Investigation on Electromagnetic Interference Properties, Surface Resistivity and Antibacterial Activity of Woven Fabrics produced By Cotton/Metal Composite Yarns

Pamuk/Metal Kompozit İpliklerden Üretilen Dokuma Kumaşların Elektromanyetik Ekranlama, Yüzey Özdirenç ve Antibakteriyel Aktivitelerinin Araştırılması

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Online Erişime Açıldığı Tarih (Available online):30 Eylül 2019 (30 September 2019)

Bu makaleye atıf yapmak için (To cite this article):
İlkan ÖZKAN, İlhami İlHAN, Ahmet Yiğit YARAR (2019): Investigation on Electromagnetic Interference Properties, Surface Resistivity and Antibacterial Activity of Woven Fabrics produced By Cotton/Metal Composite Yarns, Tekstil ve Mühendis, 26: 115, 281-288.

For online version of the article: https://doi.org/10.7216/130075992019261150

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INVESTIGATION ON ELECTROMAGNETIC INTERFERENCE PROPERTIES, SURFACE RESISTIVITY AND ANTIBACTERIAL ACTIVITY OF WOVEN FABRICS PRODUCED BY COTTON/METAL COMPOSITE YARNS

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Gönderilme Tarihi / Received: 14.03.2019  
Kabul Tarihi / Accepted: 01.09.2019

\textbf{ABSTRACT:} In this study, the effect of design parameters of metal composite woven fabric on electromagnetic shielding effectiveness (EMSE), electrical resistivity and antibacterial activity properties were investigated. For this purpose, metal composite yarn including insulated copper, stainless steel, silver wires and silver coated polyamide (Shieldex) were plied with 100\% cotton spun yarns by two-for-one twisting technique. Eight different composite fabrics were produced in hand loom with different metal composite yarn densities. 100\% cotton fabric was also produced as control sample. EMSE, surface resistivity, antibacterial activity and circular bending rigidity tests were applied to the samples. As a result, all fabrics including metal composite yarns showed EMSE up to 53 dB levels. Stainless steel, silver wires and silver coated polyamide have $10^6$ times lower surface resistivity than copper and control samples. Antibacterial activity of copper and silver samples were investigated for control purposes. Finally, antibacterial activity was not observed in both samples due to the insulation of copper and non-ionizable silver coating.

\textbf{Keywords:} Antibacterial activity, Bending rigidity, Composite fabric, Electromagnetic shielding effectiveness, Metal composite yarn, Surface resistivity

\textbf{ÖZET:} Bu çalışmada, metal kompozit iplikler içeren dokuma kumaşların üretiminde tasarım parametrelerinin elektromanyetik ekranlama etkinliği (EMSE), elektriksel direnç ve antibakteriyel aktivite özelliklerini üzerindeki etkisi incelenmiştir. Bu amaçla, izole bakır, paslanmaz çelik, gümüş teller ve gümüş kaplı poliamid (Shieldex) filament ikiye bir büküm tekniği kullanılarak % 100 pamuk ipliği ile birleştirilmiştir. El dokuma tezgâhı kullanılarak, farklı kompozit iplik yoğunluklarına sahip, bezayağı dokusunda 8 farklı kumaş, ayrıca % 100 pamuklu kontrol numunesi üretimi gerçekleştirilmiştir. Numunelere EMSE, yüzey özdirenci, antibakteriyel aktivite ve dairesel eğilme direnci testleri uygulanmıştır. Araştırma sonucunda metal kompozit iplik içeren tüm numuneler 53 dB’ye ulaşan seviyelerde EMSE göstermiştir. Paslanmaz çelik, gümüş teller ve gümüş kaplı poliamid içeren numunelerin bakır kompozit ve kontrol numunelerninden $10^6$ kat daha düşük yüzey direncine sahip olduğu tespit edilmiştir. Bakır ve gümüş içeren numunelere kontrol amaciyla antibakteriyel aktivite testi uygulanmıştır. Sonuç olarak bakır yüzeyindeki izolasyon ve gümüşün elementel (iyonize olmayan) formundan dolayı antibakteriyel aktivite ortaya çıkmamıştır.

