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# EFFECT OF GAMMA IRRADIATION ON THE DIELECTRIC PROPERTIES AND X-RAY PHASE ANALYSIS OF THE POLYPROPYLENE NA$^+$MONTMORILLONITE COMPOSITE

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Effect of γ-irradiation on the dielectric properties of polymer composites based on polypropylene (PP) with Na$^+$-montmorillonite (MMT) nanoparticles has been investigated. It has been found that γ-irradiation in the dose range of 100-200 kGy leads to slight deterioration in the dependences of $\varepsilon = f(T)$ (a) and $tg\delta = f(T)$ of the polypropylene-based composite with Na$^+$MMT filler. Further increasing the radiation dose, a sharp increase in ε and tgδ above the temperature ~ 440 K has been observed. The obtained results show that after irradiation, the active centers, radicals, and defects formed in the polymer structure interact with filler nanoparticles, reducing the mobility of the polymer chain and consequently decreasing the values of ε and tagδ of the composite. But at an irradiation dose of 300 kGy, the properties of the interphase layer deteriorate as a result of incipient destruction, which leads to an insignificant increase in the values of ε and tgδ. The results of the analyses showed that the introduction of Na nanoclays (MMT) into polypropylene in an amount of 0.5% by volume leads to some improvement in the dielectric properties before and after irradiation. It is assumed that the deterioration of the dielectric properties during irradiation in composites occurs due to changes in the crosslinking and destruction processes in the polymer matrix and at the polymer-filler interphase boundary. It is shown that due to γ-irradiation of polymer composites, it is possible to expediently control its dielectric and electrophysical properties.

**Keywords**: *Polypropylene; Na$^+$-montmorillonite; Gamma irradiation; Dielectric constant; Dielectric energy loss*
**PACS**: 71.20 Nr; 72.20 Fr

## INTRODUCTION

One of the promising fields of materials science is the creation of new composite polymer materials with unique properties. Introduction of a filler into a polypropylene (PP) matrix considerably modifies the structure and properties of composite materials due to interfacial interactions and the formation of a boundary nanolayer near the filler particles. This identifies the peculiarities of the temporary distribution of the local field in certain regions of the polymer system and the frequency dispersion of the effective complex dielectric permittivity of composite materials. The use of polymer materials enables the development fundamentally new designs of various types of products, helps to reduce their weight, operating and transportation costs, improves their quality and appearance [1-4].

Polymer composites have been known to be very sensitive to the effects of various ionizing radiation. The literature research in this direction reveals that low doses of radiation exposure led to a relative improvement in physical properties, whereas high doses lead to the destruction of materials, worsening their physical properties. In this perspective, the study of electrophysical properties of polymer composites before and after exposure to γ-irradiation is significant. Exposure to ionizing radiation leads to changes in the properties of the supramolecular structure (SMS) of the matrix and the boundary layer as a result of the processes of crosslinking, destruction and oxidation and, therefore, to a change in the electroactive properties of the composites, which consists of studying their electrophysical properties, i.e. temperature and frequency dependences of electrical conductivity (σ) and dielectric characteristics (ε and tgδ) [5-8].

Literature data analysis showed that introduction of $Dk_1$ and $Dk_2$ types of nanoclays into polypropylene in the amount of 2% by volume content leads to some improvement in physical and mechanical properties. In these works, however, there is no information about the structure of these fillers and the degree of their compatibility with the matrix. But it should be noted that depending on the deposit, montmorillonite (MMT) has different modifications and differ in particle size. The use of Na$^+$MMT as modifiers is related, firstly, to the amount of quaternary ammonium guanine cations in them and, secondly, to the good adhesion of clay with polar polyolefin molecules [9].

In this context, the presented work is aimed at studying electrophysical properties and relaxation processes in initial and irradiated polymer composites (PP/Na$^+$MMT). Despite the large-scale study of Na$^+$MMT characteristics the dielectric, electrophysical properties and peculiarities of interfacial phenomena in its composites with polymers remain understudied. In our opinion, research in this direction will end in the future with the creation of electroactive composite materials with new characteristics and converters based on them.

