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Optics

Synthesis and optical characterization of seed silver nanoparticles *in situ* coated with poly-(N-isopropylacrylamide)

M.M. Khutko[1], A.M. Lopatynskyi[1,2], V.K. Lytvyn[1], P.V. Demydov[1] and V.I. Chegel[1,2]

[1]*V. Lashkaryov Institute of Semiconductor Physics, National Academy of Sciences of Ukraine,*
41, prospect Nauky, 03028 Kyiv, Ukraine
[2]*Educational and Scientific Institute of High Technologies, Taras Shevchenko National University of Kyiv, 64/13,*
Volodymyrska str., 01601 Kyiv, Ukraine
**Corresponding author e-mail: chegelvi@outlook.com*

Abstract. Due to their outstanding physicochemical properties, noble metal nanoparticles are vastly exploited across multiple fields, including nanomedicine and sensors. In the nanostructures synthesis process, a stabilizing agent is usually introduced to prevent their aggregation. In this work, a poly-(N-isopropylacrylamide) (pNIPAM) thermosensitive polymer coating was synthesized *in situ* as a stabilizing layer during the formation of seed silver nanoparticles and characterized using UV-vis spectrophotometry and dynamic light scattering. The evolution of pNIPAM coating on the surface of seed silver nanoparticles was followed by studying the changes in the light extinction spectra of the nanoparticle solutions, caused by localized surface plasmon resonance and Rayleigh scattering. The sizes of the studied particles, determined by dynamic light scattering, indicate the formation of electrostatic chelate complexes of NIPAM or pNIPAM with silver ions or nanoparticles, which was confirmed by static and dynamic molecular mechanics (MMX) calculations in PCModel software.

Keywords: silver nanostructures, poly-(N-isopropylacrylamide), localized surface plasmon resonance, light scattering, chelate complexes, molecular mechanics calculations.

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1. Introduction

Due to their unique physicochemical properties, noble metal nanoparticles are used in various areas, involving cancer diagnosis, drug delivery, photothermal therapy, plasmon-enhanced spectroscopies, and sensors [1–5]. Useful properties of noble metal nanoparticles are determined by their size, morphology, and coating [6] due to the localized surface plasmon resonance (LSPR) phenomenon [7]. Moreover, noble metal nanoparticles are further functionalized, for example, with smart polymers, exhibiting swelling/deswelling responses to external stimuli, namely pH, temperature, ionic strength, *etc.* [8]. This responsive behavior can be harnessed to manipulate the morphological, optical, and electrical characteristics of the nanomaterial [9, 10].

One of the applications of plasmonic nanostructures coated with smart polymers is the development of nanocontainers for controllable drug release, particularly based on hollow gold nanostructures [11, 12], which are typically fabricated from silver nanostructures (AgNPs) as a template. In addition, noble metal nanoparticles with polymer coating have been used in sensitive elements for sensors based on LSPR and plasmon-enhanced fluorescence phenomena [13, 14].

Usually, the replacement of the stabilization layer is performed for polymer coating on previously prepared nanoparticles, which is a multi-stage process that involves the polymer end-group modification to bind to the noble metal nanoparticle surface [2]. To reduce costs and time, as well as to avoid aggregation processes during the nanostructure synthesis, the method of polymerization simultaneously with the synthesis of small (seed) nanoparticles [14] can be used instead.

Seed AgNPs synthesis has been typically used as a first stage in the synthesis of gold nanoshells based on the templated galvanic replacement reaction of silver for gold [15–17]. Small spherical AgNPs typically absorb light at wavelengths close to 400 nm due to the LSPR excitation [18, 19], which can be used to monitor the formation of seed AgNPs.

