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## TEMPERATURE DEPENDENCE OF PHYSICAL AND CHEMICAL PROPERTIES OF ZINC OXIDE DISCOVERED BY RAMAN SPECTROSCOPY

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**Abstract.** In this work, the phase, optical and structural properties of a ZnO powder sample were studied depending on changes in the annealing temperature. For this, the following main methods were used such as Scanning Electron Microscopy, Raman Spectroscopy, UV-Visible Spectroscopy and Powder Diffractometry-XRD method respectively. The degrees of amorphism and crystallinity were determined using Search Match!3 software. Using Raman spectroscopy, Raman spectroscopy revealed significant changes in the shape, orientation and distribution of peaks of ZnO nanopowders formed with increasing annealing (heating) temperature, as well as to explain the phonon vibration mode. The resulting high Raman intensity confirmed the high-frequency phonon mode of hexagonal wurtzite ZnO. Scanning electron microscopy was used to obtain structural images of the sample.

**Keywords:** Raman spectroscopy, heterostructures, phonon vibration mode, Raman scattering of light, longitudinal optical - transverse optical frequencies.

**1.Introduction.** The physical and chemical properties of zinc oxide (ZnO) make it a viable and extremely attractive compound to use in various nanotechnology applications [1-3]. Some of these applications include biomedicine, energy, sensors and optics. As research into ZnO nanostructures continues to grow, it has inspired a number of new innovative applications [4-5]. In addition to its unique chemical properties, it also has simple crystal growth technology and offers significantly lower production costs compared to other semiconductors used in nanotechnology. High-tech devices use new structures created using the latest technologies and modern methods [6-10]. Several processes have been developed to synthesize high-quality ZnO nanostructures, especially in the case of nanowires.

Zinc oxide (ZnO) is a multifunctional group II–VI material due to its wide band gap ( $E_g \sim 3.37$  eV) and straight band and with a large exciton binding energy of 60 MeV at room temperature. It also

has the advantages of excellent resistance to radiation damage, suitability for wet etching process, physical and chemical stability, high oxidizing ability, low cost and availability [11-14]. In recent decades, ZnO has become a very promising material used in many fields, such as solar cells, transparent conductive contacts, light-emitting devices, spintronics devices, laser deflectors, paints, antibacterial agents, biosensors, piezoelectric transducers and gas sensors [15-16]. Among these devices and components, many efforts have been made to modify the optical and/or electrical properties of ZnO through doping, ion irradiation, etc. So that it can be more widely used in undoped research areas since raw ZnO is generally inactive in carrier transfer current and is ineffective for the adsorption of solar energy. For example, when ZnO material is applied as a photocatalyst to promote the degradation of organic pollutants, less than 5% of the solar spectrum at the Earth's surface consisting of UV wavelengths can drive the photocatalytic process because the absorption edge is limited by the natural band gap of ZnO. Modification of the band gap of ZnO by metal doping is one of the promising approaches to extend light absorption to the visible wavelength region of light. In addition, these metallic impurities have also demonstrated electron scavenger properties to increase the lifetime of photo-generated electron-hole pairs [17–19].

On the other hand, when ZnO is used for the fabrication of optoelectronic devices, one of the main obstacles for the implementation of optoelectronics based on ZnO devices is the difficulty in achieving high-quality *p*-type ZnO due to the self-compensation effect that occurs due to inherent defects, as well as limited solubility and inactivation of acceptor impurities in the ZnO material. Taking into account the trend towards the creation of nanometer-sized semiconductor devices, the relevance of using optical methods for diagnosing such structures, in particular, the Raman scattering method (RSM), is increasing. In this regard, we identified these phase states for a powder sample at various annealing temperatures from 200°C to 700°C.

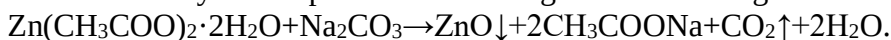
The results of this work, as well as our previous works [27-28], were obtained due to the fact that The Raman spectroscopy method allows and ensures the implementation of qualitative and quantitative analysis of the surface and volume of the sample under study, analysis of the structures of film coatings, registration of defects and stresses in the analyzed structures, control of the phase state of the structures under study, including identification of the amorphous and crystalline phases.

