ChatGPT

Materials and Reagents

- **Chemicals:** Silver nitrate ($AgNO_3$), poly(vinylpyrrolidone) (PVP, MW ~55,000), copper(II) chloride dihydrate ($CuCl_2·2H_2O$), ethylene glycol (EG), ethanol (EtOH), acetone, polyvinyl butyral (PVB) resin (e.g. Butvar/Mowital), deionized (DI) water, NaCl (for saline).
- **Substrates:** Commercial melamine sponge (e.g. "magic eraser" type), electrode holders (short polyethylene or PVC tubes), silver wire or foil.
- **Equipment:** Oil bath or hotplate (with temperature control ~150 °C), magnetic stirrer, round-bottom flask or vial (glass), centrifuge, vacuum pump or desiccator, oven (50–60 °C), pipettes, tweezers, safety gear (gloves, goggles).

1. Synthesis of Silver Nanowires (AgNWs)

Following a modified polyol method [1] [2], AgNWs are grown by reducing $AgNO_3$ in hot ethylene glycol (EG) with PVP as capping agent. $CuCl_2$ seeds promote 1D growth. Carefully control reagent concentrations and timing for reproducibility.

1. **Prepare precursor solutions:**
2. **PVP solution:** Dissolve PVP in EG to make 0.147 M (monomer units) solution. For example, dissolve ~1.6 g PVP (MW 55,000, polymer) in 10 mL EG (stir until clear).
3. **$AgNO_3$ solution:** Dissolve 0.17 g $AgNO_3$ in 10 mL EG to make 0.094 M.
4. **$CuCl_2$ solution (4 M):** Dissolve 0.68 g $CuCl_2·2H_2O$ in 1 mL EG (4 M stock) – prepare fresh or keep small volume.
5. **Heat base solvent:** Place 25 mL of EG in a glass flask with stirring. Heat to ~150–155 °C (oil bath) and stir for 1 hour to equilibrate [3]. (Use fume hood; EG vapor is toxic.)
6. **Seed with $CuCl_2$:** After 1 h heating, inject 200 µL of the 4 M $CuCl_2$ solution into hot EG. Stir and maintain temperature. (This adds ~0.8 mmol $CuCl_2$.) Continue heating 15–20 min [3].
7. **Add PVP and $AgNO_3$:** Quickly inject 7 mL of the 0.147 M PVP solution, then 7 mL of the 0.094 M $AgNO_3$ solution into the flask [3]. (Rapid addition yields uniform nanowires.) Maintain stirring and 150 °C.
8. **Growth:** Allow the reaction to proceed ~1–1.5 hours. The solution will change color (yellow → orange → greenish, then cloudy gray) as AgNWs form [4]. (An opaque gray indicates long-wire formation [4].)
9. **Quench:** Turn off the heat and cool the flask by placing it in a room-temperature water bath. This stops growth.

10. **Purification:** Transfer the reaction mixture to centrifuge tubes. Add an equal volume of acetone or ethanol to precipitate polymer. Centrifuge (~3000 rpm, 5 min) and discard supernatant [5]. Repeat washing **3×**: resuspend pellet in ethanol, centrifuge, discard (removes excess PVP/EG) [5].

11. **Disperse AgNWs:** Finally, suspend the washed AgNW pellet in fresh ethanol (or isopropanol) at a known concentration (e.g. ~1–10 mg/mL). Sonication (short pulses) can help uniform dispersion. Store this AgNW ethanolic suspension in a closed vial at 4 °C or room temperature in the dark until use [5].

2. Preparation of PVB Binder Solution

Poly(vinyl butyral) (PVB) binds AgNWs to the sponge. PVB dissolves in ethanol [6]. Make a moderate solution (e.g. 5–15 wt%). For example, dissolve 0.5–1.5 g PVB in 10 mL ethanol by stirring (gentle heating to ~40–50 °C can speed dissolution). Allow to fully dissolve (stir for ≥30 min or overnight). Let cool before mixing. No specific citation; based on common solubility.

3. Infiltration Mixture Preparation

Mix the AgNW suspension with the PVB solution to create the infiltration “ink.” For example, mix equal volumes of 1–2 mg/mL AgNW ethanol dispersion and 5–10 wt% PVB/ethanol solution. Stir gently to homogenize. (The final PVB content might be ~2–5 wt%.) This mixture contains conductive AgNWs held in a polymer binder.

4. Sponge Preparation and Vacuum Infiltration

1. **Cut sponge to size:** Trim melamine sponge into electrode-sized pieces (e.g. 1 cm cubes or cylindrical plugs matching your holder). Record dimensions for reproducibility.
2. **Clean sponge:** Rinse sponge pieces in ethanol or ethanol/DI water to remove any contaminants. Squeeze out excess solvent and air-dry briefly.
3. **Vacuum infiltration:** Place a sponge piece in a small open container (glass vial or beaker). Pour the AgNW/PVB mixture to fully submerge the sponge.
4. Connect the container to a vacuum source (e.g. vacuum desiccator or pump). Apply vacuum (~10–100 mbar) for **5–10 minutes**, watching for vigorous bubbling. Vacuum removes air from pores, allowing the solution to penetrate fully.
5. Slowly release the vacuum (to avoid splashing) so that the solution remains in the sponge.
6. **Drain and dry:** Remove the sponge; let excess solution drip off. Gently press or roll between filter papers to remove surface liquid. Place the saturated sponge on a clean dish. Dry at room temperature (several hours or overnight) or in a 50–60 °C oven for 1–2 hours until completely dry (constant weight). This leaves an AgNW–PVB coating anchored in the sponge.

