

THE PLASMA-INDUCED FORMATION OF PVP-COATED SILVER NANOPARTICLES AND USAGE IN WATER PURIFICATION

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Abstract. The contact non-equilibrium low-temperature plasma technique is used to synthesize silver nanoparticles (AgNPs) employing polyvinyl pyrrolidone (PVP) as a capping agent. Influences of PVP concentration on the formation efficiency of silver nanoparticle, their average size and stability have been studied. The synthesized silver nanoparticles had a significant antibacterial activity against two strains of Gram bacteria. Silver nanoparticles (AgNPs)-alginate composite beads with different PVP concentration were synthesized as materials for water purification.

Keywords: silver nanoparticles, plasma, poly(N-vinylpyrrolidone), composite materials, antibacterial.

1. Introduction

Unique optical, electrical and excellent catalytic properties of metal nanoparticles have spurred interest in developing different synthetic methodologies for diverse applications. Among these, the silver nanoparticles have attracted considerable attention because of their potential applications in various fields such as oxidative catalysis [1], surface enhanced Raman scattering [2], nano-electronics (single-electron transistors, electrical connects) [3], conductive coatings, biosensors [4], antibacterial activity [5], *etc.* Considering their applications in various fields, many techniques of synthesizing silver nanoparticles have been investigated. Some of them are chemical reduction, electrochemical reduction, photochemical reduction, microemulsion, γ -ray irradiation, UV-irradiation, microwave and ultrasonic [6]. One of the innovative and environmentally safe methods for preparation of nano-sized compounds is the use of plasma discharges of various configurations: plasma discharge

generated between the electrodes immersed in the liquid, at gas-liquid phase interface at reduced pressure, plasma at the atmospheric pressure in the interaction with the liquid [7].

Among plasma-chemical discharges, contact non-equilibrium low-temperature plasma (CNP) is a promising option from the point of view of practical application [8]. Plasma discharge is generated between the electrode in the gaseous phase and the surface of the liquid where the other electrode is located. Therefore, chemical transformations at the phase interface are conditioned by the combined effect of electrochemical oxidation-reduction; initiated photolysis reactions, UV radiation; flow of charged particles from the gaseous phase to the surface of the liquid medium. 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]. Previously it was shown the efficiency of using the contact non-equilibrium low-temperature plasma in comparison with the conventional method of chemical reduction in solutions and photochemical deposition [9, 10]. The efficiency of CNP use for synthesis of silver nanoparticles from the aqueous solutions of metal salts one-shot in the presence of sodium alginate, citrate, PVA (polyvinyl alcohol) is demonstrated in other published works [11-13]. Therefore, this synthesis technique seems to have potential for metal nanoparticles synthesis.

Capping agents are frequently used in colloidal synthesis to inhibit nanoparticle overgrowth and aggregation as well as to control the structural characteristics of the resulted nanoparticles in a precise manner. A number of chemicals can be used as protecting agents in the synthesis of AgNPs. However, the most commonly applied stabilizers and protective agents in nanoparticles synthesis are polymers: gelatin [14], D-sorbitol, polyvinyl pyrrolidone (PVP) [15] and PMVE (poly(methylvinylether)) [16]. Among all polymer stabilizers of silver nanoparticles, poly(N-vinylpyrrolidone) is considered an excellent dispersant as it exhibits favourable protecting properties owing to its unique structure [17, 18]. PVP is a homopolymer with a polyvinyl backbone and its repeating units contain a highly polar amide group that confers

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hydrophilic and polar-attracting properties, and also non-polar methylene groups both in the backbone and in the ring that confer hydrophobic properties [19, 20]. N and O in the polar groups have a strong affinity for silver ions and silver nanoparticles. PVP by its structural features is used in a wide variety of applications in medicine (*e.g.* as a drug carrier, a component of plasma substitute or wound dressing) and technological domains (*e.g.* in aerosol hair sprays, pigment dispersions, cosmetics) [19-23], in different types of porous composites [24, 25]. Therefore, studying the process of obtaining silver nanoparticles in the presence of PVP, used as a stabilizing reagent, under the action of plasma discharge, is of scientific and practical interest. It is reported that to practical applications AgNPs must be embedded in or adsorbed on various inorganic or organic substrates, including filter materials made of zeolite, silica or fiberglass, natural macro-porous materials, carbon materials, paper and polymers of different types [26]. While these composite materials have been shown to be qualitatively antibacterial, there are limited systematic investigations on the bactericidal efficacy of these composite materials for the purpose of drinking water treatment.