**2.Materials and methods.** In the present work, an attempt was made to prevent undesirable effects by temperature annealing a ZnO powder sample irradiated at three different temperatures. For this purpose, the following main methods were used, Raman Spectroscopy (Raman) and Powder Diffractometry-XRD, respectively. The degrees of amorphism and crystallinity were determined using Search Match!3 software. Raman spectroscopy and Raman spectroscopy revealed significant changes in the shape, orientation and distribution of peaks of ZnO nanopowders formed with increasing annealing (heating) temperature. The presence of intense Raman scattering of light in ZnO powder samples indicates the existence of a longitudinal optical phonon mode. Small shifts were detected in all ZnO modes, which indicate the presence of stress and strain in the crystal lattice. Changes in particle size were studied using confocal Raman microscopy during temperature annealing. It was shown that changes in the structure and particle size of the material improved its characteristics. It has been confirmed that nanocomposite heterostructures obtained by a simple chemical method are applicable for the creation of optoelectronic devices.

The use of the Raman scattering method, due to the patterns of changes in Raman spectra [20-22], provides a wide range of parameters of the object of study. Raman spectroscopy, or Raman spectroscopy, is applicable to determine the characteristics of research objects as a rapid non-destructive method of analysis, which allows one to estimate the size of nanocrystals from the shape and position of Raman scattering peaks, estimate the ratio of amorphous and crystalline phases, analyze the composition of crystals and evaluate deformations and stresses in crystals [23]. We studied these samples using Raman spectroscopy on an InVia Raman Spectrometer manufactured by Renishaw, UK, when excited by the lines of an RL785 Class 3B Laser laser, with a radiation wavelength of 785 nm. During the measurements, a diffraction grating with a period of 1200 lines/mm was used, and a standard Renishaw CCD Camera detector was used as a recording device [24-26].

JSM-IT210 scanning electron microscopy (SEM) was used to study the morphology of ZnO samples. The composition was analyzed using energy dispersive X-ray (EDX) spectroscopy. The concentrations of chemical elements in the composites were measured using electron probe X-ray spectral microanalyzers for energy dispersive X-ray analysis (EDXA) as part of an SEM.

**3.Method of obtaining a sample.** Zinc oxide nanoparticles were obtained by precipitation from solution. The synthesis proceeds according to the following scheme:



A 0.1 M solution of Zn (CH<sub>3</sub>COO)<sub>2</sub> was added to a 5% solution of polyvinyl alcohol on a magnetic stirrer 30 minutes, then added 0.1 M Na<sub>2</sub>CO<sub>3</sub> solution, continued stirring for 15 minutes at room temperature. The resulting precipitate was centrifuged at 3500 rpm and washed with deionized water. They were dried at room temperature.

**4.Results and discussion.** In this work, the morphological, optical, electrical and structural properties as a function of annealing temperature variation from 200°C to 700°C were investigated using Raman spectroscopy, UV-visible spectroscopy, and powder diffractometry-XRD, respectively. UV-visible spectroscopy similarly showed that ZnO samples irradiated at different temperatures had band gaps ranging from 3.46 to 3.35 eV. Film resistivity values obtained from current-voltage characteristics showed an exponential increase in resistivity with increasing annealing temperature of the ZnO sample.

