

PREPARATION OF SILVER NANOPARTICLES WITH CONTROLLED SIZE OF PARTICLES

K. Askaruly¹, S. Azat^{2,3}, A.R. Kerimkulova^{2,3}

¹Satbayev University, Satbayev st. 22a, Almaty, Kazakhstan

²Institute of Combustion Problems, Bogenbai batyr st. 172, Almaty, Kazakhstan

³Al-Farabi Kazakh National University, al-Farabi ave. 7, Almaty, Kazakhstan

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ABSTRACT

In the last decades, interest has significantly increased to to studying of nanodimensional particles, in particular, nanoparticles of various metals. In the first stage it is connected with the fact that nanoworld objects significantly differ the properties from macroobjects. Besides, there is a possibility of application of nanoparticles for receiving medical and biological medicines. In this study, we synthesized silver nanoparticles with various size and UV-VIS spectrophotometry, "Master Sizer" and "Zeta Sizer" tools characterized the nano species; we have conducted a simple and rapid one-pot synthesis method to generate nine colloids of Ag-NPs in different concentrations of TSCD and FSH. In this one-pot synthesis method, we utilized citrate as capping groups to stabilize the NPs and $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ to control the formation of shapes of NPs.

Keywords: nanoparticles, silver nanoparticles, trisodium citrate dihydrate, synthesis.

Introduction

Creation of nanoparticles of various metals are impossible without carrying out basic researches, the formed particles directed to the clarification of the nature, mechanisms and regularities of the reactions proceeding in the course of their formation. Despite considerable success in synthesizing AgNPs with different dimensions and size ranges, many of the reported methods have certain limitations in terms of their control over shape, size and stability in the dispersion system.

Silver nanoparticles, due to their special properties, are seen like a leader in the effective against morbidic microbial activity. Silver has a strong effect of slowing their activity down. Compared to the solid form of silver, the increased surface area of silver nanoparticles is feature responsible for their action in this regard [1]. This results in better contact with microorganisms, and more effective biocidal activity [2]. Silver nanoparticles are very effective from the many types of bacteria. We can see it and analyze from the next article, which the biocidal activity of silver nanoparticles has been confirmed, which includes *Acinetobacter* [3], *Escherichia* [4], *Pseudomonas* [5] and *Salmonella* [6]. The effective action of silver nanoparticles was also reported against Gram-positive bacteria: *Bacillus* [7], *Enterococcus* [8], *Listeria* [9], *Staphylococcus* [10] and *Streptococcus* [12].

Last two decades studies have shown the using of silver nanoparticles in the combination of different antibiotics [11]. It should be noted that the works also proved that silver NPs are also used against viruses by inhibiting their replication [12]. Their activity has been confirmed even against the HIV-1 [13] and influenza virus [14].

Application of silver nanoparticles

Developing of nanotechnology and their progress in creating the new methods of acquiring silver nanoparticles meant that the introduction of silver nanoparticles to a variety of consumer products has become very popular [15]. The unique properties of silver nanoparticles attracted the attention of many industries, particularly those in which an antiseptic effect is particularly desirable. This applies to food, textile, construction, medicine, cosmetology, pharmacy and other branches of industry [16, 17]. Silver nanoparticles are also used in the power industry [18] and in biomedicine, in which they act as receptors in labeling of biological materials [19]. Figures 1 and 2 illustrates the most common use of silver nanoparticles in various areas.

A particularly important issue is the use of silver nanoparticles in therapy. A process for preparing synthetic materials impregnated by silver nanoparticles, which may be used for production of catheters, has been

*Ответственный автор

E-mail: sanat_tolendiuly@mail.ru (С. Төлөндіұлы).

developed. The coating containing silver nanoparticles has antibacterial and disinfectant properties. In *in vitro* tests confirmed the inhibition of bacterial flora, with the effect maintained for a further 72 h [20].

A group of market products containing silver nanoparticles includes, among others, Acticoat Wound Care with nanocrystalline silver, and I-Flow SilverSoaker Nanosilver catheters. SilverSTAT from ABL Medical during laboratory tests that last for several minutes are destroy the *Staphylococcus aureus* – a strain resistant to methicillin and Enterococci bacteria – resistant to vancomycin [21].

