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Carbon Nonwoven Fabric Coated with Graphene/PVB as an Electrode Material for Supercapacitors

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A new type of composite supercapacitive material was developed by applying graphene nanoplatelets to non-woven carbon fiber (CNW) reinforced with polyvinyl butyral (PVB). Basic mechanical resistance of the graphene-reinforced material was examined through the tensile test. The capacitive properties of the resulting CNW/PVB/graphene composite were investigated by cyclic voltammetry and electrochemical impedance spectroscopy techniques in three electrode system, with performance compared to those of neat carbon non-woven and PVB-reinforced carbon non-woven fabrics. The specific capacitances of carbon mat, carbon mat/PVB and carbon mat/PVB/graphene, obtained from CV response registered at 50 mVs^{-1}, were 1.30 x 10^{-4}, 3.00 x 10^{-4} and 3.33 x 10^{-4} F cm^{-2}, respectively. Beyond capacitance magnitude, the graphene-modified composite also exhibited a more ideal rectangular CV shape with suppressed faradaic activity and presented a steeper low-frequency slope in Nyquist plots, indicative of improved ion diffusion kinetics and overall rate capability. These findings position the developed graphene/PVB/CNW composite as a promising, flexible electrode material for supercapacitor applications.

Key words: **graphene nanoplatelets, carbon fiber non-woven, supercapacitors, flexible current collector, electrochemical analysis, double-layer capacitance.**

Introduction

GROWING environmental concerns about the depletion of fossil fuels and greenhouse gas emissions have reinforced the global trend towards sustainable and clean energy technologies [1], [2], [3]. In this context, electrochemical energy storage systems are crucial for the transition to renewable energy, electrification of transportation, in powering portable electronics, etc. In particular, supercapacitors have gained increasing attention due to their high power density, fast charge/discharge capability, excellent reversibility and extensive lifetime [4]. The performance of supercapacitors is highly dependent on the electrode material, such as surface area, conductivity and mechanical properties. Common electrode materials for supercapacitors include carbon-based materials [5] [6] [7] [8], conducting polymers [9] [10], and metal oxides [6] [11]. Among carbon-based materials, graphene stands out due to its exceptionally high surface area, excellent electrical conductivity, and outstanding mechanical strength. These properties enable efficient charge storage and fast electron transport, making graphene a highly promising candidate for high-performance supercapacitor electrodes [3] [6]. However, beyond the intrinsic properties of active materials, the design of the electrode architecture and choice of support materials play a crucial role in determining overall device performance, especially for flexible and wearable applications [12] [13]. In this context, carbon fabrics have emerged as promising supports for advanced supercapacitor electrodes. Particularly, non-woven fabrics combine mechanical robustness with high porosity, enabling efficient charge storage and easy handling in flexible and wearable energy devices [14]. In contrast to woven carbon fabrics, non-woven fabrics of low areal weight and discontinuous/random fiber orientation possess lower mechanical strength, but higher electrical conductivity [15]. To enhance their structural resistance, polymers can be introduced [16] [17]. One effective approach is to introduce a layer of polyvinyl butyral (PVB), a mechanically strong and adhesive polymer that reinforces the carbon substrate while maintaining flexibility [18].

This work focuses on the preparation of PVB-reinforced carbon fabric further modified with graphene as a supercapacitor electrode, aiming to combine mechanical robustness with enhanced electrochemical performance. For comparison, neat non-woven carbon fabric and PVB-

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reinforced carbon fabric without graphene were also studied to evaluate the individual effects of polymer reinforcement and graphene modification on mechanical integrity and electrochemical performance. Significance of the new approach with this method of graphene/PVB coating on the carbon mat material is in the fact that it is a light-weight flexible material, but with a substantial structural integrity / mechanical strength. The introduction of a small amount of PVB binder should make the graphene coating more persistent and the carbon fiber mat more mechanically resistant, but still flexible.