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## EXPERIMENTAL PART

Composites were prepared using polypropylene (PP) as the polymer matrix and nanoscale sodium montmorillonite ($Na^+$MMT) particles as the reinforcing filler. Sodium montmorillonite is a type of smectite clay mineral, which typically forms through the long-term weathering of volcanic ash and rocks, often aided by biological activity, such as the actions of lichens and fungi. The structure of $Na^+$MMT consists of layered silicate sheets with a very fine crystal grain size, typically less than 100nm. These layers carry a net negative charge, which is balanced by exchangeable cations such as $Na^+$ located in the interlayer spaces. This charge asymmetry enables montmorillonite to interact with a wide range of chemical species. Due to its layered structure and high specific surface area, $Na^+$MMT can adsorb both cationic and anionic substances, making it highly versatile in composite and environmental applications. Even nonionic substances can be retained through secondary valence forces, such as van der Waals interactions or hydrogen bonding. Furthermore, the interlayer space within montmorillonite crystals can accommodate various molecules, allowing for intercalation and improving dispersion in polymer matrices.

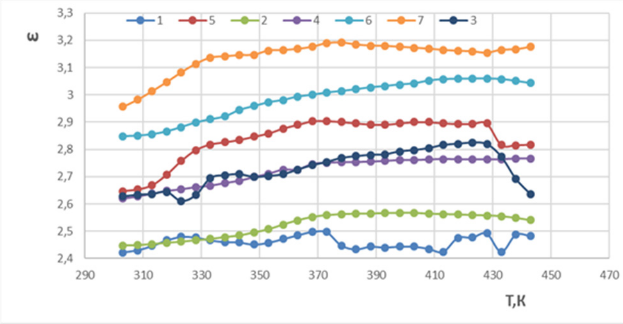
The samples were prepared from mixtures of PP and $Na^+$MMT powders in different component ratios, and then from these mixtures, by hot pressing at the melting point of the polymer matrix under a pressure of 15 MPa for 10 minutes, PP/$Na^+$MMT nanocomposites were prepared in the form of a film, with subsequent cooling. The samples were obtained in different temperature-time crystallization modes, namely, slow cooling (SC), when the samples are cooled to room temperature at a rate of 2 deg/min, and rapid cooling (RC) in an ice-water mixture at a rate of 30 deg/min [9].

For the preparation of the composites, disc-shaped samples were fabricated by homogenously mixing polypropylene with varying volume fractions (x vol.%) of $Na^+$MMT particles. The MMT filler particles had sizes up to 100 nm. The mixture was processed to ensure uniform distribution of the clay particles within the polymer matrix, which is critical for achieving reliable and reproducible dielectric properties. To characterize the electrical performance of the resulting composites, dielectric measurements were conducted using an E7-20 immittance meter. The frequency range for these measurements spanned approximately from 25 Hz to $10^6$ Hz. This wide frequency range allowed for the analysis of different polarization mechanisms and charge transport phenomena within the composite material. The results help to evaluate how the inclusion of $Na^+$MMT affects the dielectric behavior of the polypropylene matrix and can provide insight into the composite's potential applications in electronics, insulation, or barrier materials. X-ray phase analysis was carried out on a Bruker D2 diffractometer. Cu Ka ($\lambda$=1.541780 Å) was used as the light source passing through a nickel filter in the same mode. The reflection measurement error did not exceed $\theta$=±0,020 [10].

## RESULTS AND DISCUSSION

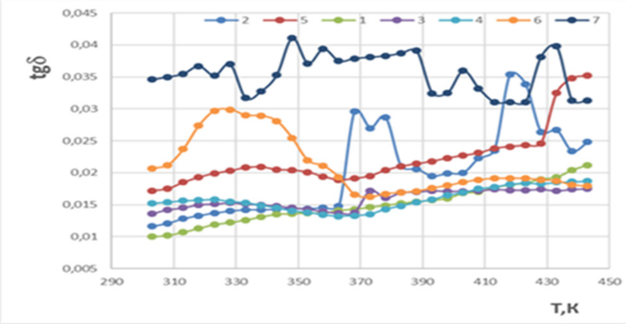
For comparison with irradiated samples, the given dependences reflect the results of the initial polymer and composite based on it. It is known that polypropylene is a polar, polycrystalline polymer and has a certain dipole moment, which is reflected in the temperature dependences of $\varepsilon$ and tg$\delta$.