The objective of this work is the study of plasma-chemical obtaining of silver nanoparticles colloidal solutions using poly(N-vinylpyrrolidone) as a capping agent and investigation of their antibacterial activity for water purification.

2. Experimental

2.1. Materials

Silver nitrate (99.8 %, Kishida), polyvinyl pyrrolidone (PVP), MW \approx 8,000), sodium alginate (AlgNa) were purchased from Merck Co. Ltd. (Darmstadt, Germany). Aqueous solutions of silver nitrate, PVP and AlgNa were prepared using ultrapure water (Direct-Q UV, Millipore) and were utilized as starting materials without further purification.

2.2. Synthesis of Silver Nanoparticles

AgNPs

The nanosized silver colloids were prepared by a plasma chemical process in aqueous solution in the presence of PVP as a stabilizing agent. PVP-coated silver nanoparticles were synthesized using a synthesis reactor [8-12]. AgNO₃ was dissolved in double distilled water to give a solution of 0.5 g/l. PVP was dissolved in water heated to 363 K to give a solution of 1.0–5.0 g/l. 2.5 ml of PVP solution were added to 40 ml of silver nitrate solution. The resulting reaction mixtures have 0.5; 1.0; 1.5 and 2.5 % of PVP. The resulting reaction mixture was

treated in the reactor with the discharge of contact non-equilibrium low-temperature plasma with fixed parameters (pressure, current strength, voltage). The parameters of plasma: $I = 120$ mA, $P = 0.08$ MPa. The electrodes were directly connected to the lines in order to apply voltage from the power sources. After plasma irradiation treatment, the resultant colloidal solutions were naturally cooled to room temperature. The final product was obtained as a colloidal dispersion. The change in color of a mixture of AgNO₃ to brown indicates the synthesis of silver nanoparticles. The strong SPR band at 400–440 nm in UV-Vis spectra thus confirms the formation of silver nanoparticles. AgNPs obtained by plasma discharges were centrifuged at 5000 rpm for 5 min. The dried powders were then used for further characterization.

2.3. Equipment

The research was carried out at the laboratory of plasma-chemical technologies of the Ukrainian State Chemical Technology University (Ukraine) on an installation of a discrete type with a reactor volume of 0.1 l. Cathode (diameter 4 mm, 18H10T stainless steel electrodes) was located in the liquid part, with the anode (diameter 2.4 mm) placed at the distance of 10 mm from the solution surface. Volume of the solution in the reactor was equal to 70 ml. Cooling of reaction mixture was ensured by continuous circulation of cold water. Pressure in the reactor was maintained at 80 ± 4 kPa. The voltage of 500–1000 V was applied to the electrodes to obtain the plasma discharge. The current strength was maintained at the level of 120 ± 6 mA.

2.4 Characterization Techniques

Spectra of colloidal solutions were obtained by means of spectrophotometer UV-5800PC using quartz cuvettes in the wavelength range of 190–700 nm (FRU, China). 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). Microphotographs of nanoparticles and particle sizes were obtained on a transmission electron microscopy JEOL TEM (Model 100 CX II; Tokyo, Japan). Disperse phase of the solution obtained as a result of plasma-chemical treatment of the solution and dried in the air at 298 K was studied using X-ray diffractometer Ultima IV Rigaku.

