Synthesis of the S/AgBr nano/micropowder in DMSO-water system

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A new synthesis method of S/AgBr nano/micropowder based on sulfur and silver halide was developed. The nano/micropowder was obtained in the DMSO-water system, in two stages. Initially, sulfur was dissolved in DMSO at 120°C. The resulting solution was cooled to room temperature and separated from sulfur by filtration. Then, pre-prepared water solutions of $AgNO_3$ and NaBr were introduced drop by drop into a sulfur solution in DMSO, at room temperature. The phase composition and structure of the precipitated powdery sediment were studied by X-ray diffraction analysis (XRD) and Raman spectroscopy. Scanning electron microscopy (SEM) was used to study the morphology and particle size. XRD showed the presence of the crystalline phase of AgBr, while the Raman spectra were characterized by the presence of sulfur and AgBr peaks. The SEM results showed that the S/AgBr is represented by oval, spherical and triangular particles of a homogeneous structure; the particle size was in the range from 300 to 800 nm.

Keywords: synthesis; nano/micropowder; sulfur; silver bromide; DMSO-water system.

ДМСО-су жүйесінде S/AgBr нано/микроұнтағының синтезі

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Күкірт пен күміс галогенид негізінде S/AgBr нано/микроұнтағың синтездеудің жаңа әдісі жасалды. ДМСО-су жүйесінде нано/микроұнтақ екі кезеңмен алынды. Алдымен күкірт ДМСО-да 120°С температурада ерітілді. Алынған ерітіндіні бөлме температурасына дейін салқындатып, күкірттен сүзілді. Содан кейін алдын ала дайындалған $AgNO_3$ және NaBr сулы ерітінділері бөлме температурасында ДМСО-дағы күкірт ерітіндісіне тамшылатып қосылды. Тұнбаның фазалық құрамы мен құрылымы рентгендік фазалық талдау (РФТ) мен Раман спектроскопиясының көмегімен зерттелді. Морфологиясы мен бөлшектердің мөлшерін зерттеу үшін сканерлеуші электронды микроскопия (СЭМ) қолданылды. РФТ AgBr кристалды фазасының болуын көрсетті, ал Раман спектрлері күкірт пен AgBr шыңдарының болуымен сипатталды. СЭМ нәтижелері S/AgBr біртекті құрылымның сопақша, сфералық және үшбұрышты бөлшектермен ұсынылғанын көрсетті; бөлшектердің мөлшері 300-ден 800-ге дейін нм диапазонында болды.

Түйін сөздер: синтез; нано/микроұнтақ; күкірт; күміс бромиді; ДМСО-су жүйесі.

Синтез нано/микропорошка S/AgBr в системе ДМСО-вода

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Разработан новый метод синтеза нано/микропорошка S/AgBr на основе серы и галогенида серебра. Нано/микропорошок был получен в системе ДМСО-вода, в две стадии. Вначале серу растворяли в ДМСО при 120°С. Полученный раствор охлаждали до комнатной температуры и отделяли от серы фильтрованием. Затем, заранее приготовленные водные растворы $AgNO_3$ и NaBr по каплям вносили в раствор серы в ДМСО, при комнатной температуре. Фазовый состав и структуру выпавшего порошкообразного осадка исследовали рентгенофазовым анализом (РФА) и Рамановской спектроскопией. Для изучения морфологии и размера частиц использовали сканирующую электронную микроскопию (СЭМ). РФА показал присутствие кристаллической фазы AgBr, в то время как Рамановские спектры характеризовались наличием пиков серы и AgBr. Результаты СЭМ показали, что S/AgBr представлен овальными, сферическими и треугольными частицами однородной структуры; размер частиц лежал в диапазоне от 300 до 800 нм.

Ключевые слова: синтез; нано/микропорошок; сера; бромид серебра; система ДМСО-вода.

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1. Introduction

Nano/micropowders are materials that are represented by a large number of varieties and are used in a wide range of fields, from electrochemistry to the space industry [1,2]. To obtain such materials, various compounds can be used that can give useful properties to the final product [1,2]. When choosing a component for nano/micropowders, scientists usually rely on the specific properties of compounds and combine them to achieve the necessary goals. Speaking of the exact compounds selected for our study, silver halides (AGX, X = Cl, Br, I) are attractive because of their ability to photocatalytic and biological activity [3-6], especially in combination with other materials such as titanium oxide (TiO_2) [7], graphite carbon nitride (C_3N_4) [8,9], zinc oxide (ZnO) [4,10], polyaniline (PANI) [11,12], silver carbonate (Ag_2CO_3) [13], silver (Ag) [7] and others. As for sulfur (S), it is one of the popular agents against microorganisms [14], but it also used in many fields of science and technology: agriculture, biotechnologies and medicine, pyrotechnics, production of batteries and other [15,16]. In general, for materials based on AgX or S can be distinguished such synthesis methods like deposition-precipitation, autoclave, solid phase, mechanical activation, hydrothermal, solvothermal and self-catalytic [16-24].

