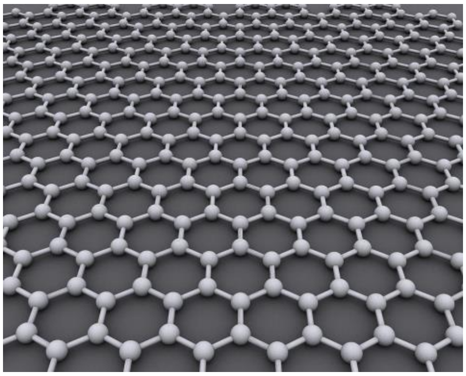

Taylor Vortex Reactor

Graphene & Graphene Oxide

Business Case

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Taylor Vortex Reactor for Graphene & Graphene Oxide

A large-scale 2D graphene producer based in the Midwest US chose our 10L Taylor Vortex Reactor to maintain their market advantage and innovative leadership in global graphene production.

The client produces single- and few-layer graphene and graphene oxide for applications in electric vehicle (EV) lithium-ion battery, nano-intermediates-thermoplastics, conductive films, energy storage, and more.

Graphene: Applications and Conventional Manufacturing Processes

Graphene is a 2D carbon material with a hexagonal molecular structure. It is one of the strongest and thinnest materials in the world. In 2010, graphene won the Nobel Prize in Physics for its extraordinary properties. Graphene's excellent electric and thermal conductivity makes it the basis of many next-generation solutions for **energy storage, electric vehicles (EVs), solar cells**, and more.

While well-known methods of graphene production exist, including Hummer's method, sonication exfoliation, and homogenization exfoliation, graphene has traditionally been expensive and difficult to produce.

Each of the above methods have significant limitations, such as environmental concerns, low yields, complex multi-step processes, and the production of small, uneven flakes. These limitations prevent industrial-scale production of graphene, which should be high quality, low cost, high yield, and environmentally friendly.

Taylor Vortex Reactor for Graphene & Graphene Oxide

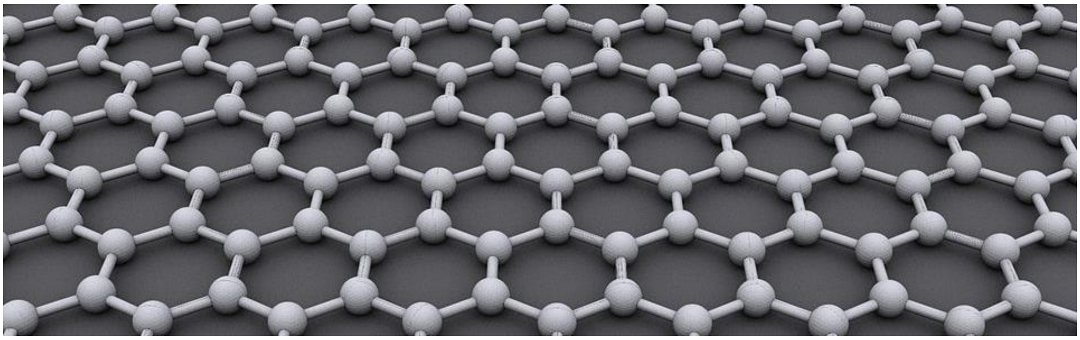
Taylor Vortex Reactor: New Method for Graphene Oxide Synthesis

Recently, the literature on graphene oxide synthesis via the Taylor Vortex Reactor has been growing. Also referred to as Taylor-Couette Flow Reactor, Couette-Taylor Flow Reactor, Stress Shearing Reactor, the reactor's production of purer, more uniform, more efficient, and higher yields of graphene oxide and graphene flakes has been replicated in several studies across the world.

In the next few pages, we summarize findings from several peer-reviewed studies that highlight the advantages of using a Continuous Vortex Flow Reactor for graphene oxide synthesis.

In short, the advantages for our graphene-producing clients include:

- Shortened reaction time with continuous synthesis
- High yield production rates
- High quality products with low defect rates
- Structurally uniform products with larger flake sizes
- Easy operation of reactor that gives control over product geometry
- Reduced water and acid waste for green synthesis



Taylor Vortex Reactor for Graphene & Graphene Oxide

Advantages of Taylor Flow Reactor for Graphene Oxide Synthesis

1. Shortened Reaction Time with Continuous Synthesis

In 2019, the Taylor-Couette Flow Reactor "revolutionized the synthesis of graphite oxide" by shortening the time of oxidation from 4 hours with the Hummer's method to **30 minutes** (AlAmer et al., 2019).

