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Synthesis and processing of polymers and polymeric composites

Синтез и переработка полимеров и композитов на их основе

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RESEARCH ARTICLE

Composite material obtained based on track-etched membranes and silver nanoparticles of different shapes

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Abstract

Objectives. To study the effect of shape on the intensity of surface-enhanced Raman light scattering when depositing nanoparticles on track membranes. The resulting composite material can be further used as a substrate for sensors. The efficiency of such sensors is determined by the effect of surface-enhanced Raman scattering of light.

Methods. Silver nanoparticles were obtained by reduction of silver ions in solution under various conditions. Nanoparticles from the obtained colloidal solutions were deposited on polyethylenimine-modified polyethylene terephthalate track-etched membranes. The samples were examined using absorption spectroscopy in the ultraviolet and visible region, scanning and transmission electron microscopy, dynamic light scattering, and Raman spectroscopy.

Results. Silver nanoparticles of spherical, triangular, and nanowire shape were synthesized. The sizes and zeta potential of the nanoparticles were determined. The obtained nanoparticles were deposited on the surface of track-etched membranes. For the composite membrane samples, the relative enhancement factors of the Raman light scattering signal of the 4-aminothiophenol test substance were calculated based on the substrate with a known enhancement factor.

Conclusions. The effect of surface-enhanced Raman light scattering was found to be greater when transitioning from spherical to various nonspherical-shaped nanoparticles. The highest value of the relative enhancement factor was $4 \cdot 10^7$ on the composite membrane with silver nanowires.

Keywords

surface-enhanced Raman scattering, silver nanoparticles, track-etched membranes, nanoplasmonics, SERS sensors

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НАУЧНАЯ СТАТЬЯ

Получение композитного материала на основе трековых мембран и наночастиц серебра различной формы

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Аннотация

Цели. Изучение влияния формы наночастиц на эффект гигантского комбинационного рассеяния света при осаждении их на трековые мембраны. В дальнейшем полученный композитный материал может использоваться в качестве подложки для сенсоров, работа которых основана на эффекте гигантского комбинационного рассеяния света.

Методы. Наночастицы серебра получали методом восстановления ионов серебра в растворе при различных условиях. Наночастицы из полученных коллоидных растворов осаждали на модифицированные полиэтиленимином полиэтилентерефталатные трековые мембраны. Для исследования образцов использовали спектроскопию поглощения в ультрафиолетовой и видимой области, растровую и просвечивающую электронные микроскопии, лазерный допплеровский микроэлектрофорез, спектроскопию комбинационного рассеяния.

Результаты. Синтезированы наночастицы серебра сферической, треугольной формы и в форме нанопроволок, определены размеры и дзета-потенциал наночастиц. Полученные наночастицы осаждены на поверхность трековых мембран. Для образцов композитных мембран рассчитаны относительные коэффициенты усиления сигнала комбинационного рассеяния света тестового вещества 4-аминотиофенола по отношению к подложке с известным коэффициентом усиления.

Выводы. Показано, что при переходе от сферической формы наночастиц к различным несферическим усиливается эффект гигантского комбинационного рассеяния света. Наибольшее значение относительного коэффициента усиления составило $4 \cdot 10^7$ на композитной мембране с серебряными нанопроволоками.

Ключевые слова

гигантское комбинационное рассеяние света, наночастицы серебра, трековые мембраны, композитные мембраны

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INTRODUCTION

Surface-enhanced Raman light scattering (SERS) spectroscopy is increasingly being applied for the qualitative and quantitative analysis of chemical compounds and biological objects at the level of low concentrations. This method is used to identify antibiotics [1], dyes [2, 3], fungicides [4], and drugs [5] at concentrations from 10^{-9} to 10^{-12} M.

Silver and gold nanoparticles are widely used to create SERS-active structures [2]. A number of studies report the use of copper [6], nickel [7], and mixed composites [8] as nanoparticle materials. However, silver nanoparticles (SNPs) enable the greatest enhancement of the Raman scattering signal [9].

