# MICRO (Multi-material, Integrated, Compact, Ready-to-plug and One-step produced): A user-friendly mobile 3D-printed electrochemical cell for on-site and forensic analysis

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## RESUMO

Neste trabalho propõe-se uma MICRO célula eletroquímica impressa em 3D inovadora, miniaturizada (dimensões < 2 cm), integrada (eletrodos de CB-PLA e corpo da célula em PETG), de baixo custo (< R$ 0,50) e prática (simples conexão sem fio com potenciostatos compactos e smartphones). Para melhorar o desempenho, a célula foi ativada em uma máquina de gravação a laser azul, no qual três parâmetros foram combinados em um planejamento fatorial ($2^3$): distância (8 a 11 mm), potência (2,75 a 5,5 W) e velocidade (600 a 1000 mm $min^{-1}$). Empregando voltametria cíclica na sonda ferricianeto, observou-se que as maiores correntes e melhor reversibilidade ($\Delta Ep$ = 117 mV e a $Ipa/Ipc$ ≈ 1,00) foram obtidas em 8 mm, 2,75 W e 600 mm $min^{-1}$. Nesta condição, procaína e lidocaína (anestésicos e adulterantes de cocaína) apresentram processos anódicos em + 0,7 V ($H_2SO_4$ 0,1 M) e + 0,8 V ($H_2SO_4$ 0,1 M) respectivamente, o que demonstra a potencialidade do dispositivo para a detecção de fármacos e drogas em campo.

*Palavras-chave: Fused deposition modeling (FDM), Carbon Black-Polylactic Acid (CB-PLA), Electrochemical sensor, Three-electrode integrated cell, Forensic voltammetry.*

## Introduction

Miniaturized analytical devices are encouraged by green analytical chemistry practices due to the miniaturized size, reduction of generated waste (microliter order), energy saving, and in situ measurements (1). In electroanalysis, miniaturized three-electrode integrated electrochemical cells such as disposable screen-printed electrodes (SPE) have been marketed and better accepted by unexperienced analysts. These portable devices have been used for environmental, clinical, food, and forensic analysis However, the cost of most of these commercial SPE is relatively high, especially in countries outside US and Europe (e.g. ≈ US$ 5.00 per device in Brazil). 3D printing (additive manufacturing) by fused deposition modeling (FDM) has emerged as a transformative technology in electroanalytical chemistry, enabling the rapid, low-cost, and reproducible mass fabrication of portable electroanalytical devices. For the fabrication of 3D-printed electrodes, Protopasta (Protoplant, USA) is the most widely commercial conductive filament. However, due to its low conductive content (≈ 20% of carbon black (CB) entrapped in a polylactic acid (PLA) matrix), post-printing activation protocol is required to improve the electrode performance. Among several treatments, laser irradiation (2) stands out due to its rapid, eco-friendly, and automated nature. In this context, a Miniaturized, Integrated, Cheap, Ready-to-plug, and One-step produced 3D-printed electrochemical cell (MICRO-$EC^{3D}$) is proposed as a novel low-cost tool for convenient analysis and forensic applications.

## Experimental

*Design and fabrication of MICRO-$EC^{3D}$*

The microdevice was fabricated in an automated process using a low-cost dual-extruder 3D printer (Sovol SV04, IDEX). This machine prints the body containing an embedded solution reservoir at nozzle 1 using PETG) and three electrodes/tracks at nozzle using CB-PLA). The image of the device is shown in Figure 1. The low-cost device (US$ < 0.10) is quickly printed (≈ 8 min), uses low sample solutions in measurements (volume from 50 to 300 µL) and is very practical (direct plug to user-friendly wireless USB-based connectors).

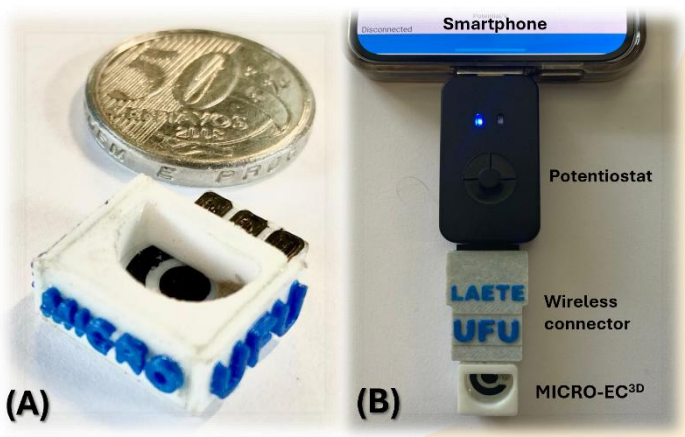


**Figure 1.** Image of (A) MICRO-$EC^{3D}$ and (B) Fully mobile system: Device connected to a handheld potentiostat through a developed wireless connector and controlled by smartphone.

*Activation of MICRO-EC$^{3D}$*

Activation was performed using a blue-laser engraving machine (P = 5.5 W, λ = 450 nm), where three key parameters were varied and combined through a factorial design ($2^3$): laser-device distance (LD) from 8 (-1) to 11 mm (+1), laser power (LP) from 50% (-1) to 100% (+1), and scan speed (SS) from 600 (-1) to 1000 mm min$^{-1}$ (+1).

