ELECTROCHEMICAL GRAPHENE EXFOLIATION FROM GRAPHITE

Producing graphene-rich carbon material from bulk graphite using electrochemical exfoliation, vacuum filtration, sonication, and purification — plus a lab SOP and training video that cut onboarding time in half.

Electrochemical Exfoliation	Graphite Processing	Vacuum Filtration	Sonication	Nanomaterial Synthesis	SOP Development

Multi- stage	3+	Graphite	1.5 days
Electrochemical exfoliation	Purification steps	Carbon feedstock	Onboarding time (from 3)

CONTEXT

Why graphene synthesis mattered to the lab

Kamkar Labs works across functional nanomaterials: aerogels, electromagnetic shielding materials, and conductive composites all depend on having a reliable supply of high-quality graphene. Sourcing it externally is expensive and introduces variability between batches. Producing it in-house through electrochemical exfoliation gave the lab a reproducible, low-cost alternative that could be tuned to the specific application.

As a first-year undergraduate with no prior co-op experience, I was asked to support this synthesis work and take ownership of running the electrochemical exfoliation process throughout my 8 months at the lab. The graphene I produced fed directly into two downstream projects: the cellulose aerogel composites (Brief 05) and magnetic field nanomaterial studies running in parallel in the lab.

Fig. 1 — Vacuum-assisted Buchner filtration setup: 9.5 CFM double-stage vacuum pump (left), filtration funnel with graphene suspension being poured in, and 1000 mL Erlenmeyer flask collecting the filtrate. This setup was used across multiple purification cycles to isolate the graphene-rich fraction from electrolyte residuals.

ELECTROLYTIC PRODUCTION

Graphite-to-graphene conversion via electrolysis

The electrochemical cell used high-purity graphite rods as the anode and a platinum cathode, submerged in a stirred ammonium sulfate electrolyte solution. Under applied DC voltage, sulfate ions intercalate between graphite layers at the anode, forcing the stacked planes apart and releasing graphene-rich carbon nanoflakes into solution through a redox-driven mechanism.

The cell was a pre-existing lab setup. My contribution was assembling the clip apparatus that suspended the electrodes over the electrolyte-filled beaker at the correct depth and spacing, setting up each run, monitoring solution behavior as exfoliation progressed, and managing the resulting suspension through downstream purification. The method runs at low temperature and requires no aggressive oxidants, making it practical and safe for routine lab-scale production.

Lab process video — electrolyte prep and electrode setup:
https://www.youtube.com/watch?v=nJ3CwtAv42M

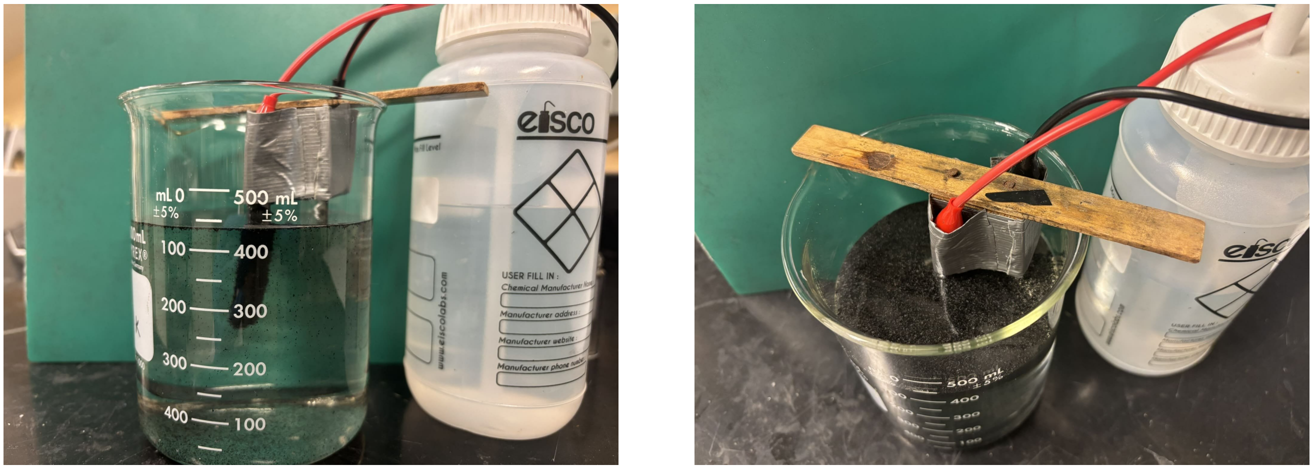

Fig. 2 — Left: graphite anode and cathode submerged in ammonium sulfate electrolyte during an early-stage exfoliation run, electrolyte still relatively clear. Right: dense graphene nanoflake suspension visible after extended exfoliation, solution turned deep black as material separated from the graphite anode.