\textbf{Anahtar Kelimeler:} Antibakteriyel aktivite, Eğilme direnci, kompozit kumaş, Elektromanyetik ekranlama, Cotton/Metal kompozit iplik, Yükse özdirenci

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\textbf{DOI:}\ 10.7216/1300739920192611508, www.tekstilvemuhendis.org.tr
1. INTRODUCTION

Recently, people are increasingly exposed to electromagnetic radiation and static electricity in parallel with technological developments. Therefore, there are a lot of scientific research in the literature on fabrics intended to protect against the effects of electromagnetic waves and static electricity. The commonly used methods to achieve this goal are coating with conductive materials, conductive nano-doped polymer fibers, blending of metal fibers with natural or man-made fibers and using conductive composite yarns produced by various methods such as core spun, plying/twisting and intermingling [1]. Researches in literature about the metal composite textile materials mostly focused on electrical and electromagnetic properties. However, fabrics provide a suitable surface for growing of microorganisms in public places. Especially, natural fibers with high hydrophilicity provide the humid environment that microorganisms need. Therefore, antibacterial properties were taken into consideration as well as surface resistivity and EMSE.

Previous researches in this field are summarized below. Su and Chern (2004) produced woven fabrics by different type of stainless steel/polyester yarns (core, covered and plied). They examined the effects of hybrid yarn density, geometry and weave type on electromagnetic shielding effectiveness (EMSE) up to 3000 MHz frequency. They noted that EMSE value increases with increasing SS yarn density, core and plied yarns show better EMSE than cover yarn, the fabrics containing SS in the single direction showed lower EMSE and 1/1 plain woven fabrics provided optimum EMSE [2]. Varnaitė et. al. (2008) investigated the electrical charging and dissipation properties of plain weave fabrics including polyester/inox (80/20 %) fibers. They used three different conductive yarn densities in the fabric samples. As a result, the authors found that the amount of PES/SS has a crucial effect on vertical electrical resistance and surface resistivity. Distance between the metal composite yarns is also effective on the shielding factor and half decay time [3]. Perumalraj and Dasaradan (2010) investigated the EMSE of plain and twill woven fabrics between 20 to 18,000 MHz frequencies. Cotton/copper wire yarns with different linear density and wire diameters used for fabric production. They examined the effects of fabric structures, yarn density per cm, yarn type, and wire diameter on EMSE. The results indicate that an increase in the warp/weft density and cover factor increased the shielding effectiveness. Increase in the copper wire diameter generally decreased the EMSE. Twill and plain woven fabrics showed 61 dB and 74 dB EMSE values respectively between 750 to 1000 MHz frequencies [4]. Palamutcu et al. (2010) used staple silver/cotton and copper wire/cotton conductive spun yarns for producing plain woven and single jersey knitted fabrics. They measured the EMSE values of fabrics in the range of 900 - 1800 MHz frequency of communication bands. The authors found that structural parameters of woven and knitted fabrics, number of fabric layers, ratio and diameter of copper wire are effective factors on the EMSE. According to the results, EMSE values decreased with increase of copper thickness and decrease in layer of specimens [5]. Sekerden (2013) investigated the surface electrical resistivity and vertical electrical resistance of woven fabrics with three different weave patterns. In that study, four different stainless steel/cotton yarns were used as weft. Results indicated that the weft yarn type and weave type had significant effect on the surface resistivity and vertical resistance of the fabrics. In addition, increasing of the distances between conductive yarns decreased the conductivity of the samples [6]. Yu et al. (2016a) investigated the antibacterial activity, surface resistance, far infrared emissivity and EMSE properties of metal composite fabrics. They used hollow spindle technique for producing stainless steel core yarns. In structure of hybrid yarn, antibacterial nylon and BC-PET used as inner and outer wrapped yarns, respectively. Increasing of metal content in the structure decreased the surface resistance and improved the EMSE of the woven fabrics. The authors mentioned that the produced fabrics may be used in areas such as hospitals and working places against electromagnetic radiation and microbial cross-infection [7]. Erdumlu and Sarıçam (2016) produced woven fabrics with plain and twill weave types by using cotton/copper and cotton/stainless steel wrapped yarns. They measured electromagnetic shielding components of produced fabrics as single and double layer structures between 0-3000 MHz frequencies. The authors stated that the differences of EMSE values of plain and twill fabrics were not statistically significant and the layered structures of fabrics including stainless steel have better shielding effectiveness than the fabrics including copper hybrid yarns in a wider frequency range [8]. Marciniak et al. (2016) investigated the effect of pitch of metallic filament coil in hybrid yarn and density of these yarns as well in woven fabrics on the shielding effectiveness. The hybrid yarns were fabricated by wrapping technique. Woven fabrics having different hybrid yarns densities were produced with a double shuttles hand-weaving loom. The reference fabrics were produced with the same technique for comparison. They found that the fabrics containing hybrid yarns showed 60% higher SE than the reference ones at 30 MHz frequency [9]. Yu et al. (2016b) aimed to improve the comfort properties of multifunctional woven fabrics containing stainless steel wire. For this purpose, they used antibacterial nylon antibacterial activity and stainless steel wire to provide antibacterial activity and electromagnetic shielding features. They used the hollow spindle technique for producing multifunctional yarns. In that study, metal hybrid yarns were used as weft and polyester filaments were used as warp. Authors determined that the drying rate of the fabrics was improved with using crisscross section polyester and wicking and drying abilities of the samples affected wrapping amounts of hybrid yarns significantly. In addition, the electromagnetic shielding characteristics between 300 kHz–3 GHz frequency closely related with lamination angles and number of layers of fabrics [10].