The synthesis of nano/micropowder based on S and AgX is still not studied. Our research group suggested two methods of the synthesis of materials, like S@AgCl-Ag_2S nanocomposite in water media [25] and S-AgI-Ag_2S nanocomposite in DMSO media [26]. The application of the DMSO as reaction media is explained by its specific properties. DMSO is polar and aprotic solvent, which can dissolve polar and nonpolar substances and it is a cheap material that can be used in large quantities [27]. From the point of view of safety, DMSO is less of a threat compared to the other aprotic solvents, like carbon disulfide (CS_2) or toluene (C_7H_8), which have high toxicity and a wide range of concentration limits of explosivity. DMSO can be dangerous only when toxic substances are dissolved in it, and therefore it is used in biotechnologies, pharmaceuticals and medicine [27,28]. Hence, this work is devoted to the development of the synthesis method of nano/micropowder based on S and AgBr (S/AgBr) in DMSO-water system.

2. Experiment

2.1 Materials

Sodium bromide (NaBr) (JSC «Brom», Crimea), Sulfur (S) (Agros Group, Republic of Bashkortostan), silver nitrate ($AgNO_3$) (Sigma Aldrich, Germany), Dimethyl sulfoxide (DMSO, $(CH_3)_2SO$) (OJSC Mikhailovsky Plant of Chemical Reagents, Russia). All reagents were of analytical grade and deionized water was used in experiments.

2.2 Characterization

The prepared sample was characterized with help of different methods of analysis. For investigation of the phase composition X-ray diffraction (XRD) with Rigaku MiniFlex 600 X-ray diffractometer (copper radiation, λ = 0.15405 nm) and ICCD-PDF2 release 2016 database was used. For confirmation of the XRD the Raman spectroscopy was conducted in the National Nanotechnology Laboratory of Open Type (NNLOT) of Al-Farabi Kazakh National University, with Solver Spectrum (NT MDT Instruments, Russia) (1800/500 diffraction grating, which provides a spectral resolution of 1 cm^{-1}). The morphology and size of the particles was studied, through the Scanning electron microscopy (SEM) with Quanta 200i 3D microscope (FEI, Netherlands) in the NNLOT of Al-Farabi Kazakh National University.

2.3 Dissolving of S in DMSO

For dissolving of S in DMSO the installation in Figure 1 was used. The installation equipped with the chemical condenser (Figure 1a, (1)), thermometer (Figure 1a, (2)) three-neck round bottomed flask (Figure 1a, (3)), oil bath (Figure 1a, (4)) teflon magnet (Figure 1a, (5)), magnetic stirrer with heating (Figure 1a, (6)) with stirring speed and temperature controllers (Figure 1a, (7 and 8)). In routine experiment 5 g of S added into 100 mL of DMSO, then the system is exposed to the constant stirring and gradual heating. After reaching of the 120°C, S is totally dissolved in the DMSO. Such temperature regime is hold during the 20 min and then, the heating is stopped. The dissolution of S in DMSO was described in [29]. According to [29] the solubility of S at room temperature is negligible – 0.918 g/L, if we apply the same calculation to our system, we can say that the solubility of S at 120°C is about 50 g/L. After the dissolution process, the reaction mixture was cooled to room temperature and filtered on a paper filter. After separating S from the liquid, the mass of the paper filter was measured. Taking into account the difference in the filter weight before and after filtration, about 0.9563 g of S remained in the solution.

