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STUDY OF ELECTROPHYSICAL AND OPTICAL PROPERTIES OF NATURAL CELLULOSE FIBERS DOPED WITH IODINE AND $KMnO_4$

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Abstract. In this article, the physical properties of cotton fibers (CF) sort of "Komolot-79" and "Xorazm-150" treated with radioactive, chemical and thermal treatment were studied. The main results of the research are as follows. The electrophysical, photoelectric and optical properties of cotton fibers irradiated with γ-^{60}Co radiation source at different doses, mercilized and doped with iodine and $KMnO_4$ were studied. The research was carried out in the temperature range of 300-360 K and voltage in the range of 0-100 V. The structural changes in the samples were studied using the IR spectroscopy method. Temperature dependence of electrical conductivity of CF was studied and the electrical conductivity increased exponentially with the activation energy E_{t1}=0.54 eV in the $KMnO_4$-doped sample and E_{t2}=1.41 eV in the iodine-doped sample. The photoluminescence spectrum of cotton fiber doped with iodine was studied. According to the results, photoluminescence maxima were observed in the visible light range (394, 487 and 521 nm) when the samples were illuminated with ultraviolet light (340 nm). It was proved that the current flowing from all samples prepared as a research object obeys Ohm's law. It was found that the photoconductivity caused by ultraviolet and visible light in the CF sample of "Komolot-79" is mainly related to the formation of inter-zone electron-hole pairs. In the sample doped with iodine, it was found that the electrical conductivity increased significantly due to the photogeneration of charge carriers.

Keywords: *Photoluminescence, cotton fiber, photoconductivity, mercilization, electrical conductivity, I-V characteristic.*

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1. Introduction

The interest in the use of cotton fibers (CF) as a polymer material is constantly increasing, mainly due to the many advantages associated with low cost and renewable material (Alessio Becheri *et al.*, 2008). Also, for the past 30 years, extensive research has been conducted to understand the electrical conductivity of natural fibers, the nature of charge transport in these materials (Gunes *et al.*, 2007; Abd El-Kader *et al.*, 2004). As an electrical conductor, polymer-coated textiles are part of a family of composite materials that can be used in many fields. The development of conductive yarns is essential for various applications in wearable electronics such as smart clothing actuators, information management devices, and biomedical sensors (Xuqing *et al.*,

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2010; Shirley *et al.*, 2007). Also, the increasing demand for semiconductor materials requires the discovery of new semiconductor materials and conducting fundamental research on them. After it was determined that CF treated chemically and thermally with various elements has semiconducting properties, research is being conducted on them (Askerov *et al.*, 2018; Oksegendler *et al.*, 2002; Ilanchezhiyan *et al.*, 2015; Giorgio *et al.*, 2011; Akobirova *et al.*, 2008) It has been determined that the electrophysical properties of CF are mainly manifested in its cuticle layer (Ilanchezhiyan *et al.*, 2015). Due to the fact that the cuticle layer has a different structure in different varieties of CF, it is explained that its electrophysical properties change. Cellulose combines multiple functionalities, optical transparency and processability. The mechanism of interaction of aqueous NaOH solution with cellulose is of interest to researchers because it has many applications in fiber modification, dissolution and regeneration by chemical treatment of cellulose with NaOH solution (Ying & Yulin, 2009). Studies have shown that individual natural organic polymer fibers, cellulose-based fabrics and papers can be successfully coated with conductive polymers using several coating methods (Zakirov *et al.,* 2011).

The mechanism of electrical conductivity in polymers is mainly characterized by such parameters as the density of charge carriers and the mobility of charge carriers. Doping has a very good effect on the conductivity properties of polymers (Gill, 1972). Iodine has been found to increase the electrical conductivity of a number of organic materials formed by hydrocarbon-iodine complexes (Kaiser and Park, 2005). Chemical, photochemical, or electrochemical doping is used to introduce external charge carriers into organic semiconductors (Manjunath *et al*., 2016). Depending on the effect of the macromolecular structure, additives reduce the resistance of polymers to different degrees (Aleshin, 2007). Optical properties of CF have been studied by several researchers. According to the result, it was determined that the bandgap energy is approximately E_g=3.36 eV through the conduction spectrum (Zakirov *et al.*, 2011).

In this work, we present the study of the electrical and optical properties of natural cotton fibers doped with iodine and $KMnO_4$ as a function of changes in the molecular structures of CF during radioactive and chemical treatment. It was found that the photoluminescence (PL) intensity and electrical conductivity can be increased compared to the original material by properly controlling the CF morphology as a result of radioactive and alkaline treatment.

