ELECTROMAGNETIC SHIELDING PROPERTIES OF POLYPYRROLE TREATED LEATHER: A COMPARATIVE STUDY

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ABSTRACT. Due to the increase in electromagnetic waves emitted by the electrical and electronic equipment surrounding us, protection from electromagnetic waves has become a very important subject. It is thereby proposed that conductive textiles and fabrics are used for protection of sensitive equipment and personnel from electromagnetic interference. Conductive textiles are considered to be a particular sub-genre of technical textiles and exhibit favourable electromagnetic shielding properties together with a list of other attributes that make them suitable for such applications. In more detail, conductive textiles exhibit light weight and low volume while being comfortable to wear, conforming to different shapes of surfaces and everyday objects (thus being easily integrated within the surrounding environment), being aesthetically appealing, etc. In this context, most of the literature-available shielding textiles or fabrics used are either conventional or conductive fabrics that are further coated with metal layers for extra conductivity and shielding. However, metal shielding exhibits certain disadvantages: it is prone to corrosion while corrosion-resistant materials (such as silver or gold) are extremely expensive; it is difficult to process; and it compromises the comfortability and aesthetic appeal of the final product. Herein, we propose the usage of electrically conductive leather for efficient shielding from electromagnetic interference. The motivation for this work is twofold: first, leather materials are seldom if not at all used for electromagnetic shielding and we wanted to close that gap for an industry sector (leather clothes and garments) that is thriving. Second, we are using one or more layers of polypyrrole treatment (PPy) to inherit conductivity to leather surfaces. We show that PPy is sufficient treatment for inheriting very satisfactory electromagnetic shielding properties all the while maintaining the comparative advantages of a non-metal-processed fabric surface (e.g. comfortability, aesthetic appeal etc.). We demonstrate a variety of different PPy treated leather samples and illustrate that electromagnetic shielding is proportional to the number of PPy layers used for leather processing. The measurements results are obtained using RF and microwave high-frequency equipment in a controlled laboratory environment within the X-microwave band (8-12 GHz).

KEY WORDS: electromagnetic shielding, X-band, microwave shielding, leather materials, polypyrrole treatment

PROPRIETĂȚILE DE PROTECȚIE ELECTROMAGNETICĂ ALE PIELII TRATATE CU POLIPIROL: UN STUDIU COMPARATIV

REZUMAT. Datorită creșterii undelor electromagnetice emise de echipamentele electrice și electronice din jurul nostru, protecția împotriva undelor electromagnetice a devenit un subiect foarte important. Prin urmare, se propune utilizarea textilelor și țesăturilor conductoare pentru protecția echipamentelor sensibile și a personalului împotriva interferențelor electromagnetice. Textilele conductoare sunt considerate a fi o subcategorie specială de textile tehnice și prezintă proprietăți favorabile de ecranare electromagnetică, împreună cu o serie de alte atribute care le fac potrivite pentru astfel de aplicații. Mai în detaliu, textilele conductoare prezintă greutate redusă și volum redus, în timp ce sunt confortabile de purtat, se conformează diferitelor forme ale suprafețelor și obiectelor de zi cu zi (fiind astfel ușor de integrat în mediul înconjurător), fiind atractive din punct de vedere estetic etc. În acest context, majoritatea textilelor de ecranare întâlnite în literatură sau țesăturile utilizate sunt fie țesături convenționale, fie conductoare, care sunt acoperite cu straturi metalice pentru conductivitate și ecranare suplimentare. Cu toate acestea, ecranul metalic prezintă anumite dezavantaje: este predispus la coroziune, în timp ce materialele rezistente la coroziune (cum ar fi argintul sau aurul) sunt extrem de scumpe; acesta este dificil de procesat și compromite confortul și atractivitatea estetică a produsului final. În această lucrare propunem utilizarea pielii cu proprietăți de conductivitate electrică pentru o ecranare eficientă împotriva interferențelor electromagnetice. Motivația pentru această muncă este dublă: în primul rând, pielea este rareori sau chiar deloc folosită pentru ecranarea electromagnetică și am vrut să reducem acest decalaj pentru un sector industrial (haine și confecții din piele) care este înfloritor. În al doilea rând, folosim unul sau mai multe straturi de tratament cu polipirol (PPy) pentru a conferi conductivitate suprafețelor din piele. Demonstrăm că tratarea cu PPy este suficientă pentru conferirea unor proprietăți de ecranare electromagnetică foarte satisfăcătoare, menținând în același timp avantajele comparative ale unei suprafețe de țesătură neprelucrată cu metal (de exemplu, confort, atractivitate estetică etc.). Demonstrăm o varietate de mostre diferite de piele tratată cu PPy și ilustrăm faptul că ecranarea electromagnetică este proporțională cu numărul de straturi PPy utilizate la prelucrarea pielii. Rezultatele măsurătorilor sunt obținute folosind echipamente de frecvență radio (RF) și de înaltă frecvență și microunde într-un mediu de laborator controlat, cu microunde în banda X (8-12 GHz).