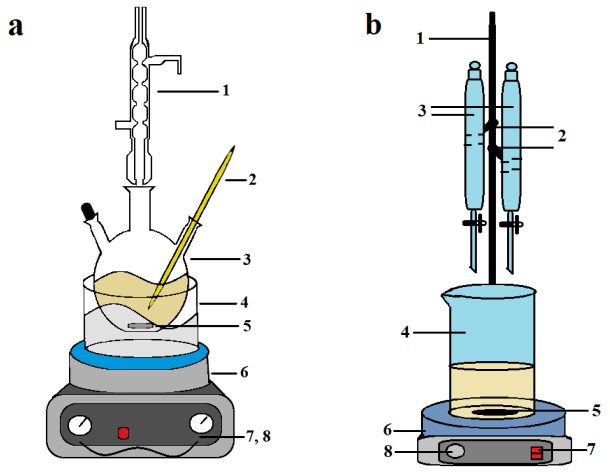

Figure 1 – The installations for synthesis: a) for dissolving of S in DMSO; b) for obtaining of S/AgBr

2.4 Synthesis of the S/AgBr nano/micropowder

AgBr was obtained on the bases of simple ion change chemical reaction (1):

$$NaBr + AgNO_3 = AgBr + NaNO_3 \quad (1)$$

Solutions of NaBr and $AgNO_3$ were prepared in water media. 0.8218 g of NaBr was dissolved in 100 mL of deionized water and 1.3564 g of $AgNO_3$ was dissolved in 100 mL of deionized water. Prepared solutions were used in subsequent synthesis of S/AgBr nano/micropowder.

For synthesis of S/AgBr nano/micropowder was used installation which is illustrated in Figure 1. The installation consists of laboratory stand and clamps for it (Figure 1b, (1 and 3)), drop funnels (Figure 1b, (2)), chemical beaker, for stirring of the reactional mixture (Figure 1b, (4)) and magnet stirrer with controllers of electric power supply and speed (Figure 1b, (6, 7 and 8)). The process of stirring was conducted by teflon magnet (Figure 1b, (5)). For the synthesis of nano/micro-powder S/AgBr, the prepared solution of S in DMSO was poured into a beaker, and the initial water solutions of NaBr and $AgNO_3$ were poured into a drop funnels. Then, with constant stirring, the gradual drop addition of water solutions to the solution S in DMSO begins. At the end of the process of adding the initial water solutions, the reaction mixture is centrifuged (4000 rpm, 20 min) and dried at 70°C for 16 hours. The final product was studied using physico-chemical analysis methods.

3. Results and Discussion

3.1 XRD analysis

XRD patterns of the S/AgBr sample represented in Figure 2. According to the obtained results, experimental sample has characteristic peaks of the AgBr. The diffraction peaks at 2Θ of 26.69°, 30.92°, 44.29°, 52.46°, 54.99°, 64.43°, 73.17° and 81.52° were related to the (111), (200), (220), (311), (222), (400), (420) and (422) crystals plans of the cubic AgBr (PDF Card No. 01-071-3754) [30]. However, the S peaks were not detected. For this reason, Raman spectroscopy was performed for the presence of S in the composition of the sample.

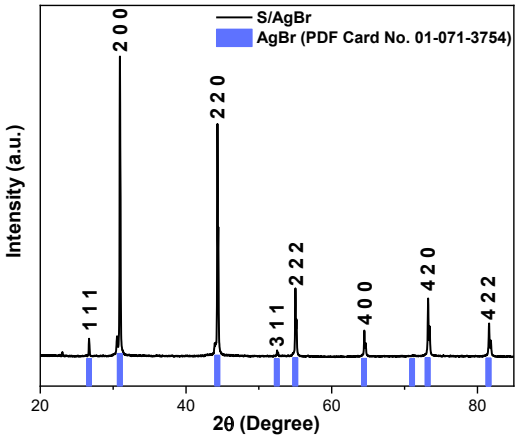

Figure 2 – XRD patterns of the S/AgBr

3.2 Raman spectroscopy

Raman spectra of the S/AgBr sample are shown in Figure 3a, b. For a better understanding of the composition of the synthesized material, various areas were considered and all characteristic peaks were shown. Figure 3a shows the characteristic peaks of S – 83, 154, 219, 472 cm^{-1} (marked with blue numbers). There are also some peaks with the next wavenumbers: 240, 390, 560, 1120 cm^{-1}, what probably can correspond to Ag_2O (marked as numbers with black color) [31].