The key characteristics of metal nanoparticles that influence signal enhancement include the material, size, and stabilizer. In addition, the shape of nanoparticles also makes a significant contribution [10, 11]. When transitioning from spherical nanoparticles to various nonspherical forms, the charge of surface atoms is localized at the angles and roughness. This leads to an increase in the electric field density between nanoparticles by more than an order of magnitude [12, 13], thus significantly enhancing the Raman signal. Hence, the synthesis and deposition of nanoparticles of various nonspherical shapes is a pressing research topic.

Nonspherical nanoparticles can be produced using spatially confined systems, such as membrane pores, or by adding reagents that are selectively adsorbed on individual faces of nanoparticle nuclei. Polyvinylpyrrolidone [14], cetyltrimethylammonium bromide [15], as well as salts of citric [14] and ascorbic acids [16], are capable of exhibiting this ability. In this case, nanoparticle growth is confined to individual directions, which enables the production of nanocubes, nanorods, triangular nanoprisms, and other shapes.

SERS spectroscopy can be conducted both in colloidal solutions and on planar substrates. The use of track-etched membranes (TMs) as a substrate permits the separation and concentration of the analyzed sample, thus improving the analysis selectivity. TMs are produced by irradiating polymer films with high-energy ions, which form latent tracks. These tracks are then converted into pores through further selective etching. The advantage of TMs consists in their ability to control pore concentration and size. To immobilize nanoparticles to the TM, its surface should be modified. In the case of SNPs, compounds containing carboxyl, thiol, or amino groups are typically used. Polyethyleneimine (PEI) is the most suitable modifier due to the presence of NH_x^+ terminal groups. PEI binds SNPs to the TM surface electrostatically as well as chemically due to the lone electron pair on the nitrogen atom [17]. PEI outperforms other modifiers in terms of its high solubility in water.

Thus, changing the SNP shape will significantly increase the Raman signal. Additionally, the use of TM as a substrate will improve the selectivity of SERS analysis. In this study, we aim to synthesize SNPs of various shapes and immobilize them on the TM surface in order to study the effect of nanoparticle shape on SERS intensity.

EXPERIMENTAL

Reagents and materials

The following reagents and materials were used in the work: $Na_3C_6H_5O_7 \cdot 5.5H_2O$ (98%, *PanReac*, Spain); branched polyethyleneimine (M_n = 60000, 50% aqueous solution, *Acros Organics*, Belgium); 4-aminothiophenol (4-ATP) (97%, *Sigma-Aldrich*, USA); ethanol (99.9%, *Merck*, Germany); $AgNO_3$ (chemically pure, *LenReaktiv*, Russia); $NaBH_4$ (99%, *Acros Organics*, Belgium); H_2O_2 (special purity grade, *Khimreaktivsnab*, Russia); NH_3 (25% aqueous solution, *PanReac*, Spain); NaOH (chemically pure, *LenReaktiv*, Russia); cetyltrimethylammonium bromide (99%, *Sigma-Aldrich*, USA); $C_6H_{12}O_6$ (chemically pure grade, *Vekton*, Russia); polyvinylpyrrolidone (M_w = 55000, *Sigma-Aldrich*, USA); deionized water (*Milli-Q*, *Millipore*, Germany) with a specific resistance of 18 μOhm·cm at 22°C; TM from polyethylene terephthalate (thickness 19 μm, pore concentration $2.7 \cdot 10^8$ cm^{-2}, pore diameter 0.4 μm) obtained at the Flerov Laboratory of Nuclear Reactions of the Joint Institute for Nuclear Research (Dubna, Russia) according to the method presented in [18].

SNP synthesis

Spherical SNPs were prepared using the citrate method based on the procedure described in [19]. To that end, 12.5 mL of a 10^{-3} M silver nitrate solution were added dropwise to 50 mL of a 10^{-3} M sodium citrate solution heated to 95°C. Prior to the synthesis, 1 M sodium hydroxide solution was added to the sodium citrate solution until a pH value became 9.8. The resulting mixture was maintained under constant stirring at the same temperature for 1 h.

