

DEVELOPMENT OF FLEXIBLE ELECTRODE FOR BIOELECTROCHEMICAL SYSTEMS

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**SCHOOL OF INTERDISCIPLINARY RESEARCH
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Development of Flexible Electrode for Bioelectrochemical Systems

by

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School of Interdisciplinary Research

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CERTIFICATE

This is to certify that the thesis titled **Development of Flexible Electrode for Bioelectrochemical Systems**, submitted by **Mr. Rahul Kandpal**, to the Indian Institute of Technology, Delhi, for the award of the degree of **Doctor of Philosophy**, is a bonafide record of the research work done by him under our supervision and guidance. The contents of this thesis, in full or in parts, have not been submitted to any other Institute or University for the award of any degree or diploma.



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ABSTRACT

The growing global demand for sustainable wastewater treatment necessitates energy-efficient and resource-recovering technologies that can address pollution while generating bioelectricity. Bioelectrochemical systems (BES), including microbial fuel cells (MFCs), offer a promising solution but suffer from key challenges such as low power output, inefficient electron transfer, and the rigidity of conventional electrodes. The development of flexible, high-performance electrodes is crucial to enhance BES efficiency and scalability, especially for treating real textile wastewater a complex industrial effluent with high organic load and synthetic dyes.

This PhD research focuses on the development of flexible electrode materials tailored for BES applications. Three different fabrication strategies were explored: (i) polycarbonate embedded with carbon black using twin-screw melt extrusion, (ii) gold-sputtered paper-based substrates followed by electrochemical polymerization of polyaniline (PANI), and (iii) polyester microfiber nonwoven (PMN) substrates dip-coated with carbon nanotube (CNT) ink followed by PANI electrochemical polymerization. Among these, the PANI/CNT/PMN electrode demonstrated superior flexibility, electrical conductivity, and microbial compatibility, making it the most effective for real wastewater treatment applications.

The PANI/CNT/PMN flexible electrode was optimized using AC electrochemical polymerization, resulting in a mesoporous structure (BET surface area: 3.2212 m²/g, pore diameter: 65.96 nm) that facilitated enhanced biofilm attachment, electron transfer, and mass transport. Electrochemical characterization confirmed significant improvements in charge storage capacity, reduced impedance (R_{ct} : 2.08 Ω), and high electrocatalytic activity. Cyclic Voltammetry (CV), Differential Pulse Voltammetry (DPV), and Electrochemical Impedance Spectroscopy (EIS) demonstrated the electrode's redox stability and fast electron transfer kinetics, surpassing traditional carbon cloth electrodes.

The developed electrode was tested in a Multielectrode Assembly Bioelectrochemical Reactor (MEABR) coupled with a Biophotovoltaic (BPV) cell for real textile wastewater treatment. The system achieved power and current densities of $1500 \pm 4.5 \text{ mW/m}^2$ and $500 \pm 6.5 \text{ mA/m}^2$, representing a nearly 2000-fold improvement over carbon cloth electrodes. The pollutant removal efficiency was also significantly enhanced, with 85.9% COD removal, 81.06% TOC removal, and 82.7% dye decolorization.

Microbial community analysis using 16S rRNA sequencing revealed that the biofilm on the PANI/CNT/PMN electrode was dominated by an uncultured filamentous, nanowire-producing *Lysinibacillus* strain, known for its high extracellular electron transfer (EET) efficiency. Advanced biofilm characterization, including Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS), provided 3D imaging and depth profiling, identifying malonic acid secretion as a key metabolic byproduct, indicating enhanced biofilm activity and electrochemical performance.

To further optimize biofilm-electrode interactions, twelve different nanomaterials and thin films were integrated into the PANI/CNT/PMN electrode, leading to material-specific microbial enrichment and enhanced biodegradation in the MEABR system. The integration of the BPV cell enabled an energy-positive configuration, demonstrating the feasibility of a self-sustaining wastewater treatment system.

This research presents a scalable and cost-effective approach for integrating flexible electrodes into BES for industrial wastewater remediation, bridging laboratory research with real-world applications. The findings contribute to the advancement of microbial electrochemical technologies, offering a sustainable solution for energy generation, pollutant degradation, and resource recovery. Future research should focus on further optimizing electrode architecture, microbial community engineering, and long-term system stability, particularly exploring the synergistic role of *Lysinibacillus* and other electroactive microbes to enhance BES performance and commercialization potential.

संक्षेप

स्वच्छ जल की वैश्विक मांग में निरंतर वृद्धि के कारण ऊर्जा-कुशल और संसाधन पुनर्प्राप्ति करने वाली जल शोधन तकनीकों की आवश्यकता बढ़ रही है, जो न केवल प्रदूषकों को हटाए बल्कि जैव-विद्युत भी उत्पन्न कर सके। जैव-विद्युत प्रणाली, जैसे कि सूक्ष्मजीव ईंधन कोशिकाएँ, इस दिशा में एक आशाजनक समाधान प्रदान करती हैं, लेकिन कम विद्युत घनत्व, इलेक्ट्रॉन स्थानांतरण की सीमित दक्षता और कठोर इलेक्ट्रोड सामग्री जैसी चुनौतियाँ इसके व्यापक उपयोग को सीमित करती हैं। इस शोध का मुख्य उद्देश्य लचीले और उच्च-प्रदर्शन वाले इलेक्ट्रोड विकसित करना है, जो वास्तविक टेक्स्टाइल अपशिष्ट जल के उपचार के लिए विशेष रूप से उपयुक्त हों। इस शोध में तीन प्रकार के लचीले इलेक्ट्रोड विकसित किए गए:

१. कार्बन ब्लैक युक्त पॉलीकार्बोनेट, जिसे ट्विन-स्कू मेल्ट एक्सट्रूजन द्वारा तैयार किया गया।
 २. सोने की परत चढ़ाए गए पेपर-आधारित सतह, जिस पर पॉलीएनिलीन का विद्युत-रासायनिक पॉलिमराइजेशन किया गया।
 ३. पॉलिएस्टर माइक्रोफाइबर नॉनवॉवन सतह, जिस पर कार्बन नैनोट्यूब स्याही का डिप-कोटिंग किया गया और फिर विद्युत-रासायनिक पॉलिमराइजेशन द्वारा पॉलीएनिलीन की परत बनाई गई।
- इनमें से पॉलीएनिलीन/कार्बन नैनोट्यूब/पॉलिएस्टर माइक्रोफाइबर नॉनवॉवन इलेक्ट्रोड ने अधिक लचीलापन, विद्युत चालकता और सूक्ष्मजीवों की अनुकूलता प्रदर्शित की, जिससे यह वास्तविक अपशिष्ट जल उपचार अनुप्रयोगों के लिए सबसे प्रभावी साबित हुआ। इस इलेक्ट्रोड को विद्युत-रासायनिक पॉलिमराइजेशन द्वारा उन्नत किया गया, जिससे इसकी छिद्रयुक्त संरचना (सतह क्षेत्र: तीन दशमलव दो दो एक दो वर्ग मीटर प्रति ग्राम, छिद्र व्यास: पैसठ दशमलव नौ छह नैनोमीटर) बेहतर हुई। इस इलेक्ट्रोड का परीक्षण बहु-इलेक्ट्रोड संयोजन जैव-विद्युत रिएक्टर और जैव-फोटोवोल्टाइक सेल में किया गया,