5. Electrode Assembly

1. **Insert into holder:** Place the dried AgNW–PVB sponge into the electrode body (a short PE or PVC tube, e.g. ~1 cm length) so that one face will contact the scalp. The sponge should fit snugly without gaps.

2. **Attach lead wire:** Press a small silver wire or foil into the sponge from the open end. Secure good contact by applying conductive silver epoxy or silver paint at the interface between wire and sponge. Alternatively, wrap fine wire around the sponge *while* wet or use a copper coin coated in Ag paint pressed onto it. Ensure the wire leads out through the tube. Allow any adhesive to cure fully.
3. **Seal edges (optional):** Seal the edges of sponge/tube interface with epoxy or silicone to prevent movement. Leave the contact face exposed.

6. (Optional) Saline Activation

To create a "semi-dry" electrode, soak the AgPMS sponge in physiological saline (0.9% NaCl) before use. Immerse the electrode tip in saline for a few minutes so the sponge absorbs it. Excess drip off. The retained electrolyte can improve skin contact and reduce impedance during EEG recordings [7].

7. Safety Notes

- **Chemical safety:** EG is high-boiling and toxic if ingested or absorbed – use in a fume hood and wear gloves/goggles. $AgNO_3$ is an oxidizer and can stain skin/clothing. $CuCl_2$ is toxic and irritant. PVB is flammable. Work in a ventilated area and keep flame sources away. Dispose of silver waste per regulations.
- **Heat precautions:** Oil baths at 150 °C can splatter – use stable setups and a lab jack. Do not seal the reaction flask (to avoid pressure buildup). Use a cap tilted for vapor escape.
- **Personal protection:** Wear lab coat, eye protection, and gloves at all times. Follow your institution's chemical hygiene protocols.

8. Reproducibility and Storage

- Use the same brand/thickness of melamine sponge for consistency. Document all volumes, masses, and times.
- For reproducibility, maintain constant stirring speed and temperature during synthesis. Record hotplate settings.
- Store unused AgNW–ethanol suspension in a sealed amber vial at 4 °C to slow oxidation [5]. Prepare infiltration mix fresh or shortly before use.
- Keep PVB solution sealed (glass bottle) to prevent ethanol evaporation.
- Finished electrodes: store dry in a desiccator or sealed container. If saline-soaked, wrap ends in plastic to retain moisture.

9. Characterization (Suggested)

After fabrication, verify structure and performance:
- **SEM imaging:** Cut a small piece of AgPMS and image by scanning electron microscopy to see AgNWs on the sponge skeleton. SEM confirms nanowire coverage and network morphology. (Lin et al. report a self-locked AgNW network on the sponge surface [8].)
- **X-ray Diffraction (XRD):** Powder or plate the dried AgNW/PVB on glass; XRD should show Ag metal peaks (e.g. (111), (200) at $2\theta \approx 38°$, 44°). This confirms crystalline Ag.
- **Conductivity:** Measure electrical conductivity of the impregnated sponge. For example, press the sponge between two electrodes or four-point probes and measure resistance. Lin et al. measured ~917 S/m for

AgPMS [9]. Ours should be on the order of 10^0–10^3 S/m (depending on loading).
- **Mechanical stability:** Compress the sponge (e.g. 10% strain) for thousands of cycles and monitor resistance. A robust AgNW network shows little change, as reported [10].
- **Electrochemical/EEG testing:** Perform impedance spectroscopy on a skin phantom or saline-soaked gel to check electrode impedance (<10 kΩ at 10 Hz is desirable [7]). Finally, record a test EEG or ECG from a volunteer to compare signal quality against a standard electrode.

By following these steps, students can reproducibly make AgPMS electrodes similar to Lin et al.'s AgNW–PVB–melamine sponge EEG electrodes [7] [9]. Ensure careful measurements and note any deviations. Quality controls (e.g. SEM, conductivity) help verify success. Good lab practices and documentation will lead to reliable and safe results.

References: Protocol steps and materials adapted from reported methods [11] [2] [7] [9] (Lin et al. 2019) on AgNW/PVB/melamine sponge electrodes.

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[1] [4] [11] **Application Note: Synthesis of Silver Nanowires**
https://www.sigmaaldrich.com/US/en/technical-documents/technical-article/materials-science-and-engineering/nanoparticle-and-microparticle-synthesis/application-note-synthesis-of-silver-nanowires?srsltid=AfmBOorisUimr4yl4kp_RhoNE9-nVc58NU62y5waJPtm8xpqKF_Qw4vY

[2] [3] [5] **Fabrication of Silver Nanowire/Polydimethylsiloxane Dry Electrodes by a Vacuum Filtration Method for Electrophysiological Signal Monitoring - PMC**
https://pmc.ncbi.nlm.nih.gov/articles/PMC7226850/

[6] **What is the best solvent/method to be use to prepare PVB (Polyvinyl butyral) solution? | ResearchGate**
https://www.researchgate.net/post/What-is-the-best-solvent-method-to-be-use-to-prepare-PVB-Polyvinyl-butyral-solution

[7] **State of the Art of Non-Invasive Electrode Materials for Brain–Computer Interface**
https://www.mdpi.com/2072-666X/12/12/1521

[8] **Highly Robust, Flexible, and Large-Scale 3D-Metallized Sponge for ...**
https://onlinelibrary.wiley.com/doi/10.1002/admt.201900761

[9] [10] **A Flexible, Robust, and Gel-Free Electroencephalogram Electrode for Noninvasive Brain-Computer Interfaces - PubMed**
https://pubmed.ncbi.nlm.nih.gov/31454250/