

Effect of biochar binding on dielectric properties of color catcher sheets

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Abstract. The dielectric properties in the frequency range of 10^2 to 10^6 Hz and at the temperatures of 30 to 60 °C of 0.4 mm thick color catcher sheets (nonwoven textile with ion exchange properties) both in the native state and with bound biochar were investigated using the oscilloscope method. The sample dimensions were 1×1 cm. To assess the influence of the sample thickness, samples with several (maximum 4) layers were used together with one-layer textile. It was shown that, unlike the data obtained by us earlier, the dielectric properties of the nonwoven textile without additives are caused by near-electrode processes. It was demonstrated as well that in this case, the dispersion of the frequency dependences of inverse resistance (analog of the imaginary component of complex dielectric permittivity) with respect to capacitance (analog of the real component of complex dielectric permittivity) corresponds to the Debye dispersion. Using the obtained results, the dielectric relaxation time ($2.4 \cdot 10^{-5}$ s) and the thickness of the near-electrode layer (1.5 μm) were estimated. Measurements at different temperatures and with several layers of the native textile demonstrated that the parameters of this relaxation process do not depend on both the sample thickness and the temperature. It was found that the sample resistance decreased by 3 orders of magnitude on average in the presence of biochar bound to the textile. In this case, the temperature dependence of the inverse resistance (analog of conductivity for uniform and continuous bodies) obeyed the Arrhenius law. The activation energy of the temperature dependence of the inverse resistance was 0.37 eV, which is greater than the similar value obtained in our work of 2024.

Keywords: color catcher sheets, nonwoven textile, dielectric properties, near-electrode processes, flexible systems, deformation.

<https://doi.org/10.15407/spqeo28.03.322>

PACS 68.37.Hk, 77.22.Ch, 77.22.-d

Manuscript received 06.06.25; revised version received 26.06.25; accepted for publication 03.09.25; published online 24.09.25.

1. Introduction

It was shown in [1] that specific types of color catcher sheets (CCS), which are regularly used during washing to prevent color runs, could be an interesting object for research. The CCS, which usually represent nonwoven textile with ion exchange properties, have found

interesting applications in (bio)analytical chemistry. Both native and modified sheets can be used as low-cost, planar optical sensors for analyzing various dyes and as a carrier for immobilizing specific affinity ligands. Changes of the sensor color can be observed by spectrophotometry or using an image analysis [2]. Recently, CCS were also employed for interaction with

inorganic particles. The CCS with bound montmorillonite derivatives [3] or Prussian blue particles [4] enabled efficient phenol polymerization or methylene blue decolorization due to peroxidase-like activity of the bound particulate catalysts. Moreover, other methods, including dielectric spectroscopy, can be efficiently employed to study the effect of the bound particles on CCS [5–18]. Therefore, the purpose of this work was to study the dielectric properties of a specific color catcher (which is a typical nonwoven textile material [2]) in the native state and after binding biochar particles, using a different method as compared to [1], and to compare the color catcher characteristics with the respective data obtained in [1].

2. Instruments and materials

Color catcher sheets (Iberia Protect, AC Marca Brands, Spain) were employed as a model nonwoven textile. Biochar-4073 (BCH-4073) produced by Biouhel.cz (Czech Republic) was prepared by pyrolysis of soft wood at 750 °C for 40 min. The biochar was ground using a knife laboratory mixer (Microtron Kinematika 550, Kinematica GmbH, Germany) before use. Then the samples were sieved using 100 µm sieves. A fraction of the biochar below 100 µm was used for modifying the CCS squares (1×1 cm in size) by immersing them in excess of BCH suspension in methanol (10 mg/mL) overnight under mixing. The modified CCS squares with the bound BCH were subsequently dried at room temperature.

The dielectric characteristics of the samples in the frequency range of 10 to 7·10⁶ Hz and at the temperatures of 30 to 60 °C were measured using the oscilloscope method [19]. For the studies, we used sandwich-type samples with a guard electrode. The area of the measuring electrode was 1 cm².

All the studied samples had the same geometric shape (a rectangular parallelepiped) with a square base having the side length of 1 cm and the height of 0.4 mm. To check the thickness dependence, the samples with several layers of textile (maximum 4 layers) were prepared.