The physical, chemical, mechanical and electrical properties of semiconductors and other materials change under the influence of high-energy rays, such as ionizing radiation, X-rays, gamma rays, beta particles and alpha particles. These changes are essential in adapting the properties of materials using a high-energy radiation flux to create new materials and, thus, improve the characteristics of various devices (Rashmi *et al*., 2023; Jasim & Abid, 2012)

2. Experimental part

Ripe cotton fibers were laid in a parallel direction on a straight body and combed in the direction of the fiber using a special comb. The approximate number of parallel fibers is 4500-5200 and the length is 4 mm. CF was washed with 80^0 C distilled water for 10 min. The samples were dried at 60% relative humidity and room temperature for 24 h. Radioactive, chemical and thermal treatment was carried out in accordance with the research task. We will briefly consider these processes below. Chemical treatment of CF with NaOH (mersilization process). Washed samples were kept in a bath with 20%

NaOH solution in water at a temperature of 18^0C for 2 min. After removing the samples from the bath, the excess NaOH that did not form a chemical bond was washed with distilled water, and the samples were dried at 60% air humidity and room temperature (Francis *et al.*, 1978). This processes increases the stability of the cellulose fiber to external influences and the permeability of the input.

The samples were irradiated with γ-^{60}Co-irradiation source at a dose of 65.73 ren/c for different times, from 5 to 120 hours. For doping CF with iodine, the samples were soaked for 20 min in a bath with a 5% solution of iodine in alcohol at room temperature. In order to diffuse the iodine coated on the CF surface into the fiber, thermal treatment was carried out in an air chamber with a temperature of 80^0 C for a period of 1 to 8 hours in accordance with the research task (1.5% solution in water was taken for doping with $KMnO_4$). Conductive adhesive made from a mixture of graphite and liquid glass was used in laboratory conditions to make ohmic contact with the samples.

The IR spectroscopy method was used to determine changes in the structure of radioactive, chemically and thermally treated CF samples. Fourier transform IR spectroscopic analysis of reagents and β–SDoFDA product was carried out to study the IR changes of functional groups in the Bruker Invenio S-2021 Fourier transform spectrometer in the range of 400-4000 cm^{-1}. Visualization of FT-IR signals was performed in OPUS software.

CF I-V characteristic was measured at temperature T=300 K. Researches were carried out in the dark and under UV light (λ=254 nm). To obtain high-quality results, a digital 2461-SourceMeter was used in the range of small current values, i.e. in nanoamperes. I-V characteristic measurement error is about 2-3%. To determine the temperature dependence of the electrical conductivity of the samples, a thermostat designed to maintain the temperature from +10 to +1250 C in any range was used to control the heater. The accuracy of the device is about 0.20 C. A Cu+Ct thermocouple was used to record the temperature.

The ИКМ-1 monochromator equipped with a LiF (light spectrum transmission range from 0.12 to 8.5 μm) prism was used to study the photoconductivity (PC) spectrum of the samples. Tungsten spiral lamp was used as a light source (quartz lamp: light spectrum transmission range from 0.16 to 4.35 μm). A LiF collecting lens was installed at the output of the monochromator to direct the monochromatic light separated into the spectrum to the sample. The sample was placed at the focus of the lens and uniformly illuminated over the entire surface of the sample. The DC voltage applied to the sample was 0-100 V. Since the samples are mainly of high resistance, measurements were made with an electrometer of type У1-2 to measure PC. A mercury lamp with spectrum energy hυ=5 eV was used to study PC kinetics. The "Cary Eclipse Fluorescence Spectrometer" device manufactured on the basis of Agilent technology was used to study the photoluminescence spectrum of the samples. The spectrometer is a sensitive, precise and flexible device designed to study the luminescence spectrum of solids in the wavelength range from 190 to 1100 nm. The photoexcitation source is a xenon lamp (80 Hz). The xenon lamp is capable of acquiring one data point every 12.5 ms and scanning at 24,000 nm/min without high drift. The wavelength accuracy is ±1.5 nm (Brian, 2020).

3. Result and discussion

In order to get a general understanding of the samples prepared for the study, the changes in the molecular structure of the chemically, radioactively and thermally treated samples were studied using the IR spectroscopy method (Fig. 1). Sample S1 is “Komolot-79” sort CF irradiated for 5 hours with γ-^{60}Co radiation source, Sample S2 "Komolot-79" sort CF doped with iodine, Sample S3 pure "Komolot-79" sort CF, S4 sample mercerized “Komolot-79” sort CF, Sample S5 is "Komolot-79" sort CF irradiated for 120 hours with γ-^{60}Co-irradiation source, Sample S6 is pure "Xorazm-150" sort CF, sample S7 is "Xorazm-150" sort CF doped with $KMnO_4$.

