

Article

Laser-Induced Graphene Dual Optical/Electrochemical Platform for In-Chip Sensing Applications

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Abstract

The present study addresses the development and characterization of an in-chip laser-induced graphene (LIG)-based sensor that combines optical and electrochemical transduction techniques as a proof of concept for the advancement of novel point-of-care (POC) devices. In recent years, LIG has emerged as a suitable material for next-generation diagnostic devices due to the increasing need for effective and easily accessible biosensing platforms. In this context, the presented sensors were fabricated and tested with an increasing number of laser exposures to understand how the resulting morphology, degree of graphitization, defects, and electrical resistance of LIG electrodes affect the electrochemical and optical sensing performance. To validate the dual sensor, ferrocyanide ($[\text{Fe}(\text{CN})_6]^{4-}$) was used as a redox probe and [(4-Dicyanomethylene)-2-methyl-6-(4-dimethylaminostyryl)-4H-pyran] (DCM) was used as model dye to explore the electrochemical and optical sensing capabilities. Finally, we showcase the sensor's ability to perform simultaneous optical and electrochemical on-time detection and analysis of the ferrocyanide electro-oxidation process, underscoring its potential to be used as a dual optical/electrochemical POC device.

Keywords: laser-induced graphene; optical; electrochemical; sensors; point-of-care

1. Introduction

Sensors and biosensors play a major role in daily life by being specifically tailored to patients' and regional demands. Recently, their use has become widespread in the form of point-of-care (POC) devices, which provide rapid diagnostic results without requiring expensive laboratory tests [1,2]. The principle underpinning them relies on the detection of biofluid samples which contain the target analytes (e.g., sweat, saliva and tears) that, once combined with specific bioreceptors (e.g., DNA/RNA and aptamers), produce a signal that can be converted into “readable data” by the transducer. The last can convert one form of energy (chemical, physical, etc.) into another (electrical, optical, gravimetric, thermal, etc.) [3,4]. Among the wide variety of biosensor and transducer types, optical and electrochemical biosensors are the most used in POC devices due to their sensitivity, adaptability and scalability [1–5].

In recent years, laser printing methods have become popular for the manufacturing of sensing platforms, due to their capacity for non-contact and high-precision production; roll-to-roll manufacturing; customized patterning; and their efficiency in cutting, curing



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or modifying the composition of materials [6]. This involves less material waste, quick processing, and high spatial precision [7], which are of the utmost importance considering the environmental standards/goals of the 2030 Agenda [8]. One example of the versatility of laser-based technologies is the production of laser-induced graphene (LIG). Discovered in 2014, the CO₂ laser photothermal conversion of a polyimide (PI) polymer sheet resulted in a novel type of three-dimensional, porous graphene derivative, with large surface area and high electrical conductivity [9,10]. Indeed, LIG has been extensively investigated as a versatile carbon nanomaterial that can be formed on numerous precursor substrates, including polyimide films [11–13], wood [14,15], paper [16,17], cellulose-based materials [18,19], and lignin-rich biopolymers [20,21], among others. Its properties can be tuned by adjusting the substrate type and laser parameters, being particularly attractive for sensor development, especially biosensors due to the high surface-to-volume ratio, strong interactions with biomolecules, wide light absorption, and superior biocompatibility. Additionally, the 3D porous structure enhances electron transfer, facilitates immobilization of biomolecules, allows integration with microfluidic or wearable platforms, and supports functionalization with enzymes, antibodies, aptamers, or other biorecognition elements. In this context, compared to other graphene derivatives—produced either by the scotch tape method (pristine graphene) [22], chemical vapor deposition (polycrystalline graphene) [23], or chemical/physical methods (graphene oxide (GO) or graphene quantum dots (GQDs)) [24,25]—LIG-based transducers were found to have several advantages such as relying on quick and easy fabrication processes; digital computer manipulation; and extremely promising intrinsic electrochemical and optical characteristics. As a result, LIG has been used in diverse sensing platforms for the detection of metabolites, pathogens, environmental toxins, and physiological markers, highlighting its potential for low-cost, scalable, and flexible (bio)sensing devices [26–34], and therefore suitable for POC applications.

In this work, we demonstrate a dual-mode LIG-based platform that integrates optical fibers with electrochemical LIG electrodes to enable both optical and electrochemical in-chip measurements within the same device. To produce such a platform—capable of performing both in-chip optical and electrochemical measurements—we have explored the setup characteristics to enhance performance, addressing: (i) different laser passages on LIG, (ii) various laser powers and speeds to pattern methacrylate sample chambers, and (iii) different laser types to cut the PDMS component of the platform. Afterwards, the setup was first characterized as an electrochemical sensor via cyclic voltammetry (CV) and differential pulse voltammetry (DPV) techniques using a ferrocyanide/ferricyanide redox couple ($[Fe(CN_6)]^{4-} / [Fe(CN_6)]^{3-}$), which is a model redox couple used in sensors and biosensors [6] due to its reversible and stable electrochemical response. Secondly, we explored the setup as an optical sensor using DCM dye [(4-Dicyanomethylene)-2-methyl-6-(4-dimethylaminostyryl)-4H-pyran] as a model analyte—a pink-colored organic dye—due to its stability and graphene quenching ability [35,36]. Finally, we carefully assessed simultaneous electrochemical and fluorescence measurements on the LIG platform, allowing us to improve on single-mode sensors, which could overcome a major drawback of existing POC technologies by avoiding false positives, and we therefore enhanced reliability while maintaining cost-effectiveness and scalability. This study not only demonstrates as proof of concept the feasibility of using LIG in multifunctional future biosensors but also contributes to the broader goal of advancing next-generation POC technologies that are adaptable, user-friendly, and potentially suitable for real-time monitoring in clinical and environmental contexts.

2. Materials and Methods

2.1. Electrochemical and Optical Setup Apparatus

2.1.1. LIG Synthesis

LIG electrodes were fabricated using a CO₂ laser, wavelength of 10.6 μm , Redsail M500 Laser System, Jinan, China (see Figure 1), operated through RDWorks V8 software. The parameters for the fabrication were based on previous studies by our group [37,38], which utilized different laser powers, passages and speeds to produce LIG, and compared their microstructures. This comparison served to optimize the presented laser parameters and encompasses the parameters used throughout the current work—using the same laser writing system. Therefore, a target laser power of 6.5 W, an interline distance of 80 μm and, finally, 150 mm/s of laser scan speed. The PI film (127 μm thickness) was positioned 2 cm down the laser head, slightly defocused (focus distance is 1.8 cm). LIG was produced employing 1, 2 and 3 laser passages, named LIG1, LIG2 and LIG3, respectively, in order to verify the impact of subsequent (reducing) laser passages on the formed LIG (see Figure 1a,b) and on the electrochemical/optical response of the prototype.