Cosmetic products are exposed to bacteria and fungi, in particular during their manufacture or storage. Since cosmetics have direct contact with human body, it is important to prevent their contamination with pathogenic microorganisms. In addition, the biological purity in the final products should be also ensured. For this purpose, silver nanoparticles have been also used. Ha et. al in their patent shows a method of using silver nanoparticles as an ingredient in cosmetics dyeing mixture. The color is dependent on the nanometal structure. In the case of silver nanoparticles a yellow colour is mostly achieved. The authors report that one can use a combination of nanoparticles of silver and gold and thus get a yellow-red colour. The product can be used in the dyeing of cosmetic foundations, eye shadows, powders, lipsticks, inks, varnishes or eyebrow pencils. According to Ha et al, the products with metal nanoparticles, unlike the conventionally used metallic pigments, are not harmful to human health, and may even have health benefits [22]. A soap incorporating silver nanoparticles is also known and in 2013 the

method for its preparation was patented [23]. The authors report that thanks to the presence of silver nanoparticles, the product also has anti-inflammatory activity and protects the skin against the adverse effects of the microorganisms. Silver nanoparticles can penetrate deep into the skin and remove from it any bacterial contamination, which inhibits the formation of acne [23]. Silver nanoparticles have also been used in the production of toothpaste or oral care gels. Silver as an ingredient of such products at a concentration of 0.004% is an effective factor in preventing the growth of bacteria that cause unpleasant oral smells and dental cavities. The authors stipulate that the best biocidal effect is achieved when the average size of nanoparticles is less than 15 nm [24].

An important issue is maintaining antiseptic conditions in industrial facilities which, due to the nature of the production, are particularly vulnerable to microbial contamination. An innovative approach is the use of silver nanoparticles to purify the air in meat plants. The air blown into the production halls is an agent that transfers microorganisms to meat products may provide an environment conducive to their growth. Microorganisms can penetrate into meat products during slaughter, storage or their processing. Therefore, the blowing air must be filtered. In order to avoid the risk of microbial growth, a method of impregnating the air filter with silver nanoparticles has been developed. Results of microbiological tests confirmed that the impregnation of baghouse with silver nanoparticles allows for the almost total elimination of microbiological contamination of the air [25].

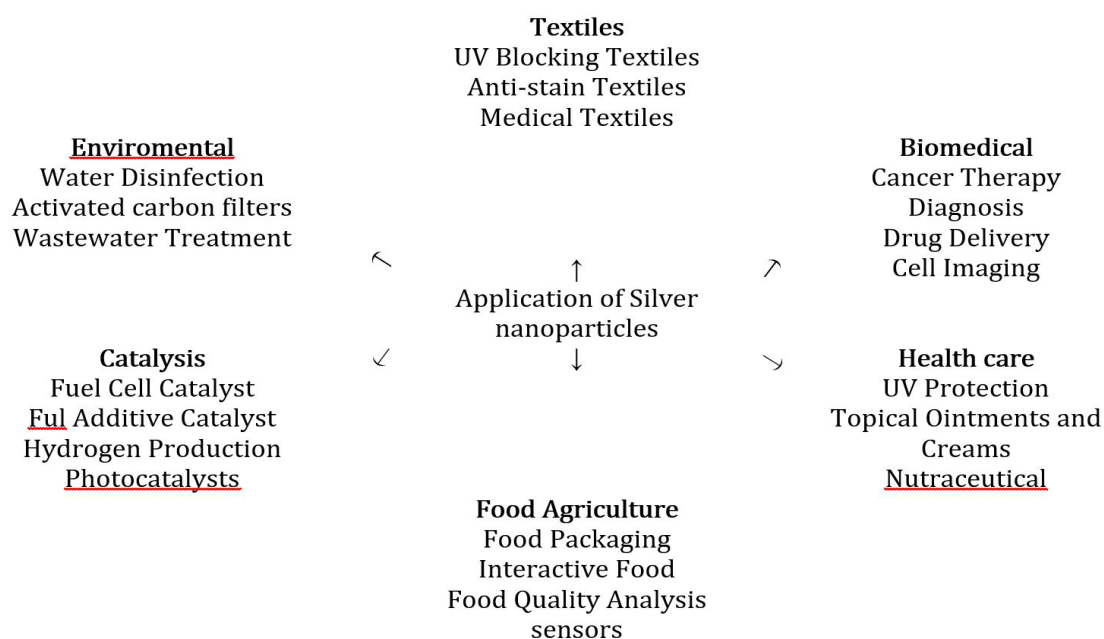


Fig. 1. Applications of silver nanoparticles [21].

