Electronic Supplementary Material (ESI) for Nanoscale.


# Electrochemical exfoliation of graphite in quaternary ammonium-based deep eutectic solvents: A route for mass production of graphane

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## SEM:

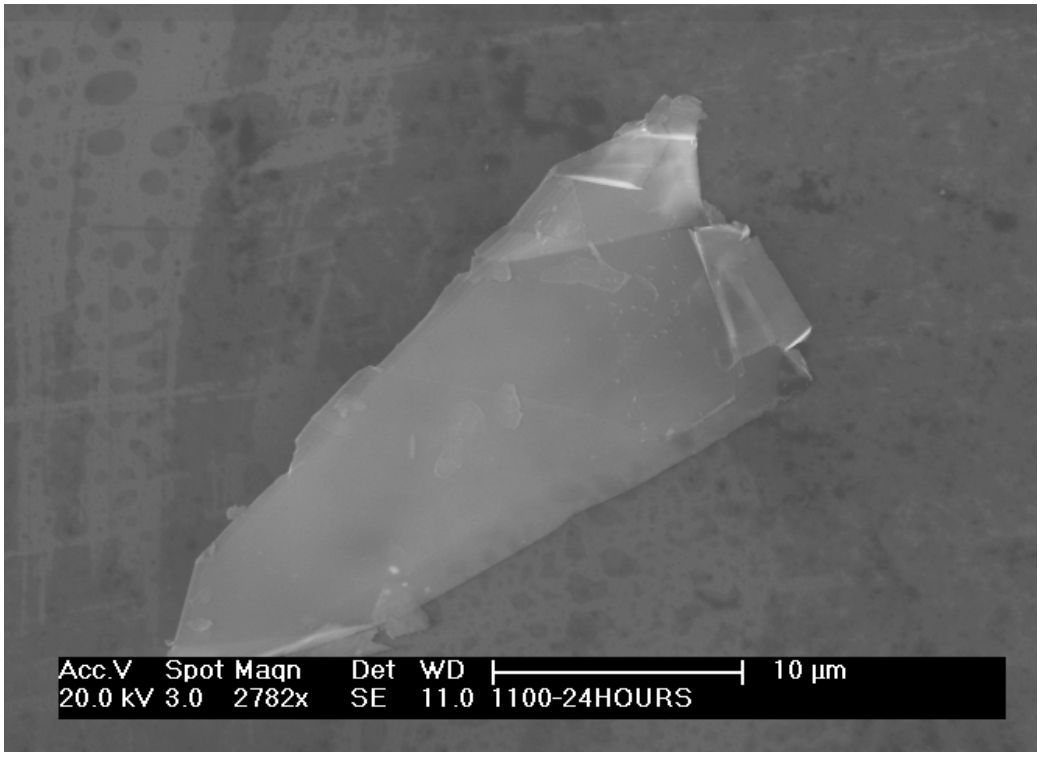



Figure S1: SEM image of the exfoliated graphene after 2 hours of electrolysis

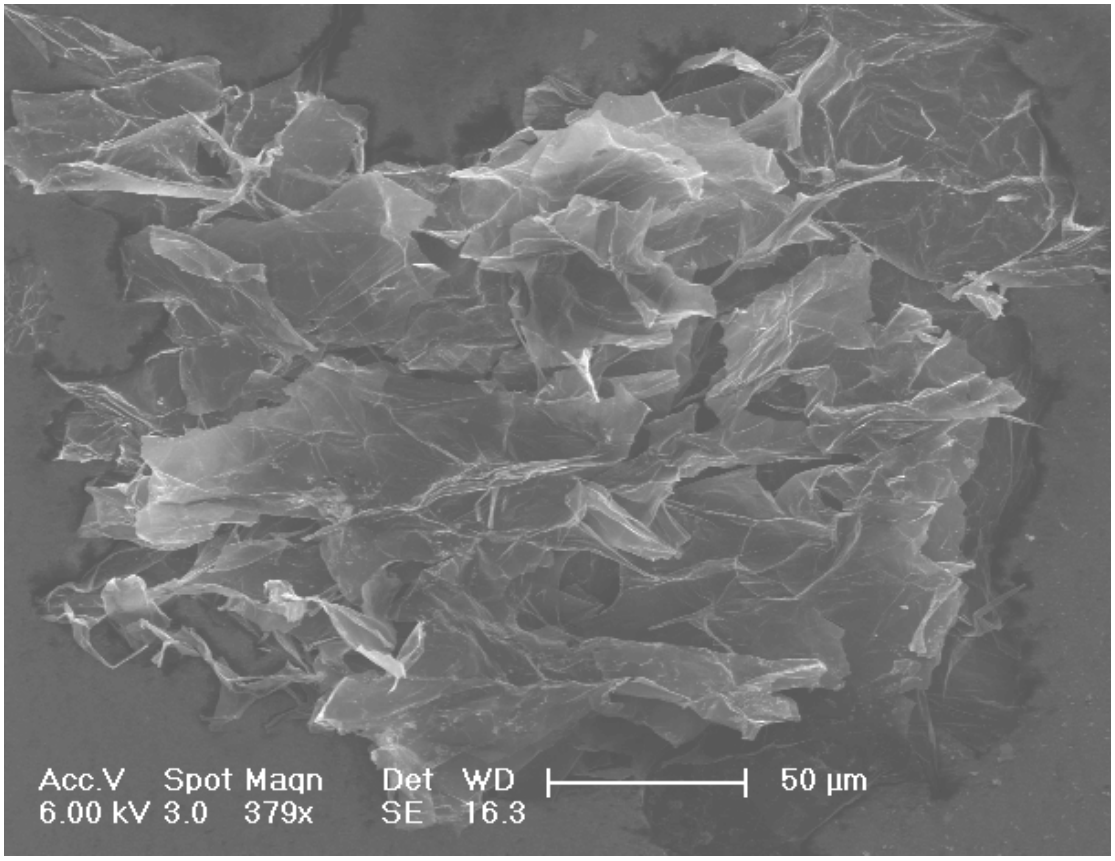



Figure S2: after 6 hours of electrolysis the flakes tend to agglomerate together in a porous structure