In Fig. 1, the change in absorption lines compared to pure cotton fiber was analyzed. In sample S3, the valence vibration absorption lines corresponding to the OH-group in the cellulose molecule are 3329.4 cm^{-1} and 3289.4 cm^{-1}, The valence vibration lines of the CH_2 group are 2910.9 cm^{-1} and 2857.2 cm^{-1}, The valence vibration absorption lines of the C=0 group are 1607.8 cm^{-1}, Absorption lines of asymmetric deformation vibration of the CH_2-group are 1426.6 cm^{-1}, It was observed that there are absorption lines in the region of 1201.6 cm^{-1} of the valence vibration of the C–OH group.

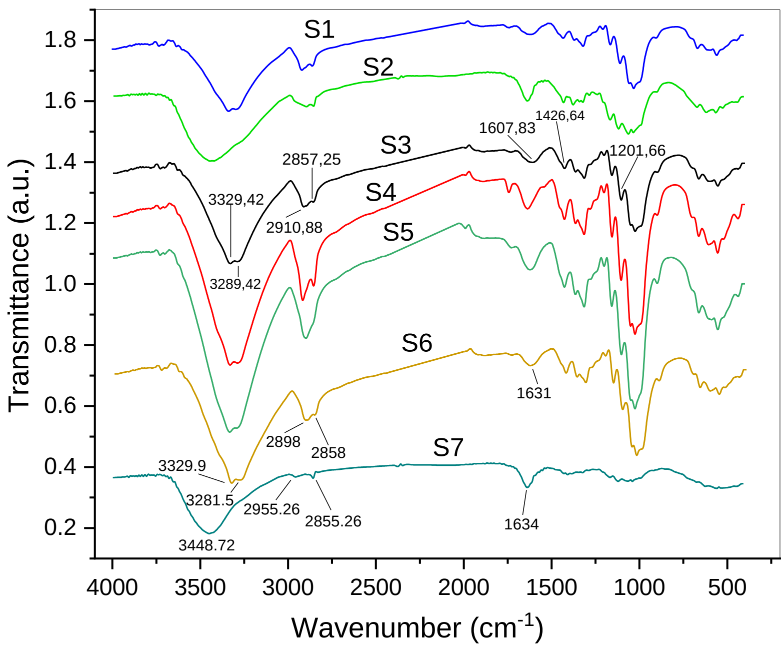

Fig 1. Radioactive chemical and thermally treated CF IR spectroscopy results

Below are the results of radioactive, chemical, and thermally treated CF IR spectroscopy, relative changes to the results obtained on pure cotton fiber. When analyzing the S1 sample irradiated for 5 hours in the γ-^{60}Co radiation source, almost no change in absorption lines was observed compared to the pure sample (S1 and S3 samples). However, the following changes were observed in sample S5 irradiated for 120 hours in γ-^{60}Co radiation source. It was found that the absorption lines were shifted from 3329.4 cm^{-1} to 3332.9 cm^{-1} at high intensity. It was observed that the absorption lines of the CH_2- group from cellulose at 2910.8 cm^{-1} and 2857.2 cm^{-1} shifted to 2898.5 cm^{-1}. The absorption lines of the C=0 group in cellulose shifted from 1607.8 cm^{-1} to

