Production of High-Quality Exfoliated graphene using Two step electrochemical approach
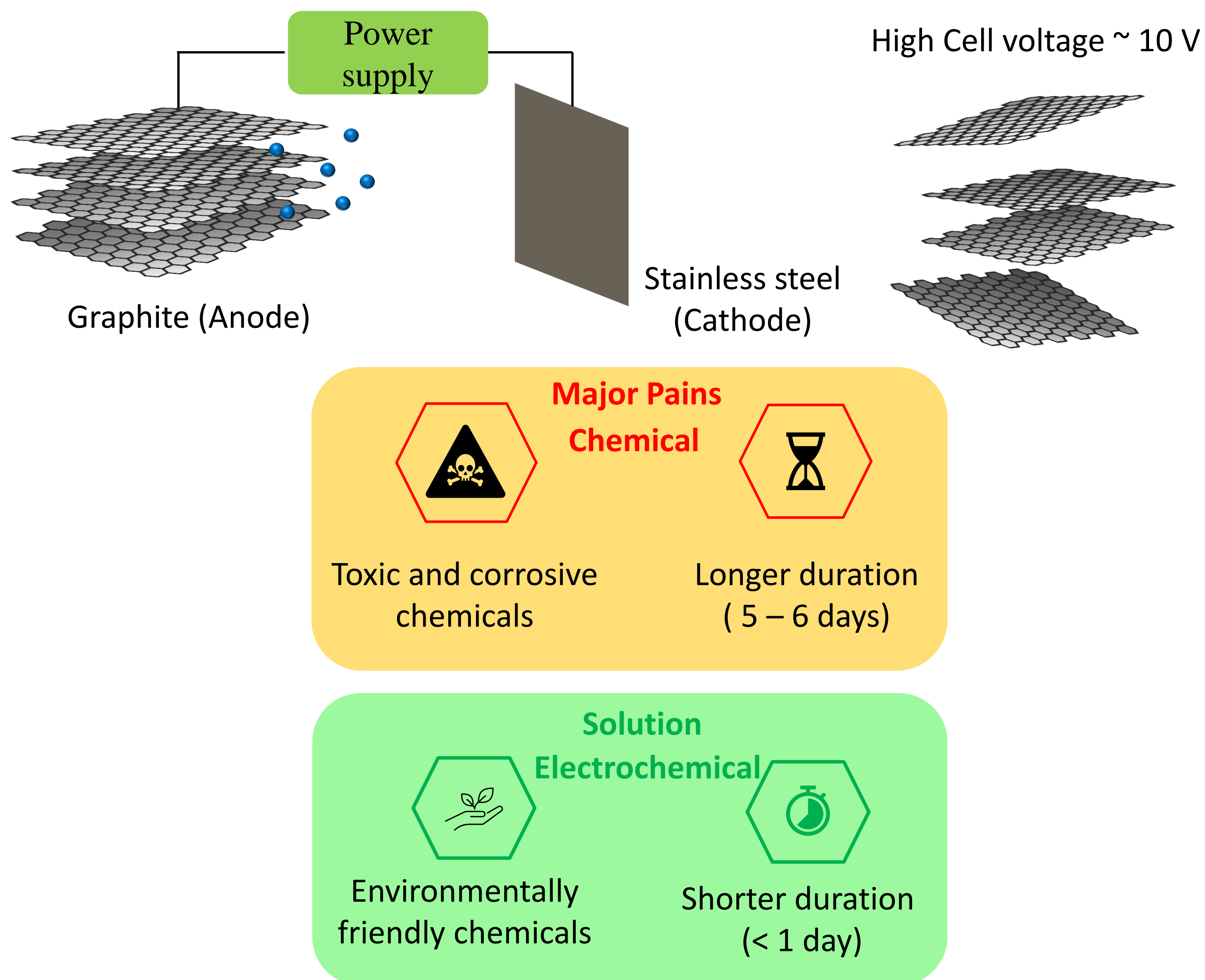
Ashutosh K. Singh[a], Maria Page-Perez[b], Nael Yasri[a], Stuart Holmes[b] and Edward PL Roberts[a]
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Chemical vs Electrochemical Approach

Power supply

High Cell voltage ~ 10 V

Graphite (Anode)

Stainless steel (Cathode)

Major Pains Chemical

Toxic and corrosive chemicals

Longer duration (5 – 6 days)

