

## Supplementary Information for

### Continuous flow photolytic reduction of graphene oxide

Thaar M. D. Alharbi, <sup>a,b</sup> Amira R. M. Alghamdi, <sup>a</sup> Kasturi Vimalanathan, <sup>a</sup> and Colin L. Raston <sup>\*a</sup>

<sup>a</sup> Flinders Institute for NanoScale Science and Technology, College of Science and Engineering, Flinders University, Adelaide  
SA 5001, Australia email: [colin.raston@flinders.edu.au](mailto:colin.raston@flinders.edu.au)

<sup>b</sup> Physics Department, Faculty of Science, Taibah University, Almadinah Almunawwarrah, Saudi Arabia

\*Corresponding authors:

Tel: +61 8 82017958 E-mail address: [Colin.raston@Flinders.edu.au](mailto:Colin.raston@Flinders.edu.au) (Colin Raston)

## **Experimental section:**

### **Chemicals and materials**

Graphene oxide (GO) (average sheet size:  $\sim 5 \mu\text{m}$  in cross section) were synthesized by a modified Hummer's method and purchased from Carbon Solution. Deionized (DI) water (Millipore Milli-Q grade) was used in all experiments.

### **Preparation of rGO**

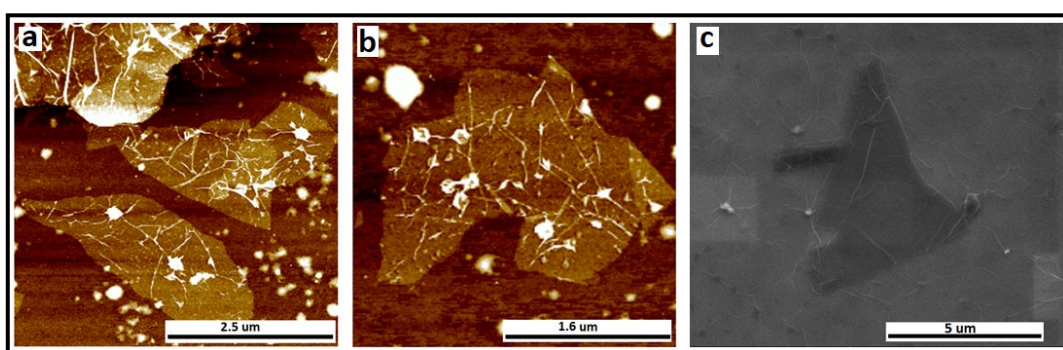
The as-received GO was dispersed in water at  $0.2 \text{ mg. mL}^{-1}$  concentration and sonicated for 30 min to afford a yellow stable dispersion. For VFD processing using the confined mode of operation of the device, 2 mL of the aqueous solution of GO was placed in the VFD quartz glass tube (20 mm OD, 17.5 mm ID diameter and 18.5 cm in length) which was spun at 45° tilt angle while irradiated with UV lamps for different reaction times, 15, 30, 60 min. A flow rate of 0.2 mL/min was used under. Optimal parameters for preparing r-GO formation were 4k rpm rotational speed of the tube, using two UV lamps each operating at 10 W with a wavelength of 254 nm, (Lamp Type – GPH212T5) and 0.2 mg/mL concentration of GO in water.

### **Characterization**

Scanning electron microscopy (SEM) performed using a FEI Quanta 450 High Resolution Field Emission SEM, with a voltage of 10 kV, and working distance of 10 mm, Atomic force microscopy (AFM) (Nanoscope 8.10 tapping mode), Transmission electron microscopy (TEM) was conducted on a TECNAI 20 microscope operated at 120 and 200 kV. The UV-vis absorption spectra were performed on a (Varian Cary 50) in the 200–1000 nm range in a quartz cell. Raman measurements were recorded at an excitation wavelength of 532 nm (5mW) at room temperature. X-ray powder diffraction (XRD) data were collected using a Bruker Advanced D8 diffractometer (capillary stage) using Co - K $\alpha$  radiation ( $\lambda = 1.7889 \text{ \AA}$ ) Samples for SEM and Raman analysis were prepared on clean silicon wafers. The thermogravimetric analysis (TGA) was recorded on a Perkin Elmerat operating at a heating rate of  $5^\circ\text{C/min}$  under a nitrogen gas flow. FTIR microscope (Nicolet iN10MX IR Microscope, Thermo Scientific). X-ray photoelectron spectroscopy (XPS) was applied using Mg K $\alpha$  radiation to determine the elemental composition and the chemical states of all components. Under the condition chosen, XPS has a probing depth of less than 10 nm. The apparatus was built by SPECS (Berlin, Germany) with a nonmonochromatic X-ray source. High-resolution scans were

