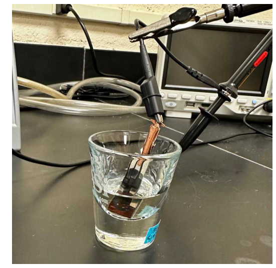
Jun 20, 2024

Growth of Chitosan nano brushes using FMED as electropolymerization method



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We use this protocol and it's working

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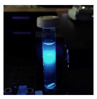
Abstract

This protocol describes the electropolymerization of chitosan nanobrushes in the working electrode of a Laser-induced graphene (LIG) sensor. The process requires approximately **36 minutes** (including baseline analysis). The electropolymerization times are not included in this protocol. A 2-channel function / arbitrary waveform generator (SINGLENT SDG 1032X) was used throughout this protocol.

This protocol uses the Microwave Synthesis of the low molecular weight deacylated chitosan protocol as an electropolymerization solution in the working electrode of an LIG. **See the protocol below.**

Protocol



NAME

Microwave synthesis of low molecular weigth deacylated chitosan

CREATED BY

Maria J Torres

PREVIEW

Guidelines

This protocol describes the electropolymerization of chitosan nanobrushes into the working electrode of a Laser-Induced Graphene (LIG) sensor. The complete process requires approximately **36 minutes** (see diagram for an overview of steps, **Fig. 4**).

A link to the Monowave Resource Manual with additional basic information on the instrument is here (**link**).

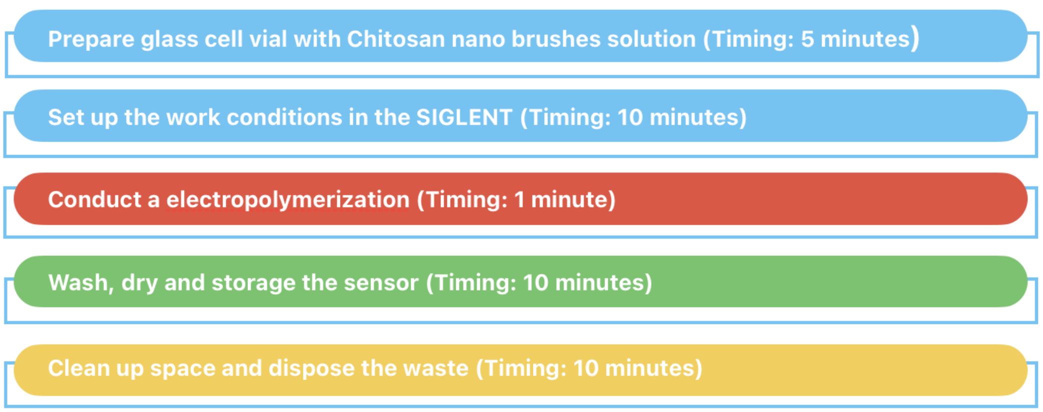


Figure 4. Process flow for the electropolymerization of chitosan nano brushes into the working electrode of a LIG sensor. The steps are separated into sections: preparation (blue), electropolymerization (red), stabilization and storage (green), and cleaning up the working area (yellow).

Materials

MATERIALS

- SIGLENT SDG1032X (**link is here**)
- Glass cell vial for voltametry (**link is here**)
- Parafilm (**link is here**)
- Metal alligator clips (**link is here**)
- Probe for Digital Oscilloscope Clip Probes (**link is here**)
- KIMTECH wipes (**link is here**)
- Paper towels
- Laboratory plastic wash bottle (**link is here**)

HARDWARE

- SIGLENT SDG1032X (**link is here**)

Safety warnings

SAFETY

General

- Lab coat, gloves, and closed-toed shoes are mandatory
- Safety issues specific to SINGLENT SDG1032X
- The maximum pressure for the glass vial is 20 bar
- The maximum volume in the glass vial at any time is 6 mL

Eye protection

- Goggles or eye protection is required when handling acidic solutions outside of a chemical hood (i.e., transport in lab).

