammonia. The interaction between copper ions and ammonia has a step character according to the chemical reaction (**2**):

$$\begin{aligned}
&Cu^{2+} + NH_3 \longleftrightarrow [Cu(NH_3)]^{2+}\\
&Cu^{2+} + 2NH_3 \longleftrightarrow [Cu(NH_3)_2]^{2+}\\
&Cu^{2+} + 3NH_3 \longleftrightarrow [Cu(NH_3)_3]^{2+}\\
&Cu^{2+} + 4NH_3 \longleftrightarrow [Cu(NH_3)_4]^{2+}
\end{aligned} \tag{2}$$

Therefore, a mixture of several ammoniates of copper will be present in solution whose quantitative ratios depend on the concentration of ammonia. Reaction of reduction of copper from copper chloride (II) by means of HH depends on value of pH of the reactionary environment as it affects character of formation of end products. It proceeds in the alkaline environment and it is described according to the chemical reaction (**3**):

$$CuCl_2 + N_2H_4 + 2NH_4OH = Cu\downarrow + N_2\uparrow + 2H_2O + 2NH_4Cl \quad (3)$$

The synthesized copper nanoparticles with CPC contain the inside layer, connected with hydrophilic groups and the external layer is connected with hydrophobic groups in aqueous solution. Thus, presence of CPC forms an inside and external layer around copper nanoparticles being at the same time the stabilizing agent.

Conclusion

We developed and offered a methodology of the synthesis of copper nanoparticles by use of chemical reduction method at the room temperature with use as a reduction of hydrate hydrazine. During the study, it was found that the change of volume of reduction agent (HH) and values of pH of the reactionary environment are the most important factors for producing of copper nanoparticles with certain characteristics. In the analysis of studies conducted by means of SEM, it was found that the copper nanoparticles with a stable smallest size from 40 to 80 nm can be obtained at volume of 25 ml HH and value of pH = 11 of the reactionary environment. It was found that this volume and value pH are optimal to obtain the best product. Obtained at these conditions copper nanoparticles have a similar size, shape, and this method is characterized by a good reproducibility. It was found that changing the volume of reduction agents, we can influence not only size of the stable nanoparticles but also on their shape. It is consistent with literature data. This methodology is a simple and cheap method of synthesis without application of complex installations, inert gases and additional heating that can be actually and economically advantageous in production of copper nanoparticles in large volumes.

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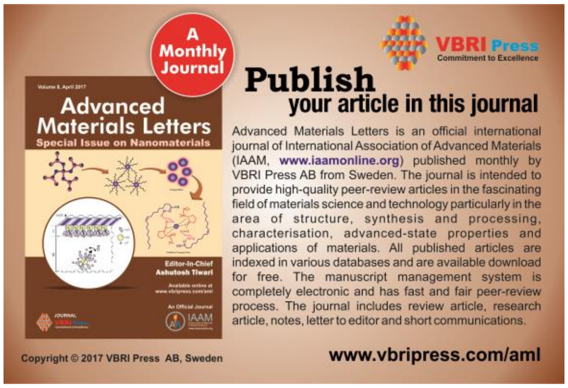

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