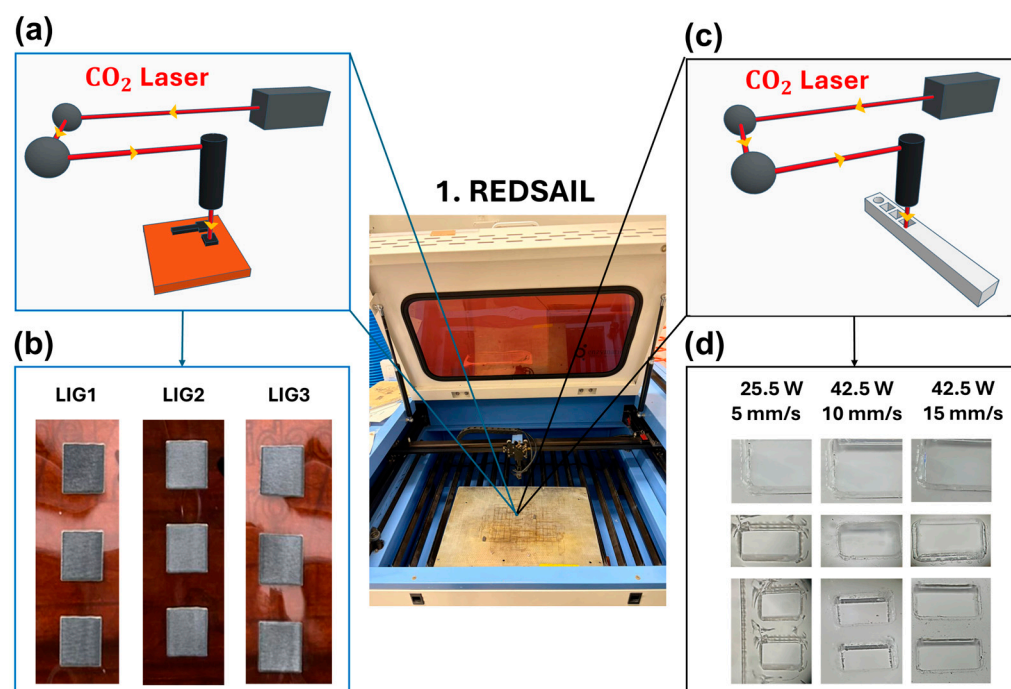


Figure 1. (a) Schematic of laser beam of LIG; (b) photograph of LIG samples with 1, 2 and 3 laser passages; (c) schematic of laser beam and cutting of methacrylate chamber plaque; (d) cutting parameters and respective photographs of methacrylate chamber plaque. Arrows between (a,b) and (c,d) represent the transition from 3D representation of the process to the real images.

2.1.2. Fabrication of Methacrylate Chambers

Methacrylate sample plaques with a size of 68.8 mm $\times$ 8 mm $\times$ 5 mm; reservoir size of 4 $\times$ 4 $\times$ 5 mm³; maximum capacity of 145 μL ; and spacing between the chambers of 3.6 mm were fabricated with different parameters (see Figure 1c,d) using the same continuous CO₂ laser system (Redsail M500). A power output of 42.5 W, a laser head scan speed of 10 mm/s, and a distance between the laser head and the methacrylate plate of 2 cm were chosen as the optimal parameters for smoother, crack-free production of methacrylate chamber plaque.

2.1.3. PDMS Layer Production and Cut

A thin PDMS film (thickness of 0.68 mm) was produced using 10:1 silicone elastomer base:polymer curing agent, and cured for 3 h under 100 $^{\circ}\text{C}$. Then, as illustrated

in Figure 2, two different methods were used to test and optimize PDMS cutting in our system: continuous CO₂ laser (1), and pulsed picosecond UV laser (2).

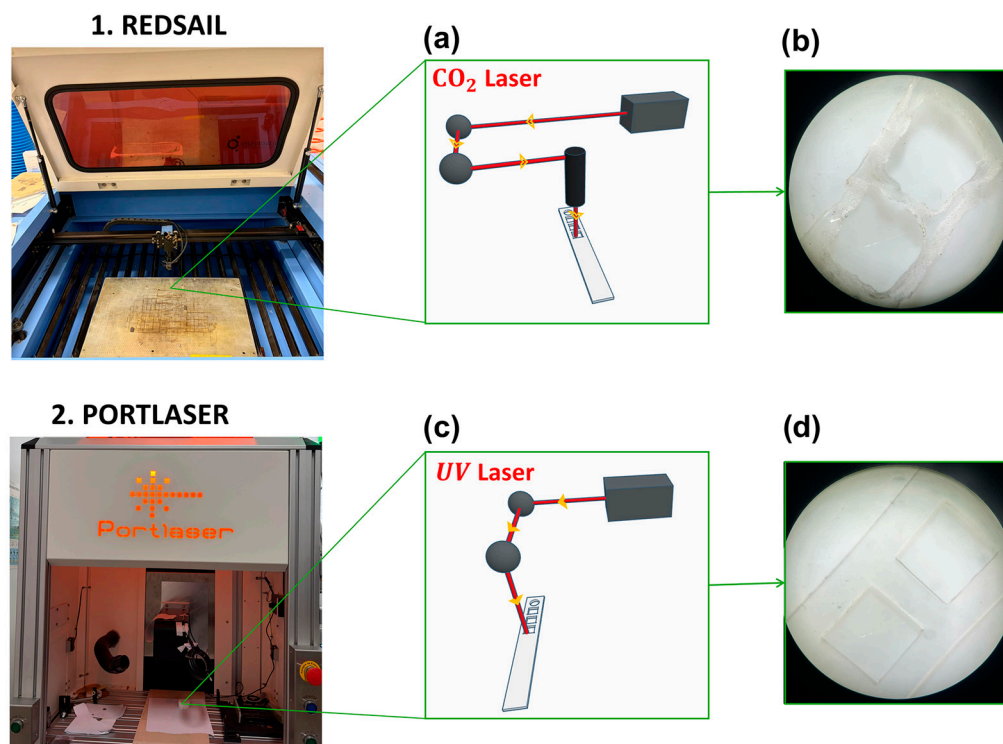


Figure 2. PDMS cut through different laser systems: (a) Redsail laser beam path; (b) PDMS sample cut with CO₂ laser; (c) PORTLASER laser beam path; (d) PDMS sample cut with UV laser.

- (1) CO₂ laser—The Redsail system with a power of 42.5 W and 5 mm/s speed was used, with 2 cm between the laser head and PDMS. Distilled water was used, before and after soaking the PDMS in isopropanol for 10 min, to remove the charred regions formed in the cut edges (see Figure 2a,b).
- (2) UV laser—The Portlaser system, Barcelos, Portugal was used with a power of 100 W and a velocity of 100 mm/s, and a distance of 0.34 mm for 10 cycles. After, the PDMS samples were treated with isopropanol for 10 min using an ultrasonic bath (operated in water), to remove the impurities (see Figure 2c,d).

As readily seen from Figure 2, the pulsed picosecond UV laser can deliver high peak power with minimal thermal damage to surrounding areas, thus allowing for a very precise and clean PDMS cutting without structural deformation, contrarily to the highly thermal continuous CO₂ laser.

2.1.4. Chip Assembly

Figure 3 shows a step-by-step diagram for construction of the chip. After consecutive production and cutting of the LIG, methacrylate chambers and PDMS layer, the chip was assembled by placing the LIG, PDMS (UV patterned) and methacrylate chambers, respectively. The PDMS layer interconnects the methacrylate chamber plaque with the PI sheet containing the LIG electrodes in a compact structure to avoid leakage and to minimize interference with the LED light transmission. This layer also enables the reuse of the entire system (chip) and, therefore, only LIG electrodes are disposable—which is a step forward in terms of reusability.

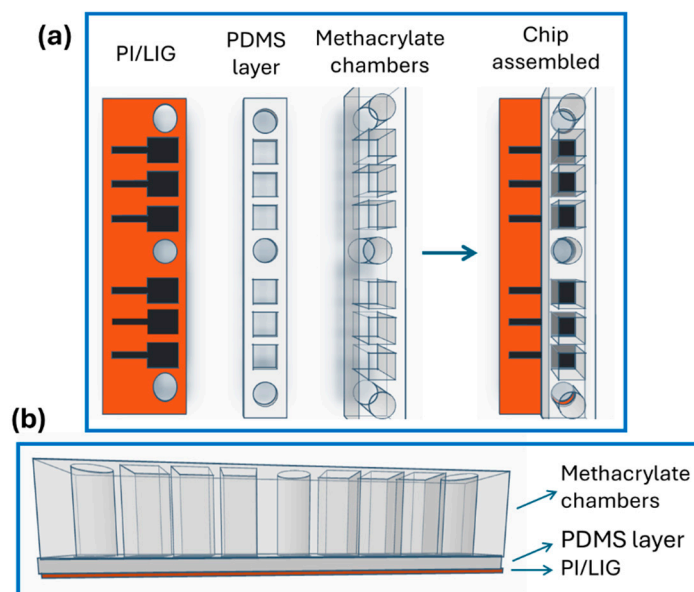


Figure 3. Step-by-step diagram for constructing the chip: (a) the separate components; (b) the order of the components for the construction of the sensor.

