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The Electrical Properties of Bast Fibre Fabrics Change after Nanoparticles of Metals and their Oxides are Applied

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***Abstract.* In the paper changes of electrical properties of bast fibre fabrics after deposition of metals and metal oxide nanoparticles have been investigated. Moisture content of samples after storing them in different climatic conditions was determined using the thermogravimetry method. Electrical resistance changes depending on moisture content were investigated using impedance spectorscopy.**

The analysis of measurements shows that samples with surface treated in plasma attract water better than samples with untreated surface. In turn metal coatings reduce water sorption. The research shows that coatings decrease electrical resistance of samples. Samples stored in normal climatic conditions can be characterized with capacitive/reactive resistance, ie. they have expressed dielectric properties but moist samples in low-frequency range have ohmic/active resistance, ie. they have expressed electric conductivity properties that turn into dielectric properties in high-frequency range. It can be concluded that electric resistance of bast fibre fabrics is influenced by moisture content in sample and coating material.

***Keywords:* bast fibres, impedance spectroscopy, metal coatings, thermogravimetry.**

I. Introduction

Functionalization of textile materials is widely used in the world, and that allows to change properties of textiles and use them in different areas. Textile materials with metal and oxide coatings is a research area of particular interest. Surface metallization and covering with metal oxides makes it possible to develop new products – for protection against radiation, conductive and antibacterial products etc. [1; 2; 3] Metal items are replaced by metallized textiles, as it reduces their weight and gives the fabrics characteristic properties, for example, pliability, air permeability etc. [4; 5; 6]

Mainly synthetic metallized fabrics are produced now because of their regular structure. But synthetic fibres are obtained from oil products, so they are not produced from renewable resources. That is why in recent years major attention has been paid to the use of natural resources. Natural fibres in comparison to synthetic fibres make it possible not only to reduce consumption of oil products significantly, but also harm the environment less. That is because natural materials degrade in nature and it is a renewable resource. Bast fibres are commonly used for technical needs because of their high strength. Natural textile materials are heterogeneous and hygroscopic. [7]Such characteristics conserve after metallization as well. So that properties of metallized textiles have to be investigated in relation to moisture content in the material. Production of such natural fibre fabrics would provide market for hemp and flax fibres obtained in Latvia. [8]

The goal of the work is to investigate changes in electric properties of bast fibre fabrics after the deposition of metals and their oxide nanoparticles.

II. Materials and methods

In the experimental part of the work, the following characteristics were determined:

• Moisture content was analysed by using thermogravimetry method

• Electrical properties were determined by using impedance spectroscopy method

Bast fibre fabric woven in SIA Limbažu Tīne Ltd. was investigated. Warps of the fabric are uncoloured and unbleached hemp fibre yarn, wefts – flax fibre yarn. Thickness of the fabric is 1,36 mm, mass of one m^2 – 510 g/m^2. Warp density in fabric is 22 warps/10 cm, weft density – 65 wefts/ 10 cm.

Fabrics were covered with Ni, NiO, Cu, CuO and Al coatings. Thin metal and their oxide coatings are applied using DC magnetron metal coating and reactive oxide coating obtaining methods. Only the P7 sample was covered in voltage mode (see the table), other samples were modified in power mode. A 500 nm Ni coating was applied to the P2 sample on both sides, for all other samples only one side was coated. For the P5 sample surface etching was performed and coating was not applied to be able to evaluate changes of fabric properties after cleaning the surface. The technical data of thin film deposition is given in Table.[9; 10; 11]

The thermogravimetry analysis was performed for the samples held in normal climatic circumstances -air relative humidity – 65±5%, temperature - 20±2°C and the samples held in environment with moisture content 95 – 98 %. The temperature amplitude was 35 – 230 ^{0}C and it increased linearly with speed of 10 ^{0}C/min. During the experiment mass loss (mg) was measuredevery second. [12; 13; 14]

