
SELF-ASSEMBLED STRUCTURES
AND NANOASSEMBLIES

New Nanocomposite Material Based on Polyurethane Foam Modified with Silver Triangular Nanoplates as a Solid-Phase Analytical Reagent for Determination of Mercury(II)

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Abstract—A method for determination of mercury(II) using polyurethane foam modified on the surface with silver triangular nanoplates that have an average edge length of 52 nm and thickness of 4 nm is developed. The method is based on the oxidation of silver nanoplates with mercury(II). This process is accompanied by a decrease in the surface plasmon resonance band of nanoparticles which allows us to consider the nanocomposite material as a solid-phase analytical reagent for the determination of mercury(II). The influence of the reaction time and pH on the sensitivity of mercury determination is studied. The detection limit of mercury under the selected conditions is equal to 50 µg/L; the range of determined contents is 150–1000 µg/L. The increase in the volume of the analyzed solution from 5.0 to 100.0 mL via concentration reduces the detection limit of mercury to 5 µg/L.

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INTRODUCTION

Recently, silver nanoparticles (NP) and materials based on them are increasingly used in analytical chemistry due to their unique optical properties and non-standard methods of an analytical signal appearance. They are used to determine metal ions, inorganic anions, and organic compounds in a wide variety of objects using the methods of optical molecular absorption spectroscopy such as colorimetry, spectrophotometry, and diffuse reflectance spectroscopy [1–4]. Silver NP and their nanocomposites are characterized by the surface plasmon resonance (SPR) phenomenon due to the interaction of conduction electrons of particles with external electromagnetic radiation. As a result of this interaction, an intense absorption band arises in the visible spectral region in the absorption spectra of NP. Its intensity, position, and shape strongly depend on the morphology, size, and degree of aggregation of NP, which is used in the chemical analysis [5–13].

In recent years, interest in the synthesis and study of the physical and chemical properties of anisotropic silver nanostructures [14–17], including triangular nanoplates (TNP) [18, 19], has noticeably increased. The possibility of obtaining various nanocomposite materials with silver NPs having specific optical properties opens the way to the manufacture of new solid-phase reagents based on them. In particular, silver

TNP and solid-phase analytical reagents based on them have already found application for the determination of chlorine [20], iodine [21], chlorides [22], iodides [23–27], nickel [28], copper [29, 30], cysteine [31, 32] and other thio-compounds [33], uric acid [34, 35], and glucose [36] using methods of optical molecular absorption spectroscopy.

The aim of this work was to assess the prospects of using a nanocomposite material based on polyurethane foam (PUF) and silver TNP as a solid-phase analytical reagent for the determination of mercury(II).

EXPERIMENTAL

Reagents, materials, and equipment. Silver(I) nitrate (PZTsM-Vtormet, analytical grade), sodium citrate (Sigma-Aldrich, ≥99.5%), polyvinylpyrrolidone (Acros Organics, $M_w = 58\,000$ g/mol, 99%), hydrogen peroxide (Sigma-Aldrich, 30% aqueous solution with stabilizer, ACS) and sodium borohydride (Acros Organics, 99%) were used to obtain silver TNP. Mercury(II) nitrate (Sigma-Aldrich, ≥98.0%), nitric acid (OAO NAK “Azot,” 68% aqueous solution, reagent grade), acetone (Sigma-Aldrich, ≥99.5%), sodium hydroxide (OOO “AO Reakhim,” reagent grade), hydrochloric acid (OOO “AO Reakhim,” 36% aqueous solution, reagent grade), and glacial acetic

