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Exploring some overlooked parameters in Laser Induced Graphene (LIG) production

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RESUMO

Os eletrodos LIG recebem destaque devido a sua excelente resposta eletroquímica e versatilidade. As suas propriedades podem ser ajustadas pelos parâmetros do laser, porém, alguns deles não são comumente relatados na literatura, como a orientação e resolução (linhas mm^{-1}). Portanto, esse estudo avalia a influência desses parâmetros nas suas propriedades. Inicialmente, as C_{dl} obtidas foram de: 281 µF na horizontal e 403 µF na vertical a qual foi mantida devido ao maior valor. Em seguida, a resolução foi variada de 5 a 20 linhas mm^{-1}, sendo observado um aumento na C_{dl} de 227 a 651 µF, indicando que foi obtida uma maior área eletroativa. Entretanto, na presença da sonda redox, $K_3[Fe(CN)_6]$, o menor ΔE_p foi para a resolução 10 (156 mV). Uma hipótese é que, ao passar o laser em regiões adjacentes geram-se mais poros, porém o grafeno é convertido em grafite, diminuindo a cinética de transferência de carga. Concluindo, esses parâmetros impactam significativamente sua resposta eletroquímica.

Palavras-chave: Laser Induced Graphene, LIG, 450 nm, Diode Laser.

Introduction

The development of novel electrodes for electrochemical measurements remains an active area of research. In 2014, a new photothermal technique (termed as LIG) using a CO_2 laser was developed to convert a polyimide sheet into a porous carbon composed mainly of graphene/graphene oxide (1,2). The morphology, composition and chemical properties of LIG electrodes can be tuned by adjusting some laser parameters, production atmosphere, laser type or substrate. For instance, using an excessive laser power tends to increase the porosity but increases the formation of graphite over graphene, which enhance the electroactive area at the cost of charge transfer kinetics. The LIG's versality comes from the large number of substrates and lasers that can be used. As an example, a recent study reported its production using a 450 nm diode laser coupled with a 3D printer, which reduces cost while retaining some key morphological and composition features (3). Similar parameter adjustments apply to this laser type. However, certain parameters (e.g., lines per millimeter) lack systematic study. Here, we evaluate the impact of these overlooked variables on LIG's physical and electrochemical properties.

Experimental

LIG Production

The electrodes were produced in a polyimide sheet (Kapton®; ϕ = 0.15 mm) using a TTS-55 Pro® laser engraver with 35.5 % (1.95 W) of power and 2729 mm min^{-1} of speed. Immediately after production, a nail polishing coating was applied to the electrode stem and dried in ambient for 6 hours before the electrochemical measurements. Initially, the laser orientation was switched from vertical to horizontal. Ther vertical orientation was maintained and then the resolution (lines mm^{-1}) was varied (5, 8, 10, 15, and 20 lines mm^{-1}). The electrode design was created using inkspace® software, and the engraver was controlled by the LaserGRBL® (version 7.14.11) software. The working area of the electrode was 0.2375 cm^2.

Electrochemical Measurements

Electrochemical measurements were performed with a Potentiostat/Galvanostat PGSTAT 101 Metrohm-Autolab connected to a laptop and managed with the NOVA® (version 2.1.8) software. The electrochemical cell was assembled with a 10 mL beaker, an $Ag/AgCl/KCl_{(sat)}$ as the reference electrode, a platinum wire as the counter electrode and a LIG as the working electrode. Double layer Capacitance (C_{dl}) was determined via cyclic voltammetry varying the scan rate from 0.04 to 0.09 V s^{-1} in the interval of +0.2 to 0.0 V with KCl 0.1 mol L^{-1}. For each scan rate three measurements were made. The electrochemical characterization was performed by using cyclic voltammetry with $K_3[Fe(CN)_6]$ 0.1 mol L^{-1} (prepared in KCl 0.1 mol L^{-1}) as the redox probe. The potential was scanned from +0.8 to -0.2 V at 100 mV s^{-1}. The values of i_{pc} and ΔE_p represent an average of the consecutive measurements. For all the parameters a batch of three electrodes was made.

Results and Discussion

For a better presentation of the data, the electrodes were named following this pattern: Orientation – Resolution. The results obtained for the C_{dl} can be observed in Table 1.

Table 1. Values of C_{dl} obtained by cyclic voltammetry.

