EFFECTS OF STERILIZATION IRRADIATION ON PROPERTIES OF COMMERCIALLY AVAILABLE PET MATERIALS USED IN THE PRODUCTION OF VACUUM TUBES FOR BLOOD SAMPLING

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Abstract

The effect of irradiation with accelerated electrons (energy 8.5 MeV, dose 5 kGy) on the physical properties of medical products made of polyethylene terephthalate (PET) was studied by methods of assessing the visible light transmission. The identified post-radiation changes from irradiation with electrons of 5 kGy confirm some changes in PET products during the radiation sterilization procedure.

1. INTRODUCTION

The introduction of radiation technologies into the production or post-processing of products made of polymers and plastics makes it relevant to study the processes occurring in such materials under the influence of radiation [1]. Polyethylene terephthalate (PET) is the most widely consumed in the polyester fiber segment. PET packaging is currently actively replacing such traditional types of pharmaceutical, medical and laboratory packaging as glass and cardboard. However, due to their heat sensitivity, PET medical products require so-called “cold” sterilization methods. To date, there are methods of sparing "cold" sterilization of medical devices with low-temperature atmospheric plasma [2], which allows achieving the necessary bactericidal effect while maintaining the necessary physical and chemical properties of medical devices. At the same time, an industrial method for sterilizing medical devices, which makes it possible to sterilize objects directly in a sealed package, is the treatment with accelerated electrons with energies below the threshold for the occurrence of nuclear reactions (usually up to 10 MeV). The method allows you to process large volumes of products in a short time without opening the factory packaging. The state standart ISO 11137-1-2011, which regulates the radiation sterilization procedure in the Russian Federation, defines a sterilization dose range of 15–25 kGy. The choice of dose from the normalized range is primarily carried out from the conditions of ensuring the established requirements for sterility. On the other hand, the radiation sterilization procedure should not significantly impair the consumer properties of products [3]. In this regard, the search for sensitive methods for assessing the post-radiation changes in the physicochemical properties of PET medical products seems to be relevant.

The material for the study was samples of medical PET tubes for blood sampling produced by Zdravmedtech-E JSC. Samples were irradiated with accelerated electrons at the Innovation and Implementation Center for Radiation Sterilization of the Ural Federal University (UrFU). The irradiation dose was 5 kGy at an electron energy of 8.5 MeV. At the same time, the paper did not set the task of establishing the lower threshold of the sterilization dose, but the main purpose of the work was to assess the sensitivity of the method to determining the initial stages of changes in the physicochemical properties of irradiated materials [4].

It is known [5] that exposure to ionizing radiation leads to a change in the physicochemical properties of materials. Also, according to the study [6], for this material, an electron irradiation dose of 1 MGy leads to a drop in ultimate strength by 15–30%. Due to the fact that their vibrational and optical spectra also depend on the chemical composition and structure of substances, it was decided to analyze the change in the transmission of visible light through test tubes [7].

2. RESULTS AND DISCUSSION

Two series of tests were carried out to evaluate the change in the optical properties of PET tubes. In the first series, the task was to analyze the change in the refractive index. For this purpose, an optical system was assembled, shown in Fig. 1.

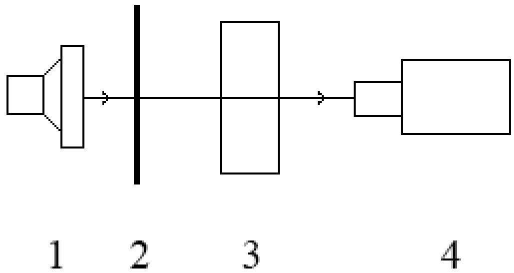

FIG. 1. Optical installation for the analysis of samples on a transmission. The presented installation includes an illuminator (1), a matte diffuser (2), an object (3), a video camera (4).

The illuminator is a powerful source of uniform radiation - an LED assembly with a luminous flux of 880 lm. As a matte diffuser, a substrate made of a randomly reinforced composite material with cellulose filler was used, on which a scale grid was previously applied. The samples were placed in a horizontal plane between a matte diffuser and a video camera on a special mount. The mount ensured the identity of the position of the irradiated and non-irradiated samples during the shooting process. Registration of a uniform light flux, as well as the light flux passing through the irradiated and non-irradiated samples, was performed on the camera in turn. A black-and-white camera Videoscan-415 was used as a video camera with the possibility of programmatically changing the exposure time. Within each series of tests, the exposure time was selected so that the radiation brightness in the controlled area was in the range of average values.

During the experiments, several series of surveys were made. Based on the obtained images, the change in the intensity of the light flux passing through the samples was analyzed, and a comparison table was compiled between the samples that were irradiated and the samples without irradiation. The camcorder generates 8-bit black and white images with a range of gray shades, expressed in signal levels from 0 to 255.