Impedance spectroscopy measurements were carried out for bast fibre fabric samples without coating, for the etched sample, samples with Ni, Cu, Al, NiO and CuO coatings from one side and for the sample with Ni coating on both sides. The measurements were performed for the samples held in normal climatic circumstances and the samples were fixed in the measuring device with the coating upwards and downwards. The samples are considered to be moist, if they are held in exicator with relative air moisture content of 95-98 % for at least 24 hours. All measurements were performed by taking into account constant area of the measuring electrode of sample holder – 11,34 cm2. [15]

TABLE

TECHNICAL DATA FORDEPOSITION OF THIN COATINGS

Sample Number	Coating Material	Sides Covered	Power, W	Gas Flow, sccm	Dissipation Time, min	Thickness of the Applied Coating, nm
P1 (157)	Ni	I	580	Ar = 6,5	20	500
P2 (159)	Ni	I	580	Ar = 6,5	20	500
		II	580	Ar = 6,5	20	500
P3 (164)	NiO	I	580	Ar = 6,0 O_2 = 1,5	7	380
P4 (170)	Cu	I	350	Ar = 6,5	12	680
P5 (171)	Kodināts	I	-	Ar = 6,5	25	-
P6 (172)	Cu	I	350	Ar = 6,5	30	1700
P7 (195)	Al	I	56	Ar = 5,0	30	200
P8 (194)	Al	I	400	Ar = 5,0	20	970
P9 (179)	CuO	I	300	Ar = 5,0 O_2 = 2,0	15	595
P10(176)	CuO	I	300	Ar = 5,0 O_2 = 2,0	15	730

III. RESULTS AND DISCUSSION

Thermogravimetry and impedance spectroscopy measurement data are summarized and analysed.

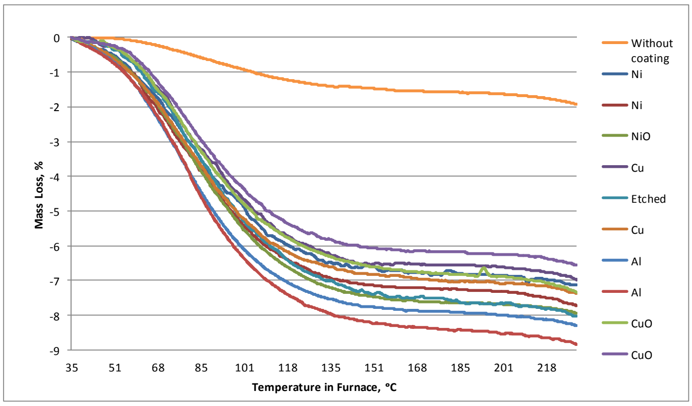

Fig. 1. Mass Loss of Samples Held in Normal Climatic Circumstances, % from Sample Mass,vs. Temperature Changes

Thermogravimetry measurements for the samples held in normal climatic circumstances are given in Figure 1. Mass losses are mainly influenced by ability of samples to attract water.For the P0 sample without coating much smaller mass losses are observed than in case of etched and coated samples.It can be seen that for the samples held in normal climatic circumstances the mass is rapidly decreasing until about 120 ^{0}C, mass decrease is smaller at higher temperatures. For the P0 sample decrease of mass is flatter and more uniform.