Experimental part

Composite samples preparation

For the preparation of the composites, the following materials were selected: PVB powder Mowital B60H (Kuraray), single-layer graphene nanoplatelets (GNPs, US Research Nanomaterials), ethanol, 96% purity (Reachem), carbon non-woven mat, surface density 20 g/m^2 declared as a type that has high resin uptake (Faserverbundwerkstoffe, Composite Technology). For the selected graphene nanostructures, the provider declared >99.3 wt% graphene purity, the thickness of the nanoplatelets 0.55–1.2 nm and their diameter 1–12 μm, having a specific surface area 500–1200 m^2/g and electrical conductivity 1000–1500 S/m. Carbon non-woven had a declared thickness (at 10 kPa) of 0.24 mm, fiber length of 6 -12 mm and fiber diameter of approximately 7 μm [19].

For the examination of the isolated effect of graphene nanoplatelets on the properties of PVB, the composite films were fabricated using a solvent-casting technique. Initially, graphene nanoplatelets were dispersed in ethanol by ultrasonic processing (Bandelin Sonopuls HD 4100, at 40 W of power, frequency of 20 kHz, for 5 minutes). This step was necessary in order to achieve deagglomeration and stable dispersion. Following this, PVB powder was gradually added to the graphene dispersion in ethanol and stirred on a magnetic stirrer at 400-500 rpm, until the added PVB was fully dissolved. The resulting homogeneous mixture was poured into PTFE molds and left to dry, allowing ethanol to evaporate under ambient conditions. This way, the uniform thin film formation was provided. The concentration of PVB in ethanol was 10 wt. %, and the amount of used graphene nanoplatelets was calculated to be in concentration of 1 wt. % related to PVB.

To fabricate flexible composites for electrochemical applications, the graphene/PVB solution was deposited onto carbon fiber non-woven mats. These mats had the role of a conductive and chemically stable substrate, capable of supporting the polymer-based thin film during measurements. The coatings were applied using different immersion strategies:

- PVB-only reference sample was made by immersion of the carbon mat in a beaker containing only the PVB-ethanol solution, without graphene. The areal weight after drying was 30,121 g/m^2.
- immersion of the carbon mat in a GNP/PVB solution, with areal weight after drying 31,133 g/m^2.
- immersion of previously made neat PVB-coated carbon mat in graphene dispersion in ethanol, under 1-minute ultrasonication, so the nanoplatelets from the dispersion could adhere onto the PVB surface. The areal weight after drying was 30,126 g/m^2.
- ultrasonically enhanced dispersion: immersion in a GNP/PVB solution subjected to ultrasonic probe treatment for 1 minute, with areal weight after drying 30,132 g/m^2.

Probable reason for higher areal weight of the carbon mat immersed in a GNP/PVB solution, regardless the low concentration of graphene, might be a higher viscosity of this solution of PVB with GNPs, thus it has a higher retention on the carbon mat. In case where neat PVB-coated carbon mat was immersed in graphene dispersion in ethanol, probably PVB started to dissolve in a small extent, so we cannot claim 100% which was the precise amount of the GNPs adhered to this sample. For the last sample, the ultrasonication was expected to help GNPs penetrate between the carbon fibers of the mat, but the total amount of the polymer absorbed was not much higher compared to the first two samples.

All the samples were dried on a flat Teflon surface, with no leaking during the drying, so it may be considered that there was controlled quantity of the impregnation on the mat’s surface. These different methods enabled comparison of coating uniformity and GNP incorporation levels. Figure 1 illustrates the sequence of the composite fabrication steps along with the representative specimens used in mechanical testing.

