

Study of Carbonaceous Hybrid Nanomaterials for Energy Storage/Conversion Devices

**SUMMARY SUBMITTED FOR THE AWARD OF THE DEGREE
OF**

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SUMMARY

Energy is the most crucial factor in human life. It plays a significant role in every second of life [1,2]. As per the international energy agency reports, consumption is increasing daily because of the population increase throughout the universe. That is continuously reflected in the energy demand. Since traditional energy sources such as oil, coal and natural gas have some limitations and are decaying day by day [3]. Alternate energy technologies are the need of the hour, which motivates researchers to work in the new and renewable energy technology field. These needs are fulfilled by alternative energy production options such as wind, hydropower, solar, and geothermal energy [4,5]. However, these renewable energy sources also have limitations in terms of geographical situations and environmental factors [6,7]. So sustainable alternatives are required to explore. These are very important as per substitutional ability. So many efforts have been made in this field to overcome the mentioned issue; the storage technology is at the top most as per the significance of energy utilization on the unavailability of natural energy sources [8,9]. Storage technology plays a vital role in the supply when the natural source may get restricted, as well as the simultaneous extra energy storage when the production is over to direct utilization [10–12]. In storage technology, the primary and secondary battery, supercapacitor and fuel cell play a significant role. Out of all the mentioned storage technology, the secondary battery and supercapacitor show a very rigorous performance [13–15]. The secondary battery shows higher energy density and moderate power density. So as per application requirements, the secondary battery is used where the continuous energy requirement at fix current. However, it lacks in boosted supply because of its low power density [16,17]. Modern supercapacitors show superior performance (Fig. 1). It is due to advanced power and energy densities [18–20]. The traditional capacitor has limited performance and application, but the supercapacitor with higher power and energy density has a broader application. The supercapacitors differ from

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the traditional capacitors in terms of charge storage mechanism. The pure capacitors stores charge electrostatically (polarization), while the pseudo capacitors stores charge via the EDLCs and electrochemically (redox reactions are contributing but surface phenomena). The most updated version of the hybrid capacitor stores the charge in both ways, as EDLC and the reversible redox reactions contribute predominantly [18,20–22].

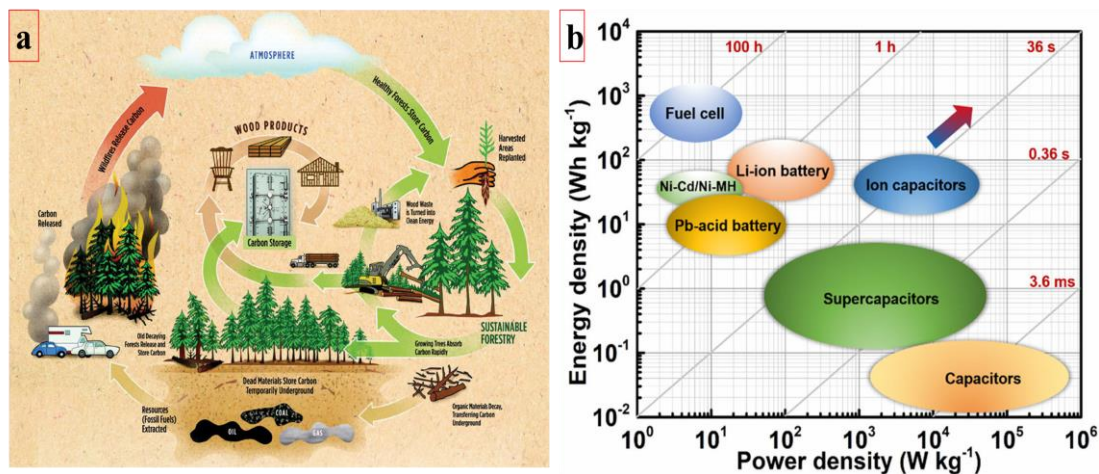


Fig. 1 (a) Depiction of the energy sources (b) Ragone plot of energy storage device: Power density versus Energy density.

In general, energy storage devices are classified as thermal energy storage, mechanical energy storage, electrochemical energy storage, geothermal energy storage and nuclear energy storage systems. Out of all energy storage systems, the most common energy storage devices are electrochemical energy storage systems [23]. These energy storage devices consist of two electrodes, separated by a semi-permeable membrane and an electrolyte (solid or liquid). The electrode comprises different materials, such as carbon or metal oxide, and the external voltage creates a polarization between the electrodes, so the current starts to flow [24–27]. Thus these energy storage devices convert chemical energy into electrical energy, which is a reversible process. Batteries and supercapacitors are the most commonly exercised energy storage devices [28–30]. However the energy storage process is similar for both systems, but the very significant differences between the two, particularly the chemical storage process [31]. The secondary battery stores charge via the deep reversible faradic reactions, which involve the oxidation and reductions at the cathode and anode successively. The anode releases the electrons to the circuit with the applied voltage difference, whereas

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the cathode gains electrons from the external circuit. Higher stability is required at the electrode-electrolyte interface for the proper functioning of these reactions. There are two main types of batteries, primary and secondary batteries [31–33]. The primary battery is designed for single use; these are discarded after the complete discharge. The secondary (rechargeable) battery can restore the charges to their initial condition by reversing the current flow after the battery is fully discharged. Zinc-air, alkaline batteries and carbon zinc are a few examples of primary batteries. In these batteries, the electrode material is mainly metal-based, generally like Zn, Mg and Al etc. These materials are mainly made up of metals. The possible reason behind this is abundance, lower cost, environment friendliness, suitable discharge voltage and longer cyclic life. These properties make the metals the most suitable electrodes for primary battery materials [34–37]. These batteries create environmental issues due to unchangeable behaviour; nowadays, researcher is recovering metals from these primary batteries and making an electrode for supercapacitors and other energy storage devices. The modern electronic and technological developments based on wireless communication devices and the Internet of Things (IOTs) work on rechargeable batteries making the primary batteries less relevant. Rechargeable systems available in the international market as well as intensively protonated research prototypes, particularly lead-acid (Pb-acid) batteries, Lithium-ion batteries (LIB), Nickel metal hydride (NiMH) batteries, nickel-cadmium (NiCd) battery and sodium-ion battery (NIB) [37,38]. Graphitic carbon and its allotropes, transition metal oxide, and hydroxide are the intercalated- based electrode most exercised for energy storage systems. On the other hand, the supercapacitor, with its long life cycle, durability, and fast charging-discharging ability, opens a new path for making modern energy storage devices. The supercapacitors are super because of their advanced storage mechanism, which includes electrostatic and redox reaction-based storage. Carbon and its derivatives are used on a large scale in making supercapacitor electrode materials in the traditional era. Due to their variable oxidation, high conductivity and high surface values, the oxide and hydroxide materials are most exercised in the last 20 years. These materials are favourite for making flexible electronic devices as well. Based on the charge storage mechanism, the modern supercapacitor can be divided into the following classes: **(a)** Electric double-layer Capacitor, **(b)** Pseudo Capacitor, **(c)** Hybrid Capacitor, **(d)** Battery-type Capacitor.

