

Structure, optical spectroscopy and cytotoxicity of dielectric zirconia-based nanoparticles for biophotonic applications

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Received December 27, 2024

Revised February 13, 2025

Accepted April 07, 2025

The results of the study of $\text{ZrO}_2 - 30 \text{ mol.\% Yb}_2\text{O}_3$ nanoparticles of different sizes, which can be used for the treatment of superficial tumors, are presented. Excitation by laser radiation with $\lambda = 980 \text{ nm}$ and a power density of $J = 0.9 \text{ kW/cm}^2$ led to heating of all samples. The cytotoxicity of particles was studied on the cell culture of mouse hepatoma Mh22a, as well as on spheroids formed from this cell culture. It was found that particles with a coherent scattering region size of 99 nm are characterized by the highest cytotoxicity at concentrations of 50 and 25 mg/ml when exposed to 980 nm laser radiation.

Keywords: nanoparticles, rare earth ions, cytotoxicity.

DOI: 10.61011/EOS.2025.05.61651.30-25

Introduction

The excitation of dielectric particles with a high content of rare earth (RE) ions by intense laser radiation can lead to a significant increase in their temperature up to the appearance of „white“ radiation [1–3]. This effect can be used to enhance the heating of biological tissue under the action of laser radiation [4]. We showed in Ref. [4] that the preliminary application of ytterbium-containing particles to the surface of rat skin leads to much more pronounced thermal damage under the action of laser radiation with a wavelength of 980 nm than without particles. Subsequent experiments *in vitro* and *in vivo* with particles of different compositions and radiation with different wavelengths revealed that the amplification effect is most pronounced for compounds based on zirconium dioxide with ytterbium in combination with radiation with $\lambda = 980 \text{ nm}$ [5,6]. For example, exposure to laser radiation of the specified wavelength with a power density of 865 W/cm^2 (power 1 W) led to heating of biological tissue (skin surface of chicken breast) to 33°C , and when using nanoparticles $\text{ZrO}_2 - x \text{ mol.\% Yb}_2\text{O}_3$ ($x = 30, 50$) — up to $180\text{--}200^\circ\text{C}$. At the same time, heating to 100°C took in average 2 s . Moreover, we demonstrated the possibility of using particles of $\text{ZrO}_2 - 30 \text{ mol.\% Yb}_2\text{O}_3$ (cutaneous or intracellular location) and the specified radiation for the treatment of melanoma in Ref. [6] during experiments *in vivo* on mice. At the same time, a more intense inhibition of tumor growth and a greater median survival rate of animals were observed with the intra-tumor administration of a suspension of these particles. Along with the thermal effect on tumor cells, there may be a cytotoxic effect of the particles themselves. The purpose of this paper was to study

the effect of the particle sizes of $\text{ZrO}_2 - 30 \text{ mol.\% Yb}_2\text{O}_3$ compounds on their emission spectra and cytotoxicity.

Materials and methods

The particles of $\text{ZrO}_2 - 30 \text{ mol.\% Yb}_2\text{O}_3$ were obtained by co-deposition. The initial reagents used were $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$ (Nevatorg, 99.99%) and $\text{YbCl}_3 \cdot 6\text{H}_2\text{O}$ (Nevatorg, 99.99%). For synthesis in 30 ml . $0.1 \text{ mol.\%}$ of an aqueous solution of a mixture of zirconium and ytterbium salts (the ytterbium chloride content was $30 \text{ mol.\%}$) was added with stirring to 50 ml $25 \text{ mass\%}$ ammonia solution (the ammonia solution was taken with a 20% excess). The resulting precipitate was aged for one hour, then washed and dried in a drying cabinet at 80°C . The resulting product was ground in a mortar and divided into three parts, which were annealed at various temperatures — $500, 800$ and 1150°C for 6 h .

The phase composition of the samples was studied by X-ray diffractometry using an Emyrean diffractometer (Malvern Panalytical, Great Britain) with a vertical goniometer and detector PIXcel 3D; radiation $\text{CuK}\alpha$, $\lambda = 1.5414 \text{ \AA}$. The morphology and sizes of the obtained particles were studied by transmission electron microscopy (TEM) using JEOL 2100 microscope (Japan) at an operating voltage of 200 kV . The particle sizes were also estimated by dynamic light scattering using the NanoFlex nanoparticle size analyzer (Microtrac, Japan).

A semiconductor laser diode with $\lambda = 980 \text{ nm}$ (maximum output power 2 W , continuous operation) was used as an excitation source for spectroscopic studies of the samples. The laser power was monitored using the Standa 11 PMK-30H-H5 PowerDetector power meter (Lithuania).

The radiation was focused on the surface of the sample using a lens. The beam waist diameter was determined using a scanning knife [7] and was $350\mu\text{m}$. The Aurora 4000 spectrometer was used as a radiation receiver. The light filter SZS-5 with a thickness of 2.1 mm was used to cut off exciting radiation. All measurements were carried out at room temperature. The color temperature of the radiation was estimated by the spectral pyrometry method described in Ref. [8] from the radiation spectra adjusted for the spectral sensitivity of the installation. In this case, the radiation of a tungsten lamp with a color temperature of 2796 K (Thorlabs) was used as gray body radiation, which was also recorded using the SZS-5 light filter. Images showing the dynamics of the radiation color of the samples were obtained by extracting frames from a video (30 frames per second) recorded using a SonyNex F3k digital camera.

