SYNTHESIS OF GRAPHENE OXIDE ON COTTON FABRIC FOR MANUFACTURING FLEXIBLE SUPERCAPACITOR ELECTRODE

SINTEZA OXIDULUI DE GRAFENĂ PE SUBSTRAT DE BUMBAC PENTRU REALIZAREA DE ELECTROZI DESTINAȚI SUPERCONDENSATOARELOR

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Abstract: *Flexible supercapacitors (SCs) may be an adequate solution of energy harvesting with various applications. This paper presents a manufacturing method for SC electrodes, via synthesizing Graphene-Oxide (GO) on cotton fabrics by Hummers' method. GO has an especially high active surface, suitable for SC's electrodes. The surface conductivity of the electrodes was measured via the four-point measuring method and the reflectance spectra of materials were recorded in visible range to show the change in optical properties due to oxidation and reduction. The spectral confirmation of chemical processes involved by Hummer method and the surface conductivity of* $1.25 \cdot 10^{-3}$ *S/m represent promising results to further develop flexible SCs.*

Keywords: supercapacitors, flexibility, energy harvesting

Rezumat: *Supercondensatorii (SC) flexibili pot reprezenta o soluție adecvată pentru alimentarea cu energie electrică pentru diferite aplicații. Articolul*

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prezintă o metodă de realizare a electrozilor pentru SC, prin sinteza de oxid de grafenă (GO) pe substraturi din bumbac, pe baza metodei Hummer. GO are o suprafață activă mare, utilă în realizarea electrozilor. Conductivitatea de suprafață a electrozilor a fost determinată prin metoda de măsurare în patru puncte, iar spectrul de reflectanță al materialelor a fost achiziționat în domeniul vizibil cu scopul de evidențiere a modificării proprietăților optice în urma oxidării și a reducerii. Confirmarea pe cale spectrală a proceselor chimice implicate de metoda Hummer, precum și conductivitatea de suprafață de 1.25·10^{-3} S/m, reprezintă rezultate bune pentru dezvoltări viitoare de SC.

Cuvinte cheie: supercondensatoare, flexibilitate, generare energie electrică

1. Introduction

Energy harvesting via flexible and eco-friendly materials is an important topic within the European context. Flexible supercapacitors (SCs) are characterized by very high charging and discharging rates and a very large number of charge-discharge cycles (over 100,000 cycles). Flexible SC may be used in a wide range of applications, including powering wearable electronics, bendable and foldable devices, or flexible sensors, actuators and antennas. Attempts have been made to design materials exhibiting good electrochemical properties combined with flexibility and lack of toxicity that could be compatible with textiles. Storing energy in flexible electronics is crucial for the supply of integrated components.

Carbonaceous materials such as activated carbon, carbon nanotubes and graphene are extensively studied as electrically conductive compounds due to their low cost, high specific surface area, chemical inertness and compatibility with a wide range of materials [2, 3, 4, 5, 6, 7]. Graphene is a promising carbon material and the structural unit of allotropes like graphite, carbon nanotubes and fullerenes. Even if its sheets restack due to the Van der Waals interactions between them, thus reducing its coulombic efficiency [3], it exhibits remarkable mechanical, thermal and electrochemical properties. Another important feature of graphene is its ability to chemically bind to textiles [2]. Also, [8] showed that it is compatible with chitosan, a biopolymer with antibacterial properties that can act as a binder in the manufacturing of electrodes. Upon treatment with strong oxidisers like potassium perchlorate and fuming nitric acid, it leads to the formation of graphene oxide (GO), an important derivative, which can act both as a semiconductor and insulator, depending on the degree of oxidation [2]. By reducing GO, a material that is similar to graphene, reduced graphene oxide (rGO), can be synthesized. This

is commonly done by Hummers' method [1, 9, 10]. The method consists in the use of strong oxidizing couples like potassium permanganate ($KMnO_4$) – sulfuric acid (H_2SO_4). In this work, rGO was synthesized using Hummers' method, for the development of textile-based, flexible SC. The achieved SC is meant for energy supply of integrated devices of smart textiles and wearable products.