जिसमें एक हज़ार पाँच सौ दशमलव चार पाँच मिलीवाट प्रति वर्ग मीटर पावर घनत्व और पाँच सौ दशमलव छह पाँच मिलीएम्पीयर प्रति वर्ग मीटर करंट घनत्व प्राप्त हुआ, जो पारंपरिक कार्बन क्लॉथ इलेक्ट्रोड की तुलना में लगभग दो हज़ार गुना अधिक था। चक्रवृद्धि वोल्टमेट्री, विभेदन पल्स वोल्टमेट्री और विद्युत-रासायनिक प्रतिबाधा स्पेक्ट्रोस्कोपी जैसे विद्युत-रासायनिक विश्लेषणों से इस इलेक्ट्रोड की बेहतर चार्ज भंडारण क्षमता, कम चार्ज स्थानांतरण प्रतिरोध (दो दशमलव शून्य आठ ओम) और उच्च इलेक्ट्रॉन स्थानांतरण दक्षता की पुष्टि हुई। टाइम-ऑफ-फ्लाइट सेकेंडरी आयन मास स्पेक्ट्रोमेट्री द्वारा किए गए गहन अध्ययन से त्रि-आयामी इमेजिंग और जैव-पर्दा की गहराई प्रोफाइलिंग की गई, जिससे मैलोनिक एसिड साव की पहचान हुई, जो सूक्ष्मजीवों की उच्च जैव-विद्युत गतिविधि का संकेत देता है। १६ एस राइबोसोमल आरएनए अनुक्रमण से यह पता चला कि एक विशेष लिसिनिबेसिलस जीवाणु प्रजाति इलेक्ट्रोड पर विकसित सूक्ष्मजीवी जैव-पर्दा में प्रमुख रूप से पाई गई। यह प्रजाति नैनोवायर निर्माण और बाह्य कोशिकीय इलेक्ट्रॉन स्थानांतरण की उच्च दक्षता के लिए जानी जाती है, जो जैव-विद्युत प्रणालियों में ऊर्जा उत्पादन और कार्बनिक पदार्थों के अपघटन को बढ़ाती है। इस उन्नत प्रणाली द्वारा पचासी दशमलव नौ प्रतिशत रासायनिक ऑक्सीजन मांग हटाने, इक्यासी दशमलव शून्य छह प्रतिशत कुल कार्बनिक कार्बन हटाने और बयासी दशमलव सात प्रतिशत रंग विघटन दक्षता प्राप्त हुई, जिससे यह ऊर्जा-सकारात्मक अपशिष्ट जल उपचार समाधान के रूप में स्थापित हुई। इसके अतिरिक्त, इस प्रणाली में बारह विभिन्न नैनो-सामग्रियों और पतली परतों को इलेक्ट्रोड में जोड़ा गया, जिससे विभिन्न सूक्ष्मजीवों की भौतिक-रासायनिक अनुकूलन क्षमता और जैविक गिरावट में वृद्धि देखी गई। यह शोध एक विस्तार योग्य, लागत-प्रभावी और उच्च प्रदर्शन वाली तकनीक को प्रस्तुत करता है, जो औद्योगिक अपशिष्ट जल उपचार और नवीकरणीय ऊर्जा उत्पादन के लिए जैव-विद्युत प्रणालियों के व्यावसायीकरण को गति प्रदान कर सकता है। भविष्य में, इलेक्ट्रोड की सतही संरचना और सूक्ष्मजीव समुदाय की गतिकी पर अधिक ध्यान देकर इस प्रणाली की दक्षता और दीर्घकालिक स्थिरता को और बढ़ाया जा सकता है

Contents

CERTIFICATE	i.
ACKNOWLEDGEMENTS	ii.
ABSTRACT	iv.
List of figures	xi.
List of tables	xxviii.
List of symbols	xxx.
List of abbreviations	xxxii.
1 Introduction	1
1.1 Global water crisis and the need for sustainable wastewater treatment	2
1.2 Overview of Bioelectrochemical System (BES).	6
1.3 Need for flexibility in BES	10
1.4 Integration of textiles in BES	16
1.5 Concept of flexible electrodes in BES	23
1.6 Challenges in textile wastewater treatment and BES limitations	28
1.7 Future research directions in BES and flexible electrodes	31
1.8 Objectives of the present study	36
1.9 Thesis organisation	37
2 Literature review	38
2.1 Global water challenges and the need for sustainable treatment technologies.	39
2.2 Microbial Fuel Cells: Principles, applications, and advantages	40
2.3 Limitations and challenges in MFC technology	42
2.4 Advances in electrode materials for enhanced MFC performance	45
2.5 Microbial interactions and biofilm formation in MFCs.	48
2.6 MFCs for textile wastewater treatment.	50
3 Optimization of Polyaniline electrochemical polymerization using Electrochemical Impedance Spectroscopy	55
3.1 Introduction and sub-objectives	56

3.2	Experimental methodology	62
3.3	Results and discussion	70
3.4	Conclusions	123
4	Flexible electrode fabrication and optimization	125
4.1	Introduction and sub-objectives	126
4.2	Experimental methodology	129
4.3	Characterization of electrodes and results.	143
4.4	Further characterization and optimization of selected electrodes.	159
4.6	Conclusions.	193
5	Electroactive biofilm enrichment from real textile wastewater of dyeing industry	195
5.1	Introduction and sub-objectives	196
5.2	Electrode selection overview	198
5.3	Biofilm enrichment process	200
5.4	Comparative results	204
5.5	Discussion	254
5.6	Conclusions	260
6	From Flexible Electrode to Functional Reactor: A Coupled MEABR–BPV System for Textile Wastewater Treatment and Energy Recovery cell	261
6.1	Introduction and sub-objectives	262
6.2	Evolution from rigid to flexible electrodes	263
6.3	MEABR-BPV system design.	274
6.4	Characterization of modified PANI/CNT/PMN electrodes	282
6.5	Textile wastewater treatment performance	297
6.6	Discussion	326
6.7	Conclusions	331
7	Summary & Conclusions	333
8	Future Scope	338

References	342
List of publications	377
Conferences	379
Awards	382
Brief biodata	384

List of Figures

1	(a) Polyaniline powder, (b) PANI structure, (c) Properties of PANI	56
2	Different oxidation states of PANI	59
3	Schematic illustration of the electrochemical polymerization of polyaniline (PANI) on Au-coated cellulosic paper electrodes using cyclic voltammetry (CV), chronoamperometry (CA), chronopotentiometry (CP), and electrochemical impedance spectroscopy (EIS) techniques	67
4	Illustration of the three-electrode system, showing the platinum working electrode, carbon cloth counter electrode, and Ag/AgCl reference electrode immersed in the 0.1 M aniline in 1 M HCl electrolyte, with the Autolab workstation controlling the AC potential and EIS measurements. Electrochemical polymerization of PANI conducted at different applied potentials (0.55 V to 0.90 V) using electrochemical impedance spectroscopy (EIS)	69
5	Schematic for the electrochemical polymerization of PANI on Au/Si E-QCMD substrate using EIS at different applied potentials ranging from 0.55 V to 0.90 V vs Ag/AgCl in 3 M KCl reference electrode and Pt counter electrode	69
6	Electrochemical polymerization of PANI on Au/Cellulosic paper via CV to obtain PANI _{CV} /Au/Cellulosic paper electrode. CV was performed by using a voltage in the range from -0.2 V to 0.9 V; Scan rate = 50 mV/s; Number of scans = 41 (equivalent to 1800 s); start and stop potential of 0.4 V in a solution containing HCl (1 M) and aniline (0.3 M) to deposit PANI coating over the working electrode. Reference electrode used was made of Ag/AgCl (in saturated KCl solution, 0.197 V vs SHE) while carbon cloth was chosen as a counter electrode.	72
7	Electrochemical polymerization of PANI on Au/Cellulosic paper via CA to obtain PANI _{CA} /Au/Cellulosic paper electrode. CA was performed using HCl (1 M) and aniline (0.3 M) solutions on Au/Paper working electrode by keeping the same reference electrode (Ag/AgCl, saturated KCl, 0.197 V vs SHE) and carbon cloth was employed as a counter electrode by applying at a potential of 0.75 V for 1800 s to get a current vs time curve for the electrochemical polymerization of PANI	72
8	Electrochemical polymerization of PANI on Au/Cellulosic paper via CP to obtain PANI _{CP} /Au/Cellulosic paper electrode. CP was performed using the same concentration of HCl and aniline solutions on working (Au/Paper), reference (Ag/AgCl, sat. KCl, 0.197 V vs SHE) and counter (carbon cloth) electrodes at a set current of 800 μ A for 1800 s to get a potential vs time curve of PANI polymerization	73
9	Electrochemical polymerization of aniline on Au/Cellulosic paper via EIS to obtain PANI _{EIS} /Au/Cellulosic paper electrode. EIS was carried out by using the same concentration of HCl and aniline (as mentioned above) along with working electrode, reference and counter electrodes by applying an AC potential of 0.75 V for 1800 s to polymerize PANI on Au/Paper. The AC signal frequency range utilized for EIS operation was from 100 kHz to 0.1 Hz	74

