Investigating the Effect of Sonication Time and Centrifugation Speed on Graphene Production

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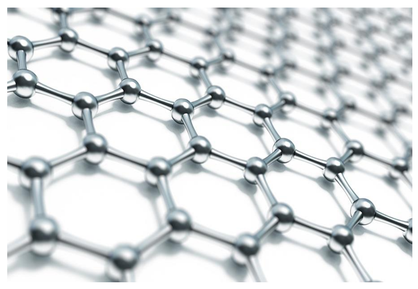
Abstract

Graphene, the 2D form of graphite, has been of great interest in recent years. Its structure gives it **remarkable electronic and mechanical properties** [1], which can be used for an extensive range of applications. This study focusses on using sonication and centrifugation to create graphene from graphite and observing how changing these variables affects the size of the graphene flakes produced.

Introduction

What is graphene?

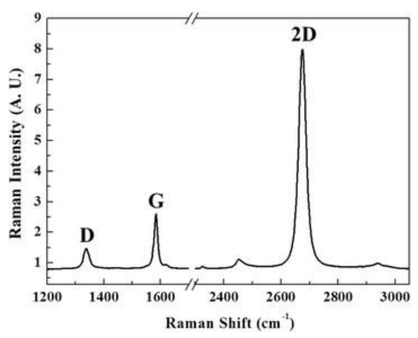
- Carbon atoms arranged in a hexagonal pattern
- Single layer of graphite
- Used in many areas, such as energy storage in solar cells [2] and drug delivery [3].



Structure of a monolayer of graphene [5]

Graphene can be made by many different processes, including liquid-phase exfoliation. Sonication uses **ultrasound waves** to break apart layers of graphite, and subsequent centrifugation **separates** the larger flakes from the smaller ones.

Raman spectroscopy with a single 532nm laser was used to analyse the flakes. From the Raman data, the area ratios of the **D and G peak** were used to estimate the lateral flake size, and the **2D peak** was used to estimate the number of graphene layers [4].



Raman spectrum of the monolayer graphene excited with a 532-nm laser [6]

Objectives

The main aims of this experiment were to see the effect of :

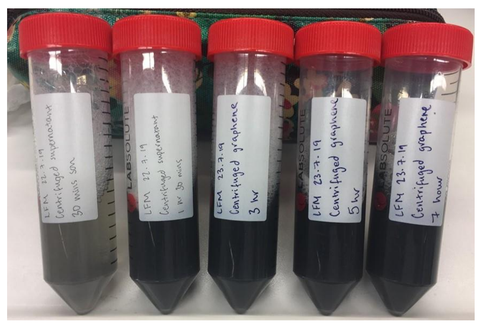
- Increasing sonication time
- Increasing centrifugation speed

on the size and quality of graphene flakes using a liquid-exfoliation method with a solution of water and sodium dodecyl sulphate (SDS).

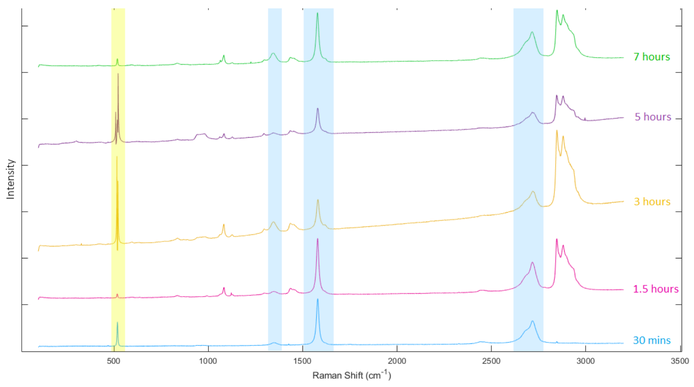
Different applications may require different sizes of graphene flake, so size optimisation is of great importance.

Effect of Sonication Time

The times for sonication were **30 mins, 1.5 hrs, 3 hrs, 5 hrs, and 7 hrs.** The samples were then centrifuged at **500rpm** for **one hour**. Increasing sonication time gave clear visible differences in solution, with the **longer samples being darker** and more opaque. The samples were also shinier before centrifugation with increasing time as the graphite was broken down into smaller flakes.

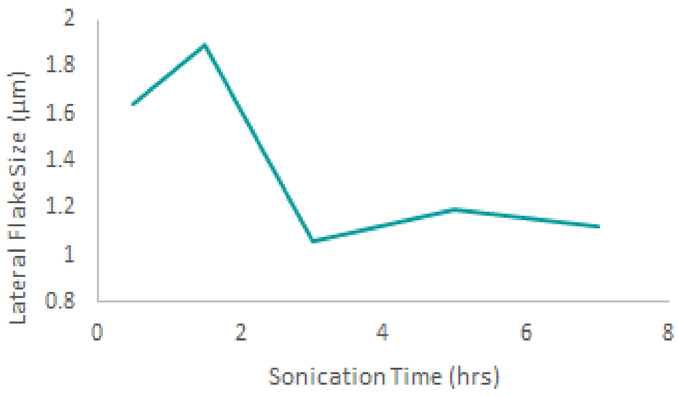


3 flakes from each sample were analysed using Raman spectroscopy, and the results were averaged.