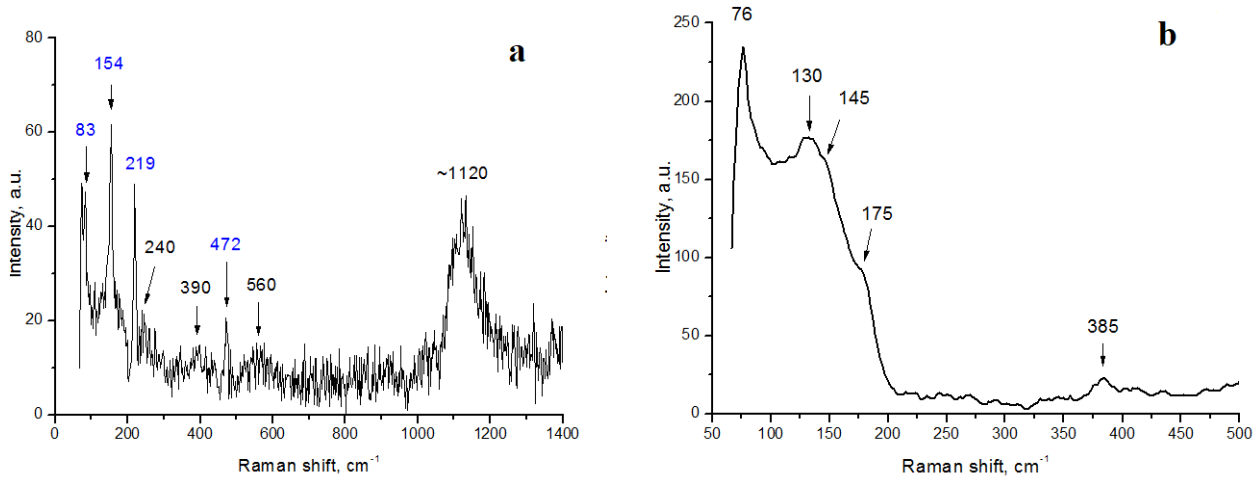

Figure 3 – Raman spectra of the S/AgBr: a) characteristic peaks of S and Ag_2O; b) characteristic peaks of the AgBr

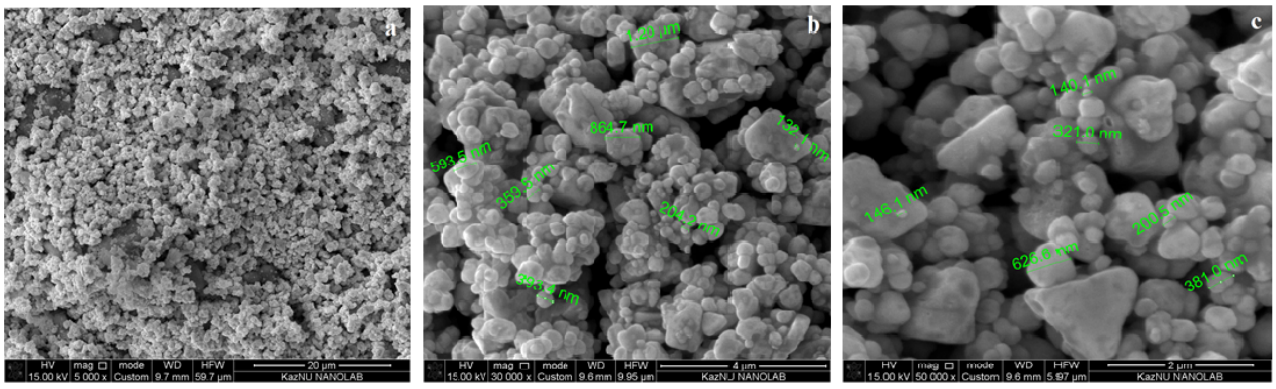

Figure 4 – SEM of the S/AgBr at different magnifications: (a) 5000x magnification; (b) 30000x magnification; (c) 50000x magnification

The formation of the Ag_2O can be explained by the influence of the laser on the AgBr and subsequent decomposition and formation of the oxide. In Figure 3b represented smoothed Raman spectra of the S/AgBr, where peaks of AgBr can be clearly seen (76, 130, 145, 175 cm^{-1}).

3.3 SEM

SEM are shown in Figure 4. Prepared sample was studied at different magnifications (Figure 4a-c). According to the analysis, obtained sample has homogenous structure with particles of smooth spherical or oval shape. There are also can be seen large triangle or rectangular grains, with the size from 1 to 2 µm. However, the average size of the sample lays in range from 300 to 800 nm.

4. Conclusions

A new and simple two-stage method for the synthesis of nano/micropowder S/AgBr in the DMSO-water system was developed. The experimental sample was characterized using physico-chemical analysis methods. The XRD showed diffraction peaks of only cubic AgBr. Raman spectroscopy proved the presence of S and AgBr. SEM showed that the sample is represented by a homogeneous system, with smooth and straight grains of oval and spherical shape. The presence of large triangular particles ranging in size from 1 to 2 µm was also observed. As for the average size, it fluctuated from 300 to 800 nm. Thus, the proposed method is of interest for further study because the resulting nano/micropowder can show itself as a universal material applicable in various fields.

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