The cytotoxicity of particles ZrO_2 –30 mol.% Yb_2O_3 was studied on a cell culture of mouse Mh22a hepatoma (Institute of Cytology of RAS; LLC „BioloT“). In the exponential growth phase, the cells were dispersed into a 96-well plate at a concentration of 5000 cells/well and were incubated for 24 h under standard conditions on DMEM medium with the addition of 10 % FBS and antibiotics (penicillin-streptomycin) at 5% SD_2 and a temperature of 37°C .

Samples of ZrO_2 – 30 mol.% Yb_2O_3 particles annealed at 500 (group 2), 800 (group 3) and 1150 (group 1) $^\circ\text{C}$, were transferred to suspension in isotonic sodium chloride solution. Before adding the suspension to the wells of the tablet, the particles were treated with ultrasound at a power of 50 W (Sapfir UZV-1.3 TTHz, Russia). The cytotoxic effect was determined at particle concentrations in the well of the tablet: 3.125, 6.25, 12.5, 25, 50 mg/ml. 24 h after the suspension was applied, the cells were exposed to laser irradiation at a power density of 1.5 W/cm^2 (the focusing lens was not used and, thus, the entire area of the well was exposed) and until the dose reached 180 J/cm^2 , which corresponded to 2 min exposure in each well. The positive control was cells that were irradiated with the same laser with the above exposure mode, but without introducing particles.

24 h after irradiation, the cells were trypsinized. The detached cells were cleaned of particles and transferred to another 96-well plate with medium. The cell viability was assessed using the MTT test after 24 h incubation [9]. To do this, the medium in the tablets was replaced with a 5% MTT solution (3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyl). MTT was reduced to formazane crystals, which was evaluated using an inverted light microscope. Then the medium was removed, $150\mu\text{l}$ DMSO was added and shaken for 20 min at 37°C . The optical density was measured on a Varioskan Lux device (Thermo Scientific, USA) at a wavelength of 570 nm, using an optical density of 650 nm as a reference.

Cell viability in all experimental groups was assessed relative to a control series of wells (negative control), in which no particle samples were placed and no laser irradiation was performed.

The differences in cell culture viability were evaluated with the number of repeats of the experiment $n = 3$. The significance level of the differences was determined using U-test Mann-Witny with a critical significance level $p < 0.05$). The viability values were expressed as the mean, $\pm$ standard deviation.

The cytotoxicity of particles on a 3D model of tumor growth was evaluated on a cell culture of mouse Mh22a hepatoma (Institute of Cytology of RAS), from which spheroids were formed. The cells were cultured for 24 hours on DMEM medium. The cells were removed from the vial using a trypsin solution, counted using a counter RWD C-100 (China) and placed in a tablet for the formation of spheroids SPL3D, SPL Livescience (Korea) at a concentration of 10,000 per well. The formation of spheroids was observed using an inverted Micromed microscope (Russia). After 5 days of formation, the spheroids were transferred to a 96-well plate. After formation, the spheroids were divided into three groups. The concentration of ZrO_2 – 30 mol.% Yb_2O_3 in the well of the spheroid tablet was 12.5, 25, 50 mg/ml. The spheroids were incubated with added particles for 24 h. 24 h after the suspension was applied, the cells were subjected to laser irradiation at a power density of 1.5 W/cm^2 until the dose reached 180 J/cm^2 , which corresponded to 2 min exposure in each well. Spheroids irradiated with the same laser with the above exposure mode, but without introducing particles were used as a positive control.

The viability of the spheroids was assessed 24 hours after incubation after laser exposure. The spheroids were cleaned of particles by repeatedly changing the medium. The medium with the particles was removed. PBS with fluorescent dyes ethidium bromide (EB, stains the nuclei of dead cells in red color) and acridine orange (AO, stains the nuclei of living cells in green color, stains the cells in apoptosis in yellow or orange color) were added to the spheroids to visualize viable spheroids (in the absence of ethidium bromide fluorescence). The staining time was 30 min. The fluorescence of spheroids was evaluated using an inverted luminescent microscope BM35FXT, ICOE at an excitation wavelength of 460–480 nm for AO and 520–530 nm for EB. The viability of the spheroids was assessed by the intensity of EB fluorescence using image analysis software Image J [10].

2.5% tween-80 solution was used to calibrate the method, which exhibits high cytotoxicity against Mh22a cells and causes intense cell glow when stained with ethidium bromide. Spheroids were used as a negative control, to which a DMEM medium without particles was added.