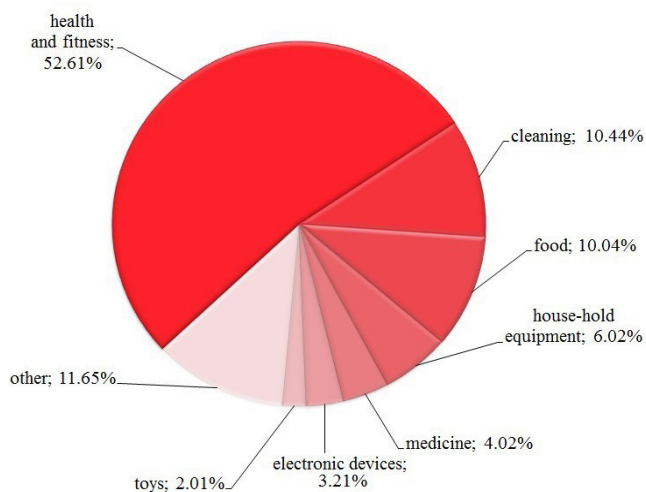


Fig. 2. Categories of products containing silver nanoparticles [19].

Synthesis of silver nanoparticles

There are many processing methods for obtaining nanosilver particles. The most common kinetic regularity of formation of nanodimensional particles is the combination of high speed of origin of a crystal phase to the small speed of its body height. These features of synthesis of nanoparticles define technological paths of its exercise [26].

There are two apparent approaches to the synthesis of nanomaterials and manufacture of nanostructures – «top down» and «bottom-up». The methods «top down» are based on decrease of the size of initial particles by crushing, etching, etc. The grind of substance by means of mills or etching in a lithograph is typical examples of receiving nanomaterial by method «top down». The main problem of receiving nanomaterials by methods of decrease is deterioration in structure of a surface of nanoobjects owing to formation of defects. These defects have significant effect on properties of nanomaterial as the surface relation to volume at nanomaterials is essential. Nevertheless, methods of receiving nanostructures decrease of the size remain widely used for receiving nanostructures and nanomaterials thanks to high efficiency and repeatability of processes. «Bottom-up» obtaining methods are synthesis or assembly of nanoobjects from atoms and molecules. The applied synthesis methods are leading to emergence and body height of nanoobjects, as a rule. A typical synthesis of similar materials happens by accession to an atom nanoparticle behind atom, a molecule behind a molecule or a cluster behind a cluster. Various ways of synthesis of the same material can lead to receiving materials, various on chemical composition, on crystallinity and a microstructure because of distinctions in kinetics of processes [27, 28].

Methods of receiving nanomaterials can be also grouped in a sign of environment in where is body height of nanoobjects:

Processes of body height of nanomaterials happen to use of a gas phase. The necessary component is transferred to a gas phase by evaporation, dispersion, combustion, etc. Formation of nanoparticles results from condensation or chemical reactions between components of a gas phase. Methods include also various ways of decomposition of volatile mother compounds (precursors). Precursors can react among themselves when heating or plasma exaltation mixture of gases. In many cases, precursors can be exposed to pyrolysis (thermal decomposition) or decay at influence of plasma or potent laser radiation. For receiving laminas use a particle flux (atoms or molecules) directed to a substrate on which they are condensed. Perhaps also formation of independent atomic bunches which at collision on a substrate form connections which corresponds to a ratio of streams of atoms.

Body height of nanoparticles is in a liquid phase due to chemical reactions of exchange, decomposition, polymerization, a crystallization, etc. In a liquid phase also nanolayers of various materials can be grown up, and there are also more difficult educations, for example threads, disks, etc.

Solid-phase processes, for example, disintegration of a solid solution on separate phases, selection of nanocrystals at heat treatment of glasses and ceramics. Also, photochemical processes are possible in solid matters, for example, in polymers from which a new phase results can be formed.

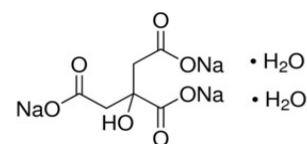
The hybrid methods using chemical processes on boundary of phases. Also photochemical processes are possible in solid matters, for example, in hybrids from which a new phase results can be formed [29].