Solution Electrochemical

Environmentally friendly chemicals
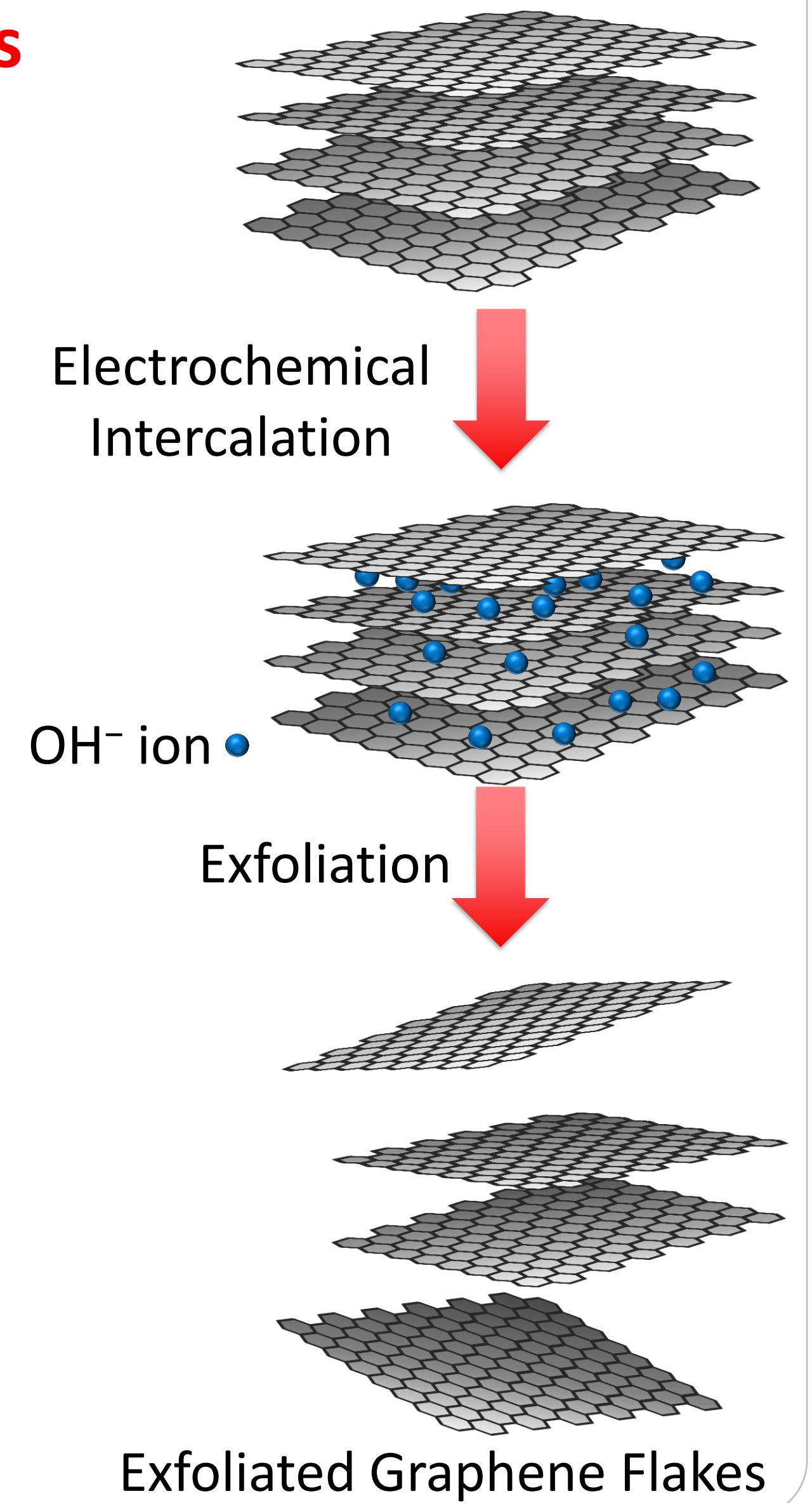
Shorter duration (< 1 day)