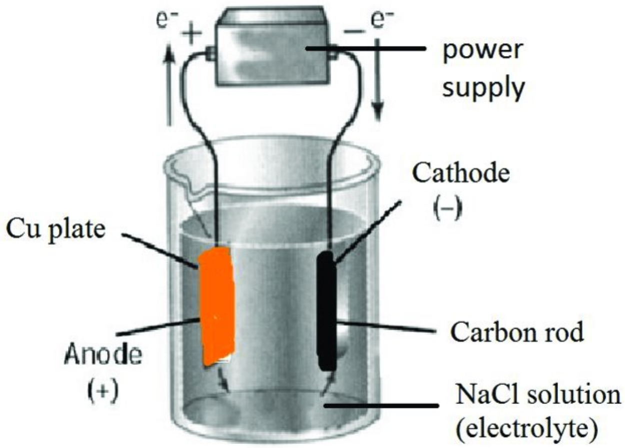

Fig. 3 — Electrochemical cell schematic showing power supply connections, copper plate anode (+), carbon rod cathode (-), and NaCl/electrolyte solution. The same cell topology was used in the lab with ammonium sulfate as electrolyte and graphite as the exfoliation anode.

PURIFICATION AND PROCESSING

Filtration, washing, and sonication

Raw exfoliation product is a suspension containing graphene nanoflakes alongside residual graphite particles, electrolyte salts, and other processing byproducts. Isolating the graphene-rich fraction required a multi-stage purification workflow run repeatedly across multiple batches.

> **Vacuum-assisted Buchner filtration**
>
> The suspension was filtered through nanoscale membrane filters using a vacuum-assisted Buchner filtration setup. Multiple filtration cycles progressively removed residuals and concentrated the graphene-rich fraction.

Washing

Between filtration cycles, the filter cake was washed to remove electrolyte salt residues that would otherwise contaminate the final product and interfere with downstream conductivity and composite performance.

Sonication in deionized water

After filtration the collected material was re-dispersed via sonication in deionized water. This step de-agglomerated stacked nanoflakes that had consolidated during filtration and ensured an even, stable dispersion, critical for consistent integration into aerogel formulations and other composite systems.

The final product was a stable, conductive graphene paste ready for integration into ongoing lab projects. Consistency across batches was confirmed visually and through dispersion stability over time.

Lab process video — filtration workflow:
https://www.youtube.com/shorts/cfUMsoMk1mo

SOP AND KNOWLEDGE TRANSFER

Systematizing the process for the lab

Partway through my time at Kamkar Labs, Prof. Kamkar asked me to document the exfoliation workflow formally. I wrote a step-by-step Standard Operating Procedure covering every stage from electrolyte preparation through final product storage. On my own initiative, I also produced an accompanying training video to make the SOP easier to follow for students who had never run the process before.

Within a single semester, five incoming students and two co-op hires were trained using these materials. Onboarding time dropped from 3 days to 1.5 days. The systematization also reduced the lab's reliance on external graphene procurement. The in-house process now produced consistent material that met the quality requirements of the aerogel and magnetic nanomaterial projects.

Writing a process document that someone else can follow without asking questions requires understanding every step well enough to anticipate where things go wrong. That was a useful forcing function for my own understanding of the chemistry.

Fig. 5 — Kamkar Lab group at the University of Waterloo. The SOP and training video were used to onboard 5 incoming students and 2 co-op hires, cutting training time from 3 days to 1.5 days within a single semester.

RETROSPECTIVE

What I'd do differently

01 **Characterize each batch with Raman spectroscopy before use**

Graphene quality was assessed visually and through dispersion stability, which gave no quantitative measure of how many layers the exfoliated flakes actually had. Running Raman spectroscopy on each batch, the standard technique for confirming graphene layer count and defect density, would have made it possible to correlate synthesis conditions with product quality and optimize the process more deliberately.

02 **Track electrolysis parameters systematically across runs**

Voltage, current, electrolyte concentration, and run time were set based on established protocol but not systematically logged against yield or product quality per run. Keeping a structured run log would have made it easier to identify which parameter combinations produced the best product and diagnose batch-to-batch variation.

03 **Include a yield calculation in the SOP**

The SOP covered the process steps but did not include a formal yield calculation: how much graphene-rich material was recovered per gram of starting graphite. Adding that metric would have made the SOP more useful for planning batch sizes and given incoming students a concrete benchmark to aim for.

REFERENCE

Technical specifications

Lab	Kamkar Labs (MMD), University of Waterloo
Supervisor	Prof. Milad Kamkar, Chemical Engineering, UWaterloo
Duration	8 months (2025), ran throughout the full lab placement

Process	Electrochemical exfoliation of graphite under DC voltage
Anode	High-purity graphite rod
Cathode	Platinum electrode
Electrolyte	Stirred ammonium sulfate solution
Mechanism	Sulfate intercalation at anode, redox-driven nanoflake release
Method advantages	Low-temperature, no aggressive oxidants, scalable at lab scale
Purification steps	Vacuum-assisted Buchner filtration, washing, sonication in DI water
Final product	Stable conductive graphene paste
Downstream use	Cellulose aerogel composites (Brief 05); magnetic nanomaterial studies
SOP	Written on request from Prof. Kamkar, step-by-step process documentation
Training video	Self-initiated, produced alongside SOP to support new lab members
Onboarding impact	7 people trained (5 students + 2 co-ops); onboarding 3 days to 1.5 days
Lab videos	youtube.com/watch?v=nJ3CwtAv42M | youtube.com/shorts/cfUMsoMk1mo