2. Materials and methods

The experimental part included the manufacturing of the electrode for the subsequently assembly of the SC. The two electrodes were manufactured by synthesis of GO on the surface of cotton fabrics. The scoured cotton fabrics had a specific mass of 326 g/m^2, a thickness of 0.73 mm and a yarn's density of 340 yarns / 10 cm in warp and 220 yarns / 10 cm in weft. GO was synthesized on the surface of the cotton fabric via the Hummer method. Fine graphite powder (particle size < 100 μm) was purchased from Merck. Sulfuric acid (H_2SO_4, 95-97%) was purchased from Fluka. Potassium permanganate ($KMnO_4$) was purchased from Sigma-Aldrich. Hydrogen peroxide (H_2O_2, 30%) and hydrochloric acid (HCl, 37%) were supplied by Consors SRL.

2.1. Synthesis of graphene oxide on cotton for the electrodes

Two grams of graphite powder was added to a vessel containing 25.5 ml of 95-97% H_2SO_4 and it was left under magnetic stirring for 23 h (Figure 1).

Figure 1. Equipment used in the synthesis of graphene oxide; digital magnetic stirrer and flask containing the reaction mixture

Then, one gram of graphite and another 25.5 ml of 95-97% H_2SO_4 were added to the mixture. The vessel was immersed in a bigger vessel containing crushed ice and water. Next, 7 g of $KMnO_4$ was added and the mixture was stirred at 50 °C for 2 h. Then, 140 ml of H_2O and 10 ml of H_2O_2 were added and the obtained mixture was stirred for 30 min. The obtained GO was filtered through a filter paper overnight and 200 ml of 5% HCl was subsequently added. The resulting mixture, having a

*p*H of 1, was homogenized using a glass rod, ultrasonicated for 15 min and subsequently kept under magnetic stirring. Then, it was filtered using filter paper and washed with 200 ml of ultrapure H_2O, thus raising the pH to 3. A total of seven filtrations and six washings were performed, leading to *p*H 4 (Figure 2). Finally, the obtained GO was dried at 90 °C for 30 min.

Figure 2. Filtration of the mixture using filter paper

2.2 Preparation of the GO-coated fabric

Five fabric samples were dipped into a 50 ml solution of GO for 30 min. Then, the coated samples were dried at 50 °C for 30 min. The dipping and the drying process were performed five times to increase the GO adsorption.

2.3 Preparation of the rGO-coated fabric

Two solutions containing reducing agents were prepared – one sodium dithionite ($Na_2S_2O_4$) solution (100 ml, 25 mM) and one ascorbic acid ($C_6H_8O_6$) solution (100 ml, 25 mM). The obtained solutions were used on the coated fabrics at 95 °C for 1 h. Two cotton fabric samples were reduced with sodium dithionite and three samples with ascorbic acid. To remove the excess reducing agents, the samples were washed with ultrapure H_2O five times and then dried at 90 °C for 30 min.

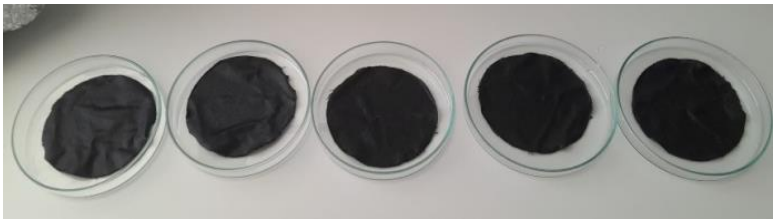

Figure 3. Graphene-coated fabrics obtained using $Na_2S_2O_4$ and $C_6H_8O_6$ as reducing agents

The manufactured GO-reduced samples were further characterized regarding physical-mechanical properties, electrical surface and bulk conductivity and spectral analysis.

3. Results and Discussion

3.1. Physical-mechanical properties

The physical-mechanical properties of the samples in a raw state, before the reduction and after the reduction were analysed, in terms of specific mass, yarn's density in warp and weft and sample thickness. Figure 4 presents the increase of the specific mass from the raw state of the sample with 326 g/m^2, up to 333 g/m^2 for the sample before reduction and 343 g/m^2 for the sample after reduction. This behaviour is given by the additional coating material deposited after each treatment.