Experimental Part

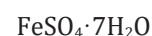
Methods and objects of research

Description of initial substances

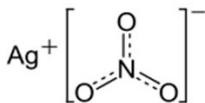
Trisodium Citrate Dihydrate (TSCD) with molecular weight 294.10 g/mol, which produced by «Sigma-Aldrich Chemie GmbH» (Germany) was used without further purification.



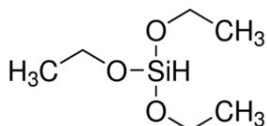
Ferrous sulfate heptahydrate (FSH) with molecular weight 278.01 g/mol, which produced by «Sigma-Aldrich Chemie GmbH» (Germany) was used without further purification.



Silver nitrate with molecular weight 169.87 g/mol, which produced by «Sigma-Aldrich Chemie GmbH» (Germany) was used without further purification.



Triethoxysilane (TES) with molecular weight 164.27 g/mol which produced by «Sigma-Aldrich Chemie GmbH» (Germany), 95 %



For preparation of solutions used distilled water.

Synthesis of silver nanoparticles

Citrate-sulfate method of synthesis of silver nanoparticles.

Silver nitrate, trisodium citrate and ferrous sulfate heptahydrate were used as starting materials for the preparation of silver nanoparticles. The silver colloid was prepared by using chemical reduction method. All solutions of reacting materials were prepared in distilled water. The synthesis was carried out in the magnetic stirrer (Fig. 3). AgNO_3 salt was melted in a distilled water. Initial substances (1% Trisodium Citrate Dihydrate and 6 g/l Ferrous sulfate heptahydrate) were measured and prepared in a different concentrations (Table 1) and they were added to solution of AgNO_3 . Stirring was carried out for 1 h.

Experiment results and their analysis

Experiment results from spectrophotometer

We have conducted a simple and rapid one-pot synthesis method to generate nine colloids of Ag NPs in different concentrations of TSCD and FSH. In this one-pot synthesis method, we utilized citrate as capping groups to stabilize the NPs and $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ to control the formation of shapes of NPs. By controlling various amounts of salts in the same mixture of AgNO_3 , sodium citrate, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, and H_2O solution, we generated various sizes and shapes of Ag NPs in each colloid, as described in the experimental section. $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ served as reducing agent for synthesis of the colloidal Ag NPs. As $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ was added into the mixture, it rapidly reduced Ag^+ to Ag, which acts as nucleation precursors. As $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ concentration increases, the amount of nucleation precursors increases. Therefore, the shapes of Ag NPs depend upon precursor concentration, which can be tuned by the amount of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$. They were have used UV-3600 Shimadzu UV-VIS-NIR spectrophotometer in order to obtain the absorption spectra of synthesized silver nanoparticles in solution. The absorption band in the 350 nm to 450 nm region is typical for the silver nanoparticles. Absorption spectra of colloids of Ag NPs in Fig. 4 show peak wavelengths range from 395 to 482 nm. For the third and fourth samples single peak wavelength of absorption spectra at 395 and 398 nm was observed, respectively. All other colloids (Table 2) show a primary peak at 400, 411, 413, 465, 470, 475, and 482 nm respectively. Absorption spectra of colloids of Ag NPs after 3 h in Fig. 5 show peak wavelengths range from 402 to 495 nm. For the third and fourth examples single peak wavelength of absorption spectra at 410 and 402 nm was observed, respectively.