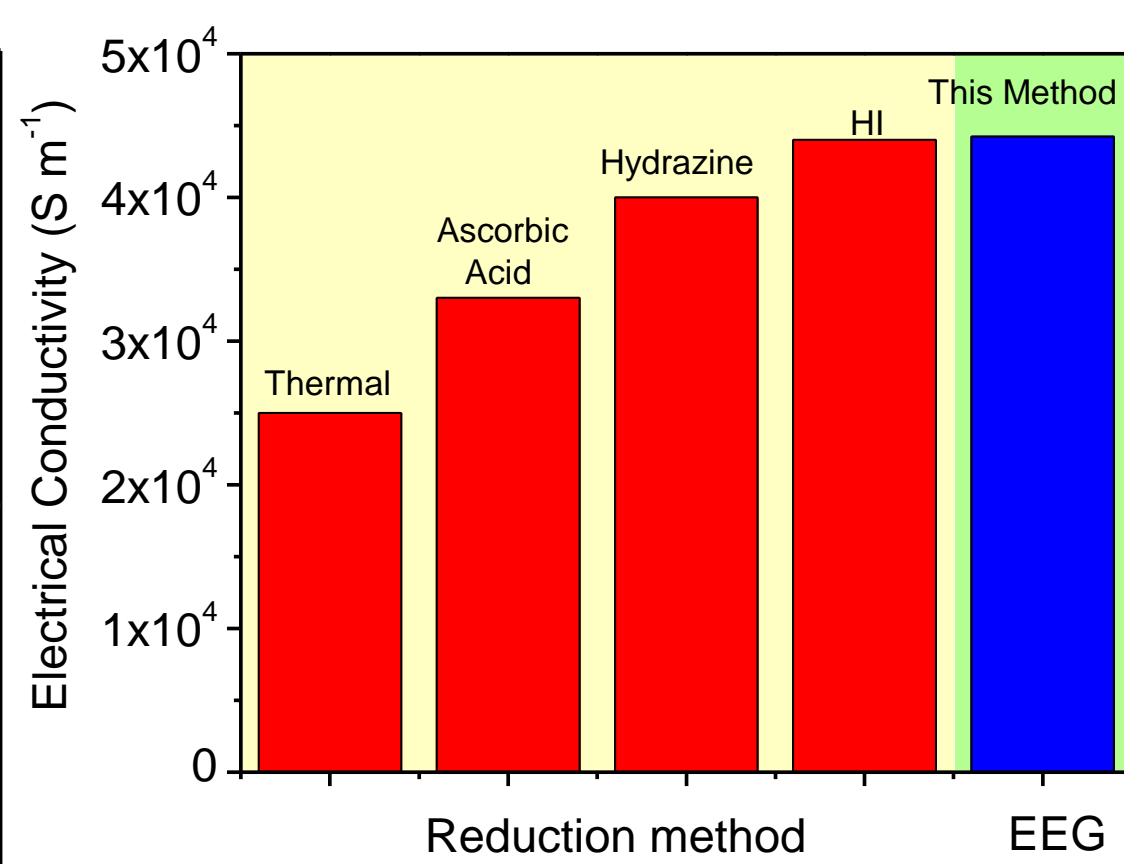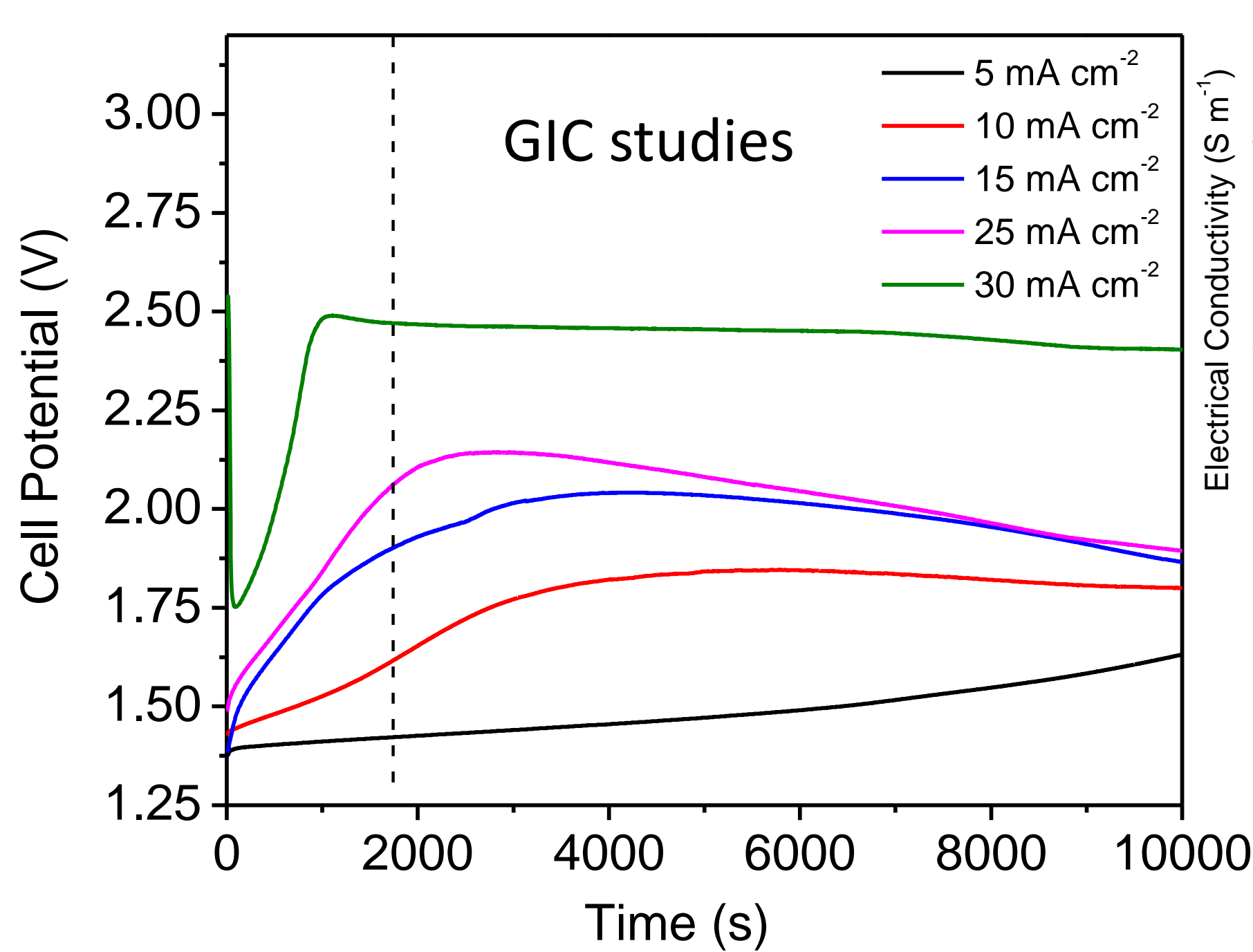## Electrochemical Preparation Process details

