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Simple and Cost-effective Fabrication of a Supercapacitor Using Carbon Nanoparticle-based Ink

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Abstract. The demand for energy storage devices such as supercapacitors is rapidly increasing in most applications, including electric transportation and portable electronics. The material's high cost and the fabrication complexity of supercapacitors suppress their mass production. In this research, a commercial low-cost conductive ink has been simply painted on flexible stainless steel sheets as supercapacitor electrodes. The ink is mainly based on carbon crystals, as indicated by the X-ray diffraction peaks, and the results of the energy-dispersive X-ray spectroscopy confirm the elemental analysis. The carbon-based ink comprises semi-spherical nanoparticles as imaged by the scanning electron microscope. The electrochemical energy storage mechanism of the carbon nanoparticle-based electrode in the sulfuric acid electrolyte depends on the electric double layer, as investigated by the cyclic voltammetry and the galvanostatic charge-discharge measurements. As a result, the carbon ink-based supercapacitor exhibits a maximum areal capacitance of 0.5 mF/cm^2 at 0.25 mA/cm^2, a maximum energy of 0.2 µWh, and a maximum power of 1600 µW. The electrochemical impedance spectroscopy shows excellent equivalent series resistance of 0.6 ohm, representing a solid attachment of the ink on the stainless steel substrate. In addition, an angle of 45^o in the low-frequency range indicates a semi-infinite diffusion of the electrolyte ions into the nanostructured electrodes. All these elemental, morphological, and electrochemical properties of the carbon-based ink promote its potential to be effortlessly implemented as electrodes, thereby contributing to the advancement of supercapacitor manufacture.

Keywords: Conductive ink, Supercapacitor, Carbon, nanomaterial, Stainless steel.

1. Introduction

In 2023, the World Bank Group reported that energy storage is crucial for maintaining a stable and consistent electricity supply, especially when demand outstrips supply, such as electric transportation and portable electronic devices [1]. One of the promising energy storage technologies is an electrochemical supercapacitor, which facilitates reversible desorption and adsorption of the electrolyte ions at the electrode-electrolyte interface, allowing for effective energy storage and release [2]. Unlike traditional electrostatic capacitors, supercapacitors exhibit high capacitance due to utilizing porous nanostructured electrodes with a large specific surface area, resulting in enhanced energy density. The capacitance (C), energy (E), and power (P) of any capacitor can be calculated by the following equations (1-4) using the amount of released charge (Q) or the constant current (I), the discharge time (Δt), and the discharge voltage (ΔV).

$$\mathrm{C} = \frac{\mathrm{Q}}{\mathrm{V}} \tag{1}$$

$$\mathrm{C} = \frac{\mathrm{I}\,\Delta\mathrm{t}}{\Delta\mathrm{V}} \tag{2}$$

$$\mathrm{E} = \frac{1}{2}\, C\, V^2 \tag{3}$$

$$\mathrm{P} = \frac{\mathrm{E}}{\Delta\mathrm{t}} \tag{4}$$

Carbon materials have been widely investigated in supercapacitors literature [3, 4]. Various forms of carbon electrodes are created, such as activated carbon [5, 6], graphene-based organic frameworks [7], graphene-based microfibers [8], graphene aerogel [9], and carbon nanotubes [10]. Zhao *et al.* [11] fabricated an asymmetric supercapacitor of activated carbon anode and NiSb/NiTe cathode, demonstrating a power density of 750 µW/cm^2. Wang *et al.* [12] used tannic acid with thermal reduced graphene oxide as a symmetrical supercapacitor in a 6 M KOH electrolyte. They measured an areal capacitance of 525 mF/cm^2 and an energy density of 72.2 µWh/cm^2 at a power density of 250.9 µW/cm^2. The high supercapacitor performance is a result of the rich redox-active functional groups of the tannic acid [12].

Supercapacitors are found in various applications, such as electric vehicles, power systems, and hybrid energy storage systems integrated with renewable energy sources [13]. Despite their benefits, their high cost-to-performance ratio hinders widespread adoption [14]. Utilizing well-produced commercial chemicals with high electrical conductivity and low cost could narrow the gap between laboratory-scale research and the industrial-scale manufacturing of supercapacitors.

