Electronic Supplementary Material (ESI) for New Journal of Chemistry.


# Micropores within N, S co-doped mesoporous 3D graphene-aerogel enhances supercapacitive performance

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# Supporting information

# List of Content



**S1: Synthesis**

**S1.1: Synthesis of GO nanoflex:**

GO nanoflex has synthesized by the improved hummer method, a 9:1 mixture of concentrated $H_2SO_4$/$H_3PO_4$ (60:6.67 mL) was added to a mixture of graphene nanoplatelets flakes (500.0 mg, 1 wt equiv) and then KMnO4 (3.0 g, 6 wt equiv.) was added slowly. The reaction was then heated to 50 °C and stirred for 6 h. The reaction was cooled to room temperature and poured onto ice cold water (60-70 mL) with 30% $H_2O_2$ (0.5 mL). Then the mixture was centrifuged (4000 rpm for 20 min) and the supernatant was decanted away. The remaining solid material was then washed in succession with 200 mL of water (2 times), 200 mL of 30% HCl, and 200 mL of ethanol (2 times) and the supernatant decanted away. The solid obtained on the filter was vacuum-dried overnight at room temperature to obtaining solid product**.**


**S1.2: pictures showing NS-rGO3 hydrogel , NS-rGO3 aerogel after freeze drying**

Figure ESI S1: NS-rGO3 hydrogel before freeze-drying (Left) , NS-rGOA3 after freeze-drying(Right).

**S.2: XRD measurements: NS-rGOA3 in comparison with GO and RGO**

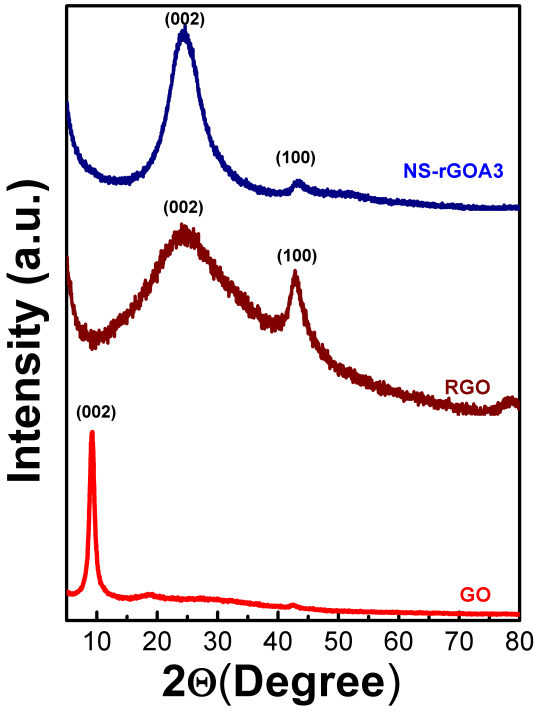



Figure ESI S2: PXRD patterns of GO, RGO and NS-rGOA3 showing various planes in materials

**S3: BET analysis**

**S3.1: BET analysis of NS-rGOA3**

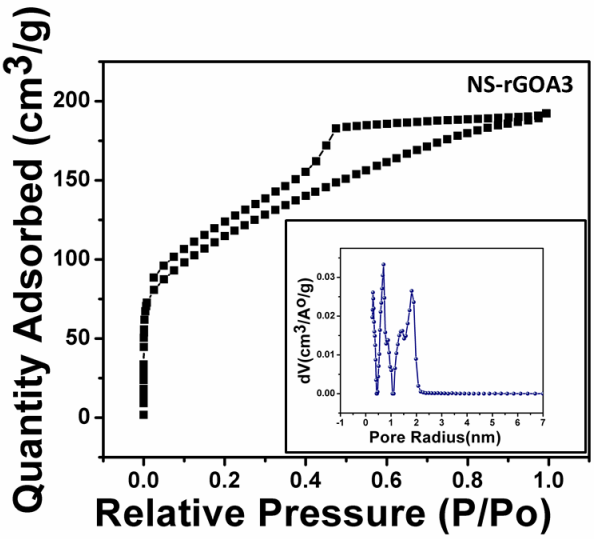



Figure ESI S3: Nitrogen adsorption isotherms at 77.3 K which presents a mixture of type I/IV (H4) hysteresis loop and the BET surface area is 412 $m^2$ $g^{-1}$and inset showing pore size distribution of NS-rGOA3 using NLDFT Model.