Despite the drastically shortened reaction time, the resulting graphite oxide sheets were uniformly structured with low defect rates and high yields.

Park et al. (2017) also compared the Hummer's method and the Couette-Taylor Reactor for graphene oxide synthesis. They found that **a 60-minute oxidation reaction in the Couette-Taylor Reactor resulted in a 98% yield of uniform, large-area flakes of few-layer graphene oxide**. In contrast, the Hummer's method produced a 34% yield within the same reaction time.

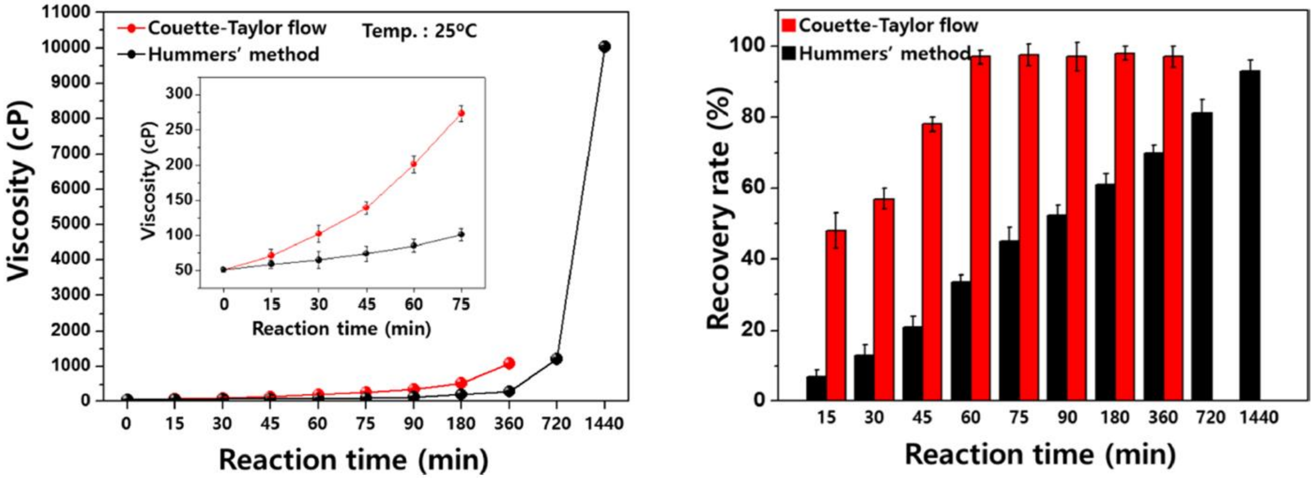


Figure 1. Comparison of the Hummer's Method and Taylor-Couette Flow Method. (Left) Viscosity of the graphite oxide mixture with varying reaction times. (Right) Recovery rate of GO in accordance with the reaction time. (Park et al., 2017)

Taylor Vortex Reactor for Graphene & Graphene Oxide

Advantages of Taylor Flow Reactor for Graphene Oxide Synthesis

2. High Yield, High Quality, Uniform Graphene and Graphene Oxide

Few-layer graphene via non-oxidative exfoliation was also produced by Tran et al. (2016) with LPR Global's Taylor Vortex Flow Reactor. The researchers concluded that our reactor demonstrates **high potential to produce high quality graphene on an industrial scale**. After measuring the AFM height of more than 250 flakes, the study found that **90% of the flakes were composed of fewer than 5 layers**.

Raman spectroscopy also indicated low defect rates, with a Raman D/G band intensity ratio (ID/IG) of 0.14. An XPS also showed no evidence of oxidation, which suggests that the Taylor Vortex Reactor produced high quality graphene flakes.