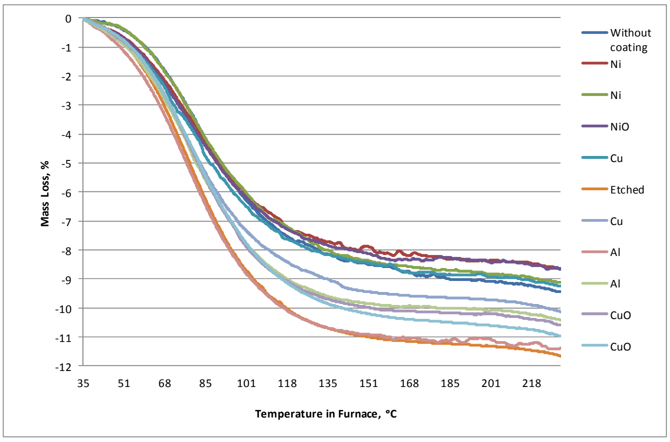

Fig. 2. Mass Loss of Moist Samples, % from Sample Mass, vs. Temperature

Mass loss % of moist samples under temperature influence can be seen in Figure 2. After holding in air moisture of 95 – 98 % mass of the P0 sample decreases similarly to mass of other samples.

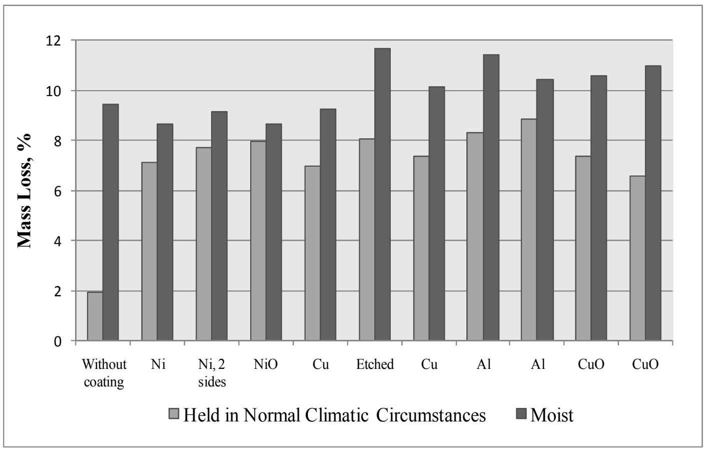

Fig. 3. Mass Losses of Samples Held in Normal Climatic Circumstances and moist samples, % from Sample Mass

In Figure 3 mass loss % for samples held in normal climatic circumstances and wet samples are compared. Mass reduction can be mainly explained by evaporation of moisture in the samples. For the P0 sample without coating much smaller mass losses are observed than in case of coated samples and etched sample. For the wet P0 sample without coating mass losses are similar to other samples. The P0sample after holding in normal climatic circumstances and heating up to 230 ^{0}C loses 2 % from its mass but after holding in environment of air moisture of 95 – 98 % it loses 9,5 % of its mass. Mass losses are influenced by surface cleaning of the samples, etching before deposition of metal coatings – that is because the surface is becoming active and attracts more water. It can be concluded that in normal climatic circumstances etched samples attract water much better than the samples without coating. Mass losses for etched samples held in normal climatic circumstances are 8 % that is a little bit less than mass losses for samples with aluminium coating. The largest mass losses are observed for wet etched samples–

almost 12 %. Samples with CuO coating have one of the lowest mass losses after holding in normal climatic circumstances. Whereas for wet samples with CuO coating mass losses are one of the largest, ie. 10,5 – 11%. The samples with Ni and NiO coatings mass losses increase only slightly after holding them in moisture content of 95 – 98 %.Their mass losses after holding in normal climatic circumstances are 7 – 8% but after holding in moisture content of 95–98 % they are 8,5–9%.[13; 14]