Eletrodo	C_{dl}* (µF)	RSD* (%)
(Horizontal – R10)	281 ± 17	6
(Vertical – R5)	227 ± 1	0.5
(Vertical – R8)	360 ± 5	1.5
(Vertical – R10)	403 ± 41	10
(Vertical – R15)	538 ± 13	2
(Vertical – R20)	651 ± 60	9

*n = 3 (for distinct electrodes).

The C_{dl} when the laser goes vertically is higher than horizontally even with the same resolution, indicating a higher electroactive area. A regard should be noted, when using the horizontal orientation, the shape of the working electrode was deformed from a circle to an ellipse, which may have contributed to this difference (Figure 1). Subsequently, the C_{dl} tends to increase with the resolution which indicates that the electroactive area is also increasing. This behavior could be explained considering that when the laser power is increased, a more porous surface is generated, thus, passing the laser multiples times over adjacent regions may cause a similar result.

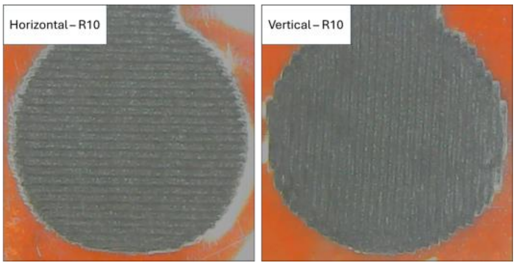

Figure 1. Photos of the electrodes with differents laser orientation.

The voltammetric responses for the $K_3[Fe(CN)_6]$ redox probe (Figure 2) were also compared. Table 2 presents the ΔE_p and i_{pc} acquired.

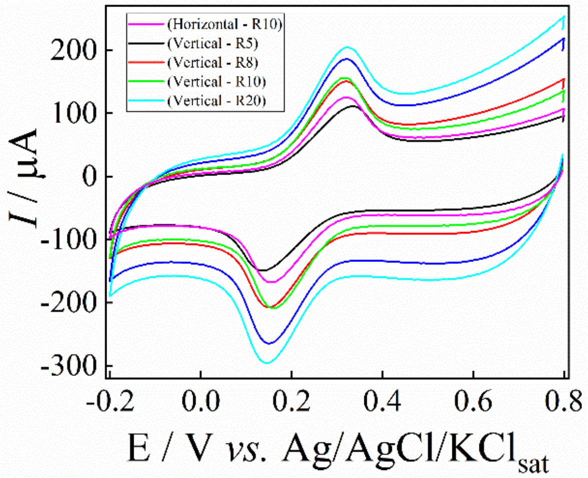

Figure 2. Cyclic Voltammograms recorded with differents LIGs in the presence of 0.1 mol L^{-1} of $K_3[Fe(CN)_6]$, prepared in KCl 0.1 mol L^{-1} at 100 mV s^{-1}.

Table 2. Values of i_{pc} and ΔE_p obtained from Figure 1.

Eletrodo	i_{pc}* (µA)	RSD* (%)	ΔE_p* (mV)	RSD* (%)
(Horizontal – R10)	97.87 ± 0.57	0.59	173 ± 7	4
(Vertical – R5)	86.01 ± 3.70	4.31	188 ± 15	8
(Vertical – R8)	109.02 ± 3.69	3.38	164 ± 11	7
(Vertical – R10)	119.09 ± 3.18	2.67	156 ± 6	4
(Vertical – R15)	131.37 ± 1.93	1.47	166 ± 2	1
(Vertical – R20)	136.10 ± 2.07	1.52	180 ± 14	8

*n = 3 (for distinct electrodes).

The LIG produced horizontally and at resolution 5 and 8 presented a resistive voltammetric profile. A less conductive and smaller electroactive area could have been generated and cause this. The ΔE_p did not tend to decrease by increasing the resolution, only the i_{pc} and C_{dl} tend to increase. The lowest value for the ΔE_p was at resolution 10. One hypothesis is that repeated laser passes over adjacent regions may increase porosity but degrade nearby graphene into graphite. This would increase the electroactive area, increasing the i_{pc} and C_{dl}, but decrease the charge transfer kinetics, increasing the ΔE_p.

Conclusions

The voltammetric response for $[Fe(CN)_6]^{3-}$ improved with increasing printing resolution up to 10 lines mm⁻¹, likely due to overlapping laser passes that enhance porosity and enlarge the electroactive surface of the LIG electrode. However, further increases in resolution impaired electron transfer, as excessive laser exposure may degrade the LIG structure, promote graphite formation and decrease electron transfer rate. This behavior highlights the need to optimize printing resolution to maximize the electrochemical performance of LIG electrodes.

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