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Chapter 1 deals with the subject background and the recent updates in the field of carbonaceous metal oxide/hydroxide electrode materials. The primary focus of this chapter is to introduce the subject terminology, materials type, and advantages of these materials in modern energy storage devices. Different types of energy storage systems with an emphasis on electrochemical energy storage devices with various parameters have been discussed. The supercapacitors based on the charge storage mechanism such as EDLCs, pseudo capacitors and hybrid supercapacitors are discussed in detail. Since the work is based on carbonaceous hybrid materials, this chapter also gives an insight into various shapes of carbonaceous materials and their advantage in energy storage devices. Since environment-friendly and sustainable electrode materials are top priority in the energy storage field, this chapter also focuses on the importance and limitation of various environment-friendly materials.

In **Chapter 2** synthesis and application of mesoporous carbon zirconium oxide hybrid materials through detailed structural, microstructural and electrochemical analysis have been described. In all samples, the 15 wt.% addition of zirconium oxide in mesoporous carbon is the optimum value, and the device comprising the 15 wt.% electrode is the best in terms of energy, power density as well as capacitance retention %. The detailed charging-discharging (GCD) based study is shown in Fig. 2. This GCD analysis exhibited the specific capacitance of the device as 125 F g^{-1} , and the energy and power density of the device were found as 25 Wh kg^{-1} at a current density of 0.5 Ag^{-1} , 1201 W kg^{-1} at 1 mA . The device showed high capacitance retention up to 82 % till 10k cycles. Columbic efficiency was found 100 % till the last segment of the test. The prototype device was fabricated and tested by a LED glow. Overall, it behaved as a potential electrode material for a device application.

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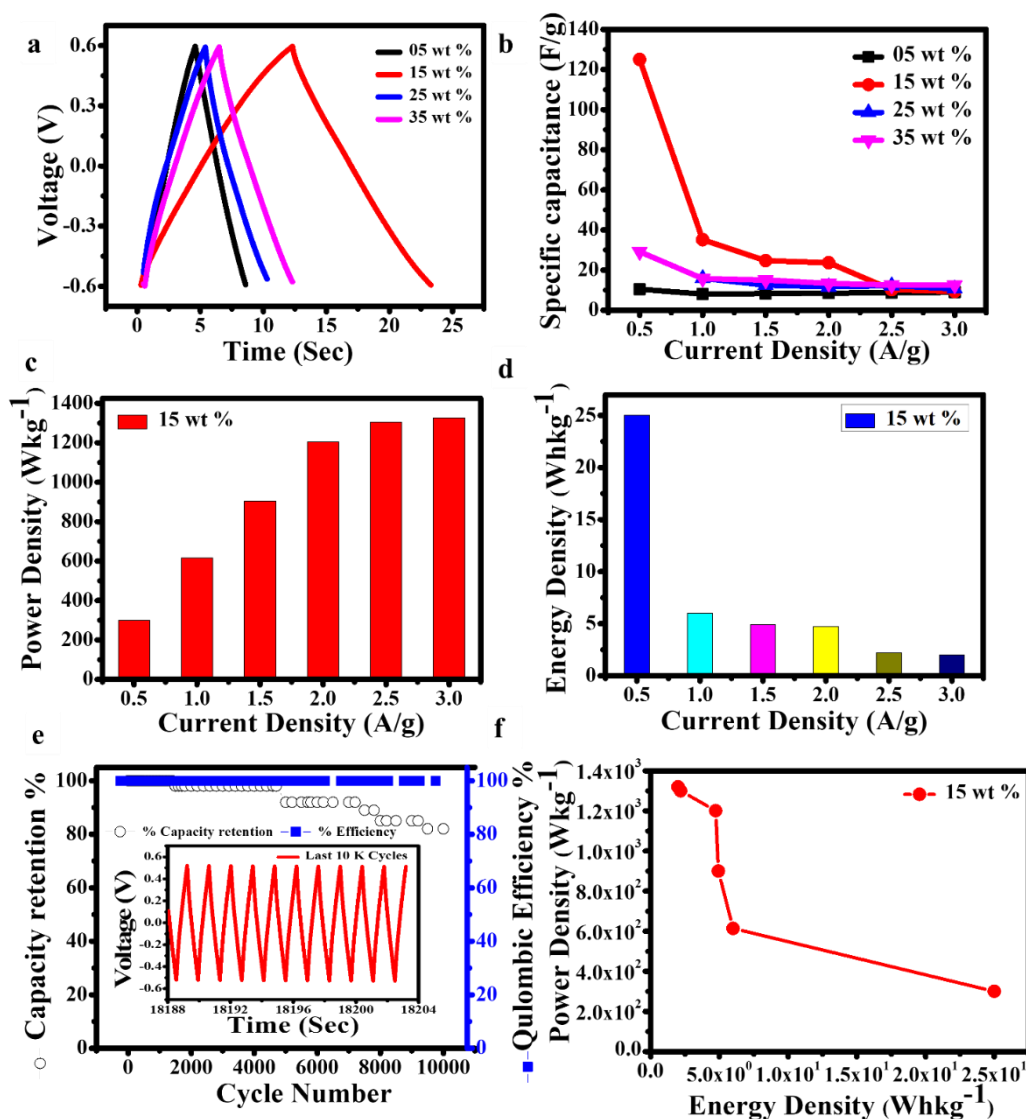


Fig. 2. Galvanostatic Charge-Discharge Analysis (a) Charging-discharging profile of the all mesoporous carbon@ZrO₂ composite electrode at fixed current (b) Current density vs specific capacitance of all samples (c) Current density vs power density of all electrodes (d) Energy density vs Current density of all electrodes (e) Coulombic efficiency, Capacitance retention % vs Cycle number, (f) Ragone plot (Energy density vs Power density).

