

# GREEN SYNTHESIS OF SILVER NANOPARTICLES AND THEIR SPECTRAL PROPERTIES

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Spherical silver nanoparticles were synthesized by the chemical condensation method using aloe vera or chamomile extracts as a reducing agent. Depending on the type of extracts and its concentration, the size of AgNpcs varied from 7 to 50 nm by simply adjusting the ratio of the starting reagents. These extracts show reducing properties due to the presence of carbonyl compounds in their composition, in particular organic acids. It is shown that regulation of concentrations and synthesis conditions allows control of particle size. A change in the synthesis conditions affects the position of the surface plasmon resonance band, and therefore the optical properties of the studied systems. It has been proven that the synthesized silver nanoparticles do not require additional stabilization. Agglomeration processes occur only with a significant increase in concentration and heating time.

**Keywords:** silver nanoparticles, optical properties, dispersion, aloe vera, chamomile.

**INTRODUCTION.** The development of nanotechnologies is constantly moving forward, and the creation of new methods of synthesis of nanoparticles of noble metals, or the improvement of existing ones, by replacing raw materials in order to optimize and reduce the price of final products, is an urgent task of modern science [1–3]. It has been discovered that silver nanoparticles (AgNPs) stop the growth and reproduction of many bacteria, such as *Bacillus cereus*, *Staphylococcus aureus*, *Citrobacter koseri*, *Salmonella typhii*, *Pseudomonas aeruginosa*, *Escherichia coli*, *Klebsiella*

*pneumonia*, *Vibrio parahaemolyticus* and the fungus *Candida albicans*, which defines their use in medicine, cosmetic industry and biotechnology. AgNPs have other physico-chemical properties, in particular, high electrical and thermal conductivity, surface combined scattering, chemical stability, catalytic activity, and nonlinear optical behavior [4]. These properties allow them to be used in inks, microelectronics and medical imaging [5–6], renewable energy sources, etc. Today, AgNPs are introduced into the composition of plastics, soaps, toothpastes, food and textile,

which is a marketing ploy and allows increase their market value [7–9].

The relevance of this research is evidenced by numerous publications on the synthesis and research of the properties of silver nanoparticles. The synthesis of dispersed systems is based on condensation and dispersion methods. Among the physical methods, the evaporation-condensation approach and the laser ablation technique are popular. These methods make it possible to obtain monodisperse systems of high purity, and exclude additional loading of the system with a solvent. Of the chemical methods, the main and most common is the method of chemical condensation. Sodium citrate, sodium borohydride, EDTA, glycerin, ethylene glycol, glucose, etc. are used as reducing agents [6,10–14]. The use of plant extracts as sources of reducing substances for the synthesis of AgNPs attracts attention due to the environmental friendliness and practicality of this method [1, 2, 12–21]. The main advantage of this synthesis is a natural reducing agent that simultaneously acts as a stabilizer. Waste from the processing of fruit and berry crops and plant raw materials is a promising source of substances (ascorbic acid, polyphenols, carbohydrates, amino acids, glycosides, and others) for use in the processes of obtaining metal nanoparticles. The studied extracts of aloe and chamomile include flavonoids (rutin, apigenin, quercetin, luteolin, etc.), glycoproteins, some acids, vitamins B, C, E, beta carotene [15, 16, 22–29]. Nanoparticles of the required size can be obtained by selecting a plant extract. The disadvantages of this method include the presence of secondary compounds and metabolites, temperature limitations characteristic of enzymes, but the advantage is the stability of such systems. Of course, each method has

its advantages and disadvantages, which can be avoided by analyzing the studied systems in detail. The choice of method depends on the field of further application of the obtained AgNPs. If we focus on the application of silver nanoparticles in the cosmetic and medical industry, the green method of synthesis is of particular interest. It is not only a modern and promising method of synthesis, but also avoids the use of additional stabilizers, which reduces the chemical and toxic load on the body, skin or hair. Thus, nanosystems obtained by reducing silver with natural extracts are biologically active dispersed systems of a wide range of applications.

