**Submission ID #: 68313**
**Scriptwriter Name: Poornima G**
**Project Page Link: https://review.jove.com/account/file-uploader?src=20835513**

# Title: Scalable Syntheses of Graphene Oxide and Reduced Graphene Oxide Using Cascade Design Oxidation and Highly Basic Reduction Reactions

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# Author Questionnaire

**1.** We have marked your project as author-provided footage, meaning you film the video yourself and provide JoVE with the footage to edit. JoVE will not send the videographer. Please confirm that this is correct.

√ Correct

**2. Microscopy**: Does your protocol require the use of a dissecting or stereomicroscope for performing a complex dissection, microinjection technique, or something similar? **No**

**3. Software:** Does the part of your protocol being filmed include step-by-step descriptions of software usage? **No**

**4. Proposed filming date:** To help JoVE process and publish your video in a timely manner, please indicate the proposed date that your group will film here: **06/20/2025**

When you are ready to submit your video files, please contact our Content Manager, Utkarsh Khare.

**Current Protocol Length**

Number of Steps: 13
Number of Shots: 30

# Introduction

1.1. **Chi Nhan Ha Thuc:** Our research scope is oxidation–reduction technologies for producing graphene oxide and reduced graphene oxide nanomaterials. We develop protocols for chemical oxidation–reduction reactions with optimized safety and efficiency.

1.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 2.1.2*

What technologies are currently used to advance research in your field?

1.2. **Thi Bang Tam Dao:** I think new technologies that improve safety and efficiency in Hummers oxidation reactions and alkaline reduction reactions are important to the production of graphene oxide and reduced graphene oxide.

1.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 2.3.1*

What are the current experimental challenges?

1.3. **Trung Do Nguyen:** Scale-up of graphite oxidation reaction requires a safe and efficient protocol. Besides, cost effectiveness, eco-friendliness and graphene restacking are challenges in chemical reduction reactions.

1.3.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 2.5.1*

What significant findings have you established in your field?

1.4. **Hon Nhien Le:** We presented cascade-design oxidation reaction harnessing exothermic energies for self-heating reactions, saving chemicals, energy and time. Reduction reaction using basic ammonia is simple and effective for non-stacked reduced graphene oxide.

1.4.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 4.1.1*

What research questions will your laboratory focus on in the future?

1.5. **Van Hieu Le:** Our laboratories will develop the protocols of oxidation – reduction reactions for scale-up productions and practical applications of graphene-based materials. Especially, as-synthesized graphene is useful for water purification, photocatalysts, energy storage and conversion.

1.5.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 4.2.1*

# Protocol

**2. Scalable Production of Graphite Oxide Using Cascade Design Oxidation Reaction**

**Demonstrator: Hon Nhien Le, Thi Bich Duyen Luu, Lam Nhu Pham**

NOTE: Author revised timestamps included by the scriptwriter. Use relevant part from author provided timestamps

2.1. To begin, add 5 grams of graphite powder to a 50-milliliter glass beaker and add 50 milliliters of 97 percent sulfuric acid to the beaker **[1]**. Stir the three suspensions of graphite and sulfuric acid magnetically at ambient conditions for at least 1 hour **[2]**.

2.1.1. 1.1.1 Prepare 50 mL graphite H2SO4.MOV Timestamps: 00:30 – 00:45.

2.1.2. 1.1.2 Stir three graphite H2SO4 suspensions.MOV Timestamps: 01:30 – 01:45.

2.2. In a 250-milliliter glass Erlenmeyer flask, add 100 milliliters of 97 percent sulfuric acid **[1]**. Slowly add 10 grams of potassium permanganate to the sulfuric acid solution under magnetic stirring to dissolve and prepare about 100 milliliters of manganese and sulfuric acid solution **[2]**.

2.2.1. 1.2.1. Add 50 mL H2SO4.MOV Timestamps: 00:05 – 00:20 .

2.2.2. 1.2.1. Add 10 g KMnO4.MOV Timestamps: 00:00 – 00:20 and 1.2.1. Stir three Mn VII solutions.MOV Timestamps: 00:40 – 00:55.