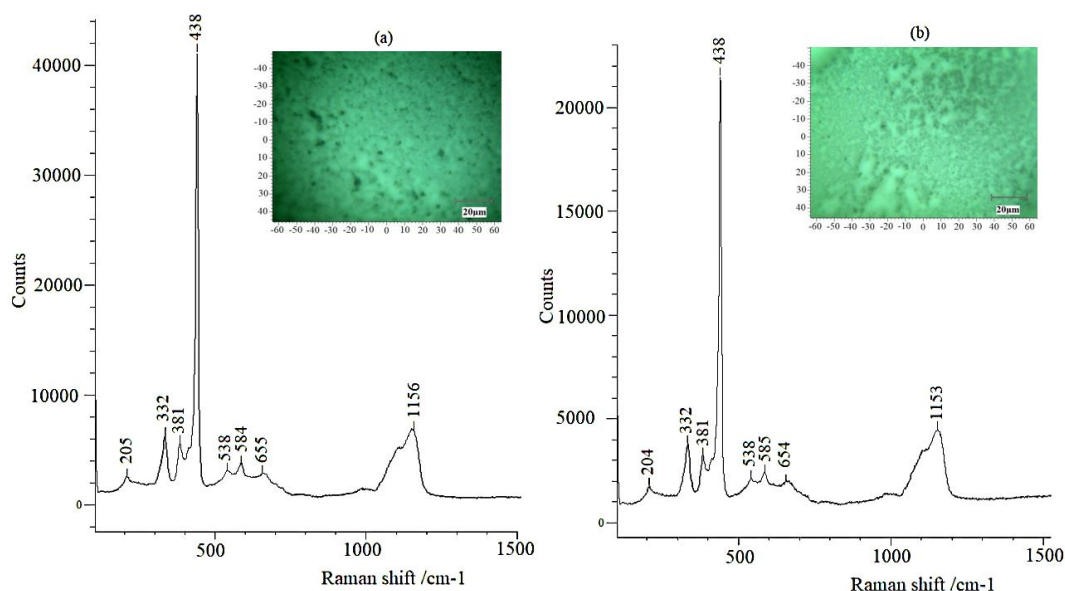


**Fig. 1.** Raman spectrum of a ZnO sample, as well as an image of a portion of the surfaces of the studied ZnO sample at different annealing temperatures. a) Initial ZnO sample. b) ZnO at annealing temperature  $T=200^{\circ}\text{C}$

In figures 1-2. a), b), c), d) the Raman spectra of nanostructures are presented, as well as an image of a portion of the surfaces of the ZnO sample under study. As can be seen from the given data for the initial sample, the intensity of the peak is 438 cm<sup>-1</sup> 42000, subsequent annealing at a temperature of 200°C leads to a decrease in the intensity of this peak to 24000, and at a temperature of 500°C 20000, this suggests that with increasing temperature up to 500°C the intensity decreases. At an annealing temperature of 700°C, the intensity of this peak sharply increases to 56000.

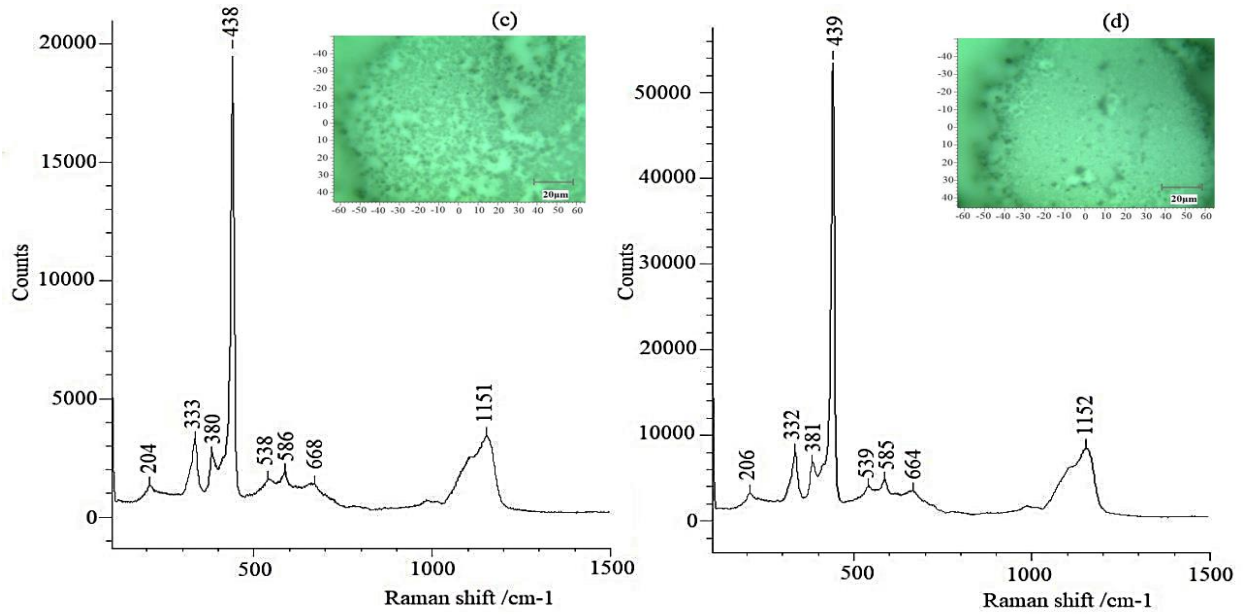


**Fig. 2.** Raman spectrum of a ZnO sample as well as an image of a section of the surfaces of the studied ZnO sample at different annealing temperatures c)  $T=500^{\circ}\text{C}$ , d)  $T=700^{\circ}\text{C}$