CUVINTE CHEIE: ecranare electromagnetică, banda X, ecranare cu microunde, piele, tratament cu polipirol

PROPRIÉTÉS DE BLINDAGE ÉLECTROMAGNÉTIQUE DU CUIR TRAITÉ AU POLYPYRROLE : UNE ÉTUDE COMPARATIVE

RÉSUMÉ. En raison de l'augmentation des ondes électromagnétiques émises par les équipements électriques et électroniques qui nous entourent, la protection contre les ondes électromagnétiques est devenue un sujet très important. Il est ainsi proposé que des textiles et des

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tissus conducteurs soient utilisés pour protéger les équipements et le personnel sensibles contre les interférences électromagnétiques. Les textiles conducteurs sont considérés comme un sous-genre particulier de textiles techniques et présentent des propriétés de blindage électromagnétique favorables ainsi qu'une liste d'autres attributs qui les rendent adaptés à de telles applications. Plus en détail, les textiles conducteurs présentent un poids léger et un faible volume tout en étant agréables à porter, s'adaptant à différentes formes de surfaces et d'objets du quotidien (s'intégrant ainsi facilement dans l'environnement), étant esthétiquement attrayants, etc. Dans ce contexte, la plupart des textiles ou tissus de blindage rencontrés dans la littérature utilisés sont des tissus conventionnels ou conducteurs qui sont en outre recouverts de couches métalliques pour une conductivité et un blindage supplémentaires. Cependant, les blindages métalliques présentent certains inconvénients : ils sont sujets à la corrosion alors que les matériaux résistants à la corrosion (comme l'argent ou l'or) sont extrêmement coûteux ; ils sont difficiles à traiter, et cela compromet le confort et l'attrait esthétique du produit final. Nous proposons ici l'utilisation de cuir électriquement conducteur pour un blindage efficace contre les interférences électromagnétiques. La motivation de ce travail est double : premièrement, les matériaux en cuir sont rarement, voire pas du tout, utilisés pour le blindage électromagnétique et nous voulions combler cet écart pour un secteur industriel (maroquinerie et vêtements en cuir) en plein essor. Deuxièmement, nous utilisons une ou plusieurs couches de traitement au polypyrrole (PPy) pour conférer de la conductivité des surfaces en cuir. Nous montrons que le PPy est un traitement suffisant pour conférer de propriétés de blindage électromagnétique très satisfaisantes tout en conservant les avantages comparatifs d'une surface de tissu non traitée en métal (par exemple confort, attrait esthétique, etc.). Nous démontrons une variété de différents échantillons de cuir traités au PPy et nous illustrons que le blindage électromagnétique est proportionnel au nombre de couches de PPy utilisées pour le traitement du cuir. Les résultats des mesures sont obtenus à l'aide d'équipements RF et haute fréquence et micro-ondes dans un environnement de laboratoire contrôlé, avec des micro-ondes en bande X (8-12 GHz).

MOTS CLÉS : blindage électromagnétique, bande X, blindage micro-ondes, cuir, traitement au polypyrrole

INTRODUCTION

Electromagnetic pollution is known to be harmful for both human health and sensitive electronic devices. Therefore it is essential to be protected from the hazardous effects of electromagnetic radiation. As electric and electronic devices and also the accessories are increasing rapidly, transmitting electro-magnetic power through the different frequency bands used on the markets, restricting and preventing electronic equipment from all sources of interference has become important. Electromagnetic shielding materials are used for protection from electromagnetic waves. Since most of the electromagnetic shields are materials including metals, they have several disadvantages such as poor handle properties, difficulties in processing, high cost, high weight, sensitivity to corrosion and so on. On the antipode, our garments cannot protect us due to the fact that they are not conductive. The smart garments need to be made conductive in order to provide shielding effects.