10	Visual comparison of different PANI films grown by different electrochemical polymerization techniques	76
11	(a–d) Nyquist plots of PANI films electrochemically polymerized on Au/Cellulosic paper using (a) CV, (b) CA, (c) CP, and (d) EIS techniques. (e) Comparison table of polarization resistance (R_p) values obtained from equivalent circuit fitting of EIS data, highlighting the superior performance of the PANIEIS/Au/Cellulosic paper electrode with the highest percentage reduction in R_p (83.95%), indicative of enhanced charge transfer characteristics. (f) Bar graph comparison of R_p values across the different polymerization techniques (CV, CA, CP, and EIS), further emphasizing the advantage of the EIS-based AC method in achieving low polarization resistance and improved electrochemical conductivity.	77
12	SEM images of (A) PANI/Au/Cellulosic paper and (B) PANI/Au/Nylon paper electrodes synthesized via different electrochemical polymerization techniques: (1) CV, (2) CA, (3) CP, and (4) EIS, along with (5) control (Au/Cellulosic paper), shown at different magnifications (a–e). Cellulose fibres ($\sim 40\ \mu\text{m}$ diameter) were irregularly distributed, leading to non-uniform PANI deposition and motivating the use of relatively flatter Nylon paper as an alternative substrate. Among all techniques, EIS produced the most uniform and well-adhered PANI coating, highlighting the superiority of AC-based methods over DC-based techniques	79
13	SEM images of PANI/Au/Nylon paper electrodes synthesized via (a) CV, (b) CA, (c) CP, and (d) EIS. CV, CA, and CP produced granular and fibrous PANI layers with average grain diameters ranging from ~ 500 – $765\ \text{nm}$, whereas EIS yielded fibrous PANI structures of larger diameter ($\sim 1.12\ \mu\text{m}$). The micron-scale fibrous morphology obtained by EIS provided a porous network favourable for exoelectrogenic biofilm attachment and growth	81
14	(a) FE-SEM image of PANI/Au/Nylon paper electrode synthesized via EIS at 5 kx magnification showing the formation of interconnected PANI microfibers. (b) High-magnification FE-SEM image (20 kx) highlighting granular morphology of PANI grains distributed along the microfibers. (c) SEM image indicating the location selected for point EDX analysis. (d) EDX spectrum confirming the elemental composition of PANI with predominant carbon (C), nitrogen (N), and chlorine (Cl), validating the successful electrochemical polymerization and doping of PANI via EIS	82
15	Atomic force microscopy (AFM) images ($20\ \mu\text{m} \times 20\ \mu\text{m}$) of PANI/Au/Nylon paper electrodes synthesized via different electrochemical polymerization techniques: (a) CV, (b) CA, (c) CP, and (d) EIS. The calculated surface areas were $798.9\ \mu\text{m}^2$, $625.5\ \mu\text{m}^2$, $555.1\ \mu\text{m}^2$, and $779.8\ \mu\text{m}^2$, respectively. CV produced the highest surface area due to granular morphology, while EIS generated a uniform fibrous structure with high stability, as also validated by SEM analysis (Figures 12 and 13)	84
16	FTIR spectra of (a) PANI/Au/Nylon paper electrode and (b) control Au/Nylon paper. Characteristic absorption peaks of PANI are observed at $\sim 1618\ \text{cm}^{-1}$ and $\sim 1466\ \text{cm}^{-1}$ (quinoid and benzenoid ring stretching), $1298\ \text{cm}^{-1}$ (C–N stretching of aromatic amine), $1108\ \text{cm}^{-1}$ (C–H in-plane bending of quinoid ring), and a broad band at $3440\ \text{cm}^{-1}$ (N–H stretching of $-\text{NH}_2$ groups). These bands confirm	85

	the successful polymerization and deposition of PANI onto Au/Nylon paper, while the absence of such peaks in the control spectrum validates the absence of PANI in bare Au/Nylon paper	
17	(a) Confocal microscopic image of Au/Nylon paper showing the yellow coloration of the sputtered gold layer. (b) Confocal microscopic image of PANI/Au/Nylon paper synthesized via electrochemical polymerization using the EIS technique, displaying a light green coloration indicative of successful PANI deposition. (c) Raman spectra of Au/Nylon and PANI/Au/Nylon electrodes recorded at 785 nm excitation. The PANI/Au/Nylon electrode exhibited characteristic bands at $\sim 1595\text{ cm}^{-1}$ ($\nu(\text{C}=\text{C})$ stretching of quinoid rings, $\nu(\text{C}=\text{N})$ imine stretching, $\delta(\text{N}-\text{H})$ deformation), $\sim 1460\text{ cm}^{-1}$ (benzenoid ring vibration), and $\sim 1332\text{ cm}^{-1}$ (semi-benzenoid polaronic $\nu(\text{C}-\text{N}^+\bullet)$), along with $-\text{CH}$ in-plane bending vibrations below 1200 cm^{-1} , confirming the presence of doped polyaniline. In contrast, the Au/Nylon spectrum showed weaker features at $\sim 1599\text{ cm}^{-1}$ and $\sim 1336\text{ cm}^{-1}$ with no imine or $\delta(\text{N}-\text{H})$ vibrational peaks, validating the absence of PANI.	87
18	Frequency Response Analysis-Electrochemical Impedance Spectroscopy (FRA-EIS) time scan plots for PANI electrochemically polymerized on a Pt wire at different applied DC potentials: (a) 0.9 V, (b) 0.85 V, (c) 0.80 V, (d) 0.75 V, (e) 0.70 V, (f) 0.65 V, (g) 0.60 V, and (h) 0.55 V	93
19	Representative differential pulse voltammograms (a-h) of PANI samples (synthesized on Pt wire) at different AC potentials (0.90 V to 0.55 V). The highlighted box (PANI@800mV and PANI@850mV) shows the dominant peaks in the E1 and E2 regions, characteristic of the desired emeraldine oxidation state.	95
20	(a-h) FE-SEM images for PANI (synthesized on Pt wire) at different AC potentials (0.90 V to 0.55 V), (i) Atomic % of different elements from EDX, (j) C/N ratio of PANI samples. (k) Average fiber diameter of PANI synthesized at different potentials	97
21	FE-SEM for PANI synthesized at an AC potential of 0.90 V by electrochemical polymerization using EIS (425 nm avg fiber diameter SD: 0.02269)	98
22	FE-SEM for PANI synthesized at an AC potential of 0.85 V by electrochemical polymerization using EIS (387.5 nm avg fiber diameter SD: 0.05483)	98
23	FE-SEM for PANI synthesized at an AC potential of 0.80 V by electrochemical polymerization using EIS (505.1 nm avg fiber diameter SD: 0.0793)	99
24	FE-SEM for PANI synthesized at an AC potential of 0.75 V by electrochemical polymerization using EIS (425.7 nm avg fiber diameter SD: 0.02778)	99
25	FE-SEM for PANI synthesized at an AC potential of 0.70 V by electrochemical polymerization using EIS (509.1 nm avg fiber diameter SD: 0.05638)	100
26	FE-SEM for PANI synthesized at an AC potential of 0.65 V by electrochemical polymerization using EIS (528.3 nm avg fiber diameter SD: 0.04869)	100
27	FE-SEM for PANI synthesized at an AC potential of 0.60 V by electrochemical polymerization using EIS (871.4 nm avg fiber diameter SD: 0.24731)	101
28	FE-SEM for PANI synthesized at an AC potential of 0.55 V by electrochemical polymerization using EIS (928.1 nm avg fiber diameter SD: 0.07461)	101