This nature of the growth of this peak suggests that annealing will first lead to the decomposition of the nanolayers, and further annealing will again lead to ordering of the sample structure. The same picture is observed for other peaks of this ZnO material. To make it easier to understand, we have divided the spectra into two parts, from 200 to  $510\text{ cm}^{-1}$  and from 530 to  $1300\text{ cm}^{-1}$ , as shown in figures 1-2. ZnO peaks at 331, 383 and  $436\text{ cm}^{-1}$  were clearly observed. Several peaks at 584, 660, 1101 and  $1154\text{ cm}^{-1}$ , respectively, were also detected in the high wavenumber region.

Peaks at  $352$  and  $654\text{ cm}^{-1}$  -  $A_1$  symmetry mode, the peak at  $438\text{ cm}^{-1}$  is attributed to non-polar optical phonons of ZnO with a high  $E_2$  mode, and the peak at  $381\text{ cm}^{-1}$  corresponds to  $A_1$  symmetry to the frequencies of transverse optical (TO) background modes. Peaks at 585 and  $1154\text{ cm}^{-1}$  -  $E_1$  symmetry corresponds to the longitudinal optical modes LO and 2LO, respectively. In addition, the intensities of all observed vibrational modes decrease with increasing annealing temperature to  $500^{\circ}\text{C}$ , and then increase again at an annealing temperature of  $700^{\circ}\text{C}$ . As a result, the size of nanostructures decreases. In addition, their line shapes become asymmetrical.

**5. Electron-phonon connection.** It is well known that as particle size decreases to the nanoscale or lattice distortion increases, the selection rule  $k=0$  for first-order Raman scattering relaxes and phonon scattering will not be limited to the center of the Brillouin zone; therefore, phonon dispersion near the center of the Brillouin zone should also be taken into account. As a result, regimes will be observed, not only shifts and broadening of the optical phonon of the first order scattering mode, but also forbidden symmetry. In this paper, we provide information on electron-phonon coupling from Raman scattering (RS), which has previously been rarely reported for ZnO nanopowders.

The relationship between the second- and first-order scattering cross sections turned out to be very sensitive to the electron-phonon coupling force [23, 24]. For bulk ZnO materials, the intensity ratio  $E_1$  (2LO) and  $E_1$  (LO) was calculated to be 2.85 [25]. This can be clearly observed for the Raman modes  $E_1$  (LO) and  $E_1$  (2LO), respectively. Consequently, as follows from the above, phonon scattering will be limited by the center of the Brillouin zone, the ratio between the second- and first-order Raman scattering cross sections increases. In the wide hump in the range of  $560\text{--}610\text{ cm}^{-1}$ , lattice vibrations in

$E_1(\text{LO})$  mode in pristine ZnO with peaks at  $574\text{ cm}^{-1}$ .  $E_1(\text{LO})$  mode refers to the characteristic of random orientation of ZnO crystallites [27]. This is due to internal defects such as oxygen

vacancies, interstitial Zn and resonance effect in the ZnO lattice crystal at the excitation wavelength [28]. This indicates the presence of a compressive stress effect on the crystal lattice of heterostructural materials.  $E_1$  and  $A_1$  are polar Raman and infrared active because they have LO and TO Raman frequencies. Other ZnO peaks detected at 333, 382, 417–424 and 574  $\text{cm}^{-1}$  are assigned to  $E_2$   $H-E_2$  modes  $L$ ,  $A_1$  (TO),  $E_1$  (TO) and  $E_1$  (LO). These modes exhibit Raman frequency shifts. Moreover, the LO phonon peaks in zinc blende ZnO indicate the existence of a form of heterostructures.

Fig. 3 shows typical examples of SEM images of the surface of ZnO nanopowders. Zinc oxide nanoparticles were obtained by precipitation from sample solutions. From figure 3 it is clear that after temperature annealing, the grain sizes in the ZnO sample vary from a few to several tens of micrometers (figure 3, a, b, c). As mentioned above, the composition was analyzed using energy dispersive X-ray (EDX) spectroscopy.

