

## COMPARATIVE ANALYSIS OF DIELECTRIC AND STRUCTURAL CHARACTERISTICS OF THE SAMPLES BASED ON POLYETHYLENETEREPTALATE

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**Abstract:** Widespread use of polymeric materials from the items for daily use to electrical engineering, electronics and medical applications causes a need for their detailed examination under various external influences. Dielectric spectroscopy allows monitoring changes in the structure of polymers under various external influences. In this paper we analyzed  $\beta$  relaxation recorded in polymer woven structures and meshes based on polyethyleneterephthalate. Based on the results obtained, a comparative analysis of dielectric parameters and structural characteristics of the samples was done.

**Keywords:** dielectric spectroscopy, polymeric woven structure, polyethyleneterephthalate.

### 1. INTRODUCTION

Dielectric spectroscopy is a non-invasive, sensitive and reliable experimental method, frequently used for the material characterization. By means of examining the temperature and frequency dependencies of dielectric properties of polymeric materials under the influence of periodic electric field, it is possible to observe their behavior under such conditions as well as their structural changes. Relative dielectric permeability of a material describes its polarization (dipole rotation and dipole distribution). Dielectric loss tangent represents energy losses occurring due to the motion or rotation of atoms or molecules within the dielectric. The importance to determine dielectric parameters of polymer woven structures used in the industry for filtration lies in the fact that dielectric parameters, besides electrical resistance, are indirect indicators of the tendency of fibrous materials to generate static charge. This phenomenon causes many disturbances, such as impairment of filtering properties of the material as well as the fire risk that can be caused by the spark appearance.

Dielectric relaxation spectroscopy is used to examine the dynamics of the bonds and the motion of molecular groups within polymeric materials under the influence of the electric periodic field in a wide range of temperatures and frequencies. Several

different relaxation mechanisms are observed in polymers under the influence of electric periodic field with varying temperatures [1–3]. Each relaxation can be attributed to specific molecular changes. Different peaks of the relaxation spectrum are usually marked by Greek letters  $\alpha$ ,  $\beta$ , and  $\gamma$  starting from higher temperatures or lower frequencies. Macromolecule polymer chains have a large number of internal degrees of movement freedom, caused by the rotation around single bonds. Above the glass transition temperature  $T_g$  in amorphous regions, chaotically ordered chains are susceptible to conformational changes, resulting in the coordinate cooperative motion of the long segments. Molecular mechanisms leading to the relaxation processes in the polymeric material below  $T_g$ , are classified into [4]: internal rotation of end groups in side chains, proton tunneling, methyl groups motion, molecular motion at defects regions and the effects due to the impurities or additives.

Polyethyleneterephthalate (PET) is a polymer with excellent mechanical, electrical and thermal properties, which make it one of the most commonly used polymers. PET contains polar groups such as low polarity ester groups [5], which causes low values of dielectric parameters. These characteristics make PET fibers suitable for electrical insulation. The oriented PET in the form of fibers has a fibrillar semicrystalline structure with a degree of crystallini-

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ty 0.5–0.6, and in the case of highly oriented fibers 0.7–0.8 [5]. Examining plastic deformation of polyester foils and fibers, Prevorsek (1973) [6] et al. have proposed a three-phased micro-fibrillar model of the fibrous structure. According to this model, the structure of fibers consists of highly ordered crystalline phase and non-crystalline areas of two types: one is amorphous and the other is an ordered "non-crystalline" phase. The molecular chains in the crystalline region contains only the *trans* – conformational elementary units, "non-crystalline" amorphous phase contains *trans* conformers outside the crystalline phase, while in the amorphous area *gauche* - conformations are observed [7,8]. Alves (2002) et al. [9] established, based on examinations using DSC method in the semi-crystalline PET, the existence of two amorphous phases with different conformational mobility (phase I and phase II). Phase I shows the glass transition in the same temperature range as amorphous PET and conformational rearrangement as in the purely amorphous phase. Phase II contains polymer chains between the crystalline lamellae making possible movements more restricted. The glass transition temperature in phase II has higher values than in phase I.