29	SEM-EDX for PANI synthesized at an AC potential of 0.90 V by electrochemical polymerization using EIS	102
30	SEM-EDX for PANI synthesized at an AC potential of 0.85 V by electrochemical polymerization using EIS	103
31	SEM-EDX for PANI synthesized at an AC potential of 0.80 V by electrochemical polymerization using EIS	104
32	SEM-EDX for PANI synthesized at an AC potential of 0.75 V by electrochemical polymerization using EIS	105
33	SEM-EDX for PANI synthesized at an AC potential of 0.70 V by electrochemical polymerization using EIS	106
34	SEM-EDX for PANI synthesized at an AC potential of 0.65 V by electrochemical polymerization using EIS	107
35	SEM-EDX for PANI synthesized at an AC potential of 0.60 V by electrochemical polymerization using EIS	108
36	SEM-EDX for PANI synthesized at an AC potential of 0.55 V by electrochemical polymerization using EIS	109
37	(a) FTIR and (b) Raman spectra for PANI synthesized at different AC potentials	111
38	(a) I-1580/I-1190 ratio as a function of PANI polymerization potential (b) I-1040/I-1190 ratio as a function of PANI polymerization potential	111
39	Raman spectra for PANI synthesized at different AC potentials by electrochemical polymerization using EIS from 1100 cm ⁻¹ to 1700 cm ⁻¹	112
40	(a-h) X-Ray Diffraction (XRD) and (i-p) Thermogravimetric Analysis (TGA) for PANI synthesized at different AC potentials	115
41	Comparative analysis of TGA data for PANI samples electrochemically polymerized at 8 different potentials	116
42	(a-h) HR-TEM for PANI synthesized at different AC potentials, (i) Average particle size and zeta potential for PANI synthesized at different AC potentials	117
43	Fixed-time deposition of PANI films on Au/Cr/SiO ₂ EQCM substrates at different applied potentials vs Ag/AgCl reference electrodes	120
44	AFM conducted on PANI film coated Au/Cr/SiO ₂ EQCM substrate to calculate the surface roughness of PANI film followed by the measurement of film thickness using Ellipsometry	121
45	Substrates utilized for the fabrication of flexible electrodes	127
46	Chemical structure of polycarbonate (PC), a thermoplastic polymer commonly used as a flexible substrate matrix. The repeating carbonate linkage (–O–(C=O)–O–) in the backbone contributes to its mechanical strength, flexibility, and thermal stability, making it suitable for embedding conductive fillers during electrode fabrication.	130
47	Schematic representation of carbon black (CB) particles, illustrating their nanoscale morphology and characteristic surface features. The diagram highlights functional groups such as carboxyl (–COOH) and hydroxyl (–OH) present on the particle surface, which enhance interfacial interactions with polymer matrices. Key physicochemical attributes such as primary particle size, aggregation, and high specific surface area are also indicated, all of which play a critical role in	131

	governing the electrical conductivity and dispersion of CB in composite electrodes.	
48	Schematic representation of the chemical synthesis of polyaniline (PANI) via oxidative polymerization of aniline in acidic medium using ammonium persulfate as the oxidant, highlighting the formation of the conductive emeraldine salt	132
49	(a) Schematic for conical twin-screw extruder and (b) Xplore Micro Compounder MC 15 used for melt extrusion of polycarbonate/carbon black composites, illustrating the twin-screw configuration that enables uniform dispersive and distributive mixing for optimal filler distribution.	132
50	Schematic for the electrode fabrication process using the twin screw melt extrusion and the table showing loading percentage of CB in PC	134
51	Schematic for the electrode fabrication process using the paper based substrates	136
52	Schematic illustration of the flexible electrode fabrication process using Polyester Microfiber Nonwoven (PMN) textile based substrate	140
53	Schematic illustration of the flexible electrode fabrication process: (a) PMN substrate preparation through cutting, washing, and drying; (b) CNT ink preparation and stable dispersion by probe sonication; (c) sequential dip-coating of PMN to achieve uniform CNT coverage; (d) AC electrochemical polymerization of PANI on CNT/PMN using a three-electrode cell configuration; and (e) schematic of complete process showing uncoated PMN substrate followed by CNT and PANI coatings	141
54	Variation of electrical resistance of PC/CB composite films as a function of CB loading (0–50 wt%). A sharp decrease in resistance is observed between 17–33 wt% CB, indicating the percolation threshold where a continuous conductive network forms within the polymer matrix. Measurements were performed using a Keysight U1731C handheld LCR meter at 1 kHz with 1 cm probe spacing.	145
55	Dependence of flexural rigidity on carbon black (CB) weight fraction in PC/CB composite electrodes. Flexural rigidity was determined from bending length measurements obtained using the Paramount Stiffness Tester. An increase in CB content beyond 33 wt% results in a pronounced rise in flexural rigidity, reflecting reduced mechanical compliance. Sample E (33 wt% CB) represents the optimized composition, achieving a balance between sufficient mechanical flexibility and enhanced electrical conductivity.	147
56	Electrical resistance of paper-based electrodes measured using a handheld LCR meter (1 kHz). The plot compares bare cellulosic and nylon paper, gold-sputtered electrodes, and electrodes further modified with electrochemically polymerized PANI, highlighting the substantial reduction in resistance achieved by Au and PANI deposition	150
57	Flexural rigidity of paper-based electrodes. The bending stiffness increases progressively with gold sputtering and subsequent PANI deposition on both cellulosic and nylon papers. PANI/Au/cellulosic paper exhibits the highest rigidity, while PANI/Au/nylon paper maintains lower flexural rigidity, indicating greater mechanical compliance	152
58	(a) Flexural rigidity (μNm) and (b) electrical resistance ($\text{M}\Omega$) of PMN-based electrodes with successive CNT coating cycles and PANI polymerization. The	156

	plots illustrate the progressive increase in mechanical stiffness and the corresponding decrease in electrical resistance, highlighting the effect of CNT reinforcement and PANI deposition on the mechanical and electrical properties of the electrodes	
59	Nyquist plot for electrochemical polymerization of PANI on Au-coated cellulosic paper substrate, showing the variation in charge transfer resistance (R_{ct}) during polymerization. The data illustrate the influence of the Au–cellulose interface on deposition kinetics and uniformity	160
60	Nyquist plot for electrochemical polymerization of PANI on Au-coated nylon paper substrate. The results highlight the effect of substrate morphology and porosity on R_{ct} and the controlled growth of PANI films	160
61	Nyquist plot for electrochemical polymerization of PANI on CNT-coated PMN substrate. The CNT coating significantly reduces R_{ct} and facilitates uniform polymer growth due to improved conductivity and surface area	161
62	Raman spectra of (a) Au-coated Nylon paper and (b) PANI/Au/Nylon paper electrodes. The Au/Nylon paper shows characteristic substrate peaks, while the PANI-coated electrode exhibits new bands corresponding to quinoid and benzenoid ring vibrations, C–N stretching, and –CH bending, confirming successful PANI polymerization and formation of the conductive emeraldine salt form	167
63	Raman spectra of (a) pristine PMN fabric, (b) CNT-coated PMN, and (c) PANI/CNT/PMN electrodes. The PMN spectrum shows characteristic PET peaks, while CNT coating introduces D and G bands indicative of high-quality CNTs with low defect density. Deposition of PANI on CNT/PMN results in new peaks corresponding to benzenoid and quinoid vibrations, confirming successful polymerization and formation of the conductive emeraldine salt form.	169
64	FESEM images of various electrodes showing surface morphology and polymer/CNT deposition. (a) Au/Cellulosic paper showing the fibrous structure of the cellulose substrate. (b) Magnified view of a single cellulosic fiber from (a). (c) PANI/Au/Cellulosic paper at the same magnification as (a) illustrating uniform PANI coating over multiple fibers. (d) Magnified view of a single cellulosic fiber from (c) highlighting granular PANI deposition. (e, f) PANI/Au/Nylon paper showing complete coverage of nylon fibers with granular PANI. (g) CNT-coated PMN fabric with magnified view of a PET microfiber, CNT agglomerates indicated by arrows. (h, j) PANI/CNT/PMN electrodes; (h) magnified view showing PANI grains over the CNT network, (j) densely packed PMN fabric with PANI grains and CNT coating on PET microfibers, demonstrating uniform deposition and continuous conductive pathways.	171
65	EDX analyses of different electrodes: (a) point EDX spectrum of a single PANI grain in PANI/Au/Nylon and PANI/Au/Cellulosic paper, confirming the presence of C, N, O, and Au; (b) elemental mapping of PANI/CNT/PMN showing uniform distribution of C, N, O, Na, Mn, S, Cl, K, Cr, and Fe across the matrix; (c) elemental mapping of CNT/PMN highlighting C, O, Na, S, and Al; (d) elemental mapping of pristine PMN showing dominant C and O signals; (e) point EDX	173

