

# Graphene oxide gel fabrication using ultrafast laser ablation



UNIVERSITY OF  
**WATERLOO**

[uwaterloo.ca](http://uwaterloo.ca)

**M. Irannejad**, K.H. Ibrahim, A. Ramadhan, M.  
Hajialamdari, J. Sanderson and M. Yavuz

Waterloo Institute of Nanotechnology(WIN)  
&  
Dept. of Mechanical and Mechatronics Engineering

# Outline

---

- Introduction
- fs-laser assisted graphene oxide gel
  - Laser parameter
  - Laser processing
- Graphene oxide gel Characterization
  - Absorption Spectroscopy
  - Raman Spectroscopy and XRD analysis
- Summary

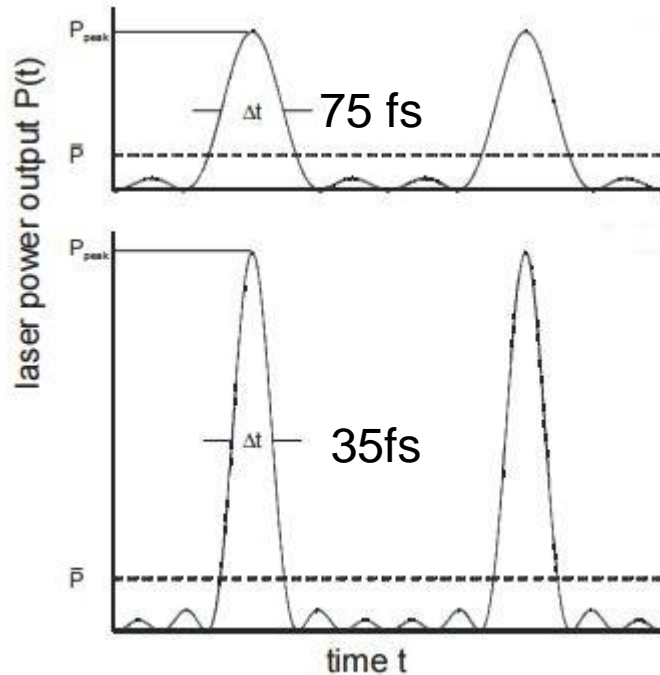
# *Introduction: Graphene oxide gel*

- The graphene oxide hydrogel/gel have been extensively used as a soft material in many applications like drug delivery, tissue engineering, sensors and actuators
- They have several advantages over the conventional graphene/graphene oxide sheets such as large surface area, high electrical and high thermal conductivity.
- In self-assembly method, the gelation of the graphene oxide sheets is acquired by different molecular interaction, includes electrostatic interaction,  $\pi$ - $\pi$  stacking and hydrogen bonding
- To produce the graphene oxide gel from aqueous graphene oxide solution, a chemical agent like *nickel foam*, *Pluronic copolymer*, *Ferrocene*, *DNA*, *PVA*, *divalent and trivalent metals* ( $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Cu}^{2+}$ , and  $\text{Ce}^{3+}$ ) is an essential requirement.

# Laser-assisted Graphene Oxide gel: *Laser parameter*

## • Laser Parameter

- $\lambda=800$  nm
- Rep. rate: 1KHz
- Pulse with: 75 fs and 35 fs



- Pulse intensity =  $4 \times 10^{13}$  to  $4 \times 10^{15}$  W/cm<sup>2</sup>  
=  $8 \times 10^{15}$  to  $8 \times 10^{19}$  W/cm<sup>2</sup>
- Focal length=5 cm and 10 cm