The choice of the extract depends on the field of application of the synthesized nanoparticles. There are already many products on the market of cosmetic products, including creams, shampoos, lotions, shaving gels, which contain AgNPs, which increases their antibacterial activity. The idea of this work is the synthesis and additions of AgNPs into the composition of hair dye in order to intensify the color and reduce the irritation of the scalp. This will confirm the thesis regarding the sensitizing action of AgNPs. Taking into account the fact that the obtained colloids are planned to be used in hair products, the extracts were selected with this in mind. Natural alcohol extracts aloe vera and *Matricaria chamomilla* with a concentration of active components  $C = 4\%$  were chosen as starting reagents.

**EXPERIMENT AND DISCUSSION OF THE RESULTS.** For the study, water-alcohol-glycerin extracts of *aloe vera*, *matricaria chamomilla* produced by the company "Vilarus" were taken. The starting solutions consist from extract 4.0%, glycerol 10%, ethyl alcohol 20%, sodium benzoate 0.08%, water >20%. One of the conditions for obtaining stable dispersed systems

is realization of synthesis in dilute solutions, which will prevent solvation of particles of the dispersed phase and rapid growth of their nuclei. Immediately before use, the extracts were diluted 10 times, so the concentration of the initial extracts for synthesis was 0.4%.

The synthesis was carried out in a water-alcohol environment. The concentration of the initial solution of argentum nitrate in ethanol was 1.0%. The initial ratio of alcoholic solution of silver nitrate: extract was 1:1 and 1:2, the concentration ratio was 2.5:1 and 5:1, respectively. 1 ml of 0.4% extract solution was added to 1 ml of 1% solution of argentum nitrate, the resulting mixture was brought up to 10 ml with water. To achieve pH=7.5, 1–3 drops (depending on the extract) of 0.1M NaOH aqueous solution were added dropwise to the mixture while stirring. After establishing the required pH level, 40 ml of boiling distilled water was added to the solution with intensive stirring and heated for 20–40 min, depending on the selected reductant extract. A sign of the formation of a colloidal silver solution is the appearance of color (the color depends on the extract). The fastest recovery occurred in the system with aloe vera extract, when heated. The resulting dispersed systems have a bright light brown color, which is due to the presence of AgNpcs in the system. All synthesized systems were investigated by the method of electronic absorption spectroscopy and by electron microscopy.

Absorption spectra of solutions were recorded on a Specord M40 spectrophotometer in the range of 300–600 nm.

The presence of a surface plasmon resonance (SPR) band in electronic absorption spectra, which is a collective vibration of metal particles smaller in size than the wavelength of electromagnetic radiation, indicates the presence

of nanoparticles in the system [13, 26–28]. Silver nanoparticles with plasmon resonance (PR) have found application in nanobiotechnology and nanomedicine. Thanks to the ability to adjust the spectral position and amplitude of the PR by changing the nature of the metal, the size, shape, structure of the particles and their dielectric environment. The latter means both the local environment formed by the adsorbed biomolecules and the global dielectric properties due to the buffer medium or metal/dielectric substrate on which the molecules can be adsorbed. Such PR changes induced by the adsorption of biomolecules are usually quite small, they are successfully used for the detection of biospecific binding of macromolecules and clinical rapid diagnostics. In addition, the shape and position of this band can be used to draw conclusions about the dispersion and morphology of the particles [26–29].

In order to investigate the influence of the nature of the extract on the dispersion of the system, 2 hours after synthesis, the electronic absorption spectra (EAS) of all samples were recorded and the SPR band was identified. The obtained result of spectral studies is in good agreement with the experiment in Fig. 1.