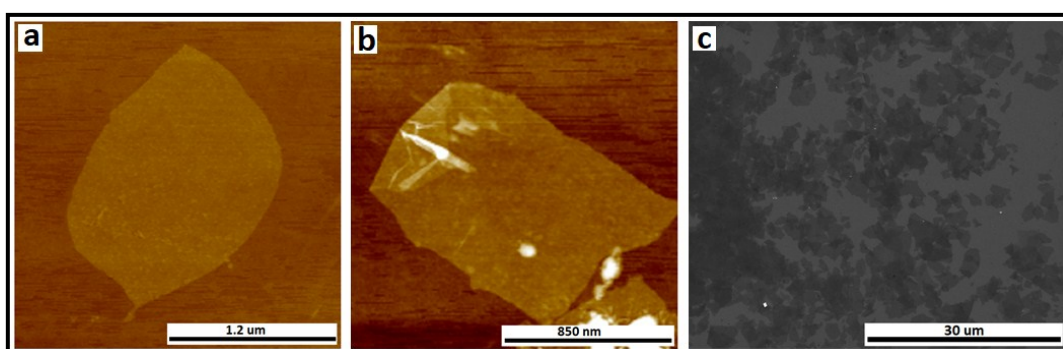
recorded for carbon (C), oxygen (O), at a pass energy of 10 eV. Survey scans were carried out over 1200-0 eV binding energy range with 1.0 eV steps and a dwell time of 100 ms. Narrow high-resolution scans were run with 0.05 eV steps and 250 ms dwell time. Base pressure in the analysis chamber was  $1.0 \times 10^{-9}$  torr and during sample analysis  $1.0 \times 10^{-8}$  torr.

**1. Additional AFM & SEM images of GO (as received) before processing in the VFD**



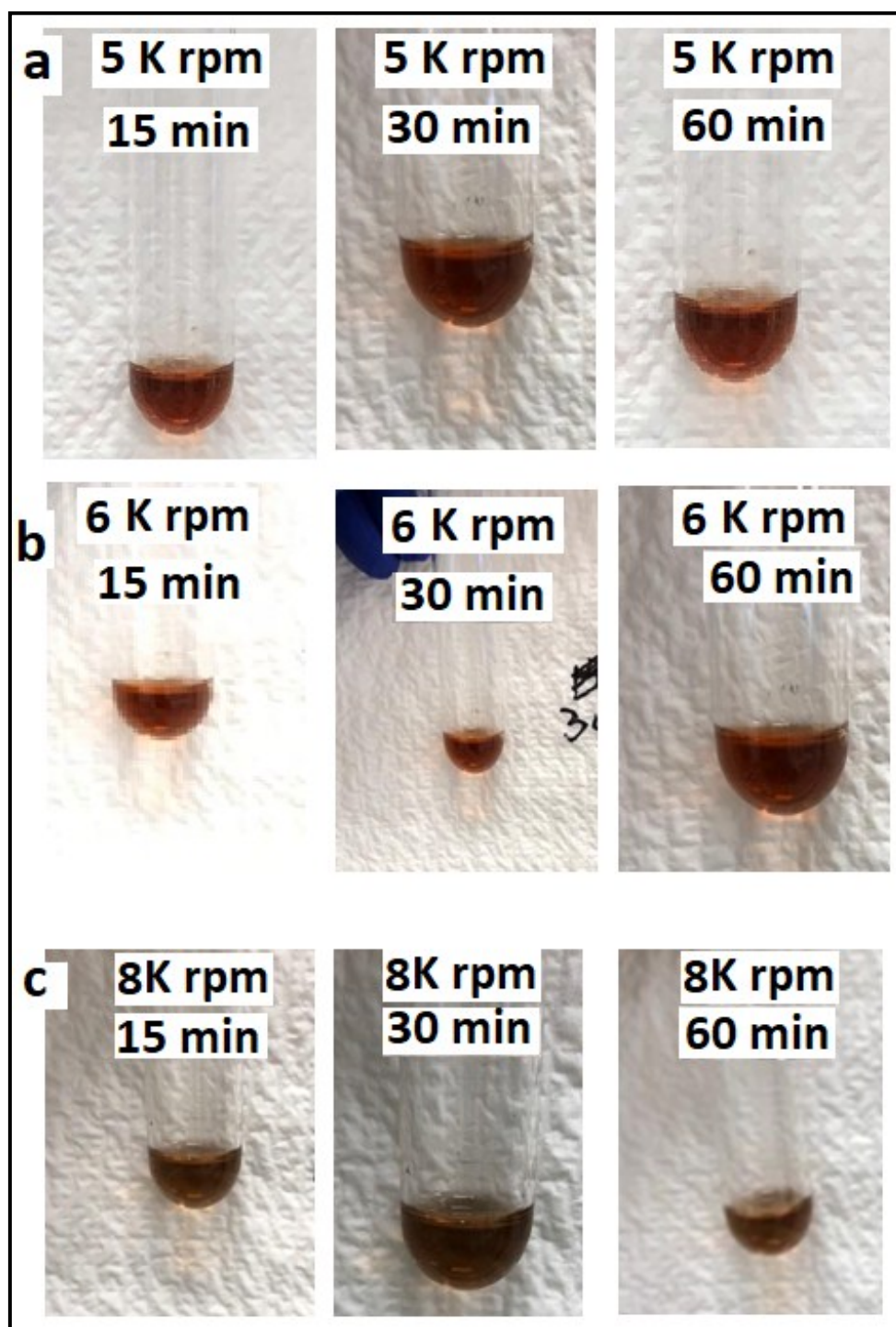
**Figure S1.** AFM and SEM images of GO sheets as received and before processing in the VFD.