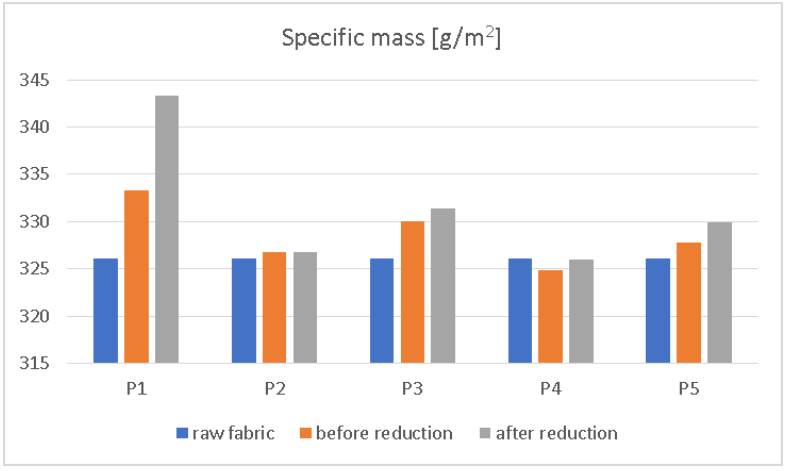

Figure 4. Comparison between raw fabric, fabric before reduction and fabric after reduction in terms of specific mass

Figures 5 and 6 present the contraction of the fabric in terms of warp and weft yarn's density, which is proven especially by the tighter warp yarn's density after each treatment.

The thickness of the samples increases with each additional treatment, due to the coating of the surface, with about 0.2 mm.

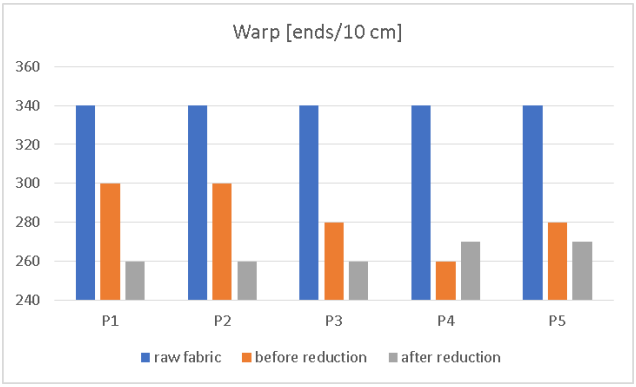

Figure 5. Comparison between raw fabric, fabric before reduction and fabric after reduction in terms of yarns density in warp

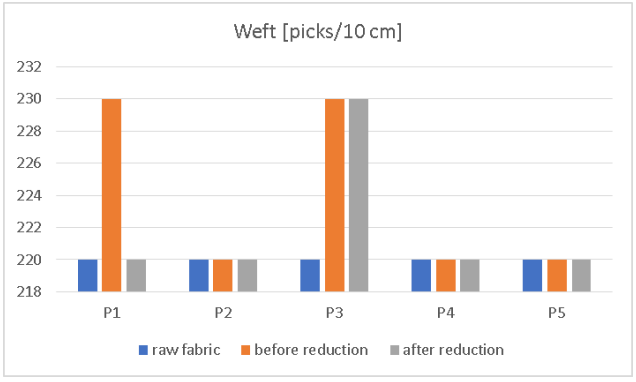

Figure 6. Comparison between raw fabric, fabric before reduction and fabric after reduction in terms of yarns density in weft

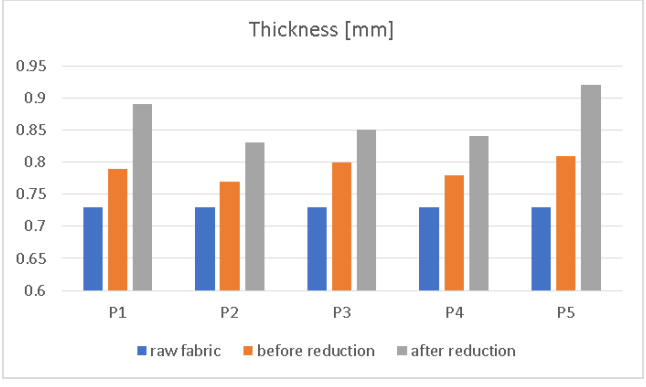

Figure 7. Comparison between raw fabric, fabric before reduction and fabric after reduction in terms of thickness

3.2. Electrical measurements of the electrode

The electrical conductivity of the textile electrode was measured via two methods:

- **The first method** was via a four-point probe measurement system (distance between outside pins 3.58 cm – surface conductivity;
- **The second method** was via two electrodes and a cut sample with dimensions of 2x4 cm^2 and 2x2 cm^2 – bulk conductivity.

First method – surface conductivity

The circular electrode sample was fastened onto the four-point probe measurement system (Figure 8). The experimental setup consisted of PC, Keithley 2700 – multimeter, measurement electrodes and HP 4145B Semiconductor Parameter Analyzer (Figure 9).