PET can be produced either in amorphous state or with a controlled amount of crystallinity, due to which it is used as an important model for studying relaxation processes in semicrystalline polymers. Molecular motions within the samples based on PET were monitored by means of recording relaxation transitions and were intensively examined using various techniques [10–21]. Based on these studies, two relaxation types were found:  $\beta$  – relaxation that occurs in amorphous fractions, and  $\alpha$  – relaxation that occurs due to segmental motions in the amorphous phase above the glass transition temperature.

The first studies of dielectric parameters of PET were published by Reddish (1950) [10]. In the frequency interval from  $10^2$  to  $10^7$  MHz, at temperatures range from  $-80$  °C to  $180$  °C, he recorded three dielectric transitions and analyzed them in the function of the sample crystallinity degree and absorbed water. The transition at  $100$  °C was related to dipole motion in the main polymer chain, while the other transition recorded at  $-50$  °C was related to thermal motion of  $-OH$  groups in amorphous phases. Reddish has noticed that a change in the value of dielectric parameters of the amorphous samples is more significant compared to the crystalline ones. He has established a connection between the sharp change of dependency at low frequencies and high temperatures, with the dc conductive effects i.e. with the conduction of charge through the material.

On the basis of the results of  $^1H$  and  $^{13}C$  Solid-state NMR on the various PET samples English (1984) [11] has identified four separate motion processes: rapid reorientation of polymer segments (angular oscillations of methylene and aromatic groups) in the crystalline region, a slower specific motion of the benzene rings (perhaps flipping) which are being connected with  $\beta$  relaxation, somewhat slower motions of methylene groups (*trans-gauche* isomerization) that correspond to  $\alpha$  relaxation, and the rearrangement of some polymer segments in the amorphous phase, which are temperature-dependent. Based on the conducted analysis, it is established that there is no difference between the results obtained for the powder, fibers and film of comparable crystallinity.

Maxwell (1998) and his associates have considered the origin of the relaxation process within the amorphous PET samples by comparing the results of NMR [15] as well as DMA and DRS [16]. The peak recorded within the temperature range from  $-120$  °C to  $80$  °C, at frequencies from 1 Hz to 10 kHz, was related to the local molecular movements that correspond to  $\beta$  – relaxation. Comparing the mechanical measurements that display all the relaxation processes in the polymer, with the dielectric measurements that show only movements of the polar groups, they examined the origin of this relaxation. It was found that the activation energy for the phenyl ring movement was higher than for the carbonyl group. By adding the additives of low molecular weight as the plasticizers, the peak obtained using DMA method at higher temperatures is suppressed, while the side at the lower temperatures remains unchanged. Based on obtained results, they have concluded that the side of the peak at lower temperatures is connected with non-cooperative motions of carbonyl dipole groups i.e. with carbonyl relaxation. The high temperature side of relaxation is attributed to the cooperative motions of nonpolar phenyl rings.

Using the FTSDC method, Neagu and Neagu (2006) [20] have recorded the spectrum within which they identified five different transitions that occur due to dipole movements and the space charges movements in semi-crystalline PET sample. They assumed that  $\gamma$  – relaxation at low temperatures occurs due to movements of hydroxyl end groups around 100 K, and the movements of carbonyl groups (C=O) in the amorphous phase around 150 K and in the crystalline phase around 210 K. The  $\beta$ -relaxation is connected with the local movement of phenyl rings around 240 K in the amorphous phase and around 290 K in the crystalline phase.

Polymer woven structures are non-homogenous materials, with a very complex structure, whose behavior in real conditions of use depends on numerous factors (basic polymeric substances, geometric and structural parameters as well as external influences). Polymer fabrics are fibrous structures that make up two-phase system: fiber-air [22–25]. Bal and Kothari (2009) [26] published a review of methods used so far to examine dielectric parameters of these structures. A large number of parameters as well as their complex interdependencies make it difficult to predict the behavior of these materials in use.

The results presented in this paper are a part of research of polymer fibrous materials, fabrics, nonwoven materials and meshes that have different raw composition and various constructive parameters [27–35].

The aim of this paper was to conduct a comparative analysis of dielectric parameters and structural characteristics of polymer woven fabrics and meshes, based on polyethyleneterephthalate, in a wide range of temperatures and frequencies, in order to obtain materials of adequate quality in terms of

dielectric properties. The paper compares samples, obtained from the same basic substance, designed to make the samples with different structural characteristics: thickness, fabric weight and yarn obtained from the filament and short fibers. The dielectric measurements are performed within the frequency interval from 90 kHz to 2.85 MHz during heating the samples within the temperature range from 120 K to 355 K.