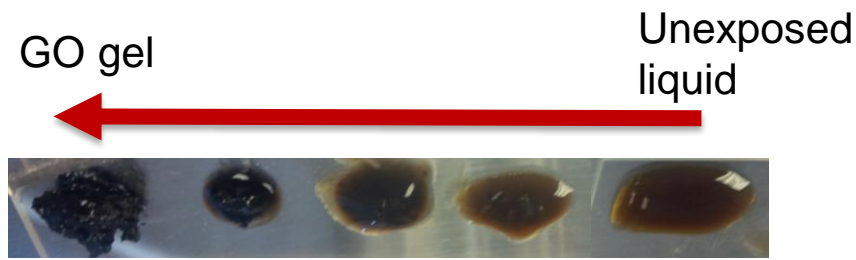
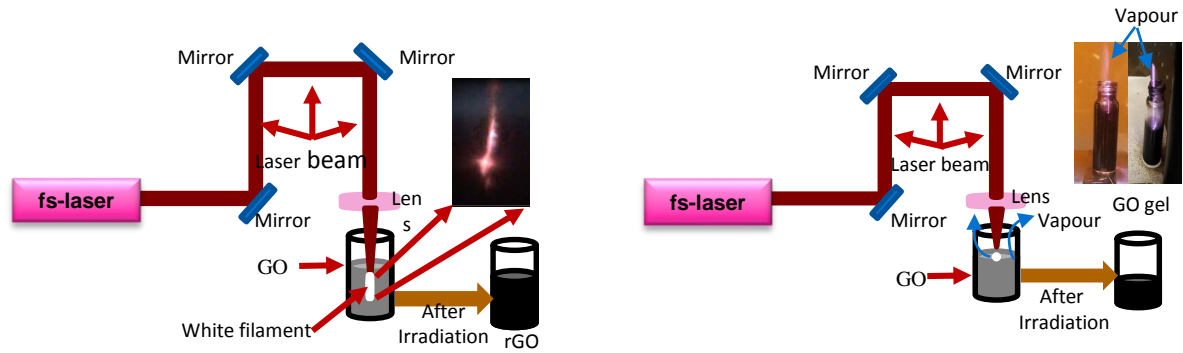
## • Exposure Conditions

- Exposure time: 8 min to 7hrs
- Room temperature

## • Graphene oxide

- Dispersion in water
- Concentration: 6.2mg/mL
- Flake size: 0.5-5  $\mu$ m

# Laser-assisted graphene Oxide gel: *Laser Processing*



# Laser-assisted Graphene Oxide gel: *Processing parameters*

Pulse energy (mJ)	Pulse duration(fs)	Focal length (cm)	Exposure time	Volume (mL)
250×10 <sup>-3</sup> (rGO)	75	5	7 hour	5
250×10 <sup>-3</sup> (GO gel)	75	5	7 hour	5
2	35	5	58 min	5
2	35	5	20 min	1.5
2	35	10	8 min	1.5
250×10 <sup>-3</sup>	35	10	15 min	1.5

Time	Observation (P=250mW, 75 fs, 5 cm)
0	Starting Irradiation
1 hr	Regular vaporization
1.5 hr	Nearly 20% of the volume has vaporized. Volume loss is faster than usual.
4 hr	~50% volume lost
7 hr	gel