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Chapter 3 reports the synthesis of activated carbon@FeOOH series with molarity variation of FeOOH composites, and detailed device performance evaluated through various electrochemical tools. The structural, microstructural and electrochemical investigation validated its formation, morphology and potential as an energy storage device. The electrode exhibited a specific capacitance of 342 (F g⁻¹) at 10 mV s⁻¹, which is also supported by the GCD analysis. The EIS analysis shows the bulk resistance (R_b) of 1 Ω for optimized ACF1. The energy density E_d and power density P_d values were calculated for ACF1 as 40 Wh kg⁻¹ and 4200 W kg⁻¹. The ACF1 showed the finest cyclic stability up to 10k cycles with capacitance retention and coulombic efficiency of 80% and 96%. The capacitive and diffusive contributions are shown in Fig. 3. The detailed electrochemical storage behaviour is presented in Fig. 4 which shows the retention profile as well the Ragone plot. This also characterizes the advanced energy storage ability of the activated carbon@FeOOH electrode material.

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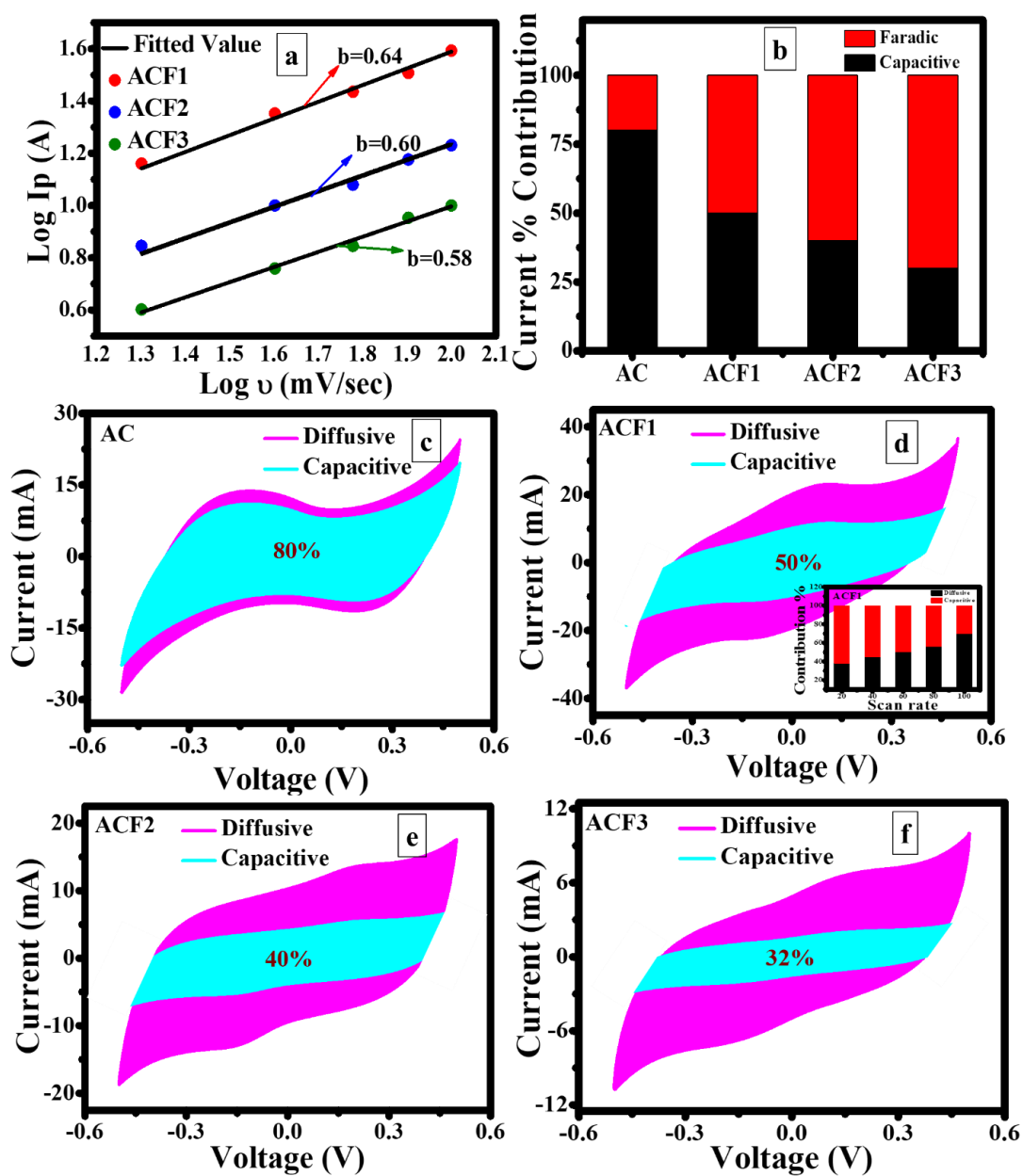


Fig. 3. (a) b value for all electrode materials (b) Current % contribution for all samples by b value calculations (c) Capacitive & diffusive current % of AC (d) Capacitive & diffusive current % of ACF1, inset show scan rate vs current % contribution for ACF1 (e) Capacitive & diffusive CV of ACF2 (f) Capacitive & diffusive CV of ACF3.

