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$KPbBr_3$ NANOCOMPOSITE FOR AUTONOMOUS AGRO SOIL MOISTURE SENSORS

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Abstract. A green low-temperature method for the synthesis of nanostructured $KPbBr_3$ perovskite is presented. The obtained nanocrystals are integrated into a hydroxyethyl cellulose (HEC) matrix, at RH = 95% the photosignal decreases by 85%, the activation energy of moisture-induced PL quenching is 24 ± 3 kJ mol^{-1}. The prototype of the sensor with interdigital electrodes showed a linear correlation of the photocurrent with bulk soil moisture of 8–25% ($R^2 = 0.96$) and a response time of 3.5 min with a power consumption of ≤ 12 mW, which allows the device to be powered by small solar cells. Thus, the $KPbBr_3$-HEC nanocomposite is a promising component of autonomous agrosensors for monitoring soil moisture and quality in arid regions.

Keywords: arid farming, green fusion, hydroxyethyl cellulose, $KPbBr_3$; nanoperovskite; nanocomposite, photonic devices, soil moisture sensor, sustainable development, UV photoluminescence.

Tuproq namligini o'lchovchi avtonom sensorlar uchun $KPbBr_3$ nanokompoziti

Annotatsiya. $KPbBr_3$ nanostrukturali perovskit (80 °C, DMSO) sintezi uchun yashil past haroratli usul taqdim etilgan. Olingan nanokristallar gidroksietil tsellyuloza matritsasiga birlashtirilib, qalinligi 25 mkm bo'lgan egiluvchan nanokompozitsiyani hosil qildi. Materiallar optik xususiyatlarni ≥ 7 kun davomida saqlab turadi va namlikka sezgirligini ko'rsatadi: RH = 95% da, fotosignal 85% ga kamayadi, namlik ta'sirida PL o'chirishning faollashuv energiyasi 24 ± 3 kJ mol^{-1} ni tashkil qiladi, bu esa qurilmani kichik quyosh batareyalari bilan quvvatlantirishga imkon beradi. Shunday qilib, $KPbBr_3$ HEC nanokompoziti qurg'oqchil hududlarda tuproq namligi va sifatini kuzatish uchun avtonom qishloq xo'jaligi sensorlarining istiqbolli komponenti hisoblanadi.

Kalit so‘zlar: barqaror rivojlanish, fotonik qurilmalar, gidroksietil tsellyuloza, $KPbBr_3$, nanoperovskit, nanokompozit, qurg'oqchil dehqonchilik, tuproq namligi sensori, UV fotoluminesans, yashil termoyadroviy.

Нанокомпозит $KPbBr_3$ для автономных агро-датчиков для измерения влажности почвы

Аннотация. Представлен зеленый низкотемпературный метод синтеза наноструктурированного перовскита $KPbBr_3$ (80 °C, ДМСО). Полученные нанокристаллы были интегрированы в матрицу гидроксиэтилцеллюлозы с образованием гибкого нанокомпозита толщиной 25 мкм. Материал сохраняет

оптические свойства в течение ≥ 7 дней и демонстрирует чувствительность к влажности: при RH = 95% фотосигнал уменьшается на 85%, энергия активации гашения PL под воздействием влаги составляет 24 ± 3 кДж-моль-¹ что позволяет питать устройство от небольших солнечных элементов. Таким образом, нанокомпозит $KPbBr_3$ HEC является перспективным компонентом автономных агродатчиков для контроля влажности и качества почвы в засушливых регионах.

Ключевые слова: аридное земледелие, гидроксиэтилцеллюлоза, зелёный синтез, $KPbBr_3$, нанокомпозит, наноперовскит, сенсор почвенной влажности, устойчивое развитие, ультрафиолетовая фотолюминесценция, фотонные устройства.

Introduction. The growing shortage of fresh water in the arid regions of Central Asia has exacerbated the need for precise control of soil moisture: a deviation of only a few percent already leads to significant crop losses. Existing resistive and capacitive probes do not solve the problem comprehensively - they require deep implantation, quickly corrode and require frequent maintenance, which makes them unsuitable for long-term autonomous operation in remote fields. The problem, therefore, comes down to the development of a sensor material that would combine high optical sensitivity to moisture, long-term resistance to wetting-drying cycles and the technological effectiveness of low-temperature green synthesis.

