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M.I. Skiba^a, *O.A. Pivovarov*^a, *A.K. Makarova*^a, *V.D. Parkhomenko*^b**ONE-POT SYNTHESIS OF SILVER NANOPARTICLES USING DISCHARGED PLASMA IN THE PRESENCE OF POLYVINYL ALCOHOL**^a Ukrainian State University of Chemical Technology, Dnipro, Ukraine^b Ukrainian Institute of Scientific and Technical Expertise and Information, Kyiv, Ukraine

A simple method has been developed for the preparation of silver nanoparticles (Ag NPs) based on the use of discharged plasma in the presence of a stabilizer (polyvinyl alcohol, PVA). The UV-vis spectrum of AgNPs/PVA exhibited a broad surface plasmon absorption around 400–440 nm which originated from the formation of Ag NPs. The effects of different Ag⁺ concentration, the amount and the molecular weight of PVA and the duration of treatment with plasma discharge upon UV-visible absorption spectral characteristics of silver nanoparticles have been studied. The content of PVA in the reaction medium (1.25–10.0 g L⁻¹) is sufficient for nanoparticles stabilization, depending on the initial concentration of Ag⁺ (0.04–0.5 g L⁻¹). The obtained results show that the average size of the silver nanoparticles decreases with increasing the amount of PVA in the solution, but increases with increasing its molecular weight. Plasma action during 5–6 minutes provides formation of silver nanoparticles for the investigated range of Ag⁺ concentration. Characterization of AgNPs was made using scanning electron microscopy, X-ray diffraction and zeta potential analysis. The results indicated the formation of spherical, nanometer-sized particles. XRD analysis revealed that the particles were face-centered cubic. Nanoparticles thus prepared are found to be stable in aqueous solution over a period of two months at room temperature.

Keywords: silver, nanoparticles, discharged plasma, polyvinyl alcohol, stabilization.**Introduction**

In recent years there has been a tremendous growing interest in the field of silver nanoparticles (AgNPs) and their applications with a range of synthesis methods for metal NPs. Many different routes to the production of silver nanoparticles have been investigated, each showing characteristic advantages and disadvantages. A large number of reports are available on the synthesis of metal nanoparticles in solution by different methods such as photochemical, electrochemical, chemical reduction, microwave processing, ultra-sound processing and gamma irradiation [1–2]. Hence, synthesis strategies that result in controlled AgNPs size, distribution, shape and stability are still an area of interest.

More recently, stable generation of plasmas at atmospheric pressure [3] has given the opportunity to explore a new range of approaches for nanomaterials synthesis and various configurations and to synthesize AgNPs directly in liquids (e.g. plasma generated by electrodes immersed in liquids [4], gas-liquid interfacial low-pressure plasmas [5], atmospheric pressure plasma interfacing with liquid

[6,7], etc.). Among plasma-chemical discharges, discharged plasma is a promising option from the point of view of practical application. The use of discharged plasma, when the cathode is in a liquid phase and the anode is at some distance from the liquid surface, allows carrying out nonequilibrium oxidation processes in the liquid being treated. In the liquid media the oxidation processes occur which cannot be realized by using conventional electrolysis or by the action of arc- crown-, townsend- or barrier-discharge plasma on the liquid. By varying the composition of liquid phases it is possible, in some degree, to manage the paths of chemical transformations and composition of obtained products [8].

In the previous works, the authors showed the efficiency of using the discharged plasma in comparison with the conventional method of chemical reduction in solutions and photochemical deposition [9]. The efficiency of the use of discharged plasma for fabrication of silver nanoparticles from the aqueous solutions of metal salts one-shot in the presence of sodium alginate is demonstrated in publication authors [10]. Therefore, this synthesis