1621.87 cm^{-1}. But in samples S1 and S5, there was no change in the fingerprint area at all. According to the conclusion, when CF was exposed to radioactive light, molecular deformation occurred, that is, point defects were formed in the molecular lattice (Frenkel pairs, i.e., vacancies and interstitial atoms). In order to reduce the H mobility of the OH group on carbon atoms 2 and 3 in the mercilized S4 sample, when treated with NaOH, H is replaced by Na, and absorption lines were observed in the area of 439.56 cm^{-1}. In sample S2 doped with iodine, the valence vibration of the OH group is shifted from 3329.4 cm^{-1} and 3289.4 cm^{-1} to 3446.5 cm^{-1}, The valence vibration of the CH_2 group was shifted from 2910.9 cm^{-1} and 2857.2 cm^{-1} to 2896 cm^{-1} and 2855.57 cm^{-1}, The valence vibration of the C=0 group was shifted from 1607.8 cm^{-1} to 1635.8 cm^{-1} and a small change was observed in the fingerprint area. According to the conclusion, iodine molecules are located in point defects in CF and are connected by valence, electrostatic and Van-Der-Waals bonds. When the pure S6 sample is compared to the doped S7 sample, when doped with $KMnO_4$, the valence vibration of the OH group is shifted to 3448.72 cm^{-1}, the absorption maxima of the CH_2 group are shifted to the area of 2955 cm^{-1} and 2855 cm^{-1}, It was observed that the absorption lines belonging to the C-O carbonyl group were shifted from 1631 cm^{-1} to 1634 cm^{-1}, and the intensity in the fingerprint area disappeared and went to a semi-solid state. In the summary, when "Xorazm-150" cotton fiber is doped with $KMnO_4$, it is explained that $KMnO_4$ molecules are mainly located in the defects between cellulose molecules (Prech *et al*., 2006).

When natural polymer materials are processed, their structure and morphology change to a certain extent. In turn, this polymer changes the physical properties of materials. At the next stage, the research results of electrophysical properties of chemically, radioactively and thermally treated CF are presented.

According to researches, high-energy particles and γ-quanta light effects are a very effective tool for influencing the electrophysical and optical properties of semiconductors. The study of lattice defects formed in nuclear conductors under the influence of radioactive radiation and the determination of the relationship between the defects formed in them are of great scientific interest and are part of the current problems of modern solid state physics. The advantages of radioactive beam treatment of semiconductors provide the ability to control the dose with high accuracy and to create certain types of defects in the materials in strictly defined concentrations. Simple defects (Frenkel pairs, spatially separated vacancies, and interstitial atoms) are formed when the energy exerted by radioactive bombardment particles on a lattice atom exceeds the energy required to displace the atom from the lattice site. These areas are called defect clusters (Watkins, 1968; Gaidar, 2011).

Fig. 2 shows the I-V characteristic of "Xorazm-150" sort CF doped with $KMnO_4$ at a constant temperature of 80^0C and at different times. It is possible to consider the obtained result as a production dependence of doping wattage on electrical conductivity. Fig. 2 in line 1, a very small amount of current of 0.2 nA at 100 V was observed in the pure S6 sample. However, in samples doped with CF $KMnO_4$ at different times, it was observed that the electrical conductivity increased significantly depending on the doping time. Doping times t, hours: S9-1.0; S8-3.0; S7-6.0; S6-0.0. This phenomenon is an example of the process of diffusion of solutes. This is the simplest and most striking example of diffusion in solids. In this case, the diffusion mechanism moves atoms from one nodal interval to another. Assuming that there is a density gradient of particles diffusing along the x-axis. Diffusion in it consists of a sudden jump of one of the particles at a distance Δx along the x-axis.

In order to better understand the mechanism of electrical conductivity of "Xorazm-150" sort CF, the dependence of electrical conductivity on temperature was studied. Usually, in semiconductors, with increasing temperature, the electrical conductivity increases according to the law $\sigma=\sigma_0\exp(-E/kT)$. For example, the larger the bandgap value, the more energy the electrons must have to move into the conduction band, which means that the semiconductor must be heated to a higher temperature in order for its conduction to be significant. Fig. 3 shows the results of measuring the temperature dependence of the electric current passing through samples of "Xorazm-150" sort CF doped with iodine and $KMnO_4$. As can be seen from the figure, the electrical conductivity of both samples increased exponentially with the activation energy of E_{t1}=0.54 eV in the sample doped with $KMnO_4$ and E_{t2}=1.41 eV in the sample doped with iodine, respectively. This result means that the inclusions depend on the interaction of the cuticle layer and a certain part of the cellulose on the CF surface.

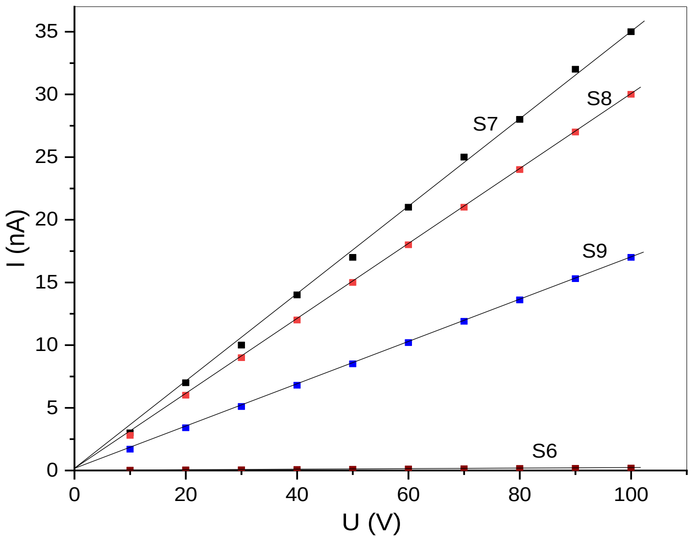

Fig 2. I-V characteristic of CF sample doped with $KMnO_4$ at different times at T=297K