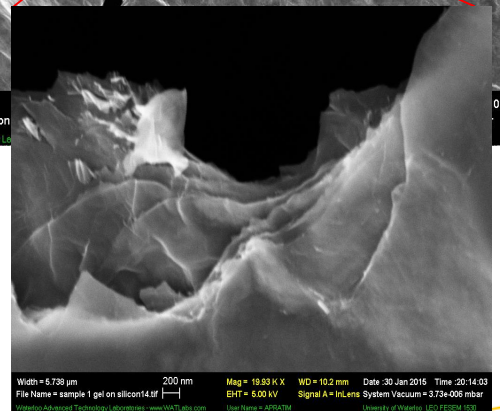
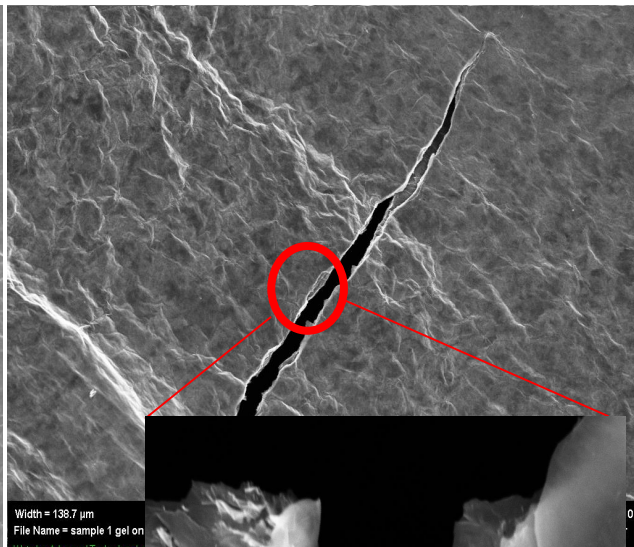
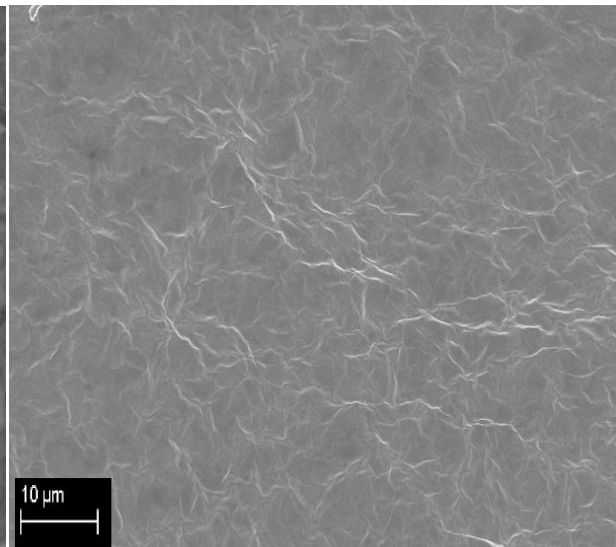
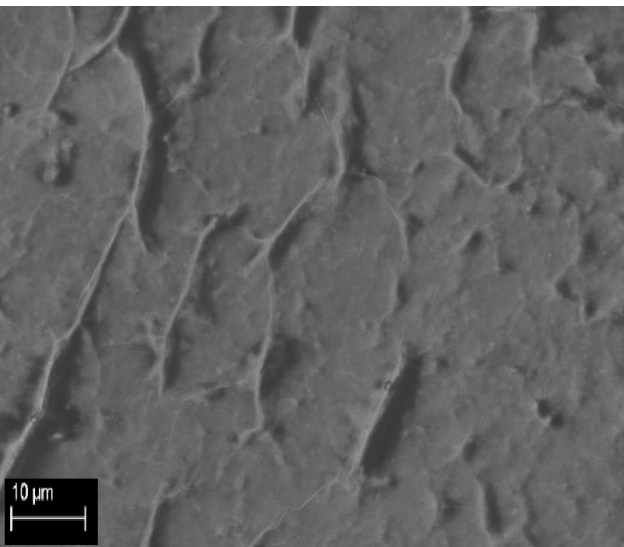
# Graphene Oxide gel: *Surface morphology*

➤ **SEM**

Unexposed

rGO

GO gel



	Carbon	Oxygen
	(At%)	