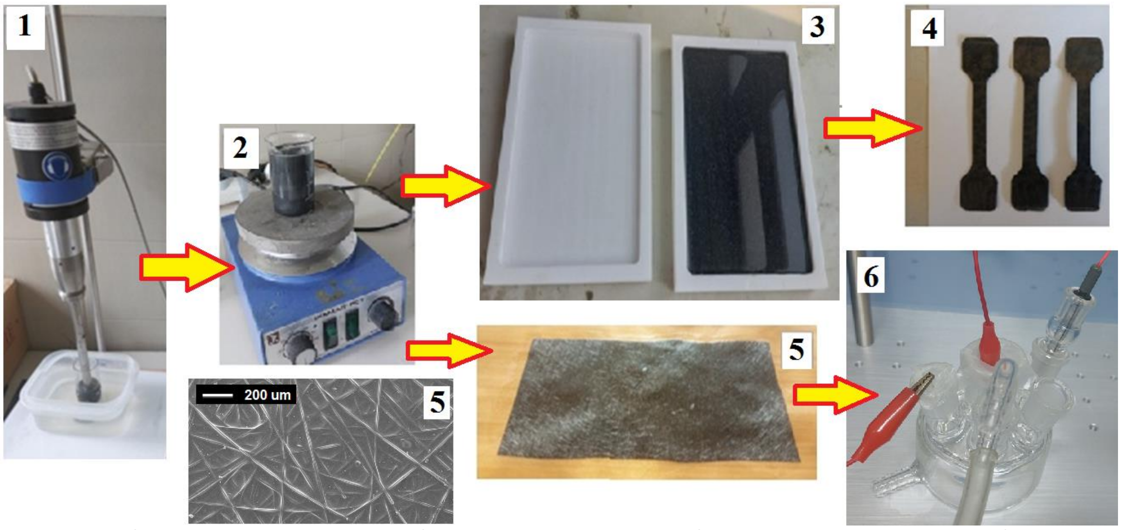


Figure 1. Preparation of graphene/PVB composites workflow: 1 – ultrasonic dispersion of GNPs in ethanol, 2 – dissolution of PVB in the graphene dispersion, 3 – solvent casting of PVB and PVB/GNP composite films in Teflon molds, 4 –specimens of PVB/GNP film cut for tensile test, 5 – carbon mat coated with PVB/GNP, 6 – electrochemical testing apparatus.

Characterization methods

The quality of impregnation of the carbon non-woven mat with PVB film was examined by scanning electron microscope, SEM, JEOL JSM-6610 LV, with a tungsten cathode, connected with an energy dispersive spectrometer, EDS, Oxford Max.

Tensile testing of PVB and PVB/GNP films was performed using an INSTRON 1122 universal testing machine. The speed of the clamps was 5 mm/min, and the test was done at the room temperature, approximately 20 °C.

Electrochemical measurements

The electrochemical performance of the samples was evaluated by cyclic voltammetry (CV) and potentiostatic electrochemical impedance spectroscopy (PEIS). Measurements were performed in a standard three-electrode setup, using a platinum wire as the counter electrode and a saturated calomel electrode (SCE) as the reference, with 0.5 M H_2SO_4 as the electrolyte (Figure 1 – 6). CV tests were conducted at sweep rates of 500, 200, 100, 50, 10 and 2 mV s^{-1}, while PEIS measurements were carried out at an open circuit potential over a frequency range of 100 kHz to 15 mHz. All experiments utilized a potentiostat/galvanostat (SP-200, Bio-Logic SAS, France). The dimensions of the tested samples were 1x1 cm. Since the recorded EIS data exhibited capacitive-like behavior, they were modeled using a transmission line equivalent electrical equivalent circuit (TLEEC) with distributed time constants, implemented in ZView (v. 3.2b, Scribner Associates Inc., Southern Pines, NC, USA). The TLEEC model comprises resistors and capacitors arranged in the form form RΩ-[C_0-(Rp,1–(C_1-(Rp,2–(...(Rp,n–Cn)...))], where each C corresponds to the capacitance belonging to the region beyond the sum of the resistances $\sum R_i$, and RΩ represents the ohmic resistance of the electrolyte.

Results and discussion

SEM/EDX analysis

SEM micrographs of the prepared composites are given in Figure 2, confirming that the carbon non-woven mat was evenly coated with the PVB/GNP film, i.e. that the fibers of the carbon mat were well immersed in the binder matrix (left pannel) and that he GNPs are well dispersed in the PVB matrix (right pannel of Figure 2). EDS spectrum of the observed site on the sample (Figure 3) has confirmed the presence of carbon, but since the carbon mat itself and graphene are made of C atoms, it was not easy to determine graphene nanoplatelets distribution in the sample. Due to this, EDS mapping was not able to provide proof of the PVB/GNP coating uniformity.