In order to achieve these electrical properties, from the traditional textiles, different methods can be used, such as incorporating conductive fibres/yarns, or treating them with conductive coatings, polymers or inks. Moreover, several techniques that can be used to integrate conductive fibres/yarns into a textile structure such as knitting, weaving, embroidering, sewing and needle felting, conductive textiles can be obtained also by treating the surface of the traditional textiles. This last method can be achieved by coating them with a conductive layer, by coating with conductive polymers, deposited onto the surface of textile substrates or by printing with conductive inks [1-13].

In this study we propose the chemical treatment of the leather, in order to be conductive. This can be achieved by coating it with different processes such as: electroless deposition, electroplating, Physical Vapour Deposition (PVD), Chemical Vapour Deposition (CVD), sputtering and coating with conductive polymers, where the last one is more used on textiles, and this paper focuses on this technique to make it conductive, but on leather [14-18].

This study focuses on the development of electrically conductive leather for electromagnetic shielding applications and investigation of their properties. Different groups have worked on leather shielding materials. Shen *et al*. has prepared natural composites based on leather and high-Z elements, which were used as gamma-ray shielding materials. They showed low density, high strength and wearable behaviours [19]. Gao *et al*. [20] developed a three-step procedure to produce flexible nanofibers films as shielding materials against electromagnetic interference (EMI), based on hydrolysate of waste leather scraps. The nanofiber films that they produced showed a high EMI shielding efficiency. Zeng *et al*. has fabricated a novel foldable leather solid waste conductive paper, which has offered a new avenue toward recycling of leather waste and displayed promising potential for applications in flexible shielding materials or wearable clothing [21]. While Zhao *et al*. propose a green chrome-free tanned electromagnetic shielding leather using *in situ* poly method with polyaniline. They were able to improve leather tanning and electromagnetic shielding performance to a large extent [22].

To the best of our knowledge this is the first time to report on electromagnetic shielding performance of electroconductive leather coated with polypyrrole, as alternatives to metals. Polypyrrole was chosen because it has a wide thermoelectric application and it is easy to be prepared, besides it is a low-cost polymer. Furthermore, intrinsically conducting polymers are able to absorb as well as reflect electromagnetic waves, exhibiting a significant advantage over metallic materials, which cannot be used as electromagnetic wave absorbers since high conductivity corresponds to a high reflection coefficient and very small penetration depth [23-24].

MATERIALS AND EXPERIMENTAL SETUP

Materials

The leather samples selected for conductivity treatment were white sheep crust Albanian leather chosen according to standard method [25]. Sheep crust leather samples have undergone the same preparation stage. They are tanned, dried, but not fully finished. The leather thickness varies from 0.9-1.1 mm.

Five samples were analysed in this study, sample number 0 is the reference sample which is not treated with PPy, while the other four samples, S1, S2, S3 and S4, are treated with PPy, as described in Table 1. All the reagents used, such as pyrrole (PPy), ferric chloride, ($FeCl_3$) anthraquinone-2- sulfonic acid sodium salt monohydrate [(AQSA), were purchased by Sigma-Aldrich].

Chemical Preparation of PPy-treated Leather Samples

Leather samples were treated using two chemical oxidative polymerization techniques: single *in situ* polymerization and double *in situ* polymerization. In order to obtain conductive leather, polypyrrole was chosen as a conductive polymer. The leather samples (10 x 10 cm) were prepared and conditioned before the treatment according to ISO standards [25-26], while after the thickness was measured based on the ISO standard [27].

The polymerization on leather samples was carried out using pyrrole monomer, ferric chloride as oxidant and AQSA as a dopant. The concentration of all the reactants, pyrrole, AQSA and $FeCl_3$ were used with different concentration and optimized in order to give maximum conductive properties to leather samples. In some cases, in order to raise the conductivity of the leather samples one parameter is changed, the others are kept constant [16].