Results and discussion

X-ray phase analysis of particles of ZrO_2 – 30 mol.% Yb_2O_3 confirmed that all of them are single-phase and represent solid solutions with a cubic structure (PG $Fm\bar{3}m$) [11] (Fig. 1). The sizes of

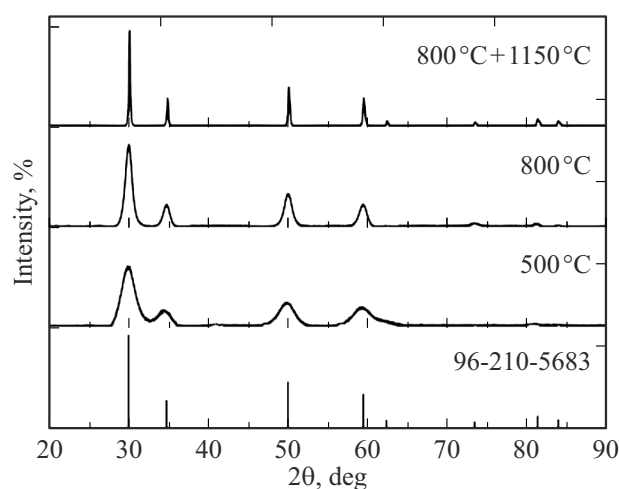


Figure 1. Diffractograms of solid solutions of $\text{ZrO}_2 - 30 \text{ mol.}\% \text{Yb}_2\text{O}_3$ annealed at various temperatures. PDF-96-210-5683 — cubic phase [11].

the coherent scattering regions (CSR) increased with the increase of annealing temperature and amounted to 5, 10, and 99 nm for 500, 800, and 1150 °C, respectively. A similar trend was noted by the authors of Ref. [12] for zirconium dioxide stabilized with erbium oxide.

The crystallite sizes estimated using TEM images (Fig. 2, *a–c*) correspond to the size of the CSR. In this case, the particle sizes range from 100–400 nm and also increase with the increase of the annealing temperature. The results of measurement by dynamic light scattering are consistent with the TEM data (Fig. 2, *d–f*).

A study of the spectral and luminescent characteristics of particles of $\text{ZrO}_2 - 30 \text{ mol.}\% \text{Yb}_2\text{O}_3$ revealed the following. When they were excited by continuous laser radiation with $\lambda = 980 \text{ nm}$ and different values of power density, broadband „white“ radiation in the range of 400–900 nm was observed. As an example, Fig. 3, *a* shows the emission spectra at a power density of $J = 1.3 \text{ kW/cm}^2$. The shape of the described radiation contour is similar to the contour of the radiation spectra of nanoparticles of $\text{ZrO}_2 - x \text{ mol.}\% \text{Yb}_2\text{O}_3$ ($x = 5 - 50$) [4], as well as particles of $\text{Y}_{0.95(1-x)}\text{Yb}_{0.95x}\text{Er}_{0.05}\text{PO}_4$, YbPO_4 [3] and the radiation spectrum of a heated gray body (tungsten lamp with color temperature 2796 K). Based on this similarity, it can be concluded that the radiation of particles of $\text{ZrO}_2 - 30 \text{ mol.}\% \text{Yb}_2\text{O}_3$ is also thermal. The change in the annealing temperature of the particles and, accordingly, their sizes did not significantly affect the shape of the contour of their radiation spectra. Thus, the color temperature of the radiation decreases from 2651 to 2312 K at $J = 1.3 \text{ kW/cm}^2$ with an increase in the size of the CSR.

The dependence of the intensity I of the broadband radiation on the excitation power P was measured at a wavelength of 590 nm (Fig. 3, *b*). This wavelength falls within the maximum sensitivity region of the detector and corresponds to the region of the particle emission

spectrum in which there is no ytterbium luminescence band. The tangent of the slope of the dependence $I(P)$ for the studied samples differed slightly and averaged $N = 4.4$, which indicates the nonlinear nature of the dependence $I(P)$. The threshold value of the excitation power density for all samples was also the same and equal to $J = 0.9 \text{ kW/cm}^2$. The values N and J differ from the previously obtained $N = 11$ and $J = 0.52 \text{ kW/cm}^2$ [4] for particles $\text{ZrO}_2 - x \text{ mol.}\% \text{Yb}_2\text{O}_3$ ($x = 5 - 30$), which may be attributable to different particle sizes. There was no radiation in the samples at a power density of exciting radiation below $J = 0.5 \text{ kW/cm}^2$.

A study of the temporal dynamics of the color of particle radiation has shown a characteristic tendency for it to shorten the burn-up time with an increase in the power density of the exciting radiation. Thus, for particles annealed at 500 °C, the burn-up time at J from 1.3 kW/cm^2 was on the order of 0.3 s, increasing J to 2.1 kW/cm^2 resulted in a two-fold reduction in burn-up time (Fig. 4). When samples subjected to annealing at different temperatures were excited by radiation with the same power density, thermal radiation flared up faster in samples with the smallest particle size (500 °C) than in the others (Fig. 4). So, for $J = 2.1 \text{ kW/cm}^2$, this value was 0.15 s (500 °C) and 0.3 s (800 and 1150 °C).

Thus, studies of spectral luminescent properties have shown that solid solutions of $\text{ZrO}_2 - 30 \text{ mol.}\% \text{Yb}_2\text{O}_3$ with different particle sizes are similarly capable of heating when excited by laser radiation with $\lambda = 980 \text{ nm}$. Therefore, all of them can be suitable for enhancing the thermal effect of this radiation on biological tissue. Cytotoxicity studies *in vitro* of particles of $\text{ZrO}_2 - 30 \text{ mol.}\% \text{Yb}_2\text{O}_3$ have shown the following. No decrease of viability was observed in the negative control group. Exposure to particle-free laser radiation also did not significantly decrease the percentage of cell viability.