2.1.5. LIG Characterization

Raman spectra were acquired using a Wasatch 532 ER spectrometer, Logan, Utah, USA coupled to an Olympus BH2 microscope, Tokyo, Japan (100 $\times$ lens) and a Cobolt 532 nm laser, Stockholm, Sweden (40 mW). The LIG samples' morphology was characterized by scanning electron microscopy (SEM) using a TESCAN Vega3 SB SEM, Brno, Czech Republic, at an accelerating voltage of 15 kV across magnifications of 200 $\times$, 2000 $\times$, and 20,000 $\times$. A working distance of 4 mm was maintained. Areas of interest were selected based on the presence of both macropores and micropores.

Next, 6 $\times$ 6 mm² LIG samples were produced for the sheet resistance (R_s) measurements. The R_s was measured through the Van der Pauw method [39] in 3 squares for each sample type using a homemade system, comprising a KEYSIGHT Precision Source/Measure Unit, Santa Rosa, California, USA, an Arduino Uno controller, Turin, Italy and MATLAB R2024b software. A total of 15 measurements were performed for each LIG type.

2.1.6. Electrochemical Cell and Optical Fiber Assembly

Figure 4 shows the assembling of the optical detection setup and the electrochemical setup. A green LED was used as the light source, and an optical fiber was used to collect the light and transmit it to a spectrometer (FLEX RES+ Vis/NIR Sarspec, Vila Nova de Gaia, Portugal) with 0.19 nm resolution and a detection range between 180 and 890 nm. The employed optical fiber was a segment of silica step-index multimode fiber, with a core diameter of 600 μ m (FP600URT, Thorlabs, Newton, NJ, USA). A plier was used to cut the segment into the present length and polished using paper to make sure that both ends of the segment were perfectly sleek. One of the ends was used to collect the light, and the other one was placed in an optical fiber connector to transmit the light to the spectrometer.

The LED optical positioner was produced in PLA using a 3D printer (Bambu, X1 Carbon, Shenzhen, China) to maintain the distance and position of the LED in each measurement. The LED was connected to an electronic circuit to switch on/off (Figure 4a).

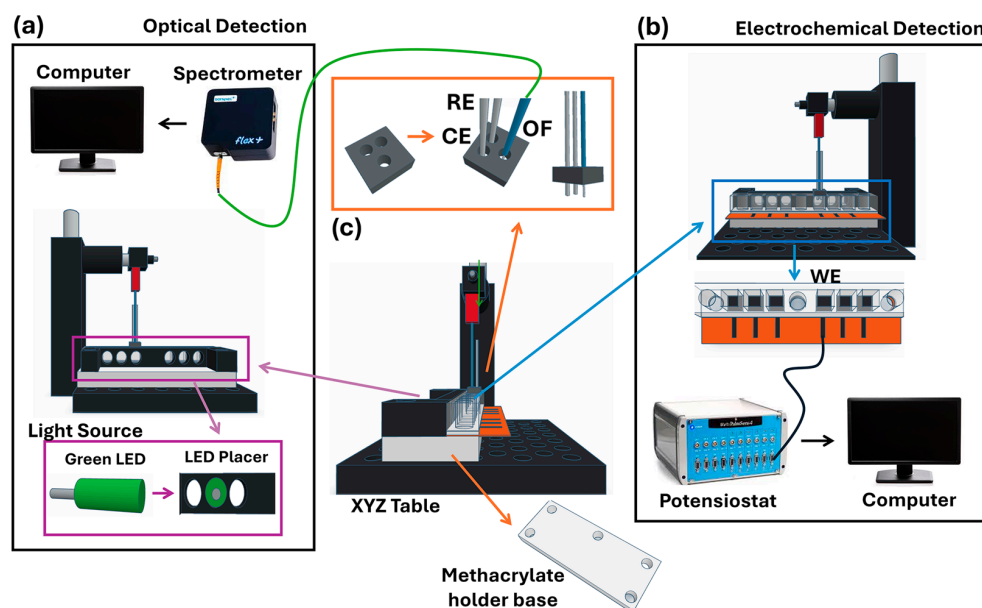


Figure 4. Dual optical/electrochemical platform for in-chip for biosensing applications: (a) optical detection components, (b) electrochemical detection components, and (c) setup.

For the production and assembly of the electrochemical cell (Figure 4b), LIG was used as the working electrode (WE) and Platinum wire (Sigma Aldrich, Louis, Missouri, USA) was used as the counter (CE). A homemade Ag/AgCl reference electrode (RE) was produced by (i) covering a silver wire (Sigma Aldrich) with Ag/AgCl ink (60/40, Sigma Aldrich); (ii) leaving it to cure at room temperature (RT) for 3 days; and (iii) finally insulating uncoated regions of the Ag/AgCl wire with epoxy, leaving a 2 mm Ag/AgCl active area. The RE was stored in 1 M KCl solution at RT.

A 3D piece of polylactic acid (PLA) was also produced (Figure 4c) using a 3D printer (Bambu, X1 Carbon) to assemble the electrodes with the optical fiber, allowing one to maintain the same placement/distance of electrodes throughout the measurements. Consequently, a small-sized device was produced, allowing both optical and electrochemical detections.

2.1.7. Setup Assembly

The LIG-based modular device is assembled according to the images in Figure 5, as follows: firstly, an XYZ table (Figure 5a) was mounted onto the optical stage to allow precise control of the alignment in all dimensions during optical and electrochemical tests. The base (Figure 5b) was then mounted onto the table to create a stable environment to place both the final “sensor” and the LED placer (Figure 5c). Previously mounted CE, RE and optical fiber were stacked into the 3D-printed holder (Figure 5d). After preparation of the “setup”, the sensor was then constructed by first placing the LIG electrode (Figure 5e), PDMS film (Figure 5f) and, finally, methacrylate chambers (Figure 5g). The assembly (Figure 5h) was tightened to the base with screws. Overall, the setup allowed one to connect to the WE from one side while optical excitation was achieved through the other side simultaneously, and the optical signal was measured via the optical fiber placed perpendicularly (Figure 5i).

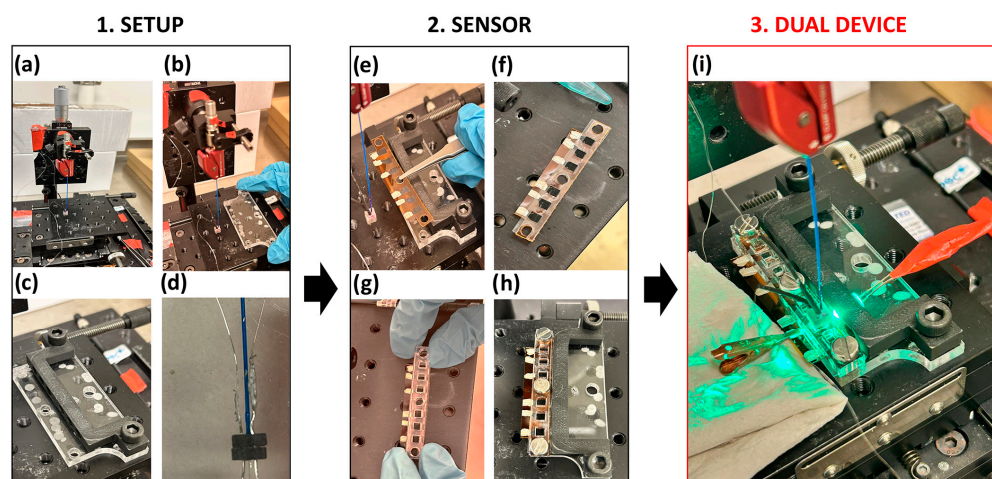


Figure 5. Setup assembly. 1. SETUP: (a) XYZ table and 3D positioner; (b) methacrylate base; (c) LED positioner; (d) 3D-printed holder. 2. SENSOR: (e) LIG electrodes; (f) PDMS on LIG electrodes; (g) chips on PDMS and LIG (final sensor); (h) assembly on base with screws. 3. DUAL DEVICE: (i) dual-sensing device.