Final experiments for *in situ* polymerization treatment were performed using pyrrole concentrations (0.15 M), AQSA concentration was 10 wt % based on the weight of pyrrole and ferric chloride concentration (0.4 M).

For single *in situ* polymerization treatment, first a mixed solution is prepared of 0.15 M pyrrole and AQSA (10 wt % based on the weight of pyrrole) by dissolving pyrrole in AQSA solution with vigorous stirring for 30 min. Then, the white sheep crust leather samples were first soaked in the mixed solution pyrrole/AQSA and are rotated in a rotating mixer for 1 hour at 10 rpm to uniformly mix. After the treatment, 0.4 M ferric chloride solution is added to the mixture as an oxidant to initiate the polymerization. The polymerization was carried out for 2 h at 5°C in 10 rpm. Treated leather samples were rinsed 4 times with distilled water and dried at 35 °C.

Experiments for double *in situ* polymerization were performed following all the procedures like the single *in situ* polymerization, but after the first bath the leather was treated again in a second bath containing the same concentrations of reactants, following the same procedure to obtain double *in situ* PPy coated leather. At the end the coated leather was washed 4 times with distilled water and dried at 35°C, as it is shown in Figure 1.

This polymerization method produces conductive and black coloured leather sample only by using the chemical polymerization treatment in a bath.

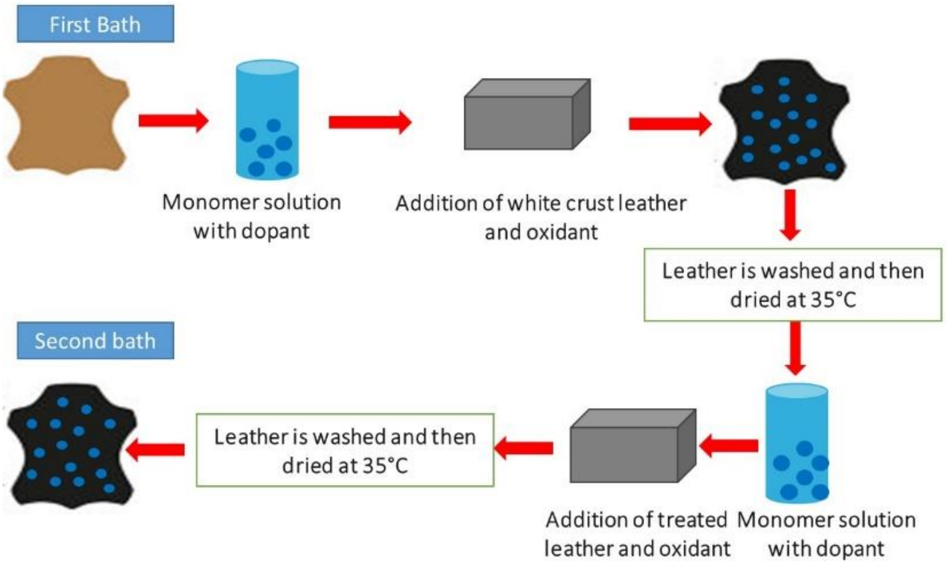


Figure 1. Scheme of the double *in situ* polymerization of pyrrole [18]

Resistance Measurements

The resistance of square shape leather samples was measured by using Van Der Pauw method [14]. The method consists of placing four probes along the circumference of the sample (in this case in four corners), two adjacent corners are used to apply a current by a DC power source and then by placing a multi meter into two opposite corners we measure the voltage drop. The procedure is repeated four times by changing corners one by one in clockwise direction. Then we calculate two resistances called horizontal and vertical resistances by using Ohm’s law. After that we solve the equation (1) regarding surface resistance.

$$e^{-\pi\frac{R_{vertical}}{R_S}} + e^{-\pi\frac{R_{horizontal}}{R_S}} = 1 \qquad (1)$$