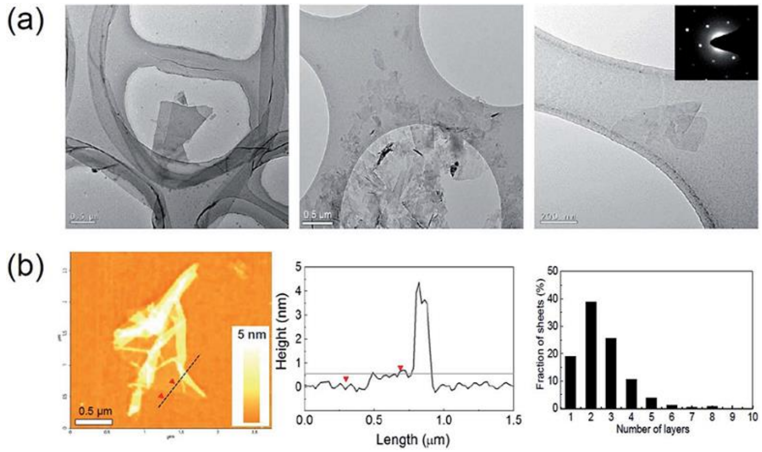


Figure 2. a) TEM images and b) AFM images where the corresponding height is ~0.6nm, and histogram of number of graphene layers (Tran et al., 2016).

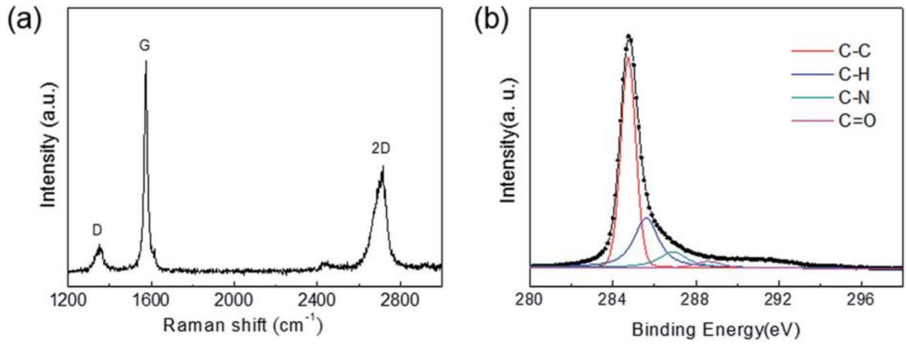


Figure 3. a) Raman spectroscopy and b) XPS Spectra of exfoliated graphene flakes (Tran et al., 2016).

Taylor Vortex Reactor for Graphene & Graphene Oxide

Advantages of Taylor Flow Reactor for Graphene Oxide Synthesis

2. High Yield, High Quality, Uniform Graphene and Graphene Oxide

Moreover, Park et al. (2017) also found that the lateral size of the graphene oxide sheets was easily manipulated by simply adjusting the rotational speed of the Taylor Vortex Flow Reactor and the reaction time. This finding is a significant improvement from sonication and homogenization methods, both of which tear graphene flakes into small, uneven pieces.

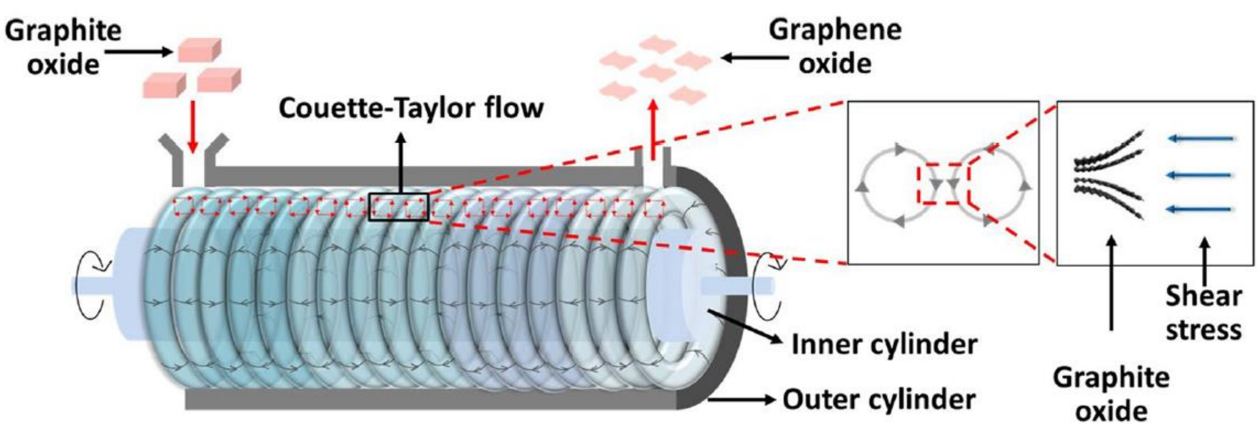


Fig 4. Conceptual illustration of Taylor-Couette Flow method of graphite oxide exfoliation via shearing stress. (Park et al., 2017)

