

1 **Supplementary Information**

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3 **Facile Synthesis and Characterization of Reduced Graphene Oxide / Halloysite**
4 **Nanotubes / Hexagonal Boron Nitride (RGO/HNT/h-BN) Hybrid Nanocomposite**
5 **and its Potential Application as Hydrogen Storage**

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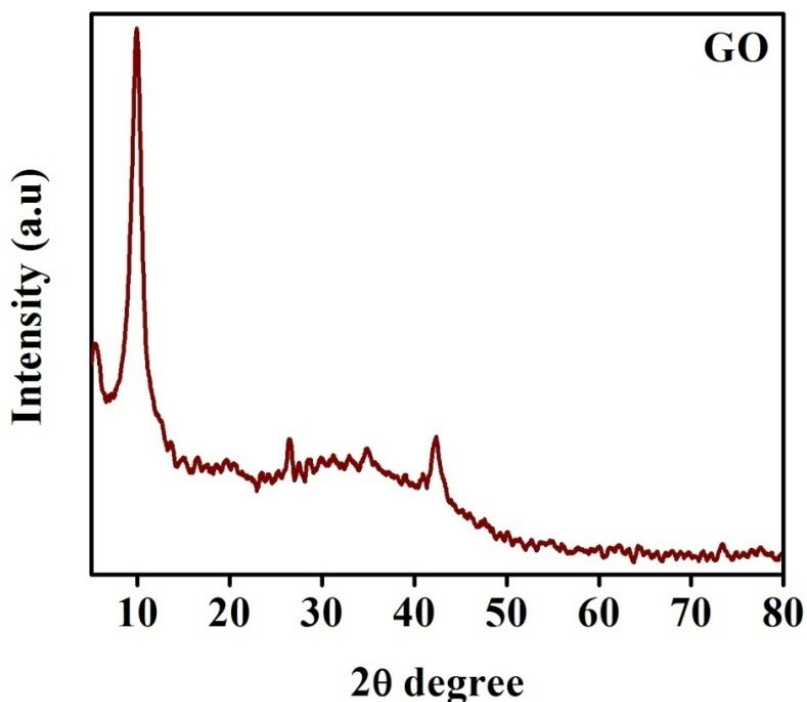
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9 Nadu, India

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11 **XRD Analysis**

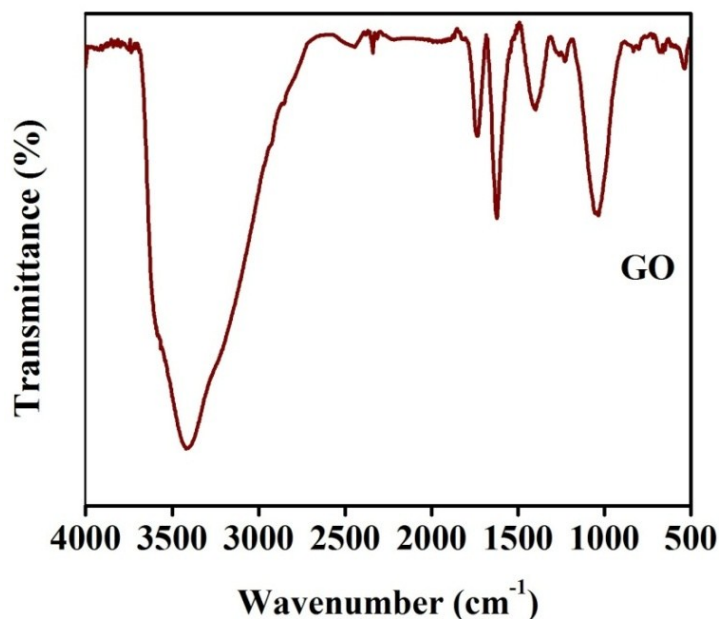
12 Fig. S1 shows the XRD pattern of graphene oxide (GO) where a strong and sharp
13 diffraction peak is observed at $2\theta = 12.02^\circ$ with an interlayer spacing of 0.77 nm along the (002)
14 orientation [1,2].