The capacitance of commercial EDLC supercapacitors ranges from 1 to 5000 F [15]. Specific examples of commercial supercapacitor performance are compared in a review article by Yassine and Fabris [2]; Panasonic offers a supercapacitor with 0.10 F, 2.30 V, and 0.08 mΩ; another supercapacitor available by PowerStor exhibits 2.20 F, 2.5 V, and 4.57 Ω. Graphene and carbon nanotubes-based inks have been respectively produced by Vorbeck Materials and Nanocyl [16, 17]. Inks based on conductive polymers and metal oxides have been manufactured by DuPont, Sigma-Aldrich, and Pedotinks [18]. For supercapacitor applications that require stretchability and long cyclic stability, carbon-based inks are recommended, while conductive-polymers-based inks are recommended for high-specific capacitance and energy density applications. Cabot Corporation provides hybrid materials-based inks such as graphene and metal oxides [16], which can utilize the advantages of each material.

In this research, commercial conductive ink and flexible stainless steel (SS) sheets are selected to facilitate the fabrication of a cost-effective, flexible, and binder-free supercapacitor. The flexible stainless-steel sheet was used as substrate in our supercapacitor because it has high electrical conductivity, corrosion resistance, mechanical strength, cost-effectiveness, and thermal stability [19, 20]. The determination of the elemental and morphological properties of the ink supports the understanding of the relationship between the material properties and the supercapacitor performance. The energy storage mechanism of our supercapacitor is investigated through the results of the electrochemical measurements. Our research aims to assess the effectiveness of such ink products in supercapacitors, potentially accelerating the implementation of cost-effective alternatives in energy storage technologies.

2. Experiments

A commercial conductive ink (Electric Paint, Bare Conductive brand, US$ 80 for 50 mL, Fly Thinking Dili Asia MBMChip Store, China) was used without any treatment or additives. It was cast on a glass substrate to analyze the crystallinity using an X-ray diffraction (XRD). In addition, the ink was also painted on an SS sheet to image its morphology, calculate its particle size, and determine its chemical element using a scanning electron microscope (SEM) equipped with a secondary electron detector and energy dispersive x-ray spectroscopy (EDX) detector.

For the supercapacitor fabrication, two pieces of SS substrates with a 4 cm^2 area are coated with the conductive ink on one side of each substrate and used as electrodes with SS terminals, as shown in Fig. 1 (a,b). The painting was done manually at room temperature without additives or binders. The dielectric material of the electrolyte is composed of sulphuric acid (H_2SO_4), amount of 1g, and polyvinyl alcohol (PVA), amount of 1g, and all mixed with 6 mL of distilled water. Then, the mixture was put on a hot plate at a temperature of 90°C with a magnetic movement estimated at 400 revolutions per minute (rpm) for several minutes until becoming a homogenous white gel. [21] Finally, the gel electrolyte was cast on the painted side of the electrodes and then placed face by face, as imaged in Fig. 1 (c).

Our supercapacitor was connected to the electrochemical workstation (CH Instrument Company, USA) as imaged in Fig. 1 (c) and tested by three experiments: cyclic voltammetry (CV), galvanostatic charge-discharge (CD), and electrochemical impedance spectroscopy (EIS) at room temperature. The supercapacitor current in the CV experiment is produced by applying a liner voltage as an independent variable at different voltage scan rates. This technique is used to study the reversibility of redox reactions if present, determine the optimal voltage window, and measure the resulting current. The data obtained from the GCD experiment is in the form of charge-discharge times within the optimal voltage window under a constant current application. This relationship is crucial to calculate the capacitance of the supercapacitor. The EIS variables are sinusoidal voltage waves of 5 mV amplitude in a frequency range from 0.1 Hz to 100 KHz as an independent variable; it is graphed in a Nyquist plot with the real and imaginary parts of the impedance as a dependent variable. The benefit of this experiment is to determine the equivalent series resistance of the substrate, electrode, and electrolyte.