Since real products were used as the object under study, it is possible that during the manufacturing process their walls may have some differences in thickness, even within the same batch, so there was a need to apply a technique for analyzing the size of the zone and the contribution of defects to the study pattern. Measurements of the signal levels characterizing the brightness of an object were carried out by averaging the values around the selected point for areas of 2x2, 3x3, 4x4 and 5x5 pixels in order to select the optimal size of the area.

Initially, about 15 measurements were made for each sample at various random points. This was done in order to collect a large amount of data and take into account possible errors and inaccuracies associated with the geometric dimensions of various samples, as well as wall thickness parameters. The calculation of the values was carried out in several stages:

1. The values of the signal intensities around each point for each area were entered into a separate array.
2. The arithmetic mean value of the signal level for each array was found.
3. The results were then compared between samples, after irradiation and without irradiation, and all samples were compared with the original image of the frosted diffuser.

For example, the data obtained from two points for different objects (Fig. 2).

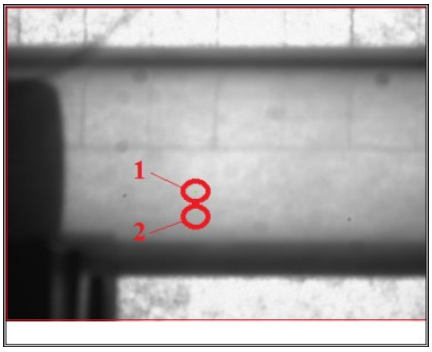

FIG. 2. Location of signal levels characterizing the center of the measurement area, 1 – with coordinates (332;342), 2 – with coordinates (332;382).

Formula for calculating intensity distribution:

$$I = \frac{(I_{pure.} - I_{irrad.})}{I_{pure.}} * 100\%,$$

where, I_{pure} is the arithmetic mean of the intensities at each point for the selected area in the unirradiated sample, and $I_{irrad.}$ is the same value for the sample after irradiation, taken for a region of the same size and at the same point. The value is given as a percentage, and its positive value indicates that for a pure sample, the signal levels are on average higher than for an irradiated one.

Similarly, the difference in intensity between the original image and all samples was calculated. The data are presented in Table 1.

TABLE 1. AVERAGE INTENSITY OF THE ORIGINAL IMAGE AND LIGHT TRANSMISSION

Sample	332;342				332;382			
	2x2	3x3	4x4	5x5	2x2	3x3	4x4	5x5
9 ml irradiated	23.54%	23.10%	23.47%	23.44%	29.42%	29.77%	30.35%	30.31%
9 ml pure	22.02%	22.15%	21.95%	21.82%	27.58%	27.77%	28.34%	28.53%
5 ml irradiated	10.40%	10.66%	11.18%	11.14%	14.20%	14.97%	16.24%	16.79%
5 ml pure	4.55%	3.97%	3.91%	3.98%	11.54%	11.61%	12.13%	11.98%
2 ml irradiated	13.64%	14.03%	14.16%	14.27%	19.51%	19.83%	20.40%	21.07%
2 ml pure	12.42%	12.28%	11.94%	11.93%	17.88%	17.74%	17.96%	17.91%

On average, a relative change in intensity of 3-4% to non-irradiated samples was observed. The change in intensity indicates that the object began to transmit less light after irradiation, which probably indicates a deterioration in its optical properties, i.e. about a proportional increase in the refractive index. With an increase in the field of analysis, the spread of values increases by 0.5-2%. From the point of view of geometry, the most accurate values seem to be for samples with a volume of 9 ml. Their lateral surface is the largest of those presented, and, probably, therefore, the effect of geometry with an increase in the size of the controlled area was less, and the discrepancies in the values for different areas do not exceed 0.5%. The same cannot be said about samples of smaller size, in which, in addition to the processes described above, associated with a decrease in the transparency of the sample under the action of irradiation, the geometry of the sample also has a significant effect. The spread of values for such samples is about 2%.

According to classical ideas about glasses and other optically transparent objects, as the refractive index increases, the reflection coefficient also increases in them [8], in order to test our previous theory, it is enough to establish that the reflection coefficient also increases. In this case, if we place the light source away from the chamber, as shown in Fig. 3, and record the light reflected from the walls of the tube, then its intensity for the irradiated samples should be higher.

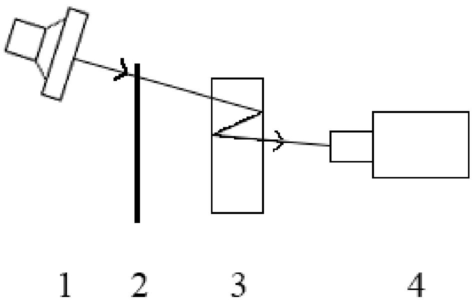

FIG. 3. Optical system for reflectance analysis and an example sample image. The numbering of the objects of the optical system corresponds to Fig. 1

For this series of tests, many points in various segments of the image were taken into account, and the most characteristic ones were selected from them. Figure 4 shows the location of points for one of the samples. The points were chosen at some distance from the light source and from the edges of the sample in order to exclude the influence of the geometry and position of the samples. The results of a series of tests fully confirmed the hypothesis put forward and showed an increase in the intensity of the reflected flux by 7–10% for various points in the region of the sample. The data is presented in Figure 5.