2.3. Slowly pour one previously prepared graphite and sulfuric acid suspension into the manganese and sulfuric acid solution under magnetic stirring in the 250 milliliter glass Erlenmeyer flask **[1]**. After 9 minutes in a water bath, continue stirring the reaction mixture at room temperature. Allow the reaction temperature to peak between 48 and 52 degrees Celsius and gradually return to room temperature **[2]**. Stir all three mixtures of graphite, manganese, and sulfuric acid magnetically at room temperature **[3]**.

2.3.1. 1.3.2. Conduct the first cascade step.MOV Timestamps: 00:00 – 00:45.

2.3.2. 1.3.3. A reaction unit in ambient.MOV Timestamps: 00:27 – 00:42.

2.3.3. 1.3.5 Stir three reaction units.MOV Timestamps: 00:30 – 00:45.

2.4. Next, carefully pour the first Erlenmeyer flask of graphite, manganese, and sulfuric acid mixture into a 2 liter beaker of water under agitation **[1]**. Use an infrared thermometer to measure the reaction temperature, which rises to approximately 59 degrees Celsius

**[2]**.

2.4.1. 1.4.2. 1.4.3. 1.4.4. Conduct the second cascade.MOV Timestamps: 00:04 – 01:04.

2.5. Then, slowly add the second Erlenmeyer flask of graphite, manganese, and sulfuric acid mixture to raise the temperature to about 80 degrees Celsius **[1]**. Add the third Erlenmeyer flask of mixture to increase the temperature to approximately 94 degrees Celsius **[2]**. Gradually pour 450 milliliters of 5 percent hydrogen peroxide solution into the beaker, observing a slight temperature rise from the exothermic reaction **[3]**.

2.5.1. **File name:** 1.4.2. 1.4.3. 1.4.4. Conduct the second cascade.MOV **Timestamps:** 01:57 – 02:47, 03:08 – 03:18.

2.5.2. **File name:** 1.4.2. 1.4.3. 1.4.4. Conduct the second cascade.MOV **Timestamps:** 03:40 – 04:15, 05:05 – 05:15.

**File name:** 1.4.5. Agitate the reaction.MOV **Timestamps**: 00:04 – 00:19

2.5.3. 1.5.2. Add 450 mL H2O2 solution.MOV Timestamps: 00:05 – 00:20, 01:23 – 01:33.

2.6. Now, pour the entire reaction mixture into 50 milliliter centrifuge tubes **[1]**. Centrifuge at 1500 *g* for 5 minutes at ambient temperature **[2]**. Collect the sedimented solids to mix them with 1 liter of 5 percent hydrochloric acid solution **[3]**. After agitating the acidic suspension for at least 1 hour, centrifuge at 1500 *g* for 10 minutes, and use a vacuum filtration system with pure water to wash the sedimented graphite oxide solid **[4]**.

2.6.1. 1.6.1. Centrifuge the suspension.MOV Timestamps: 00:26 – 00:36.

1.6.1. Centrifuge the suspension.MOV Timestamps: 00:55 – 01:05.

1.6.1. Centrifuge and collect the solid.MOV Timestamps: 01:25 – 01:30.

2.6.2. 1.6.4. Washing GrO in vacuum filtration.MOV Timestamps: 00:25 – 00:35.

2.7. Next, dry the washed graphite oxide slurry in a drying oven set to 80 degrees Celsius **[1]**. Then, using a stainless steel or ceramic mortar and pestle, grind the dried material to produce a graphite oxide powder **[2]**.

2.7.1. 1.7.1. Dry GrO at 80 C.MOV Timestamps: 00:20 – 00:40.

2.7.2. 1.7.1. Grind GrO material.MOV Timestamps: 00:34 – 00:45, 01:03 – 01:12.

**And**

1.7.1. Collect GrO powder.MOV Timestamps: 00:12 – 00:22

2.8. Use scanning electron microscopy and energy-dispersive X-ray spectroscopy to characterize the obtained graphite oxide and graphene oxide **[1]**.