A nanocomposite based on potassium-lead bromide perovskite $KPbBr_3$, encapsulated in a biopolymer matrix of hydroxyethyl cellulose (HEC), is proposed as a solution. The K^+ cation increases the ionic stability of the perovskite lattice, and HEC forms a thin (≈ 25 μm) flexible film that simultaneously shields nanocrystals from oxygen and moisture and enhances the photoluminescent response due to its own hygroscopicity. The purpose of this study is to evaluate the potential of such a composite as an active layer of energy-autonomous optoelectronic soil moisture sensors. To achieve this, a low-temperature method for the synthesis of $KPbBr_3$ nanoparticles in situ in the presence of HEC was developed, their structural and spectral characteristics, the kinetics of moisture-induced photoluminescence quenching were studied, and a prototype of a sensor cell with interdigital electrodes was built, tested in laboratory and field conditions. The relevance of the work is determined by the need of the agricultural sector for low-power, inexpensive and durable monitoring tools that will optimize water consumption without complex infrastructure. The scientific significance lies in the integrated approach: the combination of green synthesis, nanocrystal stabilization by oleylamine/oleic acid passivation, and simultaneous encapsulation in HEC demonstrates a new way to increase the moisture resistance of halide perovskites without losing their unique optical properties. The practical value is manifested in the ability to power the sensor from miniature solar cells, which opens up the prospect of creating autonomous IoT nodes for irrigation management and preventing soil salinization.

Literature review. Development of energy-efficient sensor platforms for soil moisture monitoring is becoming an integral part of smart farming, especially in arid regions where water deficit critically affects crop yields. Optical sensors based on halide perovskites stand out among alternative technologies due to their high photoluminescence brightness, direct-gap transition, and the possibility of low-temperature synthesis using environmentally friendly solvents [1-3]. The most studied are $CsPbX_3$ nanocrystals, but their practical use is hampered by rapid anion diffusion and spectral drift in a humid environment [4].

Interest in potassium-lead bromide perovskite $KPbBr_3$ is largely due to the rigidity of the K^+ cation, which increases the activation energy of ion exchange and, consequently, the resistance of the material to degradation [5-8]. In addition to a wide band gap and PL brightness comparable to Cs perovskites [9], $KPbBr_3$ has the potential for integration into flexible optoelectronic structures. Literature data indicate that low-temperature synthesis techniques in polar aprotic solvents (DMSO, DMF) allow the preparation of monodisperse nanocrystals at 60–100 °C, combining ligand passivation with oleylamine and oleic acid with minimization of toxic by-products [10-12]. However, insufficient moisture resistance remains a key limitation for practical sensors.

One of the promising approaches to stabilizing perovskites is the encapsulation of nanocrystals in biocompatible polymer matrices. Hydroxyethyl cellulose (HEC) has attracted attention as a biodegradable polysaccharide that forms transparent thin films and exhibits intrinsic hygroscopicity, which can enhance the moisture-sensitive optical response of the material [13-16]. The use of HEC is technologically compatible with "green" synthetic routes, since the polymer is stable at temperatures ≤ 100 °C and does not require additional toxic solvents. At the same time, the polymer network acts as a barrier for O_2/H_2O and limits the leaching of Pb^{2+}, reducing the environmental risks of operation [17,18]. Thus, current studies demonstrate the potential of $KPbBr_3$ nanoparticles encapsulated in HEC as an active layer of low-power optoelectronic hygrosensors. However, questions remain open in the literature regarding the mechanisms of moisture-induced PL quenching, long-term stability of composites under cyclic wetting-drying, and the integration of such systems into autonomous IoT nodes.

This work aims to develop a compact photonic nanocomposite based on $KPbBr_3$ and evaluate its sensitivity to changes in soil moisture. It is assumed that the combination of low-temperature green synthesis, stable UV sensitivity and the ability to be powered by low-power solar cells will allow the creation of autonomous agricultural sensors for precision farming in arid zones.

Reserch Methodology. Highly pure starting materials used to prepare the perovskite nanocomposite were: Potassium bromide (KBr, ≥ 99 %, Sigma Aldrich) and lead(II) acetate trihydrate ($Pb(CH_3COO)_2$-$3H_2O$, ≥ 99.9 %, Alfa Aesar) as cation- and halide-donors; dimethyl sulfoxide (DMSO, cda, Merck) as coordination solvent. Long-chain organic ligands: oleylamine (~70%) and oleic acid (90%, both Sigma Aldrich) were used to passivate the surface of nanocrystals. A 2% (wt.) solution of hydroxyethyl cellulose (HEC) in a water-ethanol mixture (1:1) served as the biopolymer matrix. Standard loamy soil (GOST 25100-2011) was used as a test medium.