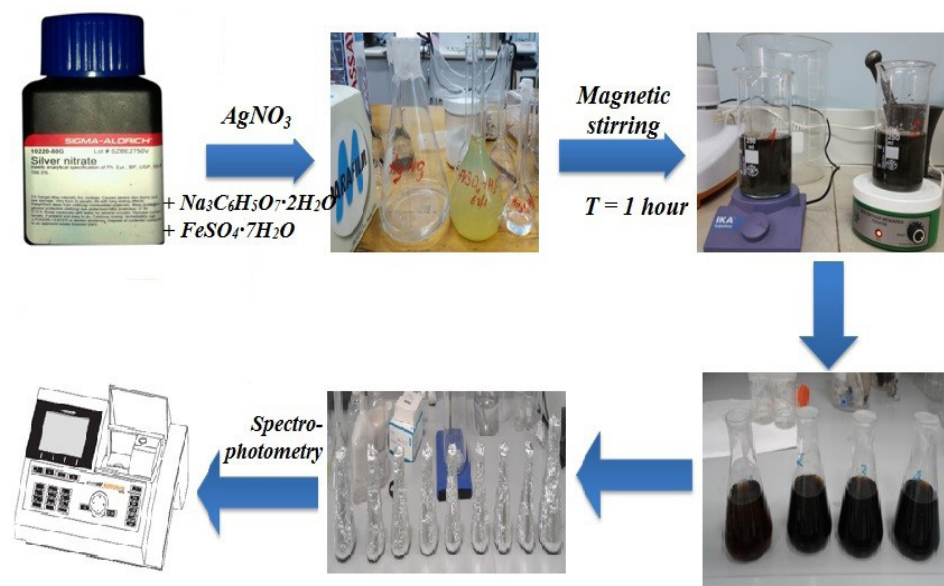


Fig. 3. Schematic representation for synthesis of silver nanoparticles.

Table 11% $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ and 6 g/l $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ salts ratio

Number of sample (10ml)	Volume of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ in a sample (ml)	Volume of $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ in a sample (ml)
1	1	9
2	2	8
3	3	7
4	4	6
5	5	5
6	6	4
7	7	3
8	8	2
9	9	1

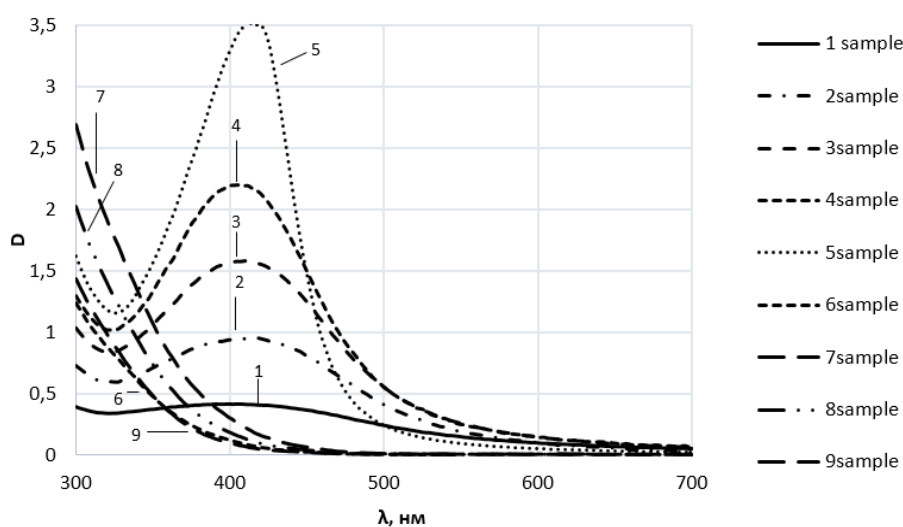


Fig. 4. Schematic representation for synthesis of silver nanoparticles.

All other colloids (Table 2) show a primary peak at 407, 416, 431, 460, 480, 482, and 495 nm respectively.

For the investigation of the sizes of synthesized silver nanoparticles we used a “Master sizer” and “Zeta sizer” tools. We have conducted a simple and rapid one-pot synthesis method to generate nine colloids of Ag-NPs in different concentrations of TSCD and FSH. In this one-pot synthesis method, we utilized citrate as capping groups to stabilize the NPs and $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ to control the formation of shapes of NPs. By controlling various amounts of salts in the same mixture of AgNO_3 , sodium citrate, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, and H_2O solution, we generated various sizes and shapes of Ag-NPs in each colloid, as described in the experimental section. $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ served as reducing agent for synthesis of the colloidal Ag-NPs. As $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ was added into the mixture, it rapidly reduced Ag^+ to Ag, which acts as nucleation precursors. As $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ concentration increases, the amount of nucleation precursors increases. Therefore, the shapes of Ag-NPs depend upon precursor concentration, which can be tuned by the amount of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$. Ag-NPs in colloids from “Master Sizer” tool show sizes in micrometer measurements. The results of Ag NPs sizes and their percentage in solution from

“Master Sizer” tool were summarized in following table (Table 3).