	spectra of a CNT agglomerate versus bare PET microfiber in PMN, showing significantly higher C intensity at CNT-coated sites, confirming CNT deposition	
66	FESEM and EDX analysis of CNT/PMN electrodes. (a) FESEM image of single-walled CNT-coated PET microfibers with a diameter of 11 μm , highlighting regions for point EDX analysis. (b, c) Point EDX spectra for spots 1 and 2, showing the elemental distribution of CNT coatings. (d, e) FESEM images of multi-walled CNTs utilized for electrode scalability. (f-i) EDX elemental mapping of multi-walled CNTs, confirming uniform distribution on the electrode surface	175
67	Energy-dispersive X-ray spectroscopy (EDX) spectra for (a) PMN fabric, (b) CNT-coated PMN, and (c) PANI/CNT/PMN electrodes. The spectra illustrate the presence of nitrogen from PANI and carbon enrichment from CNTs	176
68	Elemental composition analysis of PANI/CNT/PMN, CNT/PMN, and PMN fabric electrodes using energy-dispersive X-ray spectroscopy (EDX). The weight percentages of carbon, oxygen, nitrogen, and other elements confirm the successful incorporation of PANI and CNTs onto the PMN substrate	177
69	Cyclic voltammogram of the PANI/Au/Cellulosic paper electrode recorded in 1 M HCl (pH 7) over a potential window of -0.3 V to $+0.9\text{ V}$ vs. Ag/AgCl at scan rate (50 mV/s). Distinct redox peaks corresponding to the leucoemeraldine-emeraldine and emeraldine- pernigraniline transitions of PANI are observed, confirming electroactive behaviour of the PANI film on the cellulosic paper substrate	179
70	Cyclic voltammogram of the PANI/Au/Nylon paper electrode under identical conditions (1 M HCl, -0.3 to $+0.9\text{ V}$, Scan rate: 50 mV/s). The CV curves show clear anodic and cathodic peaks characteristic of PANI redox transitions, indicating good reversibility and stable electrochemical activity of the PANI layer on the nylon paper substrate	180
71	Cyclic voltammogram and Differential Pulse Voltammogram of the PANI/CNT/PMN electrode (PBS, pH 7, ferro/ferricyanide, -0.3 to $+0.9\text{ V}$ vs. Ag/AgCl, scan rate: 10 mV/s). The CV curves exhibit pronounced redox peaks corresponding to PANI doping/de-doping, with increasing current response at higher scan rates, demonstrating good reversibility, high electroactive surface area, and excellent charge storage capability of the composite electrode	181
72	Cyclic voltammogram of the PANI/CNT/PMN electrode (PBS, pH 7, ferro/ferricyanide, -0.3 to $+0.9\text{ V}$ vs. Ag/AgCl, scan rates: 1, 10, 100 mV/s). The CV curves exhibit pronounced redox peaks corresponding to PANI doping/de-doping, with increasing current response at higher scan rates, demonstrating good reversibility, high electroactive surface area, and excellent charge storage capability of the composite electrode	182
73	Nyquist plot of the PANI/CNT/PMN electrode recorded in PBS containing ferro/ferri ions (pH 7). The spectrum displays a depressed semicircle in the high-to-mid frequency region, attributed to charge transfer resistance (R_{ct}), followed by a Warburg tail at low frequencies representing ion diffusion. The fitted curve (solid line) corresponds to the equivalent circuit model shown in Figure 44. The extracted R_{ct} value was $2.08\ \Omega$, demonstrating the superior charge transfer kinetics of the PANI/CNT/PMN hybrid electrode	184

74	Equivalent circuit (RCR) used to fit the Nyquist spectrum of the PANI/CNT/PMN electrode. Here, R_s represents the solution resistance, R_{ct} the charge transfer resistance, and C_{dl} the double-layer capacitance. Fitting yielded an R_{ct} of 2.08 Ω , confirming efficient interfacial charge transfer and validating the high conductivity of the CNT–PANI composite structure	185
75	3D AFM topography of PANI/Au/Cellulosic paper electrode (20 × 20 μm scan) obtained using Gwyddion software. The projected surface area was measured as 708.8 μm^2 , showing a uniform PANI layer with nanoscale roughness favorable for microbial attachment	186
76	3D AFM topography of PANI/Au/Nylon paper electrode (20 × 20 μm scan) generated with Gwyddion. The projected surface area was calculated as 779.8 μm^2 , indicating higher surface roughness compared to Cellulosic paper, beneficial for electroactive interface development	187
77	Advantages of flexible electrodes in microbial fuel cells (MFCs). Flexible electrodes enhance electron transfer through conducting polymers and graphene, improve mechanical stability by allowing bending and moulding, support better biofilm formation by accommodating natural microbial growth patterns, increase electrode-biofilm contact area, and enable scalable and versatile designs for diverse MFC configurations	197
78	Comparison of paper-based substrates (cellulosic and nylon) with PMN fabric for bioelectrochemical system (BES) applications. Unlike nonwoven PMN fabric, which provides a 3D porous structure with sufficient interstitial space to support microbial colonization and biofilm development, cellulosic and nylon papers have limited surface area and compact morphology, restricting microbial attachment and growth	199
79	(a) Schematic representation of the experimental workflow for electroactive biofilm growth and application on flexible electrodes (PANI/Au/Nylon paper and PANI/CNT/PMN). The process includes: Task 1 – Biofilm enrichment using chronoamperometry; Task 2 – Measurement of electrochemical parameters (current density, power density, charge, resistance, and pH); Task 3 – Characterization of biofilm-coated electrodes; and Task 4 – Application of enriched biofilm for treatment of real textile wastewater from the dyeing industry. (b) Experimental setup for chronoamperometric biofilm growth showing the tested working electrodes (PANI/Au/Cellulosic paper, PANI/Au/Nylon paper, PANI/CNT/PMN, and carbon cloth as a standard control). The schematic also highlights textile wastewater composition (pH 7.51, BOD 1340 mg/L, soluble COD 1281 mg/L) and identified dye constituents, along with the three-electrode reactor configuration (WE: PANI/Au/Nylon paper, PANI/Au/PMN and carbon cloth, CE: carbon cloth, RE: Ag/AgCl in 3 M KCl).	202
80	Chronoamperometric response (current vs. time) recorded during electroactive biofilm enrichment on the PANI/Au/Nylon paper electrode. The gradual increase in current over time reflects microbial colonization and extracellular electron transfer activity, confirming progressive biofilm growth and establishment of electroactive species on the electrode surface	206

81	Chronoamperometric monitoring of electroactive biofilm enrichment on the carbon cloth electrode. The figure presents correlated parameters illustrating biofilm growth and electron transfer dynamics over time: (a) Current density and pH vs. time showing microbial activity and its influence on bulk solution chemistry; (b) Power density and pH vs. time indicating energy recovery trends; (c) Charge accumulation and pH vs. time reflecting progressive electron harvesting; (d) Current density and resistance vs. time showing the inverse relationship between microbial activity and interfacial resistance; (e) Power density and resistance vs. time correlating power output with reduced charge transfer resistance; (f) Resistance and pH vs. time capturing biofilm-induced conductivity shifts alongside metabolic acidification/neutralization; (g) Current density and charge vs. time demonstrating direct electron recovery as a function of biomass growth; (h) Power density and charge vs. time illustrating overall electrochemical efficiency; and (i) Resistance and charge vs. time highlighting conductivity improvements concurrent with enhanced charge storage	209
82	Chronoamperometric monitoring of electroactive biofilm enrichment on the PANI/CNT/PMN electrode. The figure depicts the evolution of multiple interdependent electrochemical and physicochemical parameters during biofilm growth: (a) Current density and pH vs. time showing microbial activity and pH modulation in the bulk medium; (b) Power density and pH vs. time highlighting energy recovery under changing biochemical conditions; (c) Charge accumulation and pH vs. time demonstrating progressive electron harvesting with metabolic shifts; (d) Current density and resistance vs. time capturing the interplay between microbial electron transfer and interfacial conductivity; (e) Power density and resistance vs. time correlating enhanced power output with declining charge transfer resistance; (f) Resistance and pH vs. time showing conductivity improvements influenced by biofilm-mediated chemical processes; (g) Current density and charge vs. time illustrating cumulative electron recovery with biofilm growth; (h) Power density and charge vs. time reflecting electrochemical efficiency and energy conversion; and (i) Resistance and charge vs. time validating conductivity gains with enhanced charge storage	211
83	Power density comparison of biofilm-enriched electrodes. The PANI/CNT/PMN electrode exhibited a ~2000-fold increase in power density compared to the standard carbon cloth electrode, demonstrating its superior performance and applicability in bioelectrochemical systems. PANI/Au/Nylon paper showed moderate power output, highlighting the advantage of high-surface-area, flexible electrodes for efficient microbial fuel cell operation	213
84	Electrochemical characterization of biofilm-coated PANI _{EIS} /Au paper electrodes. (A, B) Low-scan-rate non-turnover cyclic voltammograms (CV, -1 V to +1 V) of biofilm on PANI _{EIS} /Au/cellulosic paper and PANI/EIS/Au/nylon paper, respectively, illustrating redox activity of the electroactive biofilm. (C, D) Differential pulse voltammograms (DPV) for biofilm on cellulosic and nylon substrates, highlighting peak currents associated with electron transfer. (E) DPV comparison of biofilm-coated and bare PANI _{EIS} /Au/nylon paper, demonstrating enhanced electrochemical activity due to biofilm enrichment	216

