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DIELECTRIC CHARACTERISTICS OF POLYVINYL ALCOHOL FILMS

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Abstract. Dielectric characteristics of polyvinyl alcohol films obtained from aqueous solutions of the polymer have been investigated. The results of low-frequency (25 Hz – 1 MHz) and high-frequency (9.8 GHz) measurements are presented. The influence of filtration of aqueous solution of polyvinyl alcohol on dielectric parameters of the studied samples was found. The IR spectra of both types of films are identical and correspond to the literature data.

Keywords*: complex dielectric permittivity, electrical conductivity, IR spectroscopy, polyvinyl alcohol*

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1. INTRODUCTION

One of the promising directions in the search for new functional materials with different properties is the production of composite materials based on polymer matrix. The choice of polyvinyl alcohol (PVA) as the main polymer for various types of nanofillers is promoted by its biocompatibility, nontoxicity, biodegradability, film-forming ability, hydrophilicity, water solubility, chemical resistance, availability and ease of processing [1, 2]. The large number of hydroxyl groups in PVA prevents agglomeration with nanofillers and promotes their uniform dispersion in its matrix, and hence improves the properties of nanocomposites [2]. Due to such properties, PVA is chosen as a base matrix for fillers in various applications [1, 3–6].

The reduction and elimination of electromagnetic radiation (EMI) pollution and new wireless telecommunication standards require a variety of affordable, lightweight, EMI shielding and radio-absorbing composite materials. Carbon nanoscale structures used as fillers in polymer composites allow the development of new approaches to create new materials with different properties and therefore different practical applications while reducing the amount of fillers and reducing the weight of composite materials. The combination of the above-mentioned properties of polyvinyl alcohol and carbon nanoscale structures allows us to consider composites based on them as promising materials absorbing EMI.

The present work is devoted to the study of electrophysical properties of PVA polymer films for their possible use as a basis for composite materials containing carbon nanoscale structures.

2. EXPERIMENT

Commercial polyvinyl alcohol grade 098–15(G) (Sinopec, China) with a mass fraction of the main substance $(CH_2CHOH)_n$ of at least 95.3%, density of 1.19–1.31 g/cm^3, melting point of 220–230 °C, degree of hydrolysis of 98.6%, and decomposition temperature of 160–200 °C without prior purification was used as a polymer material for film preparation. To obtain a **1-nF** film sample, 6 g of PVA was dissolved in 95 mL of water at 80–93 °C for several days. Then 6 g of the solution was placed in a 2.4*7.1 cm^2 Teflon container and dried for 1 day at (2–3)*10^{-2} mmHg pressure and room temperature. Then, the aqueous solution of PVA was filtered with No. 2 filter paper to free the insoluble part. The **2-F** film sample was made similarly from 10 mL of the resulting filtered solution. Freshly prepared films were used in the experimental measurements. The films were

0.025 (**1-nF**) and 0.013 (**2-F**) cm thick with respective densities of 0.95 and 0.55 g/cm^3.

The IR spectra of PVA films were recorded on a FT-02 FT-IR spectrometer (Lumex, Russia) in the wave number region 450–3500 cm^{-1} with a resolution of 4 cm^{-1}. In low-frequency measurements, the capacitance C and resistance R of PVA films were determined in a two-electrode system with a diameter of brass electrodes of 5 mm at a pressure of 2 MPa in the frequency range of 25 Hz-1 MHz using an E7–20 immittance meter (Russia). To correctly determine the values of dielectric permittivity and low-frequency conductivity (σ_{LF}), which depend on the sample inhomogeneity, these measurements were carried out in different parts of the sample and these values were averaged. The complex dielectric permittivity of the samples (CDP) at high frequency (9.8 GHz) was measured by the resonator method, for this purpose the samples were placed in glass ampoules with an inner diameter of 2–2.5 mm. The obtained values of dielectric permittivity (ε'), dielectric loss (ε ") and tangent angle of dielectric loss (tanδ) were averaged over several measurements. All measurements were performed at room temperature, and their accuracy was 10 and 20% for ε' and ε", respectively. The above methods are described in [7–10].