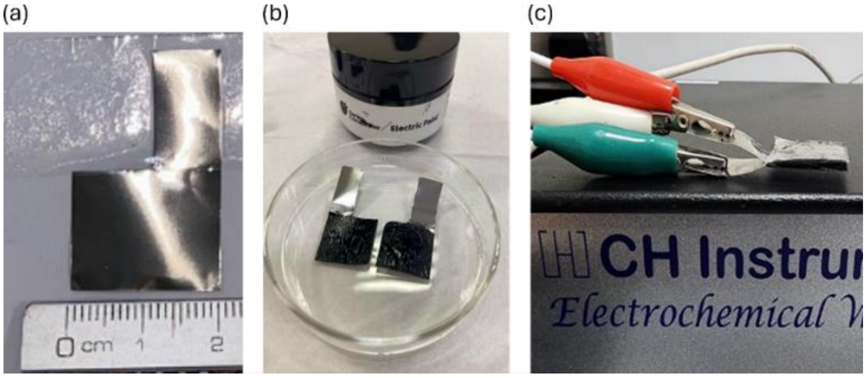
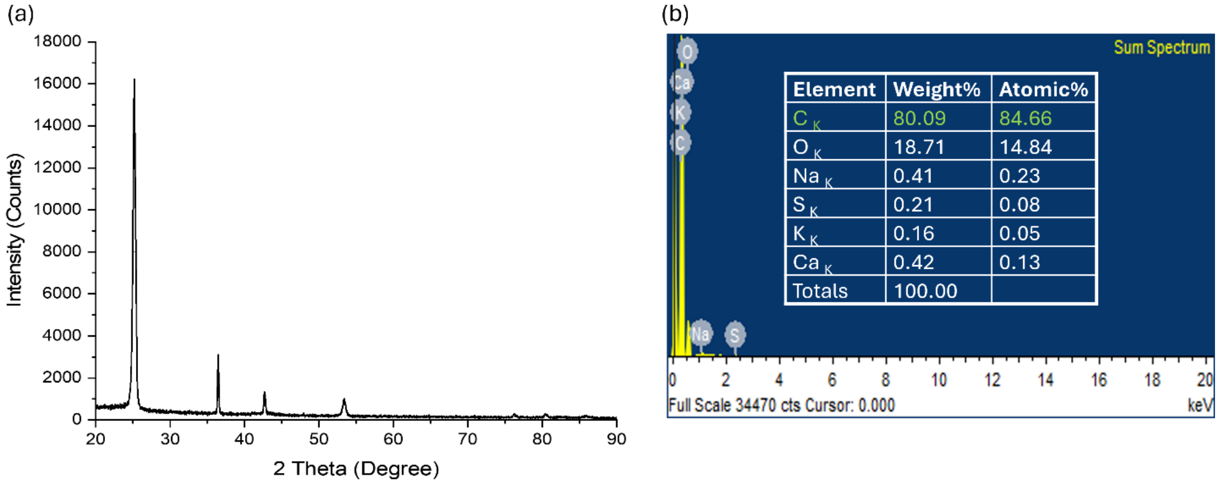


Fig. 1. Photographs of the fabrication and testing (a) a stainless steel substrate, (b) a conductive ink bottle used and coated substrates, and (c) a carbon-based supercapacitor connected to an electrochemical workstation.

3. Results and Discussion

Characterizing the crystallinity and elemental chemicals of the commercial conductive ink is essential for utilizing it as electrodes in supercomputers. Figure 2 (a) shows the XRD spectrum of the conductive ink on a glass substrate. The sharp peaks indicate that the sample consists of crystals. The centers of the peaks at 25.18°, 36.46°, 42.68°, and 53.4° match a slight shift with a reported carbon [22]. The shift in the constructive diffraction peaks can be attributed to impurities in the crystals. The EDX spectrum and its elements table in Fig. 2 (b) confirm that the conductive ink contains carbon as the majority element (80.1% weight percentage), oxygen (18.7% weight percentage), and other elements at a rate of less than 2% including sodium, sulfur, potassium, and calcium which are conductive metals. Carbon is a semiconductor material that conducts electricity at room temperature. Carbon and such metals in this commercial conductive ink cause its electrical conductivity. This property is a crucial requirement for supercapacitor electrodes.