2.2. Optical and Electrochemical Platform Testing

2.2.1. Electrochemical Experiments

All electrochemical measurements were performed in a three-electrode configuration consisting of LIG WE with $4 \times 4 \text{ mm}^2$, an Ag/AgCl quasi-RE and a platinum wire as the CE. All measurements were performed at room temperature using a PalmSens4 Potentiostat, Houten, The Netherlands. Activation of the electrodes was performed via CV in phosphate-buffered saline (PBS, pH 7.4, 0.01 M, tablets purchased from rom Fisher Bioreagents, Waltham, Massachusetts, USA) solution, from -1.0 V to $+1.0 \text{ V}$ vs. Ag/AgCl at 0.1 V/s for 10 cycles. Afterwards, measurements for system validation employed 10 mM potassium hexacyanoferrate (II) trihydrate ($\text{K}_4[\text{Fe}(\text{CN})_6] \cdot 3\text{H}_2\text{O}$, AGR) (purchased from Labkem, Barcelona, Spain) in PBS solution with increasing scan rates, namely, 0.005, 0.01, 0.015, 0.020, 0.025, 0.030, 0.035, 0.040, 0.050, 0.055, 0.060, 0.080, 0.100, and lastly, 0.120 V/s . Finally, concentrations of 0.1, 0.5, 1, 25, 50, 70, and $100 \mu\text{M}$ of ferrocyanide solution were measured via DPV using a pulse height of 30 mV and pulse width of 20 ms, in line with the optimized values in previous research [37]. Three replicas were made to gather results for each method.

After taking the mean value of the three replicas, LODs and LOQs were calculated by Equation (1) and Equation (2), respectively:

$$\text{LOD} = 3.3 \times \frac{\sigma}{S}, \quad (1)$$

$$\text{LOQ} = 10 \times \frac{\sigma}{S}, \quad (2)$$

where σ and S (sensitivity) correspond to the standard deviation of y-intercept and the slope of linear fittings, respectively.

2.2.2. Optical Experiments

Solutions of DCM dye (Lambdachrome Laser Dyes, Göttingen, Germany) were prepared in concentrations of 33.00, 16.50, 8.25, 4.13, and $2.06 \mu\text{M}$. A green LED was used as the optical source to excite the dye. The optical fiber tip was placed at a 4.5 mm distance from the WE to collect the light emitted by the dye, by using a spectrometer (FLEX RES+ Vis/NIR Sarspec) with 0.19 nm resolution and a detection range between 180 and 890 nm. All optical measurements were recorded using the LightScan Software

Version 1.1.18 with 32 ms of integration time. Three replicas were made to gather results for each method. With respect to data processing, water was used as the blank condition for optical measurements. Background subtraction was not performed. Instead, the recorded emission spectra were normalized to the corresponding blank spectra by dividing the sample intensity (I) by the blank intensity (I_0). LOD and LOQ were determined using Equation (1) and Equation (2), respectively.

2.2.3. Dual Sensing Experiments

Oxidation of ferrocyanide into ferricyanide yields a progressive change from a transparent to a yellowish-colored solution. A chronoamperometric method using a fixed potential of 0.5 V vs. Ag/AgCl was applied to oxidize an unstirred 20 mM ferrocyanide solution. The color change was followed optically for 33 min of the electrooxidation process, using an integration time of 95 ms and white LED excitation.

3. Results and Discussion

3.1. LIG Production and Characterization

Figure 6 shows the SEM images representative of all the LIG sample types produced in this work. The films denote the usual hill/valley topography arising from the successive linear irradiations of the Gaussian profile beam. The well-known 3D-like porous morphology characteristic of LIG is also patent-pores formed in different sizes with an interconnected and hierarchical arrangement, as frequently observed in LIG [37,38,40]. Increasing the number of laser exposures tends to produce larger pores and less uniform films (Figure 6a). The apparent larger macropores of LIG3 could also be a consequence of local graphene tear-off by successive laser irradiation. In general, LIG1 is composed of thinner graphene flakes compared to LIG2 and LIG3.

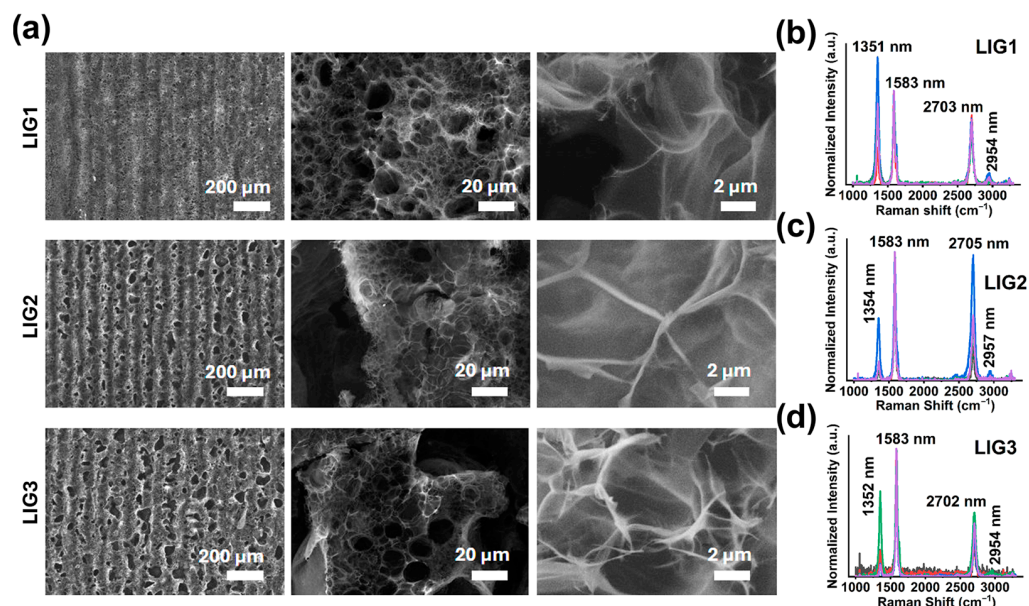


Figure 6. (a) SEM images of LIG samples, and Raman spectra of (b) LIG1, (c) LIG2, and (d) LIG3 samples, acquired using excitation wavelength of 442 nm. Different color lines between (b–d) represents the replicates ($n = 5$).

To further elucidate the structure of LIG samples, Raman spectroscopy was performed (Figure 6b–d). All samples gave an excellent match with the expected bands of LIG for an excitation wavelength of 442 nm [41] (D-band around 1351 cm^{-1} , G-band around 1583 cm^{-1} , and 2D band around 2700 cm^{-1}). LIG is formed in both hill and valley regions,

and Raman spectra were acquired from both. Using the corresponding peak area values to calculate $\frac{I_D}{I_G}$ and $\frac{I_{2D}}{I_G}$ ratios (Table 1), one can qualitatively infer the evolution of defect density and overall crystallinity of the LIG. The higher $\frac{I_D}{I_G}$ ratio for LIG1 shows higher defect density compared to other samples, indicating that the laser exposures are turning the graphene flakes into more ordered graphitic domains. Theoretically, this means a lower oxidation degree for LIG2 and LIG3. Indeed, subsequent laser irradiation of LIG results in the lowering of its oxidation degree, because of the reductive nature of the local environment created by the laser irradiation [38], which is also observable to the naked eye as LIG's appearance evolves from blackish to greyish tones [42,43] (see Figure 1b). The graphitization is also observable by the decrease in the $\frac{I_{2D}}{I_G}$ ratios after secondary laser exposures, which is consistent with an increase in the number of graphene layers in the flakes. This is aligned with the qualitative observation of thinner flakes for the LIG1 samples in the SEM images.

Table 1. Characterization results from Raman spectroscopy and Van der Pauw measurements for all LIG samples.