Skin

- Immediately rinse under water and wash with soap for at least 5 min

Disposal

- The residual chitosan should be placed in a container previously marked with the name and composition of the solution.

ADA COMPLIANCE

The following guidance is summarized from Perry and Baum [1] where relevant to this protocol.

1) General building codes for laboratory

- Minimum 2 Exits for labs ≥500 sf (150 sm) net area.
- Minimum 2 Exits for labs using chemical fume hoods or glove box
- Minimum 2 Exits for labs using flammable and combustible: liquids, gases, cryogenics, dusts and solids.
- Minimum 2 Exits for labs using oxidizers, unstable reactives, water reactives, organic peroxides, highly toxics, corrosives.

2) Egress for wheelchair 360° Turn is 1.5 m (5 ft) clearance. Wheelchair clearance must be provided for:

- Both sides of Exit and Entry doors to
- Emergency Eyewash & Safety Shower
- In front of wall benches, sinks, equipment
- In front of chemical fume hoods
- At chalk/marker board
- Between benches
- Aisles that lead to Primary Exits, back to front
- Aisles that allow passage side to side in lab

3) Standard accommodations for use of chemical hood or other exhaust air containment systems

- knee space obstructions
- adjustable work surface height
- accessible receptacles and alarm control

Common equipment

- Where visual inspection is utilized, alternative technologies should be listed as optional (colorimeters, spectro-radiometers, etc.)

References

1.Perry, J. & Baum, J. Assessing the Laboratory Environment. in *Accessibility in the Laboratory* vol. 1272 3–25 (American Chemical Society, 2018).

Before start

- Be sure to wear appropriate safety PPE throughout (lab coat, gloves, eyewear).
- Electronic or physical lab notebook may be used throughout
- See experimental plan guide for tips on planning your work

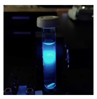
SECTION 1) Preparation

15m

1 **Prepare a glass cell vial with lower molecular weight deacylated chitosan solution.** 5m

Previous microwave-assisted synthesis of lower molecular weight deacylated chitosan solution prepared is ***required*** *following the next protocol* (**Link here**)*:*

Protocol



NAME

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PREVIEW

Note

Storage the solution at 4 °C for at least 24:00:00
This will ensure the density of the solution is accurate for electropolymerization.

2 ***Set up*** **the work conditions in the** ***SINGLET SDG1032X.*** 10m

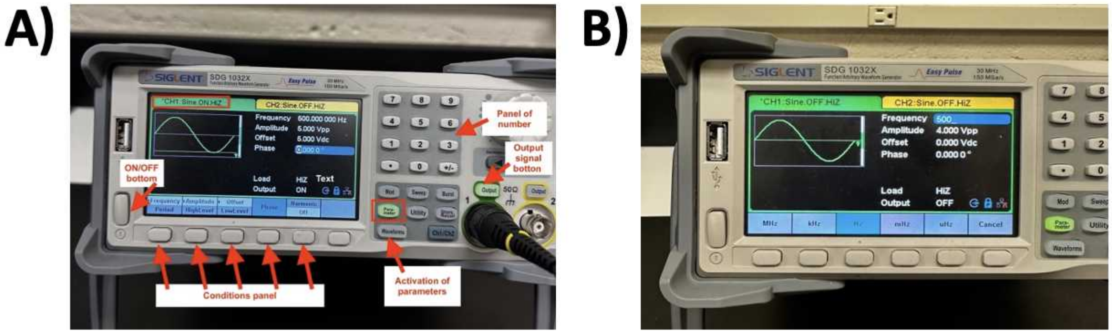

Figure 1. A) SIGLENT wave generator panel of options, including ON/OFF button, conditions panel **(left bottom)**, the panel of numbers **(upper right)**, output signal button **(bottom right)**, and activation of parameters **(middle bottom)**. **B)** Selecting the parameter conditions measurement units.