Fig.1 depicts the temperature dependences of $\varepsilon = f(T)$ (a) and tg$\delta = f(T)$ (b) of the initial polypropylene composite with 0, 0.5, 2, 3, 5, 6, 7 vol.% $Na^+$MMT fillers measured at a frequency of $\nu$ = 1 kHz. It is clear from Fig.1(a) that the dielectric constant ($\varepsilon$) of the initial PP varies slightly in the temperature range of 320-450 K. Furthermore, in the dependence of $\varepsilon = f(T)$, small maxima were observed in the temperature range of 330–333 K, 370–380 K, and 430–433 K, due to the sample inhomogeneity. According to Fig.1a, curve 2, when $Na^+$ (0.5 vol.%) is added to the polymer, the values of $\varepsilon$ do not change in the temperature range of 300-350 K, and it increases slightly with a further increase in temperature. Meanwhile, the previously observed maxima in the range of 370-380 K and 430-433 K are not observed, which may be associated with an increase in the mobility of the polymer chain. The result obtained is confirmed by our previous results. Fig.1 (a) illustrates that with further increase in Na content (2; 3 and 5 vol.%) in the polymer, $\varepsilon$ increases exponentially in the range of 320-450 K. It is also evident from Fig. 1(a) that the samples containing 2 and 3 vol.% $Na^+$MMT show a decline in dielectric constant in curves 4 and 5 over the temperature of 440 K.



a

**Figure 1a.** Temperature dependence of $\varepsilon = f(T)$ before irradiation of the PP+vol.%$Na^+$MMT composite at a frequency of $\nu$ = 1 kHz. 1-0%; 2-0.5%; 3-7%; 4-6%; 5-5%; 6-3%; 7-2%



b

**Figure 1b**. Temperature dependence of tg$\delta$ = f(T) before irradiation of the PP+vol.%$Na^+$MMT composite at a frequency of $\nu$ = 1 kHz. 1-2%; 2-3%; 3-5%; 4-6%; 5-7%; 6-0.5%; 7-0%

Figure 1b. demonstrates the temperature dependence of tgδ = *f*(*T*) before irradiation of the PP/(0, 0.5, 2, 3, 5, 6, 7 vol.%) Na$^+$MMT composite at a frequency of ν = 1 kHz. The dependences demonstrate that the introduction of Na$^+$MMT into the polymer leads to a rather pronounced maximum of tgδ in the initial composite, which is due to dielectric losses during dipole relaxation and relaxation of the accumulated charge at the interphase boundary of the amorphous and crystalline phases in the polymer matrix and matrix with Na$^+$MMT. Comparison of the curves points out that the observed maximum in ε = *f*(*T*) and the corresponding shoulder in the dependence of tgδ = *f* (*T*) of the composite is considered to be the result of the relaxation of the charge accumulated at the polymer-filler interface, which is released upon crosslinking of the polymer matrix.

To study the effect of γ-irradiation on the dielectric properties in PP/Na$^+$MMT composites, the dependences of ε = *f*(*T*) and tgδ = *f* (*T*) were investigated at T=300 K at various radiation doses. Figure 2 represents the temperature dependence of ε = *f* (*T*) after irradiation of the PP/(0, 0.5, 2, 3, 5 vol.%) Na$^+$MMT composite with 200 kGy at a frequency of ν = 1 kHz. Fig. 2(a) demonstrates that upon irradiation with a dose of 200 kGy in the dependence of ε = *f*(*T*), a sharp increase in the curves is observed at 440 K only for samples 1 and 5.