For different applications in nanomedicine, it is convenient to use smart thermoresponsive polymers, namely poly-(N-isopropylacrylamide) (pNIPAM) with a low critical solution temperature of 32 °C, which is adjustable by copolymerizing with other monomers to be close to

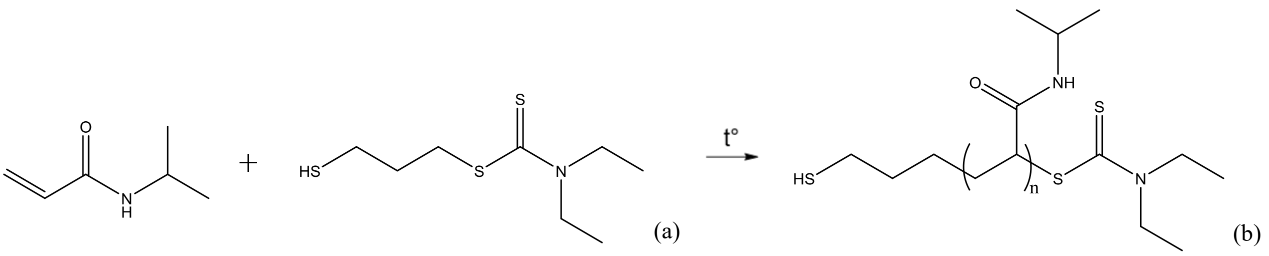

Fig. 1. Scheme of pNIPAM synthesis by RAFT polymerization *via* temperature: (a) NIPAM and 3-mercaptopropyl diethylcarbamodithioate iniferter; (b) pNIPAM.

the human body temperature of 37 °C [8, 20, 21]. Additionally, the presence of an amide group in the NIPAM molecule causes the chelation of Ag^+ ions with monomers and each other, followed by their further reduction to Ag^0 nanoparticles [19, 22]. Modification of the pNIPAM end-group to thiol is generally used to make the interactions between polymer and noble metal much stronger, providing long-term stability of the fabricated nanoparticles [23].

In this work, a new method of AgNPs synthesis, involving simultaneous formation in the presence of NIPAM with the polymerization to form a stabilizing pNIPAM layer, is proposed. Additionally, the spectral and morphological properties of the obtained plasmonic nanostructures are studied.

2. Materials and methods

2.1. Materials

To synthesize AgNPs, aqueous solutions of silver nitrate, sodium citrate or NIPAM as stabilizers, sodium borohydride as a reductant, and a saturated solution of 3-mercaptopropyl diethylcarbamodithioate in isopropanol as a reversible addition-fragmentation chain-transfer (RAFT) polymerization agent (iniferter) were used. All chemicals were purchased from Sigma-Aldrich and used as received except for the iniferter, which was custom-synthesized [24]. Ultrapure deionized water (type I, R = 18.2 MOhm·cm) from the water purification system Adrona B30 Bio was used for the aqueous solutions. Spectrophotometric measurements have shown that an aqueous solution of pure NIPAM absorbs light within the wavelength range close to 450 nm, and an isopropanol solution of pure iniferter absorbs light within the wavelength range close to 440 nm.

2.2. Nanoparticle synthesis

A known method for AgNPs formation, reported by Prevo *et al.* [15], where sodium citrate and sodium borohydride are utilized to reduce Ag^+ ions and stabilize AgNPs, was used as a reference technique. We have adapted this method by replacing sodium citrate with pNIPAM coating and sodium borohydride with iniferter. In the modified protocol, AgNPs were prepared by mixing aqueous solutions of silver nitrate and NIPAM with an isopropanol solution of iniferter and stirring for at least 2 h at 60 °C (unless otherwise specified). The concentrations of silver nitrate and NIPAM were 0.2 and 0.5 mM, respectively, in addition to 0.07 µl/ml saturated solution of iniferter in isopropanol (unless otherwise specified). Sodium borohydride with a final concentration of 2 mM was optionally added to induce further Ag^+ ions reduction to check the presence of already formed AgNPs.

2.3. RAFT polymerization

Formation of a stabilizing polymer layer was carried out using the RAFT polymerization method [25] simultaneously with the synthesis of AgNPs. This mechanism is related to the iniferter dormant and active group exchange. A radical of a polymer chain interacts with the sulfur-carbon double bond of the iniferter and goes to the carbon atom of the SH-end group, which forms a new polymer chain. Then, the latter one interacts with the sulfur-carbon double bond of the iniferter again, and the radical goes to the previous polymer chain bound with the iniferter at the first step, and so on. Another feature of the utilized iniferter is the presence of the SH-end group that provides binding of the polymer coating with AgNPs. The scheme of RAFT polymerization used in this work is shown in Fig. 1.