Graphite oxide synthesis via the Taylor-Couette Reactor was also found to produce assessed uniformly structured graphite oxide sheets with **low defect rates and high yields** by AlAmer et al. (2019). Due to the wall shear exfoliation induced by the rotating inner cylinder, the number of graphite layers decreased from ~85 layers (natural graphite) to ~8 layers.

Raman spectroscopy was also used to confirm high homogeneity in the geometry of the graphite oxide produced by the vortex flow regime.

Taylor Vortex Reactor for Graphene & Graphene Oxide

Advantages of Taylor Flow Reactor for Graphene Oxide Synthesis

2. High Yield, High Quality, Uniform Graphene and Graphene Oxide

Expandable Graphite and Few-Layer Graphene for Graphene Fiber

In a separate study, AlAmer et al. (2020) compared the Taylor-Couette Reactor to conventional batch processes for the exfoliation of natural graphite. Graphite exfoliation produces expandable graphite and few-layer graphene, which can be spun into ultralight graphene fiber and have high commercial applicability.

The high shear rates achieved during the vortex flow regime resulted in structurally homogenous few-layer graphene sheets with large lateral dimensions of over 10 µm.

This was an important finding, as flake size is a significant determinant of macroscopic fiber properties, in that larger flake sizes led to stronger fibers.

Taylor Vortex Reactor for Graphene & Graphene Oxide

Advantages of Taylor Flow Reactor for Graphene Oxide Synthesis

2. High Yield, High Quality, Uniform Graphene and Graphene Oxide

Expandable Graphite and Few-Layer Graphene for Graphene Fiber

Notably, only 1-3 hours of shearing time were required to achieve expandable graphite and few-layer graphene with almost no defects. The resulting graphene fiber (bulk density 0.35g/cm3) displayed a mechanical strength of 0.5 GPa without any modification or heat treatment.

The figure below shows that graphene fibers spun with the Taylor-Couette Reactor's expandable graphite display significantly better mechanical properties than fibers using commercially available expandable graphite.

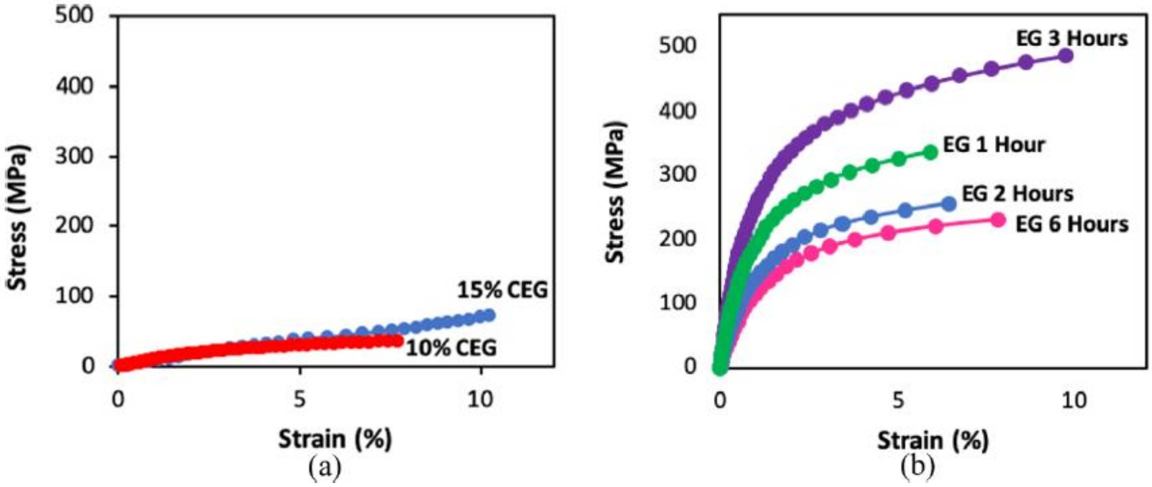


Fig 5. Mechanical properties of graphene fibers. (a) commercially available expandable graphite (CEG) and (b) expandable graphite from Taylor-Couette method. (AlAmer et al. 2020)

Taylor Vortex Reactor for Graphene & Graphene Oxide

Advantages of Taylor Flow Reactor for Graphene Oxide Synthesis

3. Green Synthesis with Reduced Acid and Water Waste

Unlike the Hummer's method, the Taylor Vortex Flow Reactor successfully produced high yields of graphene oxide at low viscosities of under 200 cP (Park et al., 2017).