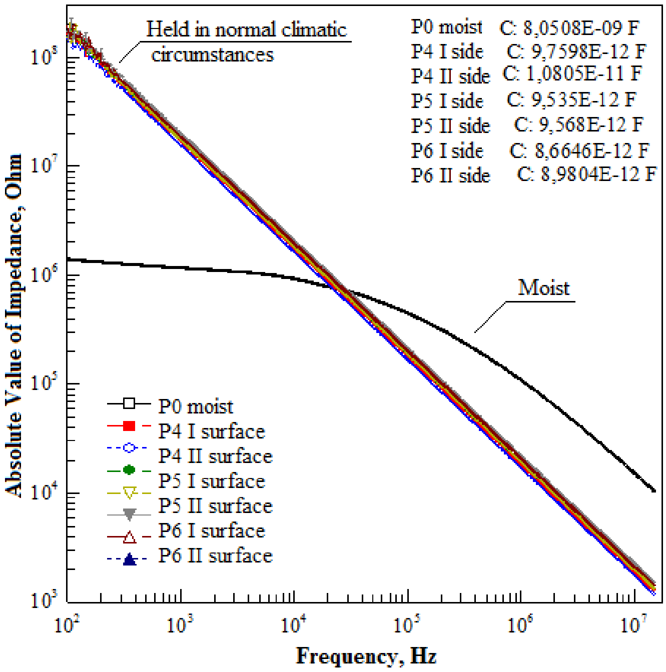

Fig. 4. Bode Plot, in which Absolute Value of Impedance |Z| Depending on Frequency is Shown for Wet Samples and Samples Held in Normal Climatic Circumstances Before Surface Modification

Electrical properties of coated samples, as well as their changes under the influence of moisture are examined by using impedance spectroscopy. Bode plots are analysed, in which absolute value of impedance vs. frequency in range from 100 Hz to 15 MHz is shown.

In Bode plot (Figure 4) impedance measurements for samples before surface modification are shown depending on frequency. Resistance of samples held in normal climatic circumstances decreases in inverse ratio to frequency from 200 MΩ to 10 kΩ. It shows that bast fabric has reactive or capacitive resistance that is characteristic for dielectrics. It is imaginary part of complex resistance that doesn’t consume energy but stores it and later gives it back to the electric circuit. Reactive resistance for alternating current is created by capacity. In Figure 4 capacity values are shown for samples without coatings. The fact that reactive resistance is characteristic of samples held in normal climatic circumstances is shown by capacity values from 8,7to 10,8 pF. The P0 sample is measured after holding it in air with moisture content of 95 - 98 %. It can be seen that the wet sample has the smallest resistance 2 - 1 MΩ, at lower frequencies – ohmic resistance is larger than reactive/capacitive resistance in comparison to samples held in normal climatic circumstances. As frequency increases above 50 kHz resistance of wet sample decreases from 2 MΩ to 10 kΩ. For a wet sample at low frequencies resistance decreases only slightly. It shows that active or ohmic resistance dominates up to frequency of 50 kHz that reflects conductivity. At about 50 kHz ohmic resistance of a wet sample becomes comparable to capacitive resistance. As frequency increases above 50 kHz, also in a wet sample capacitive resistance starts to be the dominating one. This sample accordingly has much larger capacity than samples held in normal climatic circumstances - 8,1 nF. So that increased moisture content in fabric gives it an active resistance at low frequencies.