**2. Additional AFM & SEM images of rGO after processing in the VFD**



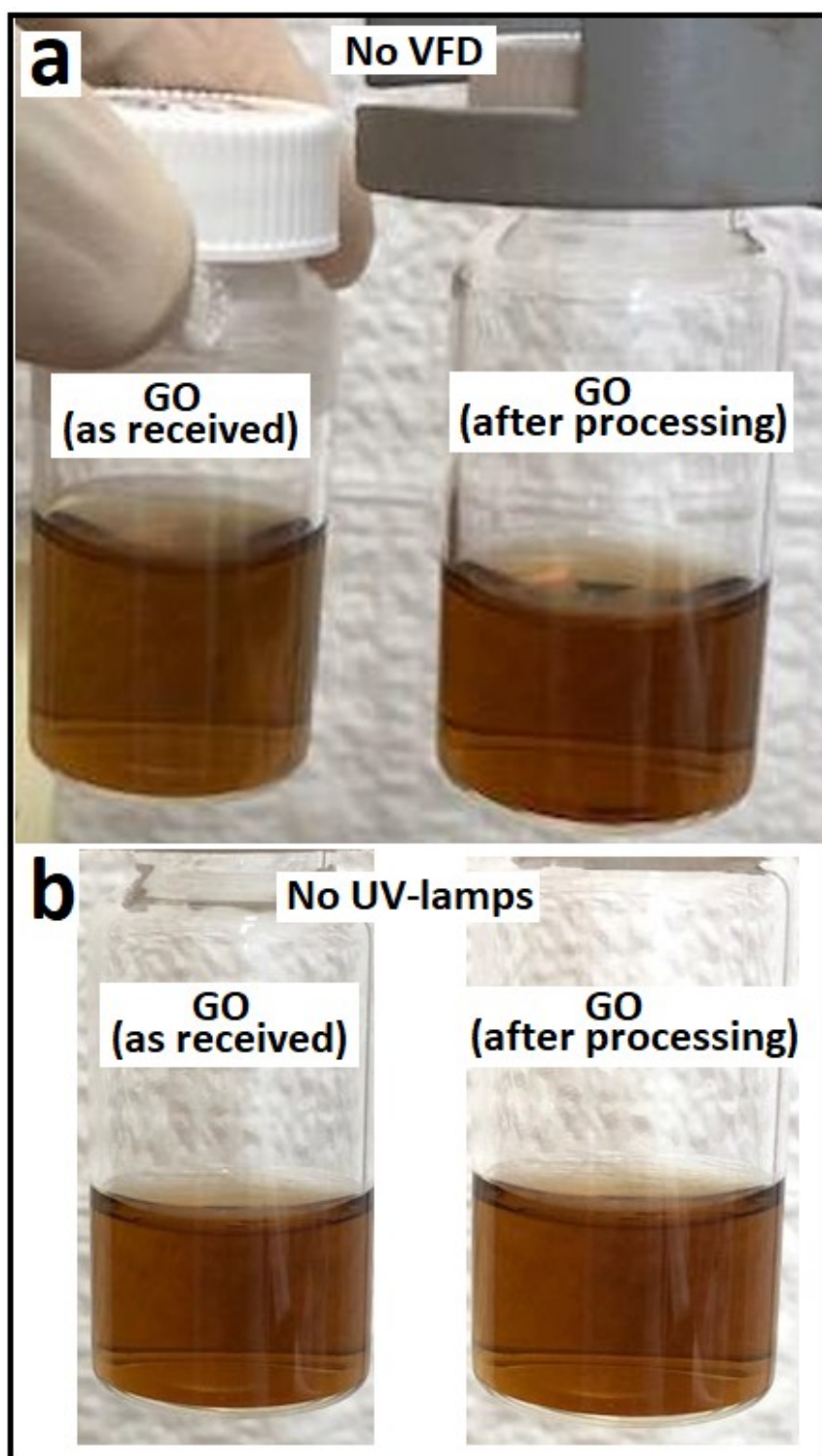
**Figure S2.** AFM and SEM images of GO after processing in the VFD at 4k rpm rotational speed, under continuous flow mode, tilt angle 45° and a flow rate of 0.2 mL/min, coupled with UV light irradiation at  $\lambda = 254$  nm at 20 W.