2.5. Antimicrobial Activity Studies

The measurement of colony-forming unit (CFU) method was used to study the antibacterial activity of the synthesized silver nanoparticles. PVP-coated silver nanoparticles were added in the spinning solution which

contains about $1.5 \cdot 10^5$ colony-forming units (CFU) of *S. aureus* and *E. coli*, respectively. The mixtures were cultured at 310 K in a shaking incubator for 12 h. Silver nitrate was also tested as a blank control and positive control, respectively. 100 ml of each of these cell solutions was seeded onto LB agar using a surface spread plate technique. The plates were incubated at 310 K for 24 h. Then the numbers of bacterial colonies (CFU) were counted. The counts were used to calculate the surviving number of bacteria [27]. Growth inhibition rate was calculated according to the following equation: $GIR = \{(\text{CFU/ml of control medium}) / (\text{CFU/ml of silver nanoparticles solution treated medium})\} \cdot 100$.

2.6. Synthesis of Silver/PVP/Alginate Composite Materials and Column Disinfection Experiment

Pre-synthesized 4 types of silver nanoparticles (AgNPs) coated with different concentration of stabilization agent (0.5–2.5 %) were incorporated into the alginate beads during the gelation. AgNPs were thoroughly mixed with a suspension of 20 g/l alginate at Ag/alginate ratio of 0.3 % (w/w). The mixture was injected into a solution of 0.3M CaCl_2 at a flow rate of 3 ml/min. The resulting beads were flushed with DI water three times with the assistance of a vacuum filter. To quantify the antibacterial effect of the composite beads, we conducted column experiments in which *E. coli* suspension was filtered through a chromatography column packed by the composite beads. Immediately after being collected, the *E. coli* samples were subjected to serial dilutions followed by plating on agar substrates. The agar plates with *E. coli* homogeneously spread on the surface were then placed into an incubator at 310 K overnight to allow the growth of visible colonies. The efficacies of these beads produced using gelation approaches of synthesis are compared, in all cases using alginate beads without AgNPs as a control.

3. Results and Discussion

The AgNP formation in all reaction mixtures was preliminarily detected by visual observation of the color changes of the reaction solutions. These color changes were attributed to the excitation of surface plasmon resonance (SPR) in the metal nanoparticles [28]. The absorbance intensities of the UV-Visible spectra provide insight into the reduction of silver ions. The SPR band appeared in UV-vis spectra is affected by the size and the shape of the synthesized AgNPs.

Influence of PVP on the formation efficiency of silver nanoparticle solutions was followed by UV-vis absorption measurement. Fig. 1 shows UV-vis spectra of

colloidal solutions obtained in the CNP plasma-irradiated AgNO_3 without PVP and with its various concentrations at the fixed silver nitrate concentration of 0.5 g/l and plasma irradiation for 4 min. Analysis of obtained data showed that the nanoparticles are formed without using PVP as a result of plasma discharge action on the solution of silver nitrate. Colloidal solutions of silver are characterized by the presence of SPR absorption maximum (λ_{max}) at 424 nm. When different amounts (0.5–2.5 %) of PVP are introduced into the reaction mixture before its treatment with CNP, a blue shift is observed in the λ_{max} of the plasmon band from 424 to 378–400 nm along with the increase in intensity. The increase in intensity of UV absorption spectra indicates the increase in number of silver nanoparticles with increasing PVP concentration. The peak shift may be attributed to the change in particle size. Further increase in PVP concentration from 1.5 to 2.5 % results in the decrease in the peak intensity; the peak is not shifted toward longer wavelength at higher PVP concentrations, which suggests the formation of less concentration particles. The increase in the polymer concentration could induce conflicting effects on the particle size. At higher polymer concentrations, more polymer molecules can cap AgNPs to decrease the particle size. While the polymer acts as a reductant, it accelerates the nucleation and growth of AgNPs to produce larger particles at relatively high polymer concentrations [29].