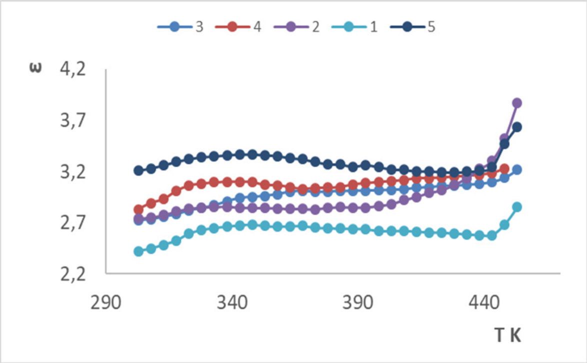


**Figure 2a**. Temperature dependence of ε = *f* (*T*) after irradiation of the PP/Na$^+$ MMT composite with 200 kGy at a frequency of ν = 1 kHz. 1-0%; 2-2%; 3-3%; 4-5%; 5-0.5%

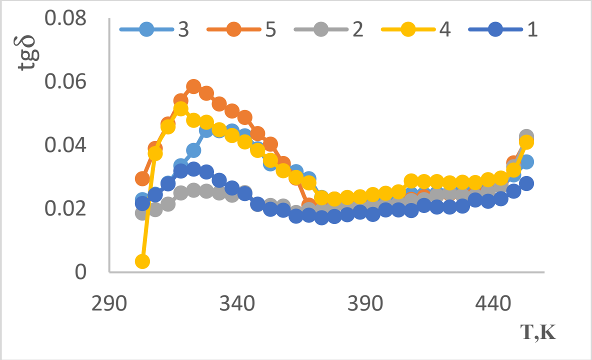


**Figure 2b**. Temperature dependence of ε = *f* (*T*) and tgδ = *f*(*T*) after irradiation of the PP/Na$^+$ MMT composite with 200 kGy at a frequency of ν = 1 kHz. 1- 0.5%; 2-5%; 3-3%; 4-2%; 5-0%

With a further increase in the radiation dose (300 kGy, Fig. 3(a)), the dielectric constant values change slightly in the temperature range of 320-440 K, and a sharp increase is observed at T>440K. This means that at T>440K, polymer degradation begins due to the interaction of the polymer matrix with radiation defects and filler nanoparticles.

Fig. 3 (b) demonstrates the temperature dependence of tgδ = *f*(*T*) after irradiation of the PP/(0, 0.5, 2, 3, 5 vol.%) Na$^+$MMT composite with 300 kGy at a frequency of ν =1kHz. Figure 3(b) reveals that in the polymer irradiated at the dose of 300 kGy, tgδ does not change in the range of 350-420K, and a sharp increase begins at the subsequent temperature rise (curve 1). In samples with introduced nanoclay (curves 2-5) in the range of 310-350 K, tgδ increases slightly after irradiation and then remains constant. At temperatures T>440 K a significant increase in tgδ occurs, indicating a decrease in the properties of the composite material and the onset of degradation.

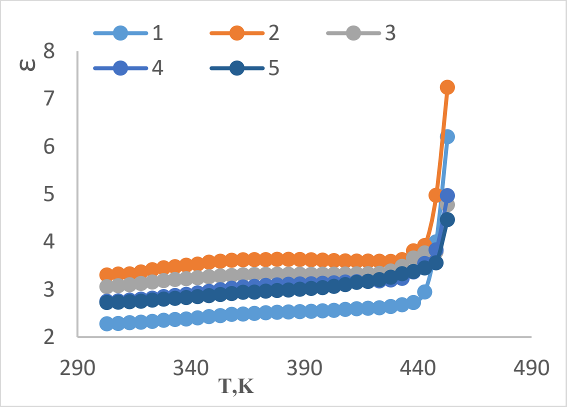


**Figure 3a**. Temperature dependence of ε = *f* (*T)* after irradiation of the PP/Na$^+$ MMT composite with 300 kGy at a frequency of ν = 1 kHz. 1-0%; 2-7%; 3-6%; 4-5%; 6-2%