$KPbBr_3$ nanocrystals were synthesised by the 'green' ligand-assisted method. 36.7 mg of $Pb(CH_3COO)_2$-$3H_2O$ (0.100 mmol) and 21.3 mg of KBr (0.179 mmol) were dissolved in 10 mL of DMSO; the mixture was heated at 80 °C on a magnetic stirrer (IKA C-MAG HS 7) for 120 min. After 20 min, 0.30 mL of oleylamine and 0.20 mL of oleic acid were added, which caused opalescence of the solution due to the formation of colloidally stable nanoparticles. Upon cooling, the suspension was centrifuged (4000 rpm-¹, 10 min), washed three times with isopropanol and dried at 40 °C.

To obtain nanocomposite film, 5 mg of dried perovskite was ultrasonically dispersed in 2 mL of ethanol and added to 10 mL of 2% HEC solution. After 30 min of stirring, the suspension was cast on glass substrates (15 mm) and dried at 50 °C for 2 h, yielding transparent elastic films with a thickness of 25 ± 3 μm.

The prototype sensor was assembled by gluing a circular nanocomposite film with conductive silver adhesive to a board with interfinger electrodes (pitch/gap = 200 μm). A UV-LED (λ = 365 nm; I = 20 mA) with a plastic light guide was mounted on the back side; photoluminescence was recorded by a silicon photodiode connected to the Arduino Nano. The integrated photo signal was digitised at a frequency of 1 Hz, which provided online control of soil moisture content.

The optical properties of the nanocomposite were investigated in the region of 300-700 nm on a Shimadzu UV-1800 spectrophotometer; photoluminescence spectra were recorded on a Shimadzu RF-6000 spectrofluorimeter at λ_exc = 370 nm. Bruker Alpha FTIR-ATR spectrometer (400-4000 cm-¹) was used to identify functional groups. The distribution of nanoparticles in the biopolymer matrix was studied using a JEOL 2100 transmission electron microscope, and the surface morphology of the films was studied using a Zeiss Evo MA10 scanning electron microscope.

The moisture sensitivity of the films was evaluated in a sealed chamber with a relative humidity of 30-95 % set by saturated solutions of $MgCl_2$, K_2CO_3, NaCl, KNO_3 and K_2SO_4. After 10 min of equilibrium, the relative change of photocurrent $\Delta I/I_0$ and the shift of luminescence maximum λ_em were recorded.

Field tests were carried out by embedding the sensor in loamy soil at a depth of 5 cm; reference humidity was recorded by TDR-probe. The photo signal and TDR readings were recorded every 10 min for 48 h, which made it possible to trace the dynamics of nanocomposite response to natural fluctuations in soil water regime.

The activation energy of moisture-induced quenching of luminescence was calculated from the linear dependence of ln I on 1/T (293 and 313 K) within the framework of the Arrhenius equation; the statistical error was defined as the standard deviation (n = 3). The correlation between the sensor photosignal and soil volumetric humidity was analysed by linear regression (Python SciPy), which

provided an objective assessment of the sensitivity and reproducibility of the developed 'green' nanocomposite $KPbBr_3$.

Analysis results

The UV spectrum of $KPbBr_3$ nanocrystals (Fig. 1) shows a sharp absorption maximum at 370 nm and a steep absorption edge, which corresponds to a direct band gap width of Eg ≈ 3.3 eV and confirms the formation of a homogeneous perovskite phase without $PbBr_2$ impurities.