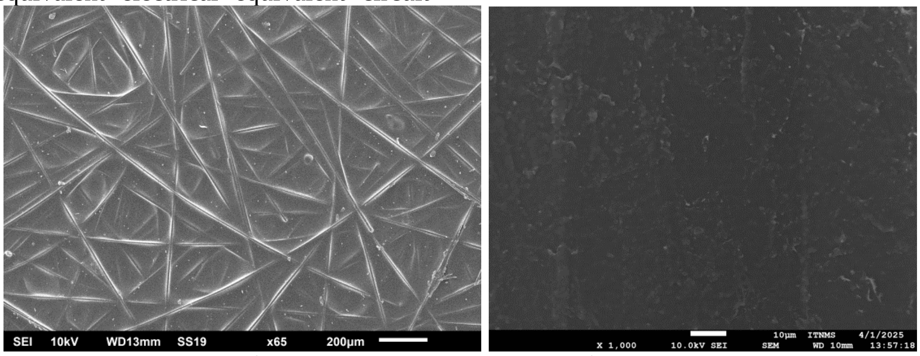
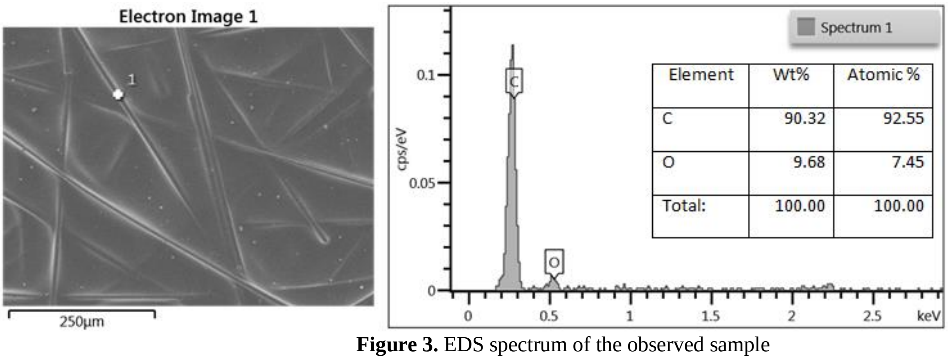


Figure 2. SEM micrograph of carbon mat coated with PVB/GNP (left) and GNPs in the PVB matrix (right)



Element	Wt%	Atomic %
C	90.32	92.55
O	9.68	7.45
Total:	100.00	100.00

Figure 3. EDS spectrum of the observed sample

The results of the tensile test are given in Table 1. For each tested film, five specimens were cut and tested, so the mean values were presented with standard deviations of the values: maximum force (F_{max}), tensile strength (σ_{max}), total elongation at break (ε_{max}), plastic strain (ε_p) and the Young's modulus (E).

Table 1. Tensile properties of PVB and PVB/GNP films

Sample/Property		F_{max}, N	σ_{max}, MPa	ε_{max}, %	ε_p, %	E, MPa
PVB	Mean value	111.38	35.71	6.44	7.77	548.03
	St.dev.	6.16	3.51	0.83	0.89	56.27
PVB/graphene	Mean value	113.13	34.11	5.01	5.32	565.1
	St.dev.	13.86	4.4	0.26	0.23	51.13

From the observed results, we may conclude that the presence of GNPs has caused a mild decrease of the tensile strength. This is actually expected, since the GNPs are a particulate filler in the polymer matrix, so here each graphene nanoplatelet represents a discontinuity in the matrix. The incorporation of graphene nanoplatelets (GNPs) into the PVB matrix resulted in a slight increase in the Young's modulus (from 548.03 MPa to 565.10 MPa), indicating a minor stiffening effect due to the intrinsically high modulus of GNPs. The total elongation at break and plastic strain were reduced, suggesting a loss of ductility. This mild embrittlement is also a consequence of the restricted polymer chain mobility caused by the presence of rigid nanoplatelets.