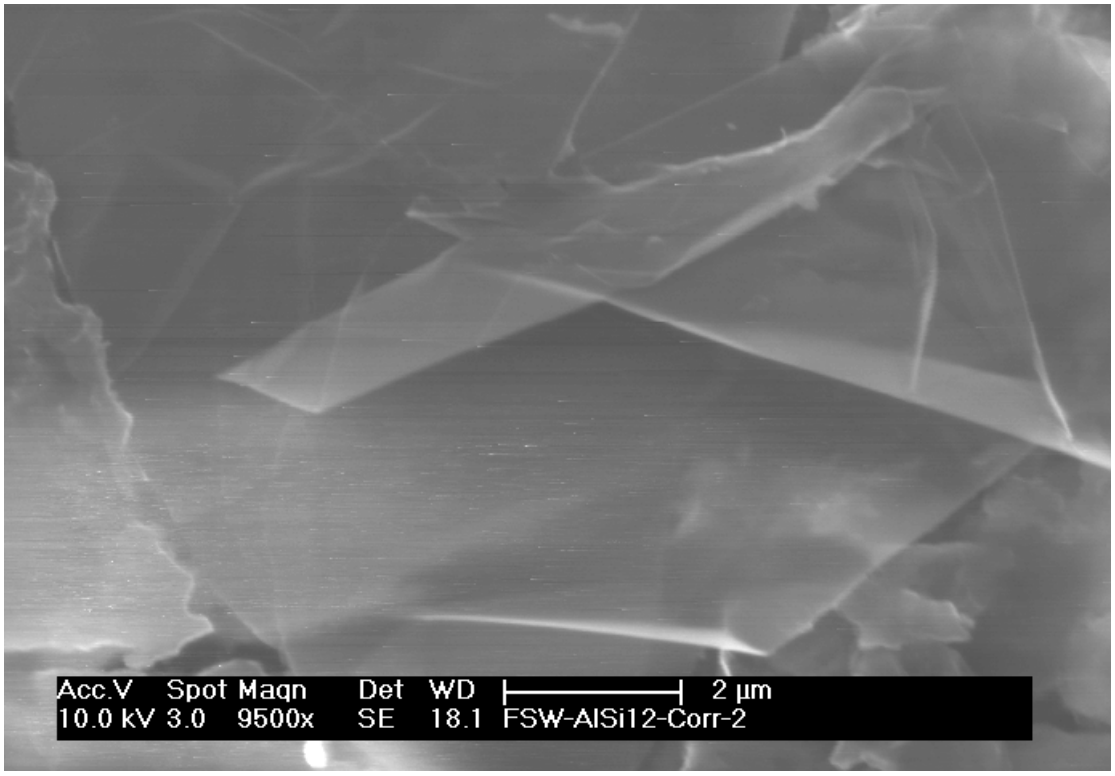