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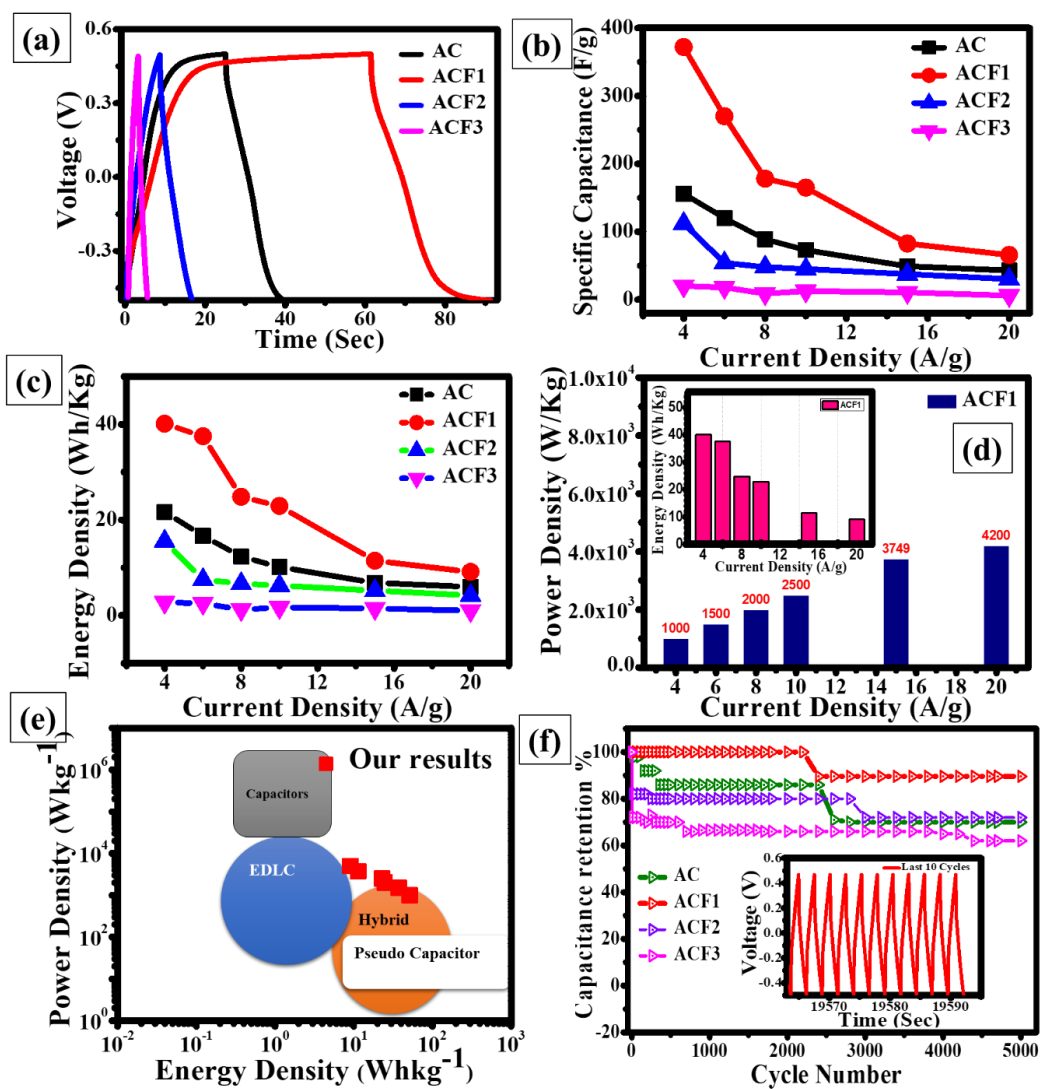
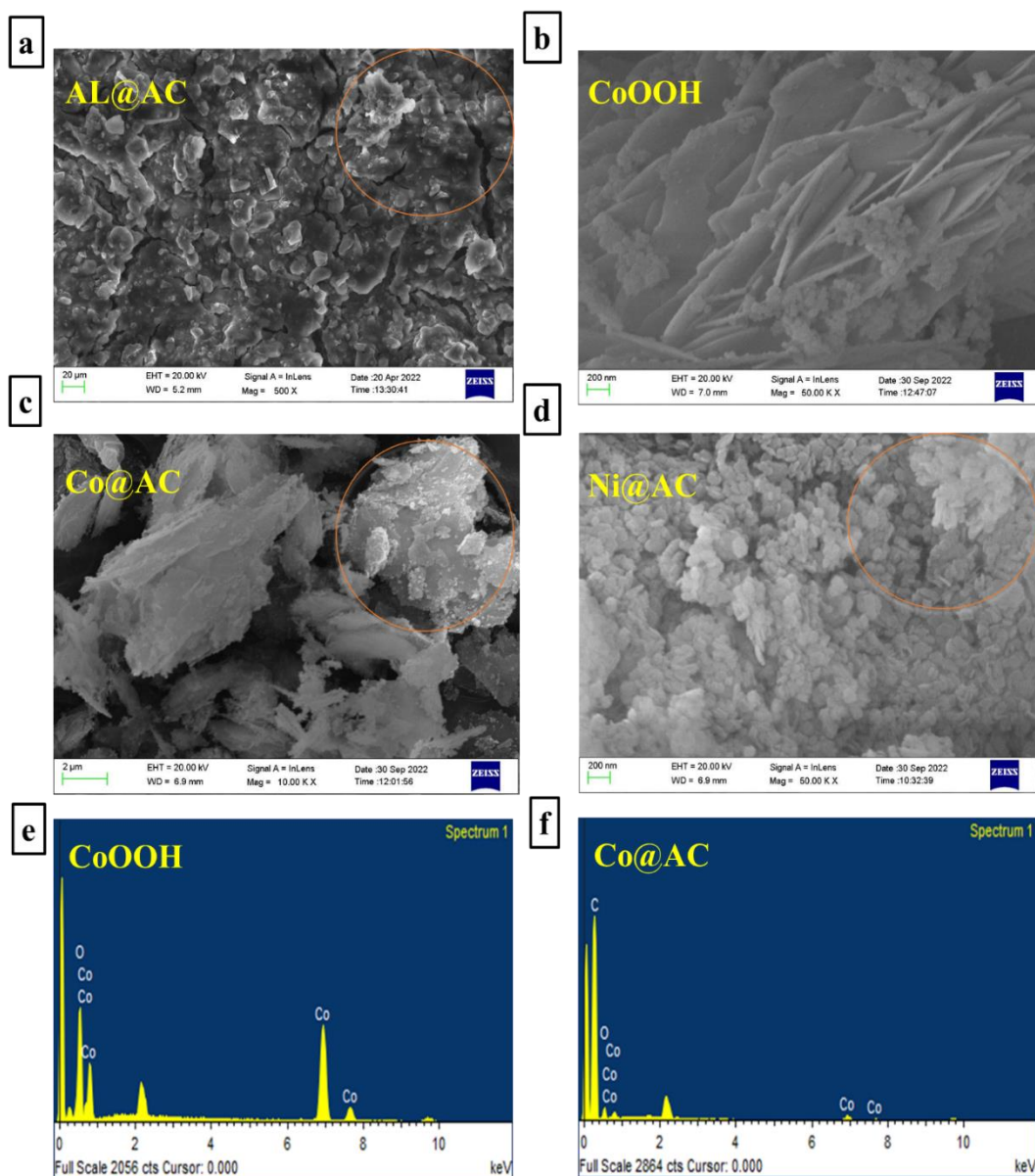


Fig. 4. (a) Charging-discharging pattern at 2 mA (b) Specific capacitance with varying current density (c) Energy density (E_d) with change in current, (d) Power density vs current values for ACF1, inset shows current density vs Energy density for ACF1 (e) Ragone Plot (f) Capacitance retention % with the change in cycle number, inset show last 10 cycles of stability test.