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16 **Fig. S1 - XRD pattern of GO**

18 FTIR Analysis

19 Fig. S2 shows the FTIR spectrum of GO and the characteristic peaks of GO appear at
20 3423, 2037, 1750, 1627, 1222 and 1051 cm^{-1} are assigned to the O-H stretching of absorbed H_2O
21 molecules, C-H stretching, C=O stretching vibrations of carbonyl and carboxylic groups, sp^2
22 unoxidized C=C stretching, epoxy C-O stretching vibrations and alkoxy C-O stretching
23 vibrations, respectively [3,4]. These modes of vibrations confirm the presence of various oxygen
24 functionalities in the prepared GO.



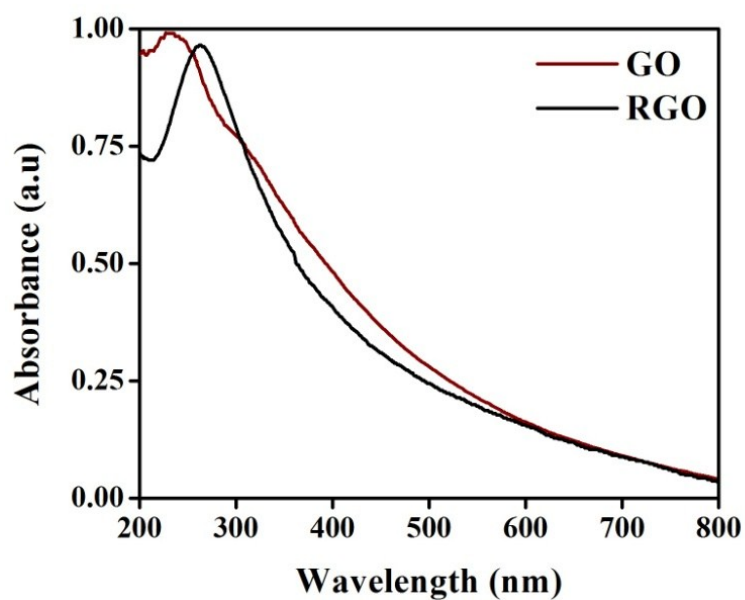
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Fig. S2 - FTIR spectrum of GO

27 UV/Vis Analysis

28 The UV-Vis spectrum of GO has a sharp peak at 233 nm with a shoulder peak at 290 nm.
29 The peak at 233 nm is attributed to the $\pi - \pi^*$ transition of C=C bonds while the band at 290 nm
30 corresponds to the $n - \pi^*$ transition of C=O bonds which further confirms the presence of
31 carbonyl groups in GO. The absorption peak at 233 nm is red-shifted during the reduction of GO
32 into reduced graphene oxide (RGO) [5].