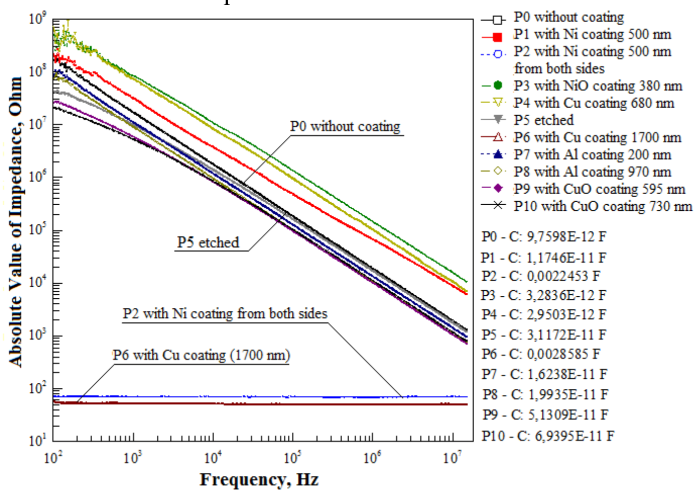

Fig. 5. Bode Plot for Samples Held in Normal Climatic Circumstances

In Figure 5 absolute value of impedance vs. frequency is shown for all samples held in normal climatic circumstances. Among results of other samples significant difference can be observed for two samples. The P2 sample with Ni coating from both sides and the P6 sample with particularly thick Cu coating (1700nm) from one side has small active resistance that is not dependant from frequency. Resistance for the P2 sample with Ni coating from both sides is only 90 Ω but for the P6 sample with a thick Cu coating resistance is 80 Ω. Capacity of P2 and P6 samples is close to 0. So metallic conductivity is characteristic for these samples. Capacitive resistance is characteristic for the other samples that in frequency range 100 Hz – 15 MHz decreases from 100 MΩ to 1 kΩ. It can be seen that the P1 sample with Ni coating, P3 sample with NiO coating and P4 sample with Cu coating has larger resistance than the P0 sample without coating and P5 – the etched sample. Conformity of resistance of the P0 and P5 sample indicates that moisture influences resistance equally. Such a result was influenced by the fact that P0 and P5 samples were held in normal climatic circumstances but P1, P3 and P4 samples were tested right after deposition of metal particles and they were dried beforehand. It confirms the fact that moisture substantially influences resistance but its influence is reduced by metal particle coatings and their different influence. The |Z| value for different samples is related to different moisture content in these samples. The largest capacity (without taking into account samples with ohmic resistance) 51,3and 69,4 pF is for P9 and P10 samples with CuO coating. They also have lower resistance that decreases from 13 MΩ to 1 kΩ, resistance of the sample with thicker CuO coating is a little bit smaller. Capacity is slightly smaller for samples No. P5 (31,2 pF), P7 (16,2 pF) and P8 (19,9 pF). Capacity of samples with larger reactive resistance is from 11,7pF to 2,9 pF.

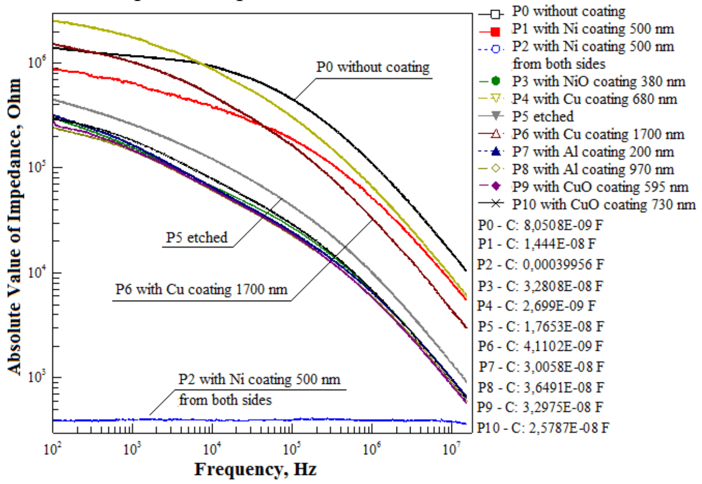

Fig. 6. Bode Plot for Wet Samples

In Figure 6the absolute value of impedance depending on frequency is shown for wet samples. Unlike the previous Bode plot with samples held in normal circumstances, here active (ohmic resistance can be observed just for the P2 sample with Ni coating from both sides. After storage in the air with moisture content of 95-98 % resistance in all frequency range is 600 Ω. It shows that for this sample after holding it in air with moisture content of 95-98 % metallic conductivity persists. For the other wet samples at low temperatures ohmic resistance dominates but, as frequency increases, capacitive resistance starts to dominate. Wet samples have a larger capacity, it can be several tens of nF. It shows that resistance decreases under the influence of moisture. The samples with CuO, NiO and Al coatings have larger capacity, 36,5 – 25,8 nF, and for the sample with Ni coating and etched sample it is 17,6 – 14,4 nF. For these samples smaller lower resistance is observed. Larger capacity corresponds to smaller ohmic resistance.