	LIG1	LIG2	LIG3
$\frac{I_D}{I_G}$	0.95 ± 0.41	0.19 ± 0.16	0.24 ± 0.23
$\frac{I_{2D}}{I_G}$	0.67 ± 0.07	0.50 ± 0.26	0.36 ± 0.08
R_s ($\Omega/\square$)	16.98 ± 0.02	11.96 ± 0.56	12.08 ± 0.11

Van der Pauw measurements were performed to investigate the impact of different laser passages' number of exposures in the final sheet resistance (Table 1). The results show the lowest R_s values for the LIG2 and LIG3 samples, with similar values. This correlates well with a lower defect density/lower oxidation degree of LIG after laser exposure. Moreover, the standard deviation of R_s is lower for LIG1 samples, indicating higher reproducibility for this sample type.

3.2. Sensor-in-Chip Validation

An electrochemical biosensor's reusability, stability, and reliability are all affected by electrochemical reversibility, which shows that the sensor can return to its initial electrochemical state following each redox cycle, allowing for repeated use without losing functionality. Theoretically, a reversible electron transfer process can be verified through linear regression obtained from the Randles–Ševčík equation, which relates the peak current (i_p) to the square root of scan rate ($v^{1/2}$), and is indicated by a high coefficient of determination ($R^2 \approx 1$), which indicates reversibility [44]. For this purpose, CV was performed and the results showed that the LIG1 and LIG2 electrodes had R^2 values of 0.99 (Figure 7a,b), suggesting high reversibility of the redox process in the range of scan rates studied. However, LIG3 exhibits a much lower R^2 value of 0.77, especially in the oxidation branch, indicating loss of reversibility. Lower electron transfer kinetics manifest by a departure from linearity at higher scan rates. This matches well the established causality between the presence of edge defects and oxygen functional groups on the kinetics of ferro/ferricyanide inner-sphere redox activity at carbon-based surfaces [37].

Reversibility is also further evidenced via the peak potential separation, annotated by ΔE_p , and anodic-to-cathodic peak ratio, $\frac{I_a}{I_c}$. In general, smaller ΔE_p values and $\frac{I_a}{I_c}$ ratios close to 1 are indicative of more reversible electron transfer behavior. As shown in Figure 8a, $\frac{I_a}{I_c}$ ratios for LIG1 and LIG2 remained close to 1.0, demonstrating symmetrical redox behavior and strong reversibility. LIG3, however, showed higher deviation with values exceeding 1.2, especially at increased scan rates, reflecting quasi-reversible or even

irreversible electrochemical behaviour. Also, as can be seen in Figure 8b, LIG1 and LIG2 showed relatively low and consistent ΔE_p values across increasing scan rates. LIG3, on the other hand, exhibited noticeably higher ΔE_p values, indicating more sluggish electron transfer kinetics.

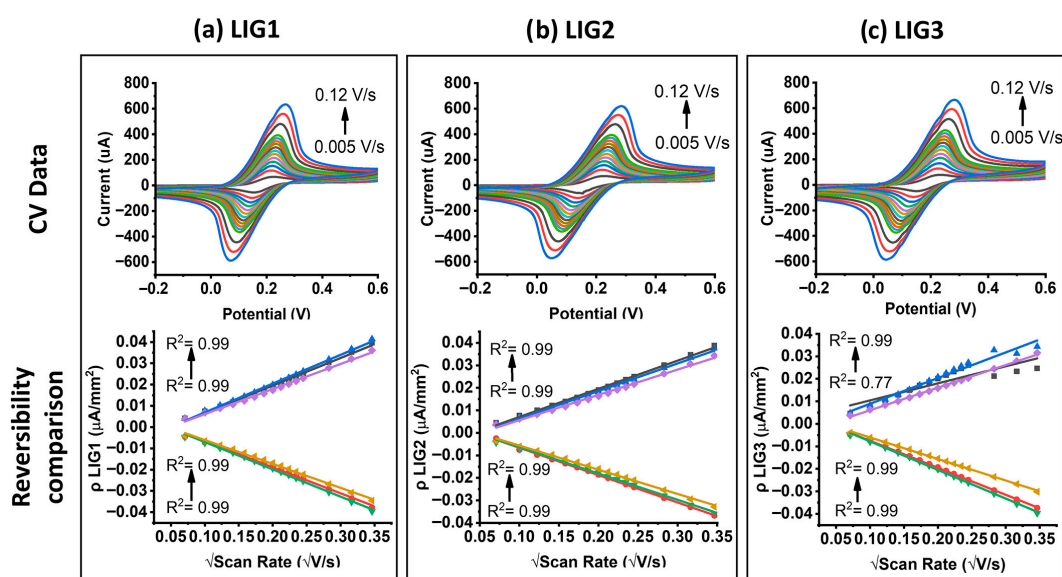


Figure 7. Characterization results from CV varying the scan rate for (a) LIG1, (b) LIG2, and (c) LIG3. Measurements acquired in 10 mM ferrocyanide in PBS solution.

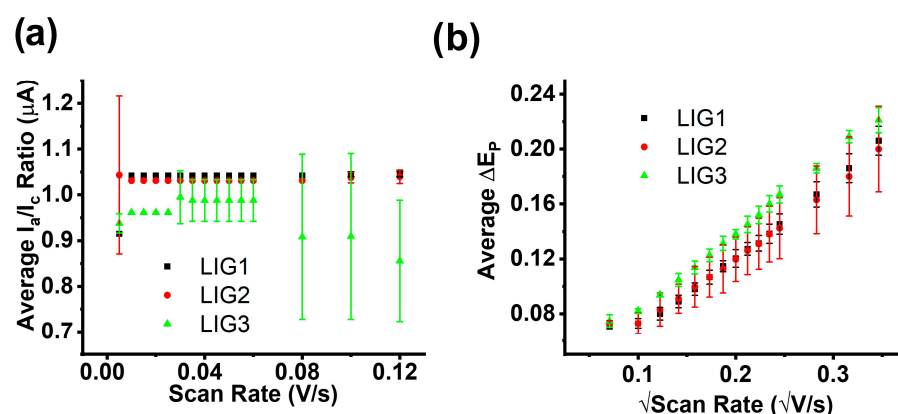


Figure 8. CV data of LIG samples: (a) anodic-to-cathodic peak intensity ratios; (b) peak potential separation comparison. Error bars are standard deviations from triplicate measurements.

DPV was employed to determine the LOD, LOQ, sensitivity and reproducibility of ferrocyanide detection with the proposed platform using LIG1, LIG2 and LIG3 electrodes ($n = 3$). The representative DPVs taken at different concentrations of ferrocyanide (from 0.1 to 100 μM) and the corresponding calibration curves are shown in Figure 9d and summarized in Table 2. LIG1 provides higher sensitivity (6.01 $\mu\text{A}/\mu\text{M}$), along with larger absolute detection currents compared to other LIG electrodes. The LOD for LIG1 is 0.24 μM , whereas for LIG2 and LIG3, it is higher (1.04 μM and 0.48 μM , respectively). These LODs are calculated using Equation (1), within the lower concentration range (see inset of Figure 9d). LIG2 and LIG3 electrodes demonstrate higher LOD (1.04 μM and 0.48 μM , respectively) and LOQ (3.14 μM and 1.45 μM , respectively) while having lower sensitivities (1.82 $\mu\text{A}/\mu\text{M}$ and 2.05 $\mu\text{A}/\mu\text{M}$, respectively), showing a restricted capacity for low concentration detection. These results suggest that LIG1 is the most promising electrode type for sensitive and reliable biosensing applications.