The synthesis was also monitored for various time intervals of the reaction medium treatment with the plasma discharge. Fig. 2 shows the UV-vis spectra of AgNPs obtained after different irradiation times. The data in Fig. 2 reveals several important findings which can be presented as follows: (i) for all samples at the early stage of reaction duration (< 20 s), the plasmon band is broad and weak indicating low conversion of Ag^+ to AgNPs at this irradiation duration; (ii) further increase in the irradiation time to 4 min (for 0.5–2.5 % PVP) and to 2 min (for 1.5 % PVP), there is a gradual increase in the absorption intensity without little red shift in the peak wavelength, which indicates that the AgNPs content increases with the increased irradiation time; the mean diameter of AgNPs does not change much. The increase in the exposure time to 5 min is accompanied by the decrease in intensity of UV absorption spectra; (iii) the strongest SPR band occurs at 400 nm (0.5 % PVP), 384 nm (1.0 % PVP), 394 nm (1.5 % PVP) and 390 nm (2.5 % PVP) implying that large amounts of silver ions are reduced and used for silver particles formation; (iv) as can be seen from the absorption intensity of SPR, the particle size does not continue to get smaller by changing the PVP concentrations from 0.5 to 2.5 % because PVP at the solid/liquid interface does not interfere with the silver diffusion-surface deposition process since the particles grow to a definite size. It is further confirmed by the particles size measurements results.

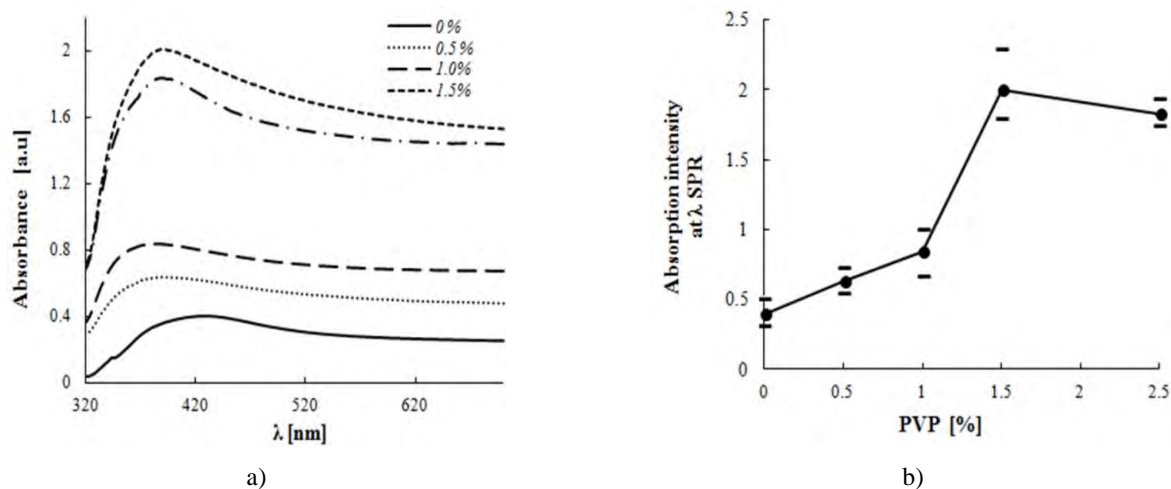


Fig. 1. The UV-vis absorption spectra of silver nanoparticles/PVP (a) and absorption intensity SPR band (b) prepared by discharge plasma at various concentrations of PVP