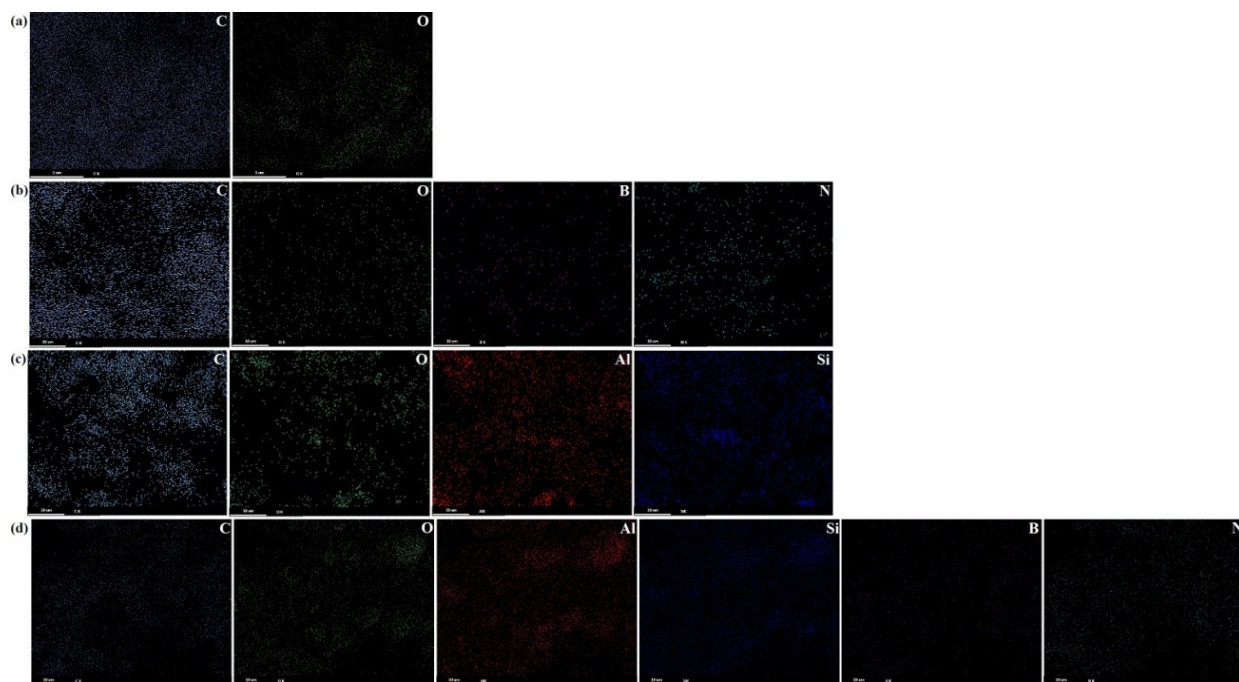
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Fig. S3 - UV/Vis spectra of GO and RGO

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36 **Elemental Mapping Analysis**



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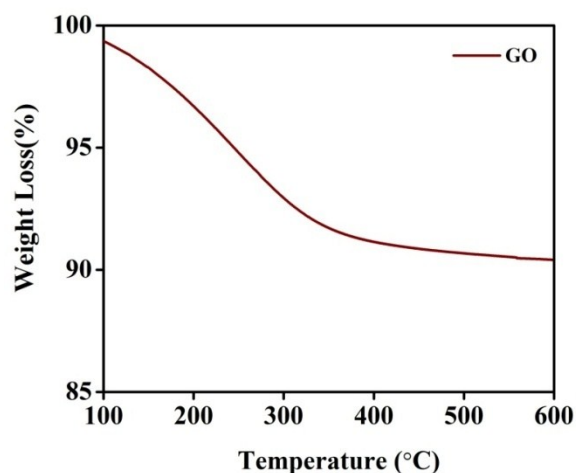
38 **Fig. S4 - Elemental mapping images of (a) RGO (b) RGO/h-BN (c) RGO/A-HNT and**

39 **(d) RGO/A-HNT/h-BN hybrid nanocomposites**

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41 **TG Analysis**

42 The TGA curve of GO is depicted in Fig. S5. It is observed that the prepared GO has the
43 initial weight loss at below 100 °C which is attributed to the evaporation of adsorbed water and
44 the second weight loss at 150 °C is due to the rapid decomposition of oxygen containing
45 functional groups. These oxygen containing groups may arise due to the trapping of more water
46 molecules between the GO layers [6]. These findings are well supported by XRD, FTIR and UV-
47 Vis studies.