The SEM images of the conductive ink on an SS substrate are depicted in Fig. 3 (a,b) at different magnifications. The low-magnification image shows the homogeneity and complete coverage of the ink on the SS substrate. The measured scale bars in the high magnification image clarify that the ink contains semi-spherical nanoparticles of diameters ranging between 50 and 100 nm. The small particle sizes and the porous morphology imply a large specific surface area, hence abundant sites of electrode-electrolyte interface in supercapacitors. The elemental and morphological characteristics of this conductive ink suggest a high possibility of constructing an effective supercapacitor



Element	Weight%	Atomic%
C_K	80.09	84.66
O_K	18.71	14.84
Na_K	0.41	0.23
S_K	0.21	0.08
K_K	0.16	0.05
Ca_K	0.42	0.13
Totals	100.00	

Fig. 2. (a) The XRD spectrum and (b) the EDX spectrum with its elements table of the conductive ink.

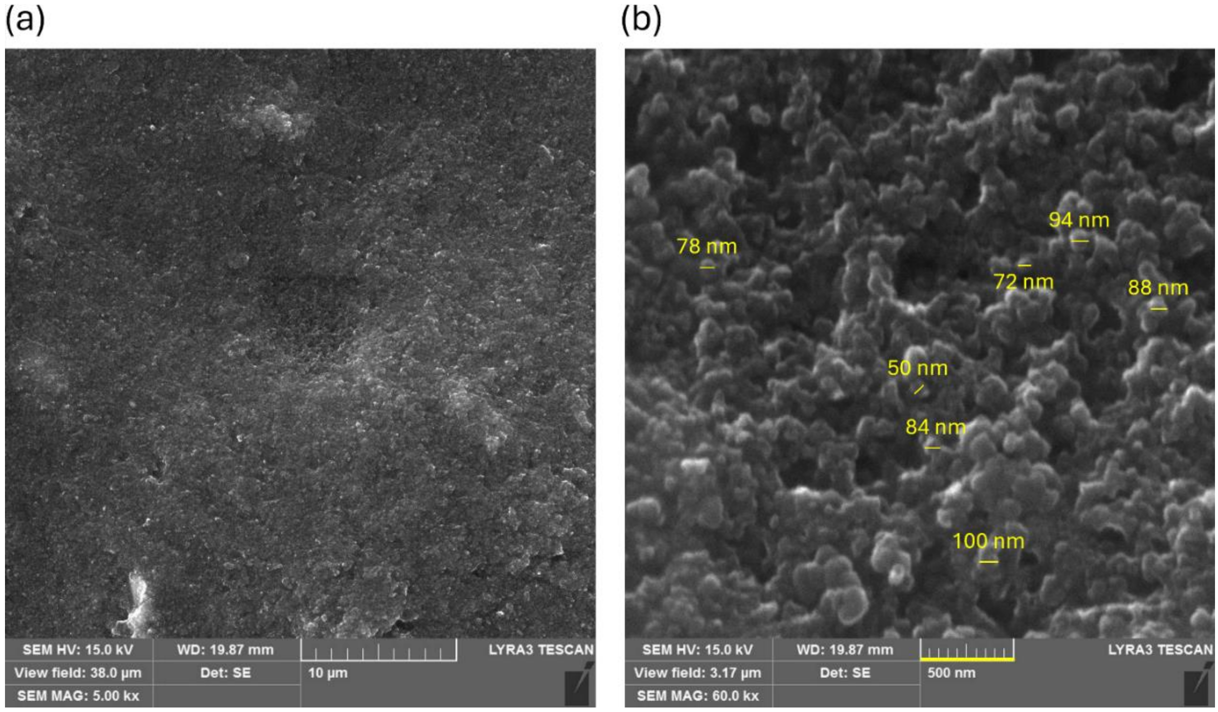


Fig. 3. SEM images of the conductive ink at (a) a low magnification of 5000x and (b) a high magnification of 60000x.