In Figure 7 all measurements of absolute value of impedance are shown for samples held in normal climatic circumstances and in moisture content of 95-98 %. Reactive resistance is characteristic for samples held in normal climatic circumstances. That shows dielectric properties of bast fibre fabric and ability to store electric charge. For wet samples at low frequencies active resistance dominates that characterizes electric conductivity. As frequency increases to 1 MHz their resistance becomes capacitive. For the P2 sample with Ni coating from both sides active resistance is observed in all frequency range. After holding in moisture content of 95 – 98 % its active resistance increases for about 510 Ω. Active resistance characteristic for the sample P6 held in normal climatic circumstances disappears after holding it in air with moisture content of 95 - 98 %. It changes from ohmic to capacitive resistance.

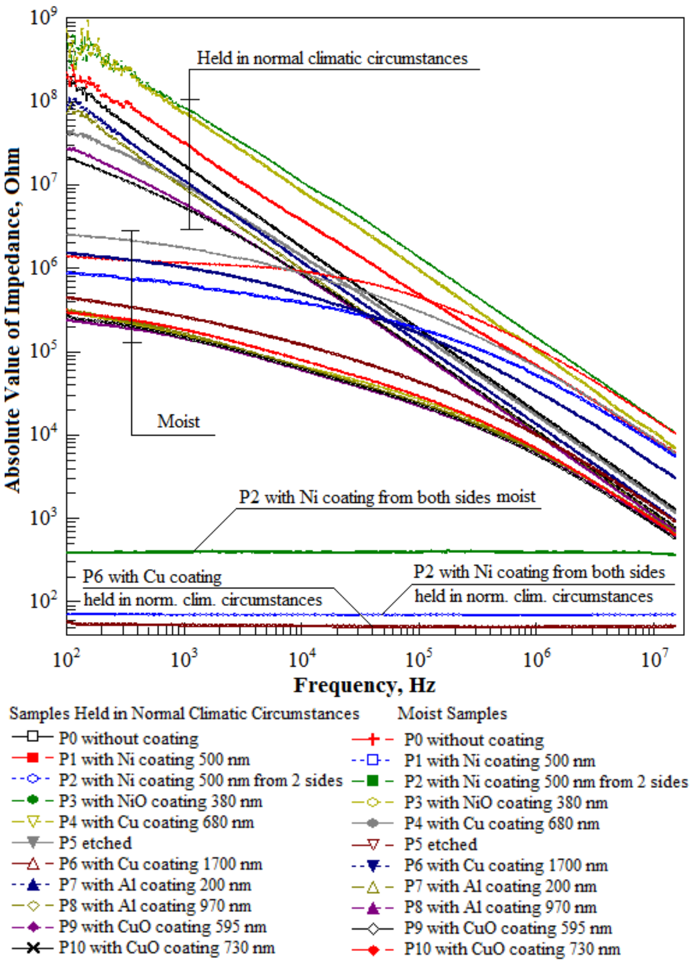

Fig. 7. Absolute Value of Impedance vs. Frequency for Wet Samples and Samples Held in Normal Climatic Circumstances

