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Multifunctional nanodispersed X-ray phosphors in a drug delivery system in X-ray photodynamic therapy

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A promising approach that can provide minimally invasive treatment, in particular, of malignant tumours located both in soft tissues and in hard-to-reach areas of the body (e.g., in the cranial cavity), is the implementation of the principles of the latest scientific and practical interdisciplinary direction – X-ray excited photodynamic therapy (XE-PDT), in which X-rays are used to activate the photosensitive component by transferring energy from phosphors. The concept of XE-PDT is actively developing worldwide, and the implementation of its results in clinical practice can provide solutions to many modern medical and biological problems, such as drug resistance, low selectivity and safety of cytotoxic drugs.
Thus, the development of functional, highly efficient, biocompatible, nanoscale phosphors that are sensitive to biologically safe, highly penetrating ‘soft’ X-ray radiation with specified luminescence spectral characteristics for the implementation of the XE-PDT method is a pressing task.
Based on the focus of the work, nanodispersed phosphors based on lanthanum fluoride (or phosphate) and hydroxyapatite activated by REE ions (terbium) ions (LaF3:Tb3+, LaPO4:Tb3+ and HA:Tb3+), obtained by co-precipitation from aqueous solutions. These phosphors are characterised by high quantum yield, significant Stokes shift, low toxicity and high biocompatibility, high chemical stability, and clear emission bands.
A comprehensive study and comparison of physicochemical characteristics was conducted, and nanostructure synthesis technologies were optimised. It was established that the X-ray luminescence spectra show bands characteristic of optical transitions in Tb3+-activated phosphors, and their spectral position under the synthesis conditions used depends little on the chemical composition of the nanocrystalline matrices. It was found that high amorphisation and defectiveness of the crystal structure of the samples have little effect on the position of Tb3+ energy levels in their forbidden zone. However, they significantly affect the intensity of X-ray luminescence, in particular due to scattering on defects in the crystal structure. In addition, water molecules on the surface of the phosphor, in the composition of crystal hydrates, as well as surface hydroxyl groups can be centres of luminescence quenching.
Morphological features were noted: LaF3:Tb3+ is characterised by spherical nanoparticles (Dav =11.17±3.09 nm), LaPO4:Tb3+ exhibits slight anisotropy in crystallite growth (Lav=19.92±4.55 nm), while HA:Tb3+ crystallites are characterised by significant anisotropy in growth and a rod-like shape (Lav=54.28±11.87 nm).
The results of XRD studies indicate the formation of single-phase crystals, for which the angular position, shape, width and intensity ratio of the reflections practically do not differ from the diffractograms of the corresponding terbium-doped nanostructure samples. The features of the diffractograms include: 1 – broadening of diffraction reflections, 2 – presence of a diffuse halo in the diffraction angle range of 15–40 degrees for all test samples. The first indicates the nanocrystalline nature of the particles, while the second is associated with partial amorphisation of the phases.
It has been shown that in an NSS medium at physiological pH values of 6–7, the ζ-potential values for the synthesized samples indicate acceptable colloidal stability of the suspensions. For LaF3:Tb3+ (ζ = 28 mV, pH = 2) and LaPO4:Tb3+ (ζ = 23 mV, pH = 3) samples are characterised by colloidal stability in an acidic environment. For HA:Tb3+, colloidal stability increases in an alkaline environment (ζ = −27 mV at pH 11).
A significant difference in the quantitative distribution of basic and acidic active centres on the surface is shown: there are both strong basic (electron-donating orbitals F−, oxygen in PO4−, OH−) and weak acidic

(electron-accepting orbitals La3+, Ca2+, Tb3+) Lewis centres.

The physicochemical characteristics of HA:Tb3+ nanoparticles functionalised with citrate groups (HA:Tb3+/Citr) were synthesised and studied. Functionalization contributes to increased colloidal stability in a wide pH range (5–11), creates additional active centers (carboxyl groups –COOH), and increases the biocompatibility of nanoparticles, which is important for materials used in medical and biological applications. In vitro biocompatibility studies conducted in collaboration with specialists from the O.O. Bogomolets Institute of Physiology of NAS of Ukraine confirmed the biocompatibility of the developed material.

Based on HA:Tb3+/Citr, a targeted delivery model was created and studied, which can be applied within the concepts of photopharmacology and photodynamic therapy. The study used a photosensitive complex compound of ruthenium-bipyridine-nicotine complex [Ru(bpy)2(Nic)2]Cl2 (RuBiNicotine), which was immobilised on the surface of HA:Tb3+/Citr nanoparticles. When irradiated with light in the wavelength range of 405 nm, 473 nm or 532 nm (for synthesised samples, X-ray-induced luminescence is recorded at 487, 542, 582 and 620 nm), it photolyzes with the release of nicotine. A quantitative determination of the released nicotine was carried out for samples under UV irradiation and X-ray irradiation. As a result of X-ray irradiation, 4.17% of nicotine was released, and under UV irradiation, 98.26% was released.

The research results may be useful for application in the fields of photopharmacology and photodynamic therapy in the development of targeted drug delivery systems and local therapy for diseases.

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