**S3.2: t-plot of all three NS-rGOAs (gives the extent of micro pores in the material).**

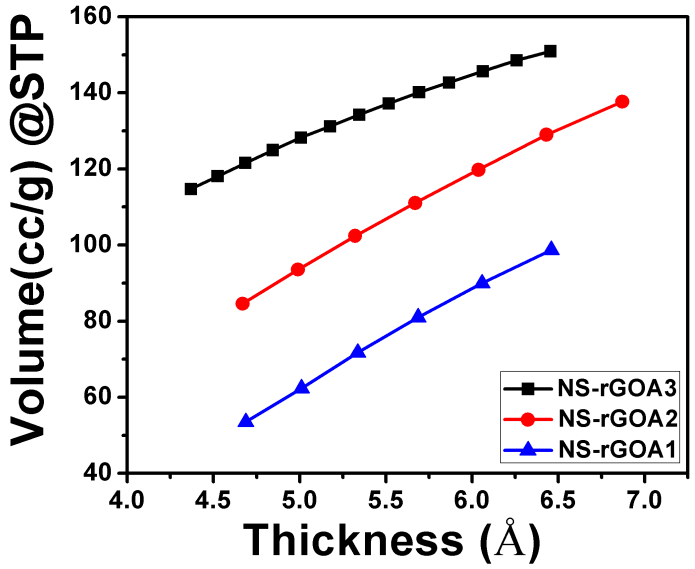



Figure ESI S4: v-t plot (t-plot) for three aerogels NS-rGOA(1-3) giving microporous and mesoporous surface area.

**S3.3: BET adsorption isotherm of all three NS-rGOAs.**

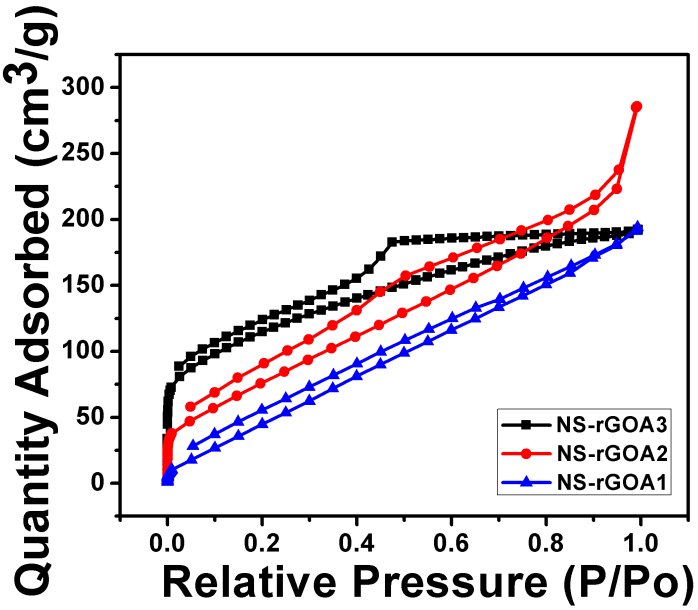
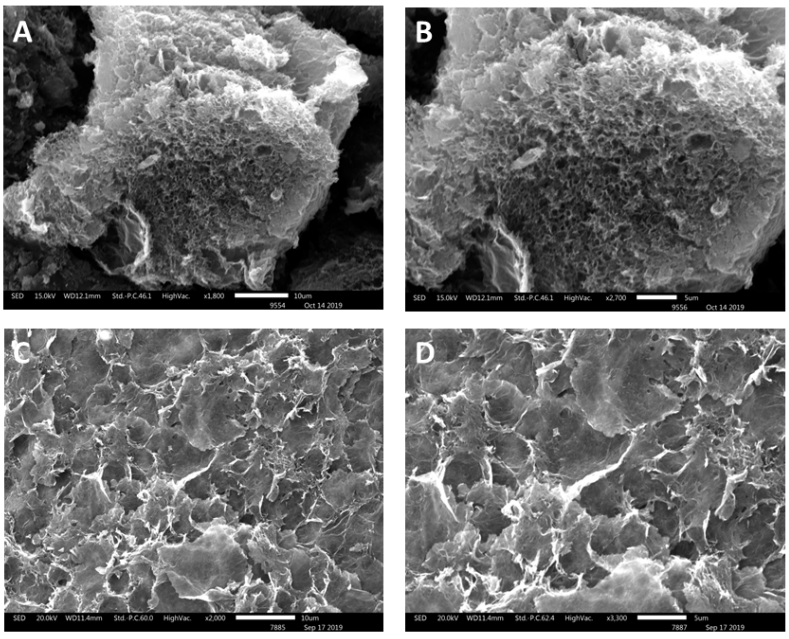