A significant inhibition to $51.9 \pm 1.9\%$ and $48.5 \pm 5.6\%$ ($p < 0.05$) for concentrations of 50 and 25 mg/ml was observed in group №1 ($\text{ZrO}_2 - 30 \text{ mol.}\% \text{Yb}_2\text{O}_3$, 1150 °C) (Fig. 5, *a*). At high dilutions, there was no cytotoxic effect in this group. According to the results of light microscopy, most of the cells were preserved, adhered to the plastic surface, and the cells had clear contours. In the group exposed to laser radiation at the same particle concentrations, the percentage of viable cells was significantly lower than in the negative control group and amounted to $16.1 \pm 5.4\%$ and $20.1 \pm 8.3\%$, respectively (Fig. 5, *a*). Fig. 5, *a* shows that with a decrease in the concentration of particles in the medium, cell viability increases to the level recorded in the control at a concentration of 12.5 mg/ml, and at a concentration of 3.125 mg/ml, culture growth was stimulated.

The death of most of the cells containing $\text{ZrO}_2 - 30 \text{ mol.}\% \text{Yb}_2\text{O}_3$ particles in the cytoplasm was noted according to the results of light microscopy (cells floated, had a rounded shape, organelles were not detected).

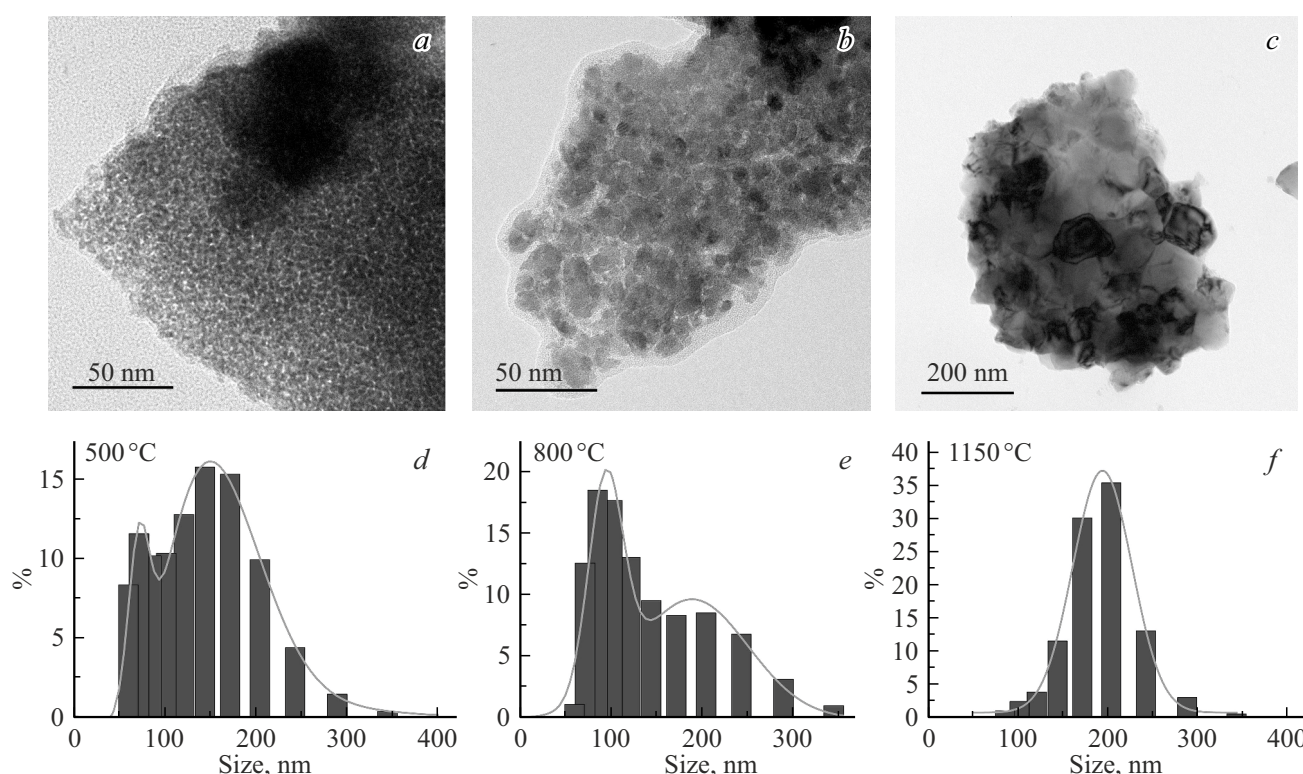


Figure 2. TEM images of solid solutions of $\text{ZrO}_2 - 30 \text{ mol.}\% \text{Yb}_2\text{O}_3$ annealed at 500 °C (a), 800 °C (b) and 1150 °C (c). Particle size distribution curves obtained by dynamic light scattering for solid solutions of $\text{ZrO}_2 - 30 \text{ mol.}\% \text{Yb}_2\text{O}_3$ annealed at 500 °C (d), 800 °C (e) and 1150 °C (f).