The figure shows the maximum intensity, the smallest half-width of the line (60 nm) and  $\lambda_{\max} = 420$  nm, which is characteristic of Ag-NPs obtained by reducing aloe (Fig. 1, curve 1). As mentioned above, recovery in this system occurred the fastest, the solution acquired a rich yellow-cognac color. This fact is apparently related to the higher content of ascorbic acid in the aloe extract compared to the chamomile extract [26] used for the study. For chamomile extract (Fig. 1, curve 4) against the background of lower intensity and shift of the band maximum by 15 nm,  $\lambda_{\max} = 436$  nm, there is also a

rather narrow line (width of 65 nm). The shift of the band maximum to the long-wavelength region may indicate the formation of larger particles. At first, a high opalescence was observed in the solutions, after which they acquired a pink color, and only after a few hours for the chamomile extract and after a day, a change in color was observed. It is obvious that the different renewable activity of the extracts is due to their different chemical composition, so the stability and dispersion of the systems requires detailed research and analysis. However, there is no doubt that the studied extracts are not only effective silver reducers, but also stabilizers of the resulting dispersed systems.

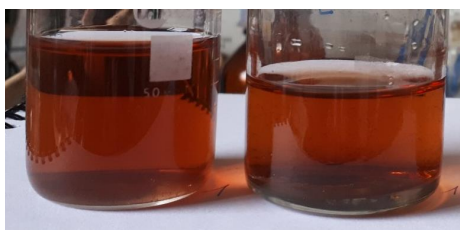
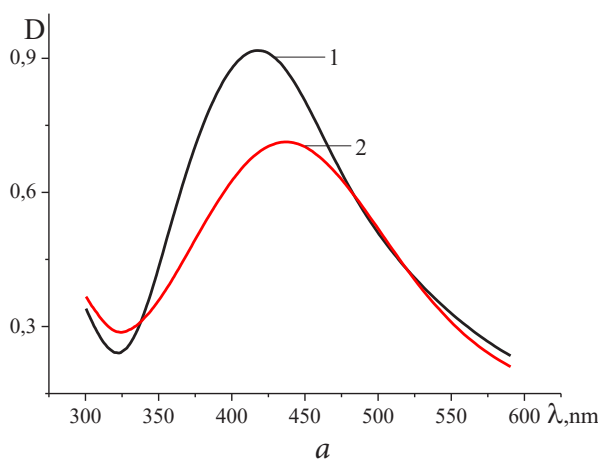


Fig. 1a Position of the SPR band of silver colloidal solutions obtained by reduction with alcohol extracts ( $\text{AgNO}_3$ : extract ratio 2.5:1): 1 – aloe, 2 – chamomile, 2.5 hours after synthesis, 1b – obtained colloidal solutions of aloe (left) and chamomile (right).

For a final conclusion about the effectiveness of the studied extracts, it is necessary to investigate the properties of the system, in particular its stability over time. The hermetically closed solutions were left, after which the electronic absorption spectra of the studied colloidal solutions were recorded for a month. As can be seen from Figure 2, curve 2 after 14 days did not shift the band maximum, but the shape changed, in particular, the intensity of the band decreased and the half-width increased to 70 nm. After 25 days, the shape of the band and the position of the maximum remained unchanged, but against the background of a slight increase in intensity, there is an increase in the half-width of the line up to 80 nm. Obviously, during the entire studied period, processes of recovery and recrystallization occur in the system, which contributes to the increase in the polydispersity of this system.

The microphotographs of dried solutions were taken on a Mira 3 Tescan electron microscope. The results of the microscopic analysis confirm the above theses. As can be seen from fig. 2a, 2d, spherical particles with a size of 9 nm are formed in the system, which is in good agreement with the shape and position of the SPR band (Fig. 1, 2, curve 1). After 25 days (Fig. 3b, 3e), the morphology of the particles is disturbed, but the dispersity practically does not change (Fig. 2, curve 2). After 2 months, significantly more significant agglomeration processes are visible, the concentration of small particles becomes smaller, particles of 30–50 nm appear, Fig. 3c, 3d. As a result of the recrystallization of small particles and their subsequent adsorption on larger particles, not only the dispersion changes, but also the morphology, the spherical symmetry of the particles is broken, they already have a slightly de-

formed shape. These results also correlate well with the position and shape of the SPR band (Fig. 2 curve 3.)