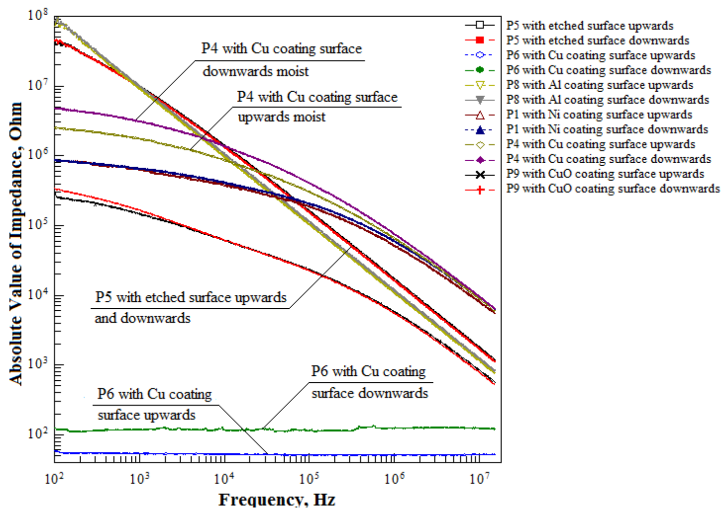

Fig. 8. Measurements of Some Wet Samples and Samples Held in Normal Climatic Circumstances with Coated Surface Upwards and Downwards

In Figure 8 plot of impedance measurements can be seen for samples depending on the position of the coated surface. Impedance |Z| depending on position of the coated surface is related to different surface resistances. As for the sample No. P5 only surface etching was performed its measurements are not dependent on the position, how the sample is fixed in the sample holder. Whereas for samples with coating a tendency can be observed – resistance is a little bit smaller, when measuring sample with coated surface upwards. It can be explained by the fact that metal and oxide coatings reduce resistance and that's why difference of resistance is observed, when measuring the coated and uncoated surface. [15]

IV. CONCLUSIONS

Electric properties of bast fibres and their possible depositions for technical needs have been investigated recently. As new technologies are developing in electronic and energetic fields, there is a need for new materials that could be used both as components of electric systems and protection means. [5; 6; 8; 12] Changes in physical properties of bast fibre fabric have been investigated after the deposition of metal and their oxide nano particles. Samples with Ni, NiO, Cu, CuO and Al coatings have been created and for one sample only surface cleaning was performed. Physical properties have been determined for samples with/without surface modification, samples held both in normal climatic circumstances and air with moisture content of 95 - 98 %.

After performing all the tasks it was concluded that:

- In the following experiments a fabric with larger density of warps and wefts and a smaller linear density should be chosen to get more precise results.
- Metal coating slightly increases electric conductivity of bast fibre fabrics but conductivity is more influenced by moisture content because of high hygroscopic properties of bast fibres.
- High ability to absorb water is promoted by the etching process that is necessary to provide attraction of metal particles.
- To obtain conductive, low density fabrics it is necessary to apply metal coating from both sides. In this case resistance slightly increases because of moisture, but the resistance stays active.
- By applying a thick metal layer from one side in normal climatic circumstances the fabric is conductive but under the influence of moisture in a low-frequency range the resistance increases and becomes a dielectric resistance in a high-frequency range.

The shortcoming of these experiments is unevenness of linear density of the fabric's warps and wefts and high porousness of the surface. The investigated fabric should be as homogenous and dense as possible to be able to compete with synthetic metallized textile materials.