85	Electrochemical analysis of carbon cloth electrodes. (a) Low-scan-rate cyclic voltammograms (CV, 1 mV/s) of bare carbon cloth and biofilm-coated carbon cloth, showing the development of redox-active sites due to biofilm enrichment. (b) Differential pulse voltammograms (DPV) of bare and biofilm-coated carbon cloth, highlighting enhanced peak currents and electron transfer activity induced by the electroactive biofilm	217
86	Electrochemical impedance spectroscopy of carbon cloth electrodes. (a) Nyquist plots of bare carbon cloth and biofilm-coated carbon cloth, showing reduced charge transfer resistance and enhanced electron transfer after biofilm enrichment. (b) Bode plots for bare and biofilm-coated carbon cloth, illustrating changes in impedance magnitude and phase angle, indicating improved electrochemical activity and biofilm-mediated conductivity	218
87	Electrochemical analysis of PANI/CNT/PMN electrodes. (a) Low-scan-rate cyclic voltammograms (CV, 1 mV/s) of bare PANI/CNT/PMN and biofilm-coated PANI/CNT/PMN, showing well-defined redox peaks indicative of robust extracellular electron transfer and enhanced electroactivity after biofilm enrichment. (b) Differential pulse voltammograms (DPV) of bare and biofilm-coated PANI/CNT/PMN, demonstrating increased peak currents and improved charge transfer, confirming the development of a highly electroactive biofilm on the high-surface-area flexible electrode	220
88	Electrochemical impedance spectroscopy of PANI/CNT/PMN electrodes. (a) Nyquist plots of bare and biofilm-coated PANI/CNT/PMN, showing increased impedance after biofilm enrichment. (b) Bode plots illustrating the enhanced impedance and altered phase behavior, reflecting the resistance contributed by the thick electroactive biofilm across the 3D electrode surface	221
89	FESEM images showing biofilm microarchitecture on polymer-coated electrodes: (A) Biofilm/PANI/Au/Cellulosic paper, exhibiting sparse and less mature biofilm formation; (B) Biofilm/PANI/Au/Nylon paper, demonstrating denser, well-structured, and more mature biofilm development, highlighting the effect of substrate morphology and conductivity on microbial adhesion and growth	223
90	FESEM and EDX analysis of electroactive biofilms enriched from textile wastewater on carbon cloth (CC) electrodes (a–f). Images show heterogeneous biofilm coverage with bridge-like extracellular structures spanning carbon fibers, highlighting microbial adhesion and electron transfer pathways across the electrode surface	225
91	Morphological and compositional analysis of PANI/CNT/PMN electrodes. (a) FESEM of uncoated PMN fabric showing the porous microfibrinous structure. (b, f) CNT coating uniformly deposited on the PMN substrate. (c, e) Dense PANI layer on CNT/PMN fabric. (d, g, h) Electroactive biofilm formed on PANI/CNT/PMN electrodes, exhibiting homogeneous coverage	226
92	FESEM images of matured anodic biofilm enriched on carbon cloth electrodes during textile wastewater treatment. The images highlight biofilm morphology and attachment to the electrode surface	227
93	FESEM and EDX analysis of electroactive biofilms enriched from textile wastewater on (a–d) PANI/CNT/PMN electrodes. The images highlight biofilm	228

	morphology and elemental mapping, emphasizing nitrogen-rich regions associated with extracellular matrix components	
94	Optical and Raman characterization of biofilm interaction with PANI/Au/Nylon paper electrodes. (A) Optical micrograph of bare Au/Nylon paper showing smooth fiber texture. (B) PANI/Au/Nylon paper exhibiting uniform greenish coating of electrochemically deposited PANI. (C) Biofilm/PANI/Au/Nylon paper showing dense biofilm attachment over the conductive surface. (D) Comparative Raman spectra of Au/Nylon paper, PANI/Au/Nylon paper, and Biofilm/PANI/Au/Nylon paper highlighting chemical interactions and peptide–PANI bonding in the biofilm-coated electrode	231
95	Raman spectra of (1) biofilm-coated carbon cloth electrodes, and (2) bare carbon cloth showing characteristic D and G bands and additional peaks corresponding to EPS and protein-related vibrations indicative of biofilm activity	233
96	Characterization of PANI/CNT/PMN electrodes. (a–d) Raman spectra of (a) pristine PMN substrate, (b) CNT-coated PMN, (c) PANI/CNT/PMN composite electrode, and (d) biofilm-enriched PANI/CNT/PMN electrode, showing characteristic molecular vibrations corresponding to each modification and biofilm interaction	236
97	ToF-SIMS and 3D morphological analysis of PANI/CNT/PMN electrode. (a) 2D surface distribution of key components: CNT, PANI, and SDBS. (b) 3D depth profile showing the spatial arrangement of CNT, PANI, and SDBS across the electrode. (c) Quantitative depth profiles of selected molecular fragments. (d) Representative ToF-SIMS spectra highlighting characteristic peaks of the composite materials. (e) FESEM image of the analyzed area (100 × 100 μm ²) with a 3D representation of the PANI/CNT layers on PET microfibers	238
98	ToF-SIMS analysis of electroactive biofilm-coated carbon cloth electrodes enriched from textile wastewater. (a) Reconstructed 3D image of the biofilm, showing dense cellular distribution on the electrode surface. (b) Visualization of malonic acid (highlighted in magenta) secreted by the biofilm and superimposed on the 3D image. (c) Intensity vs. m/z ratio plot, identifying peaks corresponding to biofilm components. (d) Detection of malonic acid at m/z = 22.9858, confirmed via Wiley Know-It-All software spectral matching. (e) Complete ToF-SIMS spectra for the biofilm-coated electrode, illustrating the chemical composition of the biofilm matrix.	240
99	Phylum-level microbial community composition in the biofilm enriched on the Biofilm/PANI/Au/Nylon paper electrode. The pie chart represents the relative abundance (%) of major bacterial phyla identified through 16S rRNA sequencing, including Proteobacteria (53.36%), Actinobacteria (28.93%), Acidobacteria (8.19%), Planctomycetes (5.77%), Firmicutes (1.91%), and Synergistetes (1.12%), along with minor taxa grouped as “Others.” The accompanying table lists the absolute abundance values for each phylum, providing a quantitative overview of community distribution across taxonomic levels	243
100	Class-level microbial community composition in the biofilm enriched on the Biofilm/PANI/Au/Nylon paper electrode	244

101	Order-level microbial community composition in the biofilm enriched on the Biofilm/PANI/Au/Nylon paper electrode	245
102	Order-level microbial community composition in the biofilm enriched on the Biofilm/PANI/Au/Nylon paper electrode	246
103	Genus-level microbial community composition in the biofilm enriched on the Biofilm/PANI/Au/Nylon paper electrode	247
104	Species-level microbial composition of the biofilm enriched on the Biofilm/PANI/Au/Nylon paper electrode. The pie chart and accompanying table depict the relative abundance (%) of dominant unclassified species identified via 16S rRNA sequencing from the genera, including Mycobacterium (28.37%), Achromobacter (15.28%), unclassified genera (13.81%), Pseudoxanthomonas (6.94%), Ochrobactrum (4.89%), Alicyclophilus (4.18%), and Pseudomonas (3.93%), among others. This representation highlights the prevalence of electroactive and dye-degrading genera within the enriched biofilm community	248
105	Microbial community structure of the biofilm enriched on Biofilm/PANI/Au/Nylon paper electrodes. (a) Krona chart depicting the hierarchical taxonomic distribution from phylum to genus level, highlighting the dominance of Proteobacteria, Actinobacteria, and other phyla. (b) Phylogenetic tree illustrating the evolutionary relationships among the major genera detected in the biofilm, emphasizing the presence of electroactive and dye-degrading microorganisms	249
106	Taxonomic distribution of microbial communities, presented at the phylum levels, illustrating the diversity of electroactive microorganisms within the biofilm-coated carbon cloth electrode and the microbial population present in the untreated textile wastewater	251
107	Taxonomic distribution of microbial communities, presented at the genus levels, illustrating the diversity of electroactive microorganisms within the biofilm-coated carbon cloth electrode and the microbial population present in the untreated textile wastewater	252
108	Fabrication of Rigid PANI-Based Nanocomposite Electrodes and MFC Assembly (a–f) Sequential fabrication steps of polyaniline (PANI)-based nanocomposite anodes comprising combinations of CNTs, MOFs, graphite (G), and TiO ₂ powders, blended with wax and compressed into 2 cm discs via cold pressing. The workflow illustrates weighing, mixing, and pressing stages for electrode preparation. (g–h) Preparation of air-breathing cathodes coated with activated carbon and PTFE binder on stainless steel mesh, ensuring enhanced oxygen diffusion and hydrophobic stability. (i) Complete single-chamber MFC assembly incorporating multiple anode types operated under identical conditions for comparative evaluation. (j) Schematic of the MFC configuration showing electrode placement, current flow, and bioelectrochemical reaction pathways. The modular setup enabled simultaneous screening of nine electrode compositions to study material-driven microbial enrichment, electrochemical behavior, and power output	264
109	Electrochemical Performance of MFCs with Multiple Rigid PANI-Based Nanocomposite Anodes (a) Voltage generation profiles over multiple semi-	267