To conduct experimental studies similar to [1], a G3-112 generator was used. A time-varying sinusoidal voltage with the amplitude of 4 V was supplied from the generator to the sample. The sample was connected in series with a resistor magazine, which served as a load resistance for the S1-93 oscilloscope. The voltage from the load resistance was supplied to the Y coordinate of the oscilloscope. The voltage was supplied directly from the generator to the X coordinate. For most frequencies, the oscillograms at voltage rises and falls differed (the oscillograms had a shape close to an ellipse). Specifically, the voltage on the load resistance during the rise of the signal from the generator U_n was greater than that the voltage during the fall U_s . Using the analysis conducted in [1], the resistance and capacitance values were calculated based on the obtained oscillograms using the following expressions.

The sample resistance was calculated as

$$R = 2R_n \frac{U_x}{U_n + U_s}, \quad (1)$$

where R_n is the value of the load resistance and U_x is the voltage, at which the capacitance and resistance were calculated, respectively.

The sample capacitance was calculated by the following expression:

$$C = \frac{U_n - U_s}{4\pi f \sqrt{U_0^2 - U_x^2}}, \quad (2)$$

where f is the frequency of the measuring signal and U_0 is the amplitude value of the measuring signal voltage, respectively.

In our research, $U_0 = 4$ V and $U_x = 2.4$ V.

The sample temperature was maintained using a thermostat and stabilization unit developed by us. The deviation from the set temperature value did not exceed 0.1 °C.

3. Experimental results and analysis

3.1. Dielectric properties of native nonwoven textile

Dielectric properties of uniform in bulk substances are characterized based on the frequency dependences of the components of the dielectric permittivity ϵ' and ϵ'' . Similar to [1], the materials studied in the present work were not continuous. Therefore, their dielectric properties could not be characterized based on the frequency dependences of ϵ' and ϵ'' .

In this work (except for individual studies), we studied the samples of the same geometric dimensions.

The components of the complex dielectric permittivity ϵ' and ϵ'' have the following relations to the sample capacitance and resistance [20]:

$$\epsilon' = \frac{Cd}{\epsilon_0 S} \quad (3)$$

and

$$\epsilon'' = \frac{d}{2\pi\epsilon_0 fSR} \quad (4)$$

where d is the sample thickness, S is its area, and f is the frequency of the measuring signal, respectively.

For textiles with non-continuous and non-uniform properties (in presence of additives), analysis of their dielectric properties can be carried out by considering the experimentally measured resistance and capacitance values.

At the initial stage, it was important to find out how the values of R and C of the native textile without additives would depend on the frequency (as was done in [1] or in a different way).

Fig. 1 shows a frequency dependence of R for one layer of native textile in the frequency range of 10² to 10⁶ Hz at the temperature of 30 °C on a bilogarithmic scale.

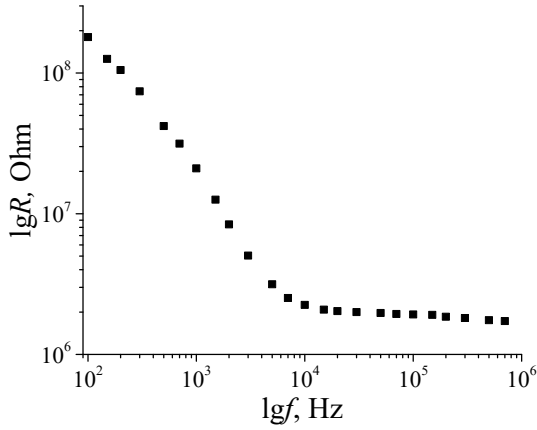


Fig. 1. Frequency dependence of the resistance of 0.4 mm thick one layer of nonwoven textile at the temperature of 30 °C on a bilogarithmic scale.

As follows from the analysis, the data for the textiles studied in this work significantly differ from the data obtained in the work [1]. Similar in nature dependences were obtained for other sample temperatures. Moreover, it was found that the temperature value (in the range of 30 to 60 °C) did not affect the resistance value.

Fig. 2 shows the frequency dependence of the capacitance for one layer of native nonwoven textile in the frequency range of 10^2 to 10^6 Hz at the temperature of 30 °C. It can be concluded based on the analysis of the obtained results that in addition to the significant frequency dependence of the resistance in the studied frequency range, the frequency dependence of the capacitance also significantly differ from the respective data reported in [1].