Unexposed	68.40	31.60
-----------	-------	-------

rGO	68.98	31.02
-----	-------	-------

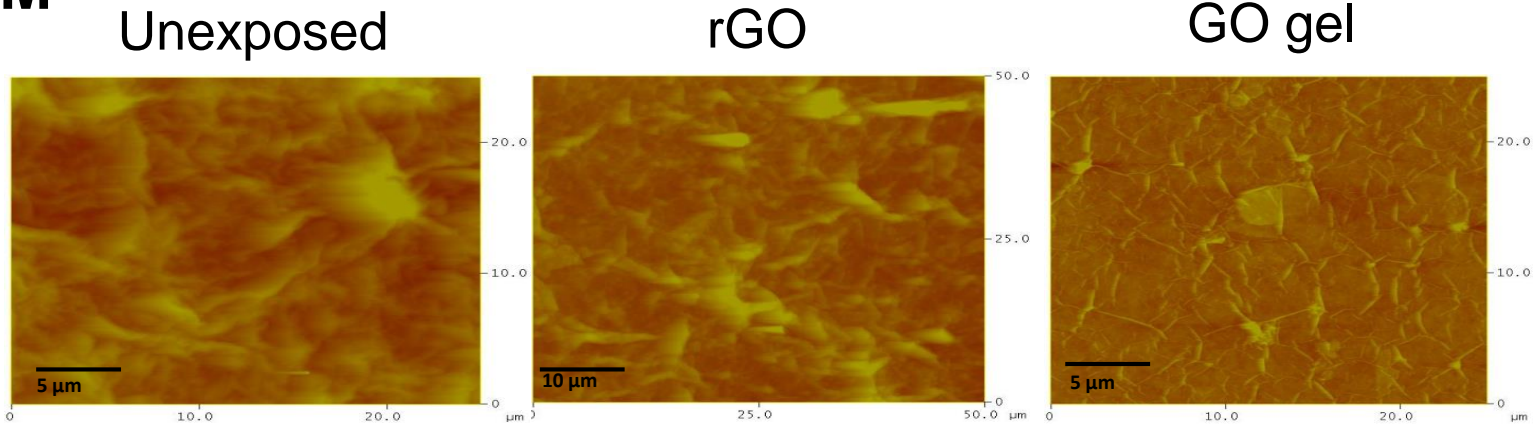
GO gel	73.59	26.41
--------	-------	-------