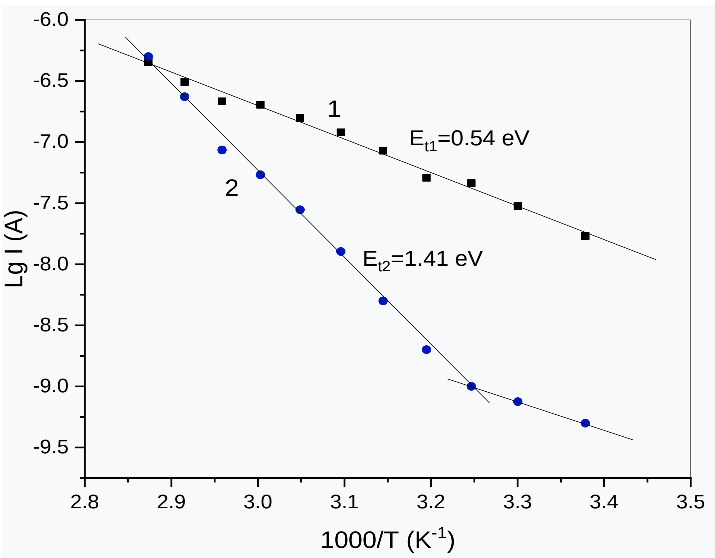

Fig 3. Temperature dependence of electrical conductivity of "Xorazm-150" sort CF doped with $KMnO_4$ (1) and doped with iodine (2). Arrhenius plot

Analysis of the temperature dependence data of electrical conductivity shows that, despite changes in the shape of the current and voltage dependences, the activation energy is constant and does not depend on the pretreatment or input concentration. According to the results, the change of activation energy can be related to the type of ligature and its interaction. According to optical studies, it was determined that the width of the CF forbidden band is approximately E_g=3.36 eV (Zakirov *et al.*, 2011).

Considering that destruction occurs at high temperatures in CF samples, at such temperatures we cannot measure the dependence σ=f(T) in the region of specific conductivity. The graph of the temperature function of the samples based on the Arrhenius law at the ratio 10^3/T is drawn. Current flow at a given constant voltage increased with increasing temperature and was characterized by a slope. Since there is a positive temperature coefficient of conductivity for both samples, it confirms that the samples have semiconducting properties. This phenomenon was also observed by several other research groups (Akobirova *et al*., 2008; Abou-Sekkina *et al*., 1986).

Pure undoped CF is an excellent electrical insulator in the dry state, contains a large number of traps, and exhibits structural deformations at a certain transition temperature (Cunha *et al*., 2007).

The photoconductivity kinetics of “Xorazm-150“ sort CF samples doped with iodine and $KMnO_4$ was investigated (Fig. 4). According to the research, when the sample alloyed with $KMnO_4$ was exposed to a constant voltage and illuminated with UV light (λ=254 nm), an increase in photocurrent was observed, and when the UV light was turned off, a long-term relaxation of photoconductivity was observed, which lasted for about 23 min (4- inside-picture). It was observed that the phenomenon of internal photoeffect was manifested. In the sample of "Xorazm-150" sort CF doped with iodine, a unique phenomenon, which is rarely seen in semiconductor materials, negative photoconductivity (NPC) phenomenon was observed (Fig. 3). When semiconductors are exposed to light above their specific absorption energy, the sum of their dark conductivity (σ_0) and photoconductivity (PC) is equal to the total conductivity (σ), or is found by the following formula:

$$\sigma = \sigma_{\mathbf{0}} + q(\mu_n \Delta n + \mu_p \Delta p) \tag{1}$$

Where μ_n and μ_p are the mobilities of electrons and holes, respectively, and, Δn and Δp are the increasing concentrations of electrons and holes under illumination. NPC is reversed if the conductivity decreases under light. NPC is a flow opposite to the direction of the field in the dark, and it is determined by the formula (2) below.

$$j_{ph} = \sigma_{ph} E; \qquad \sigma_{ph} < 0 \tag{2}$$

Various mechanisms and hypotheses have been proposed to explain the NPC phenomenon. However, no clear consensus has yet been reached about the origin of this phenomenon (Naveen *et al.*, 2022). More theory and experiments are needed on this phenomenon.