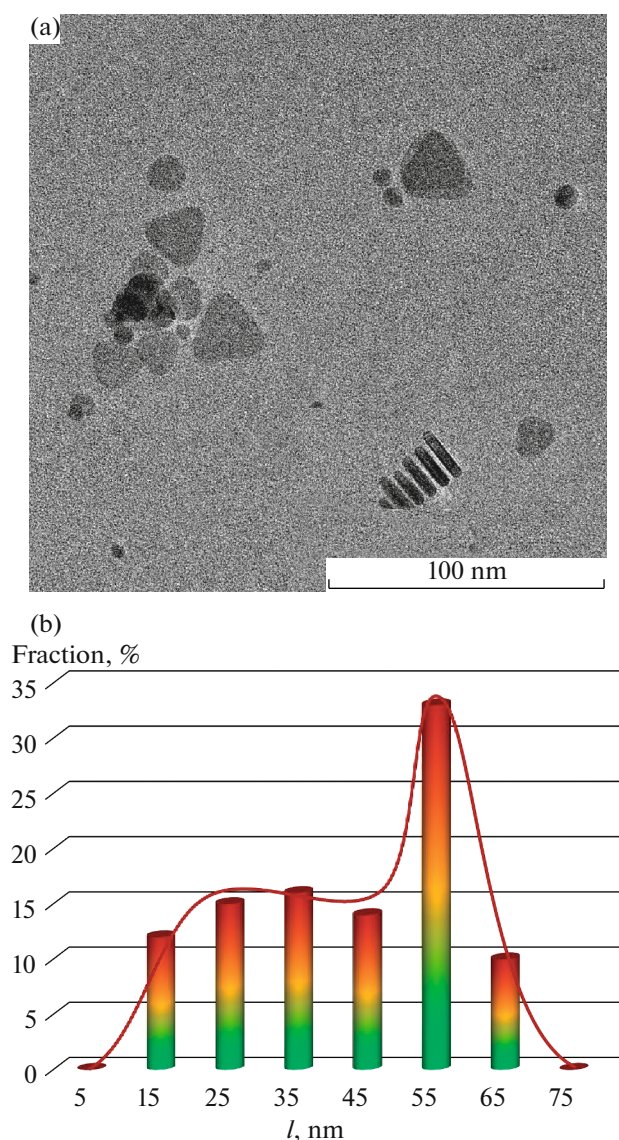


Fig. 1. (Color online) (a) Micrograph and (b) histogram of the size distribution (along the average edge length) of silver triangular nanoplates.

acid (OOO “IREA 2000,” reagent grade) were also used. Working solutions of substances were prepared by dissolving exact weights or aliquots in deionized water at room temperature.

Polyurethane foam based on polyethers in the form of cylindrical tablets (height 5 mm, diameter 16 mm, weight (20 ± 2) mg), which were knocked out of an industrial polymer sheet via metal punch, was used for the preparation of the nanocomposite material. To remove impurities, the PUF tablets were washed twice with acetone (each washing for 10 min), after which they were dried to an air-dry state.

Diffuse reflection in the visible region of the spectrum was recorded on an Eye-One Pro minispectrophotometer (X-Rite, USA). The solutions were

shaken on an Ecos PE-6500 electromechanical vibration mixer (Ecos, Russia). To measure the pH value of aqueous solutions, an Expert-001 pH meter-ion meter (Econiks-Ekspert, Russia) was used. Deionized water was obtained using a Simplicity purification system (Millipore, USA).

Electron microscopic studies of silver TNP in solution were carried out using a transmission electron microscope with a thermofield emission cathode Libra 200 (Carl Zeiss, Germany) at an accelerating voltage of 200 kV. For analysis, the samples were deposited to a copper support grid with a film of formvar and a deposited carbon layer “Formvar®/Carbon Reinforced Copper Grids 3440C-MB” (SPI, USA). Before installation under a microscope, the samples were first dried in air for 20–30 min, then in a vacuum using a Turbo Pumping Station Model 655 system (Gatan, USA) for 10–12 h.

Electron microscopic studies of polyurethane foam modified with silver triangular nanoplates (PUF/silver TNP) were carried out using a JSM 7100 F scanning electron microscope (Jeol, Japan).