UNIVERSITY OF  
**WATERLOO**



# Graphene Oxide gel: *Surface morphology*

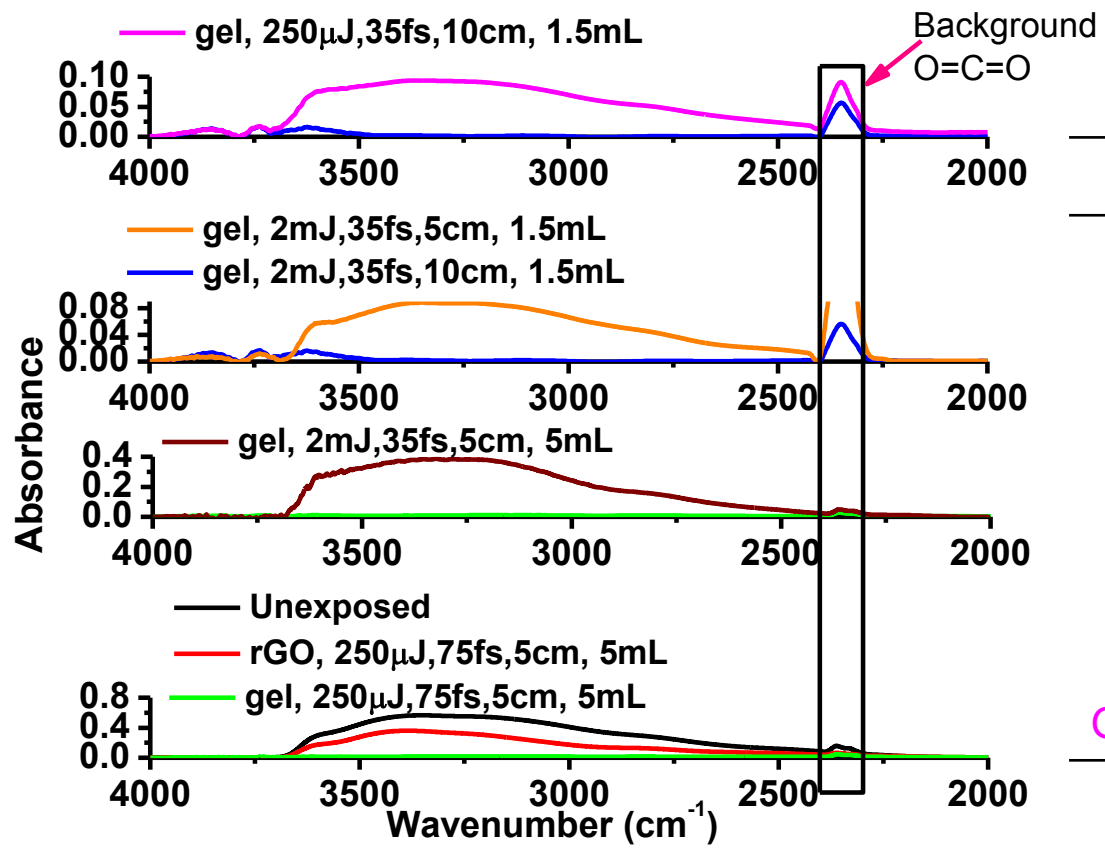
➤ **AFM**



Sample	R(nm)	t(nm)	Sample	R (nm)	t (nm)
Unexposed	11.992	23.76	2mJ,35fs,5cm	54.809	205.54
rGO	25.30	83.81	2mJ,35fs,10cm	22.57	134.54
<b>250μJ,75fs,5cm</b>	<b>5.587</b>	<b>171.95</b>	250μJ,35fs,10cm	29.171	164.40
2mJ,35fs,5cm	44.63	165.66			

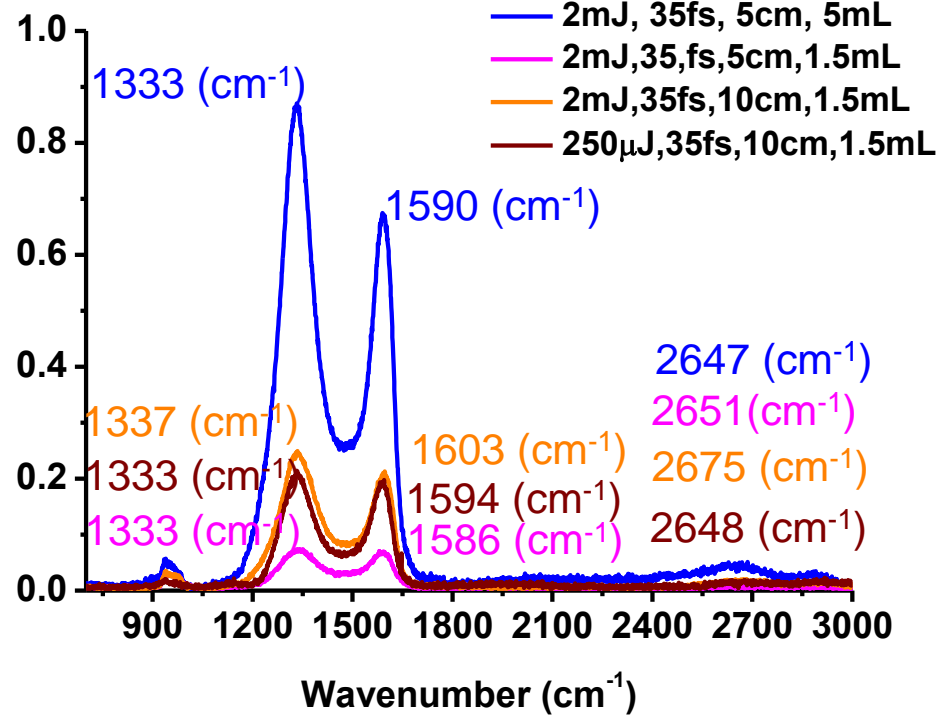
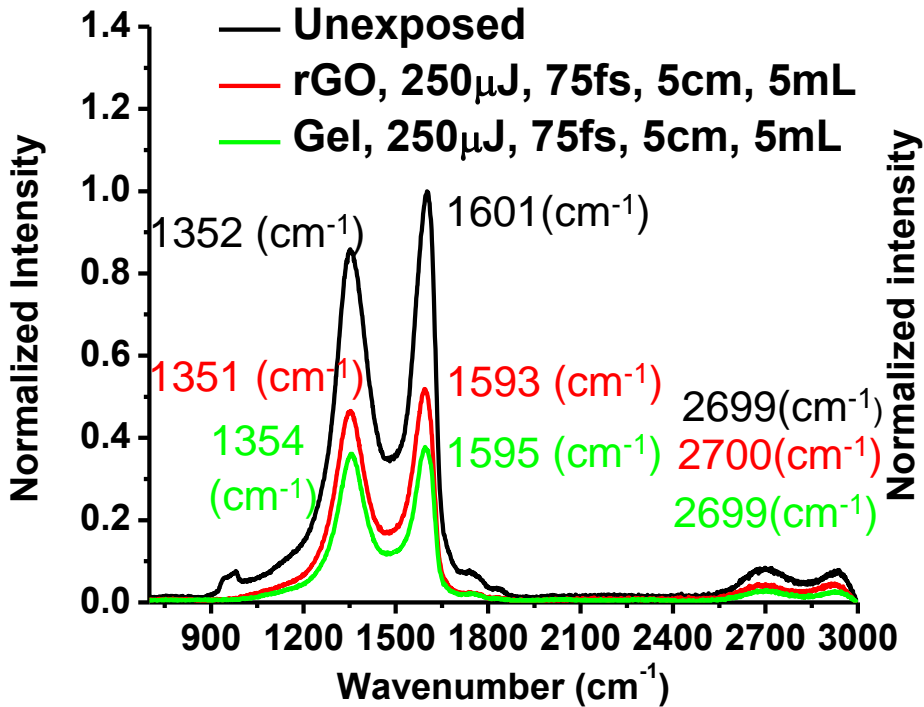