Figure ESI S5: Nitrogen adsorption isotherms at 77.3 K for three aerogels NS-rGOA(1-3).

| Parameters | NS-rGOA1 | NS-rGOA2 | NS-rGOA3 |
|---|---|---|---|
| Micropore Area | 0.00 $m^2/g$ | 0.00 $m^2/g$ | 143 $m^2/g$ |
| Mesoporous (External) area | 265.9 $m^2/g$ | 317 $m^2/g$ | 269 $m^2/g$ |
| BET Surface Area | 265.9 $m^2/g$ | 317 $m^2/g$ | 412$m^2/g$ |
| Pore Width | 2.76 nm | 3.16 nm | 1.4 nm |
| Micropore Volume | 0.00 cc/g | 0.00 cc/g | 0.062 cc/g |

Table. S1 BET results of NS-rGOA1 to NS-rGOAs

**S4: SEM images NS-rGOAs (1&2)**




ESI S6: SEM images of NS-rGOA2(A,B) and NS-rGOA1(C,D) showing their morphology.

**S5: Raman Spectrum of NS-rGOAs(1-3)**

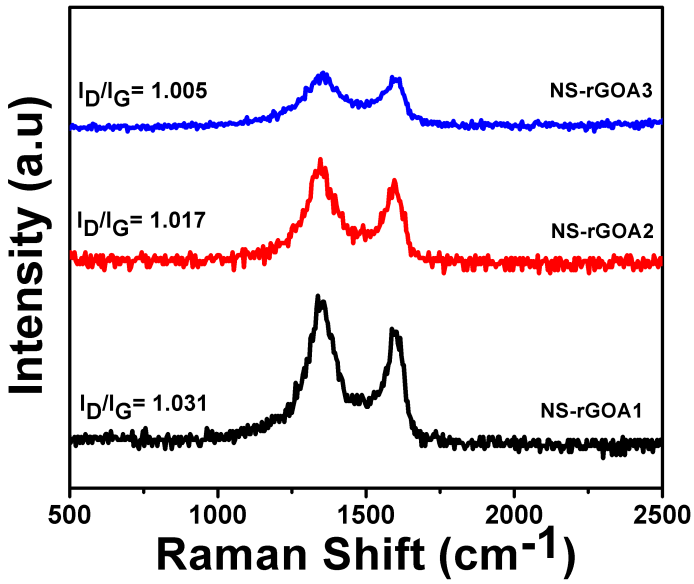



Figure ESI S7: RAMAN spectrum of NS-rGOA3 showing difference in $I_D$ and $I_G$

**S6: Electrochemical measurements: Cyclic voltammeteric analysis different materials**

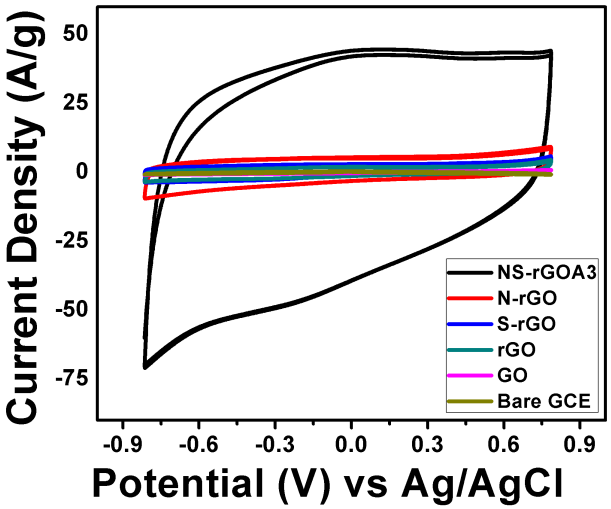



Figure ESI S8: Cyclic voltammeteric analysis of NS-rGOA3, N-rGO, S-rGO ,GO and bare GC electrode