Objective:

- Producing less oxidized graphene flakes

- Potentially replace reduction process

- Scalable process

- How about Alkaline medium ?

Other Benefits:

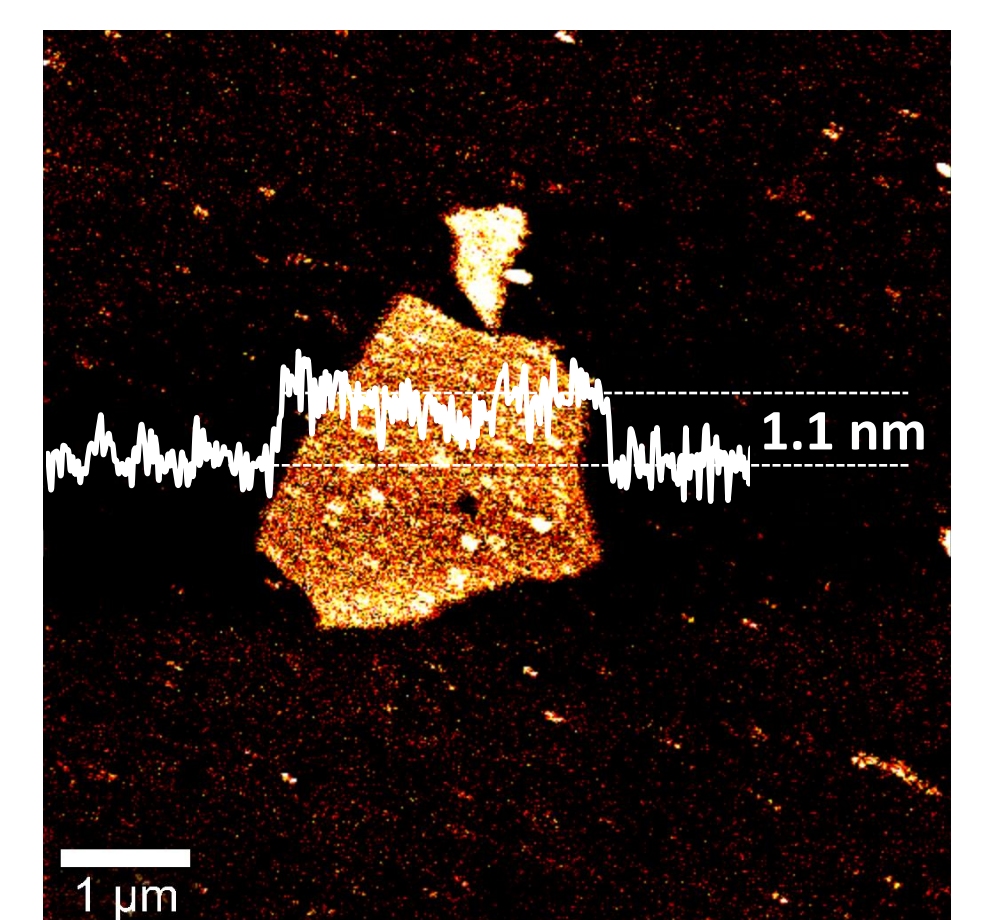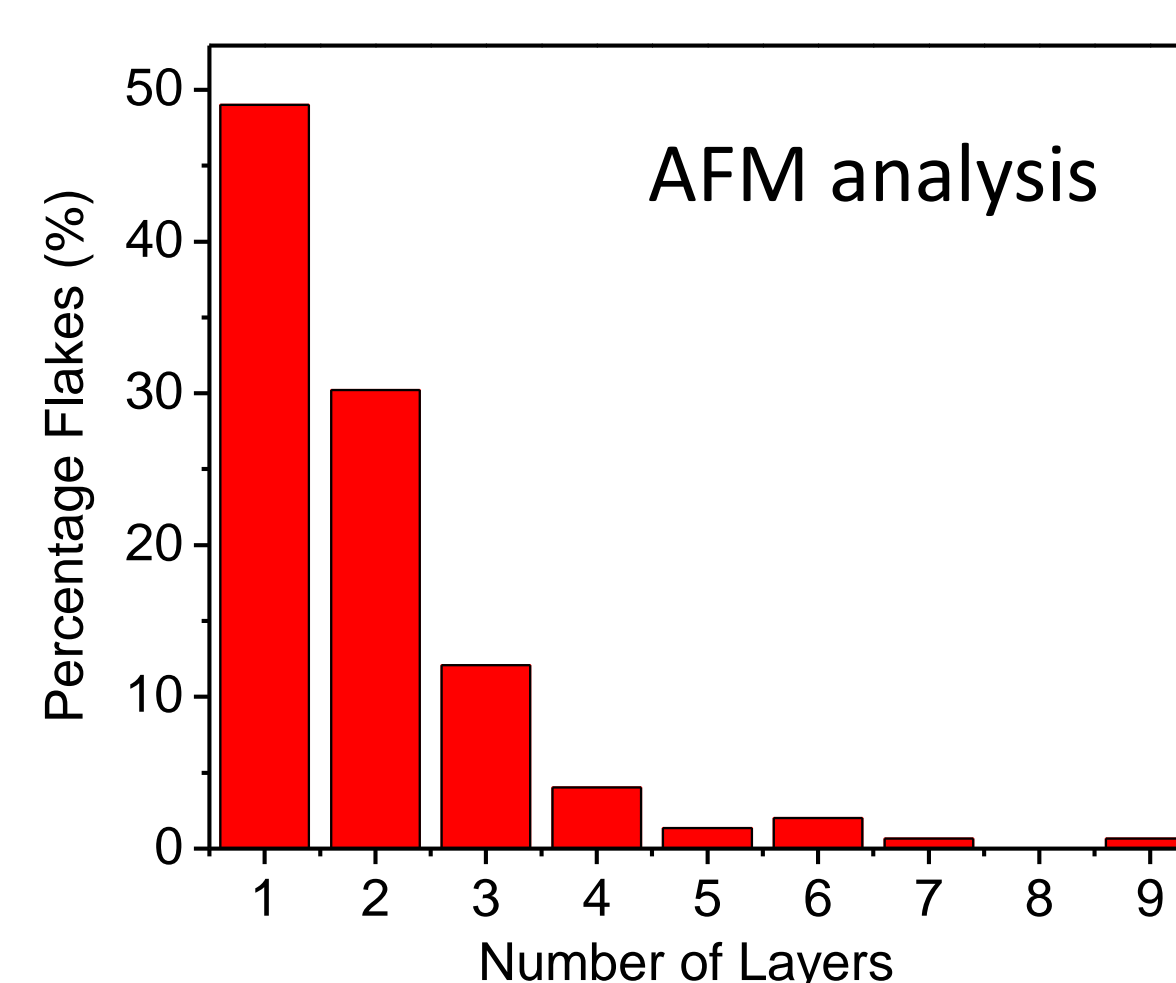- Tunable Graphene properties

Electrochemical Intercalation

OH^- ion

Exfoliation

Exfoliated Graphene Flakes

Material Characterization

GIC studies — Cell Potential (V) vs Time (s): 5 mA cm⁻², 10 mA cm⁻², 15 mA cm⁻², 25 mA cm⁻², 30 mA cm⁻²

Electrical Conductivity (S m⁻¹) — Reduction method: Thermal, Ascorbic Acid, Hydrazine, HI; This Method: EEG

Graphene materials	Characteristic absorbance peak
Graphene Oxide	230 nm
EEG (our method)	267 nm

Graphene materials	Slope factor (mL mg⁻¹ m⁻¹)
Graphene Oxide (Hummer's Method)	49*
EEG (This method)	1329