technique seems to have potential for metal nanoparticle synthesis. At the same time, silver nanoparticles can be applied in the different way: as individual silver nano-dispersions, as materials for purification and disinfection of water [11], as doping agent in the nanocomposite films [12], sensors, etc. [13]. Therefore, the use of sodium alginate may be inappropriate for such systems due to undesirable reaction. It is known that silver nanoparticles can be stabilized by sterile stabilization. Steric stabilization normally involves the adsorption of molecules such as polymers, surfactants or ligands on the particle surface providing a protective layer. Various stabilizing agents proved to be useful for protecting and dispersing nanoparticles in aqueous solutions. The polar side groups in polyvinyl alcohol (PVA) have been found especially effective in the stabilization of silver nanoparticles [14]. Polyvinyl alcohol is a bio-friendly polymer since it is water-soluble and has extremely low cytotoxicity. This allows a wide range of potential biomedical applications. It is used as a stabilizer due to its optical clarity which enables investigation of nanoparticle formation. Also, the introduction of nanosized silver into PVA matrix provides antibacterial activity. Studying of the process of obtaining silver nanoparticles in the presence of stabilizer reagent, PVA, under the action of plasma discharge, is of scientific and practical interest.

The work objective is the study of the synthesis of silver nanoparticles in colloidal solutions by discharged plasma with the use of the stabilizer, polyvinyl alcohol.

Materials and methods

The studies were carried out in a model gas-liquid plasma reactor of batch operation. Barrel-type reactor with inside diameter of 45 mm in height was used. The cooling of reaction mixture was provided by continuous circulation of cold water in outer jacket. The electrodes were made of stainless steel; one of the electrodes (with a diameter of 4 mm) was placed in the lower part of the reactor and the other one (with a diameter of 2.4 mm) was placed at a distance of 10 mm above the solution surface. To get plasma discharge, the voltage of 500–1000 V was applied to electrodes. The pressure in the reactor was held constant by vacuum pump. The volume of the solution in the reactor was 50 mL.

Silver nitrate (AgNO_3) and polyvinyl alcohol (PVA) were used to synthesize silver colloidal solution. The solutions of silver nitrate were prepared by dissolving AgNO_3 samples of P.A. purity in flasks with distilled water. PVA powders of different molar masses (15,000–1,200,000 g/mol) were employed to prepare aqueous solutions. The solutions of

polyvinyl alcohol with different concentrations were prepared by dissolving appropriate amounts of PVA in redistilled water in a 100 mL beaker using heating magnetic stirrer. A certain amount of AgNO_3 precursor was then added dropwise. Then the reaction mixture was placed in the reactor oven for the reduction of silver ions.

To characterize the nanoparticles of silver formed, the reaction mixture after the treatment was analyzed via spectrophotometry technique. The spectra of colloidal solutions were obtained by means of spectrophotometer UV-5800PC using quartz cuvettes in the wavelength range of 190 to 700 nm. Zeta potential of colloidal solutions was measured by means of the analyzer of zeta potential and particle size Zetasizer Nano-25 (Malvern Instruments Ltd., Malvern, England). The microphotographs of nanoparticles were obtained and the particle sizes were determined by scanning electron microscope JEOL JSM-6510LV (JEOL, Tokyo, Japan). The measurement was repeated three times for each sample. The dispersed phase of the solution obtained as a result of plasma-chemical treatment of the solution and dried in the air at 25°C was studied with the use of X-ray diffractometer Ultima IV Rigaku.

Results and discussion

UV-vis spectroscopy is a simple and sensitive technique to characterize the optical properties of AgNPs due to the excitation of surface plasmon resonance (SPR) in the AgNPs. We assume that the abundance of silver nanoparticles is proportional to the absorption intensity. The UV-vis spectra of the AgNPs obtained at the different concentration of silver nitrate (0.04–0.5 g/L) without and with PVA at its different concentrations are shown in Fig. 1.

The data show that the addition of PVA to the reaction mixture for all investigated concentrations of Ag^+ ions before the treatment by discharged plasma changes the intensity of the PPR peak, which characterizes metal nanoparticles of silver. However, the concentration of the polymer which provides the highest value of the absorbance for different initial concentrations of the Ag^+ is different.