**S7: Device setup**

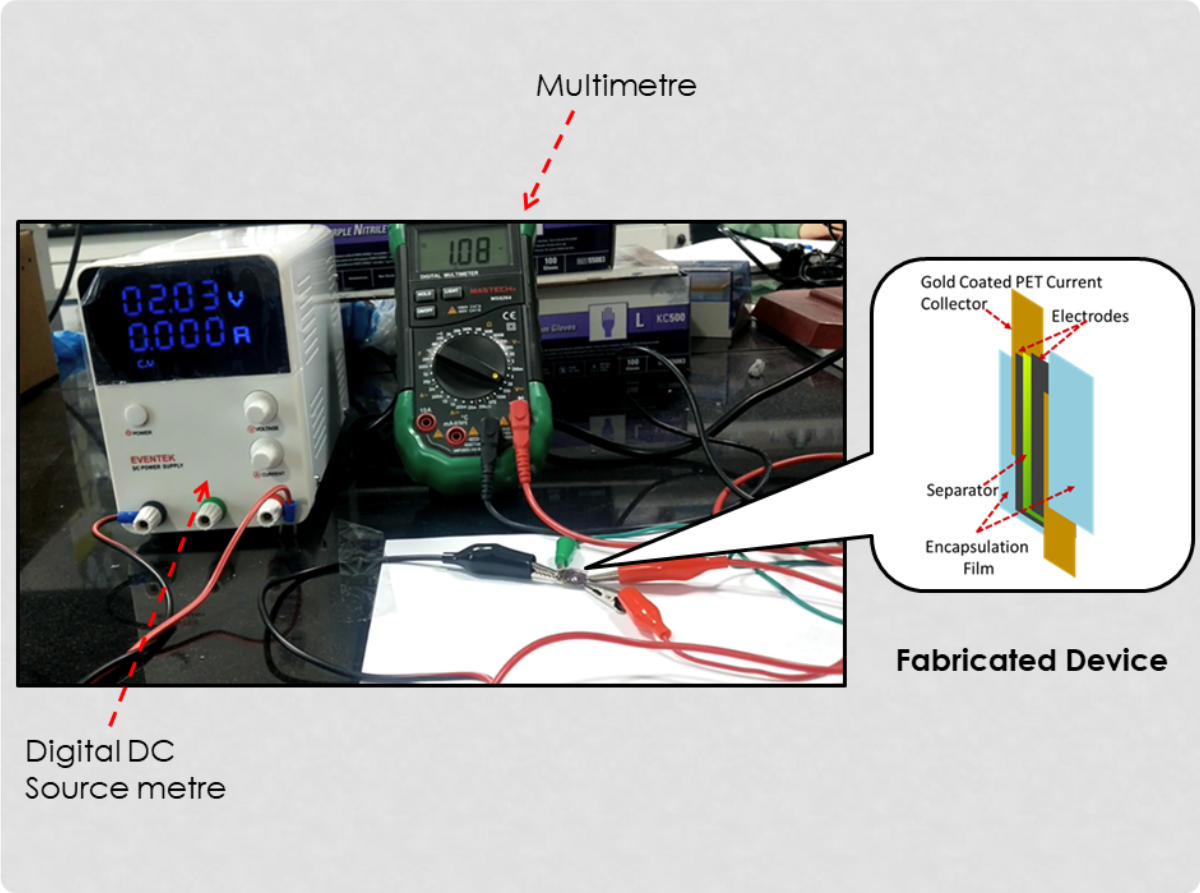



Figure ESI S9: Picture showing the fabricated device and its schematic diagram.

**S8. XPS analysis**

The X-ray photoelectron spectroscopy was carried out by Esca Lab: 220-IXL with Mg-Kα non-monochromated X-ray beam having photon energy 1253.6 eV. The percentage of all the elements present

in samples was found by calculating the area under curve by using CasaXPS software for fitting the curves. The XPS data showing that doping of nitrogen and sulphur has done successfully in the materials whereas the doping ratio if different in three aerogels.

| **Sample** | **Binding Energy(eV)** | | | | **XPS (Wt%)** | | | | **N/S** |
|---|---|---|---|---|---|---|---|---|---|
| | C 1s | O 1s | N 1s | S 2p | C | O | N | S | **Ratios** |
| **NS-rGOA1** | 282.30 | 531.05 | 399.35 | 166.96 | 90.35 | 7.04 | 2.26 | 0.35 | 6.45 |
| **NS-rGOA2** | 282.31 | 531.05 | 398.82 | 165.76 | 88.70 | 6.78 | 4.08 | 0.42 | 9.72 |
| **NS-rGOA3** | 283.64 | 529.73 | 398.83 | 167.02 | 82.61 | 11.34 | 5.34 | 0.69 | 7.74 |

Table S2. XPS results and N/S ratios of NS-rGOA1 to NS-rGOA3.