Absorbance vs Concentration (mg ml⁻¹)
Slope = 1329 mL mg⁻¹ m⁻¹
$R^2 = 0.99$
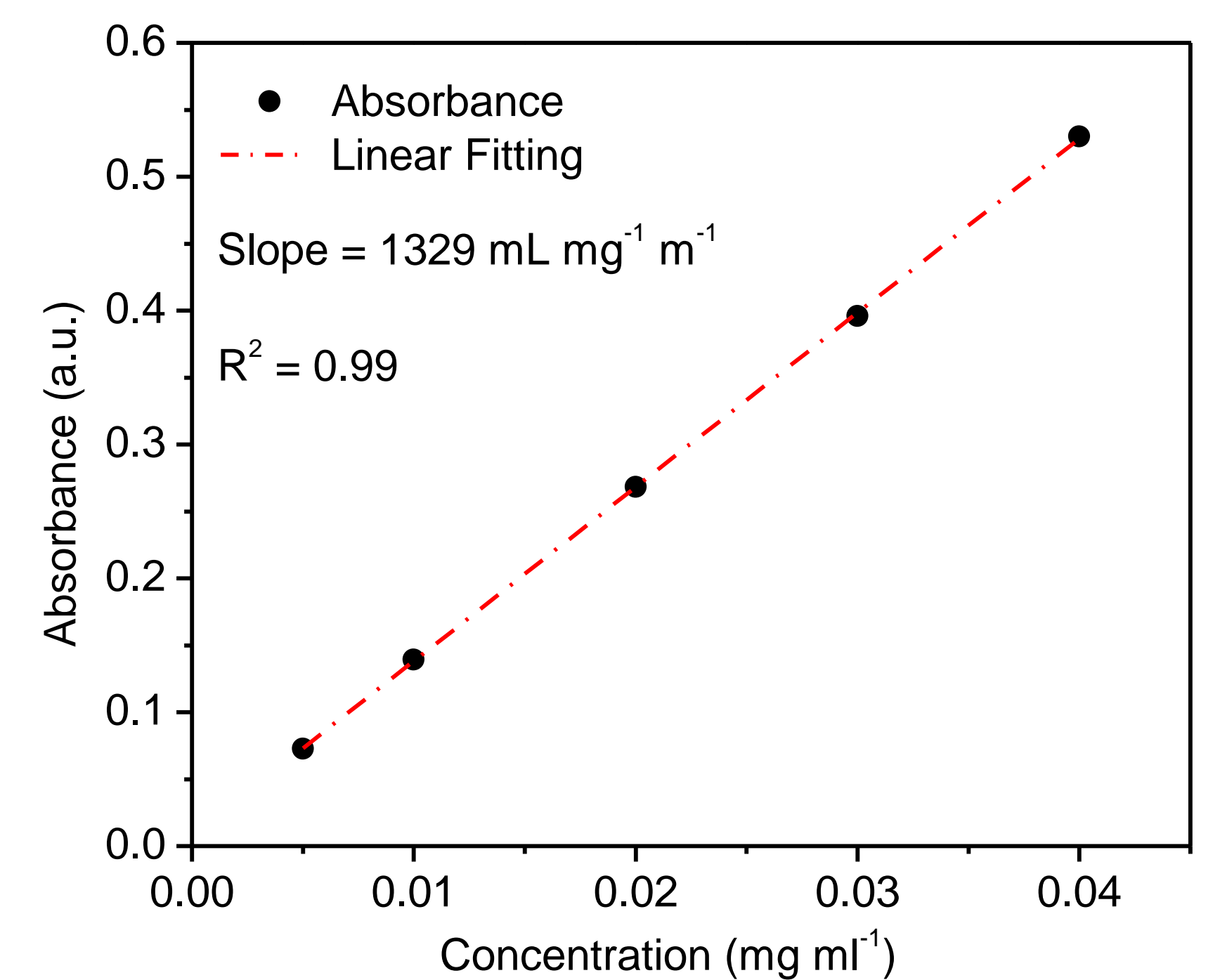
Elements detected (XPS)	Atomic Conc. (%)
Carbon	93.19
Nitrogen	0.53
Oxygen	6.28
C/O (EEG Our method)	14.8 (~15)
C/O (Graphene Oxide)	2 – 4*
C/O (reduced Graphene Oxide)	~ 30 *

Pyridinic N — Quaternary N — Pyridinic N oxides — Intensity (a.u.) vs Binding Energy (eV)

C-SO_x-C x = 2,3,4 — Intensity (a.u.) vs Binding Energy (eV)