- Turn on the SIGLET wave generator by pressing the "ON/OFF" button once and wait until the equipment turns on completely.

Note

Set up the conditions for the electropolymerization as follows:
Frequency 500 Hz
Amplitude 5.0 Vpp
Offset 5.0 Vdc
Phase 0.0 grades
Harmonic OFF
Waveform SINE

- Press the waveform button (See **Fig. 1A**) to select the Sine waveform for the conditions using the same procedure mentioned in this note.

Note

NOTE 1: To set up the conditions in each category, press the condition button once **(Fig. 1A, bottom left)**, introduce the number for the condition in the number panel **(Fig. 1A, upper right)**, select the measurement unit, press in the option that you wanted and change to the next to the right button **(Fig. 1B, center bottom)** to make changes in the other conditions.

NOTE 2: It is recommended to ensure the parameters button is activated (to know if it is activated, it must be green).

SECTION 2) Electropolymerization using FMED method

1m

3 **Conduct an electropolymerization**

- Add in a glass cell vial the 8000 µL of the previous lower molecular weight deacylated chitosan solution.

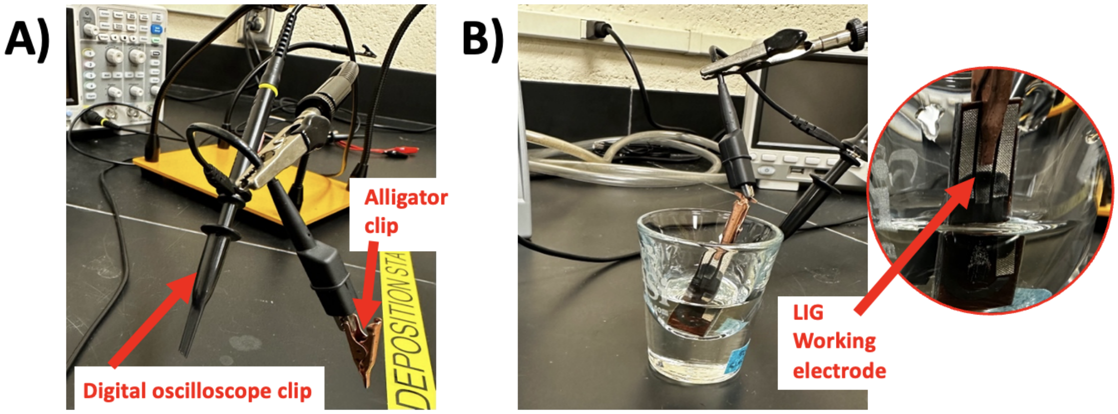

Figure 2. A) Set up the clips for the wave signal generation. **B)** Connection to the Laser-Induced Graphene (LIG) sensor in the working electrode for the FMED of Chitosan nanobrushes.

- Introduce the three-electrode LIG sensor in the digital oscilloscope clip in the lower molecular weight deacylated chitosan solution **(Fig. 2A).**

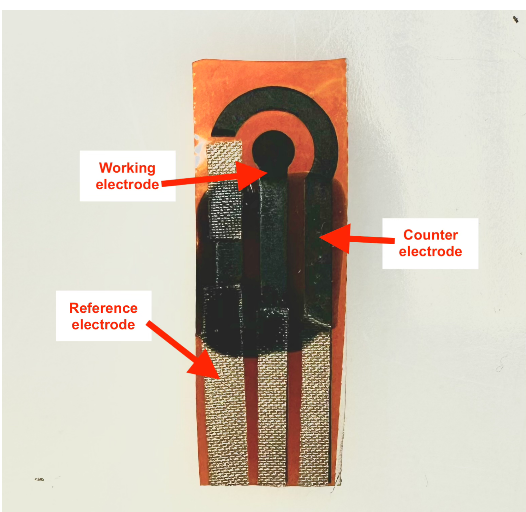

Figure 3. Laser-Induced Graphene (LIG) sensor, a three-electrode system (reference, working and counter electrode)

- Connect the alligator clip to the working electrode of the Laser-Induced Graphene (LIG) sensor. Ensure it is connected correctly in the middle of the metal tape of the working electrode.