2.4. Spectrophotometric measurements

Light extinction measurements were performed using a double beam spectrophotometer SpectroQuest 4802 (Unico, USA) within the spectral range of 350…900 nm of the solutions of NIPAM and iniferter and colloidal solutions of the AgNPs covered by pNIPAM contained in plastic semi-micro and macro spectrophotometric cuvettes with an optical path of 1 cm, with deionized water or isopropanol (for pure iniferter) as a reference sample.

2.5. Size analysis

Size distributions of the resulting particles were determined using a Zetasizer Nano ZS photon correlation spectroscopy system based on the dynamic light scattering analysis for the solutions of NIPAM with Ag ions and AgNPs coated with pNIPAM.

2.6. Computer modeling

The formation and stability of the chelate complex of NIPAM molecules with Ag atoms were studied using computer modeling in PCModel software, considering minimum energy and dynamic molecular mechanics (MMX force field) calculations with heating temperature starting from 300 K.

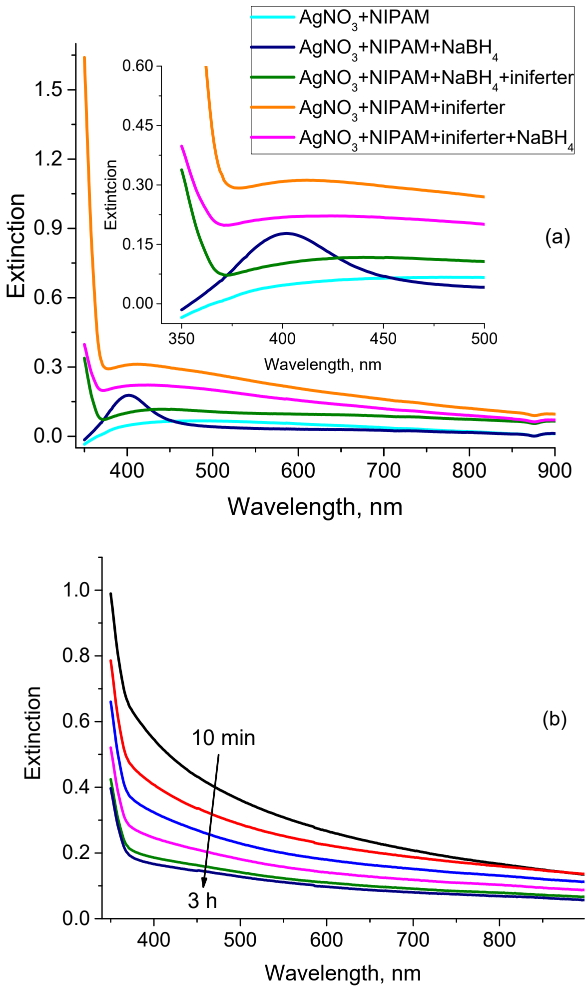

Fig. 2. Light extinction spectra of (a) mixtures of silver nitrate with NIPAM before and after the addition of sodium borohydride and/or iniferter and (b) mixture of iniferter with silver nitrate and NIPAM up to 3 hours after iniferter addition in the synthesis of AgNPs. Inset: zoom in on the absorbance peaks showing evidence of the presence of AgNPs.

3. Results and discussion

3.1. Synthesis of seed silver nanoparticles with *in situ* polymerization

To determine the optimal parameters for synthesizing stable AgNPs, a comparison of the spectral properties of seed AgNPs obtained with different sequences of reagent addition and their concentrations was carried out.

The light extinction spectra of AgNPs colloidal solutions synthesized using different routes are presented in Fig. 2a. The position in the light extinction spectra, corresponding to LSPR excitation in AgNPs, is close to 400 nm. After the addition of NIPAM to the silver nitrate solution, a broad absorbance peak close to 400 nm appeared, which may indicate the partial reduction of Ag$^+$ ions. Furthermore, after the addition of sodium borohydride to induce further reduction of Ag$^+$ ions, the solution became yellow with an absorbance peak at 402 nm, indicating the formation of AgNPs.