In this study, it was aimed to produce efficient electromagnetic protective textile material for daily and professional use. For this purpose, the effect of conductive material types, densities and settlement of metal composite yarns in woven fabrics on surface resistivity and EMSE were investigated. In addition, antibacterial activity and bending rigidity features were examined. EMSE, surface resistivity, antibacterial activity and comfort properties of the samples were evaluated together for comprehensive characterization.
2. MATERIAL AND METHOD

2.1. Experimental Design and Production of Samples

In this research, copper (Cu), stainless steel wire (SS), silver wire (Ag) and silver coated polyamide 6 were used in composite yarn samples as metal contents. The metal filaments were individually plied and twisted (500 T/m) with 100% cotton (CO) spun yarns by two-for-one twisting technique to produce metal composite yarn samples. So, four different composite yarns were obtained excluding 100% cotton spun yarn. Technical properties of the yarns are given in Table 1.

The produced yarn samples were woven into plain fabrics using a handloom by changing the metal composite yarn density and settlement direction (weft or warp). The experimental design is given in Table 2. Thus, 9 different fabric designs were obtained and all the production factors were tried to be kept as constant as possible. The average yarn densities of the fabric samples were 10 wefts/cm and 12 warps/cm. Plain weave was preferred because it provides more interlacement, more contact points for giving the best conductivity in fabric.

The magnified images of the fabric samples were taken by a digital stereo microscope for visual assessment. The images of samples and sample codes were given in Figure 1. In all images, the metallic filaments and their helical form are clearly observed. So, the metallic yarns do not form a grid structure composed of linear lines.

Circular bending rigidity, electromagnetic shielding effectiveness, surface resistivity and antibacterial activity tests were applied. The obtained data were analyzed statistically and evaluated comparatively.

2.2. Measurement of Bending Rigidity

Softness is an important comfort parameter of fabrics for daily use. This parameter is mainly affected by the stiffness of the yarns in fabric structure. The bending rigidity provides information about softness of fabric--and performance of fabric use. So, bending rigidity of the woven fabrics was tested to determine the effect of the used conductive filaments on the softness of fabrics by a digital pneumatic stiffness tester according to ASTM D 4032 test standard. The measurements were repeated 5 times for each sample.