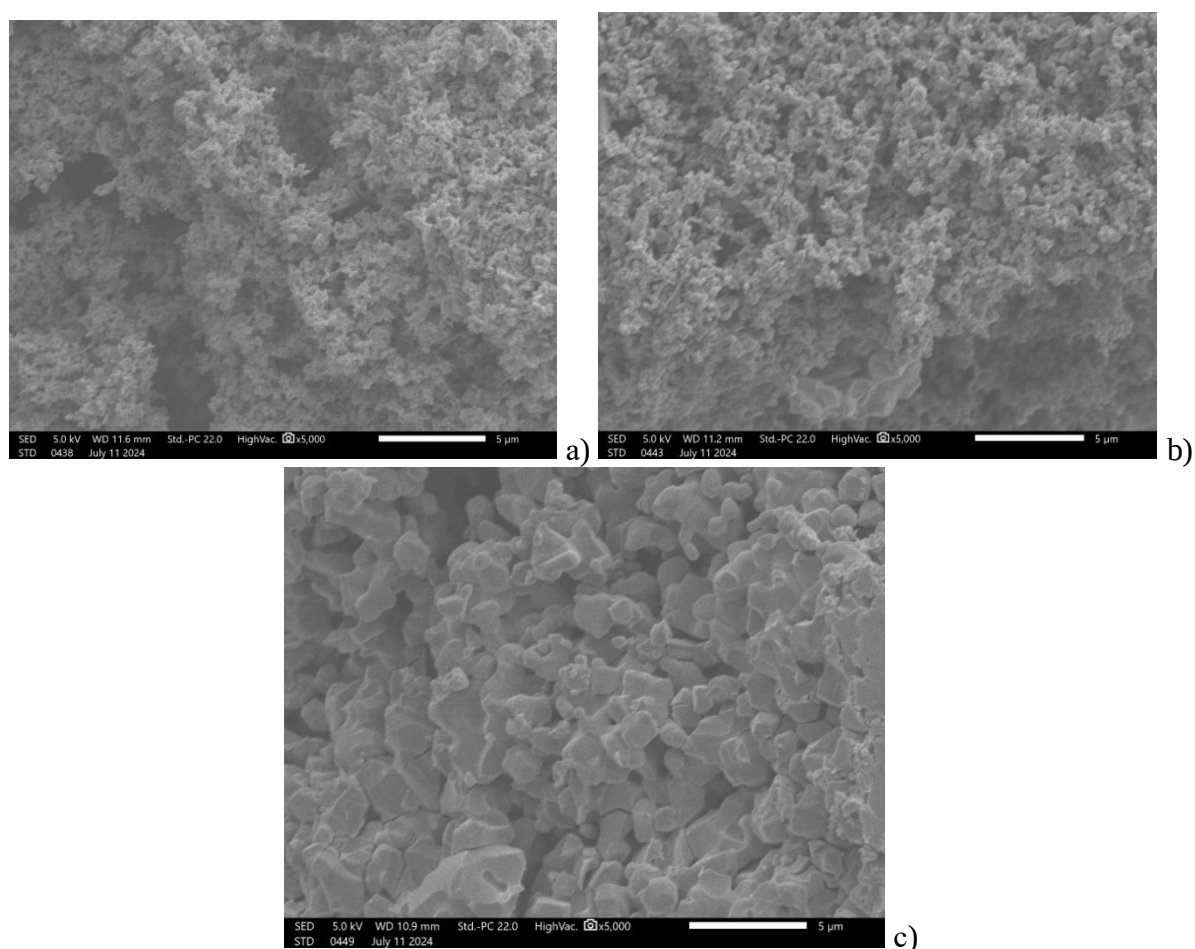


**Fig. 3.** SEM images of a) the original, b) 700<sup>0</sup>C, c) 1100<sup>0</sup>C surfaces of the ZnO sample under study at different annealing temperatures obtained by scanning electron microscope of the ZnO nanostructure

The atomic ratios of Zn and O in the nanopowders were measured using EDX, and the main results are summarized in figure 3. It is clear that there is an oxygen deficiency for all samples. However, the compositions are approximately maintained within stoichiometry, taking into account the measurement error of the light element oxygen. In addition, we also discovered figure 4(a) the presence of carbon and aluminum in the composition of zinc oxide nanopowder. Despite this, as shown by the results of SEM-EDS analysis, the sample prepared in the above way has pure ZnO phases. X-ray analysis showed that the ZnO samples have a hexagonal ZnO wurtzite structure with peaks at  $2\theta$ -positions 31.7°, 34.4°, 36.2°, 47.5°, 56.6° and 62.8° belonging to planes (100), (002), (101), (012),

(110) and (013), respectively, in figure 4 (b). We also determined the Miller indices and lattice parameters. Phase quantitative and qualitative analyzes of ZnO nanopowders were also carried out.