Fig. 5 shows the results of I-V characteristics of samples S10, S11, S12 irradiated with γ-^{60}Co radiation source for different times (5, 12, 120 hours) to CF samples of “Komolot-79” sort (after irradiation, all samples are in the same conditions doped with iodine). The number and length of fibers of all samples are almost the same.

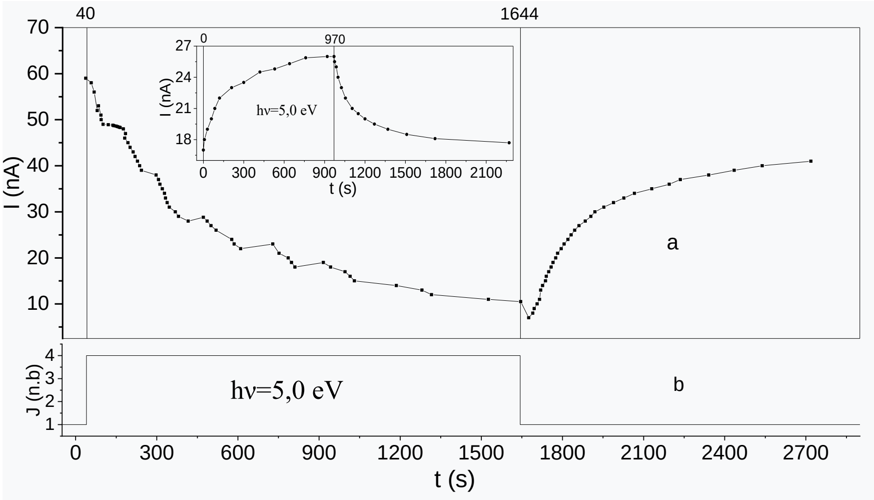

Fig 4. PC kinetics of CF sample sort "Xorazm-150" doped with iodine (inset image doped with $KMnO_4$). hυ =5.0 eV, T=300 K

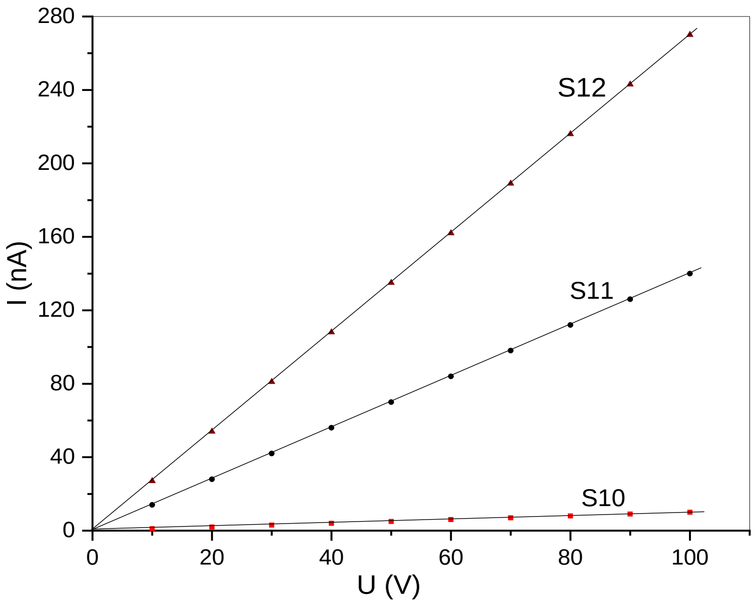

Fig 5. I-V characteristics of CF samples in the dark. T=300 K

According to the results, the electrical conductivity of the radioactively treated and iodine-doped samples increased significantly as the radiation dose increased. According to the conclusion, under the influence of temperature, iodine easily settles into the additional point defects formed in the CF sample under the influence of γ- light depending on the dose, and the iodine molecule sitting in the point defects forms an energy level in the forbidden band.

γ-^{60}Co-nurlanish manbai bilan nurlantirilgan va yod bilan legirlangan "Komolot-79" navli CF fotoo‘tkazuvchanlik spektri (yorug’lik spektri energiya diapazoni 0.26 eV dan 3.5 eV gacha) aniqlandi (6-rasm).