## 2. EXPERIMENTAL MATERIALS AND DEVICES

### 2.1 Characteristics of used materials

The samples used in this study are filter materials based on polyethyleneterephthalate (PET) (density 1,38 g/mm<sup>3</sup>) which is used in the process industry as well as for air filtration (manufactured by SEFAR and KOFIL). The table displays the basic data about the woven filter materials and mesh filters, examined in this study.

Table 1. The basic data about the woven filter materials and mesh filters

	Material composition	Type of weave	Fabric thickness [μm]	Fabric weight [g·m <sup>-2</sup> ]	Air permeability [l·m <sup>-2</sup> ·s <sup>-1</sup> ]	Fibre material warp/weft
1	PET	twill	265	235	45	monofilament/ multifilament
2	PET	twill	500	440	100	multifilament/ multifilament
3	PET	twill	550	445	55	staple / staple
					Open Area [%]	
4	PET	mesh	1000	400	44.5	monofilament/ monofilament
5	PET	mesh	45	20	44.5	monofilament/ monofilament

### 2.2. Description of experimental devices

Dielectric measurements were performed at the Laboratory for Condensed Matter and Physics of Materials, Faculty of Physics, University of Belgrade. The capacitor cell is placed in the vacuum chamber with the cold head CTI Cryogenics refrigerating model 22 and with a heater controlled by the Lakeshore model 340 temperature controller. The measurements were performed by Agilent LRC Meter 4285A at the heating rate of 3 K·min<sup>-1</sup>, from 120 K to 355 K within the frequency interval from 90 kHz to 2.85 MHz. All measurements were carried out using 1V signal. The vacuum in the chamber was

measured and maintained at 10 Pa using a Pfeiffer turbo molecular pump DUO 5 M. The samples (13 mm in diameter) are placed in the capacitor cell, whereby the measurements are carried out perpendicular to the direction of the yarn in samples. Acquisition is carried out with specially programmed software for this measurement.

Based on the obtained values of the conductance ( $G$ ) and susceptance ( $B$ ), the dielectric loss tangent ( $\tan \delta$ ), the real part of ac specific electrical conductance ( $\sigma_{ac}$ ) and effective dielectric permeability ( $\epsilon'_m$ ), are determined according to the following formulas:

$$\tan \delta = \frac{G}{B}, \quad \sigma_{ac} = \frac{G \cdot l}{S} \quad \text{and} \quad \varepsilon'_m = \frac{B \cdot l}{2 \cdot \pi \cdot f \cdot \varepsilon_0 \cdot S}$$

where -  $B_0$  is the susceptance of an empty cell,  $l$  - distance between the electrodes and  $S$  sample surface. The measurement results of such polymeric fibrous structures show an overall effect of heterogeneous fiber-air system, so that;  $\varepsilon'_m$  represents the effective dielectric permeability.

### 3. RESULTS AND DISCUSSION

For five samples of filter materials, based on polyethyleneterephthalate (PET), with different structural characteristics, temperature–frequency dependence of dielectric loss tangent ( $\tan \delta$ ), is shown in Figures 1 and 2. Within the temperature range from 120 K to 355 K at the frequencies of 90 kHz and 2.85 MHz, a wide transition is noticed for the samples 1-4, that can be related to  $\beta$  - relaxation. Based on the research of PET samples, starting with the pure amorphous sample to the sample of different crystallinity degrees, by various methods [12–18], it is determined that  $\beta$  - relaxation is performed in the amorphous fractions below the glass transition temperature. Maxwell (1998) and his associates have researched the origin of  $\beta$  - relaxation in amorphous PET samples by comparing the results of NMR [15], DMA and DRS [16]. The low temperature side of the peak is related to non-cooperative motion of carbonyl dipole groups i.e. with a carbonyl relaxation in amorphous regions. They have attributed the high temperature side of the peak with the cooperative motions of non-polar phenyl rings. Based on 2D NMR, Wilhelm and Spiess (1996) [36], have detected a slow flip of phenyl rings in the ordered non-crystalline layers, which are not typical of the crystalline and amorphous regions. The values obtained for the samples 1-3 at the frequency of 90 KHz do not differ below the temperatures 250 K. It is observed that these three samples have different values, and the trend of frequency dependence on the high temperature side of the peak. That part is connected with the motions of phenyl rings occurring in the amorphous regions of higher arrangement. The wider peak, shifted to the higher temperatures, and recorded for samples 1 and 5, can be connected with a lower content of amorphous regions i.e. with higher structure arrangement of these samples in comparison with the others [12]. The activation energy of  $\beta$  - relaxation for samples 2-4 was calculated from the Arrhenius equation and shown in ref. [34].