However, the addition of an iniferter to this system led to the nanoparticles aggregation, the appearance of a new broad extinction peak at ~ 440 nm, and a colorless solution, consequently. In another approach, adding an iniferter into the mixture of silver nitrate and NIPAM also resulted in a yellowish solution and a new absorbance peak at 412 nm, which implies the formation of AgNPs with pNIPAM coating, which manifests as the red-shifted LSPR peak position. The addition of sodium borohydride to this mixture did not change the extinction spectrum substantially, which confirms the presence of already formed AgNPs due to the iniferter addition. Therefore, iniferter destabilizes pre-prepared nanoparticles and should be added to the mixture instantly after NIPAM before the formation of AgNPs. The noted sequence of reagent addition was further used.

Broader peaks present in the top two spectra in Fig. 2a may indicate a polymerization of pNIPAM, which was further confirmed by an additional synthesis involving a mixture of iniferter with silver nitrate and NIPAM without sodium borohydride over 3 h (Fig. 2b). An increased intensity of the spectra within the UV range at the start of the synthesis was attributed to the Rayleigh scattering of light due to formation of polymer chains. Light scattering decreases with increasing reaction time in the synthesis of AgNPs and formation of a polymer coating around them, resulting in a lower concentration of free polymer chains in solution. However, the LSPR peak for AgNPs synthesized without sodium borohydride was not expressed because of the remaining Rayleigh scattering, implying that the yield of AgNPs should be optimized.

3.2. The dependence of seed silver nanoparticles stability on the time and reagent concentrations

Besides the formation of AgNPs, their stability over a long period of time is also an important parameter. To compare the stability of the synthesized AgNPs, they were prepared with concentrations of NIPAM and iniferter differing by 10 times. Fig. 3 shows light extinction spectra for colloidal solutions of AgNPs fabricated with initial (experiment 1) and 10× (experiment 2) concentrations measured at different times up to 10 days after synthesis. A weak peak close to 400 nm in experiment 1 and no expressed peak in experiment 2 registered for fresh AgNPs samples means that the formation of the polymer occurs much quicker than the formation of AgNPs in both cases, resulting in LSPR excitation being obscured by the Rayleigh scattering from the polymer. However, the stability of the resulting polymer-coated AgNPs with time differs for the two experiments. For 10 days, AgNPs in experiment 2 almost did not change their spectral properties as compared to experiment 1, where a further increase of Rayleigh scattering was observed, meaning that AgNPs obtained in experiment 2 were more stable. This result was also confirmed by the stable yellowish color of the solution, which is considered an indicator of AgNPs formation due to their size-dependent LSPR properties.

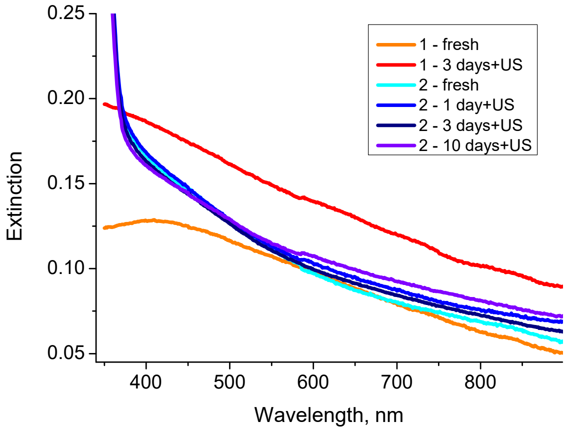

Fig. 3. Dependence of AgNPs light extinction spectra stability on the time and reagent concentrations. Concentrations of NIPAM and iniferter are 10 times larger in experiment 2 compared to experiment 1. To redisperse the particles settled on the walls of the vessel over time, an ultrasonic bath (US) was used.