In **Chapter 4** a detailed comparative analysis of pure CoOOH and all (AlOOH, CoOOH, NiOOH)@carbonaceous electrode materials were performed. The surface morphological and microstructural investigations were carried out using FESEM, XRD, which confirmed the formation of electrode materials and the sheet-like nanoarchitecture corresponding to Co@AC. The specific capacitance values for Co@AC, via CV and GCD, were found as 230

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$F g^{-1}$ and $57 F g^{-1}$ at $10 mV s^{-1}$ and $2 mA g^{-1}$. The energy and power density values for Co@AC were estimated as $37.34 Wh kg^{-1}$ and $4200 W kg^{-1}$ at a current density $2 A g^{-1}$. The capacitance retention was found 72 % for Co@AC till the last segment of GCD. The lab-scale fabricated device was found able to glow a panel of 26 LED for 5 minutes. Also, detailed theoretical calculations for optimized base CoOOH for performance prediction were carried out which validated the experimental findings. The different shape of carbonaceous electrode materials is shown in Fig. 5. The detailed energy storage contribution through faradic and non-faradic process is presented in Fig. 6.



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Fig. 5. (a) FESEM of AL@AC, (b) FESEM of CoOOH, (c) FESEM of Co@AC, (d) FESEM of Co@AC (e) EDS image of CoOOH (f) EDS image of Co@AC.

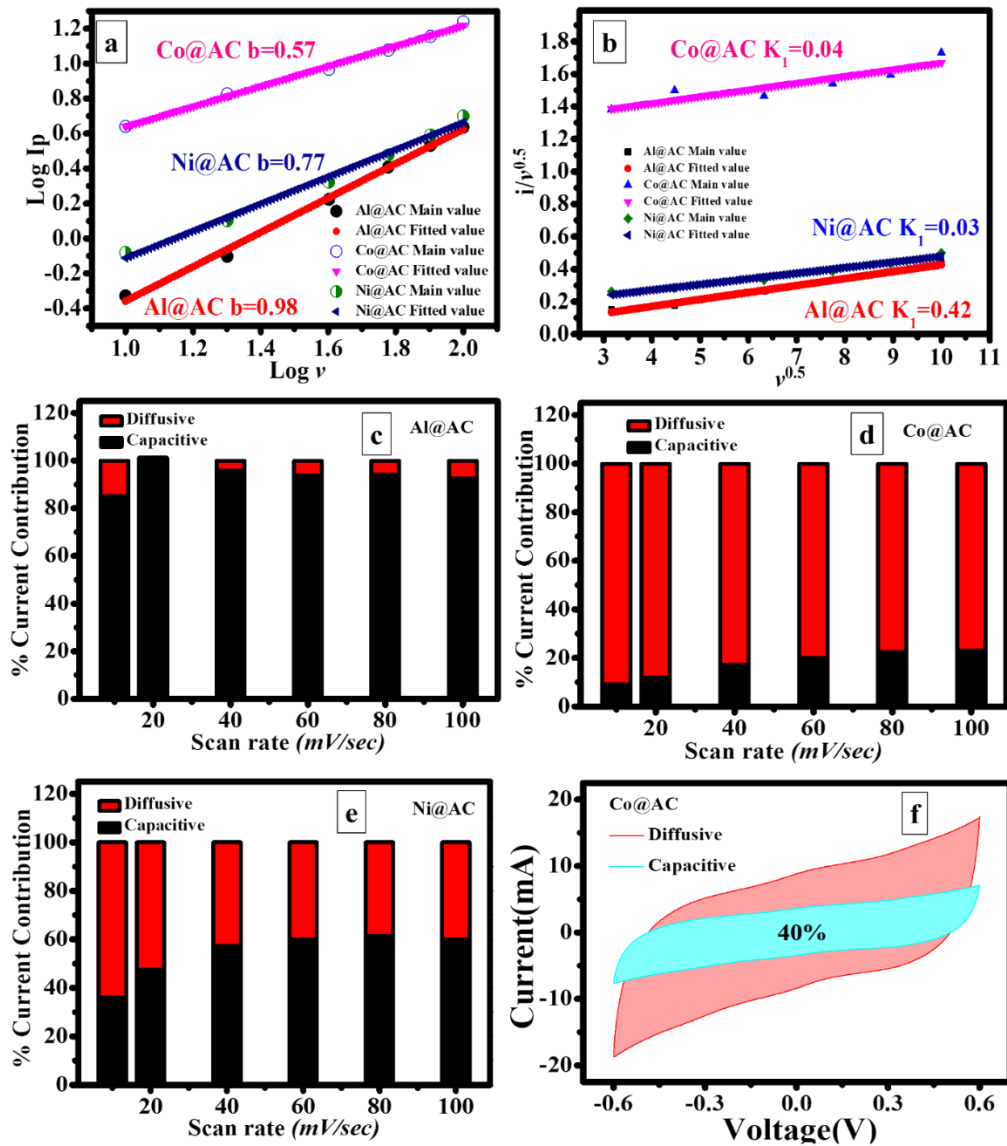


Fig. 6. Capacitive and diffusive contributions (a) $\log v$ vs $\log i$ for b value estimation (b) Plot between $v^{0.5}$ and $i/v^{0.5}$, shows the K_1 -for capacitive and diffusive estimation (c) Current, capacitive and diffusive % contributions for Al@AC, (d) Current capacitive and diffusive % contributions for Co@AC (e) Current capacitive and diffusive % Contributions for Ni@AC (f) Capacitive and diffusive % contributions at 100 mV/sec for Co@AC.

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Chapter 5 summarizes the study of Carbonaceous Hybrid Nanomaterials for Energy Storage/Conversion devices. The earth-abundant carbon@FeOOH composite series using a low-temperature hydrothermal technique was successfully synthesized. Microstructural confirmation was done by X-ray diffraction analysis; the FESEM revealed all shapes of carbonaceous electrode materials, such as fibre, rod and spherical shaped carbon particles (Fig.7). The BET estimated specific surface area as $52.7 \text{ m}^2 \text{ g}^{-1}$ for sample MCF, and the pore diameter was found as 36.71 nm. The electrochemical impedance spectroscopy (EIS) study showed that lower value of R_b (bulk resistance) and improved associated parameters for MCF. The MCF electrode showed the maximum specific capacitance of 84.3 F g^{-1} at 10 mV s^{-1} , with a potential window of 1 volt. The GCD exhibited the highest specific capacitance as 54.5 F g^{-1} at a current density of 1 A g^{-1} . The energy density and power density calculated for MCF were 7.5 Wh kg^{-1} and 5000 W kg^{-1} at current density 10 A g^{-1} . The detailed electrochemical investigations, which validated the storage performance are shown in Fig. 8. Overall, the study met its aims and MCF may be commercially used as the best material for the device fabrication for the electrochemical performance.

