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ANTIBACTERIAL AND DIELECTRIC PROPERTIES OF TEXTILE MATERIALS MODIFIED WITH HERBAL EXTRACT OF *Picea omorika* AND THE COPPER FERRITE NANOPARTICLES

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Abstract: In this paper, cotton, polyester and cotton/polyester fabrics were modified by using herbal extract of *Picea omorika* and copper ferrite nanoparticles and their antibacterial and dielectric properties were investigated. Antibacterial activities of all samples were examined against *Escherichia coli* and *Staphylococcus aureus*. Most of the fabrics modified by copper ferrite showed antibacterial activities against *Escherichia coli*, while the addition of the herbal extract improved their antibacterial protection. Dielectric properties were measured in frequency range from 24 Hz to 75 kHz at room temperature and the results showed that the modification of all three fabrics with copper ferrite caused increase in their electrical conductivity. The obtained results point to the possibility of using investigated fabrics for antibacterial protection as well as for the electromagnetic shielding application.

Keywords: Antibacterial properties, Dielectric properties, Fabrics, Herbal extract of *Picea omorika*, Copper ferrite nanoparticles.

1. INTRODUCTION

In recent, textile materials dyed with herbal extract are called herbal textile. Herbal textile having special properties helps in providing protection against various kinds of allergies caused by synthetic colors. This dyeing process is ecologically clean technology and plant extracts used for this process are obtained from natural renewable resources. Using scientific and engineering knowledge in the field treatment of materials with herbal extracts enables the development of new textile materials with advanced properties, such as antimicrobial activity [1-3].

For the textile dyeing, dye solutions that provide a good degree of coloration, antimicrobial and deodorizing properties and UV protection were commonly used. The color of herbal extracts and their antimicrobial effect come from substances with different chemical compositions (anthocyanins, anthraquinones, flavonoids, etc.) [4, 5]. Due to the fact that the bioactive substances from plants may slow or prevent the growth of microorganisms, there is a growing interest to study their application in textile processing [6-10]. In order to enhance fastness of the antibacterial activities and coloration of textile materials modified with extracts of medicinal plants during the processing, various mordants are added, such as Glauber's salt [11], $KNaC_4H_4O_6 \cdot 4H_2O$ [12], $CuSO_4$ and $FeSO_4$ [13].

Ferrites are an important class of inorganic magnetic nanoparticles, with good optical, electrical and magnetic properties, also they have low toxicity, good catalytic and antibacterial properties [14–17]. In recent times, nanosized copper ferrite has attracted particular attention because of its large dielectric permeability at high frequency, high electric resistivity, it is easy to synthesize, excellent chemical stability and low cost [15].

By modification textile materials with ferrites, nanocomosites with unique properties can be created which could be a good combination of the properties of both components and could be used in a variety of applications such as antibacterial and electromagnetic protection [18, 19]. Polymer composites with ferrites act as better electromagnetic absorbers than bulk ferrites [20].

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Pavithradevi et al. [21] reported that dielectric permeability and dielectric loss of copper ferrite nano particles decreases, but electrical conductivity increases with frequency. S.L. Prokopenko et al. reported that an increase of copper iodide concentration in polymer composites PCTFE leads to increase in real and imaginary part of dielectric permittivity and conductivity [18].

Through this paper, we presented the synthesis copper nanoferrite and the extraction of the herbal extract of *Picea omorika.* The main purpose of the paper was to get herbal textile with good antibacterial properties. Textile materials were modified by herbal extract of *Picea omorika* and copper ferrite nanoparticles. We desided to do these modifications to increase durability of antibacterial activities and coloration. Finally, dielectric properties of all samples, modified and unmodified, were investigated.

2. MATERIALS AND METHODS

2.1. Materials

For the investigation of the changes in antimicrobial and dielectric properties of the textile materials modified with copper ferrite nanoparticles and methanolic herbal extract of *Picea omorika*, fabrics of the same construction characteristics and different raw material compositions were used. The basic characteristics of fabrics are given in Table 1.

Table 1. The basic properties of the tested fabrics

The sign of the fabric	Type of weaves	Surface mass [g/m^2]	Density [cm^{-1}]		Raw material composition
			Warp	Weft	
CO	plain	184.44	21.3	19.0	100 % cotton
PES	plain	163.50	21.4	18.4	100 % polyester
CO/PES	plain	171.01	21.2	17.9	50 % cotton/50 % polyester

All used chemicals and reagents were of analytical grade: Ferrous sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$), Copper (II) pentahydrate ($CuSO_4 \cdot 5H_2O$) and Sodium hydroxide were purchased from Lach-Ner (Czech Republic Sodium alginate (powder)). CHT-ALGINAT NV was purchased from CHT (Germany).