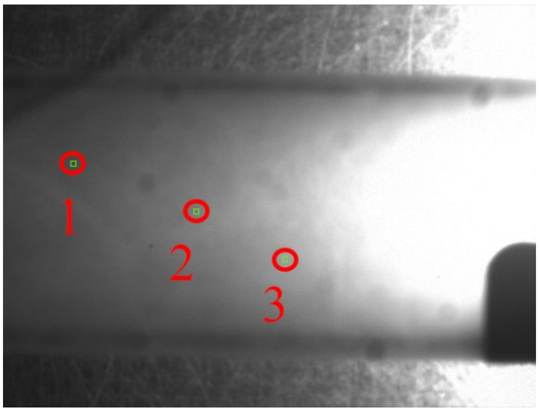

FIG. 4. Image of the sample with the designation of the studied areas for points: 1- (100; 230), 2- (280; 300), 3- (410; 370).

From the graphs in Figure 5 it can be seen that the intensity of the reflected light flux is greater where the object was irradiated, which in turn may indicate an increase in the reflection coefficient and, as a result, an increase in the refractive index. This kind of approach can also be used for optical control of products in a stream production, in the case when it is not possible to unambiguously determine the location of the sample on the conveyor belt and its exact position relative to the camera and light source. Thanks to this kind of research and more statistical measurements, this task can be accomplished.

It should be noted that when testing samples in transmission, a series of tests showed the closest possible results for different samples, only if the points were selected in the area where the signal intensity was in the range of 180-240 shades of gray. Thus, the most reliable results can be obtained for brighter regions. At the same time, when testing for the analysis of the reflection coefficient, the opposite picture was observed and the average range was chosen from 50 to 100 shades of gray, i.e. darker areas were selected for study. This is generally obvious, since when determining the refractive index, the object by its presence leads to a decrease in the final brightness of the emitter, and when determining the reflection coefficient, it is required that the background image be as dark as possible. This is necessary to enhance the contrast between the background radiation level and the signal level reflected from the sample walls.

Thus, because of the experiments, it was found that in the presented samples after irradiation, there is a completely distinguishable deterioration in the optical properties, according to a preliminary estimate, by 5-6%, more details about the nature of the change in the physical properties of the material can be learned with an increase in the dose of irradiation.

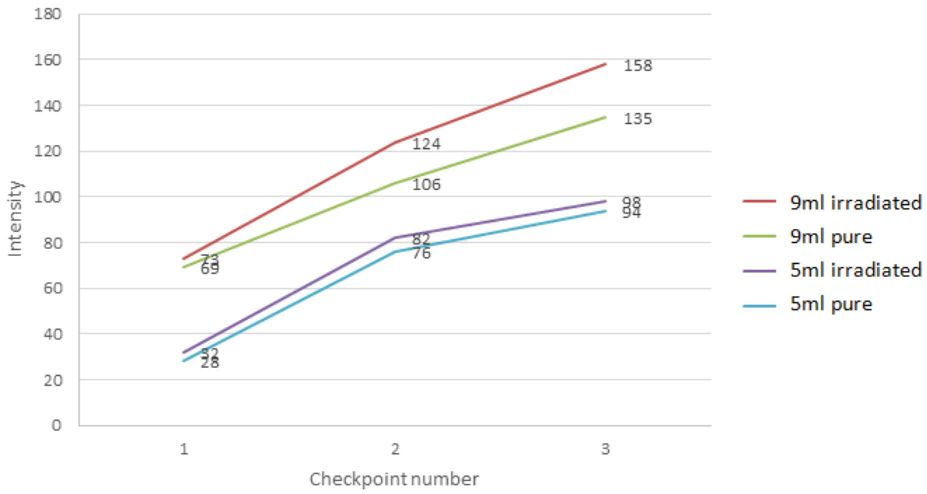

FIG. 5. Graph of the distribution of the radiation intensity of the light flux for points: 1 (100;230), 2 (280;300), 3 (410;370).

3. CONCLUSION

It has been established that exposure to irradiation of 5 kGy affects the transmission of visible light. According to the results of earlier IR spectroscopy, this effect is associated with a decrease in the number of C–H bonds [9]. The decrease in C-H bonds, apparently, is associated with the process of dehydrogenation of the test tube material, which in turn changes the optical properties of the object under study - leads to an increase in the refractive index and a proportional increase in the reflection coefficient.

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Conflict of Interest: The authors declare that they have no conflict of interest.

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