2.3. Measurement of EMI Shielding Properties

The free space measurement method was used for EMSE measurement. This is one of the basic method for SE measurement of planar materials. This method allows measurement in wider frequency range and larger sample sizes than the other measurement techniques that performed in controlled areas. EMSE measurements of the fabric samples were carried out by using Agilent PNA-L VN model microwave vector network analyzer (10 MHz - 43.5 GHz) at the range of 3–18 GHz (Figure 2). In measuring process, two horn antennas were used and placed on the same axis. The fabric samples were placed at the midpoint of the gap (15 cm) between the two antennas.

| Yarn Types | Count (Tex) | Technical Properties |
|------------|-------------|----------------------|
| Standard Yarn | Cotton carded ring spun yarn | 59.07 | - |
| Composite Plied Yarns | Ag coated polyamide 6 / standard yarn | 67.50 | Shieldex® 54/12 dtex (silverized), Z turns, 99% pure silver coated, non-antibacterial |
| | Ag metal filament / standard yarn | 66.23 | 54.00 dtex, diameter 50µ |
| | Cooper metal filament / standard yarn | 66.49 | 178.44 dtex, insulated, diameter 50µ |
| | SS metal filament / standard yarn | 78.58 | 163.96 dtex, diameter 50µ |

Table 1. Technical properties of yarns

| Sample Code | Yarn Type | Yarn Pattern | Yarn Type | Yarn Pattern |
|-------------|-----------|--------------|-----------|--------------|
| 1           | CO        | full         | CO        | full         |
| 2           | CO/Cu and CO | consecutively alternating | CO/Cu and CO | consecutively alternating |
| 3           | CO/Cu     | full         | CO/Cu     | full         |
| 4           | CO/SS and CO | consecutively alternating | CO/SS and CO | consecutively alternating |
| 5           | CO/SS     | full         | CO/SS     | full         |
| 6           | CO/Ag PA 6 and CO | consecutively alternating | CO/Ag PA 6 and CO | consecutively alternating |
| 7           | CO/Ag PA 6 | full         | CO/Ag PA 6 | full         |
| 8           | CO/Ag and CO | consecutively alternating | CO/Ag and CO | consecutively alternating |
| 9           | CO/Ag     | full         | CO/Ag     | full         |

*All yarns are same type,
**The types of yarns are alternated consecutively in the woven structure
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Figure 1. 30x magnified images of the fabric samples

Figure 2. Schematic diagram for measurement principle of EMSE

Attenuation property is the basic characteristic of the conductive fabric. Specific material that placed between the electromagnetic receiver and transmitter attenuates the electromagnetic energy due to the reflection, absorption and multi-reflection losses [5]. Shielding effectiveness is described as the logarithmic form of the ratio between the field intensity in a place without shielding material (E₀) and the field strength in the same measurement with shielding material (Eᵣ). It is calculated by the Equation 1 [11].

\[ SE = 20 \cdot \log \frac{E₀}{Eᵣ} \]  

(1)

2.4. Measurement of Surface Resistivity

Surface Resistivity of the samples were measured by using ELME MULTIMEG megohmmeter at 53.0% relative humidity (RH) and 25.0 °C. Measurement of surface resistivity was carried out according to “TS EN 1149-1: Electrostatic properties- Part 1: surface resistivity” standard. The measurements were repeated 10 times for each sample in different directions. Test data were analyzed statistically and evaluated comparatively. The classification of materials according to their surface Resistivity (ohm) is shown in Table 3.
Table 3. Surface resistivity classification of materials [12]

| Classification       | Surface Resistivity (ohm/sq) |
|----------------------|------------------------------|
| Conductive           | <10^5                        |
| Dissipative          | 10^5 to 10^12                |
| Insulating           | >10^12                       |

2.5. Measurement of Antibacterial Properties

AATCC 100 test method provides a quantitative evaluation for the antibacterial activity level of the fabrics. So, this method was preferred for determining the antibacterial activity against *K. pneumonia* which causes various diseases in humans such as respiratory tract infections, nosocomial infections etc. According to test procedure, the samples were cut as circular swatches with 4.8 ± 0.1 cm diameters and placed separately in sterile glass plates. Inoculations were carried out with bacteria culture having a concentration of 1.5–2.0 × 10^5 CFU/mL. Contact time was preferred as 24 hours in inoculation step. The inoculated samples were added to the 100 ± 1 mL of neutralizing solution and shaken for a minute. Serial dilutions were done with sterile deionized water for obtaining colonies on countable level. A 100 μL suspension of the last dilution was poured on agar plates and incubated for 24 h at 37 °C. After incubation, the numbers of colony were counted visually and the antibacterial activity was calculated using Eq 2.