Triangular nanoplates were prepared using the procedure described in [20]. Thus, 10^{-2} M silver nitrate (0.5 mL), 1% sodium citrate solution (2.3 mL), 2% polyvinylpyrrolidone solution (0.6 mL), 3% hydrogen peroxide solution (1.2 mL), and $2 \cdot 10^{-2}$ M sodium borohydride solution (1 mL) were successively added into 4.1 mL of water. Prior to adding sodium borohydride, the reaction mixture was heated to 55°C. The synthesis duration was 2 min.

Silver nanowires were synthesized by the hydrothermal method based on the procedure detailed in [21]. To that end, 0.17 g of silver nitrate was dissolved in 20 mL of water, then a 1 M aqueous ammonia solution was added to obtain silver ammine with a concentration of 10^{-3} M. 2.8 mL of the resulting silver ammine solution, 1.7 mL of a $5 \cdot 10^{-3}$ M solution of cetyltrimethylammonium bromide, and 5.6 mL of a 10^{-3} M glucose solution were added to the autoclave. The reaction mixture was maintained to 120°C for 8 h.

TM modification

The surface modification of the TMs was conducted based on the method described in [22]. The TMs were pre-washed in ethyl alcohol and water followed by immersion in a 0.1% aqueous solution of PEI and maintained on a laboratory shaker for 30 min. Following modification, the TMs were rinsed with water for 5 min.

The immobilization of spherical and triangular SNPs was conducted on PEI-modified TMs; native TMs were used for nanowires. For this purpose, 20 mL of the nanoparticle solution was filtered through the TMs in an Amicon Stirred Cells filtration cell (*Millipore*, Germany).

Methods

In order to evaluate the stability of colloidal SNP solutions, the zeta potential was measured using dynamic light scattering with a Zetasizer Nano ZSP particle size analyzer (*Malvern*, United Kingdom).

The shape of silver nanoparticles was determined by transmission electron microscopy (TEM) using a Talos F200iS/TEM microscope (*Thermo Scientific*, USA). Copper TEM grids with an amorphous carbon film (*SPI*, USA) were used as substrates. In order to deposit the SNP, the substrate was immersed in the colloidal silver nanoparticle solution followed by drying.

The presence of SNPs on the TM surface was determined from micrographs obtained by scanning electron microscopy (SEM) using an SU 8020 scanning electron microscope (*Hitachi*, Japan). A 5-nm thick layer of a platinum-palladium alloy was sputter-deposited on the samples.

The SNP size was determined using the JMicroVision 1.3.4 software[1] from the obtained micrographs.

The optical properties of the resulting SNPs were studied using ultraviolet (UV) and visible absorption spectroscopy on an Evolution 600 dual-beam spectrophotometer (*Thermo Scientific*, USA) at an optical path length of 1 cm.

The SERS effect on the resulting composites was determined using Enspectr R532 (*Spectr-M*, Russia) and Enspectr R638 (*Spectr-M*, Russia) Raman spectrometers with excitation wavelengths of 532 nm and 638 nm, respectively. Excitation was performed for 1 s in 10 repetitions. A solution of 4-ATP in ethanol was used as the test substance. 2 μL of 4-ATP solution at a concentration of 10^{-5} M were applied to the TM with immobilized SNPs, and Raman spectra were obtained after evaporation of the solvent.

RESULTS AND DISCUSSION

The conducted synthesizes resulted in stable colloidal solutions with spherical, triangular, and nanowire-shaped nanoparticles.

The following zeta potentials were obtained for the colloidal solutions of nanoparticles: −51 ± 5 mV for spherical nanoparticles, −29 ± 6 mV for triangular nanoplates, and +8 ± 1 mV for nanowires. The citrate and borohydride methods for synthesizing nanoparticles result in a negative charge of the obtained spherical and triangular nanoparticles. Therefore, these nanoparticles were immobilized on a PEI-modified membrane with a positive surface charge. Silver nanowires had a positive zeta potential; therefore, native TM, whose surface is negatively charged due to the presence of carboxyl groups, was used for nanoparticles deposition.