Microwave Testbed and Electromagnetic Shielding Measurements

The microwave characterization of the shielding properties of the provided leather samples was performed in the premises of the Department of Electrical and Electronic Engineering, University of West Attica, Athens, Greece. We used a R&S ZVA24 Vector Network Analyzer (VNA) that is suitable for measurements up to 24 GHz, as well as appropriate cables, connectors and waveguides. The waveguide equipment consisted of type WR-90 waveguides operating in the X-band (8.20 to 12.40 GHz) thus limiting the frequency range of the final setup. The testbed included two straight waveguide components and two end-launchers for connection to the VNA. Initially, we calibrated the waveguide testbed by connecting the straight parts together without any sample in between. An overview of the one end of the testbed with an end-launcher and a straight waveguide component is illustrated in Fig. 2(a), while the calibration testbed is illustrated in Figs. 2(b) and 2(c) below. The careful reader may discriminate the characteristic cutoff frequency displayed in the attached monitor in Fig. 3(c).

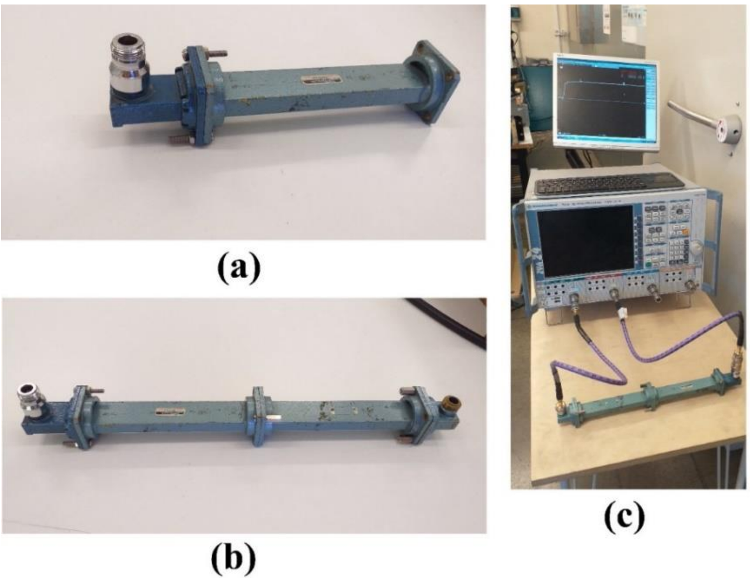


Figure 2. Waveguide testbed setup and VNA connections for initial calibration; (a) end-launcher and straight waveguide; (b) waveguide testbed; (c) final measurements setup

Subsequently, all the provided leather samples (reference samples and samples #1 through 4) were sequentially attached in the gap between the straight waveguide components and held together by applying gentle force through the supporting end-launchers. For each sample, the VNA display was photographed, printed to .jpg figures and the measurements were stored through the VNA user interface into .csv files. Photos of a leather sample attached to the testbed is illustrated in Figs. 3(a) and (c) below, while a close-up of the VNA display is provided in Fig. 3(b).

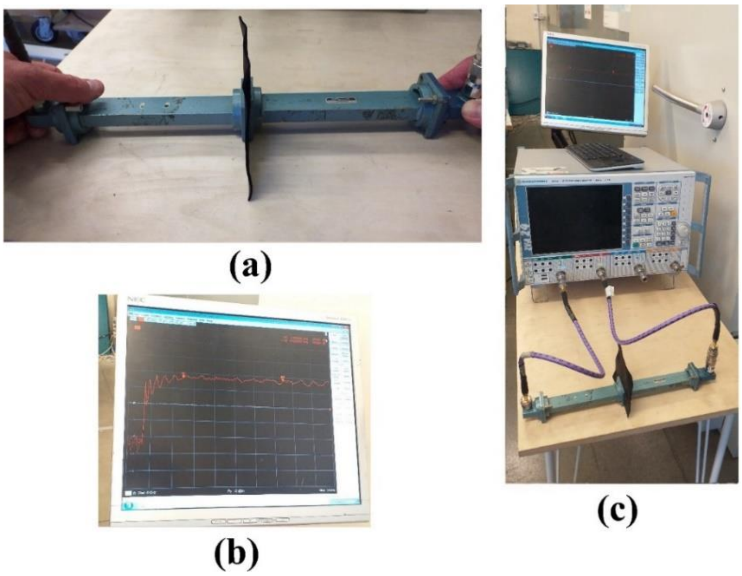
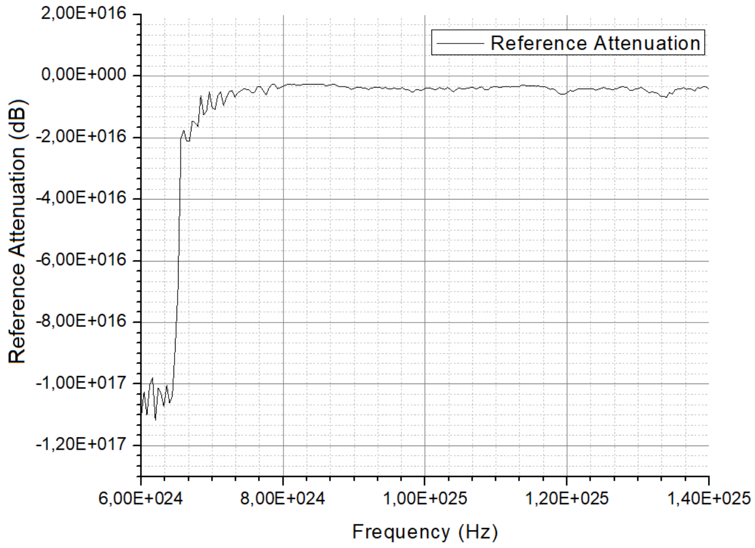