2.2. Solution of the copper ferrites nanoparticles preparation

The sodium alginate was dissolved in distilled water by magnetic stirring at 55 °C for 30 min in order to have an 8% alginate solution [22]. 695.05 mg of copper (II) sulfate pentahydrate and 177.27 mg of ferrous sulfate heptahydrate were added until complete dissolution in order to have $Fe^{3+}/Cu^{2+} = 3.5$. NaOH solution (5M) was then added in order to obtain pH10.

2.3. Modification of fabrics

In order to modify fabrics with copper ferrite nanoparticles all fabric samples were immersed in obtained copper ferrite solution and mixed by magnetic stirring at 50 °C for 30 min. After immersing all samples were dried at 45 °C. Second modification was obtained at the same way by immersing samples in the copper ferrite solution and solution of the herbal extract Picea omorika (50 mg/ml) in ration 1:1. Study of color fastness to washing is performed according to the standard [23]. The synthesis of copper ferrite and textile modification of textiles are shown in Figure 1.

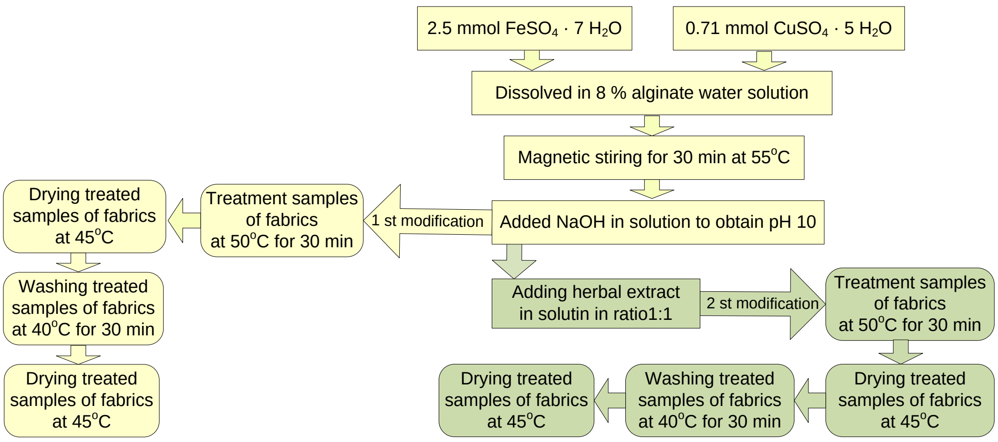


Figure 1. Flow chart of copper ferrite synthesis and textile modification

2.4. Determination of color difference (ΔE) spectrophotometrically (CIELAB)

The CIE $L^*a^*b^*$ color coordinates of the modified fabrics were determined using a portable spectrophotometer CM-2600d (Konica Minolta, Japan), under illuminant D65, with a 10° standard observer, measuring geometry d/8 and aperture 8 mm. Value a^* presents the degree of redness and greenness, taking positive values for reddish colors and negative values for greenish, while b^* is the degree of yellowness and blueness, taking positive values for yellowish colors and negative values for bluish. L^* is the degree of lightness, an approximate measurement of luminosity, which is the property according to which each color can be considered equivalent to a member of the grey scale, between black and white, taking values within the range of 0 – 100. The color differences (ΔE) between the bleaching and dyed cotton knitwear, was calculated according to the following equation [24].

$$\Delta E = \sqrt{(\Delta a^*)^2 + (\Delta b^*)^2 + (\Delta L^*)^2} \qquad (1)$$

where ΔL^* is the color lightness difference between the two samples, Δa^* is the red/green difference between the two samples, and Δb^* is the yellow/blue difference between the two samples.

2.5. Antibacterial activity of herbal fabrics

The antibacterial activity of the modified fabrics, on the bacteria *Staphylococcus aureus* ATCC 25923 and *Escherichia coli* ATCC 25922, was investigated by the method of diffusion in agar [25, 26]. For this investigation, the modified fabric samples were cut into patches of dimensions 2,5 x 2,5 cm.

The investigation procedure was performed as follows:

1. 100 μL of each culture was added to the surface of a sterile Mueller-Hinton agar plate.
2. The culture suspension was carefully distributed on the surface of the whole petri dish with a sterile glass L-stick.
3. Fabric samples were placed on the surface of the seeded culture.
4. Petri dishes were incubated for 24-48 hours at 37 °C.

After incubation, zones of inhibition were measured from the following equation:

$$Z_i = \frac{(T-D)}{2} \text{ (mm)} \qquad (2)$$

where are:
Z_i – width of zone of inhibition (mm),
T – width of sample + zone of inhibition (mm),
D – width of sample (mm).