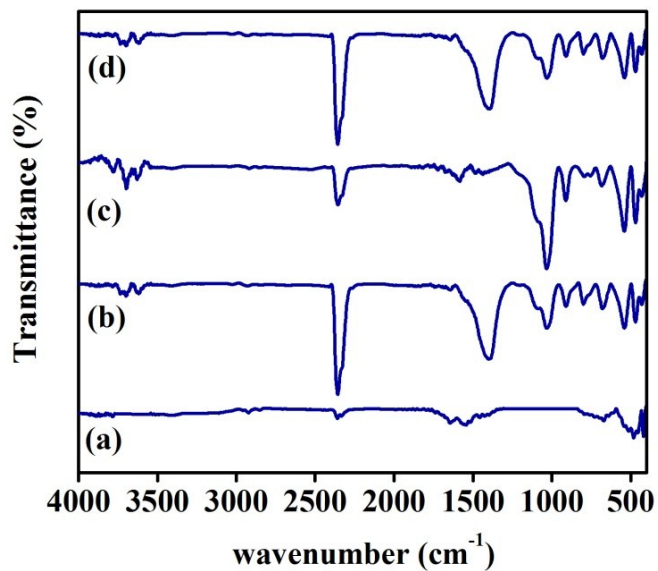
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Fig. S5 - TGA spectrum of GO

50 **FTIR Analysis for Hydrogenated Hybrid Nanocomposites**

51 The FTIR spectra of hydrogenated RGO, RGO/h-BN, RGO/A-HNT and RGO/A-HNT/h-
52 BN hybrid nanocomposites are shown in Fig. S5. It resembles the FTIR spectra of as synthesized
53 RGO, RGO/h-BN, RGO/A-HNT and RGO/A-HNT/h-BN hybrid nanocomposites except the
54 overall decrease in intensity.



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56 **Fig. S6 - FTIR spectra of hydrogenated (a) RGO (b) RGO/h-BN (c) RGO/A-HNT and**
57 **(d) RGO/A-HNT/h-BN hybrid nanocomposites**

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74 References

- 75[1] Shah MSAS, Park AR, Zhang K, Park JH, Yoo PJ. Green Synthesis of Biphasic TiO₂
76 –Reduced Graphene Oxide Nanocomposites with Highly Enhanced Photocatalytic Activity.
77 ACS Appl Mater Interfaces 2012;4:3893–3901
- 78[2] Fan W, Lai Q, Zhang Q, Wang Y. Nanocomposites of TiO₂ and Reduced Graphene Oxide as
79 Efficient Photocatalysts for Hydrogen Evolution. J Phys Chem C 2011;115:10694–701
- 80[3] Liu G, Gui S, Zhou H, Zeng F, Zhou Y, Ye H. A strong adsorbent for Cu²⁺: graphene oxide
81 modified with triethanolamine. Dalton Trans 2014;43:6977–80
- 82[4] Liu X, Pan L, Lv T, Zhu G, Lu T, Sun Z, Sun C. Microwave-assisted synthesis of TiO₂-
83 reduced graphene oxide composites for the photocatalytic reduction of Cr(VI). RSC Adv
84 2011;1:1245-9
- 85[5] Das AK, Srivastav M, Layek RK, Uddin ME, Jung D, Kim NH, Lee JH. Iodide-mediated
86 room temperature reduction of graphene oxide: a rapid chemical route for the synthesis of a
87 bifunctional electrocatalyst J Mater Chem A 2014;2:1332–40
- 88[6] Wang R, Wang Y, Xu C, Sun J, Gao L. Facile one-step hydrazine-assisted solvothermal
89 synthesis of nitrogen-doped reduced graphene oxide: reduction effect and mechanisms. RSC
90 Adv 2013;3:1194–1200

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99 **Figure captions**

100 **Fig. S1 - XRD pattern of GO**

101 **Fig. S2 - FTIR spectrum of GO**

102 **Fig. S3 - UV/Vis spectra of GO and RGO**

103 **Fig. S4 - Elemental mapping images of (a) RGO (b) RGO/h-BN (c) RGO/A-HNT and**
104 **(d) RGO/A-HNT/h-BN hybrid nanocomposites**

105 **Fig. S5 - TGA spectrum of GO**

106 **Fig. S6 - FTIR spectra of hydrogenated (a) RGO (b) RGO/h-BN (c) RGO/A-HNT and**
107 **(d) RGO/A-HNT/h-BN hybrid nanocomposites**