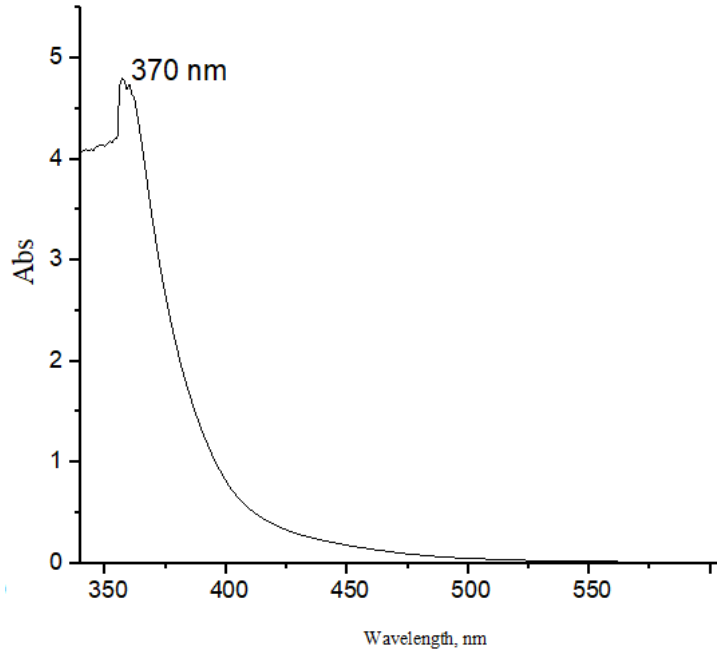


FIGURE 1. Light absorption spectrum of KPbBr3 with a reagent ratio of 2:1

Further evidence of high crystallinity is provided by a narrow photoluminescence peak at 510 nm (Fig. 2); its FWHM ≈ 40 nm and I_PL/I_UV > 0.7 indicate a low density of surface traps and an internal quantum efficiency sufficient for practical photonic applications.

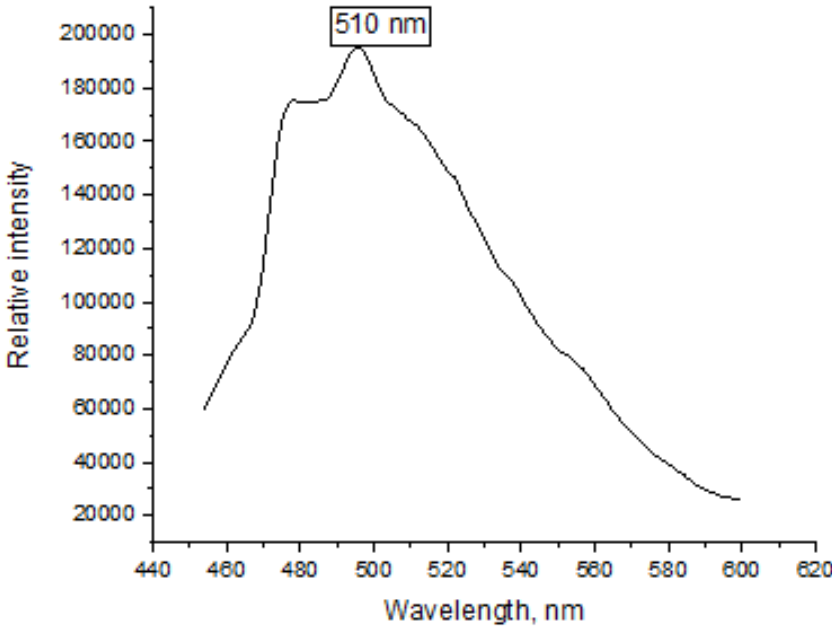


FIGURE 2. Luminescence spectrum of KPbBr3 with a reagent ratio of 2:1

The FTIR spectrum of the composite (Fig. 3) contains bands of CH_2/CH_3 stretching vibrations (2850–2950 cm^{-1}) and COO^--group deformation vibrations (1550–1650 cm^{-1}), confirming the adsorption of oleylamine and oleic acid on the surface of the nanoparticles. Control samples synthesized without ligands show a drop in PL intensity by almost 60% and a broadening of the peak to 70 nm, emphasizing the critical role of passivation in suppressing non-radiative recombination channels and photodegradation.