At the Ag^+ ions concentration of 0.04 g/L (Fig. 1,a) in the initial solution, the presence of PVA contributes to the displacement of the peak PPR from 440 nm to 400–412 nm and to an increase of its intensity. This can be explained by the formation of more particles of silver and/or by diminishing their size. At a concentration of PVA of 1.25 g/L, the PPR peak has a maximum value of the intensity. This amount of polymer is recommended, because a further increase of the polymer concentration to

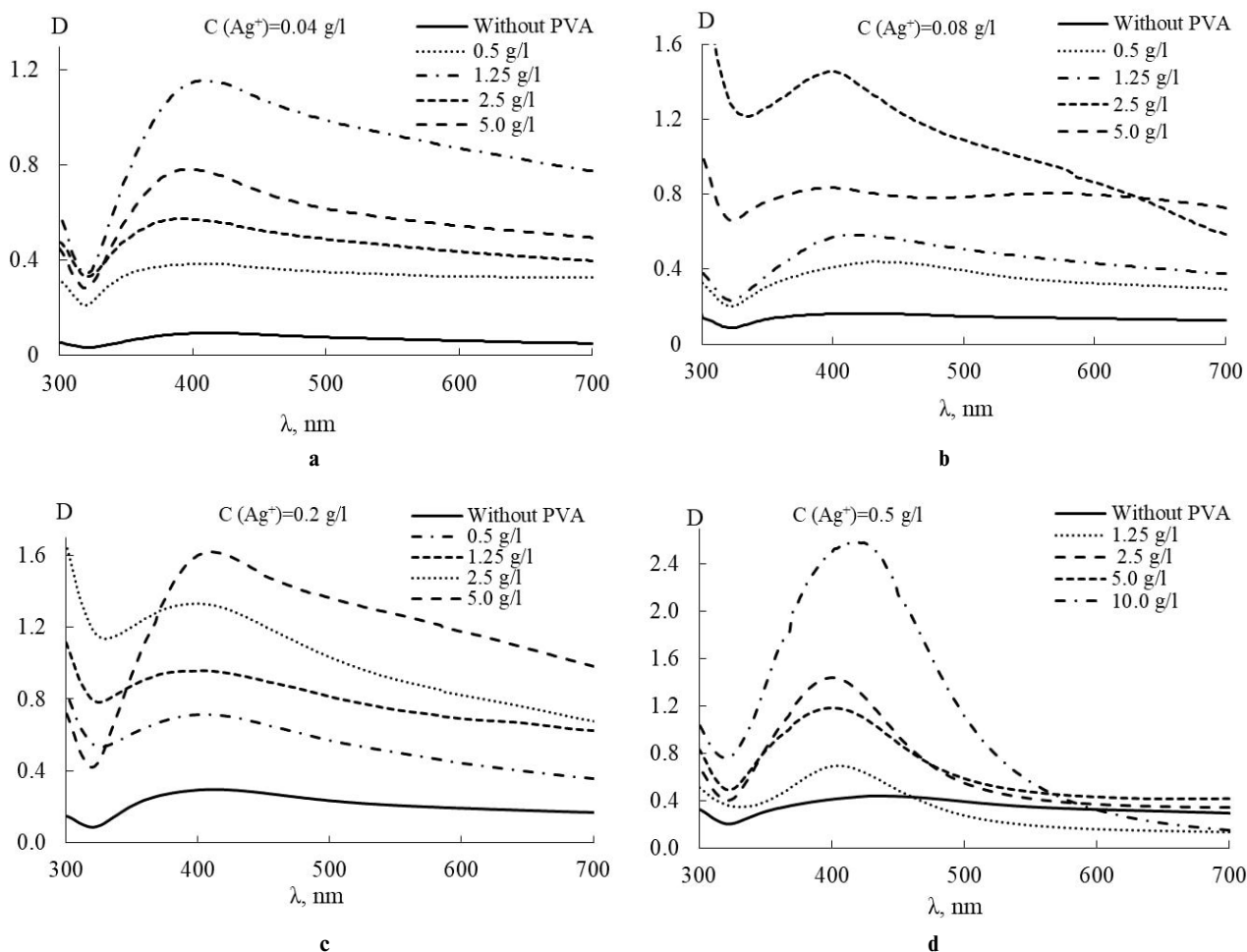


Fig. 1. Dependence of absorbance (D) of plasma-chemically obtained colloidal solution of silver on the wavelength (λ) at various concentration of Ag⁺ and PVA (a–d). I=120 mA, P=0,08 MPa, τ =5 min

2.5–5.0 g/L reduces the intensity of the PPR peak, indicating that not all the polymeric radicals may be able to reduce Ag⁺ ions due to steric reasons and some of these are being consumed in other competing reactions. Although the reactivity of PVA with hydrated electron is known to be very low, there is some possibility of the reaction of a fraction of hydrated electrons with PVA at a higher concentration. This may also be responsible for observed slightly lower reduction rate of Ag⁺ ions. A similar relationship remains at the Ag⁺ concentration of 0.08 g/L (Fig. 1,b). An increase in the initial concentration leads to a rise in the required amount of stabilizer. At a concentration of PVA of 2.5 g/L, an increase in the intensity of the PPR peak is observed. At the Ag⁺ concentration of 