However, reinforcement of the neat CNW by coating it with PVB or PVB /graphene provides a significant increase in the tensile strength of the CNW, i.e. the PVB coated and PVB/ graphene coated CNW will have significantly better structural resistance compared to the neat CNW. As declared by the producer, a carbon non-woven mat weighing 20 g/m² has a tensile strength of 36 N per 15 mm of width [19]. For the declared nominal thickness of 0.24 mm, this means that the tensile strength of the CNW is ~10 MPa. On the other hand, PVB and PVB/graphene thin films had tensile strength up to ~35 MPa.

The obtained results are in accordance with available findings on similar composites: Hoepfner et al. had studied PVB reinforced with GNP, and reported that the results of storage modulus, degree of entanglement, and adhesion factor of the polymer with the reinforcement were not significantly modified, due to weak interfacial interactions between the graphene and PVB [20].

Further improvement might include possible incorporation of the obtained non-woven mesh into a 3D printed high-porosity scaffold, which might provide even better structural properties of this functional material, but certainly the feasibility and optimization of such a production approach should be carefully analyzed in the context of process planning and technology [21].

Electrochemical characterization

Figure 4 shows characteristic cyclic voltammograms (CVs) of the carbon non-woven (carbon mat), PVB-coated CNW (carbon mat/PVB) and the PVB/graphene-coated carbon non-woven (carbon mat/PVB graphene) at different sweep rates.