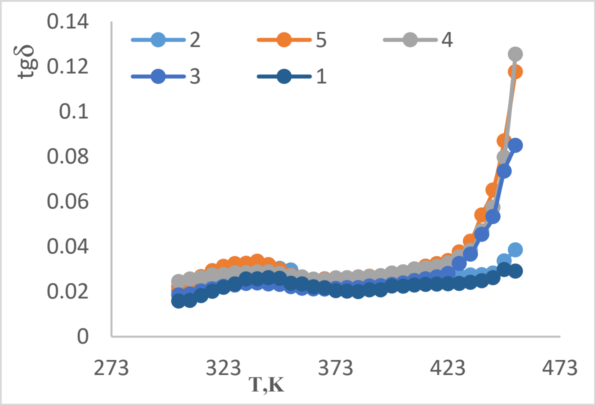


**Figure 3b**. Temperature dependence of tgδ = *f*(*T*) after irradiation of the PP/Na$^+$ MMT composite with 300 kGy at a frequency of ν = 1 kHz. 1-2%; 2-3%; 3-5%; 4-6%; 5-7%; 6-0%

To study the impact of irradiation on relaxation processes in PP/Na$^+$MMT composite, their frequency dependences of ε = *f* (ν) and tgδ = *f*(ν) at T = 298 K were comparatively analyzed. These dependencies allow estimating the intensity of molecular mobility of different relaxing processes. Fig. 4 (a) and (b) represent the dependences of ε = *f* (ν) and tgδ = *f* (ν) before irradiation measured at a temperature of T = 298 K (b). Figure 5 (a) and (b) show the dependences of ε = *f* (ν) (a) and tgδ = *f* (ν) after irradiation, measured at a temperature of T = 298 K.

As is known [11], the transition of the polymer matrix from glassy to highly elastic state in the polymer-filler system leads to a decrease in the orientation stability of dipoles and an increase in the mobility of the polymer chain. And the relaxation properties of the composite depend very strongly on these processes and the measurement temperature. At low temperatures T~ 298 K, Figs. 4(a), 4(b), 5(a), 5(b), relaxation maxima in the dependence of tgδ $= f(\nu)$ of the initial and irradiated samples of PP/Na$^+$MMT composites are not revealed and only an increase at high frequencies is observed in the measured frequency range.

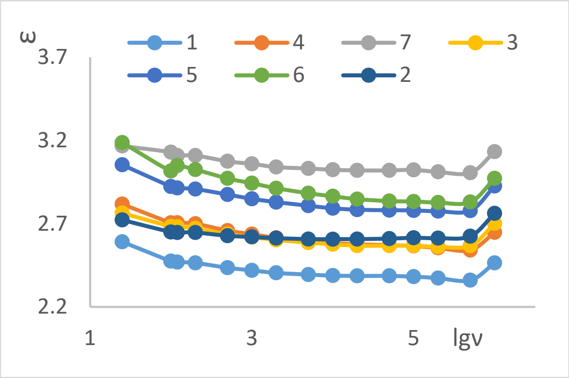


**Figure 4a**. Frequency dependence of ε $= f(\nu)$ before irradiation of the PP/Na$^+$ MMT composite at a temperature of T=298 K. 1-0%; 2-2%; 3-3%; 4-5%; 5-6%; 6-7%

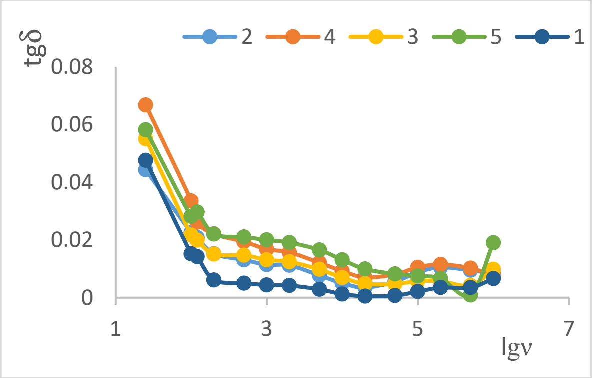


**Figure 4b**. Frequency dependence of tgδ- $= f(\nu)$ before irradiation of the PP/Na$^+$ MMT composite at a temperature of T=298 K. 1-3%; 2-5%; 3-7%; 4-2%; 1-0%