- Introduce the LIG sensor in the lower molecular weight deacylated chitosan solution.

Note

Introduce the bottom part of the LIG sensor, ensuring that the working electrode (black circle, **Fig. 3**) is completely submerged in the solution (**Fig. 2B**).

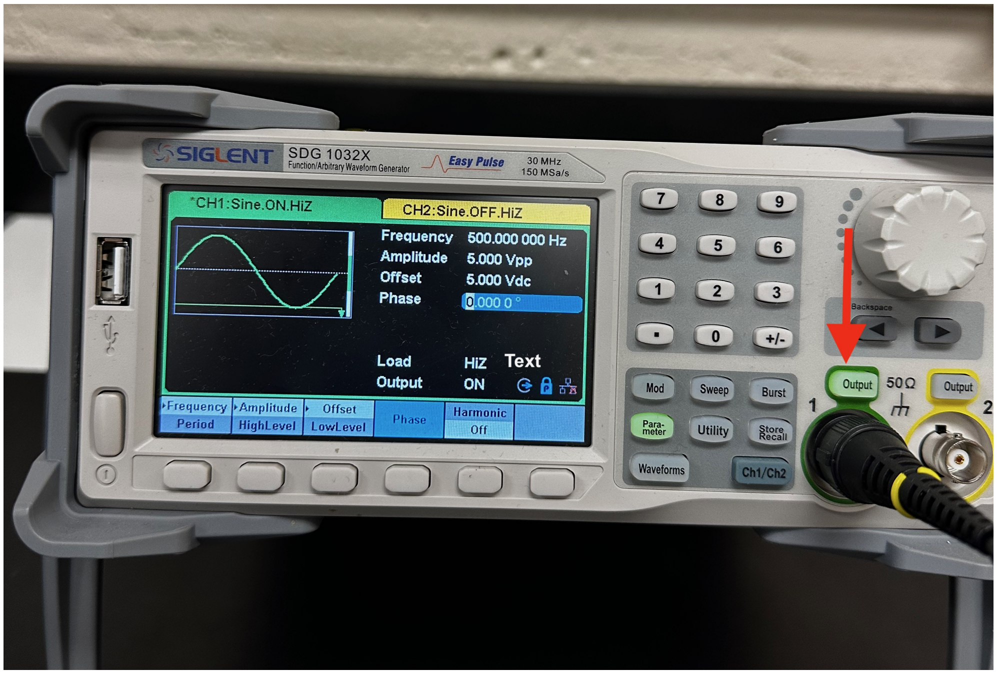

Figure 4. Output signal button. Green light indicates that itis active and functioning.

- Set up the time with a timer for the FMED electropolymerization of lower molecular weight deacylated chitosan in the working electrode of the LIG sensor. Start the time and, **at the same time**, press the Output button (**Fig. 4**). Be sure that the output button is ON (A green light indicates that it is already working).

Note

Critical step

Stop the timer when the time is already complete. ***At the same time***, press the "output" button to stop the wave generator and the FMED electropolymerization.

SECTION 4) Stabilization and storage 10m

4 **Wash, dry and storage the sensor** 10m

- Clean the LIG working electrode with DI/nano-pure water and dry the water carefully without touching the sensor electrodes. Dry from the sides with a kimitech wipe.
- Let it dry for 00:05:00 m in a paper towel and then storage the sensor in the fridge at 4 °C

SECTION 5) Clean up 10m

5 **Clean up space and dispose of waste** 10m

- Turn off the SIGLENT Wave generator and unplug it from the power outlet.
- Dispose of used chemicals according to the lab safety plan. Chitosan has a dedicated chemical disposal container.

Note

Chitosan has a dedicated chemical disposal container in the waste area.

- Wash all glass containers used in the experiment and let it dry.