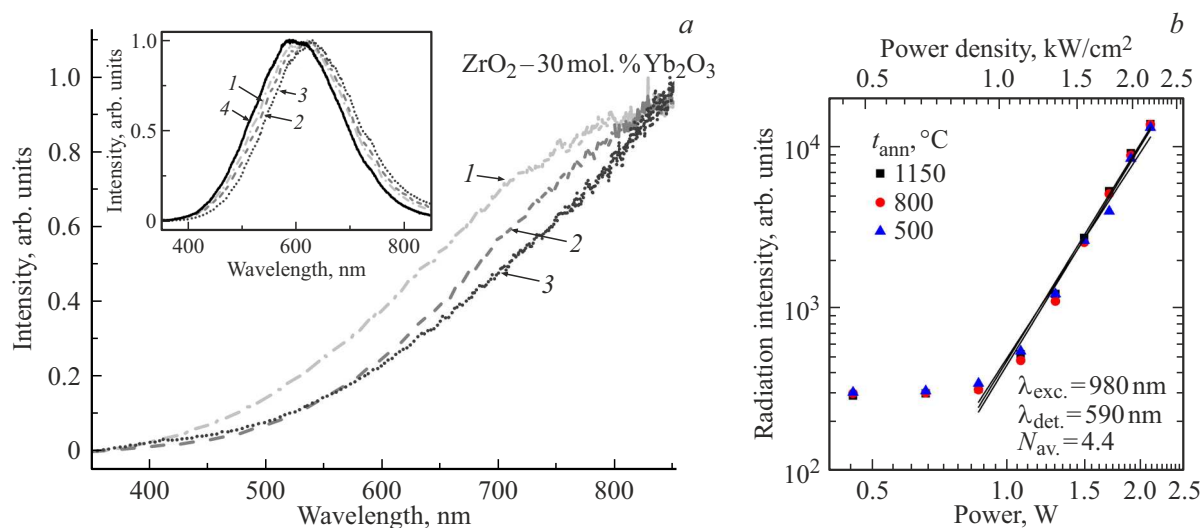


Figure 3. (a) Emission spectra of particles of $\text{ZrO}_2 - 30 \text{ mol.}\% \text{Yb}_2\text{O}_3$ with different annealing temperatures ($\lambda = 980 \text{ nm}$, $J = 1.3 \text{ kW/cm}^2$): 500 °C (1), 800 °C (2), 800 + 1150 °C (3) adjusted for the spectral sensitivity of the installation (in the insert: uncorrected emission spectra of particles and a tungsten lamp with a color temperature of 2796 K (4)). (b) Dependence of the intensity I of broadband radiation of particles of $\text{ZrO}_2 - 30 \text{ mol.}\% \text{Yb}_2\text{O}_3$ on the power P of exciting radiation, N is the tangent of the angle of dependence $I(P)$.

Intrinsic toxicity was noted in all tested concentrations for particles of the group №2 ($\text{ZrO}_2 - 30 \text{ mol.}\% \text{Yb}_2\text{O}_3$, 500 °C) (Fig. 5, b). The viability of the cell culture did not exceed 50 %, regardless of the presence or absence

of laser exposure. Significant inhibition to $16.5 \pm 4.8 \%$, $23.4 \pm 5.3 \%$, $45.3 \pm 6.6 \%$ in the range of concentrations of $50 \pm 12.5 \text{ mg/ml}$ was recorded in the group with the laser radiation exposure. The percentage of culture viability