The carbon-based supercapacitor is investigated by the CV and GCD results in Fig. 4 (a, b). The applied voltage window is optimized to be 0.8 V to avoid the hydrogen and oxygen evolution reactions in the aqueous H_2SO_4/PVA gel electrolyte. The resultant current increases when the voltage increases from 0 V to 0.8 V during the charge curve. Then, the current decreases when the voltage decreases from 0.8 V to 0 V during the discharge curve. By increasing the voltage scan rates from 50 mV/s to 200 mV/s, the area enclosed by the current loops increases with a deviation in the semi-rectangle shapes caused by factors such as resistances. The semi-rectangle CV shapes without peaks indicate that the energy storage mechanism in the supercapacitor is based on the electrical double-layer formation between charged porous electrodes and electrolyte ions without redox chemical reactions [23].

The energy storage mechanism of our supercapacitor is confirmed by the GCD semi-tringle curves, indicating the electrical double-layer mechanism. The supercapacitor can be charged and discharged within a voltage window of 0.8 V, which matches the CV testing. By applying a constant current of 1 mA, equivalent to an areal current of 0.25 mA/cm^2, the recorded discharge time was 1.68 s. When the GCD test was repeated at 2 mA, the discharge time decreased to 0.48 s.

Lists the independent applied currents and the dependent results of discharge times. The inverse relationship between the current and the charge-discharge time is because the number of stored-released charges in a capacitor is constant according to the equation (5):

$$I = \frac{Q}{\Delta t}$$

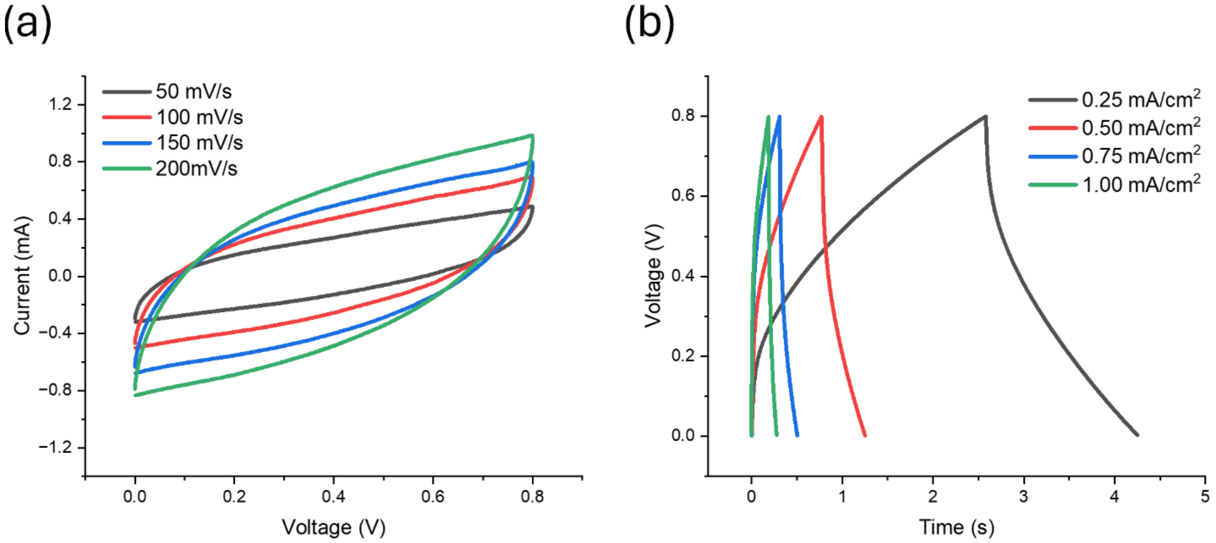


Fig. 5. Electrochemical performance of the carbon-based supercapacitor in (a) the CV curves at different scan rates and (b) the GCD curves at different areal currents.