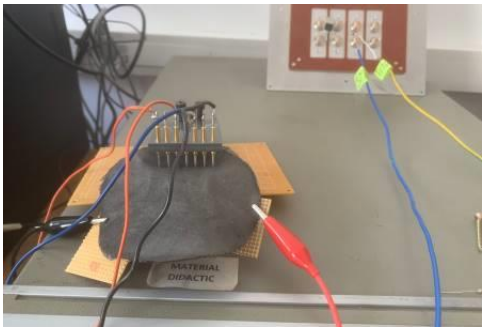

Figure 8. Four-point probe measurement of the SC electrode

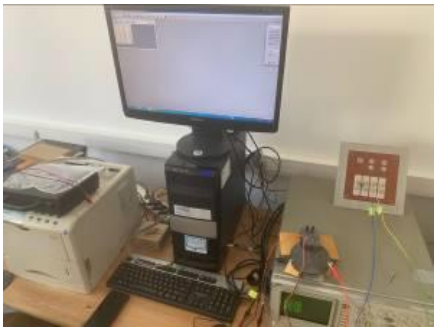

Figure 9. Experimental setup

The physical principle of four-point probe measurement of electric resistance R = V/I, is given by applying a certain electric current between the outer electrodes and measurement of an electric voltage between the inner electrodes. The schematic drawing of the four-point probe measurement system is illustrated in Figure 10.

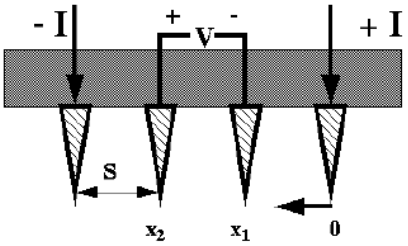

Figure. 10. Schematic drawing of the four-point probe measurement system [11]

To compute the surface resistivity from the measured electric resistance, the following relation was applied, in correlation with the geometry of the measurement electrodes:

$$\rho_s = \frac{\pi g}{\ln 2}\left(\frac{V}{I}\right) \quad (1)$$

[1]

where: ρ_s – surface electric resistivity [Ω/m];

V – electric voltage measured [V];

I – electric current applied [A];

g – fabric thickness [m].

The electrical surface conductivity is inversely proportional to the electric surface resistivity (2).

$$\sigma_s = \frac{1}{\rho_s} \quad (2)$$

σ_s - surface electrical conductivity [S/m].

The following results were obtained for the surface electric conductivity of the SC electrode fabric (table 1):

Table 1. **Surface conductivity of the electrode**

IF1P [A]	VF1P [V]	VS1P [V]	VS2P [V]	R [Ω]	Thickness substrate - g [m]	Surface resistivity - ρ_s [Ω·m]	Surface conductivity – σ_s [S/m]
-	2.00E-02	5.00E-03	3.70E-03	-	0.00073	-	-
1.00E-07	2.19E+00	5.16E-02	3.16E-02	200000	0.00073	664.4058	0.001505
2.00E-07	4.71E+00	1.03E-01	5.60E-02	233990	0.00073	777.3216	0.001286
3.00E-07	7.51E+00	1.62E-01	8.42E-02	258026	0.00073	857.1721	0.001166
4.00E-07	9.98E+00	2.24E-01	1.17E-01	267525	0.00073	888.7258	0.001125
5.00E-07	1.21E+01	2.76E-01	1.50E-01	252200	0.00073	837.8157	0.001193
6.00E-07	1.43E+01	3.24E-01	1.78E-01	244000	0.00073	810.5751	0.001233
7.00E-07	1.68E+01	3.76E-01	2.01E-01	249428	0.00073	828.6089	0.001206
8.00E-07	1.95E+01	4.35E-01	2.29E-01	257362	0.00073	854.9657	0.001169
9.00E-07	2.00E+01	4.54E-01	2.40E-01	237455	0.00073	788.8342	0.001267
1.00E-06	2.00E+01	4.55E-01	2.43E-01	212590	0.00073	706.2301	0.001415

The electric current intensity was varied between 0.1 μA and 1.0 μA (IF1P) (11 measurements) and the electric voltage was measured for each of

these values (VS1P-VS2P). The measured electric resistance was computed as ratio between the voltage and the current, while the surface resistivity and surface conductivity according to relations (1) and (2). We can conclude an average surface conductivity of $1.25 \cdot 10^{-3}$ S/m.