The surface plasmon resonance (SPR) maxima characteristic of the SNPs (Fig. 1) were determined by absorption spectroscopy in the UV and visible regions.

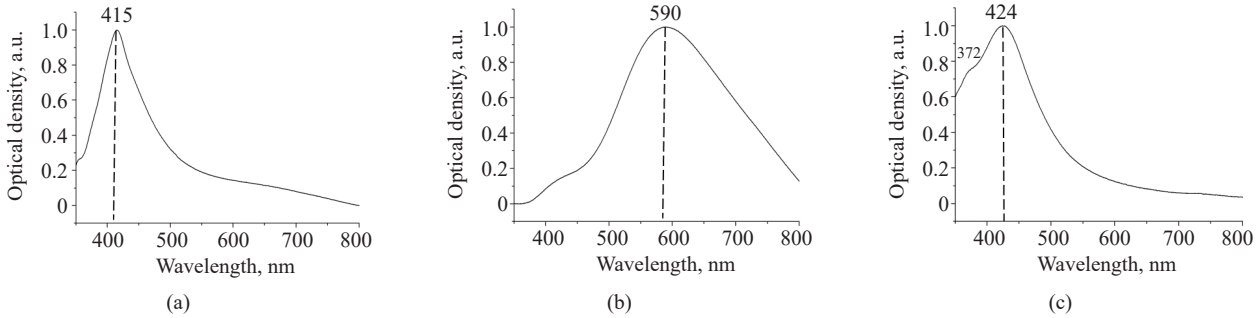
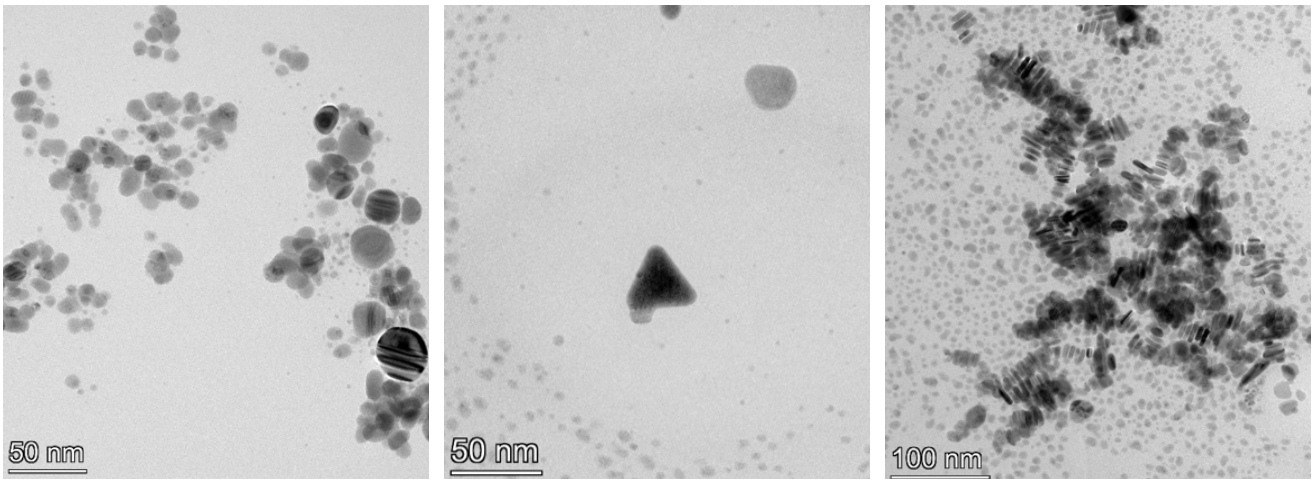


Fig. 1. Absorption spectra in the UV and visible regions of colloidal solutions of SNPs: (a) spherical; (b) triangular; (c) nanowires

[1] https://jmicrovision.github.io/download.htm. Accessed November 19, 2025.