### 3. Varying the rotational speed



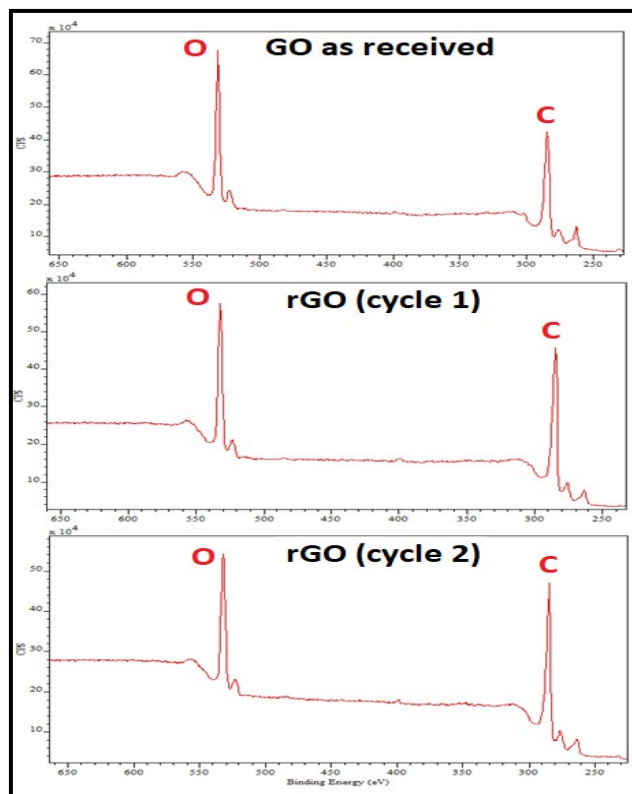
**Figure S3.** Photographs of GO after processing in the VFD at varies speeds, 5.6 and 8k rpm rotational speed, under confined mode at different reaction times (15. 30 and 60 min), tilt angle 45°, coupled with UV light irradiation at  $\lambda = 254$  nm at 20 W.

#### 4. Control experiments



**Figure S4.** Photographs of control experiments of (a) GO after processing in the absence of VFD and using UV light irradiation at  $\lambda = 254$  nm at 20 W. (b) GO after processing in the VFD (at 4k rpm, a reaction time of 60 min, tilt angle  $45^\circ$ ) and in the absence of UV light irradiation at  $\lambda = 254$  nm at 20 W. Note that the colour of the solutions are unchanged.