The obtained results of EAS and microscopic studies indicate the high reducing activity of aloe extract, which is an effective silver reducer.

Considering the high reductive ability of aloe extract, attempts were made to obtain dispersed silver systems by reduction without

heating. The process was much slower, and after 3 hours the solution acquired a barely noticeable color, and a broad SPR band of low intensity was recorded in the electronic spectra. However, after a day, the color of the solution became quite saturated, but with noticeable opalescence, and it was possible to record the SPR band (Fig. 2 curve 4) 1.3 times higher in intensity compared to the colloidal solution obtained by heating the reaction mixture.

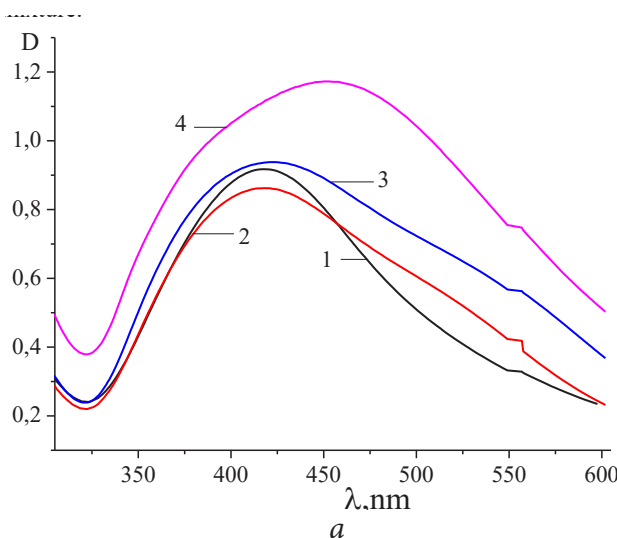


Fig. 2 The position of the SPR band of colloidal silver solutions obtained by reduction with alcoholic aloe extract (ratio  $\text{AgNO}_3$ : aloe extract 2.5:1): 1 – after 2.5 hours, 2 – after 14 days, 3 – after 25, 4 – after 1 day without heating of the original system – a, obtained colloidal solutions with a ratio of 2.5:1 – b, a ratio of 5:1 – c.

The band maximum is shifted to the long-wavelength region  $\lambda_{\text{max}}=455$ , and the half-width of the line is 90 nm. Which indicates a large particle size and polydispersity of the system. A week later, a precipitate formed at the bottom of the beaker, and the SPR band was not observed in the solution obtained without heating.

Much worse results were observed for colloidal solutions obtained with a significant excess of silver nitrate to the extract, a ratio of 5:1, respectively. As can be seen from Figure 3,

curve 1, the maximum of the band is shifted by 25 nm ( $\lambda_{\text{max}}=445$  nm) compared to the systems obtained with a smaller excess of argentum salt, and the half-width of the line is 85 nm. Already after a week, significant changes in the color of the solution were noticeable, and the results of the spectroscopic study showed a decrease in intensity and a shift of the maximum of this band to the long-wave region (Fig. 4, curve 2). This indicates that a ratio of 2.5:1 is optimal for reduction with aloe extract.