The discharge times and discharge voltage window from the GCD curves are inserted in equation (1) at each areal current value to calculate the areal capacitances and plotted in Fig. 5 (a). The maximum areal capacitance is 0.525 mF/cm^2 at 0.25 mA/cm^2. The capacitance is normalized to the area of the electrodes for the sake of the comparison with the literature. At the same areal current of 0.25 mA/cm^2, 0.406 mF/cm^2, and 0.005 mF/cm^2 were reported by Khakpour *et al.* [24] for two symmetric supercapacitors of reduced graphene oxide respectively prepared on the negative and positive feeding electrodes of bipolar electrochemistry (BPE) method. In another example, Noh *et al.*[25] applied an electrochemical oxidation treatment (EOT) of carbon nanotubes (CNT) and produced electrodes with 0.587 mF/cm^2 at 10 mV/s in PVA-Na_2SO_4 gel electrolyte, higher than 0.0182 mF/cm^2 of their untreated CNT. Furthermore, several reported areal capacitances are compared in Table 1.

Table 1. Comparison of areal capacitances of supercapacitors.

Supercapacitor Electrodes	Electrolytes	Areal Capacitance $[mF/cm^2]$	References
Commercial ink of carbon nanoparticles	H_2SO_4-PVA gel	0.525	This work
Treated graphene	Na_2SO_4 aqueous	0.406	[24]
Untreated graphene	Na_2SO_4 aqueous	0.005	[24]
Treated carbon nanotubes	Na_2SO_4- PVA gel	0.587	[25]
Untreated carbon nanotubes	Na_2SO_4-PVA gel	0.0182	[25]
Commercial ink of carbon nanofibers and MnO_2	H2SO4-PVA gel	224	[26]
Graphene foam with polyaniline	H_2SO_4 aqueous	1700	[27]
Laser-scribed graphene	H_2SO_4 aqueous	4.04	[28]
Laser-scribed graphene	H_3PO_4 aqueous	3.67	[28]
Titania nanotube arrays	KCl aqueous	0.911	[29]

For practical applications, our carbon supercapacitor's capacitance, energy, and power values are calculated using equations (2-4) and listed in Table 2. The maximum capacitance is 2.1 mF at 1 mA, where the longest discharge time is measured, releasing the maximum energy of 0.19 µWh at a power of 400 µW. However, the maximum power of 1600 µW is recorded at an energy of 0.04 µWh where the shortest discharge time is taken. Figure 5(b) clarifies this inverse relationship between the energy and the power of the carbon-based supercapacitor.

Table 2. The results of the GCD experiments of the carbon ink-based supercapacitor.

Current (I) [mA]	Discharge Time (Δt) [s]	Capacitance $(C = \frac{I\,\Delta t}{\Delta V})$ [mF]	Energy $(E = \frac{1}{2} C\, V^2)$ [µWh]	Power $(P = \frac{E}{\Delta t})$ [µW]
1	١,٦٨	٢,١	٠,١٩	٤٠٠
2	٠,٤٨	١,٢	٠,١١	٨٠٠
3	٠,١٩	٠,٧	٠,٠٦	١٢٠٠
4	٠,٠٩	٠,٥	٠,٠٤	١٦٠٠