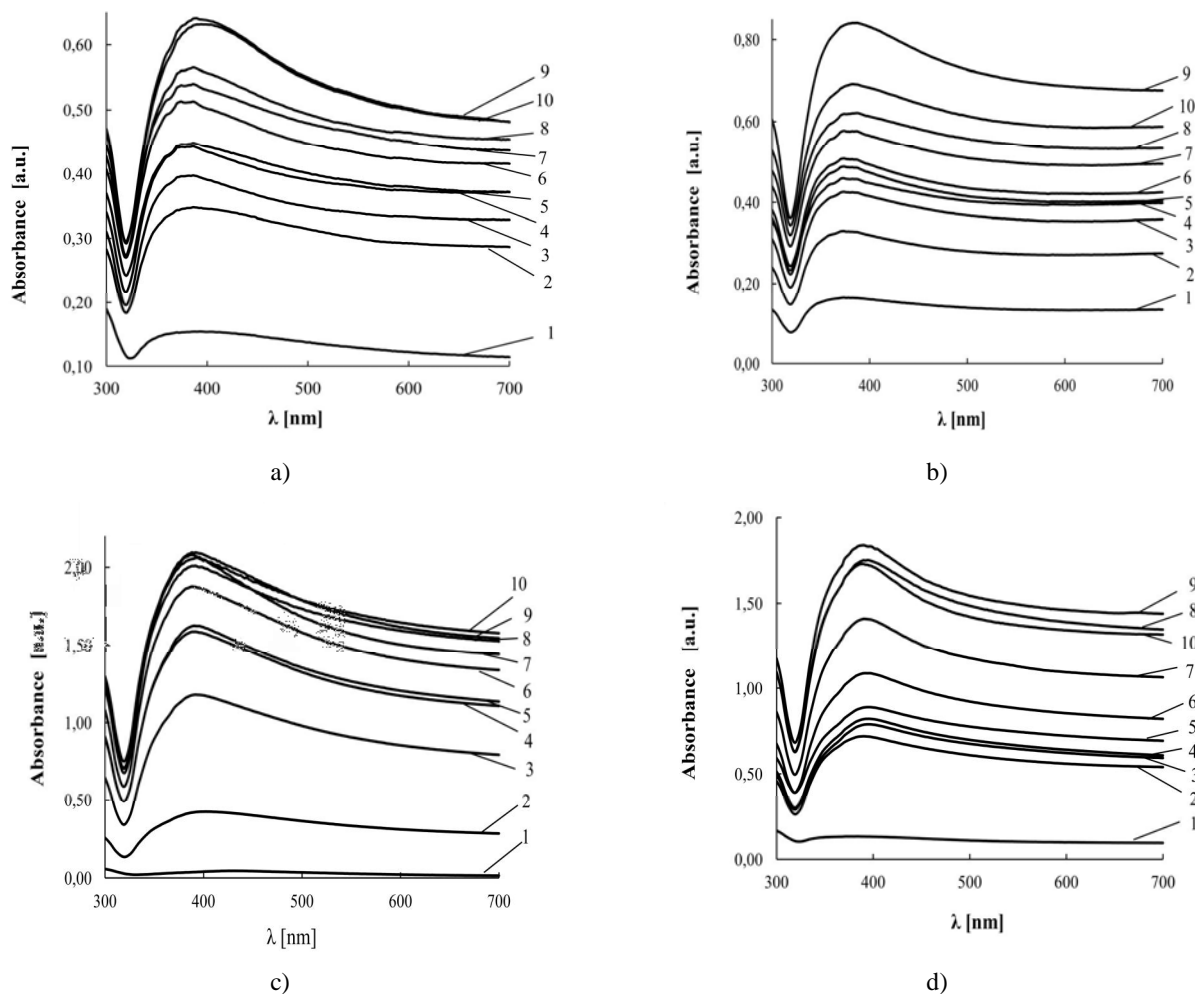


Fig. 2. UV-vis spectra of aqueous solution AgNO_3/PVP treated with plasma at various concentrations of PVP (%): 0.5 (a); 1.0 (b); 1.5 (c) and 2.5 (d). Irradiation time : 10 s (1); 20 s (2); 30 s (3); 40 s (4); 50 s (5); 1 min (6); 2 min (7); 3 min (8); 4 min (9) and 5 min (10)