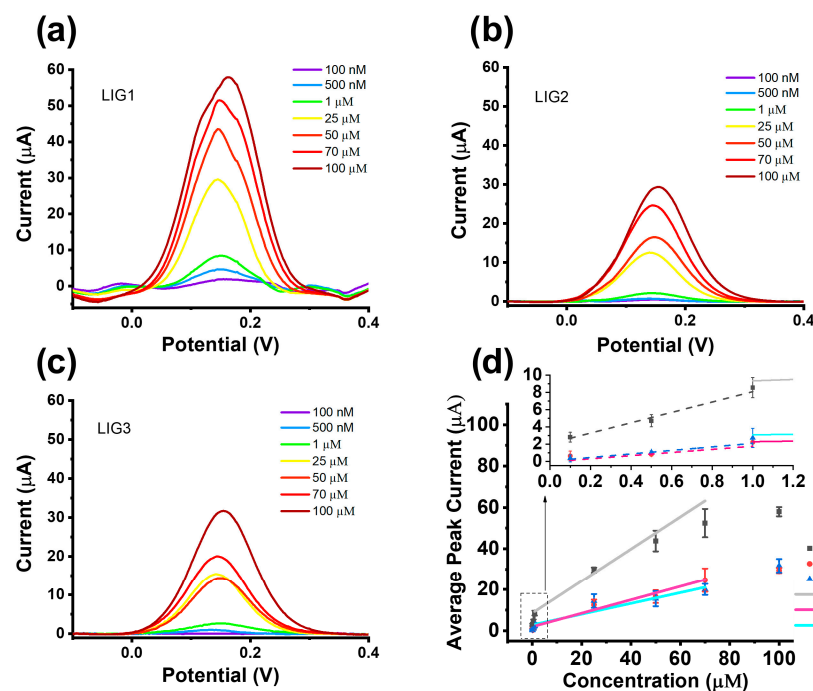


Figure 9. Analysis of background-subtracted DPV measurements as a function of ferrocyanide concentration in PBS solution, for the three LIG samples, (a) LIG1, (b) LIG2, and (c) LIG3, and (d) comparison of the LIG samples by peak currents in corresponding concentrations. Inset Figure represented with dash lines stand for concentration range of 0–1.0 μM. Error bars are standard deviations from triplicate measurements.

Table 2. Sensitivity, LOD and LOQ values for all LIG samples from electrochemical (DPV) detection of ferrocyanide and optical detection of DCM tests.

Samples	Electrochemical Test (Ferrocyanide)				Optical Test (DCM)		
	Sensitivity (Range: 0.1–1 μM) (μA/μM)	Sensitivity (Range: 1–70 μM) (μA/μM)	LOD (μM)	LOQ (μM)	Sensitivity (a.u./μM)	LOD (μM)	LOQ (μM)
LIG1	6.01	0.78	0.24	0.72	0.026	1.12	3.38
LIG2	1.82	0.33	1.04	3.14	0.034	3.31	10.0
LIG3	2.05	0.26	0.48	1.45	0.131	0.73	2.21

The optical detection capability of the platform was also tested using DCM dye as a model. The absorbance spectrum of DCM dye was first recorded using a spectrophotometer (UV2100, from Shimadzu Corporation, Kyoto, Japan) to experimentally demonstrate that the green LED is a suitable excitation source. As observed in Figure 10, the DCM has two absorption bands: (i) a lower energy band around 565 nm, which is relative to the electronic transition $S_0 \rightarrow S_1$ (fundamental singlet state for the first excited singlet state); (ii) another higher energy around 525 nm, relative to the $S_0 \rightarrow S_2$ transition (fundamental singlet for the second excited singlet state). Following Kasha's rule, both excitations relax for the lower state of vibrational energy in the first excited singlet state and emit light (Franck–Condon principle vertical transitions) [45]. Using the green LED with a maximum emission at 517 nm, we can excite the DCM dye and observe an emission band peak at approximately 587 nm, as displayed in Figure 10.

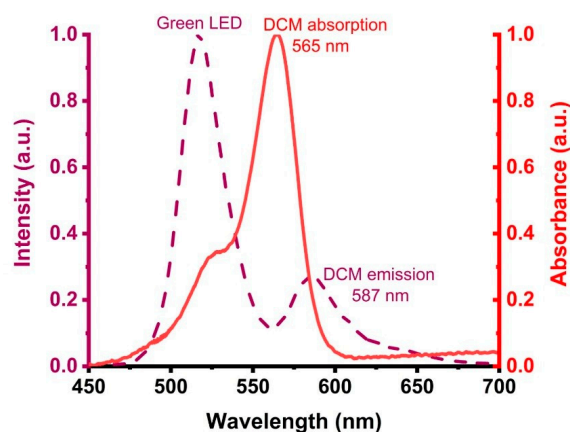


Figure 10. Absorbance spectrum of DCM dye using a Shimadzu spectrophotometer, and emission spectrum of DCM dye (66 μM) using a green LED as the light source in the developed platform.

After confirming that a green LED can be used to excite DCM dye (concentration range between 2.06 and 33 μM), we proceeded to determine the minimum concentration of the dye that can be measured with the developed platform (Figure 11). Under green LED exposure, a characteristic emission peak of DCM was observed for all the samples with a maximum emission at 585 nm for LIG1 and LIG2, and 587 nm for LIG3. These peaks remain consistent through different concentrations and increase with increasing concentrations, revealing the concentration-dependent optical response and reproducibility of DCM dye under green LED. The optical characterization was accomplished by comparing normalized intensity vs. concentration for each LIG type, as can be seen in Table 2. Considering the emission intensities for similar DCM concentrations, there is a noticeable (from data in Figure 11) increase in the output fluorescence intensity (e.g., I/I_0) with increasing laser passages: LIG3 > LIG2 > LIG1. This suggests lower DCM adsorption with increasing laser passages, subsequent decrease in fluorescence quenching, and higher effective (active) DCM present in the solution to be measured. According to this, augmented LODs compared to LIG3 are reflected in the experimental results where LIG3 showed the highest sensitivity (0.13 a.u./ μM), and lowest LOD (0.73 μM) and LOQ (2.21 μM).

Jaemyung Kim et al. [36] have reported the efficient quenching capability of DCM using GO materials at different oxidation rates, with a slight increase in quenching for higher reduced GO materials (due to higher graphitic domains). As stated previously in this manuscript, LIG1 presents higher defect density, whereas LIG3 has fewer defects (more ordered graphitic domains). These results are contradictory to theoretically expected quenching performance results described elsewhere that correlate increasing graphitic domains with increasing quenching efficiency [36]. In this context, the increase in the quenching capability of LIG materials presented in this work can instead be experimentally related to the micro/macroscale structure (as presented in SEM images, Figure 6) and surface chemistry resulting from laser passages. A synergy between (i) increasing micro/macropore structure; (ii) lower structural defects; (iii) lower expected quantity of oxygen functional groups; and (iv) consequent higher hydrophobicity of LIG materials might affect DCM and LIG interactions and decrease their quenching capability. This is in line with electrochemical measurements—where LIG1 presented better reversibility and sensitivity (e.g., higher defects, higher oxygen content)—and constitute important results regarding adsorption of biomolecules onto the sensor surface that go beyond the scope of this manuscript and need to be further addressed in the future.