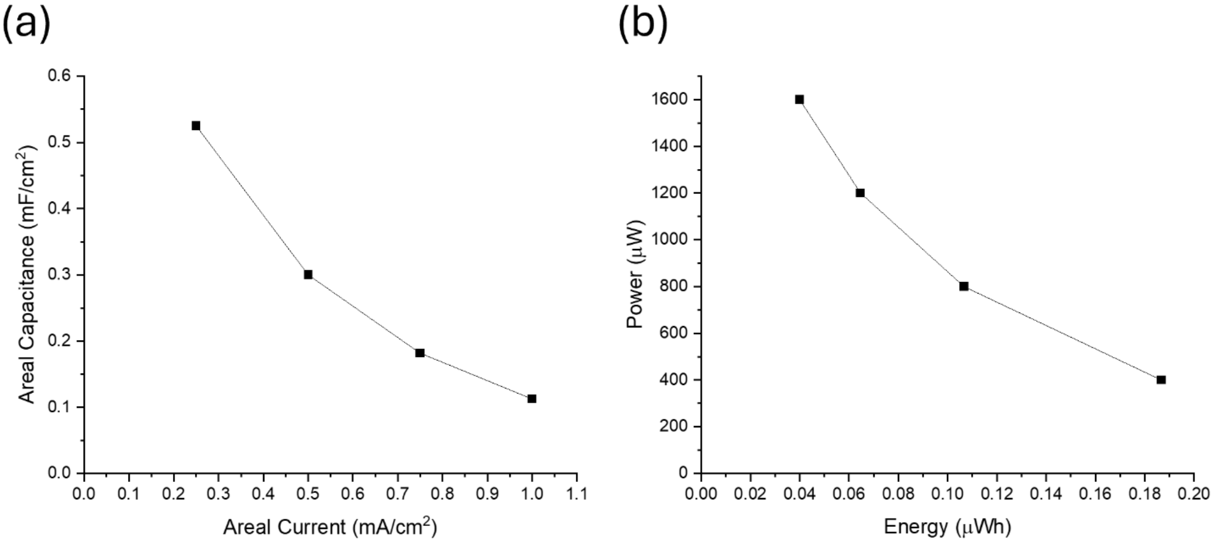


Fig. 5: (a) Areal capacitances at different areal currents and (b) powers at different energies of carbon-based supercapacitor.

Nyquist plot in Fig. 6 represents the mathematical relation between the real part of the impedance (Z') and the imaginary part of the impedance (Z”). At high frequencies, the real Z' started from 0.6 ohm when Z" equals 0 ohm. This intercept point is called the supercapacitor's equivalent series resistance (ESR), including the resistance of electrodes and substrate. The low ESR value of 0.6 ohm proves the commercial ink is a good conductor and well-pasted on the SS substrate. At low frequencies, there was a sharp, linear increase of Z" with a slope of 45^o when Z' increased from 90 to 200 ohms, considered good values. This angle of 45^o indicates a semi-infinite diffusion of the electrolyte ions into the porous electrodes.

4. Conclusion

This research investigates the validity of using a commercial conductive ink as an electrode for supercapacitors. The X-ray diffraction and the energy-dispersive X-ray spectroscopy spectra reveal that the ink contains carbon crystals and insignificant impurities. The morphology of semi-spherical nanoparticles is captured by the scanning electron microscope. The electrochemical measurements of the carbon electrodes in the H_2SO_4/PVA gel electrolyte demonstrated an electric double-layer capacitance of 2 mF at 1 mA. The maximum energy of this supercapacitor reaches 0.2 µWh, and the maximum power is 1600 µW. The electrochemical impedance spectroscopy shows a low equivalent series resistance of 0.6 ohm and a slope of 45^o. The good supercapacitor performance is attributed to several factors, including the crystallinity, the nanoparticles morphology, the strong attachment of the ink on the stainless steel substrate, and the semi-infinite diffusion of the electrolyte ions into the porous electrodes. The simple coating of flexible substrates by such commercial low-cost conductive ink could facilitate the implementation of supercapacitors in wearable electronics.