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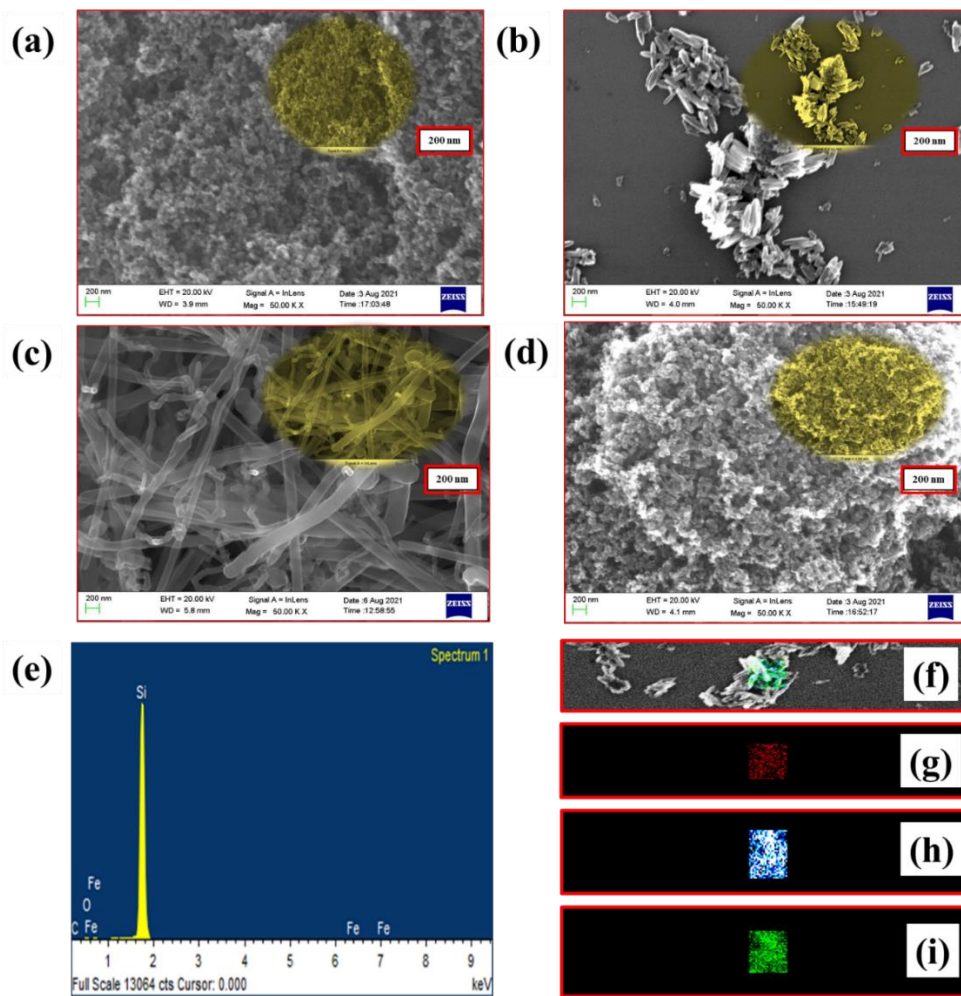


Fig. 7. FESEM Images (a) CBF (b) ACF (c) CNFF (d) MCF (e) EDS images of MCF (f) EDS-Mapping of all elements of MCF (g) EDS-Mapping of Fe (h) EDS-Mapping of oxygen (O) (i) EDS-Mapping of carbon (C).

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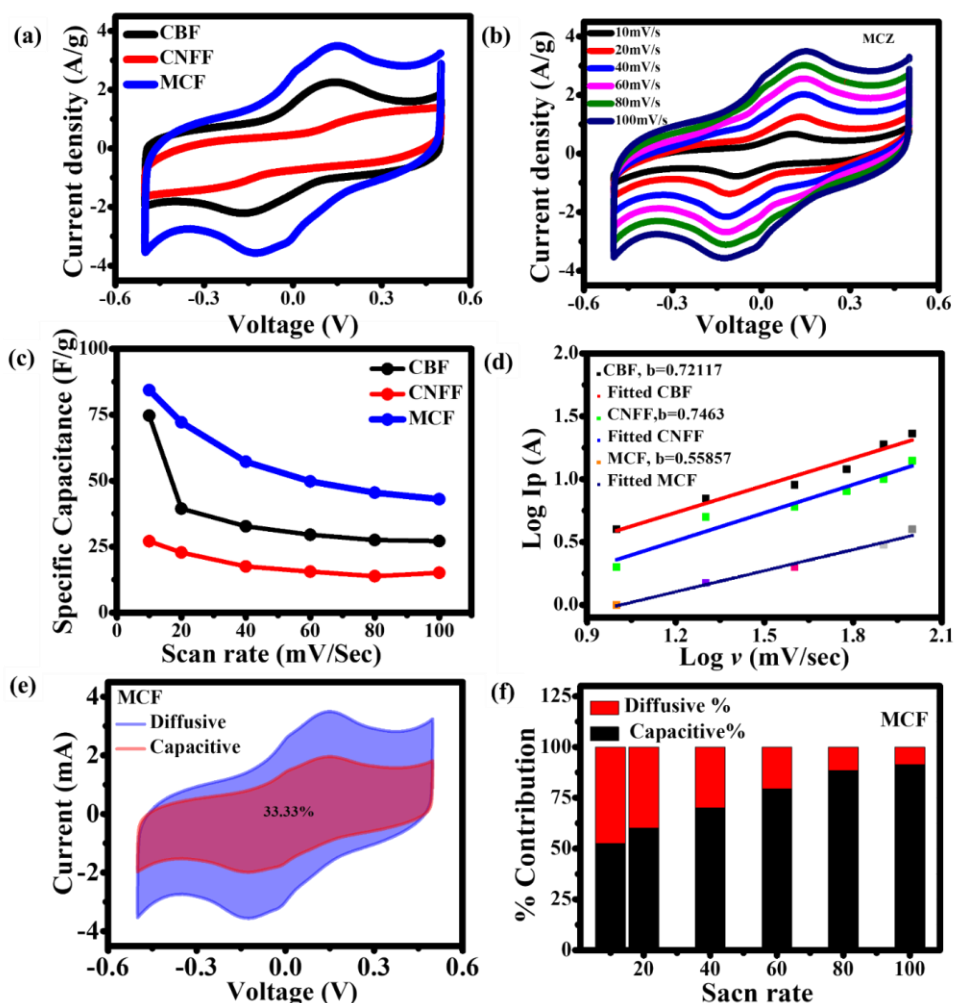


Fig. 8. Cyclic voltammetry (a) CV Graph of at 100 mV s^{-1} (b) CV curve with varying scan rate (10 to 100) mV s^{-1} for MCF (c) Specific capacitance vs scan rate for all samples at varying scan rate (d) Calculation of b value the I_p vs v (e) CV graph showing Capacitive and diffusive % contribution for MCF (f) Capacitive and diffusive % contribution for In