3. DISCUSSION OF RESULTS

The frequency dependence of dielectric characteristics of materials is described by the complex dielectric permittivity $\varepsilon(\omega) = \varepsilon'(\omega) - j\varepsilon''(\omega)$, where $\omega = 2\pi f$, f is the frequency of the applied electromagnetic field, ε'(ω) and ε''(ω) are the real and imaginary parts of the CDP characterizing dielectric polarization and losses in the dielectric, with dielectric loss angle tangent $\tan\delta = \varepsilon''/\varepsilon'$. The measured values of capacitance C and the tanδ dielectric loss angle tangent were used to calculate the low-frequency dielectric parameters. The values of ε' were determined as $\varepsilon' = Cd/S\varepsilon_0$, where d is the thickness of the sample, m, S is the area of the electrode, m^2, $\varepsilon_0 = 8.85{*}10^{-12}$ F/m is the dielectric constant of free space. The values ε" were calculated by the relation $\varepsilon'' = \varepsilon'\tan\delta$. In case of high-frequency measurements tgδ was determined from the measured values ε' and ε". The low-frequency and high-frequency conductivities (σ_{LF} and σ_{HF}) were calculated by the ratio $\sigma = \varepsilon_0 w \varepsilon''$.

Taking into account the technological process of PVA production, in which polyvinyl acetate (PVAc) is the main material, it is possible to analyze the experimental results, considering the produced PVA films as a system of PVA+PVAc+water, each component of which can contribute to their dielectric characteristics. Since polyvinyl alcohol is a hygroscopic polymer with OH-group, its properties can be influenced by many factors such as the method of its preparation, molecular weight distribution, branching, degree of hydrolysis, presence of residual acetate groups, and water [11–16]. These factors can lead to structural inhomogeneities of the films.

The measured IR spectra of PVA films (Fig. 1) contain characteristic absorption bands attributed to stretching vibrations of C-O bonds at 1050–1140 cm^{-1} [17], C-H/CH$_2$ at 2880–2960 cm^{-1}[1, 4, 17–19] and O-H at 3100–3500 cm^{-1} [1, 4, 17–20], and to bending vibrations of O-H bonds at 510–740 cm^{-1} [18] and CH$_2$ at 1320–1470 cm^{-1}[19]. Absorptions at 850 cm^{-1} and 918 cm^{-1} are attributed to vibrations of the carbon framework of PVA [4, 17]. At 1235 cm^{-1}, a weak absorption corresponding to C-H bond vibrations is observed [4, 17], and the absorption at 1660 cm^{-1} is attributed to C=O stretching vibrations of the carbonyl group of PVA [4].

The measured low-frequency parameters of the PVA films are presented in Fig. 2. For specificity, Table 1 summarizes these values for both films at frequencies of 60 Hz, 10 kHz and 1 MHz.

Table 1. Low-frequency electrophysical characteristics of polyvinyl alcohol films

Sample	f, Hz	ε'	ε"	tanδ	σ_{LF}, S/m
1-nF	60	4,99	0,35	0,07	$1{,}33{*}10^{-9}$
	10^4	4,15	0,22	0,05	$1{,}16{*}10^{-7}$
	10^6	3,40	0,28	0,08	$1{,}50{*}10^{-5}$
2-F	60	3,09	0,17	0,06	$7{,}11{*}10^{-10}$
	10^4	2,67	0,12	0,05	$6{,}67{*}10^{-8}$
	10^6	2,57	0,20	0,08	$1{,}08{*}10^{-5}$