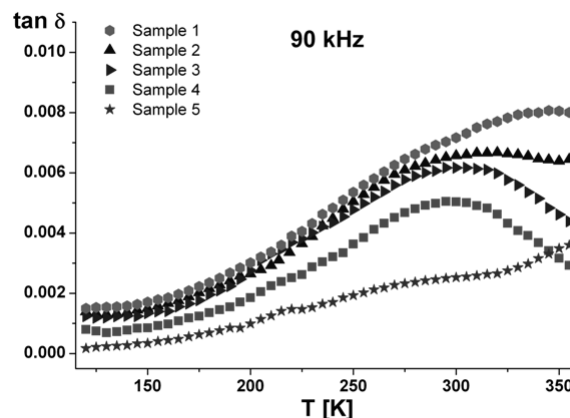


Figure 1. Temperature dependence of  $\tan \delta$  at frequency of 90 kHz

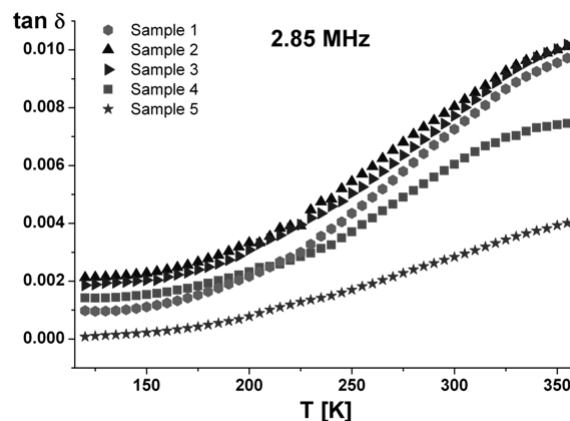


Figure 2. Temperature dependence  $\tan \delta$  at frequency of 2.85 MHz

In Figures 3 and 4,  $\tan \delta$  is shown as a function of temperature for samples 3 and 4 at various frequencies. It is observed that as the temperature increases, the recorded peak shifts towards higher frequencies [17,37], because thermal energy enables mobility of polar groups accompanying the changes of periodic electric field, which results in a decrease of relaxation time [38]. Also, the height of the peak increases with an increase of temperature and frequency, due to an increase in the number of dipoles involved in the relaxation process or due to the greater mobility caused by an increase in free volume [17]. The  $\beta$  - relaxation occurred at the temperatures around 300 K which indicates high crystallinity of the samples i.e. a high orientation of fibers. Highly oriented fibers contain a large number of crystallites whose direction coincides with the direction of fibers [1].

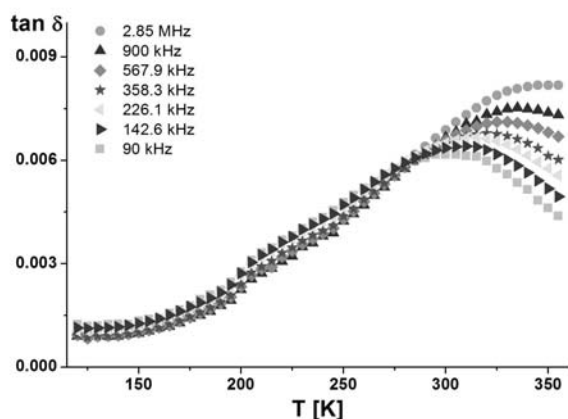


Figure 3. Temperature dependence of  $\tan \delta$  at the various frequencies for sample 3