In **Chapter 6** synthesis of the ternary carbonaceous electrode material by the facile method has been discussed. The structural and electrochemical studies confirmed the material's formation. The enhancement in the potential window was found because of PANI incorporation in activated carbon @FeOOH binary composite electrode materials. The electrochemical performance of electrode materials was verified through the cyclic voltammetry, galvanostatic charge-discharge process and the electrochemical impedance spectroscopy. The cyclic voltammetry (CV) and galvanostatic charge-discharge (GCD) examination displayed the specific capacitance of 213 F g^{-1} at 10 mV s^{-1} and 234 F g^{-1} at 2

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mA sec⁻¹ for 20 wt.%. The specific capacitance was found as 234.5 F g⁻¹ at 1 Ag⁻¹ for 20 wt.% PANI composites. The energy (E_d) and power density (P_d) of device were found to be 45 W h kg⁻¹ and 5997 W kg⁻¹ at 2 mA and 20 mA, respectively (Fig. 9). The lab scale fabricated device was tested with LED panel glow. Throughout all the materials prepared and presented in previous chapters of this thesis, the 20% PANI incorporated in activated carbon @FeOOH binary composite electrode exhibited the advanced performance.

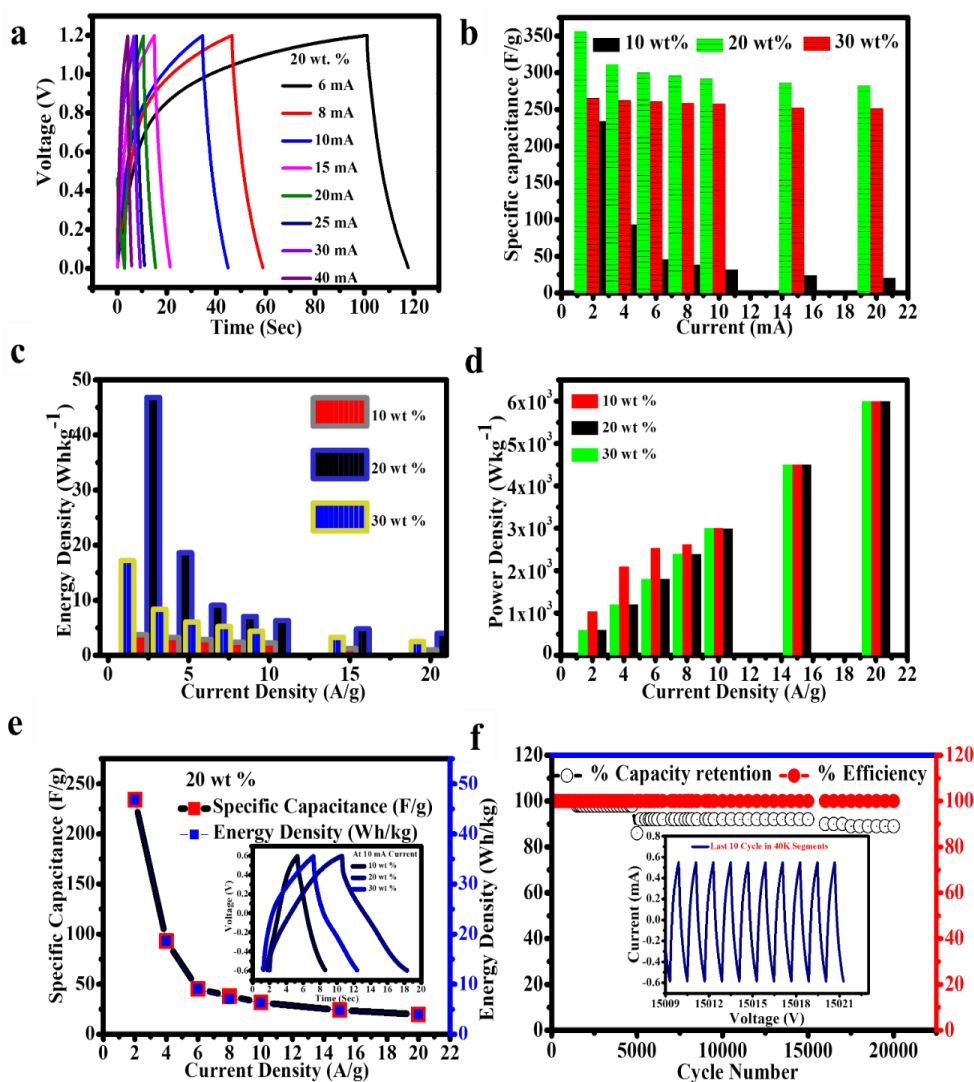


Fig. 9. (a) Charging-discharging pattern of the device (b) Specific Capacitance vs Current for all materials (c) Current density vs E_d (d) Power density vs Current density (e) Specific capacitance, Energy density vs current density, inset shows Charging-discharging pattern of all electrodes. (f) Capacitance retention and Coulombic efficiency, inset displays preceding 10 GCD sequences from 20k cycles.

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Chapter 7 summarizes the entire work of the thesis with concluding remarks and future scope. Low energy density, low life cycle, and lower potential windows are the main challenges in supercapacitor technology. Carbonaceous electrode materials are on the top priority due to advanced retention and high-power density. These materials store charge through electrical polarization (electrostatic charge storage mechanism). The devices that store charge by electrostatic and redox reaction methods show better energy density and long capacitance retention percentage. In the present work, we have successfully incorporated both functionalities by including both types of materials, pure activated carbon and oxyhydroxide, in a cumulative manner, and the materials are known as hybrid nanomaterials. Future scope for further research work in the field of carbonaceous electrode materials, and their energy storage applications has been depicted at the end of this thesis. A study of synthesis, characterization and energy storage properties of carbonaceous nanomaterials is summarized in Table 7.1:

Table 7.1 Chapter-wise Comparison of Optimized Electrode Materials.