10 µm

2 µm   0.5 µm   0.5 µm   0.5 µm

0-110   1-210

AFM analysis — Percentage Flakes (%) vs Number of Layers

1.1 nm

1 µm
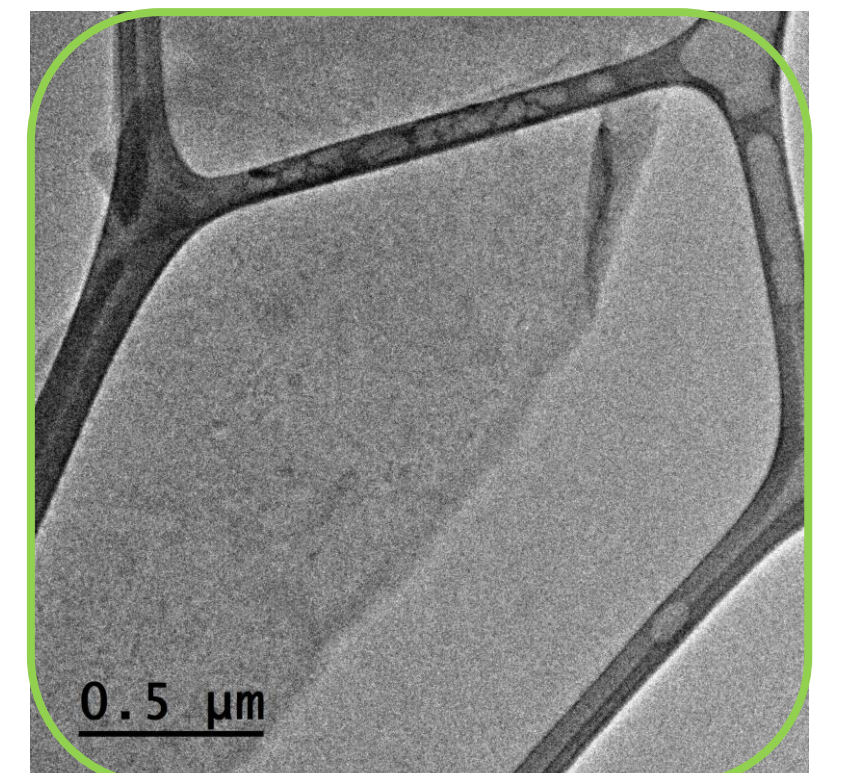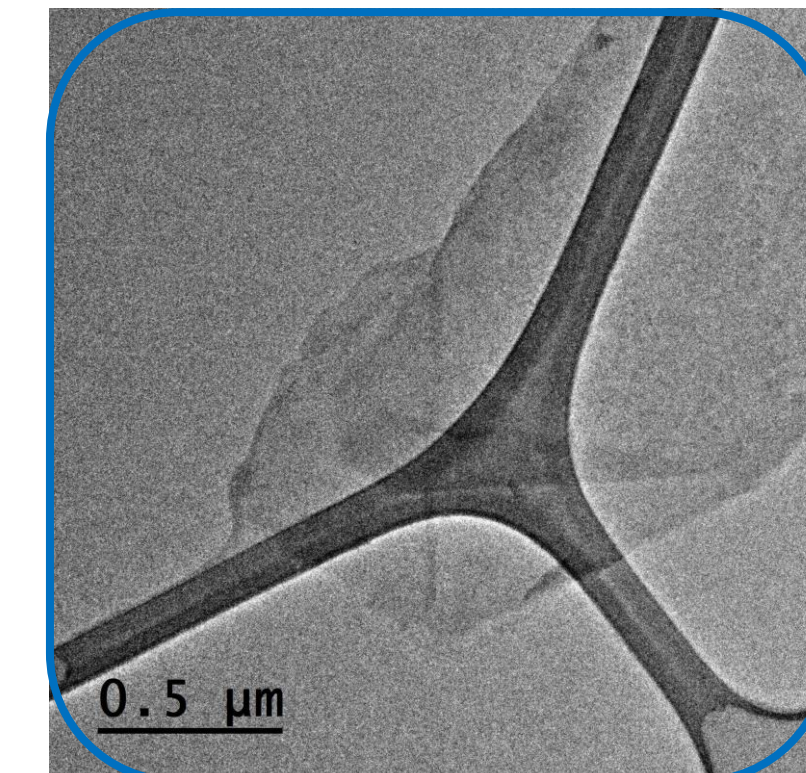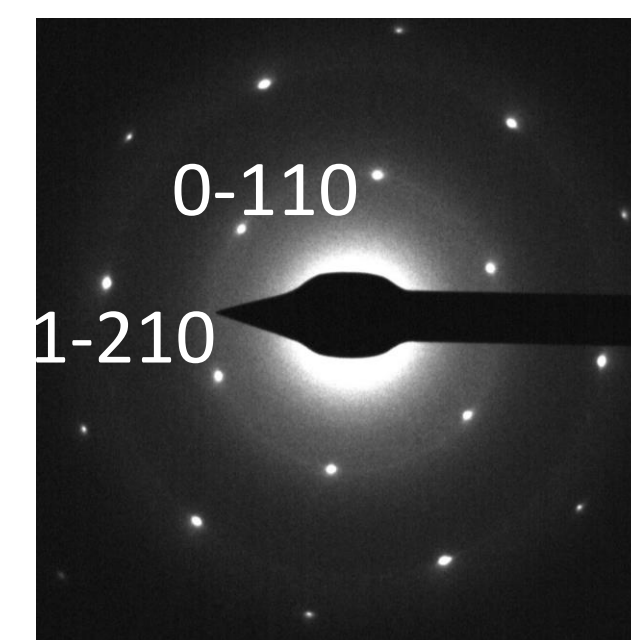