- continuous batch cycles for MFCs operated with different anode compositions: PANI–CNT/G/TiO₂, PANI–CNT/MOF/G, PANI–CNT/MOF, PANI/G/TiO₂, PANI/CNT/G, PANI–CNT/TiO₂, PANI/MOF/G, PANI/MOF, and PANI–CNT/MOF/G/TiO₂. Distinct stabilization trends correspond to varying microbial community development on each electrode surface. (b) Corresponding power density curves derived from polarization analyses showing differences in output performance as a function of electrode conductivity and composition. Hybrid electrodes integrating CNTs, MOFs, and graphite demonstrated superior current output and sustained voltage stability due to enhanced electron transfer and biofilm-electrode coupling
- 110 Comparative Power Output of Different PANI-Based Nanocomposite Electrodes Across MFC Operation Cycles. Bar graph summarizing the maximum power densities (mW/m²) achieved by each electrode composition over successive MFC operational cycles. The PANI–CNT/MOF anode exhibited the highest performance (322 mW/m²), followed closely by PANI–CNT/MOF/G (204.42 mW/m²) and PANI–CNT/G/TiO₂ (181.89 mW/m²). Electrodes containing CNTs and MOFs consistently outperformed those lacking conductive nanofillers, emphasizing the synergistic role of conductivity and porosity in facilitating extracellular electron transfer (EET). The figure highlights the material-specific influence of composite architecture on bioelectrocatalytic efficiency and demonstrates the strong correlation between electrode conductivity and power generation capacity 268
- 111 Schematic illustration of the PANI/CNT/PMN electrode fabrication and integration into the MEABR–BPV system. Process overview showing: (a) fabrication and modification of the flexible PANI/CNT/PMN electrode through CNT coating and electrochemical polymerization of polyaniline on polyester microfiber nonwoven (PMN); (b) assembly of the Multielectrode Assembly Bioelectrochemical Reactor (MEABR) comprising multiple flexible anodes and a common cathode for parallel electrochemical operation; (c) enrichment of electroactive biofilm on PANI/CNT/PMN electrodes under controlled potential; and (d) subsequent coupling of MEABR with a Bio-Photovoltaic (BPV) cell for real textile wastewater treatment. The BPV cell enhances oxygen evolution and light-assisted cathodic reactions, enabling improved degradation efficiency and sustainable energy recovery in the integrated system 274
- 112 (a) Schematic of MEABR showing thirteen vertical flexible anodes, a shared cathode (counter electrode), wastewater inlet/outlet, and flow path, (b) Diagram of MEABR reactor paired with BPV cell with anode chamber (algae on PANI/CNT/PMN electrode), cathode/counter electrode (carbon cloth), and glass chamber for sunlight exposure 275
- 113 FTIR spectra of the nanomaterials and nanocomposites used for fabricating the modified PANI/CNT/PMN electrodes, including PANI–CNT–TiO₂, rGO, GO, PANI–MOF(Zn- based), PANI–TiO₂, Ag nanoparticles, carbon black, and CNT. The spectra highlight the characteristic functional groups, including C–O, C=O, O–H, Ti–O, and Zn–O, confirming the successful integration and functionalization of the materials. Peaks are annotated to demonstrate the unique 284

	chemical signatures contributing to enhanced electrochemical properties and surface interactions	
114	FESEM and EDX analysis of the PANI/CNT/PMN electrode, showing uniform surface morphology and elemental distribution of nitrogen, carbon, and oxygen, confirming successful integration of PANI and CNTs onto the PMN substrate	286
115	FESEM and EDX images of the PANI-TiO ₂ /PANI/CNT/PMN electrode, highlighting the incorporation of TiO ₂ nanoparticles and their uniform distribution across the electrode surface, enhancing its electrochemical and biofilm-supporting properties	287
116	FESEM and EDX analysis of the PANI-CNT-TiO ₂ /PANI/CNT/PMN electrode, showing the co-presence of CNTs and TiO ₂ nanoparticles, contributing to increased surface area and improved conductivity for bioelectrochemical applications	288
117	FESEM and EDX characterization of the PANI-MOF (Zn-based)/PANI/CNT/PMN electrode, revealing the presence of Zn along with carbon and nitrogen, indicative of successful incorporation of Zn-MOF into the electrode matrix for enhanced microbial interaction	289
118	FESEM and EDX analysis of the rGO/PANI/CNT/PMN electrode, confirming the integration of reduced graphene oxide (rGO) with PANI and CNTs. The increased carbon content reflects improved conductivity and surface properties	290
119	FESEM and EDX images of the GO/PANI/CNT/PMN electrode, showing the distribution of oxygenated groups from graphene oxide (GO) along with PANI and CNTs, contributing to enhanced hydrophilicity and biofilm attachment	291
120	FESEM and EDX characterization of the Carbon Black/PANI/CNT/PMN electrode, highlighting the presence of carbon black particles and their uniform dispersion, which improve the electrode's conductivity and electrochemical performance	292
121	FESEM and EDX analysis of the Pt/PANI/CNT/PMN electrode, illustrating the presence of platinum nanoparticles embedded within the PANI/CNT matrix, enhancing catalytic activity and electron transfer efficiency	293
122	FESEM and EDX analysis of the Cu/PANI/CNT/PMN electrode, indicating uniform distribution of copper nanoparticles on the electrode surface, contributing to improved redox behaviour and microbial enrichment	294
123	FESEM and EDX characterization of the Ag/PANI/CNT/PMN electrode, revealing the presence of silver nanoparticles alongside PANI and CNTs. The addition of silver enhances antimicrobial properties and conductivity	295
124	FESEM and EDX images of the Au/PANI/CNT/PMN electrode, confirming the incorporation of gold nanoparticles into the electrode matrix, contributing to enhanced electrochemical activity and biofilm-supporting characteristics	296
125	FESEM and EDX analysis of the electrode containing All nanomaterials/Cu/Pt/Ag/Au integrated with PANI/CNT/PMN electrode. The analysis shows a synergistic distribution of materials, enhancing the electrode's multifunctionality for bioelectrochemical applications	297
126	(a-i) current, power, charge, resistance, and pH vs time plot for whole cell assembly of multielectrode assembly bioelectrochemical reactor for the treatment	301