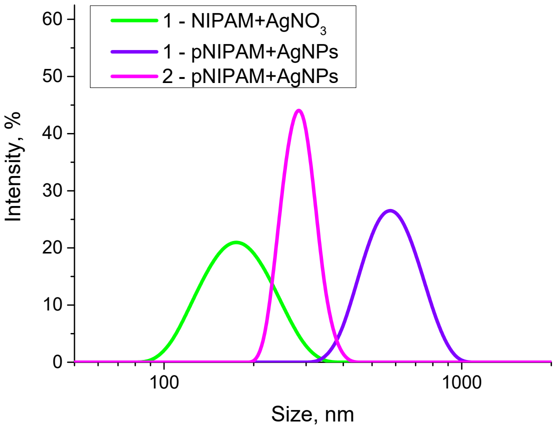

Fig. 4. Particle size distributions for silver nitrate solutions with NIPAM and obtained AgNPs with various reagent concentrations. Concentrations of NIPAM and iniferter are 10 times larger in experiment 2 compared to those in experiment 1.

3.3. Size analysis

The sizes of resulting particles in solution were determined using the dynamic light scattering analysis, which showed interesting distributions (Fig. 4). Silver nitrate solution with NIPAM (Fig. 4, experiment 1) contains particles with an average diameter close to 190 nm, which is unusually large for this stage of synthesis. After the addition of the iniferter, the average diameter of particles containing pNIPAM and AgNPs increased to 500 nm (Fig. 4, experiment 1), which may indicate their aggregation. In contrast, AgNPs prepared with 10 times larger concentration of NIPAM and iniferter exhibited a narrower peak with a smaller average particle size close to 300 nm (Fig. 4, experiment 2) even after 3 days, obviously proving their stability. This size distribution and spectral properties imply the formation of electrostatic chelate complexes of NIPAM or pNIPAM with Ag ions or nanoparticles. To confirm this conclusion, energy modeling was performed to show the existence of these chelate complexes.

3.4. Computer modeling of chelate complex of NIPAM molecules with Ag atoms – formation and stability

Seven simulations of the formation of the chelate complex between NIPAM molecules and Ag atoms with the minimum energy value within the range of [–113…–83] kcal/mol were carried out, and a typical result is presented in Fig. 5. In Figs. 5a and 5b, two NIPAM molecules with five Ag atoms were located chaotically. After several iterations, they were getting

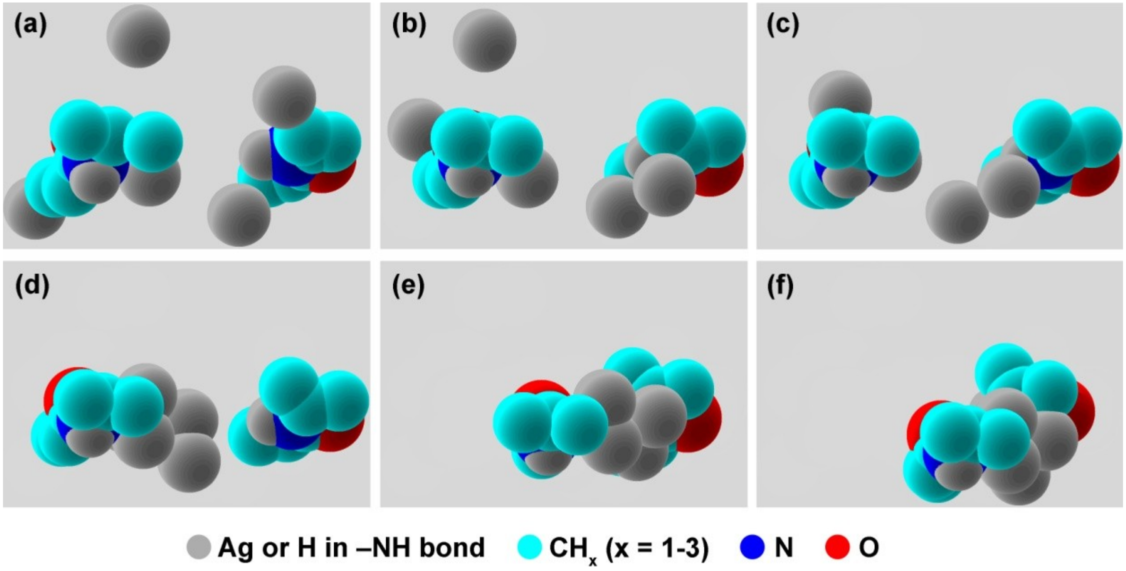

Fig. 5. Visualization of the electrostatic chelate complex formation between NIPAM molecules and Ag atoms resulting from minimum energy simulations.