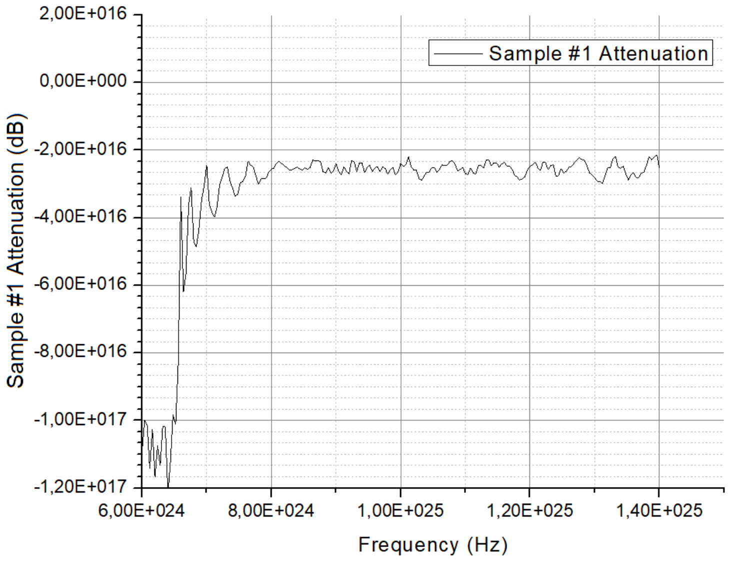
Figure 3. Waveguide testbed setup and VNA connections for initial calibration; (a) end-launcher and straight waveguide; (b) waveguide testbed; (c) final measurements setup

RESULTS

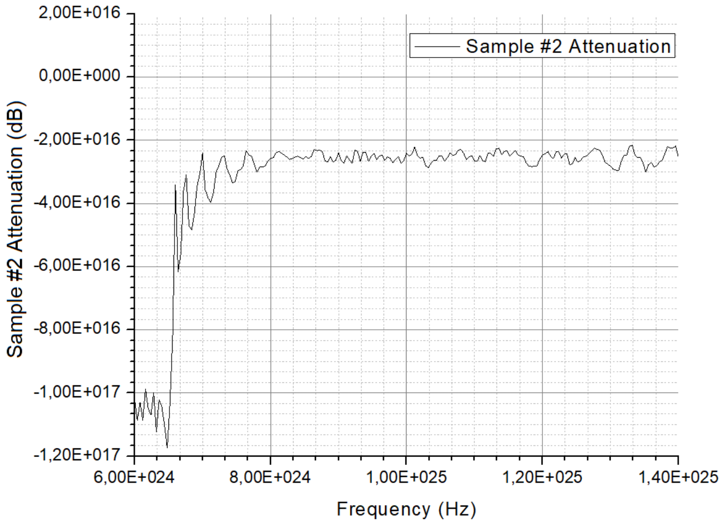
All leather samples were then measured with respect to their electromagnetic shielding properties using the testbed and methodology described in Section II above. The leather samples used and their measurements results are provided in Table 1.