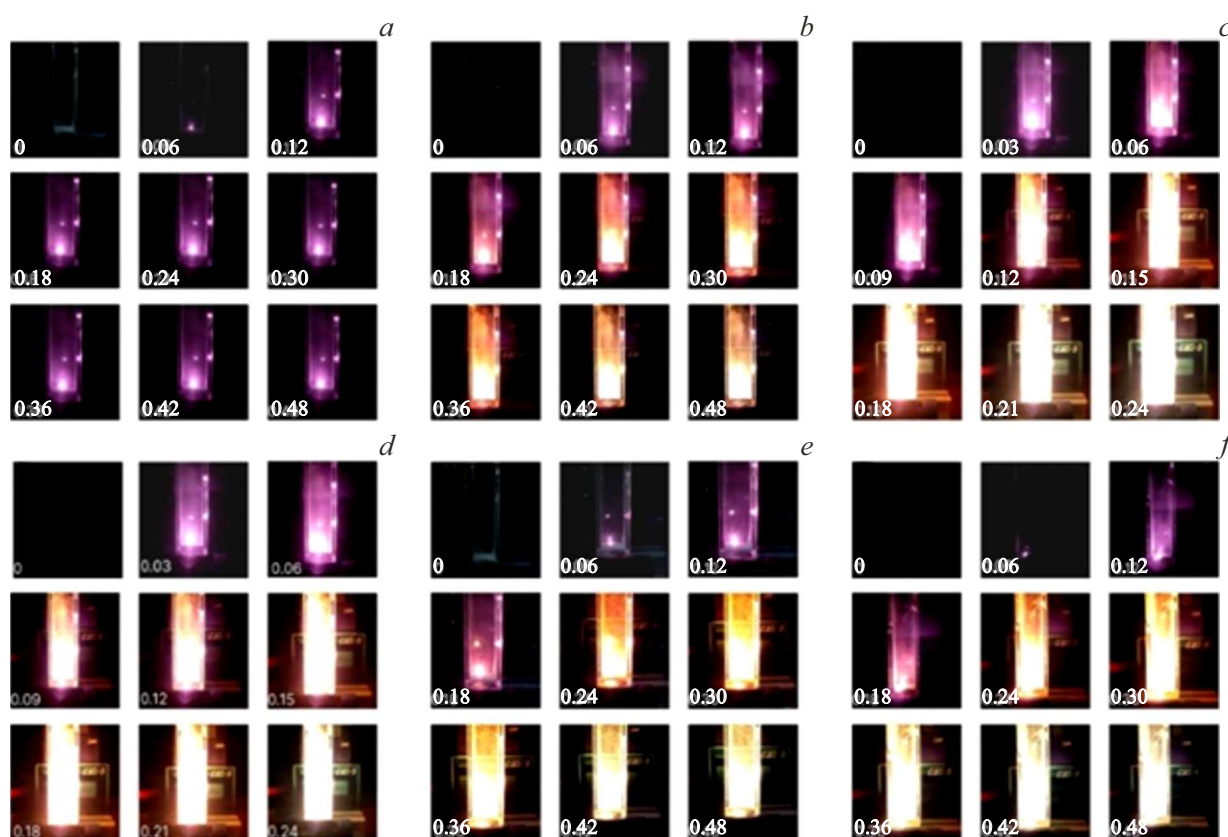


Figure 4. Change over time (indicated in seconds) in the radiation color of the sample of $\text{ZrO}_2 - 30 \text{ mol.\% Yb}_2\text{O}_3$ with an annealing temperature of 500°C when excited by continuous laser radiation with $\lambda = 980 \text{ nm}$, $J = 0.5 \text{ kW/cm}^2$ (a), 1.3 kW/cm^2 (b), 2.1 kW/cm^2 (c) and samples annealed at temperatures of 500°C (d), 800°C (e) and 1150°C (f) when excited by continuous laser radiation with $\lambda = 980 \text{ nm}$, $J = 2.1 \text{ kW/cm}^2$.

was similar in the group without laser exposure at these concentrations ($17.7 \pm 3.7\%$, $25.9 \pm 5.1\%$, $45.7 \pm 4.3\%$, respectively). IC_{50} of this sample in both experimental modes was 2.8 mg/ml (when exposed to laser) and 4 mg/ml (without exposure to cell culture).

Particles of $\text{ZrO}_2 - 30 \text{ mol.\% Yb}_2\text{O}_3$ (800°C , group №3) showed even greater cytotoxicity in contrast to previous samples (fig. 5, c). Exposure to laser radiation did not have an inhibitory effect on the detected intrinsic cytotoxicity of the particles. Significantly high toxicity was recorded at minimal dilutions (concentrations of $50\text{--}25 \text{ mg/ml}$, $p < 0.05$) for cell culture without laser exposure $6.4 \pm 3.7\%$ and $12.9 \pm 6.7\%$, respectively, and for cell culture with exposure to 980 nm exposure $7.8 \pm 4.8\%$ and $14.0 \pm 4.5\%$, respectively. IC_{50} for this composition with laser exposure with $\lambda = 980 \text{ nm}$ and without exposure to cell culture was approximately the same — 4.9 and 5.1 mg/ml , respectively.

A similar pattern was observed in two experimental models according to the results of light microscopy for samples No2 and 3 — a large cell culture death with characteristic features (the cells had a rounded shape without clearly defined organelles).

Thus, the sample of particles of $\text{ZrO}_2 - 30 \text{ mol.\% Yb}_2\text{O}_3$ (1150°C) was chosen for further study of toxicity on

spheroids. It showed inhibition of tumor cell proliferation at concentrations of 50 and 25 mg/ml when exposed to laser radiation with $\lambda = 980 \text{ nm}$, which was not observed in the experiment without laser exposure. A high toxicity of the particles in relation to the cell culture is also observed with the selected exposure mode. IC_{50} for this composition showed a concentration of 14.9 mg/ml when exposed to a laser with $\lambda = 980 \text{ nm}$, IC_{50} was 41 mg/ml without laser exposure.

Next, the cytotoxicity of zirconium dioxide particles stabilized with ytterbium oxide was studied using 3D models of tumor growth. The selected fluorescent dyes show how cells in a state of apoptosis (orange fluorescence) and dead cells (red fluorescence) are distributed in the spheroid structure (Fig. 6). Green fluorescence indicates the presence of nuclei of living cells. In all the studied concentrations exposed to a laser with $\lambda = 980 \text{ nm}$ in the selected exposure mode, apotic cells were observed in the structure of the spheroids in the violet light filter, which were evenly distributed throughout the volume; in the green light filter, a significant number of cells had red fluorescence, which may indicate large-scale cell death (Fig. 6). Significant values were noted at concentrations of 25 and 12.5 mg/ml