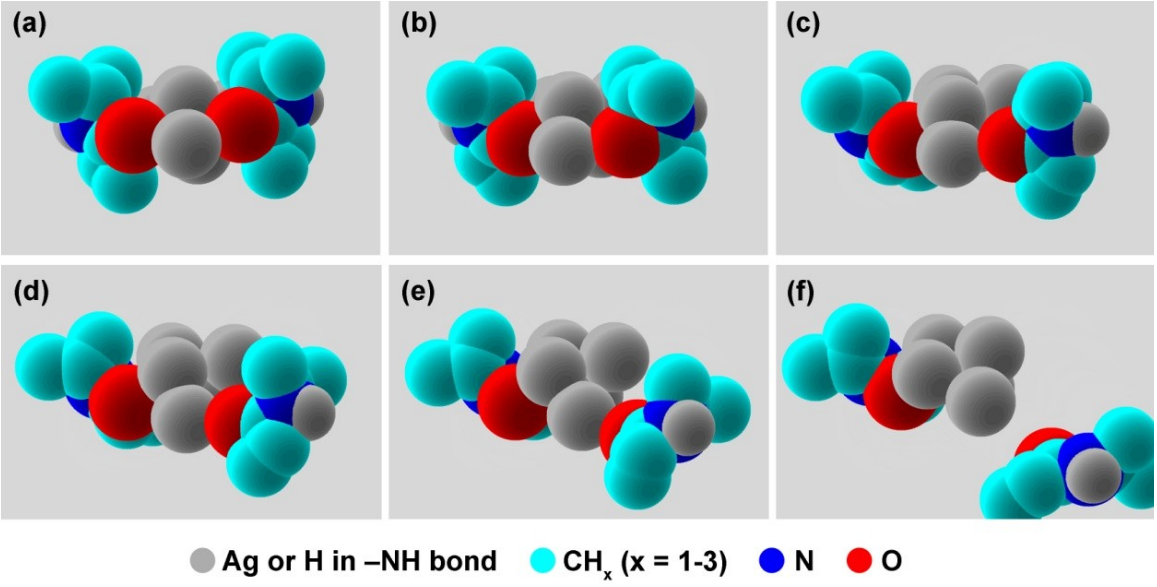

Fig. 6. Visualization of the process of dynamic oscillation of the electrostatic chelate complex between NIPAM molecules and Ag atoms resulting from heating modeling.

closer to each other (Figs. 5c, 5d) and finally combined in one unit (Figs. 5e, 5f). When Ag atoms approach NIPAM molecules and each other, they tend to agglomerate, forming large particles due to the negative charge of oxygen and nitrogen and the positive charge of silver. These chelate complexes can be quite stable, which was further proved by dynamic oscillation using heating modeling.

In heating modeling, the chelate complex changed its conformation within the temperature range 230 K…337 K, but it remains stable (Figs. 6a–6d). However, when the temperature gradually increased to 463 K, the complex started to fall apart (Figs. 6e, 6f). Yet, in real conditions, the synthesis of AgNPs is carried out at 60 °C (*i.e.* 333 K); therefore, the complex would remain stable after formation.

4. Conclusions

Using a specialized iniferter with a thiol group for binding with noble metal surface makes RAFT polymerization much easier and with fewer stages, providing the opportunity to create a thermoresponsive polymer pNIPAM coating simultaneously with the synthesis of seed AgNPs. It was found that the formation of the polymer occurred much quicker than the formation of AgNPs with the studied reagent concentrations, implying that the yield of AgNPs should be optimized. UV-vis light extinction spectra, dynamic light scattering analysis and computer modeling have proved the stability of seed AgNPs with increasing the NIPAM and iniferter concentrations due to NIPAM or pNIPAM electrostatic chelate complexes with Ag ions or nanoparticles. We believe that the proposed method for the synthesis of polymer-coated noble metal nanoparticles has the potential to streamline the development of drug delivery and sensor systems.