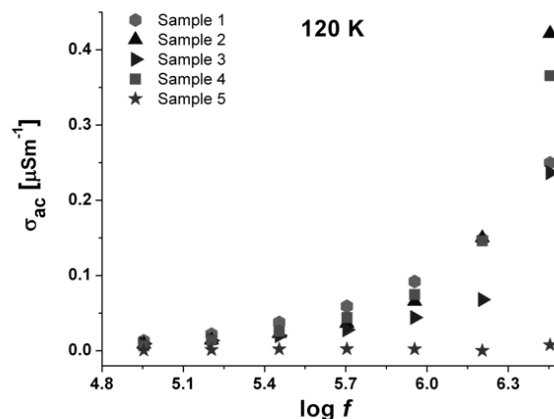


Figure 5. Frequency dependences of  $\sigma_{ac}$  at temperature 120 K

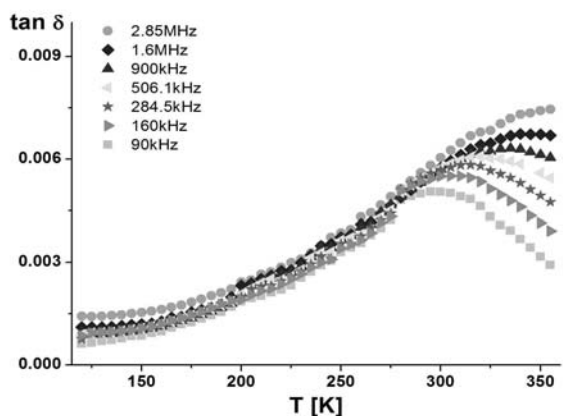


Figure 4. Temperature dependence of  $\tan \delta$  at the various frequencies for sample 4

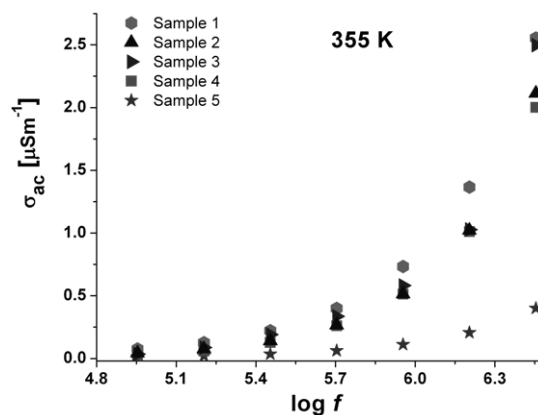


Figure 6. Frequency dependences of  $\sigma_{ac}$  at temperature 355 K

The measurement results of the electrical conductivity ( $\sigma_{ac}$ ) for the samples 1-5, in the frequency range from 90 kHz to 3 MHz, at temperatures of 120 K and 355 K, are shown in Figures 5 and 6. At lower frequencies, there is almost no change of  $\sigma_{ac}$  value [38], while as the frequencies increase, the electrical conductivity increases too [38, 39]. For the sample 5, the  $\sigma_{ac}$  value deviation is observed as well as the deviation of the trend of frequency dependence, in comparison to other samples. It is observed that electrical conductivity is lower at 120 K by about an order of magnitude, than at 355 K at almost all frequencies for all samples. As the conductivity in pure PET is a consequence of limited movements of charged impurities and electron tunneling through PET, we can say that the jump conductivity at higher temperatures is a result of the electrons release from the finite potential barriers along the PET chains as well as moving of the captured charges in non-homogenous areas at the microscopic level.

In Figures 7 and 8 frequency dependences of  $\sigma_{ac}$  are shown for the samples 1-5, at the temperature of 295 K. The measurements shown in Figure 7 are carried out in vacuum, whereas Figure 8 shows the results obtained under ambient conditions at relative air humidity of 35%. According to the manufacturer's specification, the moisture content of the fibers, used to obtain the examined filter materials at 20 °C and 65% relative humidity, is 0.4. At a lower level of relative humidity, the lower moisture sorption is expected. The absence of polar functional groups capable of forming hydrogen bonds with water molecules and the high packing density of molecules at a supermolecular level, form a spatial and energy barrier for the diffusion of the molecules that have low molecular weights in synthetic fibers. This causes low energy of the surface reactions with water and low capacity of synthetic fibers to sorb the substances with low molecular weight, primarily moisture [5]. A slight increase in the  $\sigma_{ac}$  value of the woven filter material sample is a result of the pres-

ence of polar water molecules in an empty space between the fibers at relative air humidity of 35%.

