Appendix A

Supporting information

A.1. Differential plot of intensity versus binding energy of deconvoluted XPS spectra

Figure A1: Differential plot of intensity versus binding energy (a) Ni 2p, (b) Co 2p, (c) O 1s of CNO and CNOG, and (d) C 1s spectra of CNOG

Figure A1 displays the differential plots of intensity versus binding energy of the deconvoluted peaks of Ni 2p, Co 2p, O 1s and C 1s spectra of fig. 3.10. The binding energy of the deconvoluted peaks in fig. 3.10 can be obtained from their corresponding differential plots in fig. A1

A.2. Kinetic behaviour of MXene and NiCo2O4/NiO/MXene (CNOT) supercapacitor electrode

The kinetic behaviour of the electrodes MXene and $NiCo₂O₄/NiO/MX$ ene (CNOT) are studied to know the contribution of the charge storage mechanisms, namely, diffusion controlled faradaic process, and non-diffusive capacitive process by using the following Power's law (equation A1):

$$
I(V) = av^b \tag{A1}
$$

where, $I(V)$ and v represent the current density at a fixed potential value and scan rate, respectively. α and β are two adjustable parameters [1, 2, 3]. The slope of $log I(V)$ vs. $logv$ gives the value of b. When b value is equal to 0.5, then the storage mechanism is dominated by diffusion controlled process. Whereas, b value equals to 1 suggests surface capacitive process. Dunn in their work $[4]$ reported that when b value lies very close to 0.5, i.e. $b = 0.55$, it indicates intercalation of electrolyte ions. However, when b lies in the range of 0.8-1, the undergoing process is dominantly surface capacitive. The quantitative contribution of each mechanism can be calculated by using the following relation (equation A2):

$$
I(V) = k_1 v + k_2 v^{1/2}
$$
 (A2)

where, k_1v and $k_2v^{1/2}$ signify the capacitive and diffusive contribution of current, respectively, v is the scan rate at which $I(V)$ is noted, and k_1 and k_2 are constants. The plot of $\frac{I(V)}{V^{1/2}}$ vs. $V^{1/2}$ gives the values of k_1 and k_2 . The slope gives the k_1 value, whereas k_2 is the intercept.

A.2.1. Analysis of MXene electrode

The logarithm of current density of MXene is plotted as a function of logarithm of scan rate and presented in fig. A2a. The anodic b value is 0.68, whereas the cathodic b is 0.55. The presence of both capacitive and diffusive process is revealed from the 0.68 slope value. On the other hand, the cathodic slope of 0.55 suggests the intercalation of electrolyte ions into the MXene nanosheets. The contribution ratio of capacitive and diffusive process is obtained at the scan rate $(50-170)$ mV/s as shown in fig. A2b. It is observed from fig. A2b that with increase in scan rate the capacitive contribution increases from 21% (50 mV/s) to 33% (170) mV/s, whereas the vice-versa is observed

for the diffusive process. At higher scan rate, the electrolyte ions do not get enough time to reach the active sites and initiate the redox reactions, as a result of which the diffusive current reduces. The capacitive current contribution out of the total current is visualized at 50 mV/s, as displayed in fig. A2c.

Figure A2: For MXene electrode: - (a) Plot of log (current density) vs. log (scan rate) in the range (10-200) mV/s and (30-200) mV/s for anodic and cathodic current density, respectively, (b) Relative contribution of capacitive and diffusive mechanism at scan rates (50-170) mV/s, (c) total current and capacitive current contribution at 50 mV/s scan rate.

A.2.2. Analysis of NiCo2O4/NiO/MXene (CNOT) electrode

As presented in fig. A3a, CNOT exhibits both the surface capacitive and diffusive controlled mechanism of charge storage as the anodic and cathodic b value is 0.77 and 0.81, respectively at high scan rate (30-200) mV/s. However, the 0.81 cathodic slope indicates the dominance of capacitive process at high scan rate.

Figure A3: For CNOT electrode: - (a) Plot of log (current density) vs. log (scan rate) in the range (30-200) mV/s for anodic and cathodic current density, respectively, (b) Relative contribution of capacitive and diffusive mechanism at scan rates $(3-7)$ mV/s (c) Relative contribution of capacitive and diffusive mechanism at scan rates (30-100) mV/s, (d) total current and capacitive current contribution at 50 mV/s scan rate.