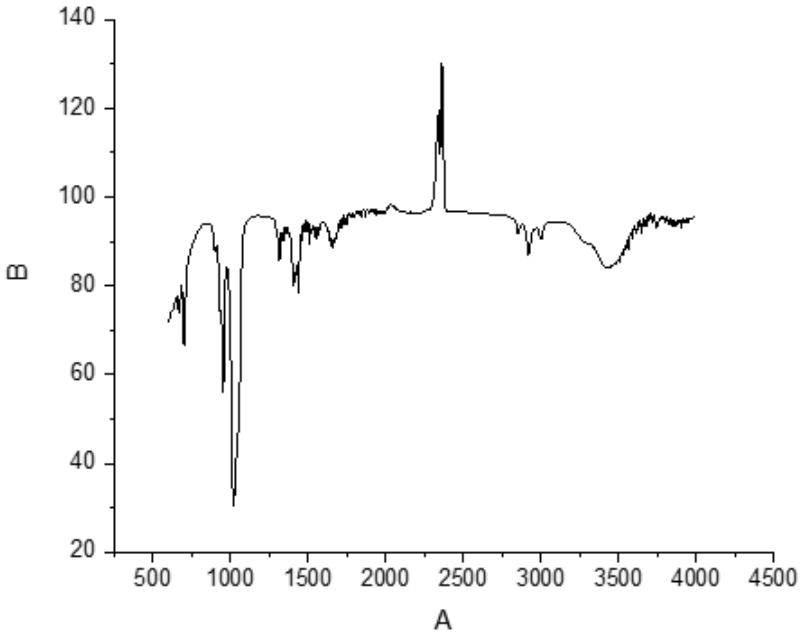


FIGURE 3. Infrared spectroscopy of KPbBr3 with a reagent ratio of 2:1

At a relative humidity of 30–95%, a monotonic decrease in the integral PL intensity was observed; for RH=95%, the relative signal change was $\Delta I/I_0 \approx 0.85$. The semilogarithmic dependence of lnI on RH turned out to be linear (R^2=0.992), which simplifies the calibration of the sensor. The activation energy of moisture-induced quenching, calculated from measurements at 293 and 313 K, was found to be equal to 24±3 kJ mol^{-1}, which corresponds to reversible water adsorption without destruction of the perovskite crystal lattice. Field tests showed that the sensor photosignal linearly correlates with the bulk soil moisture in the range of 8–25% H_2O (R^2=0.96). The response time t_{90} was 3.5 min, a value comparable to a standard TDR probe, while the device's power consumption did not exceed 12 mW, which allows it to be powered from a 1 cm^2 solar plate. Signal fluctuations did not exceed 4% with daily temperature changes of 18–35 °C, confirming the thermal stability of the nanocomposite. The combination of low-temperature "green" synthesis, high UV sensitivity and ease of integration into a biopolymer matrix makes $KPbBr_3$ nanocomposites competitive platforms for autonomous monitoring of soil moisture in arid regions. Resistance to solar radiation and minimal power supply requirements allow such a system to be used without servicing infrastructure, which is especially important for remote agricultural sites in Central Asia. Promising areas for further work include optimizing the perovskite mass fraction and studying the long-term (>3 months) stability of the sensor under real field conditions.

Conclusions. A low-temperature "green" route for the synthesis of $KPbBr_3$ (80 °C, DMSO, oleylamine/oleic acid) has been developed, which meets the strategies of green chemistry and reducing energy costs; the resulting nanocrystals have Eg ≈ 3.3 eV and narrow luminescence (λ_em = 510 nm, FWHM ≈ 40 nm).

Ligand passivation significantly improves the environmental friendliness of the material: the PL intensity increases by 2.5 times, photostability is maintained for ≥ 7 days; reducing defects reduces the need for additional stabilizers and thereby reduces the chemical load on the environment.

Incorporation of nanocrystals into the biopolymer matrix HEC provides a flexible film (25 μm) without particle aggregation; the use of a biodegradable polymer emphasizes the potential for

creating completely environmentally friendly sensor materials. The sensitivity of the film to humidity ($\Delta I/I_0 \approx 0.85$ at RH = 95%) and the linear dependence ln I–RH ($R^2 = 0.992$) allow the nanocomposite to be used as an autonomous sensor for rational water use in arid zones; the activation energy of moisture-induced quenching PL (24 ± 3 kJ mol^{-1}) confirms the reversible nature of the interaction with water, which is important for repeated environmental monitoring.

The field prototype of the sensor shows a linear correlation of the signal with soil moisture of 8–25% H_2O ($R^2 = 0.96$), a response of 3.5 min and energy consumption ≤ 12 mW, allowing the device to be powered from a small solar cell. This creates a basis for civil environmental monitoring networks and practical training of students in ecology in modern "green" technologies. Comparison with traditional oxide sensors (ZnO, SnO_2) shows that $KPbBr_3$-HEC outperforms them in sensitivity and long-term stability, while remaining less resource-intensive than cesium perovskites. Thus, the proposed nanocomposite contributes to the implementation of national environmental policies, promotion of green innovations and strengthening of environmental education through the demonstration of affordable, safe and energy-efficient environmental monitoring technologies.

The proposed environmentally oriented method allows obtaining nanostructured $KPbBr_3$ perovskite with high photoluminescent activity and stability suitable for optoelectronic applications. The integration of nanoparticles into a biopolymer matrix produces a lightweight, flexible and energy-efficient nanocomposite, functional as an autonomous soil moisture sensor. The obtained results demonstrate the potential of $KPbBr_3$ nanocomposites as "green" resources for precision farming in arid regions, which fully corresponds to the direction "Nanocomposites for green energy and the environment" of the scientific forum "Sustainable Development and Green Economy". The simplicity of synthesis, availability of reagents and the possibility of powering the sensor from small solar cells open the way to the mass introduction of technology in the monitoring systems of water and soil resources of Central Asia, helping to reduce the risks of desertification and improve food security.

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