5. The survey spectra of samples processed under continuous flow mode.



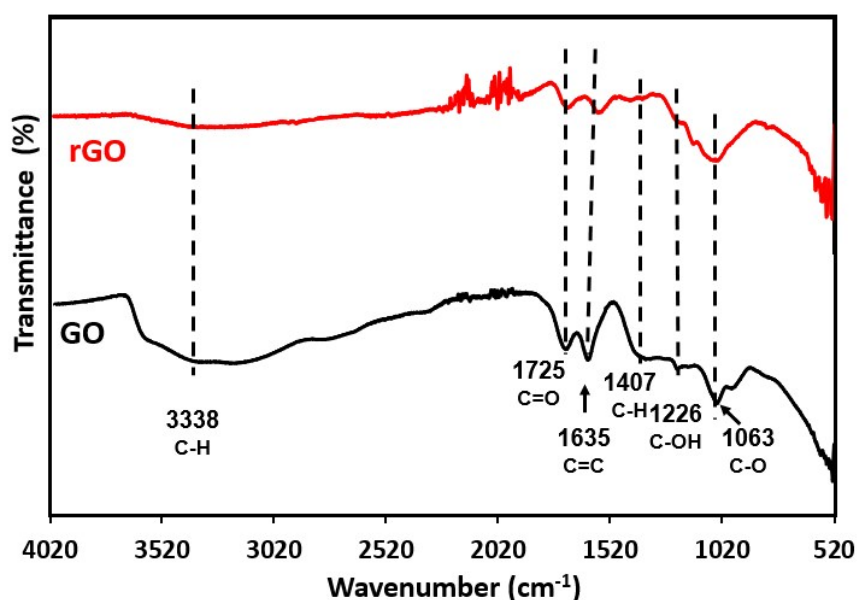
**Figure S5.** Survey XPS spectra of GO (as received) and after VFD processing (cycles 1 and 2) at 4k rpm rotational speed, tilt angle 45° and a flow rate of 0.2 mL/min, coupled with UV light irradiation at  $\lambda = 254$  nm at 20 W.

**Table S1.** XPS analysis of the C1s content for different GO (as received) and after processing in the VFD under UV irradiation at different reactions times, 15, 30 and 60 min.

Sample	C-C (%)	C-O (%)	C=O (%)	COOH (%)
GO (as rec.)	50.50	45.28	4.2	-
rGO (15 min)	65.46	15.19	9.24	6.42
rGO (30 min)	68.14	14.46	7.55	5.99
rGO (60 min)	72.08	13.83	5.33	5.42

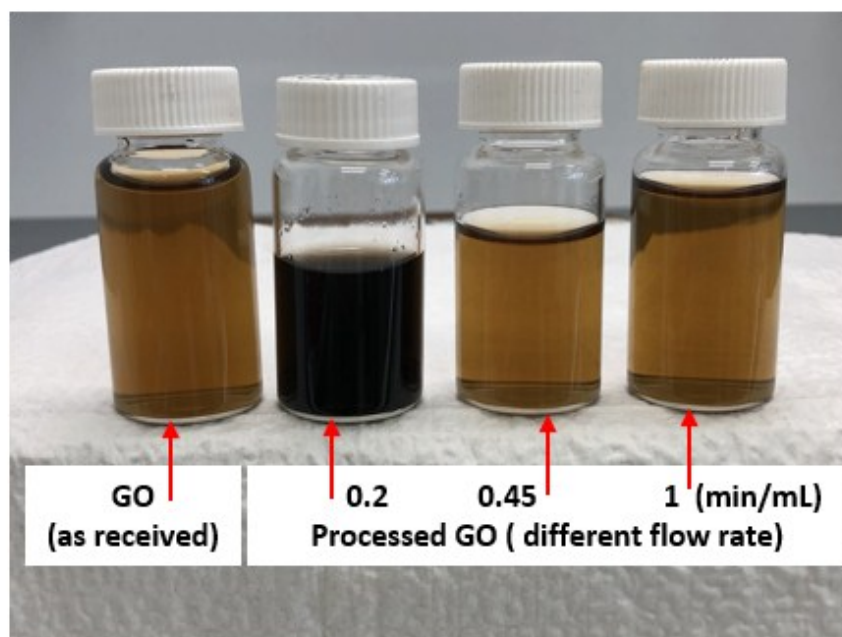
**Table S2.** . XPS analysis of the C1s content of the surfaces of different GO (as received) and after processing in the VFD while UV irradiated at 4k rpm, under continuous flow mode, tilt angle 45° and flow rate of 0.2 mL/min

Sample	C-C (%)	C-O (%)	C=O (%)	COOH (%)
GO (as rec.)	50.50	45.28	4.2	-
rGO (Cycle.1)	64.21	16.24	9.29	5.82
rGO (Cycle.2)	68.14	14.89	7.13	4.91