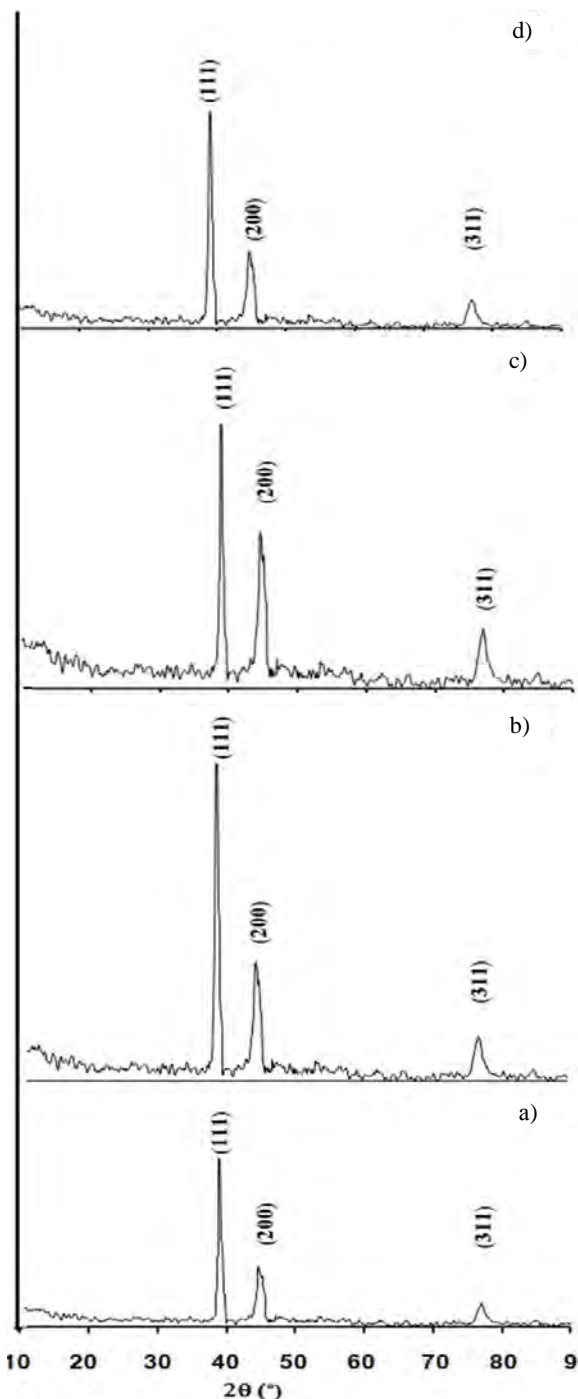


Fig. 3. X-ray diffraction pattern of silver nanoparticles prepared by discharge plasma at different concentrations of PVP (%): 0.5 (a); 1.0 (b); 1.5 (c) and 2.5 (d)

The formation of metal silver was also confirmed by powder X-ray diffraction (XRD) analysis. The conditions of the experiment were as follows: the parameters of plasma $I = 120$ mA, $P = 0.08$ MPa, $\tau = 5$ min, $C(\text{AgNO}_3) = 0.5$ g/l. Fig. 3 shows typical XRD pattern of silver nanoparticles obtained in an aqueous solution: peaks at 2θ

values of 38.1, 44.9, 77.5 deg can be attributed to (111), (200), (311) crystalline planes of the face centered cubic crystalline structure of metallic silver. The intensity of peaks reflected the high degree of crystallinity of the silver nanoparticles. The size of the particles actually formed in the aqueous solution under plasma discharge conditions was determined. To do this, the study of particle size distribution was performed, and the average particle size (Table 1) of plasmochemically prepared silver dispersions at different initial concentrations of PVP was determined. The TEM images and size distribution of the prepared Ag/PVP (same sample C (Ag^+) = 0.003 mol/l) are shown in Fig. 4. As a result of the plasmochemical impact, silver nanoparticles with a wide size distribution (7–150 nm) are formed. The data obtained (Table 1) indicate that the average diameter of the nanoparticles formed under plasma discharge impact is 40.0–48.4 nm and not significantly increases with increasing initial concentration of PVP.

Zeta potential measurements were conducted to evaluate the stability of the silver suspensions (Table 1). 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 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 concentration of Ag^+ in the solution without a stabilizer, there was a little variation in the zeta potential value of AgNPs, indicating low stability of the synthesized nanoparticles. In overall, all obtained samples solutions of silver nanoparticles/PVP depending on the initial concentration of the stabilizer, are characterized by the average value of zeta potential in the range from -20.1 to -21.3 mV, which is typical of the stable colloidal systems [30].