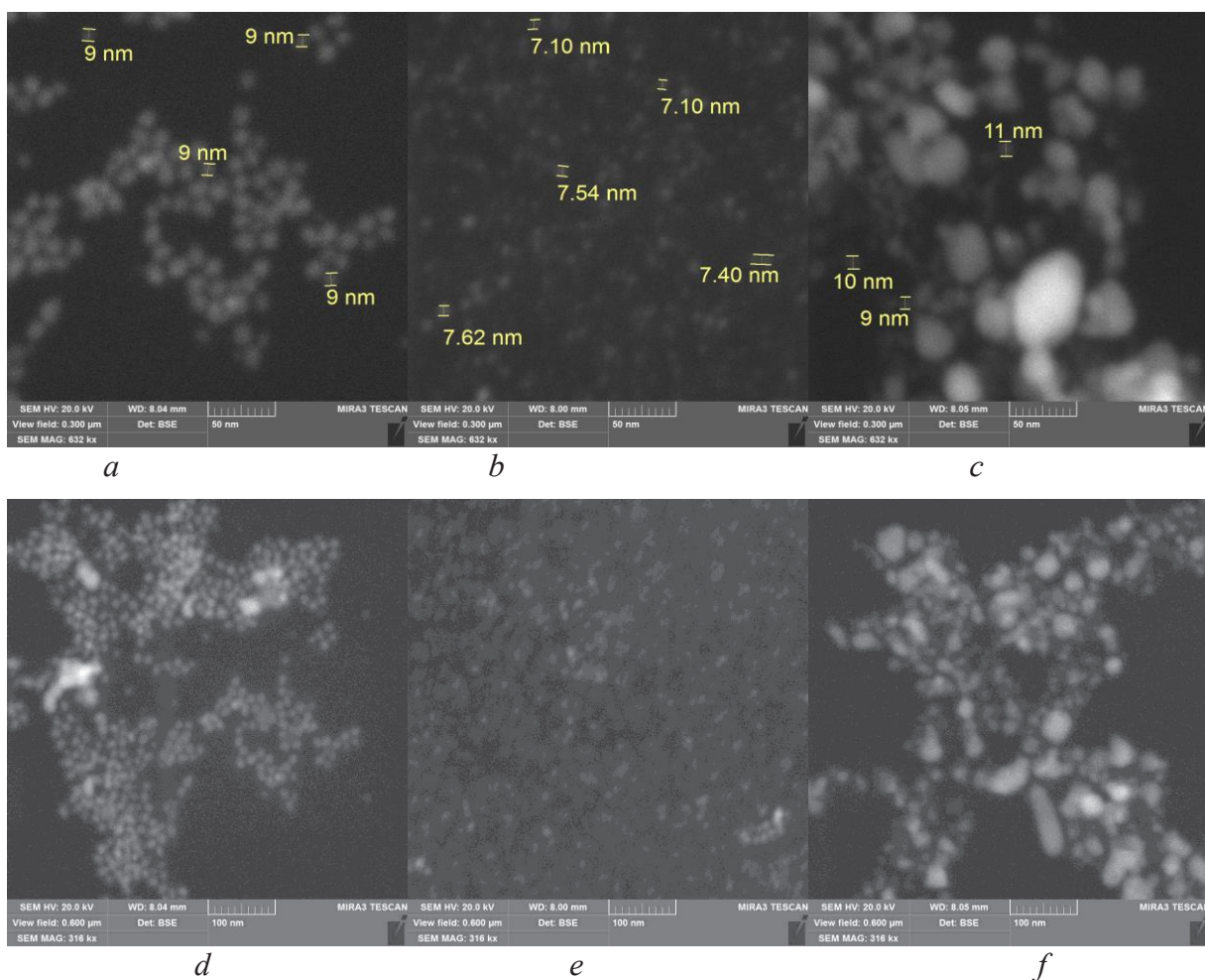


Fig. 3 SEM analysis of microphotographs of samples over time, reducing agent – aloe extract: *a, d* – 2 days after synthesis, *b, e* – after 14 days, *c, f* – after 25 days, scale mark *a, b, c* – 50 nm, *d, e, f* – 100 nm.

A completely different process was observed when chamomile extract was used as a reducing agent. In the first hours, this colloidal solution was inferior to the system based on aloe, the maximum of the band was shifted to the long-wave region (Fig. 1 curve 2, Fig. 5 curve 1) ( $\lambda_{\max}=436$  nm), however, over time the solution became increasingly transparent and saturated, which was reflected in EAS. As can be seen from the figure, the intensity of the band increased over time (Fig. 5, curve 2), and

the position of the maximum ( $\lambda_{\max}=436$  nm) and the half-width of the line did not change. The highest intensity of the band was observed on the 25th day (Fig. 5, curve 3), thereafter the SPR band remained practically unchanged, which is also confirmed by electron microphotographs of this system. Thus, colloidal silver solutions based on chamomile extract do not need additional stabilization, however, to avoid agglomeration processes, special storage conditions must be observed (a cool place, no

direct sunlight). The systems obtained with a 5-fold excess of argentum nitrate are unstable, because immediately upon mixing the components, the solution acquires a grayish hue, which is reflected in the EAS (Fig. 5, curve 4). After a day, a black sediment appears at the bottom, which indicates an intensive recovery of silver.