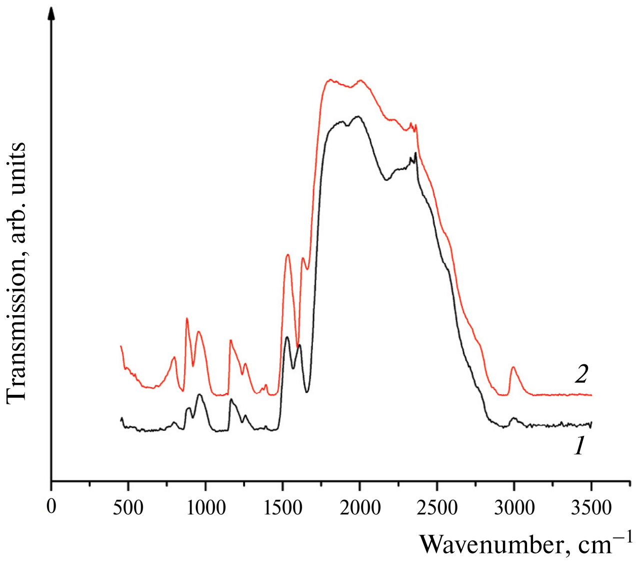

Fig. 1. IR spectra of films made from unfiltered (**1**) and filtered (**2**) aqueous solutions of polyvinyl alcohol. 1-film **1-nF**, 2-film **2-F**.

Table 2. High-frequency electrophysical characteristics of polyvinyl alcohol films at 9.8 GHz

Sample	ε'	ε"	tanδ	σ_{HF}, S/m
1-nF	2,97	1,62	0,52	0,89
2-F	2,97	2,09	0,75	1,16

The decrease of the low-frequency values of the real part of the dielectric constant ε' (Fig. 2a) and increase of σ_{LF} values (Fig. 2d) with frequency is typical for many polymers, and in the literature it is attributed mainly to dipole polarization [1, 21]. The behavior of ε" seems to reflect the structural heterogeneity of the studied PVA polymer films and the uncontrolled water content.

Filtration of aqueous polyvinyl alcohol solution resulted in changes in the low-frequency and high-frequency CDP values of PVA films. The low-frequency values of ε', ε", tanδ and σ_{LF} decreased while maintaining the trend of their frequency dependence (Fig. 2, Table 1). It can be assumed