The photoconductivity spectrum of “Komolot-79” sort CF irradiated with γ-^{60}Co-radiation source and doped with iodine (light spectrum energy range from 0.26 eV to 3.5 eV) was determined (Fig. 6). According to the result, after keeping the sample in the dark (1.5 hours), it was observed that the photoconductivity started to increase from the light spectrum hυ=0.68 eV. As the spectral energy increases, a further increase is observed at hυ=2.53 eV, and another current increase is observed when the spectrum of the light beam reaches approximately hυ=3.21 eV. This PC spectrum can be explained by the presence of a deep donor level in the upper half of the CF band gap (Fig. 6). The photoconductivity of this sample is mainly related to the process of formation of electron-hole pairs in inter-band and inter-band energy levels. If we use the classical band model of semiconductors, the above photoconductivity for CF can be explained as follows. Experiments show that doping the CF sample with iodine increases photosensitivity. This will only happen if iodine forms a deep level in the range of the CF forbidden gap.

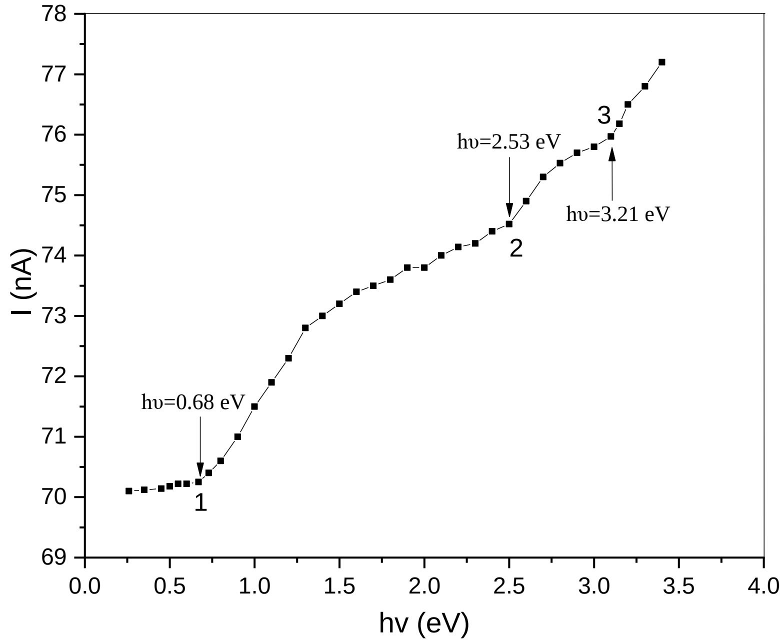

Fig. 6. Photoconductivity spectrum of "Komolot-79" sort CF irradiated with γ-^{60}Co radiation source and doped with iodine

Assuming that iodine forms a deep donor level in the upper half of the band gap, the onset of the PC spectrum is hυ=0.68 eV due to the ionization energy of iodine in CF, which forms a deep level in the band gap E_c-0.68 eV. If we consider that CF doped with iodine has n-type conductivity, then it can be assumed that the level E_t=E_c-0.68 eV is located in the upper half of the band gap, and according to the analysis of the PC spectrum, this we can say that the level has a donor property. The monotonic increase of PC in the region of 0.68≤hυ≤2.53 eV is due to the transfer of electrons from the E_t level to the C band (Fig. 6, 1st pass).

We can say that the further increase of PC from 2.53<hυ<3.21 eV is related to the transfer of electrons from the V-band to the E_t level, that is, a secondary optical transition was observed (Fig. 6, 2nd pass). The increase of the photocurrent starting from hυ=3.21 eV is explained by the direct transfer of electrons from the V-band to the C-band (3rd pass). From a technological point of view, this CF sample shows that it is a photodetector that returns the UV and visible light range. Undoped CF samples have very low conductivity values and very little photosensitivity under UV light. The absence of photoconductivity in samples doped with iodine is explained by the photoinduced charge exchange between iodine molecules and the polymer chain (Morita *et al.*, 1993).

In the wavelength range of 340-600 nm, the photoluminescence (PL) spectra of two different types of CF were compared. To them; Pure (A1) and iodine-doped (A3) "Xorazm-150" sort CF samples, as well as pure (A2) and iodine-doped (A4) "Komolot-79" sort CF samples were selected (Fig. 7). According to the research, PL spectra of different types of cotton fibers differ in intensity and shape. From Fig. 1 It can be seen that the spectrum of CF PL extends from 340 to 600 nm and almost overlaps with each other, and the intensity of the maxima varies in a large range.