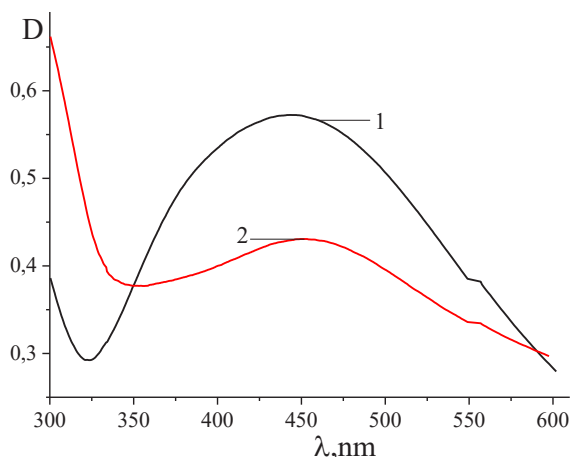


Fig. 4 The position of the SPR band of colloidal silver solutions obtained by reduction with alcohol extracts ( $\text{AgNO}_3$ :aloe extract ratio 5:1): 1 – after 2.5 hours, 2 – after 7 days.

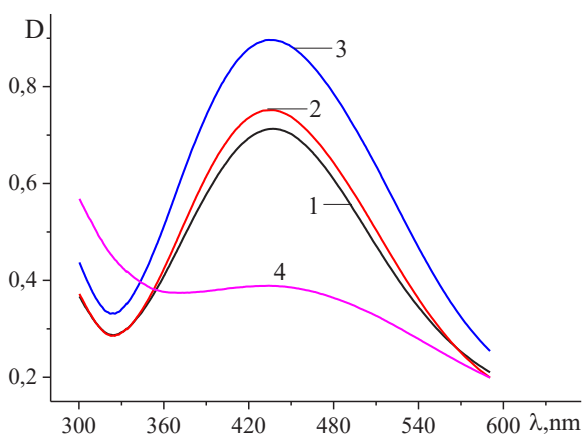


Fig. 5 Position of the SPR band of silver colloidal solutions obtained by reduction with chamomile alcohol extract: 1 – after 2.5 hours, 2 – after 14 days, 3 – after 25 ( $\text{AgNO}_3$ : extract ratio 2.5:1), 4 – after 2.5 hours with a ratio of  $\text{AgNO}_3$ : extract 5:1.

It can be seen from the microphotographs (Fig. 6) that when chamomile extract is used as a reducing agent, spherical particles are formed, but their size differs significantly and is in the range of 7–10 nm (Fig. 6a, f). After 25 days, the size of the particles increases somewhat, which is due to the processes of recrystallization of small particles (Fig. 6b), but all particles have the correct spherical shape (Fig. 6e). After 2 months, the number of small particles decreases, 20 nm particles appear, the slight difference in morphology is due to agglomeration processes (Fig. 6c, e), which confirms the increase in the half-width of the SPR line. Thus, the optical properties and stability of the studied nanosystems depend on the reducing extract, the ratio and concentration of the starting reagents.