If there is no zone of inhibition, and no growth below the sample, then it is defined as contact inhibition.

2.6. Dielectric properties of herbal textiles

Dielectric spectroscopy measurements were performed on precise LCR Hameg 8118 instrument in frequency range between 24 Hz to 75 kHz at temperature of 20.2 °C. The samples in form of thin disks (63.5 mm in diameter) are placed in the capacitor cell, whereby the dielectric measurements are carried out perpendicular to the samples, using U_0 = 1.0 V [27,28]. Conductance (G_m), and susceptance (B_m), of the samples, as well as, the conductance (G_b), and susceptance, (B_b), of an empty cell were measured using C_p model of the instrument, while the specific conductance and susceptance were calculated using the following relations:

$$G_{spec}\left(or\ B_{spec}\right) = G(or\ B) \times \frac{d}{S} \qquad (3)$$

The dielectric permittivity (ε_r) was calculated from the experimental data using the following relations:

$$\varepsilon_r = Cd/\varepsilon S \qquad (4)$$

Where $C=B/2\pi f$ and $B=B_m-(B_b-2\pi f\varepsilon_0 S/d)$ and f is the frequency, C is the capacity, d is the spacing between electrodes (equal to the thickness of the sample), S is the area of the electrode, and ε_0 is the dielectric permittivity of vacuum (8.85×10^{-12} F/m). The dielectric loss tangent was calculated as $\tan\delta=G/B$.

3. RESULTS AND DISCUSION

The values of the ΔE parameter for fabrics after modification with copper-ferrite nanoparticles and *Picea omorika* herbal extract, as well as after the washing are given in Table 2.

After the study of the color difference (ΔE), Table. 3 and Figure 2, it can be seen that the samples of fabrics modified with copper ferrite nanoparticles and extract of *Picea omorika* achieved good color fastness after washing, 14.340 for sample CO/PES-FE-W(1x), 15.649 PES-FE-W(1x) and 21.685 for sample CO-FE-W(1x) in relation to unmodified fabrics. From the histogram, Figure2, it can be seen that the best color fastness to washing was shown by the CO-FE sample, ie. the smallest color difference (ΔE=12.255) was measured after washing.

Table 2. The color difference (ΔE) for fabrics modified with copper-ferrite nanoparticles

Sample label	L^*	a^*	b^*	ΔE^*
CO	92.880	1.798	-7.726	/
CO-F	90.758	0.160	9.582	17.514
CO-FE	67.962	0.352	15.024	33.772
CO-F-W(1x)	92.736	1.314	-3.886	3.873
CO-FE-W(1x)	75.950	1.670	5.824	21.685
PES	92.468	1.680	-5.612	/
PES-F	91.906	1.288	1.266	6.912
PES-FE	70.590	0.274	15.488	30.428
PES-F-W(1x)	92.426	1.580	-4.002	1.614
PES-FE-W(1x)	80.132	2.592	3.974	15.649
CO/PES	92.806	0.896	-5.346	/
CO/PES-F	91.904	0.066	5.864	11.277
CO/PES-FE	69.264	0.248	16.444	32.085
CO/PES-F-W(1x)	93.108	0.804	-4.858	0.581
CO/PES-FE-W(1x)	81.186	1.296	3.048	14.340

F – fabric modified with copper-ferrite nanoparticles
FE – fabric modified with copper-ferrite nanoparticles and herbal extract *Picea omorika*
F-W(1x) – fabric washed after treatment with copper-ferrite nanoparticles
FE-W(1x) – fabric washed after modification with copper-ferrite nanoparticles and herbal extract *Picea omorika*