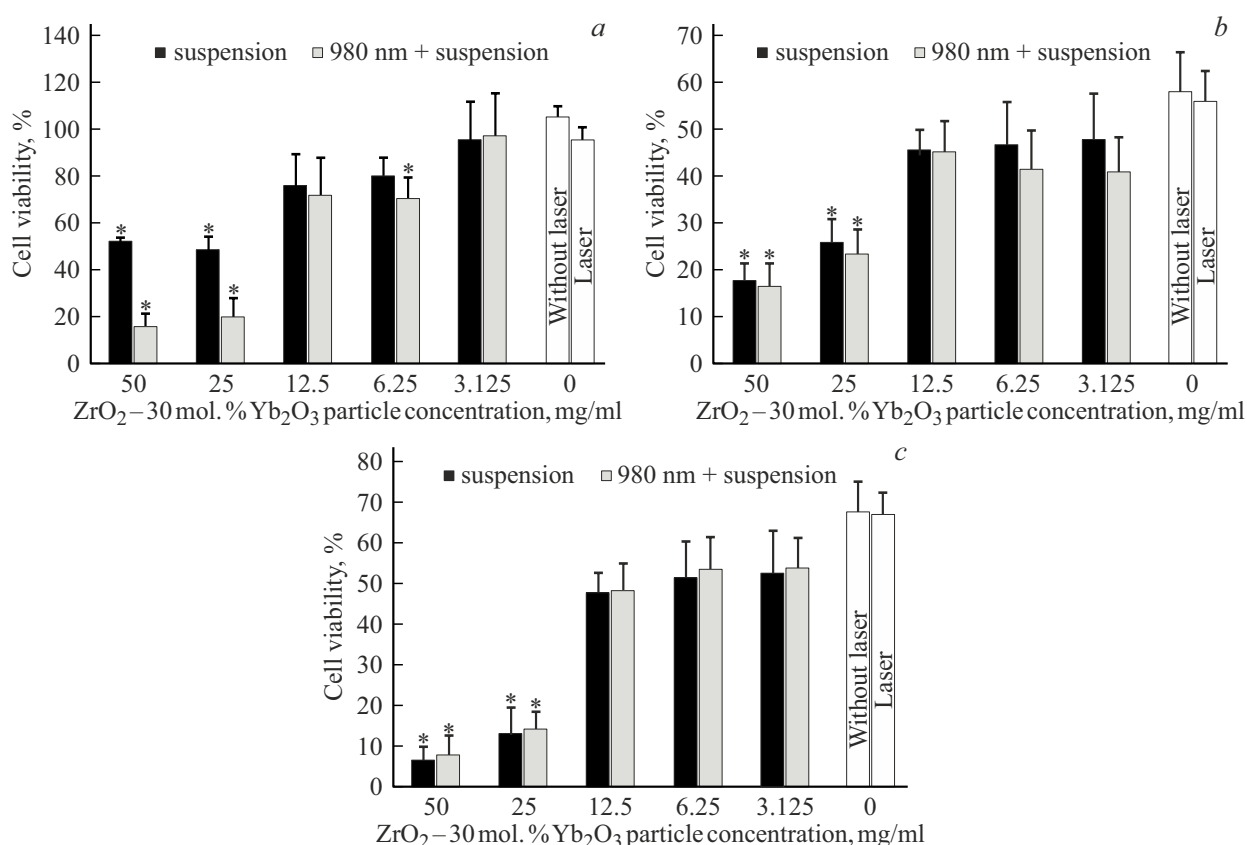


Figure 5. Cytotoxicity of various particle concentrations (a) groups №1 — $\text{ZrO}_2 - 30 \text{ mol.\% Yb}_2\text{O}_3$ (1150°C), (b) groups №2 — $\text{ZrO}_2 - 30 \text{ mol.\% Yb}_2\text{O}_3$ (500°C), (c) groups №3 — $\text{ZrO}_2 - 30 \text{ mol.\% Yb}_2\text{O}_3$ (800°C) in relation to Mh22a cells. The value marked with * is very different from the control group without exposure to radiation.

(Fig. 7), which correlates with IC_{50} for this cell culture according to cytotoxicity studies.

No differences in the studied concentrations with the control group were observed without exposure to laser radiation. Uniformly colored areas with both green and yellow fluorescence were observed in the spherical structure, which may indicate the initial stage of apoptosis. However, the intensity of the red fluorescence does not allow us to draw an unambiguous conclusion about the mass death of cells.

Thus, the studies have shown that $\text{ZrO}_2 - 30 \text{ mol.\% Yb}_2\text{O}_3$ particles annealed at 1150°C can be used for further experiments *in vivo*.

Conclusions

$\text{ZrO}_2 - 30 \text{ mol.\%}$ and Yb_2O_3 particles with various CSR sizes (5, 10, and 99 nm) were synthesized and annealed at temperatures of 500, 800, and 1150°C , respectively. The excitation of these particles by continuous laser radiation with $\lambda = 980 \text{ nm}$ and a power density of $J = 0.9 \text{ kW/cm}^2$ leads to the occurrence of thermal radiation in them. At the same time, the particle size in the studied size range does not significantly affect the characteristics of thermal radiation (contour shape, slope angle tangent of

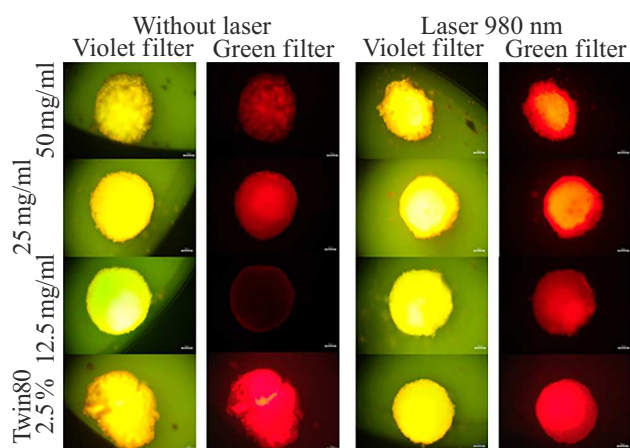


Figure 6. Fluorescence of spheroids of the Mh22a cell line 24 h after introduction of particles of $\text{ZrO}_2 - 30 \text{ mol.\% Yb}_2\text{O}_3$ in the medium (1150°C). Luminescent microscopy at an excitation wavelength of 460–480 nm for acridine orange (AO) and 520–530 nm for ethidium bromide (EB). Green fluorescence — nuclei of viable cells, orange — cell nuclei in a state of apoptosis, red — nuclei of dead cells.