# Graphene Oxide gel: *Absorption spectroscopy*



<i>Irradiation Parameter</i>	<i>A<sub>OH</sub>%</i>
Unexposed	56.5
rGO,250 $\mu$ J,75fs,5cm, 5mL	36.1
Gel,250 $\mu$ J,75fs,5cm, 5mL	2.1
Gel,2mJ,35fs,5cm, 5mL	38.3
Gel,2mJ,35fs,5cm, 1.5mL	8.8
Gel,2mJ,35fs,10cm, 1.5mL	0.2, 0.1.6
Gel,250mJ,35fs,10cm, 1.5mL	9.4

# Graphene Oxide gel: *Raman spectroscopy*



- λ= 488 nm; power= 20 mW
- Spin coating @ 500 rpm & 30 min post baking @90°C

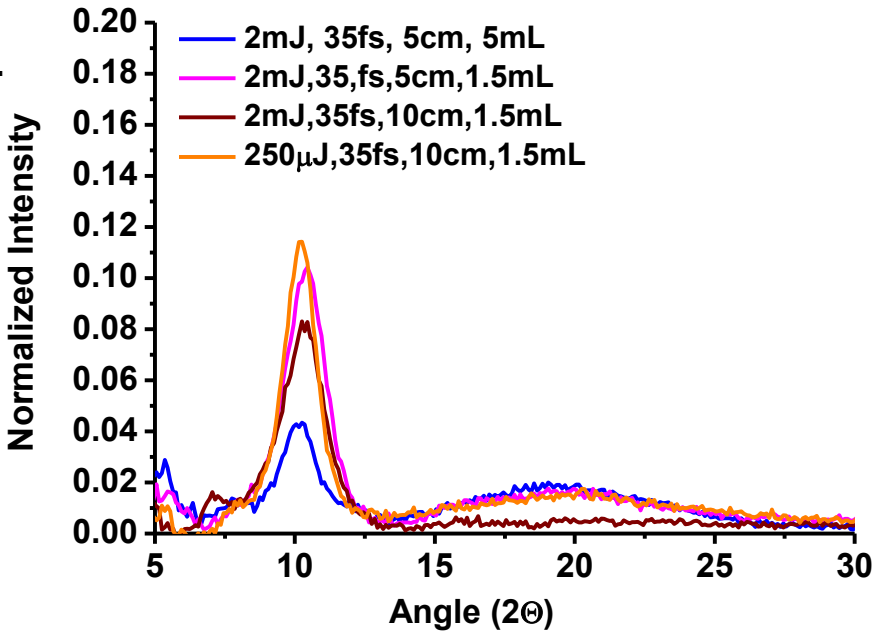
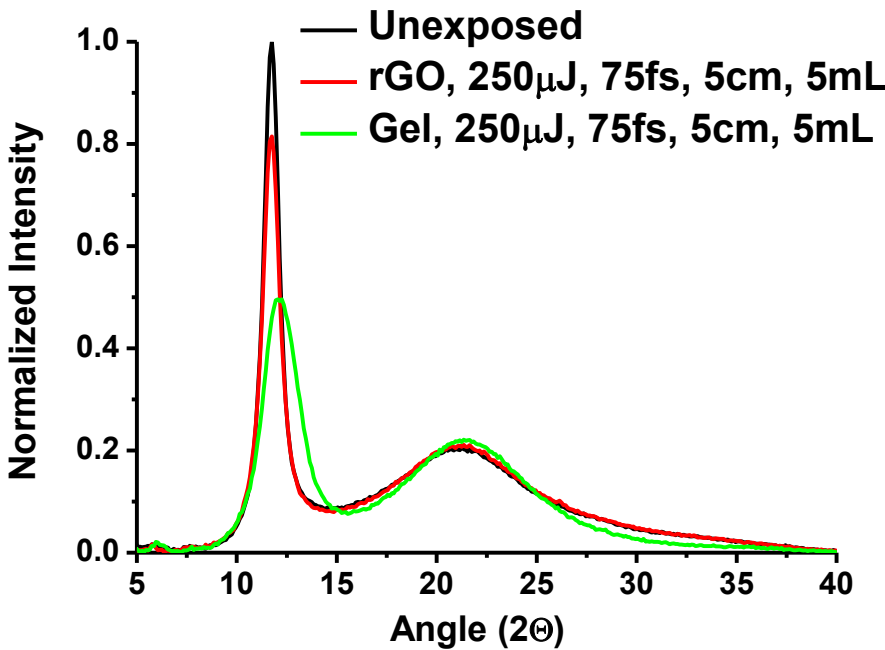
UNIVERSITY OF  
**WATERLOO**