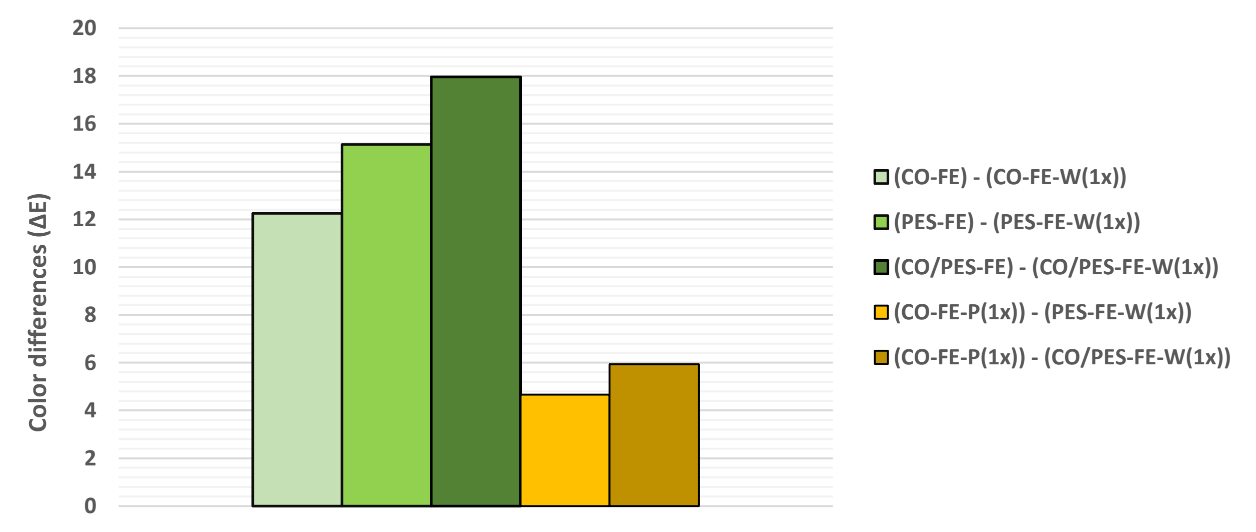


Figure 2. The Color difference between modified and washed fabrics of all samples

The results of studying the antimicrobial effect of fabrics modified with copper-ferrite nanoparticles on *Staphylococcus aureus* and *Escherichia coli* are shown in Table 3, fabrics modified with copper-ferrite nanoparticles with the addition of methanol herbal extract of *Picea omorika* are shown in Table 4 and modified fabrics after the washing are shown in Table 5.

From the results of studying antibacterial effect of fabrics modified with copper-ferrite nanoparticles, Table 3, it can be seen that all modified fabrics show antibacterial activity in the form of contact inhibition on *Escherichia coli*, while on *Staphylococcus aureus* only CO/PES-F fabric shows antibacterial activity.

Antibacterial activities of fabrics modified with copper-ferrite nanoparticles with the addition of methanolic extract of the herbal *Picea omorika* are presented at Table 4. The appearance of contact inhibition on *Staphylococcus aureus* and *Escherichia coli* in all modified fabrics was observed. Also, from the Table 4, it can be seen that the modified CO-FE and PES-FE fabrics show an enhanced antimicrobial effect in the form of the appearance of a zone of sparse growth around the patch, i.e. partial inhibition on *Staphylococcus aureus*. This is explained by the presence of herbal extract of *Picea omorika* in the copper-ferrite nanoparticle solution because the CO-F and PES-F fabrics, modified without the addition of the extract, did not show any activity against *Staphylococcus aureus*.

Table 3. Results of antibacterial activity of fabrics modified with copper-ferrite nanoparticles

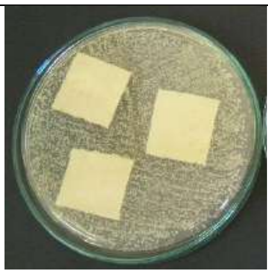
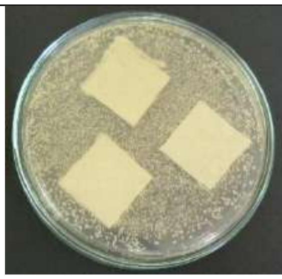
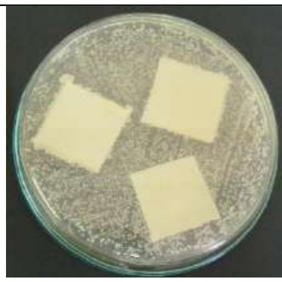
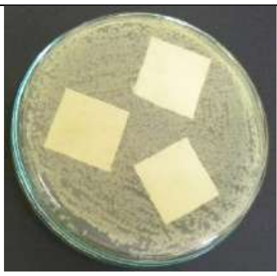
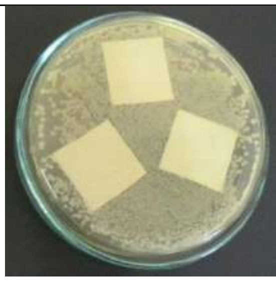
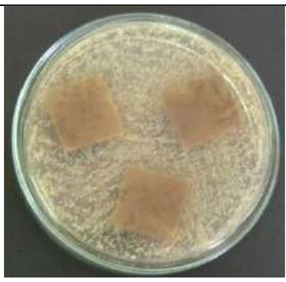
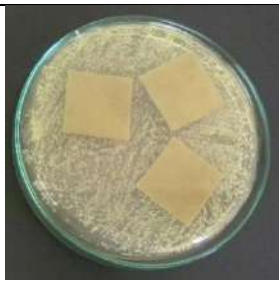
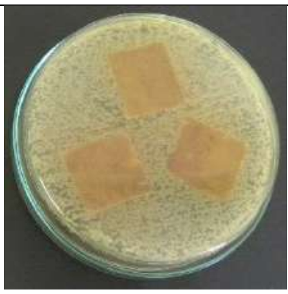
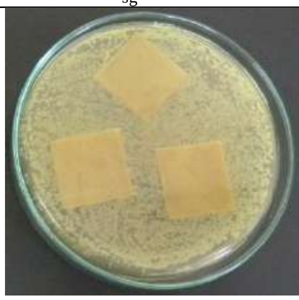
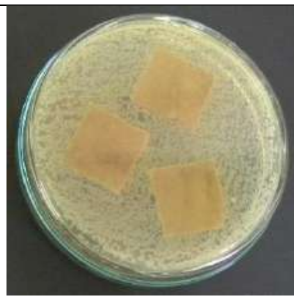
Bacteria tested	Labels of modified fabrics		
	CO - F	PES - F	CO/PES - F
Staphylococcus aureus	 NA	 NA	 KI
Escherichia coli	 KI	 KI	 KI
NA – no activity; KI – contact inhibition; Z_i – zone of inhibition; Z_{sg} – zone of sparse growth			