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Uļjana Sidoroviča, Ilze Baltiņa, Andrejs Lūsis, Jānis Zandersons. Lūksnes šķiedru auduma elektrisko īpašību izmaiņas pēc metālu un to oksīdu nanodaļiņu uznešanas
Attīstoties jaunām tehnoloģijām elektronikas un enerģētikas jomās, kļūst aktuāla jaunumaterial nepieciešamība, kurus varētu izmantot gan kā elektrisko sistēmu sastāvdaļas, gan kā aizsardzības līdzekļus. Virsmas metalizācija vai pārklāšana ar metāla oksīdiem ļauj veidot jaunus produktus - aizsardzībai pret starojumu, elektrovadošus, antibakteriālus u.c. Ar metalizētiem tekstilmateriāliem aizstāj metāla izstrādājumus, tie ir vieglāki un saglabā tekstilijām raksturīgās īpašības, piem., lokanību, gaiscaurlaidību u.c. Šobrīd ražo galvenokārt metalizētus sintētiskos materiālus, to vienmērīgās struktūras dēļ. Taču sintētiskās šķiedras iegūst no naftas produktiem, kuru krājumi ar katru gadu samazinās. Tādēļ pēdējo gadu laikā liela uzmanība pievērsta dabīgo resursu izmantošanai. Galvenā priekšrocība ir tāda, ka, metalizējot dabīgo šķiedru tekstilmateriālus, iegūst videi draudzīgus produktus, kas dabā noārdās un ir pārstrādājami.
Eksperimentālajā daļā pētīts, kā mainās lūksnes šķiedru auduma elektriskās īpašības pēc metālu un to oksīdu nanodaļiņu uznešanas. Izmantojot termogravimetrijas metodi, pēc izturēšanas dažādos klimatiskos apstākļos noteikts mitruma saturs pētāmajā materiālā. Ar impedances spektroskopijas metodi izpētīts, kā mainās paraugu elektriskā pretestība atkarībā no mitruma satura.
Veiktie pētījumi parāda, ka paraugi, ar plazmā attīrītu virsmu, daudz labāk piesaista ūdeni, nekā paraugs ar neapstrādātu virsmu. Savukārt metāla pārklājumi samazina ūdens piesaisti. Izpētīts, ka pārklājumi samazina paraugu elektrisko pretestību. Normālos klimatiskos apstākļos izturētiem paraugiem raksturīga kapacitatīvā/reaktīvā pretestība - ir izteiktas dielektriskās īpašības, bet mitriem paraugiem pie zemām frekvencēm ir omiskā/aktīvā pretestība - ir izteiktas elektrovadāmības īpašības, kas pie augstām frekvencēm pāriet dielektriskās. Tātad lūksnes šķiedru auduma elektrisko pretestību ietekmē mitruma saturs materiālā un pārklājuma materiāls.

Ульяна Сидорович, Илзе Балтыня, Андрей Лусис, Янис Зандерсонс. Изменения электрических свойств лубяной ткани после нанесения металлических и металлооксидных наночастиц
С развитием новых технологий в области электроники и энергетики появляется необходимость в новых материалах, которые могут быть использованы в качестве компонентов электрических систем и средств защиты. Металлизация поверхности или её покрытие оксидами металлов позволяет создавать новые продукты - защищающие от излучений, электропроводные, антибактериальные и т.д. Металлизированными текстильными материалами заменяют металлические изделия, они легче и сохраняют свойства текстиля, например, гибкость, воздухопроницаемость и т.д. В настоящее время производят в основном металлизированные синтетические материалы, из-за однородности структуры. Однако, синтетические волокна производят из нефтепродуктов, запасы которых уменьшаются с каждым годом. Поэтому в последние годы большое внимание уделяется использованию природных ресурсов. Основным преимуществом является то, что, металлизируя ткани из натуральных волокон, производятся экологически чистые продукты, которые подлежат естественному разложению и переработке.
В экспериментальной части исследованы изменения электрических свойств лубяной ткани после нанесения металлических и оксидных наночастиц. Используя метод термогравиметрии, определено содержание влаги в исследуемом материале, после выдерживания в различных климатических условиях. С помощью импедансной спектроскопии изучены изменения электрического сопротивления образца в зависимости от влажности.
Проведённые исследования показывают, что образцы, поверхность которых очищена в плазме, гораздо лучше впитывают влагу, чем образцы с необработанной поверхностью. В свою очередь металлические покрытия снижают эту способность. Исследовано, что покрытия уменьшают электрическое сопротивление образцов. Образцам, выдержанным в нормальных климатических условиях, характерно ёмкостное / реактивное сопротивление, т. е. присущи диэлектрические свойства. В отличие от этого, влажным образцам характерно омическое / активное сопротивление, т. е. им свойственна электропроводность, которая при высоких частотах переходит в диэлектрические свойства. Следовательно, электрическое сопротивление лубяной ткани зависит от влажности материала и его покрытия.