2.8.1. 1.8.2. SEM EDS characterization of GrO.MOV Timestamps: 00:20 – 00:40.

## 3. Reduction of Graphene Oxide Dispersion using Highly Basic Ammonia Solution

3.1. Drop 25 to 28 percent ammonia solution gradually into the aqueous dispersion of graphite oxide until the pH reaches 10 **[1]**. Sonicate the dispersion using an ultrasonic probe set to 100 watts power, with a continuous cycle and amplitude of 80 percent **[2]**.

3.1.1. 2.1.1. Prepare GrO dispersion pH 10.MOV Timestamps: 00:15 – 00:25, 01:25 – 01:35.

3.1.2. 2.1.2. Sonicate GrO dispersion.MOV Timestamps: 00:15 – 00:30.

3.2. Pour the 1 liter dispersion of graphene oxide into a 1-liter round glass reactor **[1]**. Add 111 milliliters of 25 to 28 percent ammonia solution to raise the pH above 11 and make the mixture highly basic **[2]**. Heat the reaction mixture to 90 degrees Celsius without stirring **[3]**.

3.2.1. 2.2.1. Add GO and NH3 solution.MOV Timestamps: 00:10 – 00:30, 01:30 – 01:40

3.2.2. **File name:** 2.2.2. Heat the reaction to 90 C (1).MOV **Timestamps**: 00:10 – 00:30.

3.2.3. **File name:** 2.2.2. Heat the reaction to 90 C (2).MOV **Timestamps**: 00:10 – 00:30.

3.3. Then, filter the reduced graphene oxide mixture using filter fabrics or cellulose filter paper **[1]** and collect the hydrogel in a plastic box for storage **[2]**.

3.3.1. 2.3.1. Filter RGO suspension (1).MOV Timestamps: 00:15 – 00:45.

3.3.2. **File name:** 2.3.1. Filter RGO suspension (2).MOV **Timestamps**: 00:10 – 00:25, 00:40 – 00:50

3.3.3. 2.3.2. Collect RGO hydrogel for storage.MOV Timestamps: 00:10 – 00:25.

3.4. Use scanning electron microscopy and energy-dispersive X-ray spectroscopy to characterize the reduced graphene oxide hydrogel **[1]**.

3.4.1. 2.4.2. SEM imaging of RGO structure.MOV Timestamps: 00:10 – 00:30, 00:44 – 00:54.

3.5. Finally, disperse 0.1 gram of reduced graphene oxide hydrogel in 300 milliliters of water **[1]**. Use agitation followed by sonication to ensure a homogeneous dispersion of RGO nanosheets, indicating good aqueous dispersibility **[2]**.

3.5.1. **File name:** 2.4.3. Sonicate RGO dispersion (1).MOV **Timestamps**: 00:25 – 00:40

3.5.2. **File name:** 2.4.3. RGO dispersion after sonication (2).MOV **Timestamps**: 00:25 – 00:35

# Results

## 4. Results

4.1. The X-ray diffraction pattern of graphite oxide powder revealed a peak at 10.8 degrees, corresponding to an intersheet distance of 8.19 angstroms **[1]**, and a peak at 42.2 degrees **[2]**.

4.1.1. LAB MEDIA: Figure 2A. *Video editor: Highlight the sharp peak labeled 10.8° on the left side of the XRD graph*.

4.1.2. LAB MEDIA: Figure 2A. *Video editor: Highlight the smaller peak labeled 42.2° on the right side of the XRD graph*.

4.2. SEM images confirmed the multilayer structure of graphite oxide particles **[1]**, and elemental mapping displayed uniform distributions of carbon and oxygen atoms **[2]**.