Table 4. Results of antibacterial activity of fabrics modified with copper-ferrite nanoparticles and methanol herbal extract of Picea omorika

Bacteria tested	Labels of modified fabrics		
	CO - FE	PES - FE	CO/PES - FE
Staphylococcus aureus	 KI i Z_{sg} = 1-2 mm	 KI i Z_{sg} = 1-2 mm	 KI
Escherichia coli	 KI	 KI	 KI
NA – no activity; KI – contact inhibition; Z_i – zone of inhibition; Z_{sg} – zone of sparse growth			

The results of antibacterial testing of modified fabrics after washing, Table 5, also showed better antibacterial activity against *E. coli*, except cotton fabrics (CO-F-W(1x) and CO-FE-W(1x)) for which activity is not observed. Modified fabrics after washing generally did not show any activity against *S. aureus*, except for contact inhibition in PES - FE - P (1x) fabric.

Gram-negative bacteria are known to be more resistant to the action of herbal extracts [29–31], so the good antimicrobial activity of tissues against *E. coli* can be attributed to the modification of copper-ferrite nanoparticles.

Table 5. Results of antibacterial activity of modified fabrics after washing

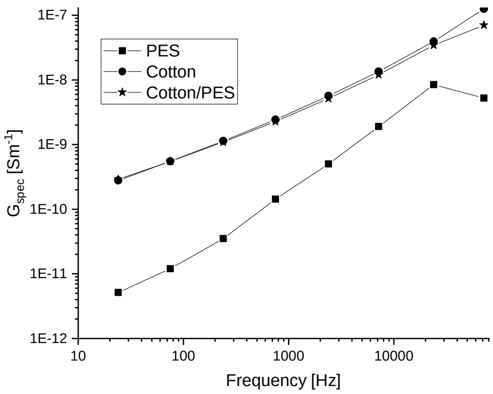
Labels of modified fabrics	Bacteria tested	
	Staphylococcus aureus	*Escherichia coli*
CO-F-W(1x)	NA	NA
PES-F-W(1x)	NA	KI
CO/PES-F-W(1x)	NA	KI
CO-FE-W(1x)	NA	NA
PES-FE-W(1x)	KI	KI
CO/PES-FE-W(1x)	NA	KI
NA – no activity; KI – contact inhibition		

Dielectric spectroscopy

In this part dielectric properties of polyester, cotton and cotton/polyester fabrics were presented. Also changes in dielectric properties of these fabrics by modification with copper ferrite nanoparticles and herbal extract of *Picea omorika* at room temperature were discussed.

At Figure 3 were presented specific conductance and susceptance of polyester, cotton and cotton/polyester. One can see increasing in both, conductance and susceptance with increasing in frequency. Specific conductance of cotton and cotton/polyester is about one order of magnitude higher than the value of specific conductance of polyester sample. This can be explained with the fact that the component with the higher values of dielectric properties has a dominant role in blend [32]. At lower frequency there are no significant difference between specific conductance of cotton and cotton/polyester samples, while at the higher frequency specific conductance of cotton is 78 % higher than for cotton/polyester sample.