**CONCLUSION.** The results of the conducted research showed that, in addition to high biological activity, the studied extracts have a pronounced reducing ability, which made it possible to use these extracts as reducing agents in the synthesis of silver nanoparticles by the chemical condensation method. Green synthesis of spherical silver nanoparticles using extracts of aloe vera, *Matricaria chamomilla* allows quick, simple and effective synthesis of nanoparticles with the required optical properties. Depending on the extract and its concentration, the size of AgNpcs was varied from 7 to 50 nm by simply adjusting the ratio of the starting reagents. These extracts show reducing properties due to the presence of carbonyl compounds in their composition, in particular organic acids. The conducted studies showed the high efficiency of natural extracts of aloe vera and *Matricaria chamomilla*. Aloe extract has the best regenerative properties, which is probably due to the presence of salicylic and

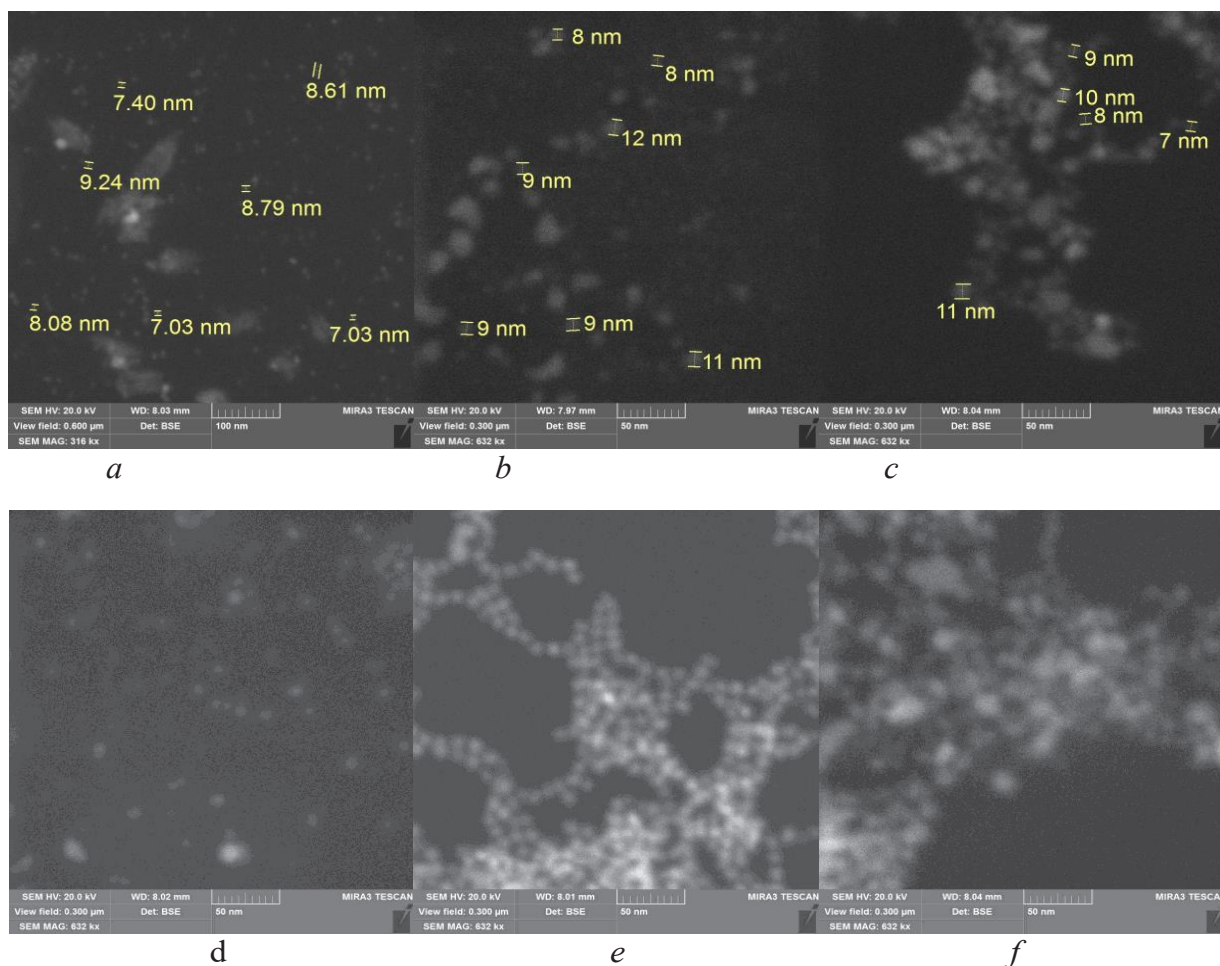


Fig. 6 SEM analysis of microphotographs of samples over time, reducing agent - chamomile extract: *a, d* – 2 days after synthesis, *b, e* – after 25 days, *c, f* – after 2 months, scale mark *a* – 100 nm, *b, c, d, e, f* – 50 nm.