\[ R(\%) = 100 \left( \frac{C - A}{A} \right) \]  

Where; \( R \) is the percentage reduction ratio, \( A \) is the number of bacteria recovered from the inoculated metal contained test specimen swatches incubated for 24 hours, and \( C \) is the number of bacteria recovered from the inoculated control specimen swatches immediately after inoculation (at “0” contact time).

3. RESULTS AND DISCUSSION

3.1. Bending Rigidity Results

Bending rigidity test data was evaluated statistically. ANOVA and multiple comparison analysis were carried out. Results showed that the use of metal monofilaments in the fabric structure significantly increased the bending rigidity of the fabric as expected. There was a statistically significant difference between the consequently alternating and full samples. The increase in amount of metal monofilament significantly increased the rigidity of samples. There was not statistically significant difference between the rigidity values of control and Ag_PA/CO samples for both fabric structures. Circular bending rigidity test results are given in Figure 3. It can be noted that the use of metal-coated PA filaments is effective on the bending rigidity. Because the flexural rigidity of multilaminate yarn is much lower than the equivalent monofilament yarn [13]. Therefore, metal-coated multifilament yarns should be preferred to obtain the more comfortable metal composite fabric.

![Figure 3. Comparison of circular bending rigidity values](image)

3.2. EMI Shielding Measurement Results

The plots of SE (Shielding Effectiveness) are given in Figure 4a and 4b. In Figure 4a, SE values of all fabrics decreased with increasing of frequency. Shielding behavior of the fabric samples including Cu/CO and Ag_PA/CO showed a similar tendency, the same situation was also observed for the SS/CO and Ag/CO samples. Moreover, Ag_PA/CO sample showed the better SE values between 3.75 – 7.20 GHz. Cu/CO and Ag_PA/CO samples showed higher peak points than SS/CO and Ag/CO samples between 9.00 – 13.50 GHz. The mentioned regular tendency did not observe over 13.5 GHz. The obtained peak values for Cu/CO, SS/CO, Ag_PA/CO and Ag/CO consecutively alternating samples were 43.04, 24.22, 33.07, 20.97 dB, respectively.

![Figure 4. Shielding effectiveness plots of (a) consecutively alternating and (b) full samples](image)
In Figure 4b, SE values of the full samples decreased with increasing frequency similar to Figure 4a. When Figure 4a and 4b were compared with each other, SE values of all the full samples were generally higher than the consecutively alternating samples. It showed that SE performance increased with increasing the amount of metal content in fabric structure. In both plots, Cu/Co samples gave the maximum peak points for SE. These peak points were 43.04 dB at 8.40 GHz for the consecutively alternating samples and 53.72 dB at 8.67 GHz for the full samples. The peak SE values obtained for Cu/CO, SS/CO, Ag_PA/CO and Ag/CO full samples were 53.72, 44.56, 39.91, 41.18 dB, respectively. It is worth noting that the mentioned frequency and SE values at peak points in both plots were close to each other.

3.3. Surface Resistivity Measurement Results

The plots of surface resistivity for the consecutively alternating and full samples are given in Figure 5.

As seen in Figure 5, the full and consecutively alternating samples including SS/CO, Ag_PA/CO and Ag/CO yarns had about $10^6$ times lower surface resistivity than the control and Cu/CO samples. As can be expected, the surface resistivity of the Cu/CO sample was lower the other composite fabrics due to the insulation of Cu wire. According to Figure 5a and 5b, it can be said that the increase in amount of metal content in fabric structure did not make any significant difference on the surface resistivity.