At Figure 4, the results of the frequency dependence of specific conductance [*G*] and susceptance [*B*] for the polyester fabric are presented. Furthermore, one can see changes in specific conductance an susceptance of polyester fabric by modification with copper ferrite nanoparticles and by herbal extract of *Picea omorika*. As expected, based on conductivity of the copper ferrite nanoparticles [21,33], the specific conductance increases by sample modification with copper ferrite nanoparticles. Similar changes were observed by Prokopenko et al. [18]. When a thin layer of polymer is placed between the fillers, charge particle transportation occurs through tunnelling and hopping process [34]. By additional modification with herbal extract of *Picea omorika* specific conductivity takes place between values of unmodified and modified with copper ferrite nanoparticles.



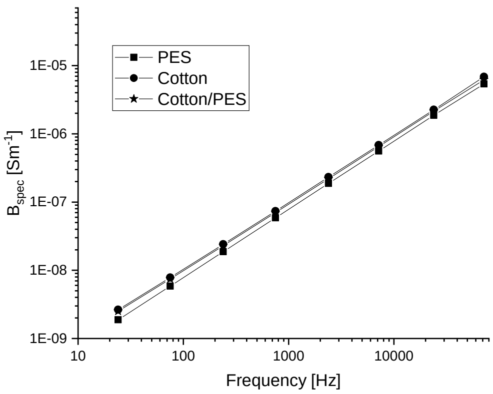


Figure 3. Frequency dependence of specific conductance and susceptance of cotton, polyester and cotton/polyester fabrics without modification at room temperature.

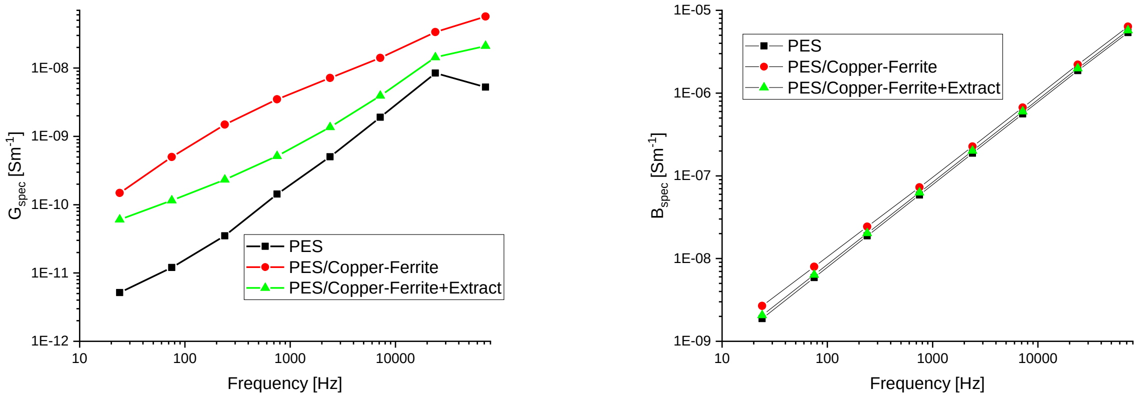


Figure 4. Frequency dependence of specific conductance and susceptance of polyester fabric, polyester fabric modified with copper ferrite nanoparticles and polyester fabric modified with copper ferrite nanoparticles and herbal extract of Picea omorika

Specific conductance of unmodified PES increase three orders of magnitude by an increase in frequency, while modified samples showed two orders of magnitude increase. At low frequency increase in specific conductance by sample modification with copper ferrite nanoparticles is two orders of magnitude, while at higher frequency increase is one order of magnitude. As the applied field frequency increases ferrites conductive grains become more effective thereby promoting electron hopping and transition between Fe^{2+} and Fe^{3+}. Due to high frequency, the electric charge carriers are not able to follow the alteration of the applied field and that is the reason why the increase in conductivity is less pronounced at higher frequencies [35,21]. Specific susceptance also showed an increase with modification of samples, but less pronounced then increase in specific conductance, see Figure 4. Frequency dependence of specific conductance of cotton fabric and cotton/polyester fabric with the same modification are similar to PES fabrics and are presented at Figure 5 and Figure 6.

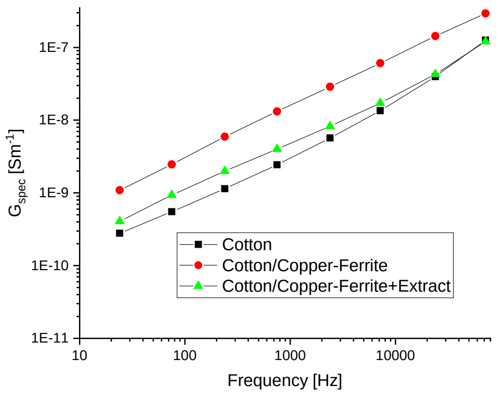


Figure 5. Frequency dependence of specific conductance of cotton fabric, cotton fabric modified with copper ferrite nanoparticles and cotton fabric modified with copper ferrite nanoparticles and herbal extract of Picea omorika.