4.2.1. LAB MEDIA: Figure 3B,C.

4.2.2. LAB MEDIA: Figure 3D E F.

4.3. EDS spectrum and elemental analysis of graphene oxide indicated 64.02% carbon atoms **[1]** and 35.98% oxygen atoms, giving a C/O atomic ratio of 1.78 **[2].**

4.3.1. LAB MEDIA: Figure 4B. *Video editor: Highlight the atom percentage values 64.02 in the right-hand column*.

4.3.2. LAB MEDIA: Figure 4B. *Video editor: Highlight the atom percentage values 35.98 in the right-hand column*.

4.4. SEM images of GO nanosheets exfoliated from graphite oxide at 1000 parts per million concentration revealed successful separation into thin sheets **[1]**.

4.4.1. LAB MEDIA: Figure 4C,D. *Video editor: Highlight the dispersed thin flake structures across both SEM images*.

4.5. XRD patterns of reduced graphene oxide hydrogel and powder revealed a broad peak at 27.7 degrees for hydrogel **[1]**, while powder exhibited sharper peaks at 10.1, 26.6, and 42.6 degrees **[2]**.

4.5.1. LAB MEDIA: Figure 5A. *Video editor: Highlight the blue broad peak at 27.7° labeled as "RGO hydrogel".*

4.5.2. LAB MEDIA: Figure 5A. *Video editor: Highlight the sharp black peaks at 10.1°, 26.6°, and 42.6° labeled as "RGO powder"*.

4.6. SEM images of dehydrated RGO hydrogel revealed non-stacked nanosheets **[1]**, and porous morphology of assembled RGO structures **[2]**.

4.6.1. LAB MEDIA: Figure 6B.

4.6.2. LAB MEDIA: Figure 6C.

4.7. Elemental mapping of the RGO hydrogel structure demonstrated uniform distributions of carbon and oxygen atoms throughout the matrix, with a C/O *(C-Oh)* atomic ratio of 4.16 **[1]**.

4.7.1. LAB MEDIA: Figure 6 D E F

4.8. SEM images of dehydrated RGO nanosheets revealed a wrinkled morphology and thin sheet structure **[1]**.

4.8.1. LAB MEDIA: Figure 7C,D.

**Pronunciation Guide:**

1. **Graphene**
- Pronunciation link: https://www.merriam-webster.com/dictionary/graphene
- IPA: /ˈgræˌfiːn/
- Phonetic Spelling: gra-feen
2. **Cascade**
- Pronunciation link: https://www.merriam-webster.com/dictionary/cascade
- IPA: /kæˈskeɪd/
- Phonetic Spelling: kas-kayd
3. **Graphite**
- Pronunciation link: https://www.merriam-webster.com/dictionary/graphite
- IPA: /ˈgræˌfaɪt/
- Phonetic Spelling: gra-fite
4. **Potassium Permanganate**
- Pronunciation link: https://www.merriam-webster.com/dictionary/potassium%20permanganate
- IPA: /pəˈtæsiəm pərˈmæŋgəˌneɪt/
- Phonetic Spelling: puh-ta-see-um per-man-guh-nayt
5. **Hydrogen Peroxide**
- Pronunciation link: https://www.merriam-webster.com/dictionary/hydrogen%20peroxide
- IPA: /ˈhaɪdrədʒən pəˈrɑːksaɪd/
- Phonetic Spelling: hy-droh-jen puh-rok-side
6. **Hydrochloric Acid**
- Pronunciation link: https://www.merriam-webster.com/dictionary/hydrochloric%20acid
- IPA: /ˌhaɪdrəˈklɔːrɪk ˈæsɪd/
- Phonetic Spelling: hy-droh-klor-ik a-sid
7. **Oxide**
- Pronunciation link: https://www.merriam-webster.com/dictionary/oxide
- IPA: /ˈɑːksaɪd/
- Phonetic Spelling: ok-side
8. **Oxidation**
- Pronunciation link: https://www.merriam-webster.com/dictionary/oxidation
- IPA: /ˌɑːksɪˈdeɪʃən/
- Phonetic Spelling: ok-si-day-shun
9. **Reduction**
- Pronunciation link: https://www.merriam-webster.com/dictionary/reduction
- IPA: /rɪˈdʌkʃən/
- Phonetic Spelling: ri-duk-shun