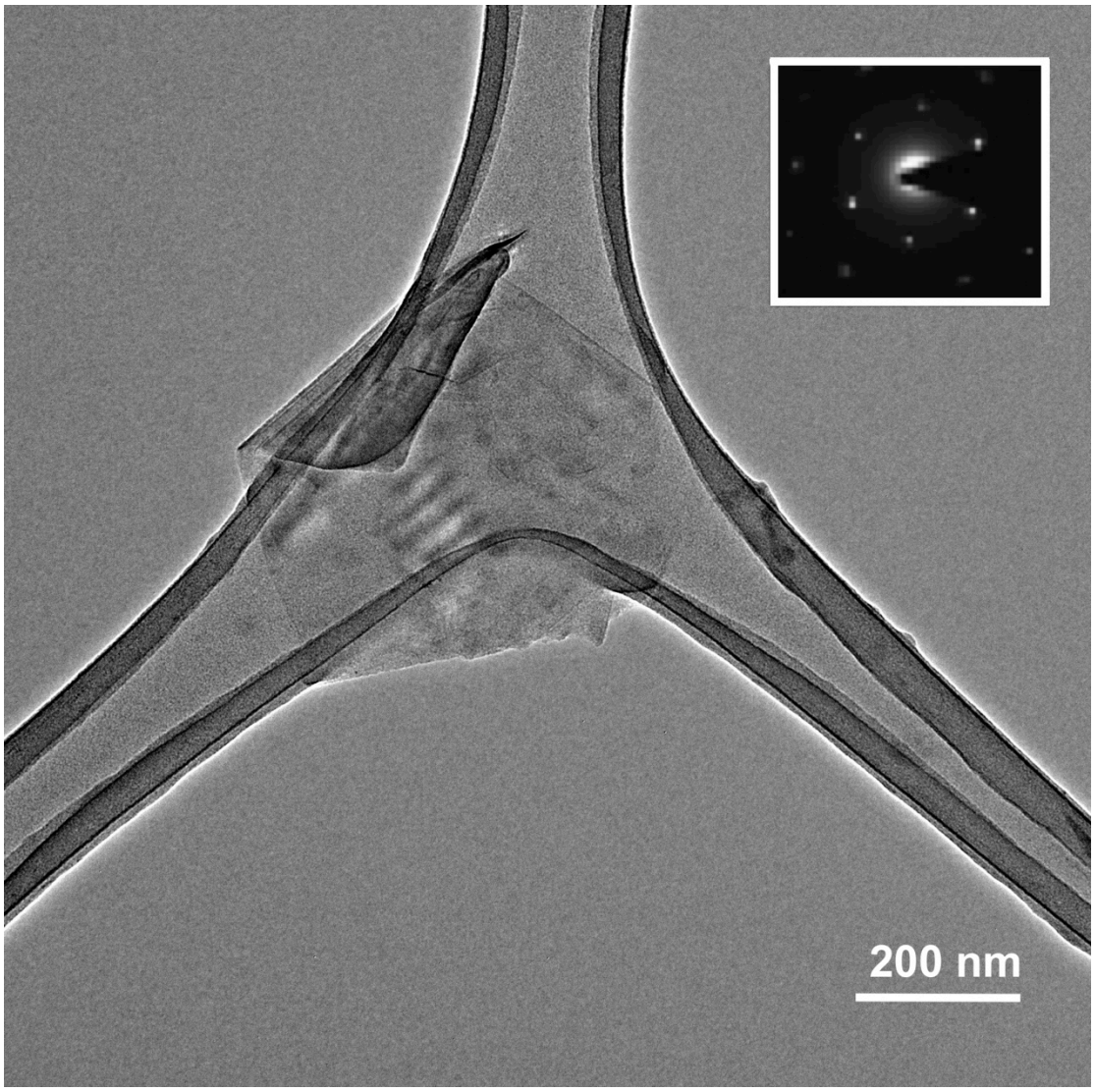