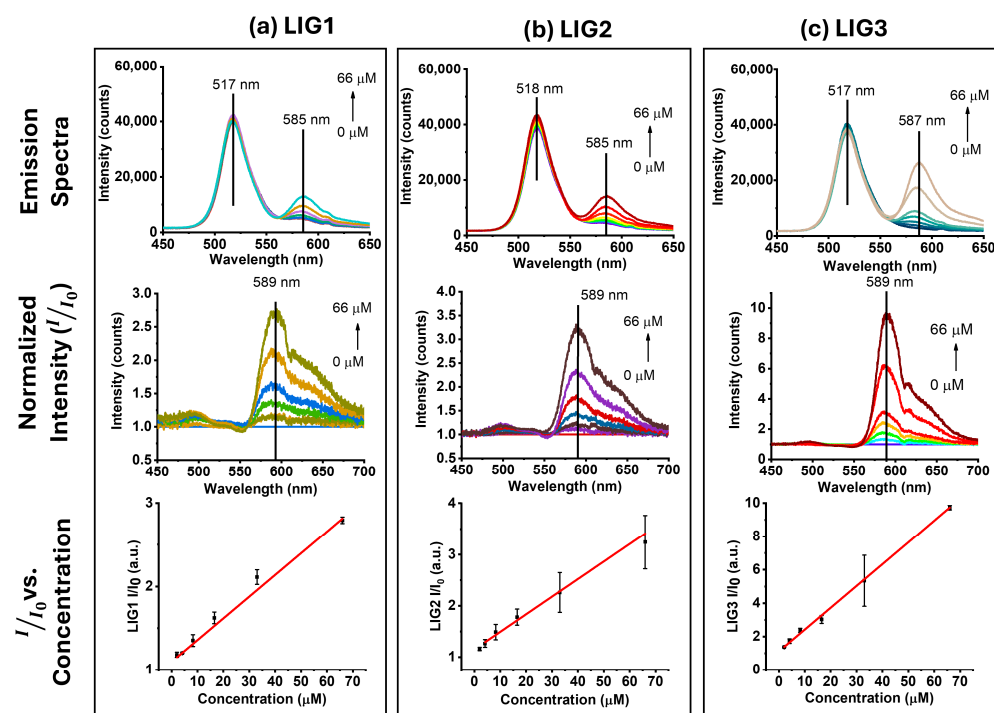


Figure 11. Optical response using green LED for the different LIG samples: (a) LIG1; (b) LIG2; (c) LIG3. Different color lines represent different concentration-dependent responses of, from bottom to top, 0–66 μM DCM.

3.3. Dual Optical/Electrochemical Sensor

The simultaneous dual sensing is demonstrated in Figure 12, combining: (i) electrochemical oxidation (diffusion-limited) by applying a stable potential of +0.5 V vs. Ag/AgCl and respective current recording as a function of time t , with (ii) optical detection by acquiring optical spectra every minute throughout 33 min in total (Figure 12a,b). Figure 12c shows the methacrylate chamber used for the configuration of the experiments and electrodes. Under applied potential, the colorless ferrocyanide ($[\text{Fe}(\text{CN})_6]^{4-}$) oxidizes at +0.5 V vs. Ag/AgCl to ferricyanide ($[\text{Fe}(\text{CN})_6]^{3-}$), turning to a yellow color (Figure 12d,e). Each ferrocyanide to ferricyanide step transfers one electron. Because water oxidation is negligible at this potential, and assuming no other faradaic activity, the current (Figure 12b) is essentially due to the oxidation of ferrocyanide to ferricyanide only, and decays as $i \propto t^{-1/2}$. By Faraday's law, the integrated charge Q (in coulombs) passed gives the moles of ferricyanide formed:

$$n_{\text{Ferri}} = Q/F, \quad (3)$$

where F ($\approx 96,485$ C/mol) is the Faraday constant.

Ferricyanide is yellow because it strongly absorbs blue light. Its visible spectrum has a peak around 420 nm [46], whereas ferrocyanide does not absorb significantly in the visible. Consequently, as ferricyanide rises, the 420–450 nm intensity drops (blue is absorbed, Figure 12b). For moderate absorption regimes such as the present, the measured optical absorbance (or loss of reflectance) at 420–450 nm is directly proportional to the ferricyanide concentration, following a Beer–Lambert-like rule. In fact, since ferricyanide is the only visible light-absorbing compound, one expects that a two-wavelength ratio—using the peak intensity at ~ 560 nm where negligible absorption occurs to account for, e.g., LED instability—to reliably track ferricyanide. Therefore, I_{560}/I_{420} should rise with the cumulative charge passed, as observed qualitatively in the raw data of Figure 12a,b and quantitatively in Figure 13.

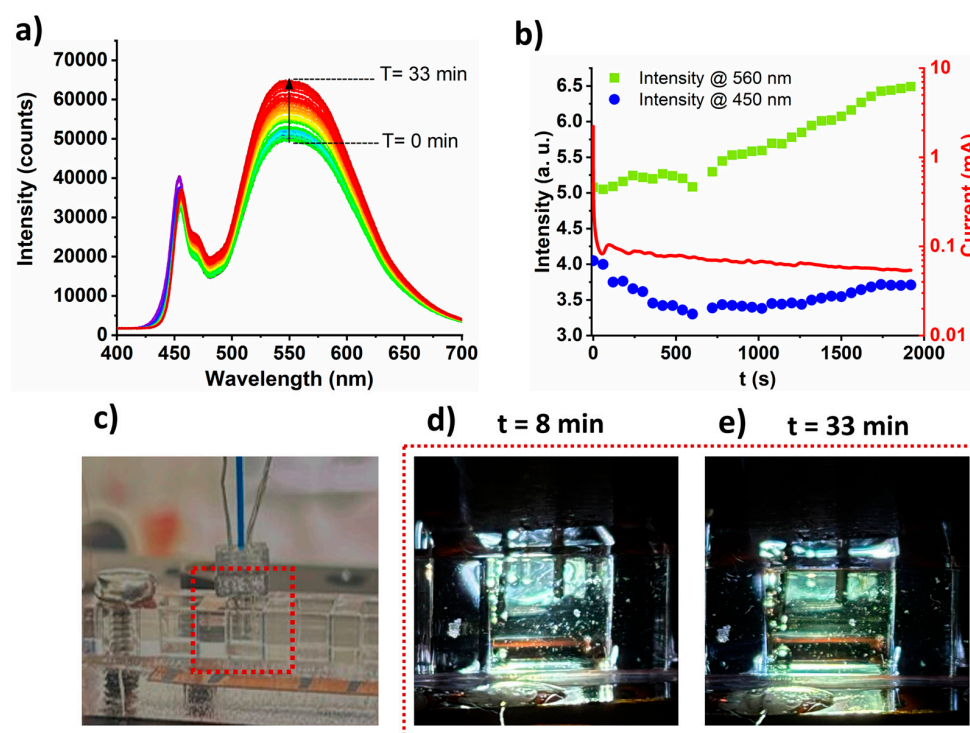


Figure 12. Optical detection of 20 mM ferrocyanide oxidation under continuous chronoamperometry: (a) spectra from 0 min to 33 min; (b) real-time intensity change through time and continuous current measured. Photographs of: (c) the setup wells; (d) naked-eye ferricyanide color after 8 min; (e) after 33 min.

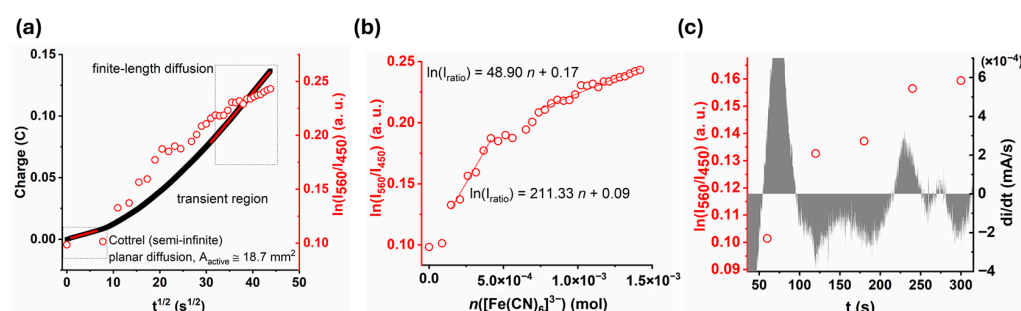


Figure 13. Chronoamperometric oxidation of 20 mM ferrocyanide: (a) mass transport regimes; (b) ferricyanide produced; (c) optical measurements tracking electrochemical events.