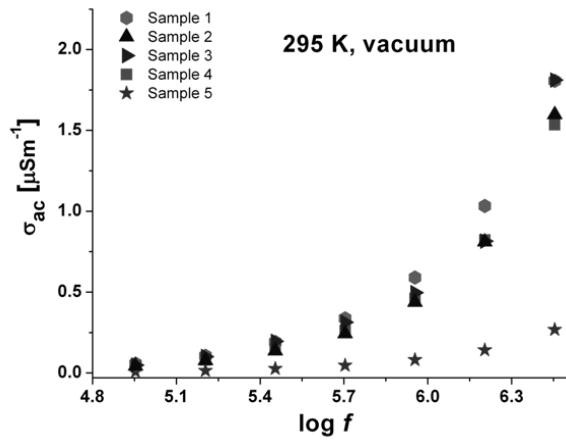


Figure 7. Frequency dependences of  $\sigma_{ac}$  at temperature 295 K, in vacuum

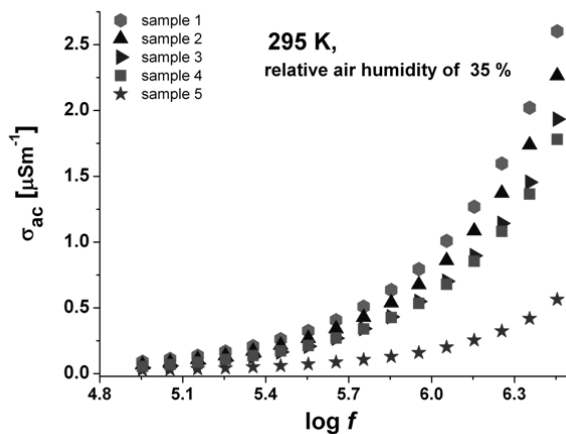


Figure 8. Frequency dependence of  $\sigma_{ac}$  at temperature 295 K and at relative air humidity of 35%

The frequency dependence of the effective dielectric permeability ( $\epsilon'_m$ ) recorded in vacuum at the temperature of 295 K, for the samples 1-5 obtained from PET, is shown in Figure 9. PET contains the polar groups such as ester groups, low polarity [5], which causes a low value of dielectric permeability. Sample 5 has lower thickness and fabric weight compared to other tested samples, so the lower  $\epsilon'_m$  values of that sample are expected. Low polarity of PET, low hygroscopicity, and low content of contaminants introduced during the synthesis causes low values of: electrical conductivity, dielectric loss tangent and effective dielectric permeability in a wide range of frequencies and temperatures.

Figure 9 displays a slight decrease of  $\epsilon'_m$  value as the frequency increases due to dispersion, [12,38–39]. As the frequencies increase, the material polarizability decreases as dipoles cannot follow the changes of external periodic field. Temperature dependence of  $\epsilon'_m$  at the frequency of 90 kHz is shown

in Figure 10. Temperature increase leads to an increase of  $\epsilon'_m$ , because thermal mobility of polymer groups and macromolecules parts increases the intensity of polarization [12,38].