- The blue regions indicate the peaks that are specific to graphene.
- The yellow region represents the peaks generated by the silicon substrate.
- The remaining peaks are produced by SDS.

The lateral flake size generally **decreased with increasing sonication time**, but the trend does not seem well established.

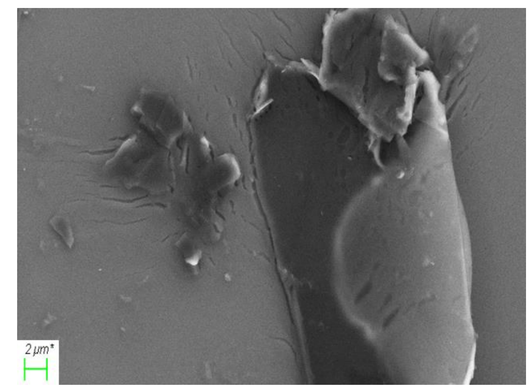


The asymmetrical shape of all of the 2D peaks indicates that it is most likely that **multi-layer graphene** was present in the areas that were analysed.

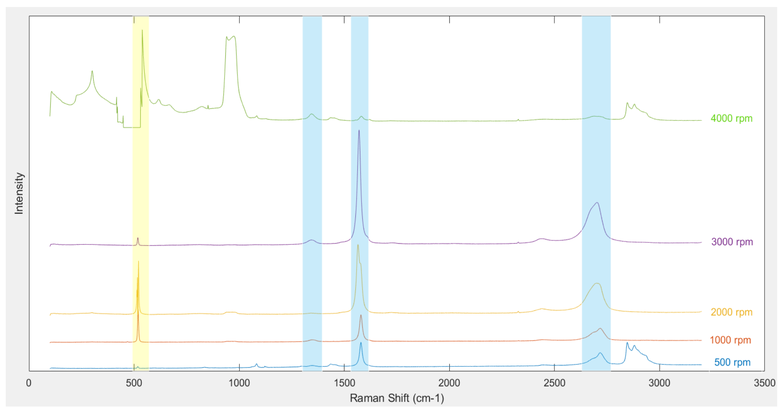
Effect of Centrifugation Speed

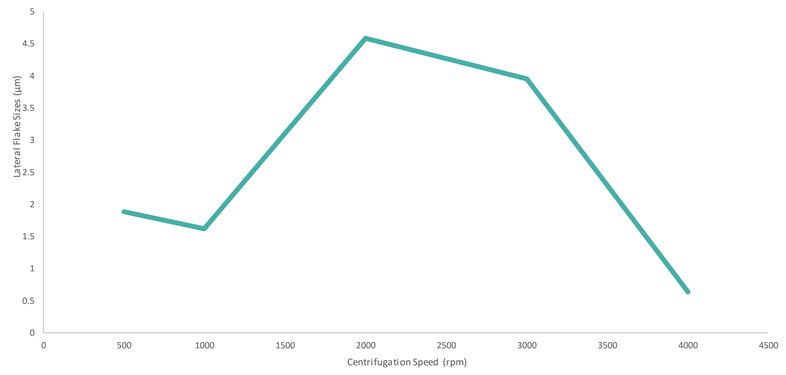
Each sample was placed in the Allegra X-15R Centrifuge at 20 degrees Celsius and centrifuged at varying speeds - **500rpm, 1000rpm, 2000rpm, 3000rpm and 4000rpm** - for **one hour**. The supernatant obtained was lighter **with increasing speed** showing that the **average flake size was decreasing**.

A few drops of the supernatant were pipetted on to small pieces of silicon to undergo Raman spectroscopy.



SEM image of a graphene flake obtained from the sample centrifuged at 1000rpm.





The **first** blue highlighted peak is the D peak and it signifies the level of defects present in the graphene.

The **second** highlights the G peak, which shows that the flake under analysis is indeed graphene.

The **third** is the 2D peak; the sharper and smoother it is the lesser the number of graphene layers present [7]. As this peak gets smoother with increasing speed, it can be inferred that **increasing centrifugation speed decreases the number of layers of graphene** obtained.

Generally, the lateral flake size of graphene **decreases with an increase in centrifugation speed**, however, as both the flake sizes obtained at 2000rpm and 3000rpm are larger than those of the 1000rpm, it is possible that larger flakes were used in the analysis of those particular cases and more flakes need to be analyzed to obtain a conclusive result.

Conclusions

Increasing sonication time:

- Generally decreased lateral flake size
- Produced more concentrated graphene solutions

Increasing centrifugation speed:

- Generally decreased lateral flake size
- Decreased the number of graphene layers

The sample sonicated for 1.5 hrs (right) can be seen to be darker and has finer graphite flakes than the 30 mins sample (left).

The results supported the hypothesis but were irregular, so analysis of more flakes for both variables would make the results more reliable and fully confirm the expected trend.

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