Reference Attenuation
Reference Attenuation (dB)
2,00E+016
0,00E+000
-2,00E+016
-4,00E+016
-6,00E+016
-8,00E+016
-1,00E+017
-1,20E+017
6,00E+024
8,00E+024
1,00E+025
1,20E+025
1,40E+025
Frequency (Hz)

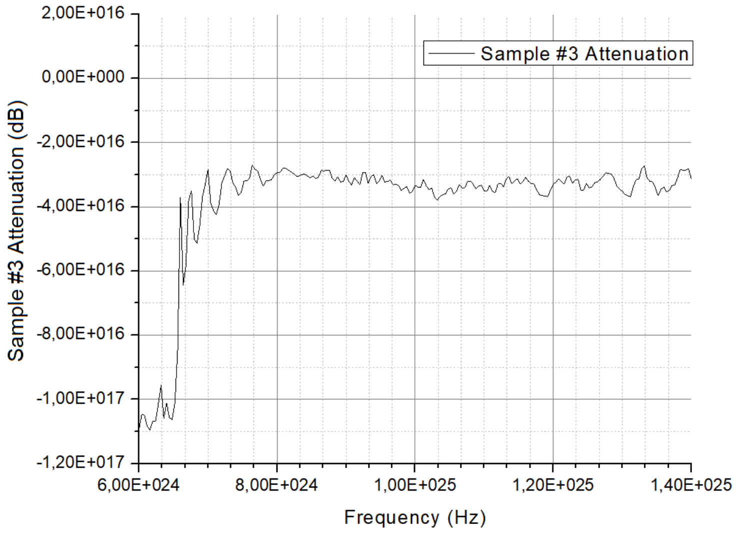
(a)


Sample #1 Attenuation
Sample #1 Attenuation (dB)
2,00E+016
0,00E+000
-2,00E+016
-4,00E+016
-6,00E+016
-8,00E+016
-1,00E+017
-1,20E+017
6,00E+024
8,00E+024
1,00E+025
1,20E+025
1,40E+025
Frequency (Hz)

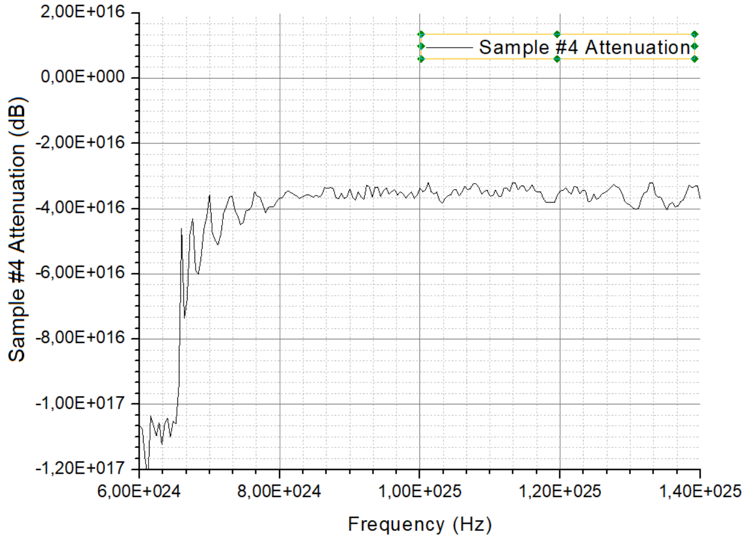
(b)


Sample #2 Attenuation
Sample #2 Attenuation (dB)
2,00E+016
0,00E+000
-2,00E+016
-4,00E+016
-6,00E+016
-8,00E+016
-1,00E+017
-1,20E+017
6,00E+024
8,00E+024
1,00E+025
1,20E+025
1,40E+025
Frequency (Hz)

(c)



(d)



(e)

Figure 4. Shielding measurements results of conductive leathers (a) reference, and (b) through (e) samples #1 through #4, respectively

First, the reference sample with no PPy treatment whatsoever was measured and the transmission coefficient (known as “S^{21}”) through the VNA and waveguide apparatus is displayed in Fig. 4(a). The horizontal axes of all sub-figures within Fig. 4 are in Hz units for the signal frequency, whereas the vertical axes are in dB. Since S^{21} refers to the transmission coefficients (output voltage to input voltage), the vertical axes in dB are calculated as 20*log (S^{21}), where S^{21} the transmission coefficient in linear scale.