In this one-pot synthesis method, we utilized citrate as capping groups to stabilize the NPs and $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ to control the formation of shapes of NPs. By controlling various amounts of salts in the same mixture of AgNO_3 , sodium citrate, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, and H_2O solution, we generated various sizes and shapes of Ag NPs in each colloid, as described in the experimental section. The results of synthesized silver nanoparticle size's on micrometer size and their percentage in prepared solution were given on the appendix E.

Ag-NPs in colloids from “Master Sizer” tool show sizes in micrometer measurements. We have investigated the results of Ag-NPs sizes and their percentage in solution from “Master Sizer” tool. Dependence of size from volume density of silver nanoparticles with the mix that was $\text{FeSO}_4 \cdot 7\text{H}_2\text{O} : \text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ (1:9) showed the results of particle sizes, as 10 nm, 21 nm, 35 nm, 40 nm, 52 nm, 67 nm, 77 nm, and 87 nm. The next sample that was $\text{FeSO}_4 \cdot 7\text{H}_2\text{O} : \text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ (3:7) showed the results of particle sizes, as 10 nm, 16 nm, 21 nm, 31 nm, 40 nm, 52 nm, 67 nm, and 77 nm.

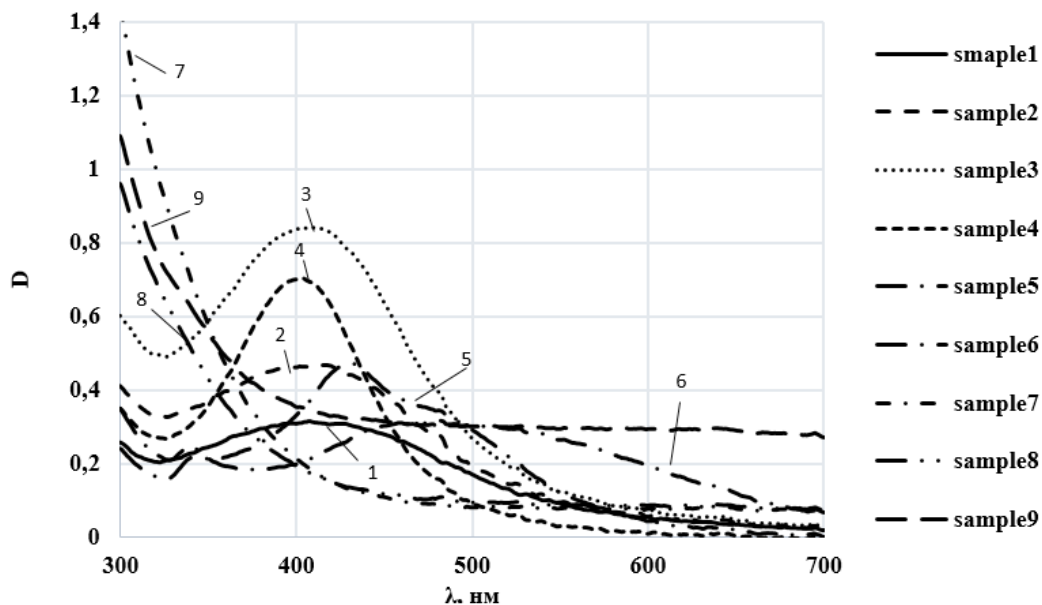


Fig. 5. Schematic representation for synthesis of silver nanoparticles.

Table 3

The summarized results of Ag NPs sizes from “Master Sizer” tool

№	1		2		3		4		5		6	
Na ₃ C ₆ H ₅ O ₇ ·2H ₂ O:	1:9		2:8		3:7		4:6		5:5		7:3	
FeSO ₄ ·7H ₂ O												
salts ratio												
Results of Ag NPs sizes and their percentage	size, nm	%	size, nm	%	size, nm	%	size, nm	%	size, nm	%	size, nm	%
	10	2	10	2.3	10	2.3	10	2.4	10	2.4	10	0.3
	21	8.6	16	9	16	9.2	16	9.2	17	9.1	16	1.3
	35	6.2	18	9.7	21	10	25	9.3	24	9.3	21	1.4
	40	5.1	21	9.9	31	8	31	7.4	31	7.4	31	1.1
	52	2.9	31	8.1	40	5.5	40	4.7	41	4.7	40	0.8
	67	1.1	40.7	5.7	52	2.9	52	2.2	53	2.1	52	0.4
	77	0.4	52.6	3.1	67	0.9	68	0.5	68	0.5	59	0.3
	87	0.1	67.9	1.1	77	0.4	77	0.2	77	0.1	67	0.1