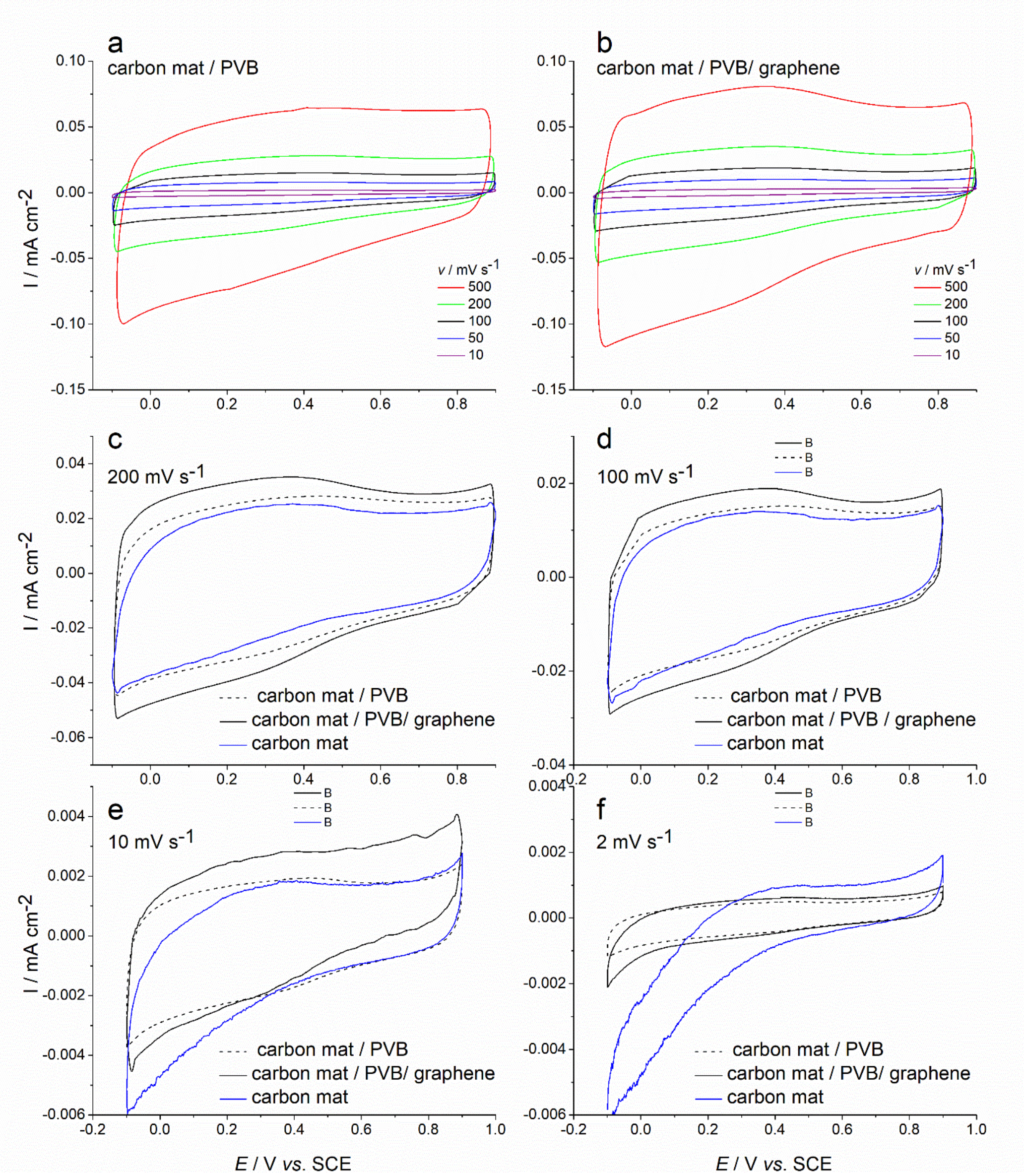


Figure 4. Cyclic voltammograms (CVs) of a) carbon mat/PVB and b) carbon mat/PVB/graphene at different sweep rates; c-f) Comparison of CVs of carbon mat, carbon mat/PVB and carbon mat/PVB/graphene at different sweep rates. Electrolyte: 0.5 M H_2SO_4.

All CV curves exhibit a quasi-rectangular shape, which is typical for capacitive materials where charge storage is governed by electric double layer capacitance. A small anodic peak near 0.4 V appeared in the CV curves of the graphene-modified sample, accompanied by corresponding cathodic peaks during discharge, suggesting a redox process. These features are likely due to proton-coupled electron transfer reactions involving residual oxygen-containing functional groups on the graphene, such as quinone-like structures [22]. The CV currents of the graphene-modified sample are higher than those of the PVB-reinforced, confirming the enhanced charge storage capacity contributed by the graphene coating. The retention of the quasi-rectangular shape at higher scan rates emphasizes the good rate capability of the graphene-modified sample. At slow rate of 2 mV/s (Figure 4f), the pure carbon mat showed a distorted CV with a pronounced cathodic tail, indicating dominant faradaic side reactions. In contrast, the carbon mat/PVB and carbon mat/PVB-graphene showed a much more regular rectangular CV shape, indicating dominant double layer capacitance and suppressed faradaic activity. This improvement can be attributed to the presence of PVB, a non-conductive polymer that acts as a passivating layer, effectively masking or reducing the accessibility of faradaic active sites on the carbon mat surface [18]. This minimizes unwanted side reactions and promotes the dominance of double-layer capacitance.

The values of specific capacitances of the prepared samples were determined from the CV curves by equation (1):

$$C_s = \frac{Q}{\Delta E * m} \quad (1)$$

where Q is the charge consumed in the potential range, ΔE and m is the mass of active materials used for electrode preparation. The specific capacitances of carbon mat, carbon mat/PVB and carbon mat/PVB/graphene, obtained from CV response registered at 50 mV s^{-1}, were 1.30 x 10^{-4}, 3.00 x 10^{-4} and 3.33 x 10^{-4} F cm^{-2}, respectively.

To better understand the capacitive behavior of carbon mat/PVB and carbon mat/PVB/graphene, their electrochemical behavior was investigated by EIS measurements. The impedance plane plots and Bode plots are shown in Figure 5 and Figure 6, respectively.