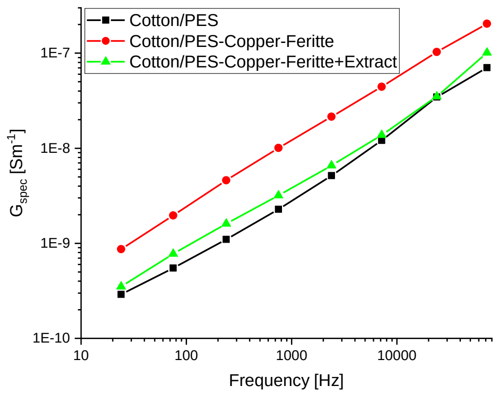


Figure 6. Frequency dependence of specific conductance of cotton/polyester fabric, cotton/polyester fabric modified with copper ferrite nanoparticles and cotton/polyester fabric modified with copper ferrite nanoparticles and herbal extract of Picea omorika.

Dielectric permittivity and dielectric loss tangent as a function of frequency for all samples modified with copper ferrite nanoparticles are presented at Figure 7 and Figure 8, it can be seen that there is a *tan δ* maximum at frequency about 200 Hz only for the PES sample modified with copper ferrite nanoparticles. By analyzing results for other samples their very similar trend of changes with increase of frequency can be seen.

Cotton fabrics, modified and unmodified, have shown the biggest increase in conductance. Dielectric permittivity and *tan δ* as a function of frequency for cotton fabric, cotton modified with copper ferrite nanoparticles and cotton modified with copper ferrite nanoparticles and extract are presented at Figure 9 and Figure 10 at frequency of 24 Hz, unmodified cotton fabric has value of the dielectric permittivity 2, while cotton modified with copper ferrite nanoparticles has value of 3.2. The dielectric permittivity of all samples decreases with increase in frequency. Taking into account the modified fabrics, this relaxation process can be identified as grain bounderies polarization [35]. Dielectric permittivity of copper ferrite modified cotton decreased rapidly at lower frequencies and it reached a constant value at higher frequencies. Such behavior can be explained on the basis of space charge polarization and electronic polarization [21]. Dielectric permittivity and *tan δ* for other samples are smaller than values for cotton fabric and they are not presented.

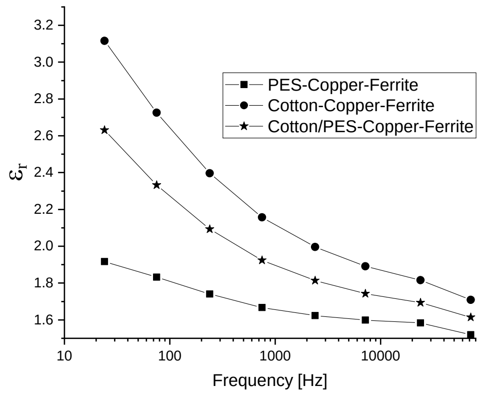


Figure 7. The dielectric permittivity as a function of frequency for all the samples modified with copper ferrite nanoparticles at room temperature.

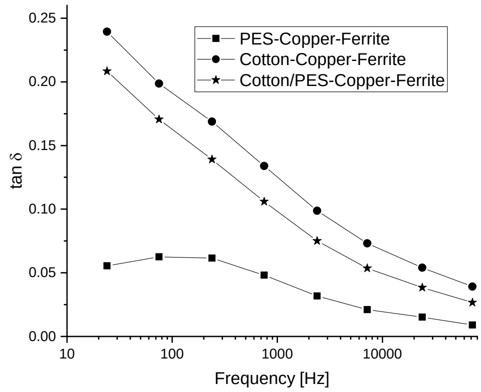


Figure 8. The tan δ as a function of frequency for all the samples modified with copper ferrite nanoparticles at room temperature.

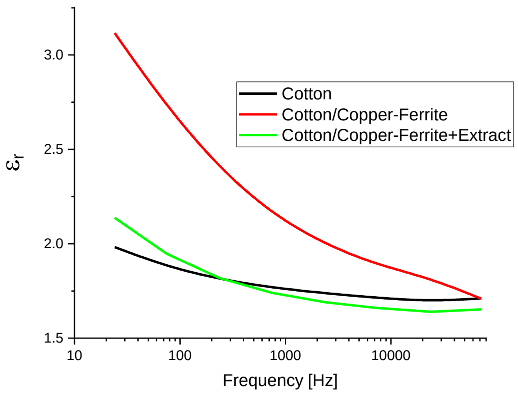


Figure 9. The dielectric permittivity as a function of frequency for the samples: cotton fabric, cotton fabric modified with copper ferrite nanoparticles and cotton fabric modified with copper ferrite nanoparticles and herbal extract of Picea omorika.