In dielectric spectroscopy, the type of the frequency dependence of ε' and ε'' is determined based on the analysis of the dependences $\varepsilon''(\varepsilon')$, *i.e.* the Cole–Cole diagrams. For the same geometric dimensions of the non-uniform samples, the equivalent of ε' is the capacitance C , and the equivalent of ε'' is $1/\omega R$ (here, $\omega = 2\pi f$ is the cyclic frequency).

Fig. 3 shows the dependence $1/\omega R(C)$ for nonwoven textile at the temperature of 30 °C calculated using the data presented in Figs. 1 and 2. As can be seen from Fig. 3, the main part of this dependence can be described by a semicircle.

For uniform and continuous bodies, this type of dispersion corresponds to the Debye dispersion with one relaxation time and is described by the following relation [20]:

$$\varepsilon^* = \varepsilon_s + \frac{\varepsilon_s - \varepsilon_\infty}{1 + i\omega\tau}, \quad (5)$$

where ε^* is the complex permittivity, ε_s and ε_∞ are the permittivity values at the frequencies $f=0$ and $f=\infty$, respectively, and τ is the dielectric relaxation time.

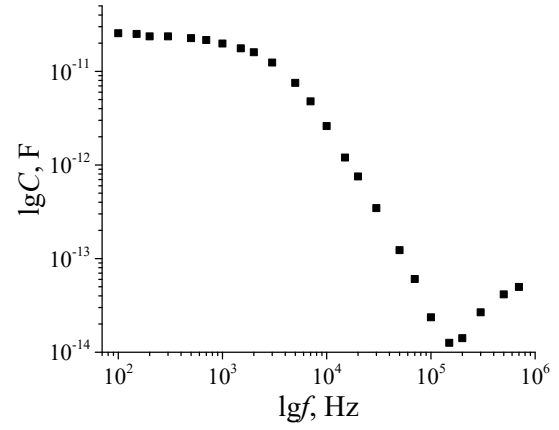


Fig. 2. Frequency dependence of the capacitance of 0.4 mm thick one layer of native nonwoven textile at the temperature of 30 °C on a bilogarithmic scale.

We believe that for non-uniform and non-continuous bodies (such as the textile under study), analysis of the obtained data may be carried out using the following relation:

$$C^* = C_s + \frac{C_s - C_\infty}{1 + i\omega\tau}, \quad (6)$$

where C^* is the complex capacitance and C_s and C_∞ are the capacitance values at the frequencies $f=0$ and $f=\infty$, respectively.

Calculations by the expression (6) provided $\tau = 2.4 \cdot 10^{-5}$ s.

The values of the samples capacitance in the studied range (10^2 – 10^6 Hz) obtained by us significantly exceed the capacitance of the samples at a uniform distribution of an electric field in the textile. It may be assumed therefore that the electric field in the textile is distributed non-uniformly, similar to, *e.g.*, a liquid crystal case. Namely, the electric field is mainly distributed within the near-electrode areas. Based on this assumption, the thickness of the near-electrode areas, where the electric field is predominantly distributed, can be estimated.

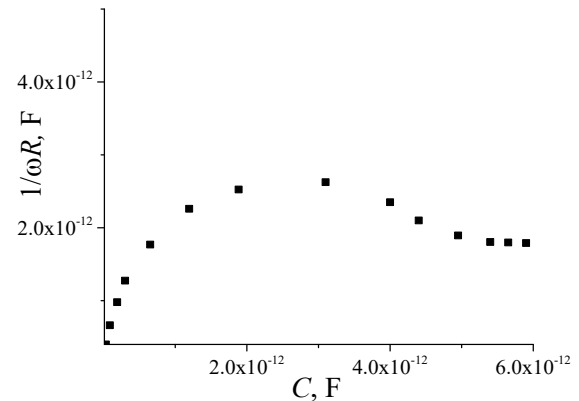


Fig. 3. $1/\omega R(C)$ calculated using the data presented in Figs. 1 and 2.

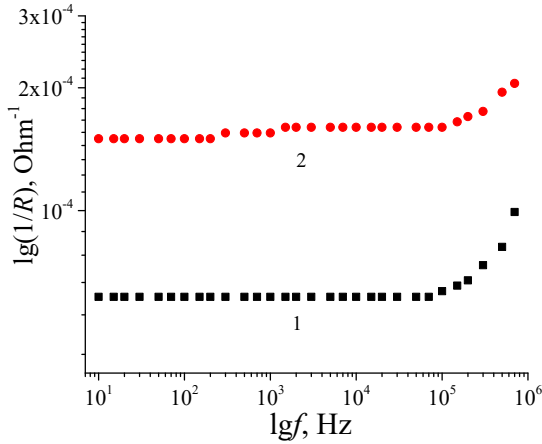


Fig. 4. Frequency dependences of the inverse resistance $1/R$ for one layer of nonwoven textile with bound biochar at 30 °C (1) and 60 °C (2).