Figure S3: a paper made of the graphene after 6 hours of electrolysis. Note that the sample start to get charged due to the hydrogenation. It was really difficult to take SEM images of the samples that was hydrogenated for longer time.

## TEM:



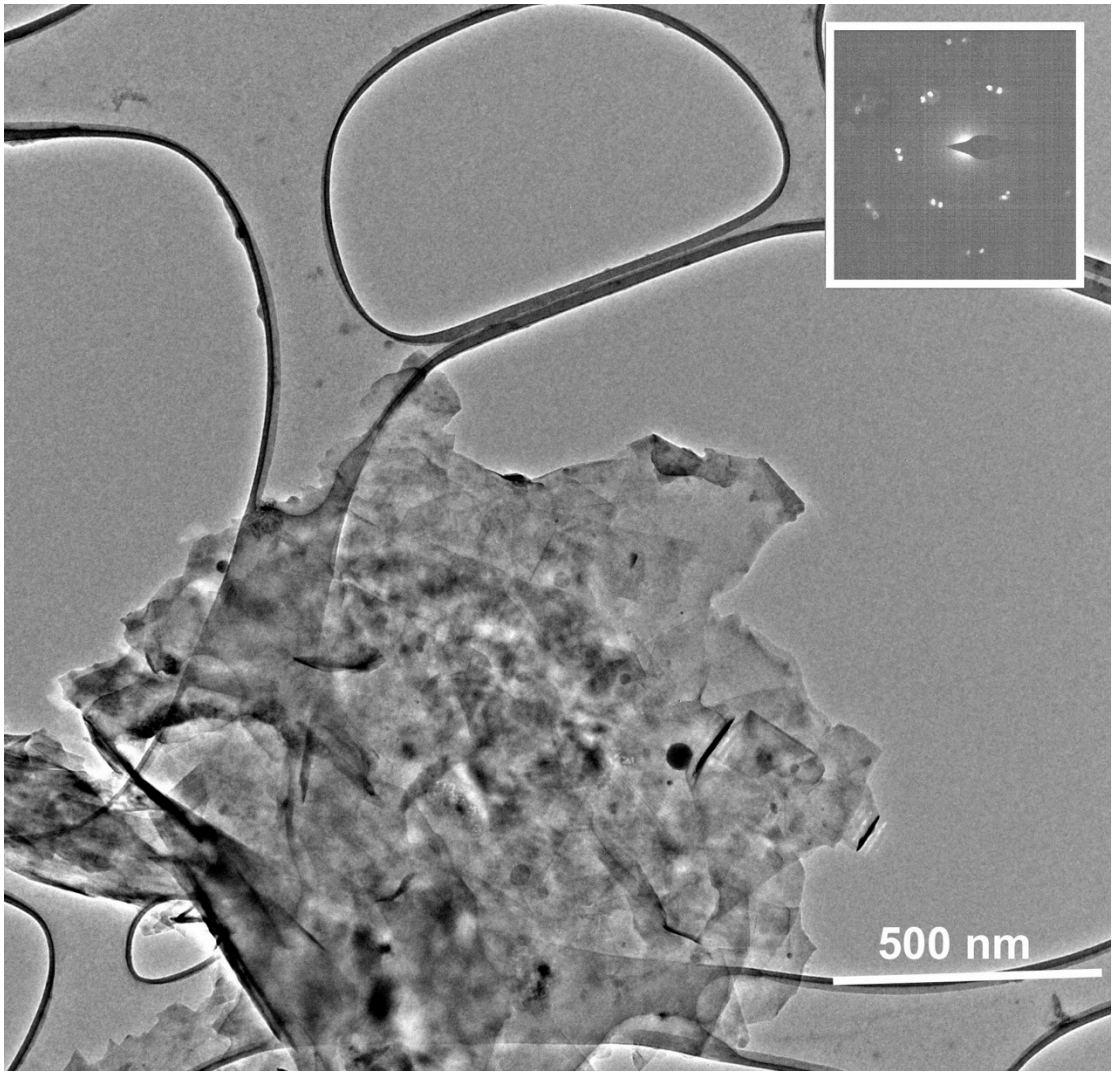
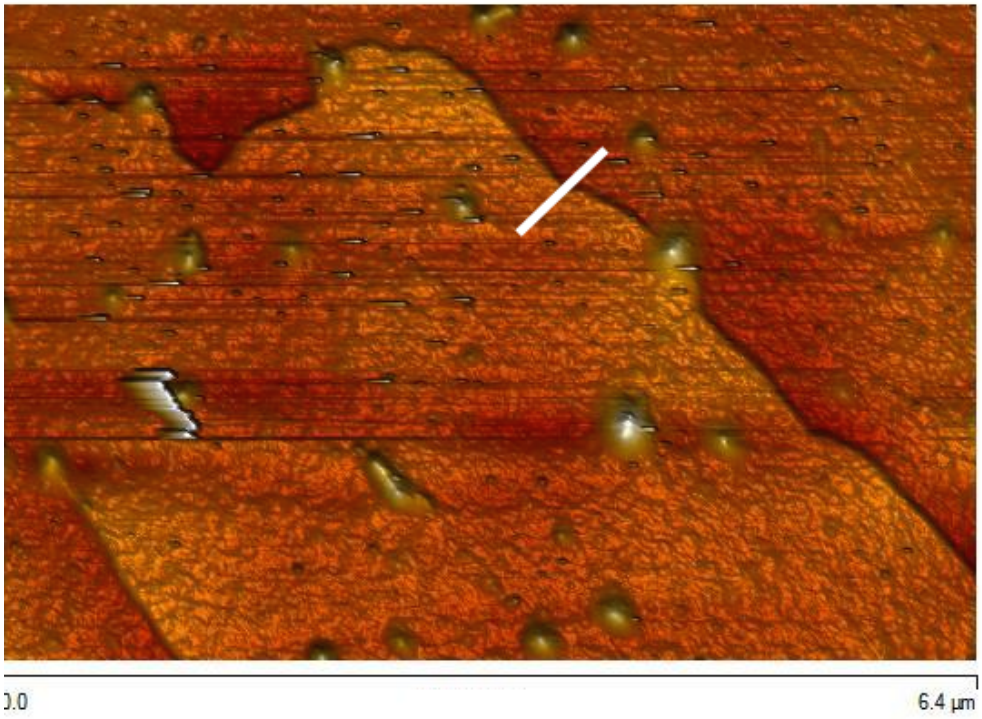



Figure S4: TEM images with the exfoliated flakes with the SAED pattern as inset; (top) the samples after 12 hours of electrolysis, electron diffraction pattern of the sheet showing the crystalline hexagonal arrangement. The relative intensity of the spots (I(1 1 0 0)/I(2 1 1 0) > 1) indicates that the exfoliation process could produce flakes as thin as single layer graphene. (Bottom) presents a few micron graphene particles produced after 3 hours of exfoliation. The presence of different diffraction spots confirms that the material is formed by more than one layer rotated to the other