Synthesis of silver triangular nanoplates. To obtain silver triangular nanoplates, a technique, proposed by Ch. Mirkin et al., with minor modifications was implemented [37]. The glassware used in the experiment was prerinse with freshly prepared nitrohydrochloric acid, thoroughly washed with distilled water, and dried in air. For the preparation of all solutions, deionized water was used. 4.3 mL of deionized water were added to 0.5 mL of a 0.01 M AgNO_3 solution, then 2.3 mL of a 1% solution of sodium citrate, 0.6 mL of a 2% solution of polyvinylpyrrolidone, and 1.2 mL of a 3% solution of hydrogen peroxide were added and vigorously stirred. Freshly prepared 0.035 M NaBH_4 solution (1.0 mL) was quickly added to the resulting colorless solution. While aging the system for 30 min, a color change was observed from yellow to blue. The resulting colloidal solution of silver TNP was stored at room temperature in a dark place. The final concentration of TNP in the solution after synthesis was $56 \mu\text{g/mL}$ (0.52 mM in terms of atomic silver).

A micrograph of the silver TNP and a histogram of size distribution (along the average length of the edge) are shown in Fig. 1. The synthesized TNP are characterized by an intense SPR band in the region of 620–700 nm. With strict control of the synthesis conditions, the position of SPR band maximum is reproduced with an accuracy of ± 5 nm. The content of silver TNP in the solutions was determined spectrophotometrically via absorption at the maximum of the SPR band using a previously obtained calibration curve.

Obtaining a nanocomposite material based on polyurethane foam and silver triangular nanoplates. To obtain solid-phase analytical reagents based on silver TNP, PUF tablets were placed in 10 mL of an aqueous solution containing 0.16 mM of TNP in terms of

atomic silver, squeezed with a glass rod to remove air and shaken on an electromechanical vibration mixer for 30 min. Then, the colored tablets were removed, dried between sheets of filter paper, and the diffuse reflection spectra of silver TNP on PUF were recorded. Colored tablets of the nanocomposite material were stored in a dark place. The content of silver TNP on PUF after synthesis was 17 $\mu\text{mol Ag/g}$.

The presence of silver TNP on PUF surface was confirmed via scanning electron microscopy (Fig. 2a). One can see that silver TNP are located on the surface of polymer membranes. The degree of their aggregation is small and the particles are mainly separated from each other.

Procedure for measuring diffuse reflection of a nanocomposite material using an Eye-One Pro minispectrophotometer. Diffuse reflection of a nanocomposite material based on silver TNP and PUF was recorded in the visible spectral region on an Eye-One Pro minispectrophotometer, which was proposed in our research group as an alternative to a diffuse reflection spectrometer [38]. This miniature USB-compatible device has a holographic diffraction grating used as a monochromator and a 128-pixel LED array as a detector. The device has a built-in radiation source of D50 type, which is a gas-filled tungsten lamp. The Eye-One Pro minispectrophotometer measures diffuse reflection coefficients R in the wavelength range from 400 to 730 nm in increments of 10 nm.

For measurements, the minispectrophotometer was connected to the computer USB port, within Microsoft Windows XP; Eye-One Share device management and data processing program was launched; and the device was calibrated against the white substrate included in the standard package. After that, samples of the nanocomposite material were measured, and the data as an array of diffuse reflection coefficients for various wavelengths were exported to Microsoft Excel 2003.

The content of silver TNP in the PUF phase was assessed by the value of the Kubelka–Munk function F at wavelength λ_{max} corresponding to the maximum of the SPR band:

$$F(R) = \frac{(1 - R)^2}{2R},$$

where R is the diffuse reflection coefficient.

To go to a value directly proportional to specific adsorption a , in all cases the values of the function were calculated:

$$\Delta F(R) = F(R) - F_0(R),$$

where $F(R)$ is the value of the Kubelka–Munk function for a PUF tablet, modified with silver TNP, and $F_0(R)$ is the value of the Kubelka–Munk function for pure PUF.

Study of the interaction of nanocomposite material with mercury(II). To study the interaction of PUF/sil-