Chapter No.	Electrode Materials	V	Specific Capacitance	E_d Wh kg ⁻¹	P_d W kg ⁻¹	Capacitance retention %	Q-Efficiency
2.	Carbonaceous@ZrO ₂	1.0	125 F g ⁻¹	25 Wh kg ⁻¹	1201 W kg ⁻¹	82 % @10000 Cycle	100 %
3.	Activated carbon@FeOOH	1.0	372 F g ⁻¹	40 Wh kg ⁻¹	4200 W kg ⁻¹	80 % @10000 Cycle	96 %
4.	Activated carbon@CoOOH	1.2	225 F g ⁻¹	37.6 Wh kg ⁻¹	4200 W kg ⁻¹	72 % @10000 Cycle	89 %
5.	Mesoporous@FeOOH	1.2	84.35 F g ⁻¹	7.55 Wh kg ⁻¹	5000 W kg ⁻¹	92 % @15000 Cycle	100 %
6.	AC@FeOOH@PANI	1.2	234.0 F g ⁻¹	45 Wh kg ⁻¹	5997 W kg ⁻¹	89 % @20000 Cycle	100 %

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7.2 Future Scope of the Work

After an extensive study of carbonaceous@oxyhydroxide electrode materials, we can say the possibility of device fabrication using the optimized ternary-activated carbon@FeOOH@PANI has been explored. However, it still requires systematic and planned analysis with lots of scope for improvement. The device aspect has to be taken up very rigorously with lots of optimization and fabrication parameters. However, the substantial scope for further improvement in their properties still exists. The following points are key to the above discussion indicating the future scope of the work:

- Extensive possibility of outdoor model of devices based on the activated carbon@oxyhydroxide electrode materials.
- For commercial applications purpose, lots of planned and systematic analysis and testing of devices at different currents is still required.
- The device aspect has to be taken up very rigorously with lots of optimization and fabrication parameters.
- Our studies found that devices comprising these carbonaceous hybrid electrodes have high structured stability, but optimizing with different electrolytes, particularly gel-electrolyte, is needed.
- Other carbonaceous oxyhydroxide hybrid materials e.g. carbonaceous@2D-dichalcogenide electrode materials can be explored for device application by fixing one activated carbon.

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LIST OF PUBLICATIONS

Part of the thesis published in the refereed journals

1. **N. Singh**, S. Tanwar, S. Kour, A. L. Sharma, B.C. Yadav, "Sustainable carbon coated ZrO₂ electrodes with high capacitance retention for energy storage devices." *Journal of Physics D: Applied Physics* 55.42 (2022): 425501.
2. **N. Singh**, S. Tanwar, A. L. Sharma, B. C. Yadav, "Advanced cyclic stability and highly efficient different shaped carbonaceous nanostructured electrodes for solid-state energy storage devices." *International Journal of Hydrogen Energy* 47.66 (2022): 28254-28271.
3. **N. Singh**, S. Tanwar, P. Kumar, A. L. Sharma, B. C. Yadav, "Advanced sustainable solid state energy storage devices based on FeOOH nanorod loaded carbon@PANI electrode: GCD cycling and TEM correlation." *Journal of Alloys and Compounds* 947 (2023): 169580.
4. **N. Singh**, S. Tanwar, A. L. Sharma, B. C. Yadav, "Economic and environment friendly carbon decorated electrode for efficient energy storage devices." *Journal of Energy Storage* 66 (2023): 107452.

Part of the thesis unpublished/communicated in the refereed journals

1. **N. Singh**, S. Tanwar, S. M. S, A. L. Sharma, B. C. Yadav "An Approach to Substitute Costly-Commercial Capattery Electrodes by Activated carbon@Co with advanced retention: Detailed device study Supported by DFT Investigation" *Advanced Composites and Hybrid Materials, Springer Nature*, **Ref: Submission ID** 971e65c-d047-490c-0afe-444fo3f2281, **(I.F=20.1)**.

Work not included in Thesis

1. **N. Singh**, S. Tanwar, B. C. Yadav, A. L. Sharma, "High efficient activated carbon-based asymmetric electrode for energy storage devices." *Materials Today: Proceedings* 57 (2022): 5-10.
2. **N. Singh**, K. Kanwar, S. Tanwar, A. L. Sharma, B. C. Yadav, "Structural, electrical and electrochemical properties of Fe doped orthosilicate cathode materials." *Advanced Functional Materials and Devices: Select Proceedings of AFMD 2021*. Springer Singapore, 2022.

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3. **N. Singh**, S. Tanwar, S. Kour, B.C.Yadav, A. L. Sharma, "Effect of Temperature on Electrochemical Performance of Activated Carbon FeOOH Composite Electrode for Energy Storage Device", *Proceedings of the 65th DAE Solid State Physics Symposium, DAE SSPS 2021*.

Papers presented in International Conferences/Proceedings

1. Participated in National Conference on "**Advanced Nanomaterials and their Applications**" (ANA-2018) Dec 21-23, 2018, at MNNIT Allahabad, Prayagraj U.P.
2. Participated in the "**Young Scientist Conference** as part of **India International Science Festival-2019**" during Nov 5-8, 2019, at Biswa Bangla Convention Centre Kolkata, Kolkata, India.
3. Participated in the 13th "**National Conference On Solid State Ionic (NCSSI-13)**" during Dec 16-18, 2019, at the Department of Physics IIT Roorkee, Roorkee, India.
4. Participated and presented research work as Oral Presentation in **International conference on advanced functional materials and devices (AFMD-2021)** organized by the Department of Physics & IQAC, ARSD College University of Delhi from 03-05th March 2022.
5. Participated and presented research work as a poster presentation at the **International Symposium of Materials of the Millennium: Emerging Trend and Future Prospects (MMETFP-2021)** held at the School of Technology, Pandit Deen Dayal Energy University, Gujrat on Nov 19-21, 2021.
6. Participated in "**Young Researcher Colloquium**" at **International Symposium of Materials of the Millennium: Emerging Trend and Future Prospects (MMETFP-2021)** held at the School of Technology, Pandit Deen Dayal Energy University, Gujrat on Nov 19-21, 2021.
7. Participated and Presented a paper in the **65th DAE Solid State Physics Symposium** Organized by Bhabha Atomic research center, held at Bhabha Atomic Research Centre, Mumbai, during 15 - 19 December 2021 Mumbai Sponsored by: Board of Research in nuclear sciences (BRNS) Department of Atomic Energy, Government of India.

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Awards/ National Level Exams/Certificate

1. Qualified for the **National eligibility test** for “Assistant Professor” **CSIR-UGC NET** December 2017 in Physical Science. **E-Certificate No-172005806.**
2. **Best poster award in National conference** on “Nanomaterials and associated Energy” at BBAU(Central University) during 01-03th Feb 2019.
3. Best Oral presentation award in **International conference (AFMD-2021)** organized by Department of Physics & IQAC, ARSD College University of Delhi during 03-05th March 2021.
4. Awarded Oral presentation award in “**Young researcher colloquium**” at **International Symposium of Materials of the Millennium: Emerging Trend and Future Prospects (MMETFP-2021)** held at the School of Technology, Pandit Deen Dayal Energy University, Gujrat on Nov 19-21, 2022.