At low scan rate (3-7) mV/s, the capacitive current (as displayed in fig. A3b) dominates the diffusive current due to the presence of MXene nanosheets. Similarly, at higher scan rate (as observed in fig. A3c), the capacitive current increases with increase in scan rate. The percentage of diffusion current reduces at higher scan rate, as the electrolyte ions do not get ample time to reach the active sites and initiate the redox reactions. On deconvoluting the CV of CNOT at 50 mV/s (fig. A3d), we observe that maximum percentage of the total current is contributed by capacitive process. Thus, in nanocomposite CNOT capacitive current dominates and the contribution of capacitive current of CNOT is higher than pristine MXene nanosheets due to the synergistic contribution from each individual component.

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List of Publications

A. In Refereed Journal

- 1. **Baruah, K.**, Kumar, A. and Deb, P. Visible light active $Au@g-C_3N_4$ core-shell plasmonic photocatalyst. *Materials Today: Proceedings*, 47:1627-1632, 2021.
- 2. **Baruah, K**., Sarmah, D. and Kumar, A. Ternary hybrid nanocomposites of polypyrrole nanotubes with 2D self-assembled heterostructures of protonated g-C3N4-rGO as supercapacitor electrodes. *Ionics*, 27(7):3153-3168, 2021.
- 3. **Baruah, K.** and Deb, P. Electrochemically active site-rich nanocomposites of two-dimensional materials as anode catalysts for direct oxidation fuel cells: new age beyond graphene. *Nanoscale Advances*, 3(13):3681-3707, 2021.
- 4. **Baruah, K.** and Deb, P. Enabling methanol oxidation by an interacting hybrid nanosystem of spinel Co3O⁴ nanoparticle decorated MXenes. *Dalton Transactions*, 51(11):4324-4337, 2022.
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- 6. **Baruah, K**., Nandi, S., Pershaanaa, M., Ramesh, K., Ramesh, S. and Deb, P. A versatile non-precious metal based electrode material endowed by layer-on-layer structure for methanol oxidation and supercapacitor applications. *Journal of Energy Storage*, 84:110867, 2024.
- 7. **Baruah, K.,** Nandi, S., Singh, A.K., Pershaanaa, M., Ramesh, K., Ramesh, S. and Deb, P. Layered MXene-transition metal oxide nanocomposite revealing its versatility in methanol oxidation and PVA/KOH hydrogel-based symmetric supercapacitor. *International Journal of Modern Physics B*, 2540047, 2024.

B. Book chapter

Baruah, K. and Deb, P. Nanocomposites of NiO/Graphene as efficient electrocatalyst in fuel cell. In Proceedings of 28th National Conference on Condensed Matter Physics: Condensed Matter Days 2020 (CMDAYS20), pages 211-216, ISBN 9789811654077 Springer Singapore, 2021.

C. Paper presented in conferences

- 1. **K. Baruah**, D. Sarmah, A. Kumar, **International Conference on Advanced Nanomaterials and Nanotechnology 2019 (ICANN-2019)**, Centre for Nanotechnology, Indian Institute of Technology Guwahati (IITG), Assam, India, December 18-21, 2019.
- 2. **K. Baruah**, A. Kumar, **International Web Conference on Recent Advances in Nanoscience & Nanotechnology for High-end Applications (IWCRANHA-2020)**, Department of Applied Science and Humanities, Assam University, Silchar, Assam, India, July 25-26, 2020.
- 3. **K. Baruah**, A. Kumar, **Online National Conference On Recent Advances in Functional Materials (RAFM-2020),** Department of Physics, ARSD College, University of Delhi, Delhi, India, December 5-6, 2020.
- 4. **K. Baruah**, P. Deb, **National conference "Condensed Matter Days 2020 (CMDAYS 20)",** National Institute of Technology Silchar, Assam, India, December 11-13, 2020.
- 5. **K. Baruah**, A. Kumar, P. Deb, **National Conference on Emerging Trends in Material Science**, Department of Physics, Tezpur University, Assam, India, May 17, 2022.
- 6. **K. Baruah,** P. Deb, **International Seminar on Environmental Issues & Sustainable Development: The Global and Indian Perspective,** Department of History and Department of Chemistry, Nanda Nath Saikia College, Titabar, Assam, India, May 3-4, 2024.

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