## Results and Discussion

The electrochemical response of each laser activated electrode was assessed via cyclic voltammetry (CV) in 1 mM ferricyanide and 0.5 M KCl. The CV parameters (peak-to-peak separation (*ΔEp*), anodic peak current ($I_{pa}$), cathodic peak current ($I_{pc}$) and ratio $I_{pa}$ / $I_{pc}$) obtained from these experiments are present in Table 1.

**Table 1.** Parameters obtained by CV in 1.00 mM ferricyanide

| Exp. | SS | LP | LD | ΔEp (V) | $I_{pa}$ (μA) | $I_{pc}$ (μA) | $I_{pa}$ / $I_{pc}$ |
|---|---|---|---|---|---|---|---|
| 1 | -1 | -1 | -1 | 0.117 | 11.89 | 11.86 | 1.00 |
| 2 | -1 | -1 | 1 | 0.141 | 8.05 | 8.31 | 0.97 |
| 3 | -1 | 1 | 1 | 0.295 | 1.34 | 1.3 | 1.03 |
| 4 | 1 | -1 | -1 | 1.199 | 1.89 | 7.09 | 0.27 |
| 5 | 1 | 1 | -1 | 1.374 | 1.11 | 4.72 | 0.23 |
| 6 | -1 | 1 | -1 | 0.359 | 5.15 | 6.46 | 0.00 |
| 7 | 1 | -1 | 1 | 1.286 | 2.16 | 6.77 | 0.32 |
| 8 | 1 | 1 | 1 | - | - | - | - |
| 9 | 0 | 0 | 0 | 1.404 | 2.72 | 4.8 | 0.57 |

As shown, the best reversibility of ferricyanide redox probe (lowest ΔEp = 117 mV and $i_{pa}$ / $i_{pc}$ = 1) and peak currents ($i_{pa}$ and $i_{pc}$) were achieved using the lowest levels of factorial design study. In real variables, these values correspond to laser-device distance of 8 mm, 50 % of laser power (2.75 W) and speed scan of 600 mm min$^{-1}$. Using this condition, the required time for the activation of one device is only 5 min. Using this activation procedure, the analytical application of MICRO-EC$^{3D}$ was evaluated for the detection of procaine and lidocaine. These substances are anesthetics present in pharmaceutical formulations and common cocaine adulterants. Figure 2 shows the cyclic voltammograms in the presence of procaine in 0.1 M $H_2SO_4$ and lidocaine in 0.1 M NaOH.

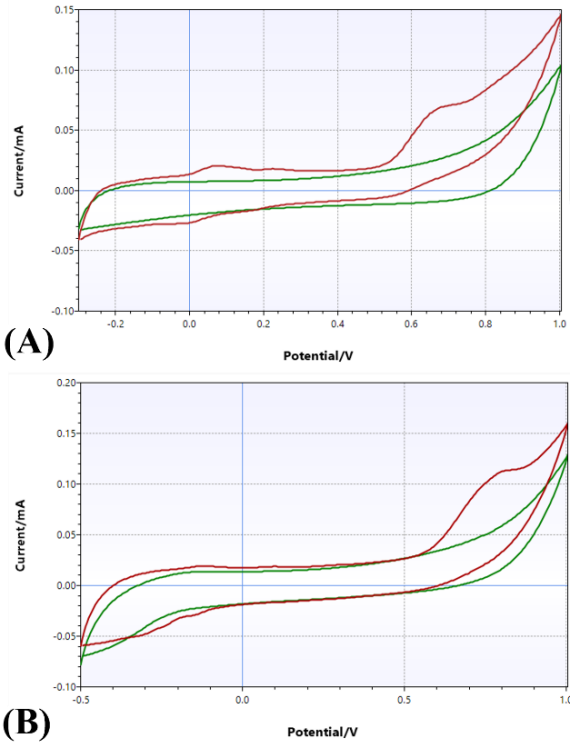


**Figure 2.** Cyclic voltammograms in the absence (green line) and presence (red line) of (A) 1.00 mM procaine in 0.1 M $H_2SO_4$ and (B) 1.00 mM lidocaine in 0.1 M NaOH. Scan rate = 50 mV s$^{-1}$.

As shown, procaine exhibits a well-defined anodic peak at + 0.7 V and a redox pair close to 0.0 V in acid solution whereas lidocaine a single irreversible anodic peak at + 0.8 V in basic solution. These results show the potential of this blue laser activated MICRO-EC$^{3D}$ for the screening and determination of procaine in pharmaceutical and drug seized samples.

## Conclusions

In this study, a novel fully 3D-printed electrochemical cell was proposed as a convenient tool for on-site analysis and drug screening. The device was named MICRO-EC$^{3D}$ to highlight its small size and features: Multi-material (PETG and CB-PLA), Integrated (body with embedded electrodes and solution reservoir), Compact (1.9 x 1.4 x 0.7 cm), Ready-to-plug (user-friendly USB connectors) and One-step produced (on a low-cost dual extruder 3D-printer). This microdevice is portable and easy to use (simple wireless interface with pocket potentiostats and smartphones). After a rapid, low-toxicity, and automated blue-laser activation protocol (2.75 W at 600 mm min$^{-1}$), good response for procaine and lidocaine detection was obtained. These results demonstrate the potential of electroanalytical microdevice for use in several on-site and routine forensic analyses, as well as for enhancing the use of electrochemical techniques, particularly among inexperienced users.

## Acknowledgements

The authors gratefully acknowledge financial support from CNPq (Grant numbers: 404210/2021-0 and 404465/2023-4) and FAPEMIG (Grant numbers: RED 00120-23 and APQ-03000-18).

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