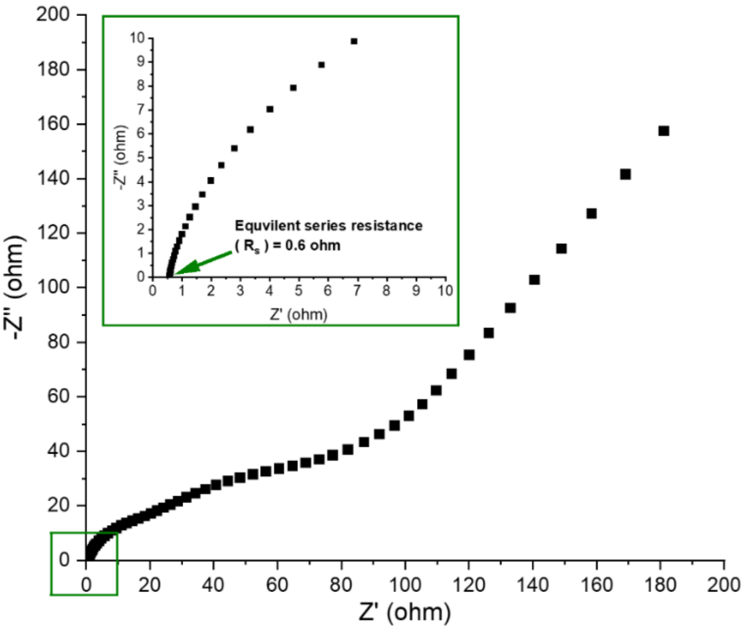


Fig. 6. Nyquist plot of electrochemical impedance spectroscopy of the carbon-based supercapacitor and an enlarged scale in the high frequencies' region.

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Conflicts of Interest

The authors declare no conflict of interest.

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تصنيع بسيط وفعال من حيث التكلفة لمكثف فائق باستخدام حبر قائم على جسيمات الكربون النانوية

نهى الحبشي [١]، و **نوف الدوسري** [٢]، و **وهاد السلمي** [٢]، و **غادة المحمادي** [٢]، و **رغد الزهراني** [٢]

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المستخلص. يتزايد الطلب على أجهزة تخزين الطاقة مثل المكثفات الفائقة بسرعة في معظم التطبيقات، بما في ذلك النقل الكهربائي والإلكترونيات المحمولة. إن التكلفة العالية للمواد وتعقيد تصنيع المكثفات الفائقة يثبطان إنتاجها الضخم. في هذا البحث، تم طلاء حبر موصل تجاري منخفض التكلفة ببساطة على صفائح مرنة من الفولاذ المقاوم للصدأ كأقطاب كهربائية للمكثفات الفائقة. يعتمد الحبر بشكل أساسي على بلورات الكربون، كما هو موضح من خلال قمم حيود الأشعة السينية، وتؤكد نتائج مطيافية الأشعة السينية المشتتة للطاقة التحليل العنصري. يتألف الحبر الكربوني من جسيمات نانوية شبه كروية كما تم تصويرها بواسطة المجهر الإلكتروني الماسح. تعتمد آلية تخزين الطاقة الكهروكيميائية للقطب الكهربي القائم على جسيمات الكربون النانوية في إلكتروليت حمض الكبريتيك على الطبقة المزدوجة الكهربائية، كما تم التحقيق فيها من خلال الفولتية الدورية وقياسات الشحنة والتفريغ الجلفانية. نتيجة لذلك، يظهر المكثف الفائق القائم على الحبر الكربوني سعة مساحية قصوى تبلغ ٠,٥ ميكرو فاراد/سم٢ عند ٠,٢٥ مللي أمبير/سم٢، وطاقة قصوى تبلغ ٠,٢ ميكرووواط/ساعة، وقوة قصوى تبلغ ١٦٠٠ ميكرووواط. يُظهر مطياف المعاوقة الكهروكيميائية مقاومة متسلسلة مكافئة ممتازة تبلغ ٠,٦ أوم، مما يمثل ارتباطًا قويًا للحبر على ركيزة الفولاذ المقاوم للصدأ. بالإضافة إلى ذلك، تشير الزاوية ٤٥ درجة في نطاق التردد المنخفض إلى انتشار شبه لانهائي لأيونات الإلكتروليت في الأقطاب الكهربائية النانوية. تعزز كل هذه الخصائص الأولية والشكلية والكهروكيميائية للحبر القائم على الكربون من إمكاناته ليتم تنفيذه بسهولة كأقطاب كهربائية، وبالتالي المساهمة في تقدم تصنيع المكثفات الفائقة.

الكلمات المفتاحية: حبر موصل، مكثف فائق، كربون، مادة نانوية، الفولاذ المقاوم للصدأ.