It is commonly known and well established that silver nanoparticles show antibacterial activity [31]. The quantitative characterization of the antibacterial activity of the synthesized nanoparticles is appropriate. The plasma chemical synthesized AgNPs were studied for their antibacterial activity. The anti-bacterial activities (ABE) of the Ag/PVP solutions against *S. aureus* and *E. coli* are shown in Table 2, and table data indicate that the Ag/PVP (1.5 %) solution has the highest ABE and this activity is quite strong.

The ABE against *E. coli* is lower compared with that against *S. aureus*, probably because of the difference in cell walls between Gram-positive and Gram-negative bacteria. The cell wall of *E. coli*, which consists of lipids, proteins and lipopolysaccharides (LPS), provides effective protection against biocides. However, the cell wall of Gram-positive bacteria, such as *S. aureus*, does not consist of LPS [32–34].

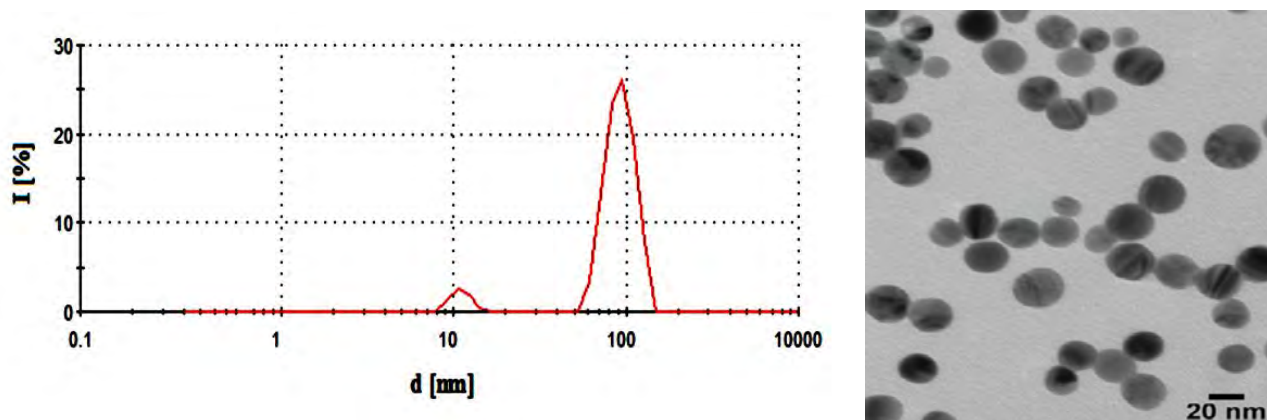


Fig. 4. Size distribution and TEM images of plasma-chemically obtained nanoparticles of silver/PVP

Table 1

Size and zeta potential of silver nanoparticles prepared in plasma treatment conditions

PVP, %	Average size of AgNPs, nm	Zeta potential, mV
–	60.1	–14.2
0.5	41.3	–20.1
1.0	44.1	–20.3
1.5	40.0	–21.3
2.5	48.4	–20.1

Table 2

Growth inhibition rates of silver nanoparticles/PVP

Tested bacterial strains	Growth inhibition rate, %				
	Blank	Ag ⁰ (0.5 g/l)/PVP			
		0.5	1.0	1.5	2.5
<i>Escherichia coli</i>	0	89	90	92	90
<i>Staphylococcus aureus</i>	0	92	94	98	93