Second method – bulk conductivity

A sample of 2x2 cm^2 was cut from the electrodes fabric and fixed at both ends with copper electrodes. The electric voltage was measured at a given current intensity in the range of 0.01 μA and 0.05 μA. Figure 11 shows the cut fabric sample of the SC electrode.

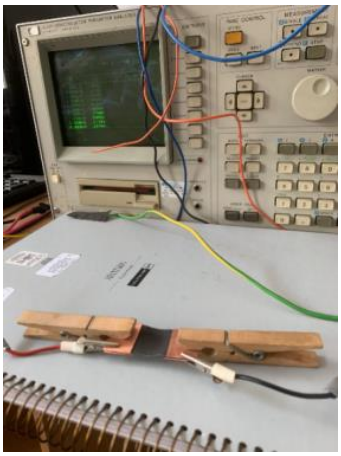

Figure 11. Image of the 2x2 cm^2 sample and the copper electrodes

The following classic relation for the bulk electric resistivity was applied:

$$\rho_b = \frac{U}{I} \cdot \frac{D \cdot g}{L} = R \cdot \frac{D \cdot g}{L} \quad (3)$$

where: ρ_b – bulk resistivity [Ω·m];

U – measured voltage [V];

I – applied current [A];

D – distance between electrodes (fabric length) [m];

L – width of the electrodes (fabric width) [m];

g – fabric thickness [m].

By applying the relation (3) we got the following results in Table 2 (by considering $D = L$):

The average value for the bulk conductivity of sample 2x2 cm^2 is $4.833 \cdot 10^{-5}$ S/m. For the 4x2 cm^2 fabric sample same considerations apply. Table 3 presents the measured results and computations for this sample (with $D=2L$).

The average value for the bulk conductivity of sample 4x2 cm^2 is $3.898 \cdot 10^{-5}$ S/m. Both the surface conductivity ($1.25 \cdot 10^{-3}$ S/m) and the bulk conductivity ($4.833 \cdot 10^{-5}$ S/m and $3.898 \cdot 10^{-5}$ S/m) are promising results for the GO synthesized SC electrode.

Table 2. **Bulk electric conductivity of the sample 2x2 cm^2 [S/m]**

IA [A]	VA [V]	Resistance R [Ω]	Thickness g [m]	Resistivity ρ_b [Ω·m]	Conductivity σ_b [S/m]
0.00E+00	-3.50E-02	-	0.00073	-	-
5.00E-09	1.83E-01	36600000	0.00073	26718	3.7428E-05
1.00E-08	2.26E-01	22600000	0.00073	16498	6.06134E-05
1.50E-08	4.37E-01	29133333	0.00073	21267.33	4.70205E-05
2.00E-08	5.51E-01	27549500	0.00073	20111.14	4.97237E-05
2.50E-08	6.75E-01	26999600	0.00073	19709.71	5.07364E-05
3.00E-08	8.22E-01	27399667	0.00073	20001.76	4.99956E-05
3.50E-08	9.80E-01	27999429	0.00073	20439.58	4.89247E-05
4.00E-08	1.13E+00	28200000	0.00073	20586	4.85767E-05
4.50E-08	1.35E+00	30000000	0.00073	21900	4.56621E-05
5.00E-08	1.54E+00	30700000	0.00073	22411	4.46209E-05

Table 3. **Bulk electric conductivity of the sample 4x2 cm^2 [S/m]**

IA	VA	Resistance R [Ω]	Thickness g [m]	Resistivity ρ_b [Ω·m]	Conductivity σ_b [S/m]
0.00E+00	4.00E-03	-	0.00073	-	-
5.00E-09	2.90E-01	57998000	0.00073	21169.27	4.72E-05
1.00E-08	4.30E-01	42999000	0.00073	15694.64	6.37E-05
1.50E-08	9.85E-01	65665333	0.00073	23967.85	4.17E-05
2.00E-08	1.58E+00	79100000	0.00073	28871.5	3.46E-05
2.50E-08	2.00E+00	80160000	0.00073	29258.4	3.42E-05
3.00E-08	2.39E+00	79796667	0.00073	29125.78	3.43E-05
3.50E-08	2.81E+00	80371429	0.00073	29335.57	3.41E-05
4.00E-08	3.25E+00	81347500	0.00073	29691.84	3.37E-05
4.50E-08	3.68E+00	81844444	0.00073	29873.22	3.35E-05
5.00E-08	4.18E+00	83676000	0.00073	30541.74	3.27E-05