Statistical analysis was also applied to the test data. The ANOVA results showed that there was a significant difference between the groups. Post hoc tests were applied to confirm where the differences occurred between groups. As a result of statistical analyses, the differences among the groups were statistically significant and the surface resistivity values could be ranked in descending order as follows; Control > Cu/CO > SS/CO > Ag/CO > Ag_PA/CO for the consecutively alternating samples. The difference between Ag/CO and Ag_PA/CO were not statistically significant and the ranking was occurred as follows; Control > Cu/CO > SS/CO > Ag/CO ~ Ag_PA/CO for the full samples. Silver composite fabric samples had the lowest electrical resistivity. Because electrical resistivity of Ag (1.47 x$10^8$ ohm-m) is lower than Cu (1.56 x$10^8$ ohm-m) and SS (5.56 x$10^7$ ohm-m) [1]. The multifilament yarn has a larger surface area than monofilament wire. Thus, Ag_PA sample exhibited lower surface resistivity than Ag sample at low densities. Therefore, it can be said that it is more convenient to use metal-coated multifilament yarns in fabrics where higher conductivity is required.

3.4. Results of Antibacterial Activity Tests

The researches in literature noted that the stainless steel has no antimicrobial properties and copper and silver are widely used in various forms due to their antibacterial properties [14-16]. It is known that only silver ions provide antimicrobial activity as confirmed experimentally. The antibacterial property of silver is related to the amount of silver ions. Silver is inert in elemental form but it gets ionized when reacts with the moisture. The ionized silver is highly reactive, it binds to tissue proteins and causes structural changes in the bacterial cell wall and nuclear membrane leading to cell disruption and death. Silver also binds to bacterial DNA and RNA by denaturing and inhibits bacterial replication [17-19]. Hence, antibacterial activity tests were only applied to Cu/CO and Ag_PA/CO full samples for investigating antibacterial effect of insulated Cu wire and Ag_PA filament. The petri dishes photos of the control sample at 0 contact time, Cu/CO and Ag_PA/CO for 24 hours contact with bacterial solution are given in Figure 6.

As seen in Figure 6, the number of K. pneumoniae colonies increased after 24 hours as compared with control sample. It was determined that the Cu/CO and Ag_PA /CO full samples did not have antibacterial activity. The reason may be that the isolated structure of copper wire prevents bacterial contact for Cu/CO full sample. In addition, silver ions could not be released from silver-coated PA filament yarns in Ag_PA/CO full sample due to the non-ionizable form of silver coating. These results approved that ionizable forms of copper and silver are required to provide the antibacterial activity in fabrics.
4. CONCLUSIONS

In this study, the effect of some design parameters of the metal composite woven fabrics on electromagnetic shielding performance, electrical surface resistivity and antibacterial activity were investigated experimentally. The obtained results summarized below.

Bending rigidity test results showed that the rigidity of samples increased with increasing in amount of metal monofilament significantly. The use of Ag_PA multifilament in fabric construction had not affected the rigidity as opposed to monofilament samples. According to SE results, SE values of all samples generally decreased with increasing frequency. The SE values of full samples were higher than the consecutively alternating samples. It can be stated that SE performance increases with increasing of the amount of metal content. Cu/Co samples gave the maximum peak points for SE.

The surface resistivity measurements showed that the SS/CO, Ag_PA/CO and Ag/CO samples had about $10^8$ times lower surface resistivity than the control and Cu/CO samples for the full and consecutively alternating structures. Cu/CO sample showed very low conductivity (high surface resistivity) as the control sample due to the insulation of Cu wire. Ag_PA sample had the lowest surface resistivity because of the larger surface area of the multifilament yarn. As a result, it can be said that the conductivity can be increased more with the use metal coated multifilament yarns instead of metal monofilament wire.

Cu/CO and Ag_PA/CO full samples did not show antibacterial activity. The reason may be that the isolated structure of copper wire prevents bacterial contact for Cu/CO full sample. In addition, the non-ionizable form of silver coating did not provide to antibacterial activity. Finally, applications that prevents ionization of the metals eliminated the antibacterial activity. However, electromagnetic shielding feature did not affected from this situation.

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