# Graphene Oxide gel: *Raman spectroscopy*

Laser Irradiation parameter	$I_D/I_G$	$I_{2D}/I_G$
Unexposed	0.86	0.09
rGO, 250 $\mu$ J,75fs,5cm, 5mL	0.89	0.09
Gel, 250 $\mu$ J,75fs,5cm, 5mL	0.96	0.07
Gel, 2mJ,35fs,5cm, 5mL	1.29	0.07

Laser Irradiation parameter	$I_D/I_G$	$I_{2D}/I_G$
Gel,2mJ,35fs,5cm,1.5mL	1.06	0.09
Gel,2mJ,35fs,10cm,1.5mL	1.17	0.08
Gel,250 $\mu$ J,35fs,10cm,1.5mL	1.08	0.07

# Graphene Oxide gel: XRD Analysis



# Graphene Oxide gel: XRD Analysis

	(001) peak	(002) peak	Interlayer distance (nm)	
	2 $\theta$	2 $\theta$	(001) peak	(002) peak
Unexposed	11.75	21.25	0.753	0.418
rGO,250 $\mu$ J, 75fs,5cm, 5mL	11.75	21.35	0.753	0.418
<b>Gel,250<math>\mu</math>J, 75fs,5cm, 5mL</b>	<b>12.25</b>	<b>21.35</b>	<b>0.723</b>	<b>0.416</b>

	(001) peak	(002) peak	Interlayer distance (nm)	
	2 $\theta$	2 $\theta$	(001) peak	(002) peak
Gel,2mJ, 35fs,5cm, 5mL	10.25	19.05	0.863	0.466
Gel,2mJ, 35fs,5cm, 1.5mL	10.45	19.05	0.863	0.466
Gel,2mJ, 35fs,10cm, 1.5mL	10.25	19.05	0.863	-
Gel,250 $\mu$ J, 35fs,10cm, 1.5mL	10.25	20.25	0.863	0.466

# Summary

---

- Graphene oxide gel were produced by using surface ablation for the first time with high concentration of *Carbons* and lower *Oxygen* concentration.
- OH absorbance was reduced by of **96%** and **99%** in a GO gel produced using laser pulse energy of 250  $\mu\text{J}$ , focal length of 5 cm and 2 mJ, focal length of 10 cm respectively
- The smaller interlayer distances was achieved for longer laser irradiation.