acetylsalicylic acids in its composition. A comparison of the stability over time of the studied systems indicates a higher stability of dispersed systems based on chamomile extract, which indicates the effectiveness of this extract, and a high ability of the dispersed system to self-stabilize.

Thus, the presence of biologically active compounds with high reducing properties in the extracts, in particular flavonoids, glycoproteins, salicylic and ascorbic acids, make them prom-

ising for the synthesis of silver nanoparticles by the chemical condensation method. The use of natural extracts expands the range of practical applications of the obtained AgNpcs.



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## ЗЕЛЕНИЙ СИНТЕЗ НАНОЧАСТИНОК СРІБЛА ТА ЇХНІ СПЕКТРАЛЬНІ ВЛАСТИВОСТІ

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Хімічною конденсацією синтезовано сферичні наночастинки срібла з використанням екстрактів алое вера та ромашки лікарської як відновників. Наявність у складі екстрактів біологічно активних сполук із високими відновними властивостями, зокрема флаваноїдів, глюкопротеїдів, саліцилової та аскорбінової кислот, роблять їх перспективними при синтезі наночастинок срібла методом хімічної конденсації. Залежно від екстракту та його концентрації розмір AgNpcs змінювався від 7 до 50 нм шляхом простого регулювання співвідношення вихідних реагентів. Вочевидь різна відновлювана активність екстрактів зумовлена їхнім різним хімічним складом. Кращі відновні властивості має екстракт алое вера, що, ймовірно, зумовлено наявністю в його складі не лише аскорбінової, а й саліцилової та ацетилсаліцилової кислот. Вже в перші години формується монодисперсна система з розміром наночастинок 9 нм. Екстракт ромашки, вочевидь, містить мен-

ші концентрації відновників, тому процес трохи зміщений в часі і в перші години після синтезу утворюється полідисперсна система з розміром 7–10 нм, проте з часом відбувається рекристалізація частинок і, як наслідок, зменшується полідисперсність – на 25 день розмір частинок стає 9 нм. Зміна концентрацій та умов синтезу дозволяє керувати розміром частинок. Зміна умов синтезу впливає на положення смуги поверхнево плазмонного резонансу, а отже, і на оптичні властивості досліджуваних систем. Форма та положення смуг в електронних спектрах поглинання та електронні мікрофотографії свідчать про утворення сферичних AgNpcs. Доведено, що синтезовані наночастинки срібла не вимагають додаткової стабілізації при дотриманні зазначених умов. Крім цього, електронні мікрофотографії зразків через кілька тижнів після синтезу свідчать про формування з часом стійких дисперсних систем. Для уникнення агломераційних процесів потрібно не допускати потрапляння прямих сонячних променів, які сприятимуть подальшому фотовідновленню наночастинок та перепадів температур, які викликать коагуляційні процеси. За тривалого зберігання розчинів у дисперсних системах протікають процеси відновлення та рекристалізації, що зумовлює полідисперсність зазначеної системи, проте на її стійкість не впливає, оскільки розподіл за розмірами в межах однієї системи не перевищує 10 нм. Використання висококонцентрованих розчинів для синтезу викликає агломераційні процеси та руйнування систем. Зелений синтез сферичних AgNpcs із використанням екстрактів алое вера (*Aloe vera*) та ромашки лікарської (*Matricaria chamomilla*) дозволяє

швидко, просто та ефективно синтезувати наночастинки з регульованими оптичними властивостями. Застосування природних екстрактів розширює спектр практичного застосування отриманих AgNpcs.

**Ключові слова:** наночастинки срібла, оптичні властивості, дисперсність, алое вера, ромашка.

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