Furthermore, please note that the nominal frequency range of operation for WR-90 waveguides is from 8.20 to 12.40 GHz. From Fig. 4(a) it can be deduced that there is a slight fluctuation of shielding vs. frequency; this is expected and attributed to the surface roughness and imperfections of the sample body. The transmission coefficients correspond to an attenuation of ~3 dB, which is rather small; this is also expected since the untreated leather does not inherently possess significantly high conductivity.

Then, all leather samples were also measured. The respective transmission coefficients are illustrated in Fig. 4(b) through (e) for the samples #1 through #4, respectively. Furthermore, Table 1 tabulates the average shielding effectiveness per sample for the entire 8.20-12.40 GHz frequency range.

It can be seen that for samples #1 and #2, with single *in situ* PPy polymerization, the shielding effectiveness is in the order of 25 dB. This represents a satisfactorily large number meaning that a single layer of PPy polymer on top of the reference sample is able to provide a significant attenuation since the output signal is in the order of ~17,8 times lower than the input signal; this corresponds to a power of the output signal that is ~ 300 times lower than the input signal.

Table 1: Measurements results of leather samples shielding effectiveness

No.	Leather samples	Thickness (mm)	Chemical treatment	Electrical resistance Ω/sq	Average Shielding effectiveness 8.20-12.40 GHz (dB)
0	Reference (white sheep crust leather (not treated)	1.015	Not Applicable	∞	-3.82
1	S_1	0.974	single *in situ* polymerization	23.90	-25.35
2	S_2	0.982	single *in situ* polymerization	21.42	-25.27
3	S_3	1.034	double *in situ* polymerization	7.27	-32.68
4	S_4	0.988	double *in situ* polymerization	8.43	-35.06

On the other hand, samples #3 and #4, with double *in situ* PPy polymerization exhibit a shielding effectiveness in the order of 34 dB, which corresponds to an output signal in the order of ~50 times lower than the input signal; this corresponds to an output signal power that is ~2500 times lower than the power of the input signal. It is also worth noting that the attenuation of samples #3 and #4 is almost 10 dB lower than in the case of samples #1 and #2. This attenuation corresponds to a significantly high level of shielding effectiveness and demonstrates our motivation in using the proposed approach for electromagnetic shielding using PPy conductive layering. It should be also noted that the higher shielding effectiveness of double polymerized samples is expected since double polymerization with a conductive layer corresponds to higher conductivity and, thus, higher microwave signal attenuation and electromagnetic shielding.

CONCLUSIONS

In this research we have studied the electrically conductive leather for efficient shielding from electromagnetic interference. The leathers were prepared with two different chemical treatments, single *in situ* polymerization and double *in situ* polymerization, by using polypyrrole (PPy) to inherit conductivity to leather surfaces. We have shown that PPy is sufficient treatment for inheriting very satisfactory electromagnetic shielding properties all the while maintaining the comparative advantages of a non-metal-processed fabric surface (e.g. comfortability, aesthetic appeal etc.). Measurements results were obtained by using RF and microwave high-frequency equipment in a controlled laboratory environment within the X-microwave band (8-12 GHz). In this study was presented that electromagnetic shielding is proportional to the number of PPy layers used for leather processing, by increasing the number of treatments the shielding effectiveness is increased as well. This is due to double polymerization with a conductive layer, which corresponds to higher conductivity and, thus, higher microwave signal attenuation and electromagnetic shielding.

The shielding effectiveness of samples treated with single *in situ* polymerization, show to have a shielding effectiveness in the order of 25 dB, while those treated with double *in situ* polymerization have a shielding effectiveness in the order of 34 dB, almost 10 dB lower than the first ones. We may conclude that 2500 times attenuation is a satisfactory result, which corresponds to a significantly high level of shielding effectiveness and demonstrates our motivation in using the proposed approach for electromagnetic shielding using PPy conductive layering.

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