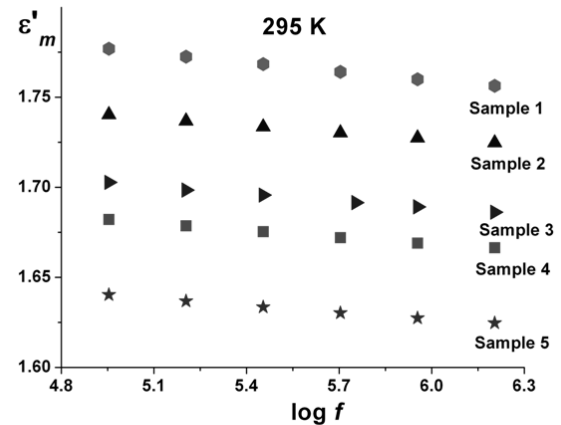


Figure 9. Frequency dependence of  $\epsilon'_m$  at temperature 295 K

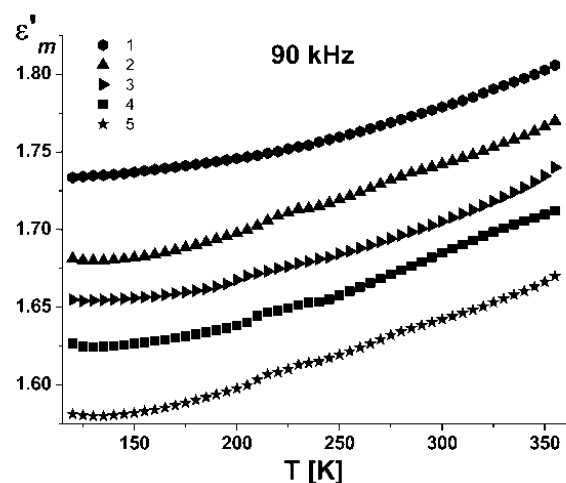


Figure 10. Temperature dependence of  $\epsilon'_m$  at frequency 90 kHz

As the woven filter materials form two-phase system fiber-air, the obtained values may be connected with the volume fraction of polymeric material in the samples. For sample 1, the volume fraction is 0.643, for sample 2, 0.638 and for sample 3 0.586 [34]. It can be seen that by reducing the volume fraction in the samples, a decrease of the  $\epsilon'_m$  value occurs.

The obtained effective permeability  $\epsilon'_m$  values of examined samples correspond with the results obtained for  $\tan \delta$  and  $\sigma_{ac}$ . The obtained data are analyzed in the light of structural characteristic of examined samples. Comparing the obtained results for the woven filter materials, it can be noticed that sample 3, whose yarns are obtained from short fibers, has lower values of dielectric parameters compared to samples 1 and 2, with filament yarn along

the weft and the warp. The obtained results for filter materials samples made of PET fibers showed that the woven filter materials had slightly higher values of dielectric parameters than the mesh samples 4 and 5.

#### 4. CONCLUSION

This paper presents a comparative analysis of dielectric and structural properties of the samples based on polyethyleneterephthalate. The commercial filter fabrics and meshes with various structural parameters (thicknesses, fabric weight, yarns obtained from filament and short fibers) were used in the analysis. Dielectric measurements were carried out in the frequency range from 90 kHz to 2.85 MHz, while the samples were heated at temperatures from 120 K to 355 K. Within this temperature and frequency range recorded  $\beta$ -relaxation is related to the motions in amorphous phases below the glass transition temperature. Comparing the results for the woven filter materials of polyethyleneterephthalate, it can be noticed that the samples whose yarns were obtained from short fibers have lower values of dielectric parameters than the samples with filament yarn along the weft and the warp. Based on the results obtained for the woven filters and the filter meshes of polyethyleneterephthalate it is observed that the values of dielectric parameters of the woven samples are higher. The measurements of dielectric parameters as a function of frequency and temperature enable to predict its behavior in real application conditions, making possible designing of the fabrics with desired properties. Determining the dielectric parameters of polymeric fibrous structures is very important in order to determine their tendency to generate static charges. The actuality of studying the dielectric parameters of woven polymeric structures at various outer influences is reflected in their more frequent use for e-textiles, electromagnetic protection and medical products.

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### КОМПАРАТИВНА АНАЛИЗА ДИЕЛЕКТРИЧНИХ И СТРУКТУРНИХ КАРАКТЕРИСТИКА УЗОРАКА НА БАЗИ ПОЛИЕТИЛЕНТЕРЕФТАЛАТА

**Сажетак:** Широка примјена полимерних материјала за израду предмета за свакодневну употребу до примјене у електротехници, електроници и медицини условљава њихово детаљно испитивање при различитим спољашњим утицајима. Диелектрична спектроскопија омогућава праћење промјена у структури полимерних материјала при различитим спољашњим утицајима. У овом раду анализирана је диелектрична  $\beta$  релаксација снимљена у полимерним тканим структурама и мрежама од полиетилентерефталата. Компарацијом добијених резултата установљена је веза између диелектричних параметара и структурних карактеристика узорака.

**Кључне ријечи:** диелектрична спектроскопија, полимерне ткане структуре, полиетилентерефталат.