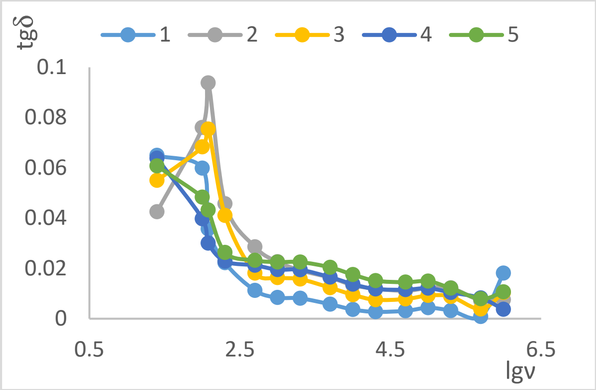


**Figure 5 a.** Frequency dependence of tgδ- $= f(\nu)$ after irradiation of the PP/Na$^+$ MMT composite with 100 kGy at a temperature of T=298 K. 1-0%; 2-0.5%; 3-7%; 4-6%; 5-5%; 6-2%

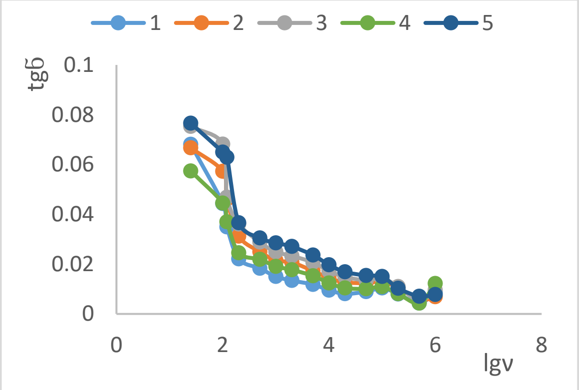


**Figure 5 b.** Frequency dependence of tgδ- $= f(\nu)$ after irradiation of the PP/Na$^+$ MMT composite with 300 kGy at a temperature of T=298 K. 1-0%; 2-0.5%; 3-7%; 4-6%; 5-5%; 6-2%

The findings indicate that after irradiation the formed active centers, radicals and defects in the polymer structure interacting with filler nanoparticles lead to a decrease in the mobility of the polymer chain and, therefore, to a decrease in the values of ε and tgδ of the composite. However, at a radiation dose value of 300 kGy, the properties of the interphase layer deteriorate as a result of the onset of degradation, which leads to a minor increase in the values of ε and tgδ. γ-irradiation is commonly known to ionize the polymer chain, resulting in crosslinking and chain rupture by free-radical mechanism. The forming ions then cause chemical interactions between molecules at different concentrations in the second phase. The degree of crosslinking depends on the polymer structure, phase morphology, gamma irradiation with controlled dose and duration, and the nature of the gamma radiation source.

Based on the literature [12], it is also known that the introduction of fillers into the polymer matrix in composites leads mainly to a decrease in the mobility of the macromolecules of the polymer chain. During gamma radiation exposure, the polymer chains interaction with the filler surface increases during crosslinking and decreases with degradation. The dependence of tgδ = $f(\nu)$ makes it clear that the maxima of the samples at radiation doses of 100 and 300 kGy are shifted towards low frequencies relative to the initial one, which is associated with crosslinking and a decrease in the mobility of polymer chains.

The Figure 6 represents the X-ray diffraction patterns of different samples of the composite. As can be seen, with increasing the amount of MMT in the composite, the intensity of the reflexes decreases in the range of values $2\theta=13\text{-}25^0$. This happens by decreasing the size of PP crystallites, i.e. due to the formation of new supramolecular structures.[13]

According to the results presented, the effect of radiation on the relaxation of volume charges accumulated in the polymer-filler interphase boundary can be observed. In the dependence of tgδ $= f(\nu)$ for the initial PP/Na$^+$MMT composite, the observed flat maximum measured at a temperature of T~ 298 K consists of two components: the relatively low-frequency one corresponds to the relaxation of the volume charge in the interfacial boundary, and the high-frequency one corresponds to the relaxation dipoles in the matrix.