0.2 g/L, an intense peak at 410 nm is observed at the amount of PVA of 5.0 g/L. At the Ag⁺ concentration of 0.5 g/L, the most intense peak characteristic of nanoparticles of silver is observed at 420 nm in the presence of 10.0 g/L PVA. Thus, the obtained data show that

the effectiveness of plasmochemical formation of nanosheets of silver rises in the presence of PVA.

The influence of the duration of plasma action on the formation of silver nanodispersion at different concentrations of Ag⁺ ions with predetermined expedient concentration of PVA was investigated (Fig. 2).

The results showed that absorbance of irradiated Ag⁺ solutions was increased up to a maximum at 4–5 min for the Ag⁺ concentration from 0.04 to 0.5 g/L. This irradiation time defined as conversion time to reduce Ag⁺ into metallic silver completely. Rising irradiation time to 8 min is accompanied with a slight increase in the intensity.

Zeta potential measurements were conducted to evaluate the stability of the silver suspensions (Fig. 3). Zeta potential is based on the mobility of a particle in an electric field and is related to the electrical potential at the junction between the diffuse ion layer surrounding the particle surface and the bulk solution. Generally, a suspension that exhibits

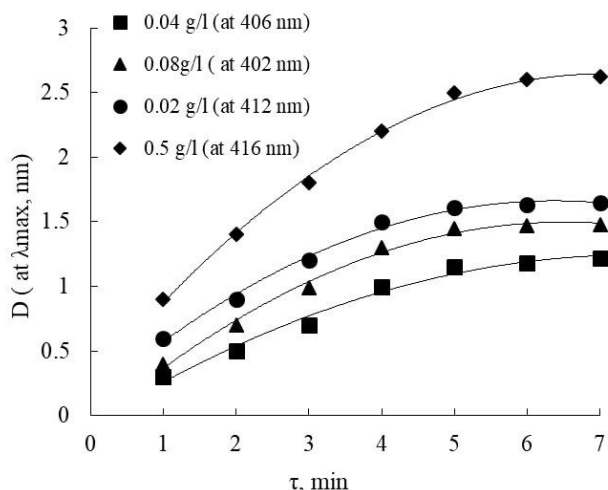


Fig. 2. Dependence of absorbance (D at λ_{max}) of plasma-chemically obtained colloidal solution of silver/PVA at various duration of the treatment with plasma discharge (τ) for various concentration of silver