The low-viscosity mixture allowed for an initial separation H_2SO_4 from the graphene oxide slurry with a simple filtration system. Subsequently, the purification of the graphene oxide product used 75% less water compared to the Hummers' method.

This finding overcomes one of Hummer's method's greatest limitations: its inability to produce high yields of graphene oxide from low-viscosity mixtures, which then requires great amounts of water for acid purification.

method	amount of mixture (g)	volume of water (mL)	final pH
Hummers' method	1	1601 ± 11	5
first filtration (fresh H_2SO_4)	1	400 ± 7	5
second filtration (one-time recycled H_2SO_4)	1	399 ± 3	5
third filtration (two-time recycled H_2SO_4)	1	402 ± 6	5

Table 1. Volume of water used in washing process for the Hummer's and Taylor Vortex / Filtration Methods. (Park et al., 2017)

Taylor Vortex Reactor for Graphene & Graphene Oxide

Advantages of Taylor Flow Reactor for Graphene Oxide Synthesis

3. Green Synthesis with Reduced Acid and Water Waste

Moreover, Park et al. (2017) found that graphene oxide synthesis with once- and twice-recycled filtered H_2SO_4 for produced comparable quality and yield. Recovery rates for graphene oxide produced with fresh, once-recycled, and twice-recycled H_2SO_4 were approximately 98.5%, 97.1%, and 97.9%, respectively.

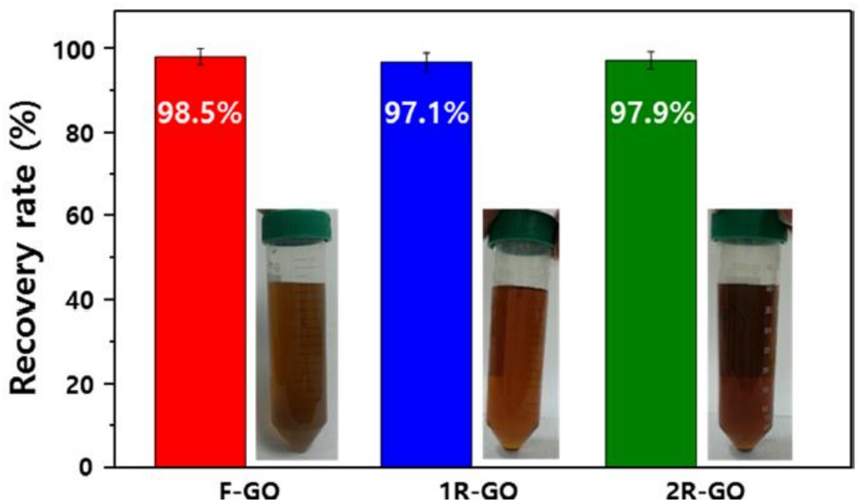


Fig 6. Recovery rate of the graphene oxide obtained with fresh (F-GO), once-recycled (1R-GO), and twice-recycled (2R-GO) sulfuric acid. (Park et al., 2017)

Taylor Vortex Reactor for Graphene & Graphene Oxide

Advantages of Taylor Flow Reactor for Graphene Oxide Synthesis

Customizable Reactor for Pilot-Scale Production

The 10L Taylor Vortex Reactor used by this client is ideal for pilot-scale production. The standard model has a maximum agitation speed of 1500 RPM and maximum reaction temperature of 90°C. Our Continuous Flow Reactor can also be customized for higher agitation speeds and reaction temperatures.

The user-friendly PLC interface also allows our client to save their reaction data, thereby facilitating their reaction optimization process.

For questions on how LPR Global's Taylor Flow Reactor can enhance your advanced materials manufacturing, please reach out to info@lprglobal.com.

Taylor Vortex Reactor for Graphene & Graphene Oxide

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