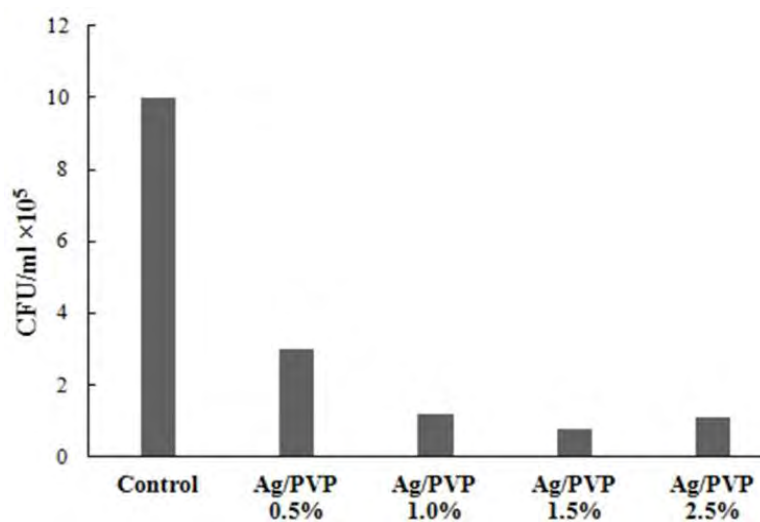


Fig. 5. The dependence number of colony forming unit per ml sample plated (columns) from the amount stabiliser agent of pre-synthesized AgNPs

Silver nanoparticles-alginate composite materials were synthesized as filler materials of packed columns for a simultaneous filtration disinfection as an alternative portable water treatment process. Alginate was chosen as the immobilization/delivery material due to its natural abundance and biocompatibility. These AgNP-alginate composite beads were packed to form porous columns through which bacteria containing water were passed. The results of the column experiments are presented in Fig. 5.

Composite beads prepared using different stabilisators were found to be effective in disinfecting the *E. coli* to different extents. With silver-alginate composite beads, the disinfection efficiency is high even with retention time up to 10 min. Different efficiency of composite samples obtained with different PVP concentration indicates different degrees of antibacterial action of plasma-chemically synthesized silver nanoparticles. In addition, this may be due to the different structural formation of the composite and the release mechanisms of silver ions [34, 35].

4. Conclusions

Silver nanoparticles were prepared in aqueous AgNO₃ solution by using the contact non-equilibrium low-temperature plasma and PVP as the capping agent. Effect of PVP concentration on the formation efficiency of silver nanoparticle, their average size and stability has been studied. The formation of silver colloidal solutions in the presence of the capping agent is characterized by the presence of peak $\lambda_{\max} = 380\text{--}400$ nm in the spectra. According to the results of our findings, introduction of the stabilizer promotes the increase in intensity of silver nanoparticles formation. The average size of formed silver particles is up to 100 nm. The stability of silver nanoparticles solutions was studied. Zeta-potential of plasma-chemically obtained colloidal solutions at various concentrations of the stabilizer varied from -20.1 to -21.3 mV. The content of nanoparticles increased with the increase of duration of plasma action on the solution; processing during 4 min provided formation of silver nanoparticles. The formation of silver metal particles was confirmed by X-ray diffraction analysis. The synthesized silver nanoparticles had the significant antibacterial activity against *Escherichia coli* and *Staphylococcus aureus*. Silver/alginate composite materials were successfully prepared using different concentrations of PVP. Composite beads prepared using different amount of PVP were found to be effective in disinfecting the *E. coli* to different extents. Specifically, composite materials were able to achieve a disinfection efficiency with 10 min retention time, indicating that the column packed with

these Ag-alginate composite beads may be a viable means for portable water purification.

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ПЛАЗМА-ІНІЦІЙОВАНЕ ОДЕРЖАННЯ ПОКРИТИХ ПВП НАНОЧАСТИНОК СРІБЛА ТА ЇХ ЗАСТОСУВАННЯ ДЛЯ ОЧИЩЕННЯ ВОДИ

Анотація. За допомогою контактної нерівноважної низькотемпературної плазми одержані наночастинки срібла (AgНЧ) із застосуванням полівінілпіролідону (ПВП) як стабілізуючого агенту. Вивчено вплив концентрації ПВП на ефективність формування наночастинок срібла, їх середній розмір та стабільність. Встановлено, що одержані наночастинки срібла проявляють антибактеріальну активність проти двох штампів грам-бактерій. Одержано композитні гранули (AgНЧ-альгінат) з різною концентрацією ПВП для очищення води.

Ключові слова: наночастинки срібла, плазма, полівінілпіролідон, композитний матеріал, антибактеріальний.