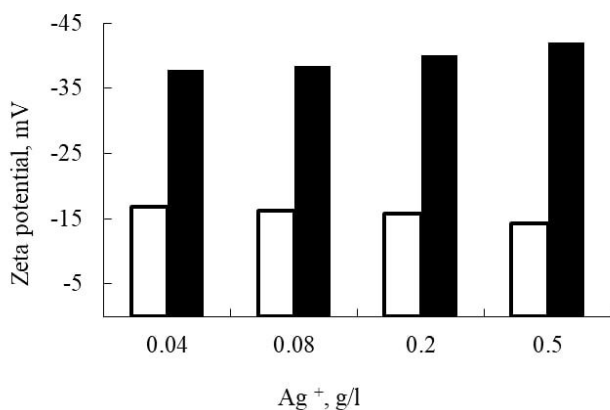


Fig. 3. Dependence of zeta potential of plasma-chemically obtained colloidal solution of silver on various concentration of Ag^+ without using stabilization agent (white columns) and with PVA (black columns)

a zeta potential less than -20 mV is considered unstable and will result in particles settling out of solution in the absence of other factors.

At different initial concentrations of Ag^+ ions in the solution without any stabilizer, there was a little variation in the zeta potential value of AgNPs, indicating the instability of the synthesized nanoparticles. In overall, all obtained samples solutions of silver nanoparticles/PVA depending on the initial Ag^+ and concentration of stabilizer are characterized by the average value of zeta potential in the range from -37.8 to -42.1 mV, which is typical of the stable colloidal systems.

The data of zeta potential analysis are confirmed by UV-vis spectroscopy data (Fig. 4). The analysis of spectra of silver colloids after aging during 4 weeks passing from the date of their obtaining showed no significant changes in the structure and intensity of SPR bands, thus evidencing the absence of noticeable (quick) aggregative processes in silver dispersions.

In order to investigate the role of PVA in stabilizing the nanoparticles, we also varied the molecular weight of PVA, which influences the average size of the silver nanoparticles. The average size of the silver nanoparticles is given in Tables 1 and 2.

Table 1

The amounts of PVA and $AgNO_3$ in each sample when the weight percent of PVA is varied

Weight percent of PVA (g/L)	Concentration of $AgNO_3$ (g/L)	Average size of silver nanoparticles (nm)
1.5	0.5	71.6
2.5		64.1
5.0		61.0
10		58.4

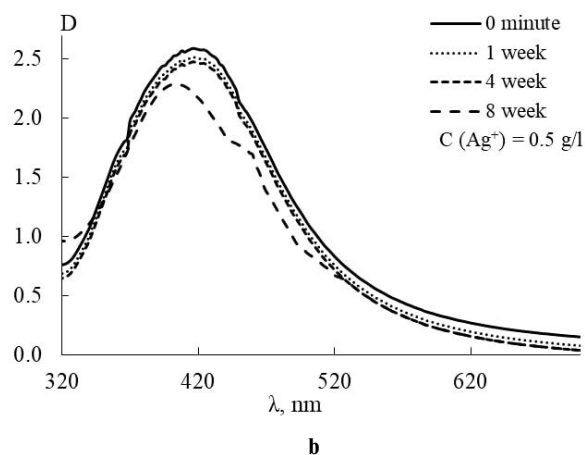
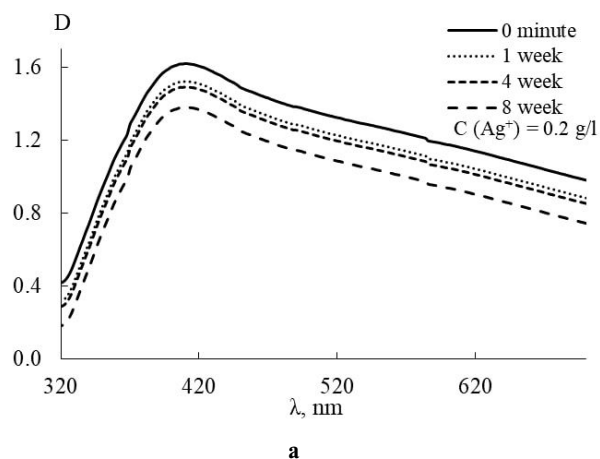