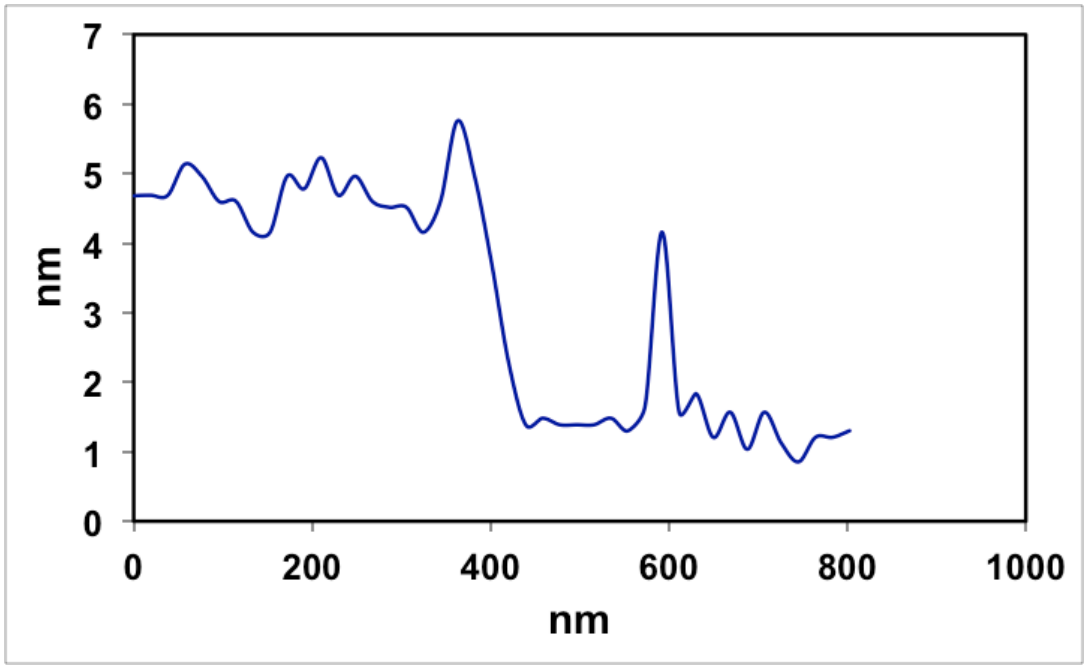




Figure S5: AFM image and height profile across the line giving clear evidence of the exfoliation to multilayer graphene after 3 hours of the electrolysis. In average, the thickness of the flakes gets thinner by increasing the electrolysis time as can be concluded from comparing Figure S5 and Figure 7 in the manuscript.