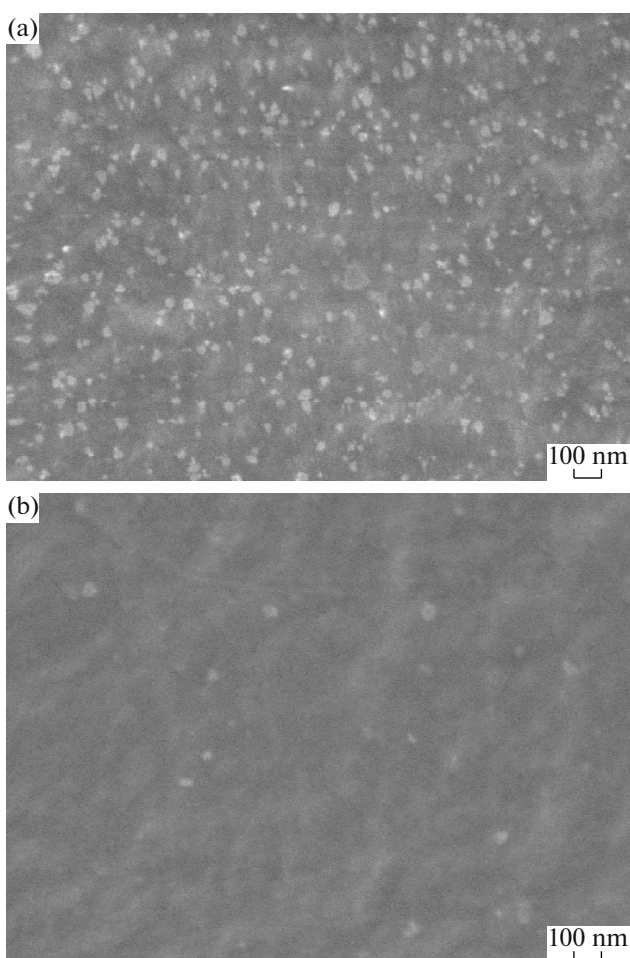


Fig. 2. Micrographs of silver triangular nanoplates on the surface of polyurethane foam (a) before and (b) after interaction with mercury(II).

ver TNP with mercury(II), 0.50 μg of analyte were introduced into polypropylene tubes and an acetate buffer solution was added to a volume of 5.0 mL. When studying the effect of pH, 0.1 M acetic acid and 0.1 M sodium hydroxide solution were mixed in different ratios in a test tube.

Then, the PUF/silver TNP tablet was placed in a test tube, squeezed with a glass rod to remove air, and shaken using an electromechanical vibration mixer at a predetermined time. After this, the tablets were removed, dried between sheets of filter paper, and the diffuse reflection spectra of silver TNP on the PUF surface were recorded.

RESULTS AND DISCUSSION

Interaction of PUF/Silver TNP with Mercury(II)

At the addition of mercury(II) to PUF/silver TNP after a few minutes, the color of the nanocomposite material weakens. In the PUF/silver TNP diffuse reflection spectra, a decrease in the intensity of the