For liquids and in particular liquid crystals, we obtained the following expression to estimate the thickness of the near-electrode area W [21]:

$$W = \frac{d\varepsilon_{\infty}}{2\varepsilon_s}. \quad (7)$$

For the studied textiles, the expression (7) transforms into

$$W = \frac{dC_{\infty}}{2C_s}. \quad (8)$$

Calculations by the expression (8) provided $W = 1.5 \mu\text{m}$.

3.2. Dielectric properties of nonwoven textile with bound biochar

Fig. 4 shows frequency dependences of the inverse resistance (analog of conductivity for a uniform and continuous medium) for one layer of textile with bound biochar at the temperatures of 30 °C (curve 1) and 60 °C (curve 2).

It can be concluded from the data presented in Fig. 4 that such dependences significantly differ from those for the textile without additives. First, the resistance of the textile is significantly reduced. Taking into account that the resistance of the textile without additive depends on the frequency, one may assume that introduction of the additive into the textile leads to a decrease in the R value by approximately 1000 times on average.

Second, unlike the native textile (without bound biochar) case, the resistance of the textile with bound biochar is practically independent of frequency (except for a small part at $f > 10^5$ Hz).

Third, the dependence of the resistance of the textile with bound biochar changes with temperature. Such temperature dependences for different textile thicknesses

due to use of several layers are shown in Fig. 5. In this figure, the dependences of $1/R$ (analog of electrical conductivity for uniform and homogeneous media) on temperature are presented in Arrhenius coordinates. The value of $1/R$ increases with the temperature, which is characteristic of the mechanism of conductivity in semiconductors.

The temperature dependence of the electrical conductivity of the nonwoven textile with bound biochar can be described by the following expression:

$$1/R = b \exp\left(-\frac{\Delta E}{kT}\right), \quad (9)$$

where b is a constant, $k = 1.38 \cdot 10^{-23}$ J/K is the Boltzmann constant, and T is the absolute temperature, respectively.

The most important parameter included in the expression (6) is the activation energy of the value $1/R$ (analog to conductivity for uniform bodies). The same slope of the straight lines in Fig. 5 indicates that the activation energy of $1/R$ does not depend on the number of layers. Fitting the experimental results presented in Fig. 5 with the expressions (9), the activation energy of the temperature dependence of $1/R$ was found to be 0.37 eV.

This value of the activation energy of $1/R$ is greater than that obtained in [1] (0.13 eV), where the electrical properties of a different type of CCS nonwoven textile with the same biochar additive were studied. This confirms once again the conclusions made earlier that, unlike the textile is made using threads, the properties of the nonwoven materials can be changed much more by introducing various types of additives. Hence, even minor modifications in the textile manufacturing technology and the properties of the additives may significantly impact the properties of the nonwoven material.

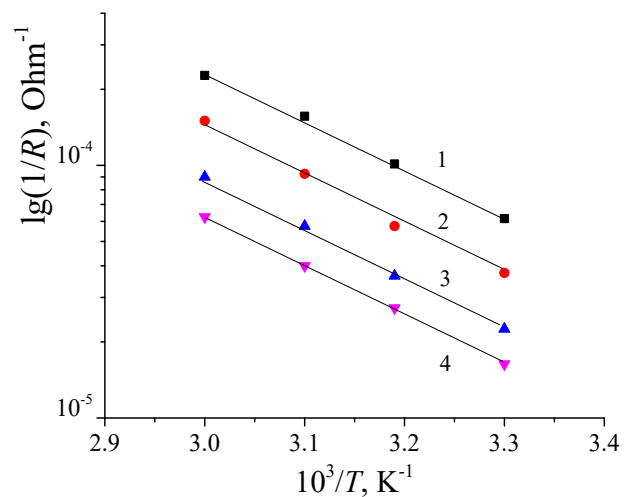


Fig. 5. Temperature dependences of $1/R$ of textile with bound biochar with different numbers of layers: 1 (1), 2 (2), 3 (3), and 4 (4). The thickness of one layer is $0.4 \mu\text{m}$. The sample area is 1 cm^2 .