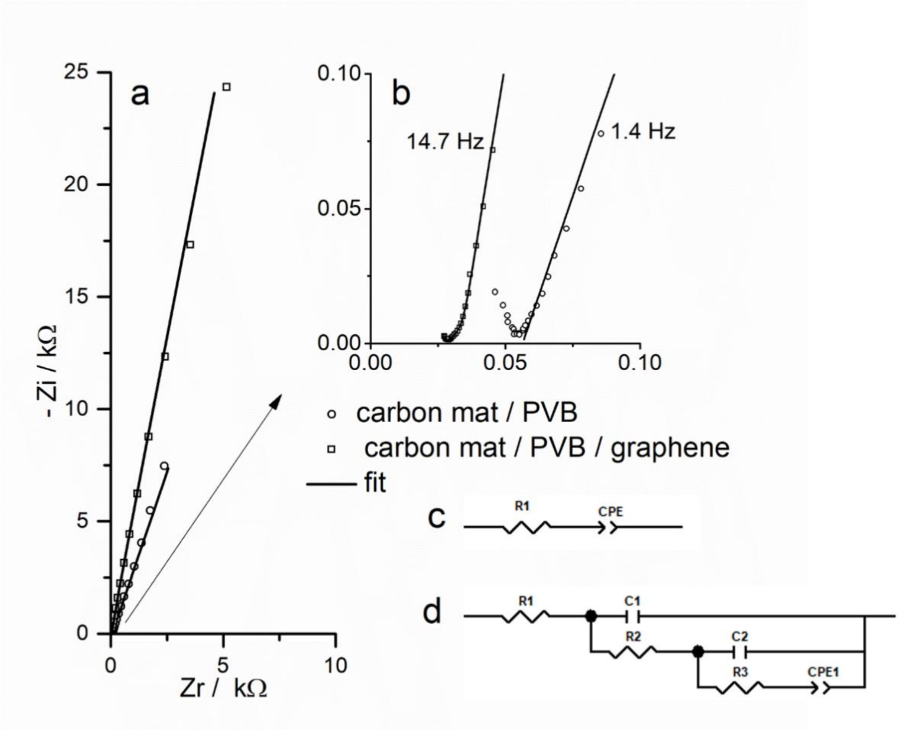


Figure 5. a) Nyquist plane plots registered for carbon mat/PVB and carbon mat/PVB/graphene from 100 kHz to 15 mHz with b) enlarged high frequency range, along with equivalent electric circuits used for fitting of c) carbon mat/PVB and d) carbon mat/PVB/graphene. Electrolyte: 0.5 M H_2SO_4; symbols: Data; lines: Fitting data.

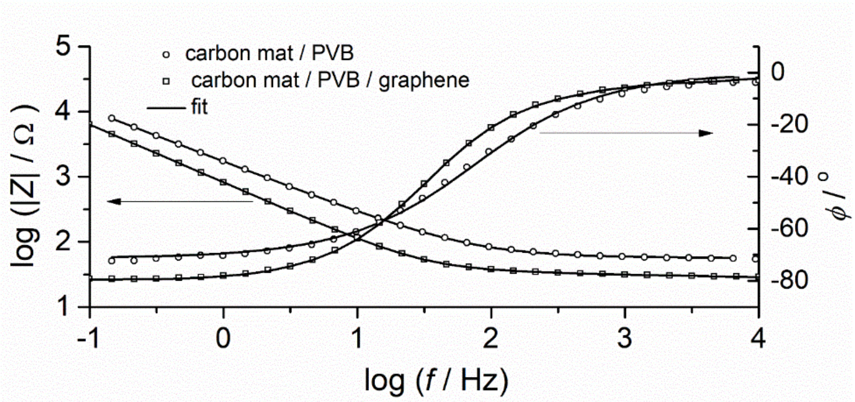


Figure 6. Bode plots registered for carbon mat/PVB and carbon mat/PVB/graphene. Electrolyte: 1 M H2SO4; symbols: Data; lines: Fitting data.