the dependence of the intensity of broadband radiation on the excitation power, ignition time). Cytotoxicity studies of

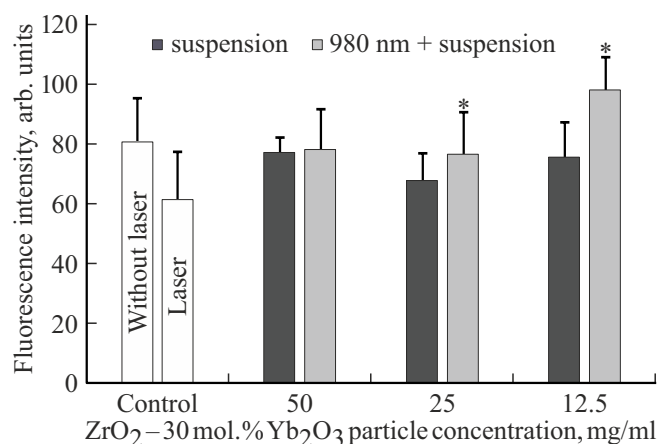


Figure 7. Quantitative assessment of changes in the fluorescence intensity of spheroids exposed to a suspension of particles of ZrO₂ – 30 mol.% Yb₂O₃ (1150 °C).

ZrO₂ – 30 mol.% Yb₂O₃ particles were performed on cell culture. The particles subjected to annealing at 1150 °C have the greatest cytotoxicity at concentrations of 50 and 25 mg/ml when exposed to laser radiation with $\lambda = 980$ nm. Studies of the cytotoxicity of particles on spheroids also showed an increase in their toxic effect under the action of laser radiation with $\lambda = 980$ nm.

Funding

The research was supported by a grant from the Russian Science Foundation № 23-72-01099, <https://rscf.ru/project/23-72-01099/>

Conflict of interest

The authors declare that they have no conflict of interest.

References

- [1] S.M. Redmond, S.C. Rand, S.L. Oliveira. Appl. Phys. Lett., **85**, 5517 (2004). DOI: 10.1063/1.1825068
- [2] S. Tabanlı, H. Cinkay Yılmaz, G. Bilir, M. Erdem, G. Eryurek, B. Di Bartolo, J. Collinse. ECS J. Solid State Sci. Technol., **7**, 3199 (2018). DOI: 10.1149/2.0261801jss
- [3] S.A. Khrushchalina, P.A. Ryabochkina, V.M. Kyashkin, A.S. Vanetsev, O.M. Gaitko, N.Yu. Tabachkova. JETP Lett., **103**, 302 (2016). DOI: 10.1134/S0021364016050064
- [4] P.A. Ryabochkina, S.A. Khrushchalina, A.N. Belyaev, O.S. Bushukina, I.A. Yurlov, S.V. Kostin. Quant. El., **51**, 1038 (2021). DOI: 10.1070/QEL17646
- [5] S.A. Khrushchalina, I.A. Yurlov, P.A. Ryabochkina, V.P. Ageev, O.N. Kulikov, V.I. Shlyapkina, M.N. Tremasov, M.N. Zharkov, A.N. Belyaev, O.S. Bushukina. In: *2022 International Conference Laser Optics (ICLO) Proceedings* (Institute of Electrical and Electronics Engineers Inc., 2022), p. 1. DOI: 10.1109/ICLO54117.2022.9840157
- [6] P.A. Ryabochkina, S.A. Khrushchalina, O.N. Kulikov, V.I. Shlyapkina, V.P. Ageev, N.Yu. Tabachkova, V.O. Veselova, T.V. Volkova. Bull. Lebedev Phys. Inst., **51**, S581 (2024). DOI: 0.3103/S1068335624601730
- [7] G. Brost, P. Horn, A. Abtahi. Appl. Opt., **24**, 38 (1985). DOI: 10.1364/ao.24.000038
- [8] A.N. Magunov. Instrum. Exp. Tech., **52**, 451 (2009). DOI: 10.1134/S0020441209040010
- [9] Y. Mazur, G. Lavie. Patent US № 6229048, 2001.
- [10] A. Walzl, C. Unger, N. Kramer, D. Unterleuthner, M. Scherzer, M. Hengstschläger, D. Schwanzer-Pfeiffer, H. Dolznig. J. Biomol. Screen, **19**, 1047 (2014). DOI: 10.1177/1087057114532352
- [11] N. Ishizawa, Y. Matsushima, M. Hayashi, M. Ueki. Acta Crystallogr. B, **55**, 726 (1999). DOI: 10.1107/S0108768199005108
- [12] V.O. Veselova, A.V. Egorysheva, I.A. Yurlov, P.A. Ryabochkina, O.V. Belova, T.D. Dudkina. Russ. J. Inorg. Chem., **65**, 1298 (2020). DOI: 10.1134/S0036023620090211

Translated by A.Akhtyamov