(a) (b) (c)

Fig. 2. TEM images: (a) spherical nanoparticles; (b) triangular nanoparticles; (c) stacks of triangular nanoparticles

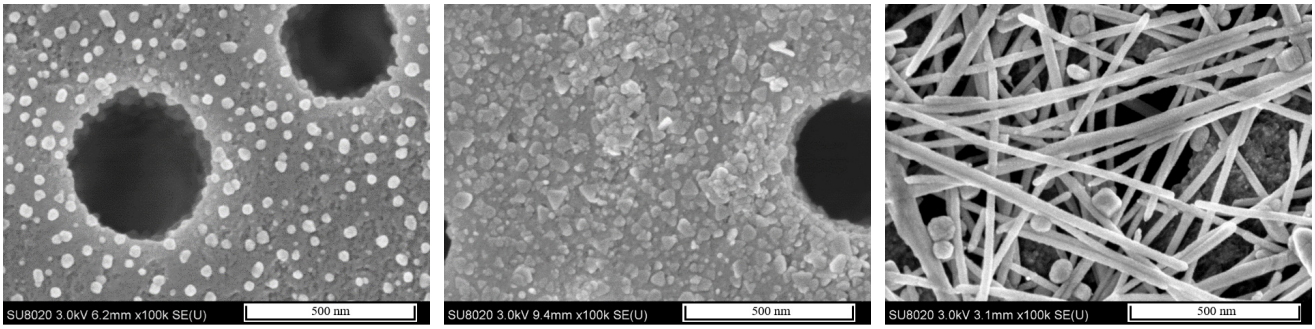
The absorption maximum of the SPR of the nanoparticles obtained by the citrate method is located at 415 nm, which corresponds to spherical nanoparticles (Fig. 1a) [23]. The absorption spectrum of the nanoparticles obtained by the borohydride method (Fig. 1b) has an absorption maximum at 590 nm, which confirms the presence of triangular nanoparticles [24]. The absorption spectrum of the colloidal solution of silver nanowires (Fig. 1c) has a maximum at 424 nm, which corresponds to the SPR of nanowire-shaped nanoparticles [25].

The TEM micrographs (Fig. 2) clearly show the shape of spherical nanoparticles (Fig. 2a), as well as triangular nanoplates (Fig. 2b), which form characteristic stacks when dried on the TEM grids (Fig. 2c).

At the next stage, the sizes of the nanoparticles were determined. The diameter of the spherical nanoparticles ranged 25 ± 5 nm, while the edge length of the triangular nanoparticles was 38 ± 8 nm, and their thickness was 4 ± 1 nm. The determination of silver nanowire length is a challenging task due to their multiple entanglement; their thickness was 26 ± 4 nm.

SEM micrographs (Fig. 3) clearly show silver nanoparticles on the TM surface, all of which are uniformly distributed. Note that the triangular nanoparticles exhibit an admixture of spherical nanoparticles. The silver nanowires exhibit an admixture of cubic nanoparticles.

The Raman spectrum of the original TM is a spectrum of polyethylene terephthalate with the most intense peaks at 1614 cm^{-1} and 1727 cm^{-1} corresponding to ν_{CC} and ν_{CO} vibrations, respectively [26]. The Raman spectrum of 4-ATP has peaks at 463 (δ_{CC}), 1085 (ν_{CS}), and 1592 cm^{-1} (ν_{CC}) [27]. It can be seen from Fig. 4 that when the signal is enhanced on spherical and triangular SNPs, the characteristic peaks of 4-ATP corresponding to ν_{CS} and ν_{CC} vibrations are preserved. At the same time, maxima also appear at 1380 and 1427 cm^{-1}, which can be attributed to ν_{NN} vibrations [27]. The discrepancy between the peak values of the Raman and SERS spectra of 4-ATP on spherical and triangular SNPs ranges within the instrumental error. The significant difference in 4-ATP spectrum on silver nanowires can be explained by 4-ATP sorption on silver nanowires not only by the SH group, but also by the NH_2 group. Figure 3c shows that the