Acknowledgements

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Authors and CV

Maria M. Khutko, PhD student in Applied Physics and Nanomaterials, Leading Engineer at the Department of Biochemosensorics, V. Lashkaryov Institute of Semiconductor Physics. Her research interests include chemical synthesis and studies of plasmonic nanostructures with dielectric coatings for plasmon-enhanced fluorescence and drug delivery applications. E-mail: mariiakhutko@gmail.com, https://orcid.org/0009-0005-9641-0497

Andrii M. Lopatynskyi, PhD in Physics and Mathematics, Senior Researcher at the Department of Biochemosensorics, V. Lashkaryov Institute of Semiconductor Physics. His research interests include development, experimental investigation and computational modeling of optical properties of disordered and ordered plasmonic nanosystems based on noble metal nanostructures as well as development of sensor devices on their basis.
E-mail: lop2000@ukr.net,
http://orcid.org/0000-0002-6847-892X

Vitalii K. Lytvyn, PhD in Technical Sciences, Researcher at the Department of Biochemosensorics, V. Lashkaryov Institute of Semiconductor Physics. His research interests include plasmon-enhanced fluorescence, nanotechnology, electronics, and development of random and highly ordered high-conductive nanostructure arrays for plasmonic and biotechnological applications.
E-mail: lytvet@ukr.net,
https://orcid.org/0000-0003-1241-4457

Volodymyr I. Chegel, Professor, DSc in Physics and Mathematics, Head of Plasmonics group at the Department of Biochemosensorics, V. Lashkaryov Institute of Semiconductor Physics. His research interests are focused on plasmonics, nanotechnology, material science, biological and chemical sensors, design of optoelectronic devices.
E-mail: chegelvi@outlook.com,
http://orcid.org/0000-0002-0190-9932

Petro V. Demydov, PhD in Applied Physics and Nanomaterials, Junior Researcher at the Department of Biochemosensorics, V. Lashkaryov Institute of Semiconductor Physics. His research interests include localized surface plasmon resonance, nanoparticles, electrical and optoelectronic engineering, computational modeling of optical properties of disordered and ordered plasmonic nanosystems based on high-conductive metallic nanostructures. https://orcid.org/0000-0002-8394-5573, e-mail: vodimed@ukr.net

Authors' contributions

Khutko M.M.: investigation, methodology, writing – original draft.

Lopatynskyi A.M.: project administration, methodology, writing – review & editing.

Lytvyn V.K.: resources, writing – review & editing.

Demydov P.V.: investigation, writing – review & editing.

Chegel V.I.: conceptualization, methodology, writing – review & editing.

Синтез та оптичні характеристики зародкових наночастинок срібла, *in situ* покритих полі-(N-ізопропілакриламідом)

М.М. Хутько, А.М. Лопатинський, В.К. Литвин, П.В. Демидов та В.І. Чегель

Анотація. Завдяки своїм видатним фізико-хімічним властивостям, наночастинки шляхетних металів широко застосовуються в різних галузях, зокрема в наномедицині та сенсориці. У процесі синтезу наноструктур зазвичай вводиться стабілізуючий агент для запобігання їх агрегації. У цій роботі термочутливе полімерне покриття з полі-(N-ізопропілакриламіду) (pNIPAM) було синтезоване *in situ* як стабілізуючий шар під час формування зародкових наночастинок срібла і досліджене методами УФ-видимої спектрофотометрії та динамічного розсіювання світла. Еволюцію покриття pNIPAM на поверхні зародкових наночастинок срібла відстежували за змінами в спектрах екстинкції світла розчинів наночастинок, зумовлених локалізованим поверхневим плазмонним резонансом та релеївським розсіюванням. Розміри досліджуваних частинок, визначені методом динамічного розсіювання світла, вказують на утворення електростатичних хелатних комплексів NIPAM або pNIPAM з іонами срібла або наночастинками, що підтверджено розрахунками статичної та динамічної молекулярної механіки (MMX) у програмному забезпеченні PCModel.

Ключові слова: наноструктури срібла, полі-(N-ізопропілакриламід), локалізований поверхневий плазмонний резонанс, розсіювання світла, хелатні комплекси, розрахунки молекулярної механіки.