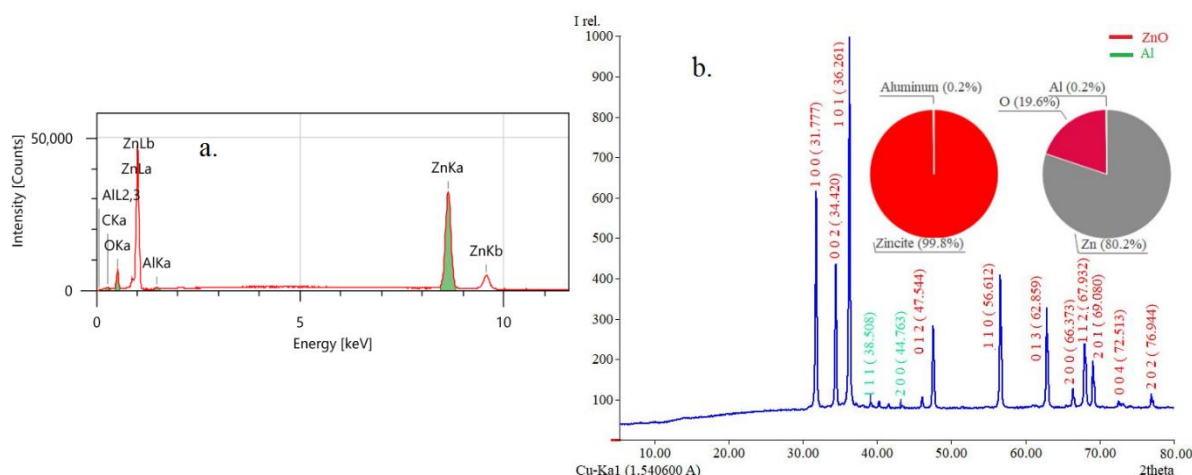


**Figure 4.** The energy dispersive spectra of the samples obtained from SEM-EDS analysis clearly show that the sample prepared in the above manner has pure ZnO phases (a), X-ray phase analysis of a ZnO sample, Miller indices and lattice parameters (b)

The experimental data obtained are in good agreement with data obtained by other methods. The degree of crystallinity and amorphism of ZnO samples was assessed by X-ray diffraction analysis using the Search and Match!3 program. For zinc oxide, it was found that the amorphous phase is - 86.27% and the crystalline phase is 13.73%, respectively. It was found that with increasing annealing temperature, the crystallinity of the ZnO sample increases slightly.

**6.Conclusion.** Thus, the Raman spectra of ZnO nanopowders showed that the strength of electron-phonon interaction increases with increasing size of nanocrystals when they are heated to 700°C. The strong  $E_2 H$  peak (high-frequency phonons) at  $438\text{ cm}^{-1}$  demonstrates the imprint of the hexagonal structure of wurtzite ZnO with good crystallinity. The vibration of the Zn heavy sublattice and the vibration of oxygen are associated with this nonpolar mode. It has relatively higher intensity compared to others, the Raman peak of pristine ZnO is narrow and sharp. Changes in particle size were also detected using confocal Raman microscopy during thermal annealing. It was shown that changes in the structure and particle size of the material improved its characteristics. Oxygen deficiency was also detected for all samples, and the compositions were maintained within stoichiometry, taking into account the measurement error of the light element oxygen. In addition, we determined the presence of carbon and aluminum in the composition of zinc oxide nanopowder.

**Competing interests.** No competing of interests.

**Authors Contributions.** **Kholikov U.Kh.:** Conceptualization, Investigation, Methodology, Data curation. **Khozhiev Sh.T.:** Validation, Visualization, Writing - original draft. **Kosimov I.O.:** Formal analysis, Writing - review & editing. **Khudaykulov I.Kh.:** Formal analysis, Writing - original draft. **Berdiyev U.F.:** Writing - review & editing, Resources, Supervision. **Bekpulatov I.R.:** Writing - original draft, Writing - review & editing, Resources, Supervision. **Farmonov N.P.:** Formal analysis, Writing - review & editing. **Gunel Imanova:** Formal analysis, Writing - original draft, Writing - review & editing, Supervision.

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