(a) (b) (c)

Fig. 3. SEM images of SNPs on the TM surface: (a) spherical nanoparticles; (b) triangular nanoparticles; (c) silver nanowires

silver nanowires are arranged to several layers and are repeatedly intertwined forming 3D sorption cavities. The peaks at 1283 and 1351 cm^{-1} can be attributed to ν_{CN} and δ_{CH} vibrations, respectively [11, 27]. Similar differences in the spectra were observed in [11] upon 4-ATP sorption between a gold substrate and gold nanoparticles forming a sandwich structure.

Enhancement factors (EFs) of the obtained samples were calculated relative to EF of a commercial substrate manufactured by *Spektr-M* (Russia). The commercial substrate represents a silicon crystal with SNPs formed from thin silver films obtained by thermal spraying, having the known EF. For the obtained samples, EFs were calculated according to the formula:

$$K_2 = K_1 \frac{N_2}{N_1},$$

wherein $K_1 = 7 \cdot 10^6$ is the EF on a commercial substrate; K_2 is the EF on a TM with immobilized SNPs; N_1 is the Raman peak intensity on a commercial substrate; N_2 is the Raman peak intensity on a TM in the region of 1465 cm^{-1}. The values were $9 \cdot 10^6$ for spherical SNPs, $1 \cdot 10^7$ for triangular SNPs, and $4 \cdot 10^7$ for silver nanowires.

When shifting from the shape of spherical SNP to various anisotropic shapes, the EF increases. The highest EF is observed for silver nanowires. The nanowires are arranged in several layers on the TM surface. Interactions occur not only between nanowires within a single layer, but also between adjacent layers, which leads to a significant amplification of the analytical signal.

CONCLUSIONS

In this study, we synthesized nanoparticles of various shapes, including spherical, triangular, and nanowire-shaped. The shape and zeta potential were determined for all nanoparticles. The diameter of the spherical nanoparticles was 25 ± 5 nm; the edge length of the triangular nanoplates was 38 ± 8 nm; and the thickness of the silver nanowires was 26 ± 4 nm. The resulting nanoparticles were immobilized on TM surface followed by studying the SERS effect on the resulting composites. It was found that the EF increases upon the transition from a spherical nanoparticle shape to anisotropic shapes. The highest EF value was $4 \cdot 10^7$ for composite TM samples with silver nanowires. This renders these materials promising as substrates for sensors operating on the SERS effect.

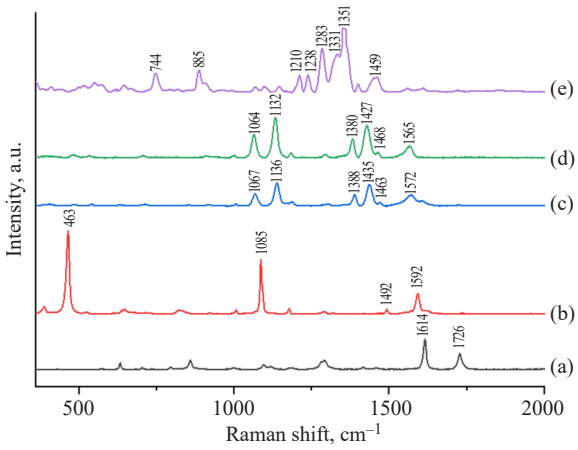


Fig. 4. Raman spectra of the native TM, 4-ATP, 532 nm laser (a); 4-ATP, 532 nm laser (b), and SERS spectra of 4-ATP on TM with SNPs of spherical, 532 nm laser (c), triangular, 638 nm laser (d) and nanowire, 638 nm laser (e) shapes

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Authors' contribution

All authors equally contributed to the research work.

The authors declare no conflicts of interest.

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