In the Nyquist plots, both the carbon mat/PVB and carbon mat/PVB/graphene electrodes exhibited characteristic inclined straight lines in the medium to low frequency range. This behavior is indicative of the diffusion-controlled capacitive response. Notably, the carbon mat/PVB/graphene electrode presented a steeper slope in this region compared to the carbon mat/PVB, signaling a closer approach to ideal capacitive characteristics. This enhanced performance is attributed to the synergistic effects of graphene, which both enhances the overall electrical conductivity of the composite network and expands the electrochemically accessible surface area, thereby expediting both electronic charge transport and ionic diffusion within the intricate electrode matrix. The impedance response of the carbon mat/PVB sample was fitted in the medium to low frequency range using an equivalent circuit consisting of a resistor in series with a constant phase element (R–CPE). In contrast, the more complex porous structure introduced by the graphene required a multi-branch equivalent circuit model to accurately fit the impedance data for the carbon mat/PVB/graphene sample. In both models, the series resistance (R1) is attributed to the uncompensated resistance of the electrolyte, the current collectors, and the contact interfaces. The use of a CPE instead of an ideal capacitor accounts for the non-ideal capacitive behavior often observed in porous materials. In the Bode plots (Figure 6), both composites exhibit a phase angle approaching -90° at low frequencies, characteristic of ideal capacitive behavior, transitioning to a dominant resistive response at high frequencies. Overall, these EIS findings support that the incorporation of graphene enhances the electrochemical performance of the carbon mat-based electrodes by optimizing both charge transfer and storage kinetics. Further research might consider incorporation of higher concentrations of graphene nanoplatelets, similar to those reported by Huang et al. [23]. This way, the new material might have an application in encapsulation of solar cells and the cooling of electronic devices.

Conclusion

A new PVB/GNP composite coating was successfully developed and applied as a thin layer on a carbon non-woven fabric. The PVB and PVB/GNP coating has reinforced the CNW mechanically, increasing its tensile strength. The composite's capacitive properties were evaluated using cyclic voltammetry and electrochemical impedance spectroscopy, and its performance was compared to that of bare carbon non-woven. The modification of the carbon mat with PVB and graphene effectively enhances its capacitive performance. Despite being an insulating polymer, PVB contributes to a significant increase in capacitance, by improving electrode integrity, electrolyte retention, and surface accessibility. The subsequent incorporation of graphene nanoplatelets further improves charge storage capability, confirming its role as an efficient conductive additive. The graphene-modified carbon non-woven reinforced with PVB thus shows promising potential as a supercapacitive material.

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Karbonska netkana tkanina obložena grafenom/PVB-om kao elektrodni materijal za superkondenzatore

Razvijen je novi tip kompozitnog materijala nanošenjem nanoljuspica grafena na netkanu karbonsku tkaninu – mat (CNW) ojačanu PVB-om. Cilj je bio da se CNW koristi kao fleksibilni kolektor struje u fleksibilnim i laganim superkondenzatorima. Za ispitivanje kvaliteta impregnacije karbonskog mata korišćena je SEM mikroskopija. Osnovna mehanička otpornost materijala ojačanog grafenom/PVB-om ispitana je testom zatezanja. Kapacitivna svojstva kompozita CNW/PVB/grafen ispitivana su tehnikama ciklične voltametrije i spektroskopije elektrohemijske impedancije u rastvoru 0,5 M Na_2SO_4, pri čemu je vršeno upoređivanje sa čistom netkanom karbon tkaninom i PVB-om ojačanim netkanom tkaninom. Dobijeni rezultati su karakteristični za kapacitivne materijale kod kojih skladištenje naelektrisanja nastaje mehanizmom elektrohemijskog dvojnog sloja. Poboljšani kapacitivni odziv kompozita CNW/PVB/grafen u poređenju sa čistim CNW i CNW/PVB pripisuje se visokoj električnoj provodljivosti i velikoj specifičnoj površini grafena, što omogućava efikasnije skladištenje naelektrisanja na granici dodira faza elektroda/elektrolita. Rezultati pokazuju da je CNW perspektivan fleksibilni kolektor struje za kapacitivne aplikacije zasnovane na grafenu.

Ključne reči: **Grafen nanoljuspice, netkani karbonski mat, superkondenzatori, fleksibilni kolektor struje, elektrohamijska analiza, kapacitet dvostrukog električnog sloja.**