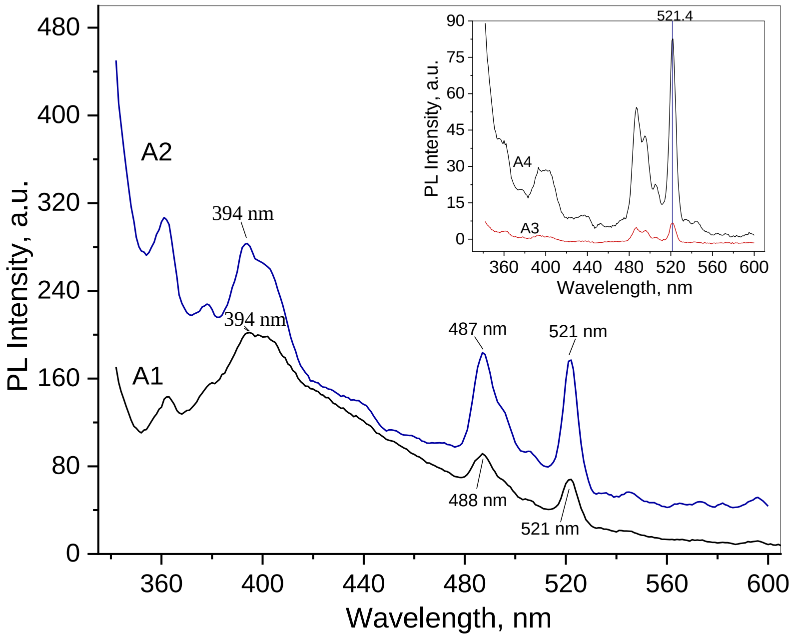

Fig 7. PL spectra of “Xorazm-150” (A1, A3) and “Komolot-79” (A2, A4) sort CF samples excited by 340 nm light

Pure "Xorazm-150" and "Komolot-79" CF PL spectra have three maxima in the regions of 394, 487 and 521 nm for both samples, which differ in intensity. An interesting situation occurred in the samples doped with iodine, i.e. according to Fig. 7, compared to the unalloyed pure sample of "Komolot-79" sort CF doped with iodine, there was an additional maximum yield in the wavelength region of 495.7 nm. A similar situation occurred in the "Xorazm-150" sort CF sample, that is, in the sample doped with iodine, an additional maximum was produced in the wavelength region of 496.6 nm. When CF is doped with iodine, it can be explained by the fact that I_2 molecules form an additional energy level in the fiber. When CF is mercerized and doped with iodine, it affects the lattice structure and the conformational defects of CF increase. Also, the order of the CF chain decreases and leads to an increase in the amorphous region. In conclusion, it can be said that by doping CF with iodine, the intensity of PL can be significantly increased compared to the original material and additional maxima can be created. FL spectra in the blue and green light range were observed in cotton fibers when excited by UV light. This chemically and thermally treated CF can be tailored for potential applications in optoelectronic devices.

4. Conclusion

In conclusion, the electrophysical and optical properties of cotton fibers of the "Komolot-79" and "Xorazm-150" sorts treated with radioactive, chemical and thermal treatment were studied. The experimental results were analyzed. It was determined that the electrical conductivity of "Xorazm-150" sort CF doped with $KMnO_4$ depends on the doping time. It was found that the electrical conductivity of CF doped with iodine, irradiated with γ-^{60}Co, depends on the radiation dose. Structural changes in the samples were determined using the infrared spectroscopy method. Temperature dependence of electrical conductivity of "Xorazm-150" sort CF doped with $KMnO_4$ and iodine was determined. A long-term relaxation of the photoconductivity of “Xorazm-150” sort CF doped with $KMnO_4$ was observed, and in the sample doped with iodine, a unique phenomenon rarely seen in semiconductor materials, i.e., the phenomenon of negative photoconductivity, was determined. Insertion molecules act as additional anchoring centers and provide bonding between polymer molecules in the amorphous sphere, resulting in the formation of charge-transfer complexes. It was analyzed that the Poole-Frenkel mechanism is the main conduction mechanism for undoped pure CF. Especially the clear photoconductivity in the UV and visible region shows that the photoconductivity is mainly related to the formation of interband electron-hole pairs. It is explained by the photoinduced charge conduction in iodine molecules and polymer sphere. Photoluminescence in the visible range was observed for CF excited by UV light. The obtained results show that CF processing plays an important role in the enhancement of light emission.

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