3.3 Spectrophotometric analyses

The graphene sheets oxidation of pristine powder during Hummers' GO synthesis was optically manifested by the brown colour occurrence in the suspension prepared [12, 13]. This feature was conferred to the textile materials treated by immersion and thermal drying, and the reflectance spectra

R(λ) were acquired on material surfaces in the range of 350÷850 nm (which includes the visible domain) to spectrally represent the colour property. The acquisitions were performed with the UV-VIS-NIR spectrophotometer *Perkin Elmer Lambda 950* in the diffuse-only mode using the integrating sphere. Before reduction, the percentage reflectance spectra of samples show a wavelength-proportional dependence (Fig. 12-a).

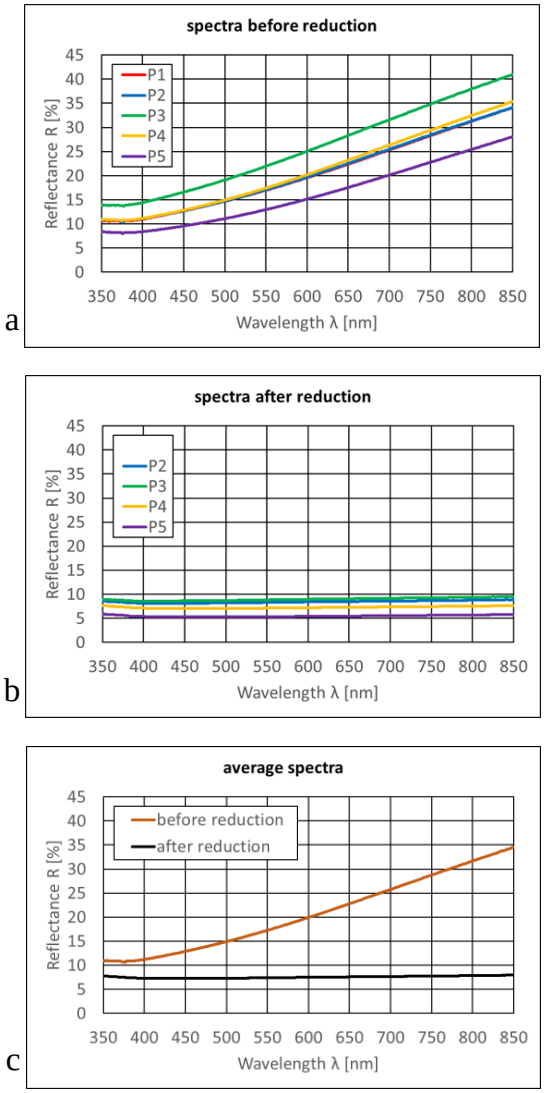

Figure 12. The spectra acquired before (a), after (b) chemical reduction process, and their averaging (c).

The prevalence of the higher wavelengths in the visible domain at the expense of lower wavelengths explains the brown colour of materials.

The GO reduction leads to the colour changing from brown to dark gray / black [12, 13]. This chemical step was performed on the previously treated materials and after the washing and thermal drying the spectra were acquired in the same conditions (Fig. 12-b). These show a low reflectance (5÷10 %) with an almost constant wavelength dependence, so the newly gotten colour is spectrally substantiated.

By averaging the spectra before, and respectively, after reduction, the representative R(λ) was obtained for each of these two fabrication stages (Fig. 12-c). The average spectra more clearly highlight the abovementioned optical characteristic after oxidation (before reduction) and after reduction.

The brownish color getting after graphene sheets oxidation is assigned to the $\pi\pi^*$ bonds density decreasing between carbon layers due to the interlayer binding of oxygenated functional groups [13, 14]. After reduction these oxidative bonds are removed restoring the initial dark color of graphitic structures [14].

4. Conclusions

This paper presents the manufacturing process of a textile electrode, meant to build a flexible supercapacitor. The reduction of Graphene Oxide as described in the Hummer method was applied on cotton fabrics. Physical-mechanical and electrical characterization of the manufactured samples was accomplished and presented good results for the surface electrical conductivity ($1.25*10^{-3}$ S/m). Spectral analysis has evidenced the changes in the surface color after oxidation and reduction, thus confirming the occurrence of chemical processes involved by Hummers' chain. These promising results will be continued.

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