CONCLUSION

In the present study, silver nanoparticles and silver nanocolloid solution were prepared chemically by the reduction of silver salt. The nano species were characterized by UV-VIS spectrophotometry, “Master Sizer” and “Zeta Sizer” tools. The synthesis process was precisely controlled and AgNPs of average size 8, 10, 15, 50, 70, 90, and 100 nm were synthesized, it is established that trisodium citrate has unique property to stabilize intermediate positively charged clusters of silver on the various structure. Times of their life were tens of minutes and hours. It is shown that concentrations of trisodium citrate and ferrous sulfate heptahydrate essentially influences the mechanism formation of clusters and

nanoparticles at radiation chemical restoration of ions of silver. These silver nanoparticles can be used in the above-mentioned area.

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Получение наночастиц серебра с регулируемым размером частиц

К. Аскарулы¹, С. Азат^{2,3}, А.Р.Керимкулова^{2,3}

¹Институт проблем горения, Богенбай Батыра 172, Алматы, Казахстан

²КазНУ им. аль-Фараби, проспект аль-Фараби 7, Алматы, Казахстан

³КазНИТУ им. К.И. Сатпаева, ул.Сатпаева 22а, Алматы, Казахстан

АННОТАЦИЯ

В последние десятилетия значительно возрос интерес к изучению наноразмерных частиц, в частности, наночастиц различных металлов. На первом этапе это связано с тем, что объекты наномира существенно отличаются по своим свойствам от макрообъектов. Это привело к открытию новых возможностей их применения для получения новых материалов с высокими качествами и другими характеристиками, которые находят все большее применение в различных областях науки и техники. Так, в последнее время наноматериалы используются для получения эффективных и селективных катализаторов, для создания элементов микроэлектронных и оптических приборов, синтеза материалов с уникальными свойствами. Кроме того, существует возможность применения наночастиц для получения медицинских и биологических лекарственных средств. В этом исследовании мы синтезировали наночастицы серебра различного размера и наночастицы были охарактеризованы с помощью спектрофотометрии UV-VIS, инструментов «Master Sizer» и «Zeta Sizer»;

Ключевые слова: наночастицы, наночастицы серебра, дигидрат тринатрийцитрата, синтез.

Бөлшек өлшемдері басқарылатын күміс нанобөлшектерінің алу

Қ. Асқарұлы¹, С. Азат^{2,3}, А.Р.Керимкулова^{2,3}

¹Жану проблемалары институты, Бөгенбай батыр көшесі 172, Алматы, Қазақстан

²Әл-Фараби атындағы ҚазҰУ, әл Фараби даңғылы 7, Алматы, Қазақстан

³Қ.И. Сатпаев атындағы ҚазҰТЗУ, Сатпаев көшесі 22а, Алматы, Қазақстан

АНДАТПА

Соңғы онжылдықта наноөлшемді бөлшектерді, атап айтқанда, түрлі металдардың нанобөлшектерін зерттеуге қызығушылық артуда. Бірінші кезеңде бұл нанообъектілері өзінің қасиеттері бойынша макрообъектілерден айтарлықтай өзгешелігіне байланысты. Бұл ғылым мен техниканың әр түрлі салаларында кеңінен қолданылатын жоғары сапалары және басқа да сипаттамалары бар жаңа материалдарды алу үшін оларды қолданудың жаңа мүмкіндіктерінің ашылуына әкелді. Мәселен, соңғы уақытта наноматериалдар тиімді және селективті катализаторларды алу үшін, микроэлектронды және оптикалық аспаптардың элементтерін жасау үшін, бірегей қасиеттері бар материалдарды синтездеу үшін пайдаланылады. Бұдан басқа, медициналық және биологиялық дәрілік заттарды алу үшін нанобөлшектерді қолдану мүмкіндігі бар. Бұл зерттеуде біз әр түрлі көлемдегі күміс нанобөлшектерін синтездедік және нанобөлшектерді УК - спектрофотометрия «Master Sizer» және «Zeta Sizer» құралдарының көмегімен сипаттадық.

Түйінді сөздер: нанобөлшектер, күміс нанобөлшектері, тринатрийцитрат дигидраты, синтез.