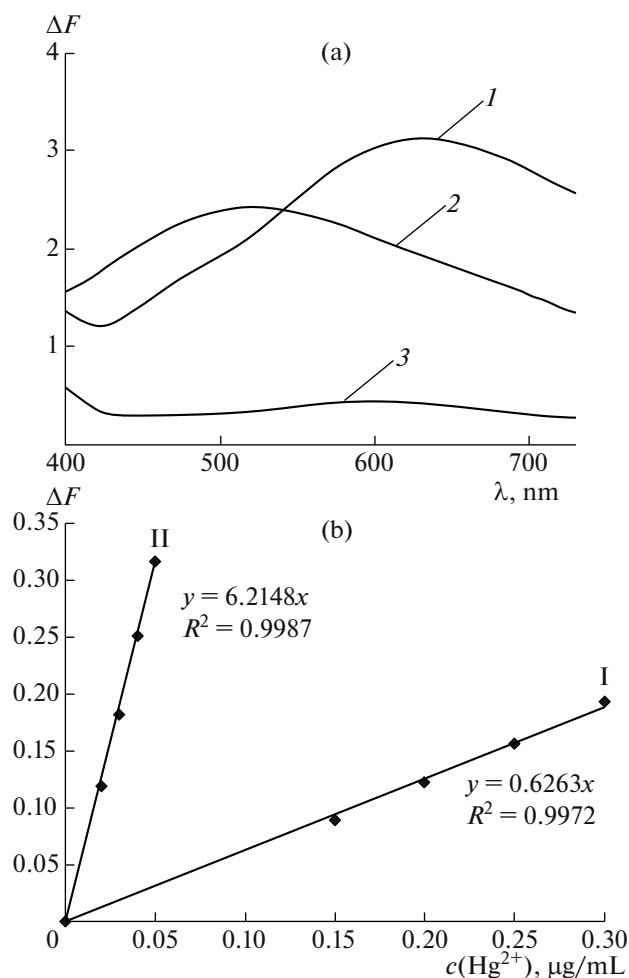


Fig. 3. (a) Diffuse reflection spectra of silver triangular nanoplates on the surface of polyurethane foam (I) before and after interaction with (2) 1 and (3) 5 $\mu\text{g/mL}$ of mercury(II) and (b) initial sections of the calibration dependences for determining mercury(II) using polyurethane foam modified with silver triangular nanoplates; $V =$ (I) 5.0, (II) 100.0 mL; $c(\text{TNP}) = 17 \mu\text{mol Ag/g}$, pH 4.5, and $t = 40$ min.

surface plasmon resonance band and its hypochromic shift are observed (Fig. 3a). The observed spectral changes can be considered as the basis for mercury(II) determination. A micrograph of silver triangular nanoplates on the PUF surface after interaction with mercury(II) is shown in Fig. 2b. Comparison of micrographs of nanoparticles shown in Fig. 2 makes it possible to conclude that most of the silver TNP after interaction with mercury(II) was oxidized and passed into solution. We note that silver TNP visible in micrographs have largely lost their initial faceting due to the oxidation of primarily the most reactive silver atoms at the vertices of triangular nanoplates, with which the observed hypochromic shift of the spectrum is probably associated [39].

Selection of Conditions for the Determination of Mercury(II)

The influence of the reaction time and pH of the solution on the sensitivity of PUF/silver TNP to mercury(II) was studied. The maximum change in the spectral characteristics of the nanocomposite material is observed 40 min after the addition of mercury(II) (Fig. 4a). Thus, according to the kinetics of interaction, the solid-phase reagent is comparable with silver TNP in solution [40]. This may indicate that the rate-limiting stage is precisely the interaction of silver TNP with mercury(II), and not interphase transfer or diffusion.

Figure 4b presents the dependence of the change in the values of the nanocomposite material Kubelka–Munk function (ΔF) in a solution containing 0.5 $\mu\text{g/mL}$ of mercury(II) from the pH value. The maximum analytical signal is observed in pH range 4–5, which is comparable to the results for silver TNP in solution [40]. At pH < 4, silver triangular nanoplates undergo changes associated with a change in shape [39], as indirectly indicated by the shift of the SPR band to the short-wavelength region. A decrease in the

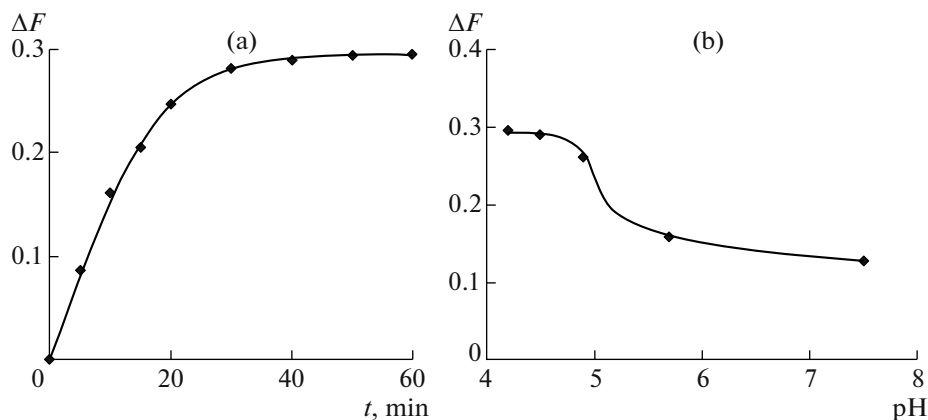


Fig. 4. Dependence of the change in the Kubelka–Munk function of a nanocomposite material based on silver triangular nanoplates and polyurethane foam in a solution containing mercury(II) on (a) time and (b) pH; $c(\text{TNP}) = 17 \mu\text{mol Ag/g}$, $c(\text{Hg}^{2+}) = 0.50 \mu\text{g/mL}$, $V = 5.0$ mL, (a) pH 4.5; (b) $t = 40$ min.