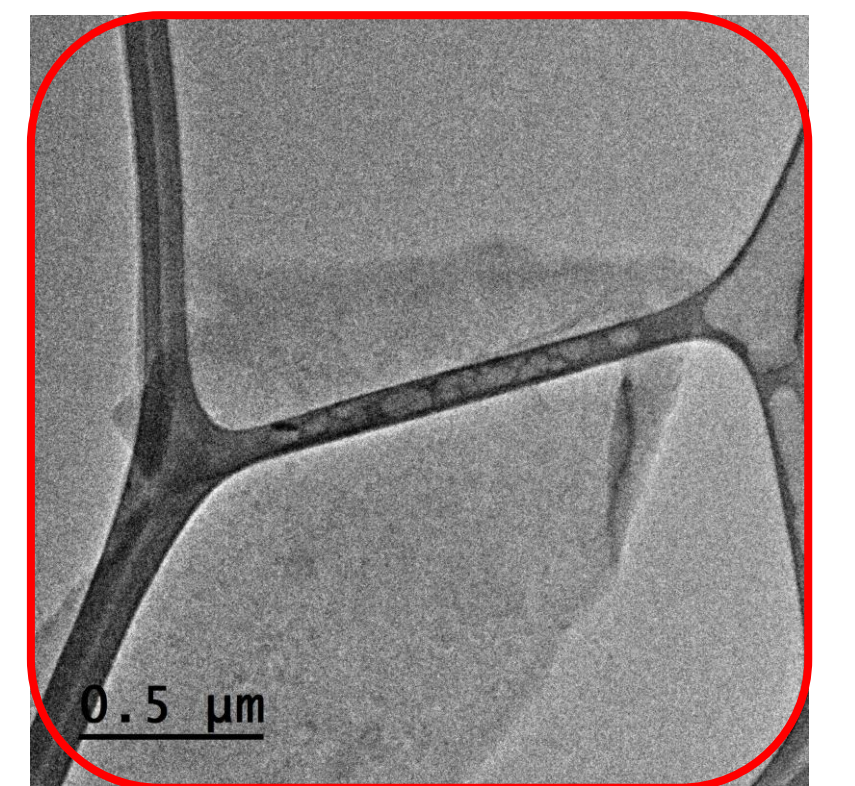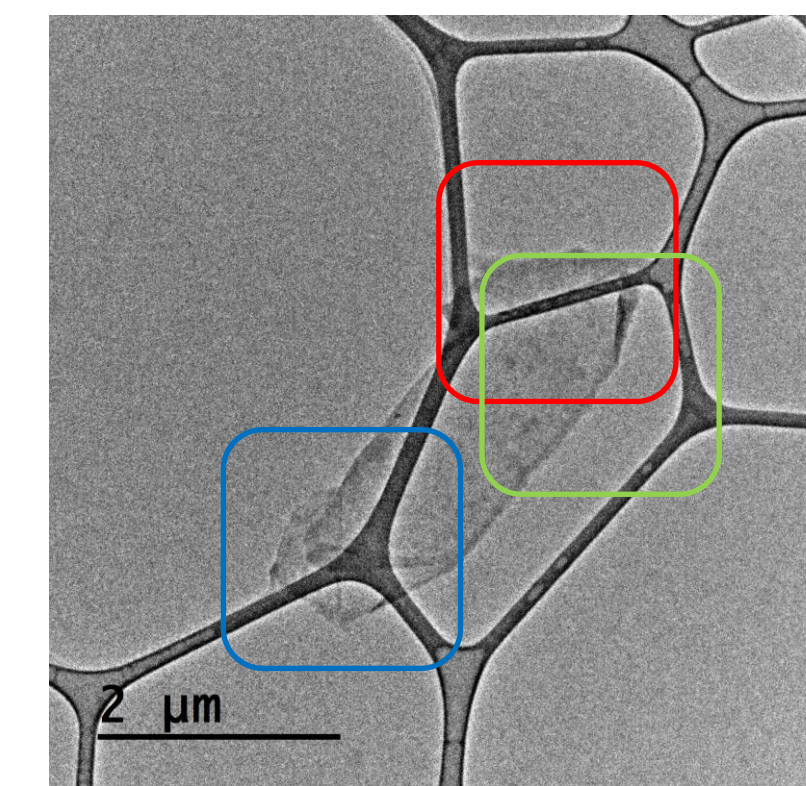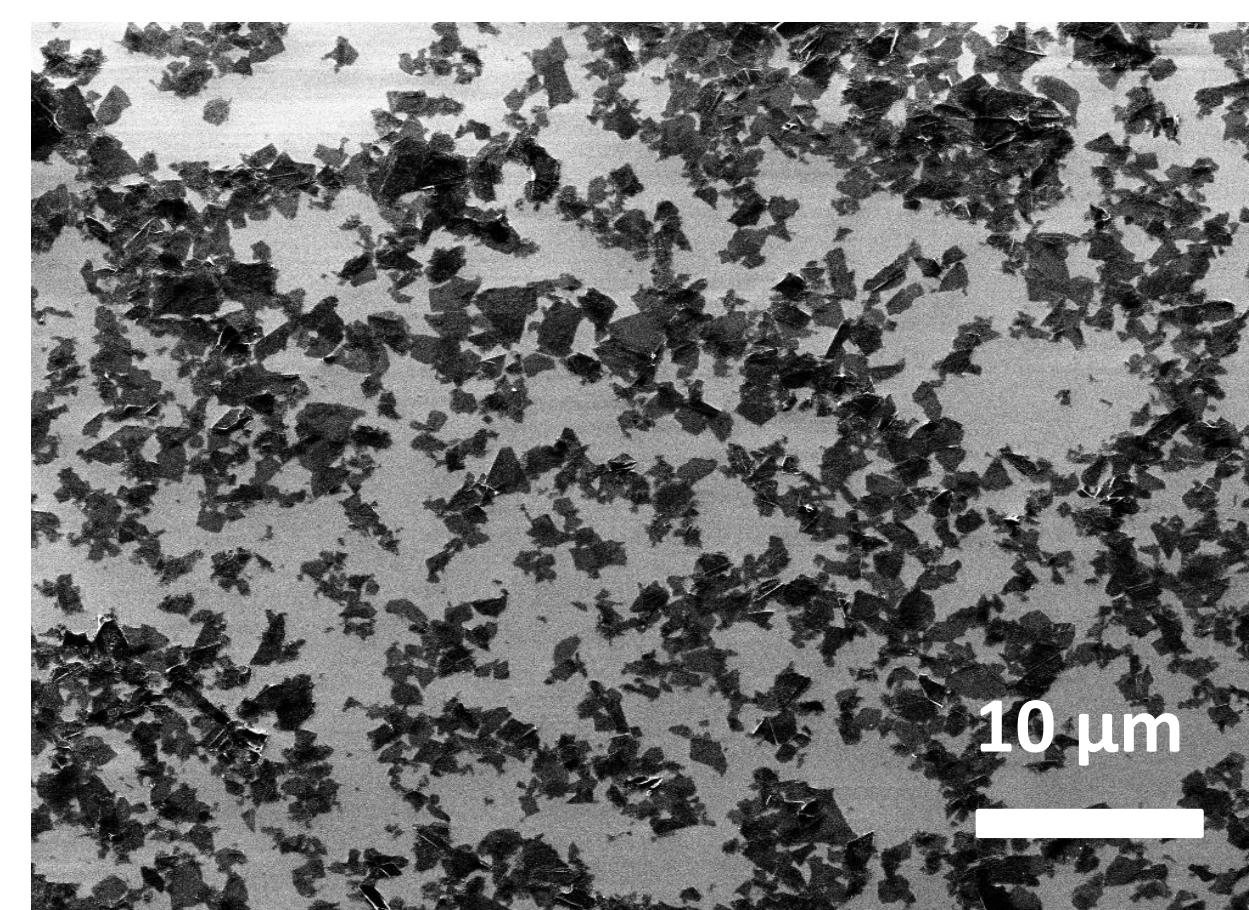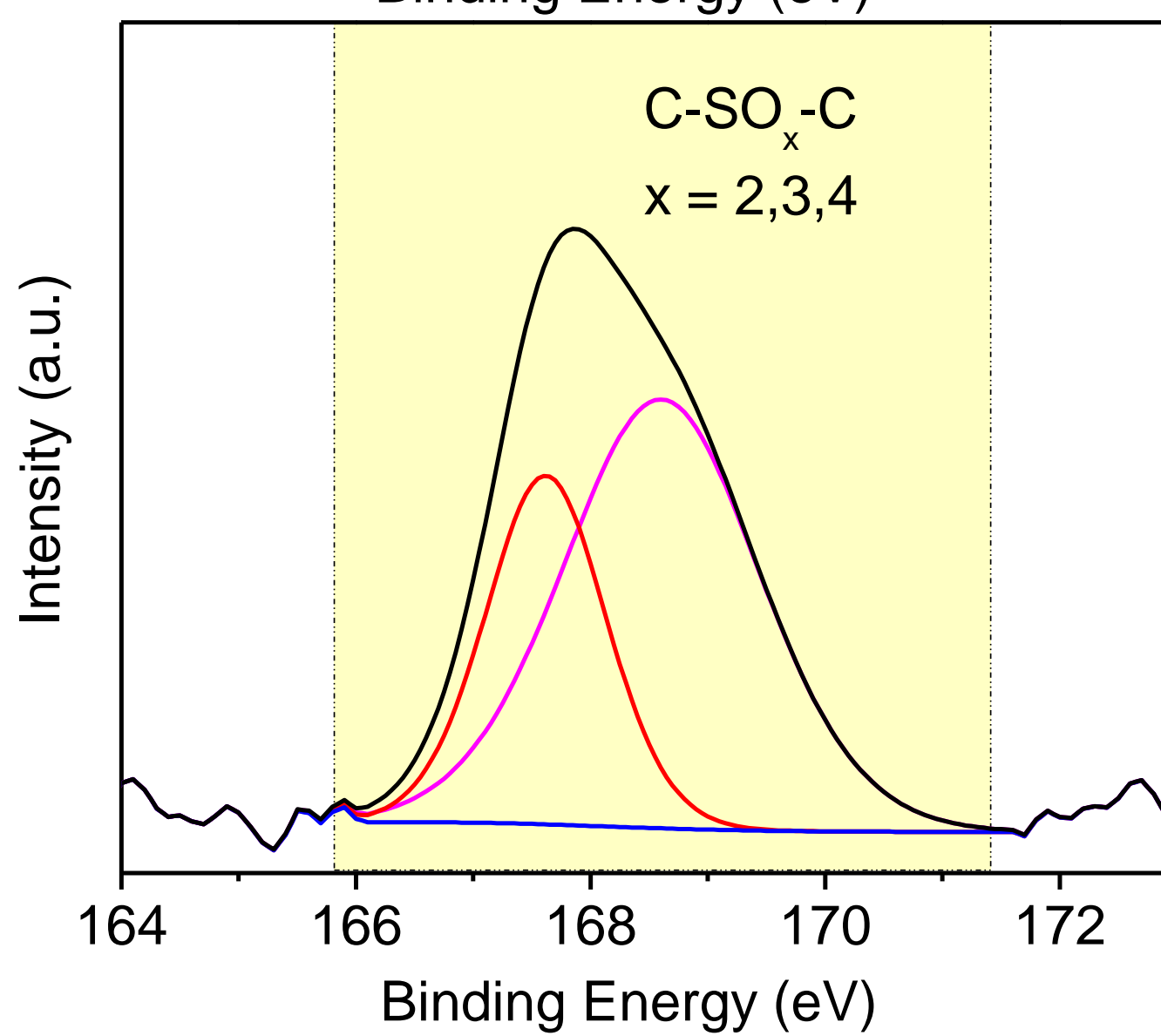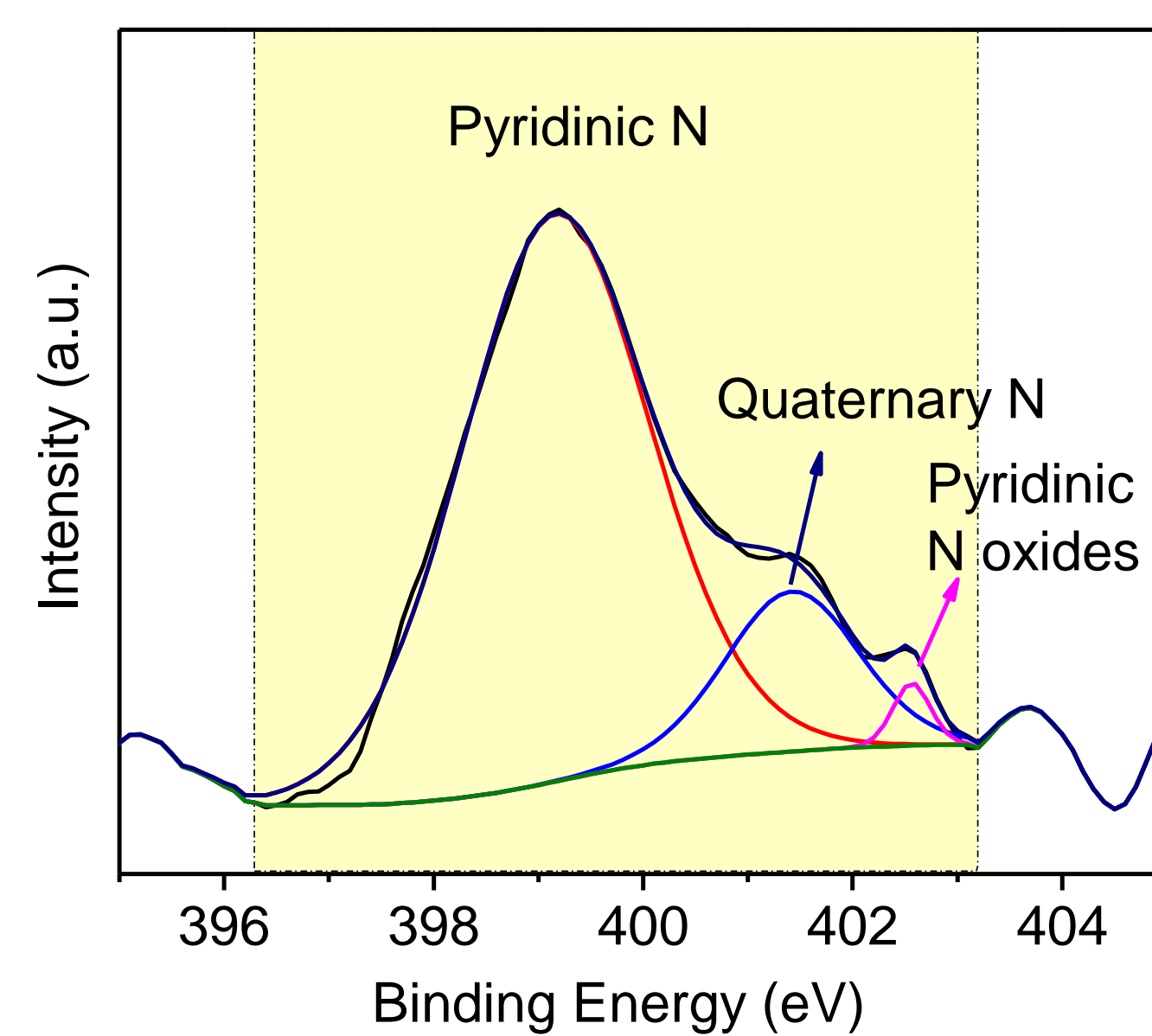
Conclusion

- **More reduced graphene produced**

- **N and S doping**

- **Less disrupted π structure**

- **92% flakes – less than 4 layers**

References

J. Am. Chem. Soc. 2017, 139, 48, 17446-17456
Singh et. al. (coming soon)

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