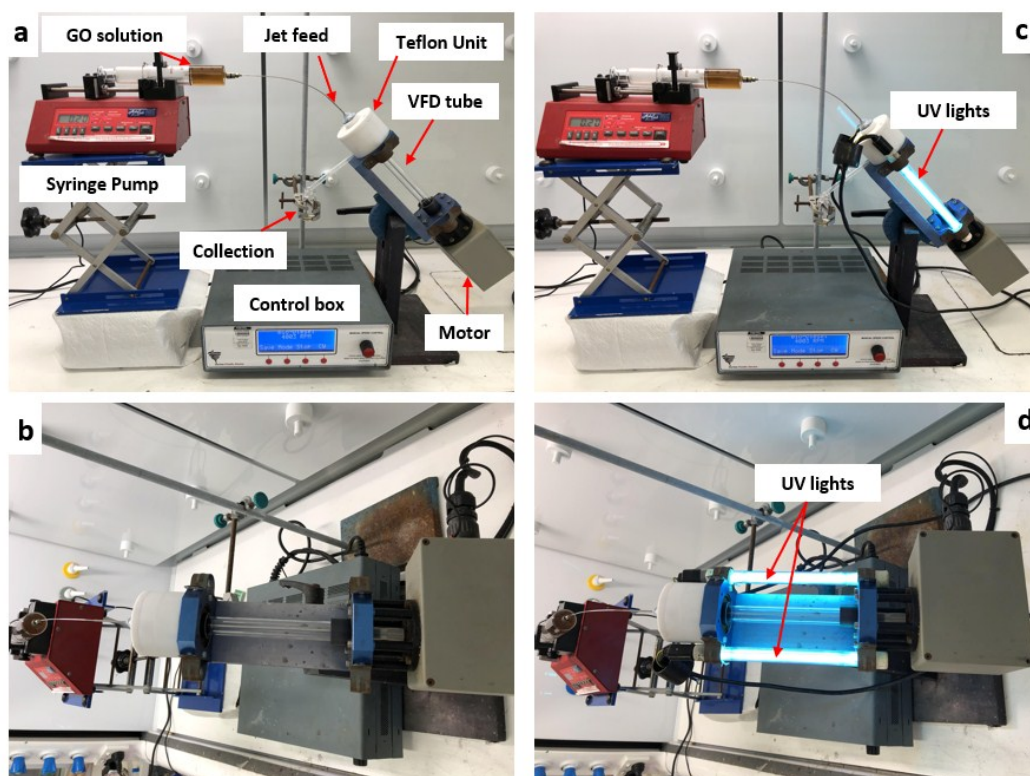


**Figure S6.** (a) FTIR for GO and reduced GO (rGO), after processing in the VFD at 4k rpm rotational speed, tilt angle 45°, under continuous flow flow rate 0.2 mL/min, coupled with UV irradiation  $\lambda = 254$  nm, 20W.

In order to further confirm the reduction process, fourier transform infrared (FTIR) spectra were recorded, as shown in the Fig. 7. Such FTIR is used to investigate the oxygen-containing functional groups of GO before and after the reduction with significant differences in the spectra between GO, and rGO. In the case of GO (prior to the reduction), different oxygen-containing functional groups can be clearly identified, with the broad peak at 3338  $\text{cm}^{-1}$  corresponding to the stretching vibration of COOH and water<sup>1</sup>. The weak peak at 1730  $\text{cm}^{-1}$  corresponds to the stretching vibrations of C=O in the carboxyl and carbonyl groups, with other peaks at 1226 and 1063  $\text{cm}^{-1}$ , corresponding to the stretching vibration of C–OH and C–O, respectively.<sup>2</sup> A peak at 1635  $\text{cm}^{-1}$  corresponds to C=C stretching.<sup>3</sup>



**Figure S7.** Photographs of a) GO (as received), after processing in the VFD at 4k rpm rotational speed, tilt angle 45°. under continuous flow, coupled with UV irradiation  $\lambda = 254$  nm, 20W, at flow rate of 0.2, 0.45 and 1 mL/min respectively. From the photographs, 0.2 mL/min is the only flow rate that the reduction process happened.



**Figure S8.** Photographs of Vortex Fluidic Device (VFD) in the absence of UV- Lights, a) side view, b) top view, the VFD in the present of UV- Lights c) side view and d) top view.

## References:

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