Table 1. Comparison of the proposed method with methods for determining mercury(II) using other nanoreagents

Nanoreagent	c_{\min} , $\mu\text{g/mL}$	Reference
Spherical silver NP stabilized by <i>L</i> -cysteine	3.2	[42]
Spherical silver NP stabilized by <i>Soap-root</i> plant extract	0.44	[43]
Nanocomposite based on paper and spherical silver NP	0.12	[44]
Spherical silver NP stabilized by sodium citrate	0.06	[45]
Spherical gold NP coated with silver	0.06	[46]
Silver TNP	0.02	[40]
Nanocomposite based on PUF/silver TNP	0.05 ($V = 5 \text{ mL}$) 0.005 ($V = 100 \text{ mL}$)	This study

NP is a nanoparticle.

optical density of solutions at $\text{pH} > 5$ is possibly due to a decrease in the concentration of free mercury(II) ions via formation of hydroxocomplexes. All further experiments were carried out at $\text{pH} 4.5$ which was created using an acetate buffer solution.

Assessment of the Analytical Characteristics of the Method

To obtain a calibration curve, a series of aqueous solutions containing 0 to 5 μg of mercury(II) was prepared, to which an acetate buffer solution with $\text{pH} 4.5$ was added to a volume of 5.0 mL. PUF/silver TNP was added to the resulting solution, the nanocomposite material was squeezed with a glass rod to remove air, and the solution was shaken using an electromechanical vibration mixer. After 40 min, PUF/silver TNP tablets were removed from the solution and dried between sheets of filter paper, thereafter diffuse reflection spectra were recorded. A calibration curve was constructed in the coordinates $\Delta F = f(c(\text{Hg}^{2+}), \mu\text{g/mL})$. The calibration curve, which is described using equation $\Delta F = 0.626c$ ($r^2 = 0.99$), is linear in the range 0.15–1 $\mu\text{g/mL}$. The detection limit of mercury(II), calculated via 3 s -criterion, was 50 $\mu\text{g/L}$.

The selectivity of the determination of mercury(II) with respect to certain cations and anions was evaluated. Na^+ ($m(\text{Hg}^{2+})/m(\text{Na}^+) = 1 : 4000$), K^+ (1 : 4000), Mg^{2+} (1 : 1000), Ca^{2+} (1 : 1000), Al^{3+} (1 : 1000), and Pb^{2+} (1 : 100) cations; CH_3COO^- (1 : 4000), NO_3^- (1 : 1000) anions do not interfere with the determination of Hg^{2+} . Cu^{2+} (1 : 100), Ni^{2+} (1 : 1), and Fe^{3+} (1 : 1) cations; Cl^- (1 : 250), Br^- (1 : 10), and I^- (1 : 1) anions interfere with the determination.

To improve the analytical characteristics of the determination of mercury(II), the effect of the solution volume on the analytical signal was studied (Fig. 3b). As a result of concentration, an increase in the volume of the analyzed solution by a factor of 20 with constant mercury(II) content results in a decrease in its detection limit by about 10 times—to 5 $\mu\text{g/L}$. This value is 4.4 times lower than the limit

of detection of mercury(II) using silver TNP in solution [40].

CONCLUSIONS

This study showed the possibility of using a nanocomposite material based on polyurethane foam and silver triangular nanoplates as a solid-phase analytical reagent for the determination of mercury(II). The high sensitivity of the nanocomposite material to mercury(II) compares favorably with most organic reagents (dithizone, diphenylcarbazone, sodium diethyldithiocarbamate, etc.) that form coordination compounds with mercury(II), the molar absorption coefficients of which do not exceed $7 \times 10^4 \text{ L}/(\text{mol cm})$ [41], as well as from other systems based on noble metal nanoparticles (Table 1). Other advantages of the nanocomposite material are ease of use, as well as the demonstrated possibility of using a cheap and compact minispectrophotometer, a monitor calibrator, for recording the analytical signal.

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