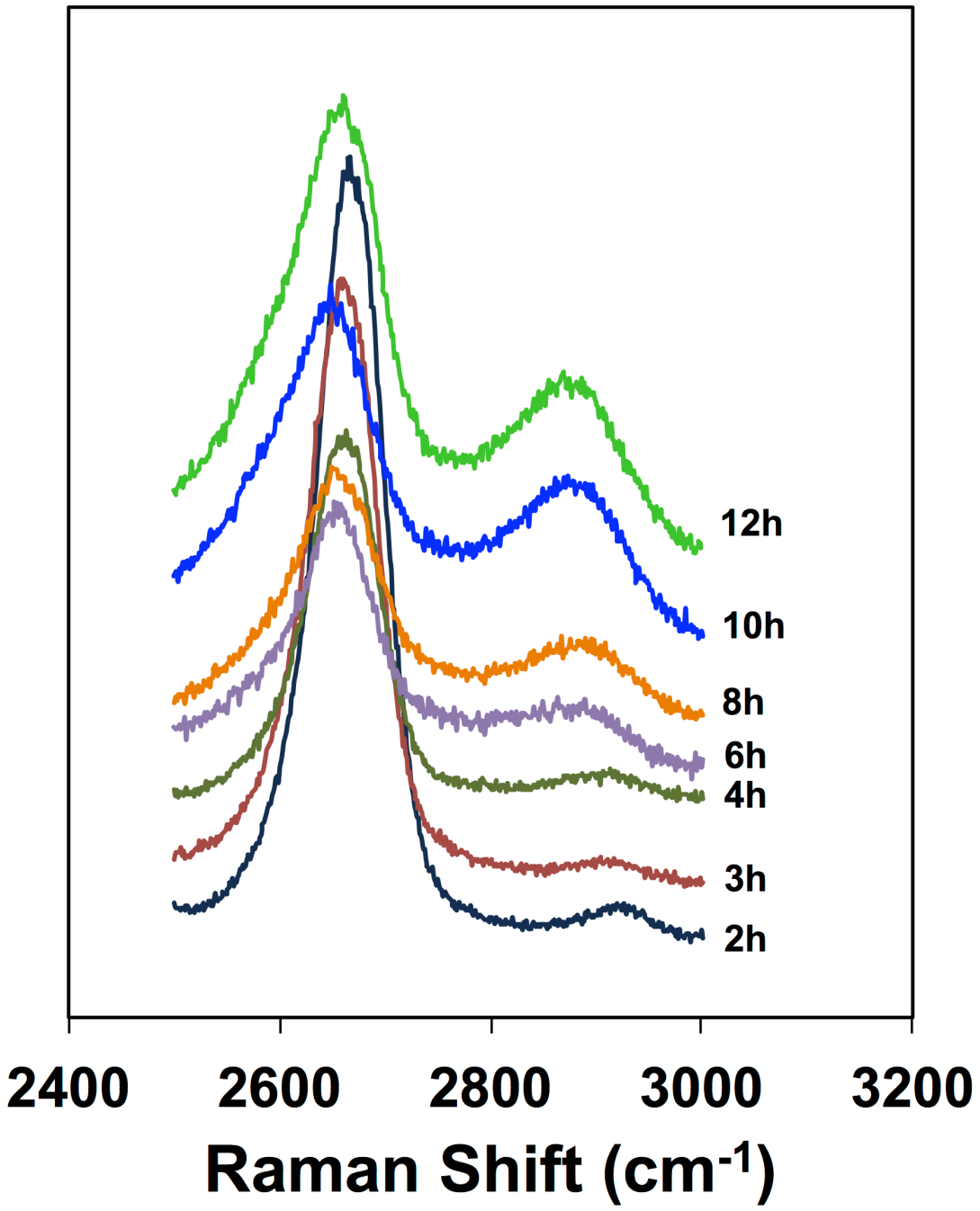



Figure S6: Raman spectra recorded using the 633 nm laser, showing the development of the G+D band as a function of the process duration.