Fig. 4. Dependence of intensity of absorbance of plasma-chemically obtained colloidal solution of silver on wavelength at various duration of storage after action of discharged plasma

Table 2
The amounts of PVA and AgNO₃ in each sample when the molecular weight of PVA is varied

Molecular weight of PVA (g/mol)	Concentration of AgNO ₃ (g/L)	Average size of silver nanoparticles (nm)
14,000	0.5	57.2
40,000		58.4
127,000		81.1
1,200,000		92.1

Note: The content of PVA is constant at 10.0 g/l in all samples.

The average size of the silver nanoparticles decreases with an increase in the amount of PVA in the solution (Table 1). However, if the percentage by weight of PVA in the solution is constant and equal to 10.0 g/L, the nanoparticle size increases with increasing the molecular weight of PVA (Table 2). Thus the size of the particles fabricated by means of plasma-chemical treatment is related to the length of the stabilizing PVA polymer's chains: the longer the polymer chains, the greater is their inhibitory action on the aggregation of the particles. The results indicate that the number of silver ions that can interact with a molecule of PVA decreases as the amount of PVA in the solution increases. As a result, the number of silver atoms presented at close range decreases, and so the average size of the silver nanoparticles decreases. For a constant percentage by weight of PVA (10 g/L), the number of total repeating units of PVA is the same for various molecular weights. However, PVA with a molecular weight of 1,200,000 has more repeating units per molecule than PVP with a molecular weight of 14,000. So, PVA with a higher molecular weight can interact with more silver ions per molecule. Because there are more silver ions in close proximity within a PVA molecule with a molecular weight of 1,200,000 than in PVA chains of lower molecular weight, the use of PVP with a molecular weight of 1,200,000 results in final silver nanoparticles of larger size.

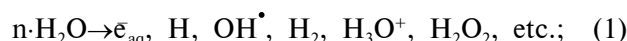
The X-ray diffraction (XRD) profile of AgNPs is depicted in Fig. 5,a. One can see all prominent peaks at respective 2θ values for zero valent FCC silver, representing (111), (200), (220) crystal planes due to Bragg's reflection at $2\theta = 38.06^\circ$, 44.24° , 64.66° . Here, the conditions of experiment were as follows: concentration of silver nitrate was 0.5 g/L and the concentration of PVA was 10.0 g/L.

The SEM images and size distribution curves of the prepared AgNPs are shown in Fig. 5,b and 5,c). The morphology of the resulting sample

indicates that relatively spherical nanoparticles are formed with an average diameter of silver of up to 100 nm.

Thus, the plasma-chemical formation of silver nanoparticles in the presence of PVA is an effective method of preparing stable colloidal solutions of silver nanoparticles. The possible mechanism of their formation is presented below.

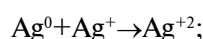
Energetic electrons deposit energy in the medium through various processes, among which the most prominent processes are the ionization and excitation of atoms, the breaking of chemical bonds, cross-linking, the disintegration of molecules, etc. During discharged plasma irradiation, the following electron-induced chemical reactions and disintegration processes can be initiated in a mixture of AgNO₃ and PVA solution:



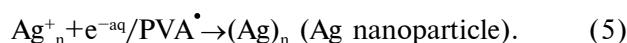
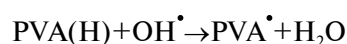
For the Ag⁺ ions, the electron capture cross section is high and, therefore, a large number of neutral (Ag⁰) silver atoms can be produced in the solution through the following reaction:



The neutral Ag atoms can encounter the excess Ag⁺ ions and produce Ag⁺² species, which progressively leads to the formation of silver nanoparticles in the solution through the following reactions:



Moreover, the reaction leading to the reduction of Ag⁺ ions can proceed through the strongly reducing hydrated electrons as well as by the polymeric radicals PVA[•]. During electron irradiation, the following chemical reactions can be induced:



In this manner, during electron irradiation, the size of the silver particle can grow and subsequently silver particles of nano-sized dimensions can be synthesized in the solution.