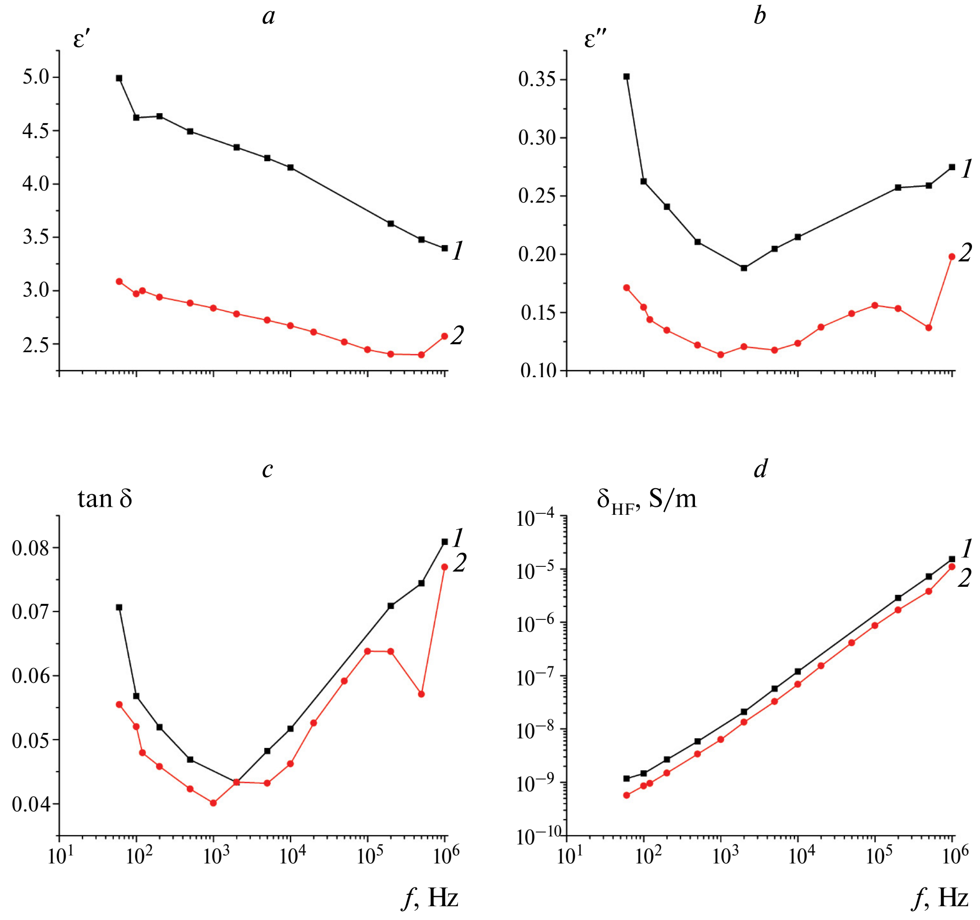

Fig. 2. Effect of filtration of aqueous polyvinyl alcohol solution on the low-frequency complex dielectric permittivities and conductivities (ε' (a), ε" (b), tanδ (c), σ_{LF} (d)) of **1-nF** (1) and **2F** (2) PVA films

that the **2-F** sample obtained from the filtered PVA solution is more homogeneous in composition than the **1-nF** sample obtained from the original solution, it may have fewer uncontrolled impurities, which is reflected in the low-frequency characteristics. Table 2 summarizes the high-frequency dielectric characteristics. The increase in the high-frequency dielectric loss values of the PVA film prepared from filtered aqueous PVA solution can be attributed to the more porous structure of the **2-F** film, as indicated by its lower density. The decrease in density seems to be due to the increase in free volume [16], i.e., the air-bound water ratio has changed.

The low-frequency and high-frequency values of ε' and ε" presented in the present work, which exceed the similar values for PVA powder [14], indicate the role of water contained in the studied PVA films. Adsorbed water affects the dielectric and physical and mechanical properties of polymer composites [13, 15, 16, 21–23]. The low-temperature regime for obtaining the studied PVA films (<100 °C) promotes water retention due to its interaction with polymer molecules. Evaporation of free water occurs at temperatures up to 200 °C, and removal of chemically bound water occurs in the range 230–400 °C [19]. In the investigated films, the values of ε' (Fig. 2a) are much lower than those for free water [24] and this may be due to the influence of adsorbed water. The binding of water and PVA molecules in the aqueous solution, the subsequent attachment of water molecules to the groups of the polymer molecule of PVA may limit their motion and increase the relaxation time compared to free water molecules [14]. Comparison of the frequency characteristics of the dielectric permittivity of water in different states (solid, adsorbed, liquid) revealed a shift of the frequency dispersion of the dielectric permittivity to the region of lower frequencies and a significant increase in the dielectric relaxation time of adsorbed water compared to water in the liquid phase [24].

Dielectric characteristics of PVA films allow us to consider the possibility of their use as a basis for polymer composite materials containing carbon nanoscale fillers that absorb electromagnetic radiation [8, 10].

4. CONCLUSION

Low-frequency (25 Hz-1 MHz) and high-frequency (9.8 GHz) measurements of complex dielectric permittivity and electrical conductivity of polymer films of polyvinyl alcohol obtained from aqueous solutions of the polymer have been carried out. The influence of filtration of aqueous solution of polyvinyl alcohol on the electrophysical parameters of the films obtained from it was demonstrated. The influence of filtration on the IR spectra of the films was not found and they correspond to the literature data. Polyvinyl alcohol can be considered as a basis for polymer composite materials with carbon nanostructured fillers that absorb electromagnetic radiation.

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