4. Conclusions

1. It has been shown that a change in the technology of manufacturing nonwoven textile can significantly affect its electrophysical properties. While for the nonwoven textile studied in [1], the charge carrier transport was jump-like in the sample bulk, in the same-type materials but with slightly different characteristics, studied in this work, the charge carrier transport was significantly influenced by near-electrode phenomena. The influence of the near-electrode processes in the nonwoven textile without additives leads to that the electric field is mainly redistributed in the near-electrode layers.

2. Analysis of the dielectric properties of the studied textiles, which are non-uniform media, was carried out considering the frequency dependences of the resistance R and capacitance C as well as the dependence $1/\omega R(C)$ (analog to the Cole–Cole diagrams for uniform and single-component media).

3. It has been shown that the Cole–Cole diagrams for the studied textile without additives have a semicircle shape, which is characteristic of the Debye dispersion with a single relaxation time. The relaxation time ($2.4 \cdot 10^{-5}$ s) and the thickness of the near-electrode layer, in which the relaxation process occurs (1.5 μm), have been estimated.

4. It has been shown that the characteristics of the near-electrode processes do not depend on the temperature in the range of 30 °C to 60 °C and the sample thickness (0.4 to 1.6 mm, i.e. 1 to 4 textile layers).

5. In the presence of biochar bound to the textile, the conductivity of the latter increases by three orders of magnitude on average and does not depend on the frequency except for the frequencies above 10^5 Hz.

6. It has been shown that the temperature dependence of $1/R$ (analog of conductivity for non-uniform media) corresponds to the Arrhenius law in the frequency range where it does not depend on the value of f .

7. It has been found that the activation energy of the temperature dependence of the conductivity of the studied samples does not depend on the number of textile layers and equals as 0.37 eV. This value is significantly higher than the respective value obtained in [1].

Acknowledgements

This research was supported by the projects: of the Slovak Research and Development Agency No. APVV-22-0060 MAMOTEX, VEGA 2/0028/25 and REA.A – Marie Skłodowska-Curie Actions & Support to Experts within the project 101182948 – RETROTRAFO – HORIZON-MSCA-2023-SE-01.

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Volokh L.V.: investigation.

Oleinikova I.V.: investigation.

Mariano J.: investigation.

Safarik I.: textile materials preparation and characterization, conceptualization, methodology, data curation, writing – original draft, writing – review & editing.

Kopčanský P.: resources, investigation, visualization, writing – review & editing.

Вплив додавання біовугілля на діелектричні властивості кольоропоглинальних листів

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Анотація. У діапазоні частот 10^2 – 10^6 Гц та температур 30–60 °C за допомогою осцилографічного методу досліджено діелектричні властивості кольоропоглинальних листів (нетканого текстилю з іонообмінними властивостями) товщиною 0,4 мм, як у вихідному стані, так і з доданим біовугіллям. Розміри зразків становили 1×1 см. Для оцінки впливу товщини зразка, крім одного шару текстилю, використовувалися зразки з кількома шарами (максимальна кількість – 4 шари). Показано, що, на відміну від даних, отриманих нами раніше, діелектричні властивості нетканого текстилю без добавок зумовлені приелектродними процесами. Показано, що в цьому випадку дисперсія частотних залежностей величини, оберненої до опору (аналог уявної складової комплексної діелектричної проникності), відносно ємності (аналог дійсної складової комплексної діелектричної проникності) відповідає дисперсії Дебая. Використовуючи отримані дані, було оцінено час діелектричної релаксації ($2,4 \cdot 10^{-5}$ с) та товщину приелектродного шару (1,5 мкм). При вимірюваннях за різних температур та кількох шарів вихідного текстилю було показано, що параметри цього процесу релаксації не залежать ні від товщини зразка, ні від температури. Було виявлено, що за наявності біовугілля, доданого до текстиля, опір зразків зменшується в середньому на 3 порядки. Було показано, що в цьому випадку температурна залежність значення, оберненого до опору (аналог провідності для однорідних та суцільних тіл), відповідає закону Арреніуса. Було виявлено, що енергія активації температурної залежності значення, оберненого до опору, становить 0,37 еВ, що більше за аналогічне значення, отримане в роботі, опублікованій у 2024 році.

Ключові слова: кольоропоглинальні листи, нетканий текстиль, діелектричні властивості, приелектродні процеси, гнучкі системи, деформація.