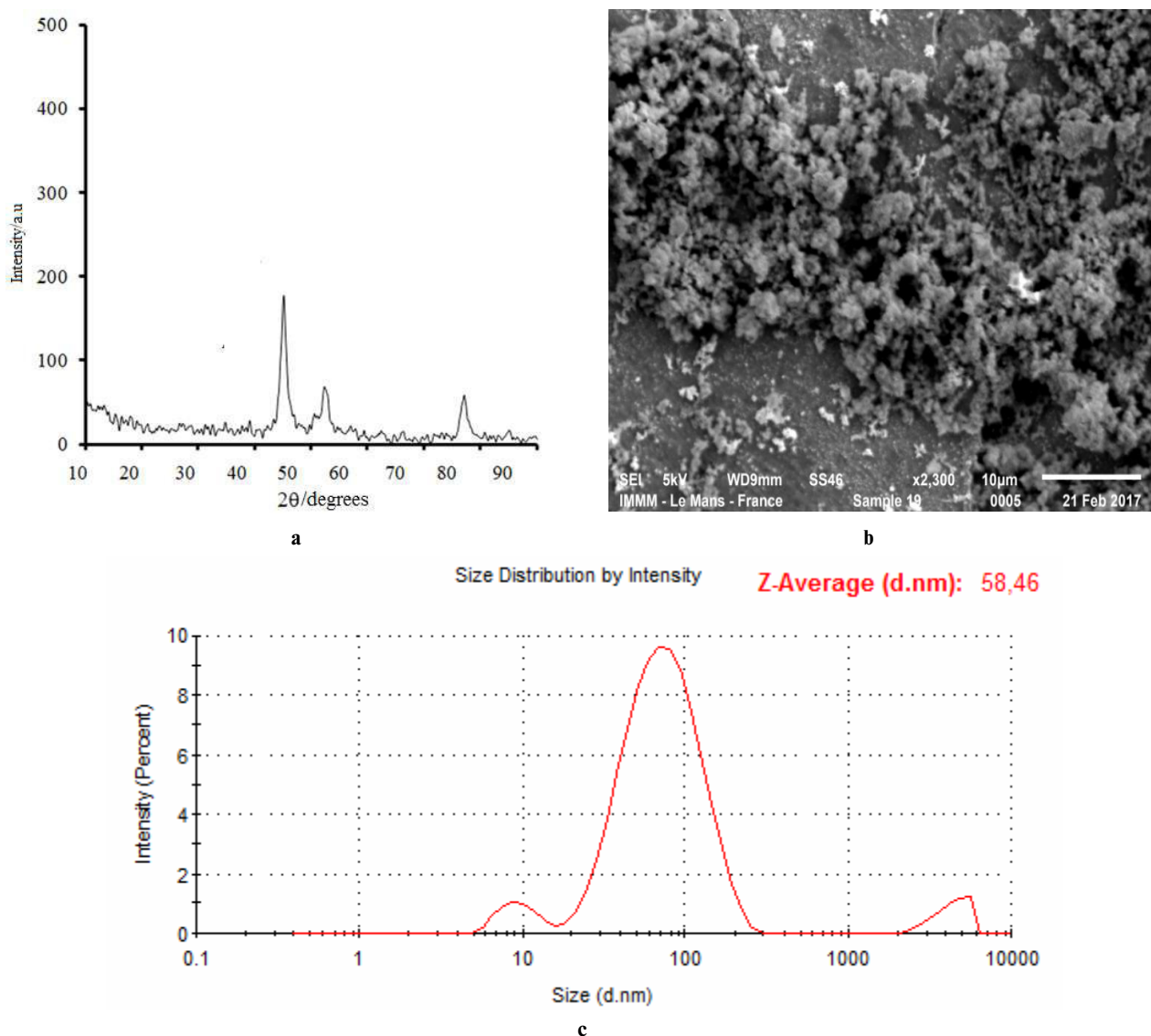


Fig. 5. X-ray diffraction pattern (a), SEM-image (b) and size distribution (c) of plasma-chemically obtained nanoparticles of silver in the presence of PVA. $C(\text{Ag}^+) = 0.5 \text{ g/L}$, $C(\text{PVA}) = 10.0 \text{ g/L}$, $I = 120 \text{ mA}$, $P = 0.08 \text{ MPa}$, $\tau = 5 \text{ min}$

Conclusions

Silver nanoparticles were prepared in aqueous AgNO_3 solution by using discharged plasma and polyvinyl alcohol as a stabilizer. According to our findings, the introduction of stabilizer promotes an increase in the intensity of the formation of silver nanoparticles. The effects of Ag^+ concentration, the amount and the molecular weight of PVA, and the duration of treatment with plasma discharge on the formation of silver nanoparticles as well as the aggregative stability of silver nanoparticles obtained using plasma-chemical method were studied. The content of PVA in the reaction medium ($2.5\text{--}10.0 \text{ g L}^{-1}$) is sufficient for nanoparticles stabilization, depending on the initial concentration of Ag^+ ions ($0.04\text{--}0.5 \text{ g L}^{-1}$). The results showed that both the

amount and the molecular weight of PVA in the irradiated solution considerably affect the average size of the silver nanoparticles. The average size of the silver nanoparticles decreases with increasing the amount of PVA in the solution, but increases with increasing its molecular weight. The content of nanoparticles grows with the increase of duration of plasma action on the solution; processing during 5 min provides the formation of silver nanoparticles for different concentration of Ag^+ ions in solutions. The results indicated the formation of nanometer-sized particles. The silver nanoparticles prepared in this way are uniform and stable in solution over a period of 2 months at room temperature (25°C) and show no signs of aggregation.