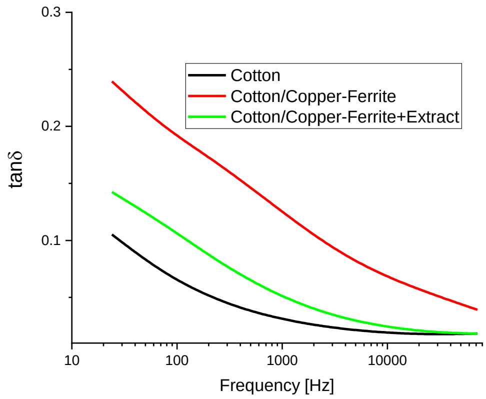


Figure 10. The tan δ as a function of frequency for the samples: cotton fabric, cotton fabric modified with copper ferrite nanoparticles and cotton fabric modified with copper ferrite nanoparticles and herbal extract of Picea omorika.

4. CONCLUSION

After the study of the antibacterial and dielectric properties of unmodified fabrics and fabrics modified with copper-ferrite nanoparticles and herbal extract of the *Picea omorika*, it was found that:

– All fabrics modified with copper-ferrite nanoparticles show antibacterial activity, which acts as contact inhibition against *Escherichia coli*, while against *Staphylococcus aureus* antibacterial activity was observed only in the CO/PES fabric modified with copper-ferrite nanoparticles.

– Antibacterial activity against *Staphylococcus aureus* and *Escherichia coli* was observed in all fabrics modified with copper-ferrite nanoparticles and methanolic herbal extract of the *Picea omorika* .

– Cotton and PES fabrics modified with copper-ferrite nanoparticles and methanolic herbal extract of the *Picea omorika* show an enhanced antibacterial activity, which acts as partial inhibition on *S. aureus*. This is explained by the presence of herbal extract of the *Picea omorika* in the solution of copper-ferrite nanoparticles, because the herbal extract shows a good effect against gram-positive bacteria, which includes *S. aureus*.

– The results of the antibacterial activities of the modified fabrics after washing also showed better antibacterial activities against *E. coli*, except in cotton fabrics in which no activity was observed.

– Specific conductance of cotton and cotton/polyester is about one order of magnitude higher than value of specific conductance of polyester sample. At lower frequency there are no significant differences between specific conductance of cotton and cotton/polyester samples, while at a higher frequency specific conductance of cotton is 78 % higher than for cotton/polyester sample.

– As expected, based on conductivity of the copper ferrite nanoparticles, the specific conductance of the all samples increases by sample modification with copper ferrite nanoparticles. The values of specific conductances of samples additionally modified with herbal extract of *Picea omorika* are between the values of unmodified samples and samples modified with copper ferrite nanoparticles.

– All results indicated that cotton fabrics modified with copper ferrite nanoparticles showed more pronounced increase in conductance than other fabrics, also if one wants to develop material with good antibacterial properties and better conductivity than pure fabrics the best choice is cotton fabric modified with both, copper ferrite nanoparticles and herbal extract of *Picea omorika*.

5. ACKNOWLEDGMENTS

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АНТИБАКТЕРИЈСКЕ И ДИЕЛЕКТРИЧНЕ ОСОБИНЕ ТЕКСТИЛНИХ МАТЕРИЈАЛА МОДИФИКОВАНИХ ЕКСТРАКТОМ БИЉКЕ *Picea omorika* И БАКАР-ФЕРИТНИМ НАНОЧЕСТИЦАМА

Сажетак: У овом раду модификоване су тканине од памука, полиестера и мјешавине памук/полиестер користећи екстракт биљке *Picea omorika* и бакар-феритне наночестице и тестиране су њихове антибактеријске и диелектричне особине. Антибактеријска активност свих узорака испитана је на бактерије *Escherichia coli* и *Staphylococcus aureus*. Већина тканина модификованих бакар-феритним наночестицама показала је антибактеријско дјеловање на *Escherichia coli*, док је додатак екстракта биљке *Picea omorika* побољшао њихове антибактеријске особине. Диелектрични параметри мјерени су у фреквентном опсегу од 24 Hz до 75 kHz на собној температури, а резултати су показали да је модификација све три тканине бакар--феритом узроковала пораст њихове електричне проводљивости. Добијени резултати указују на могућност употребе испитиваних тканина за антибактеријску, као и електромагнетну заштиту.

Кључне ријечи: антибактеријске особине, диелектричне особине, тканине, екстракт биљке *Picea omorika*, бакар-ферит наночестице.

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