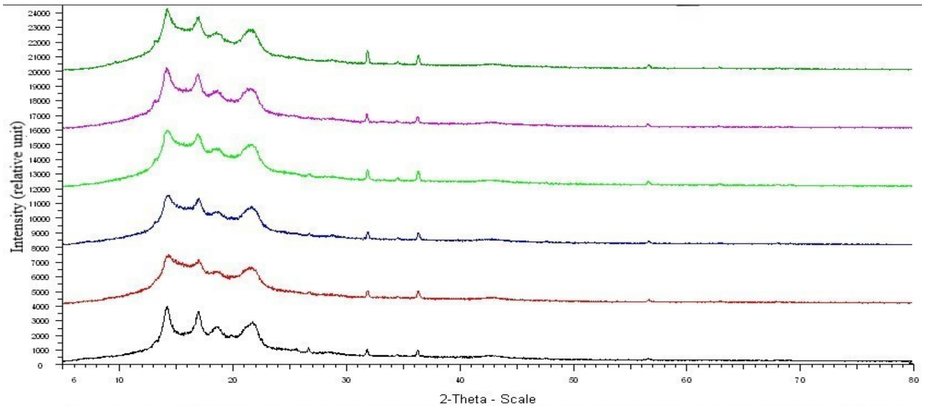


**Figure 6.** The figure represents the X-ray diffraction patterns of different samples of the composite PP+vol.% Na$^+$MMT composite (—) – 0 %; (—)- 2%; (—) - 0,5%; (—) - 3%; (—) - 4%;(—) - 5%.

## CONCLUSIONS

Hence, on the basis of investigation of frequency and temperature dependences of dielectric coefficients (ε, tgδ) in γ-irradiated polymer composites with Na$^+$ montmorillonite (MMT) nanoparticles, the nature of dielectric losses, crosslinking and degradation mechanism have been revealed.

Having compared the obtaining results of temperature and frequency dependences of electrophysical properties of the initial and γ-irradiated PP/Na$^+$MMT composite, it is possible to state that observed changes in these properties are determined by the ratio of crosslinking and degradation processes occurring in the polymer matrix and in the interphase boundary of the polymer with filler. It has been demonstrated that due to γ-irradiation of polymer composites its dielectric and electrophysical properties can be reasonably controlled. This offers opportunities for creating fundamentally new types of products.

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# ВПЛИВ ГАММА-ОПРОМІНЕННЯ НА ДІЕЛЕКТРИЧНІ ВЛАСТИВОСТІ ТА РЕНТГЕНОФАЗОВИЙ АНАЛІЗ КОМПОЗИТУ ПОЛІПРОПІЛЕН NA+МОНТМОРИЛОНІТ

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Досліджено вплив γ-опромінення на діелектричні властивості полімерних композитів на основі поліпропілену (ПП) з наночастинками Na+-монтморилоніту (ММТ). Було виявлено, що γ-опромінення в діапазоні доз 100-200 кГр призводить до незначного погіршення залежностей ε = f(T) (a) та tgδ = f(T) композиту на основі поліпропілену з наповнювачем Na+MMT. При подальшому збільшенні дози опромінення спостерігається різке збільшення ε та tgδ вище температури ~ 440 K. Отримані результати показують, що після опромінення активні центри, радикали та дефекти, що утворюються в структурі полімеру, взаємодіють з наночастинками наповнювача, зменшуючи рухливість полімерного ланцюга та, як наслідок, зменшуючи значення ε та tagδ композиту. Але при дозі опромінення 300 кГр властивості міжфазного шару погіршуються внаслідок початкового руйнування, що призводить до незначного збільшення значень ε та tgδ. Результати аналізів показали, що введення наноглин Na (MMT) у поліпропілен у кількості 0,5% за об'ємом призводить до деякого покращення діелектричних властивостей до та після опромінення. Вважається, що погіршення діелектричних властивостей під час опромінення в композитах відбувається через зміни процесів зшивання та руйнування в полімерній матриці та на міжфазній межі полімер-наповнювач. Показано, що завдяки γ-опроміненню полімерних композитів можна доцільно контролювати їх діелектричні та електрофізичні властивості.

**Ключові слова:** *поліпропілен; Na+-монтморилоніт; гамма-опромінення; діелектрична проникність; втрати діелектричної енергії*