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ОДНОСТАДІЙНИЙ СИНТЕЗ НАНОЧАСТОК СРІБЛА З ВИКОРИСТАННЯМ РОЗРЯДЖЕНОЇ ПЛАЗМИ В ПРИСУТНОСТІ ПОЛІВІНІЛОВОГО СПИРТУ

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Розроблено простий метод одержання наночастинок срібла (НЧ Ag) із використанням розрядженої плазми в присутності стабілізатора (полівінілового спирту, ПВС). На спектрах НЧ Ag/ПВС присутній пік при 400–440 нм, що свідчить про утворення наночастинок срібла. Досліджено спектральні характеристики поглинання УФ-випромінювання наночастинок срібла, одержаних при різних концентраціях Ag⁺, кількості і молекулярній масі ПВС, тривалості дії плазмового розряду. Вміст ПВС в реакційній середовищі (1,25–10,0 г/л) є достатнім для стабілізації наночастинок в залежності від початкової концентрації Ag⁺ (0,04–0,5 г/л). Результати показали, що середній розмір наночастинок срібла зменшується зі збільшенням кількості ПВС в розчині, але зростає зі збільшенням його молекулярної маси. Обробка плазмою протягом 5–6 хв є достатньою для формування наночастинок срібла в досліджуваних концентраціях Ag⁺. Характеристики НЧ Ag проводили з використанням скануючої електронної мікроскопії, рентгенівської дифракції, аналізу дзета-потенціалу. Результати показали формування сферичних нанорозмірних частинок. Рентгеноструктурний аналіз показав металеві частинки з гранецентрованою кубічною решіткою. Одержані таким чином наночастинки стабільні в водно-му розчині протягом двох місяців при кімнатній температурі.

Ключові слова: срібло, наночастинки, розряджена плазма, полівініловий спирт, стабілізація.

ONE-POT SYNTHESIS OF SILVER NANOPARTICLES USING DISCHARGED PLASMA IN THE PRESENCE OF POLYVINYL ALCOHOL

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