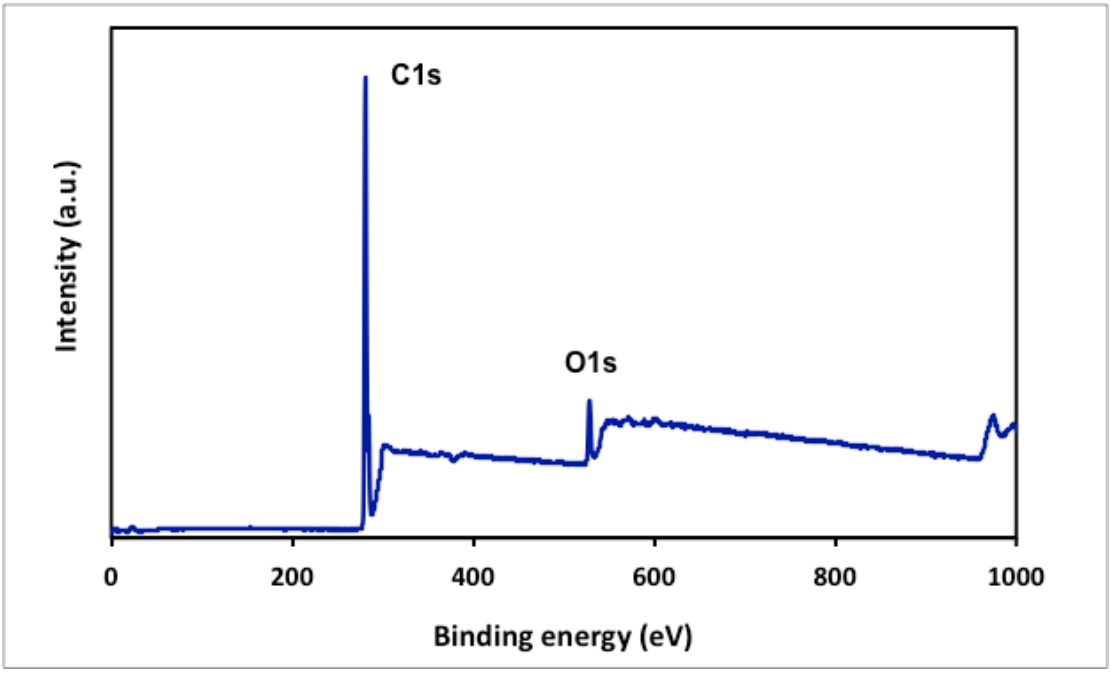



Figure S7: Wide scan XPS for the sample hydrogenated for 12 hours at 2.5 V in urea-choline chloride eutectic mixture. Showing almost no oxygen content.

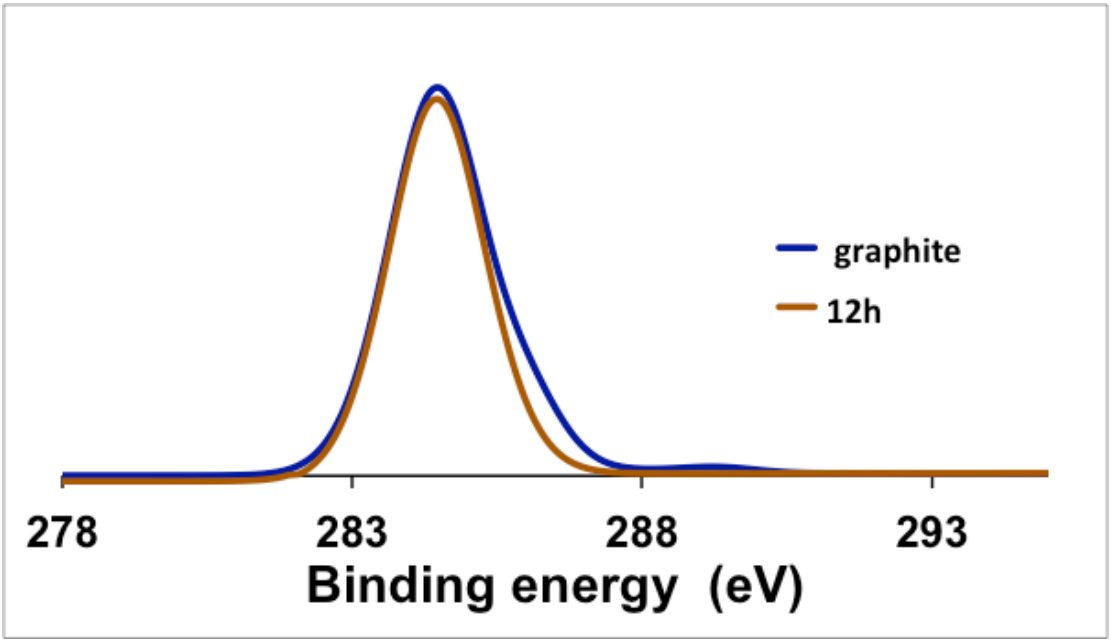



Figure S8: High-resolution scans of the C 1s position for the produced graphane and the initial raw materials showing almost identical curves.