Chronoamperometric oxidation of ferrocyanide at the LIG WE at a diffusion-limited regime reveals three distinct mass transport regimes when the accumulated charge is analyzed as a function of $t^{1/2}$, reflecting successive changes in diffusion geometry and boundary conditions (Figure 13a). At short times ($t \lesssim 100$ s), the charge Q increases linearly with $t^{1/2}$, consistent with classical Cottrell behavior under semi-infinite diffusion. In this regime, the diffusion layer remains small compared to the interelectrode spacing, concentration gradients are localized near the WE, and the CE does not influence mass transport. The response therefore reflects diffusion-limited oxidation from an effectively infinite reservoir. The calculated active electrode area A of 0.187 cm² is consistent with the geometrical area of the LIG electrode (~ 0.16 cm²), obtainable via the integral form of the Cottrell relation.

$$Q(t) = \frac{2nAFC^*(Dt)^{\frac{1}{2}}}{\pi^{\frac{1}{2}}} + Q_i, \quad (4)$$

where Q_i is a constant accounting for the rapid (initial) capacitive charging and faradaic activity of pre-adsorbed ferrocyanide, D is the ferrocyanide diffusion coefficient in KCl

aqueous solution ($\sim 7.0 \times 10^{-6} \text{ cm}^2/\text{s}$) [47] and C^* is the bulk concentration of ferrocyanide (in mol/cm^3).

At intermediate times ($100 \text{ s} \lesssim t \lesssim 1000 \text{ s}$), a pronounced deviation from $t^{1/2}$ linearity is observed. The diffusion layer thickness $(\pi Dt)^{1/2}$ at 100 s is about $\frac{1}{4}$ of the interelectrode spacing. This non-linear transient corresponds to a crossover regime in which the diffusion layer expands sufficiently to interact with the evolving concentration field of the CE. During this interval, the electrolyte between electrodes becomes progressively concentration-polarized, and the transport problem can no longer be described by either semi-infinite or fully developed finite-length diffusion.

At longer times ($t \gtrsim 1000 \text{ s}$), the characteristic length $(\pi Dt)^{1/2}$ becomes like the cell dimensions and interelectrode spacing ($\sim 2 \text{ mm}$). Linearity in charge Q versus $t^{1/2}$ is recovered with a steeper slope, marking the establishment of a finite-gap, diffusion-limited regime in which evolving concentration gradients span the entire electrolyte volume. In this state, electrolysis involves the bulk electrolyte between the electrodes. Although this late-time regime exhibits a steeper $t^{1/2}$ dependence of charge, the corresponding currents are smaller and continue to decay with time, as expected for diffusion-controlled transport ($i \propto t^{-1/2}$). The increased slope thus reflects a change in diffusion geometry and boundary conditions rather than an enhancement of mass transport rate. Overall, the observed sequence of regimes provides a consistent description of the evolution from semi-infinite diffusion to concentration-polarized, finite-gap transport in an unstirred cell.

The existence of the above regimes is corroborated by the optical measurements and I_{560}/I_{420} ratio, as seen in Figure 13b,c. At the early stages, the ferricyanide concentration is highly non-uniform and accumulates near the LIG electrode. Optical sampling volume (mainly in the electrolyte bulk) differs from the electrochemical reaction zone (electrode surface). As natural convection effects appear and the diffusion layer starts overlapping the optical sampling volume (transient region in Figure 13a), the changes in the $\ln(I_{560}/I_{420})$ ratio become much larger and show an approximately linear trend with Q (with convection-induced fluctuations) and, thus, the produced quantity of ferricyanide (Figure 13a,b). Finally, in the finite-gap diffusion regime the $\ln(I_{560}/I_{420})$ ratio also tracks the charge and thus the produced ferricyanide linearly, yet at a smaller slope, reflecting the lower rate of ferricyanide production.

The data shown in Figure 13c accurately highlight the ability of the optical measurements to closely track specific electrochemical events in the prototype. The derivative of current—which is proportional to production rate $d[\text{Ferricyanide}]/dt$ —aligns with the rate of change in the optical ratio. A lag is observed, especially at shorter times, given that the current responds instantly to the electrode surface, whereas the fiber probe detects the bulk solution concentration, so diffusion delays the optical response, especially in an unstirred cell. In short, positive spikes or steps in $i(t)$ (positive derivative) coincide with large rises in I_{560}/I_{420} , and negative ones tend to flatten out the ratio, confirming that the ratio tracks the electrochemical production of ferricyanide closely.

In summary, a maskless alternative to conventional soft lithography is demonstrated by using laser-based fabrication techniques—and therefore reducing fabrication complexity and infrastructure requirements [37]. Furthermore, the present setup relies exclusively on disposable LIG as the sensing transducer and a simple optical fiber for fluorescence detection, avoiding bulky optical components [37]. Briefly, this study improves the state of the art in the field by combining fabrication simplicity, reusability, minimal optical instrumentation, and dual-sensing capability. These characteristics enhance its potential applicability not only in high-income settings but also in resource-limited environments, with the long-term prospect of contributing to decentralized diagnostic strategies, and is particularly well suited for POC applications.

4. Conclusions

This work demonstrates a laser-induced graphene (LIG) platform that integrates electrochemical and optical transduction within a single in-chip architecture, providing a robust proof of concept for future point-of-care (POC) biosensing applications. By varying the number of laser passages (LIG1, LIG2 and LIG3), clear correlations were established between morphology, structural order, and sensing performance. Increasing the number of laser passages progressively increases macroporosity and reduces oxidation degree—as expected given the reductive nature of laser irradiation—with LIG3 exhibiting the most irregular and porous morphology.

These structural differences translate directly into complementary electrochemical and optical responses. Electrochemically, LIG1 and LIG2 exhibit fast and reversible ferro/ferricyanide redox behavior, while LIG3 shows degraded reversibility and slower kinetics. Differential pulse voltammetry identifies LIG1 as the most reliable electrochemical transducer, combining higher currents, lower LOD and higher sensitivity. In contrast, optical sensing could benefit from increased laser exposure: all LIG electrodes enable stable, concentration-dependent fluorescence detection of DCM dye in solution; however, LIG3 allows lower LOD and higher sensitivity. These results highlight the tunability of LIG through laser processing, allowing optimization toward electrochemical robustness or optical sensitivity depending on the intended sensing mode.

The full capabilities of the platform are demonstrated through simultaneous optical and electrochemical monitoring of ferrocyanide bulk electrolysis. Chronoamperometric oxidation of ferrocyanide to ferricyanide produces a clear and quantifiable colorimetric response that is tracked in real time alongside the electrochemical current. Analysis of the accumulated charge reveals successive semi-infinite, transitional and finite-gap diffusion regimes, fully consistent with classical mass transport theory in an unstirred cell. Importantly, the optical signal—expressed through a ferricyanide absorption-dependent ratio of intensities at two wavelengths—tracks the electrochemically generated ferricyanide quantitatively. This direct correlation confirms that the optical channel faithfully reports electrochemical events occurring at the electrode.

Beyond validating the physical consistency of the device, this dual ferrocyanide experiment clearly illustrates the opportunities opened by the proposed architecture for biosensing. Simultaneous electrochemical and optical interrogation provides intrinsic cross-validation, reducing false positives while preserving sensitivity—an essential requirement for POC diagnostics. It also opens space for the study of new detection mechanisms based on the electrochemical/optical interplay. Combined with its low-cost laser-based fabrication and materials, modular design, reuse of all components except the LIG electrode, and compatibility with biorecognition functionalization, this platform emerges as a practical and scalable foundation for future automated, multiplexed and reliable dual-mode biosensors. In this context, further systematic experiments with extended experimental data analysis concerning fabrication yield, alignment tolerance, reuse cycles, and inter-device variation must be conducted for lab-to-market implementation and real-world applications.

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Abbreviations

The following abbreviations are used in this manuscript:

DCM	4-Dicyanomethylene-2-methyl-6-(4-dimethylaminoethyl)-4H-phyran
I_A/I_C	Anodic-to-cathodic peak ratio
CE	Counter electrode
CV	Cyclic voltammetry
DPV	Differential pulse voltammetry
GO	Graphene oxide
GQD	Graphene quantum dot
LIG	Laser-induced graphene
LOD	Limit of detection
LOQ	Limit of quantitation
i_p	Peak current
ΔE_p	Peak potential separation
PBS	Phosphate-buffered saline
POC	Point-of-care
PLA	Polylactic acid
PI	Polyimide
E_{pulse}	Pulse height
t_{pulse}	Pulse width
RE	Reference electrode
RT	Room temperature
ν	Scan rate
SEM	Scanning electron microscopy
WE	Working electrode

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