- of real textile wastewater, (j) Current vs time plot for individual electrodes generating current from real textile wastewater, and (k) power vs time plot for individual electrodes generating power from real textile wastewater
- 127 Electroactive biofilm enriched PANI/CNT/PMN electrode generating electricity from real textile wastewater in multielectrode assembly BES: (a) Current density and pH vs time, (b) Power density and pH vs time, (c) Charge and pH vs time, (d) Current density and Resistance vs time, (e) Power density and Resistance vs time, (f) Resistance and pH vs time, (g) Current density and Charge vs time, (h) Power density and Charge vs time, (i) Resistance and Charge vs time curves 303
- 128 Electroactive biofilm enriched PANI-TiO₂/PANI/CNT/PMN electrode generating electricity from real textile wastewater in multielectrode assembly BES: (a) Current density and pH vs time, (b) Power density and pH vs time, (c) Charge and pH vs time, (d) Current density and Resistance vs time, (e) Power density and Resistance vs time, (f) Resistance and pH vs time, (g) Current density and Charge vs time, (h) Power density and Charge vs time, (i) Resistance and Charge vs time curves 304
- 129 Electroactive biofilm enriched PANI-CNT-TiO₂/PANI/CNT/PMN electrode generating electricity from real textile wastewater in multielectrode assembly BES: (a) Current density and pH vs time, (b) Power density and pH vs time, (c) Charge and pH vs time, (d) Current density and Resistance vs time, (e) Power density and Resistance vs time, (f) Resistance and pH vs time, (g) Current density and Charge vs time, (h) Power density and Charge vs time, (i) Resistance and Charge vs time curves 305
- 130 Electroactive biofilm enriched PANI-MOF/PANI/CNT/PMN electrode generating electricity from real textile wastewater in multielectrode assembly BES: (a) Current density and pH vs time, (b) Power density and pH vs time, (c) Charge and pH vs time, (d) Current density and Resistance vs time, (e) Power density and Resistance vs time, (f) Resistance and pH vs time, (g) Current density and Charge vs time, (h) Power density and Charge vs time, (i) Resistance and Charge vs time curves 306
- 131 Electroactive biofilm enriched rGO/PANI/CNT/PMN electrode generating electricity from real textile wastewater in multielectrode assembly BES: (a) Current density and pH vs time, (b) Power density and pH vs time, (c) Charge and pH vs time, (d) Current density and Resistance vs time, (e) Power density and Resistance vs time, (f) Resistance and pH vs time, (g) Current density and Charge vs time, (h) Power density and Charge vs time, (i) Resistance and Charge vs time curves 307
- 132 Electroactive biofilm enriched GO/PANI/CNT/PMN electrode generating electricity from real textile wastewater in multielectrode assembly BES: (a) Current density and pH vs time, (b) Power density and pH vs time, (c) Charge and pH vs time, (d) Current density and Resistance vs time, (e) Power density and Resistance vs time, (f) Resistance and pH vs time, (g) Current density and Charge vs time, (h) Power density and Charge vs time, (i) Resistance and Charge vs time curves 308

133	Electroactive biofilm enriched Carbon Black/PANI/CNT/PMN electrode generating electricity from real textile wastewater in multielectrode assembly BES: (a) Current density and pH vs time, (b) Power density and pH vs time, (c) Charge and pH vs time, (d) Current density and Resistance vs time, (e) Power density and Resistance vs time, (f) Resistance and pH vs time, (g) Current density and Charge vs time, (h) Power density and Charge vs time, (i) Resistance and Charge vs time curves	309
134	Electroactive biofilm enriched Pt/PANI/CNT/PMN electrode generating electricity from real textile wastewater in multielectrode assembly BES: (a) Current density and pH vs time, (b) Power density and pH vs time, (c) Charge and pH vs time, (d) Current density and Resistance vs time, (e) Power density and Resistance vs time, (f) Resistance and pH vs time, (g) Current density and Charge vs time, (h) Power density and Charge vs time, (i) Resistance and Charge vs time curves	310
135	Electroactive biofilm enriched Cu/PANI/CNT/PMN electrode generating electricity from real textile wastewater in multielectrode assembly BES: (a) Current density and pH vs time, (b) Power density and pH vs time, (c) Charge and pH vs time, (d) Current density and Resistance vs time, (e) Power density and Resistance vs time, (f) Resistance and pH vs time, (g) Current density and Charge vs time, (h) Power density and Charge vs time, (i) Resistance and Charge vs time curves	311
136	Electroactive biofilm enriched Ag/PANI/CNT/PMN electrode generating electricity from real textile wastewater in multielectrode assembly BES: (a) Current density and pH vs time, (b) Power density and pH vs time, (c) Charge and pH vs time, (d) Current density and Resistance vs time, (e) Power density and Resistance vs time, (f) Resistance and pH vs time, (g) Current density and Charge vs time, (h) Power density and Charge vs time, (i) Resistance and Charge vs time curves	312
137	Electroactive biofilm enriched Au/PANI/CNT/PMN electrode generating electricity from real textile wastewater in multielectrode assembly BES: (a) Current density and pH vs time, (b) Power density and pH vs time, (c) Charge and pH vs time, (d) Current density and Resistance vs time, (e) Power density and Resistance vs time, (f) Resistance and pH vs time, (g) Current density and Charge vs time, (h) Power density and Charge vs time, (i) Resistance and Charge vs time curves	313
138	Electroactive biofilm enriched PMN fabric generating electricity from real textile wastewater in multielectrode assembly BES: (a) Current density and pH vs time, (b) Power density and pH vs time, (c) Charge and pH vs time, (d) Current density and Resistance vs time, (e) Power density and Resistance vs time, (f) Resistance and pH vs time, (g) Current density and Charge vs time, (h) Power density and Charge vs time, (i) Resistance and Charge vs time curves	314
139	Electroactive biofilm enriched All nanomaterials/Cu/Pt/Ag/Au/PANI/CNT/PMN electrode generating electricity from real textile wastewater in multielectrode assembly BES: (a) Current density and pH vs time, (b) Power density and pH vs time, (c) Charge and pH vs time, (d) Current density and Resistance vs time, (e)	315

	Power density and Resistance vs time, (f) Resistance and pH vs time, (g) Current density and Charge vs time, (h) Power density and Charge vs time, (i) Resistance and Charge vs time curves	
140	Peak current and power generated for individual electroactive biofilm enriched electrodes in the MEABR reactor	316
141	Dye decolorization from real textile wastewater using the Biofilm/CC, Biofilm/PANI/CNT/PMN, MEABR, BPV, and MEABR + BPV sequential reactor	319
142	TOC for the samples (1) Untreated textile wastewater and, (2) Treated textile wastewater was measured using TOC analyzer. The report for the TOC experiment is attached below. TOC for untreated textile wastewater was found to be 1769 mg/L and for the treated textile wastewater was found to be 335.1 mg/L. Hence a TOC removal efficiency of 81.06 % was observed	325

List of Tables

1	Comparison of Conventional and BES Treatment Methods	5
2	Timeline for different discoveries in the field of BES	7
3	Comparison of Rigid and Flexible Electrodes in BES	15
4	Comparison of Materials for Textile-Based Electrodes in BES	23
5	Comparison of Flexible Electrode Designs in BES	28
6	Comparison of Textile Wastewater Challenges and BES Limitations	31
7	Future Research Priorities for BES and Flexible Electrodes	35
8	Assignment of Raman peaks of PANI samples	113
9	Weight of initial substrates and percentage weight gain after sequential gold sputtering and PANI electrochemical polymerization for cellulosic and nylon paper electrodes. The data illustrate the mass increase associated with each modification step, reflecting deposition efficiency and material loading on the respective substrates	122
10	Weight of the PMN substrate and percentage weight gain after successive CNT dip-coating cycles (1–5) and subsequent PANI electrochemical polymerization. The data reflect the incremental material deposition at each stage, providing insight into the buildup of conductive layers and their contribution to electrode properties	138
11	Bending length, mass per unit area, and calculated flexural rigidity of PC/CB composite films with varying carbon black concentrations. The data illustrate the effect of increasing carbon black content on the mechanical stiffness of the composites	142
12	Comparison of charge transfer resistance (R_{ct}) values for various PANI-based flexible electrodes. The present study reports significantly lower R_{ct} for PANI _{EIS} polymerized CNT/PMN and Au/paper electrodes, indicating enhanced conductivity compared to literature-reported electrodes	146
13	Charge transfer resistance (R_{ct}) values for various PANI-based flexible electrodes. The present study reports significantly lower R_{ct} for PANI _{EIS} polymerized CNT/PMN and Au/paper electrodes, indicating enhanced conductivity compared to literature-reported electrodes	166
14	Comparison of different substrate materials and fabrication processes for electrode development in bioelectrochemical systems (BES). The table summarizes the advantages and disadvantages of polymer-based, paper-based, and polyester microfiber nonwoven (PMN) textile-based substrates, highlighting key trade-offs in conductivity, flexibility, surface area, cost, and biodegradability for their application in BES	185
15	Experimental techniques employed for electroactive biofilm characterization on flexible electrodes in BES. The table outlines electrochemical, microscopic,	199

- spectroscopic, and molecular biology methods used to evaluate biofilm enrichment, redox activity, morphology, elemental composition, microbial community structure, and 3D chemical imaging
- 16 Comparative power density of electroactive biofilm-enriched electrodes. The 204
 PANI/CNT/PMN electrode exhibited the highest power density (1500 mW/m²), followed by PANI/Au/Nylon paper (296 mW/m²), while conventional carbon cloth showed the lowest performance (71.43 mW/m²), emphasizing the impact of electrode material on BES output
- 17 Comparison of Power densities obtained from PANI/CNT/PMN and CC 212
 electrodes in BES reactor with the existing literature
- 18 Dye decolorization and COD removal efficiencies for various biofilm reactor 256
 configurations including Biofilm/CC, Biofilm/PANI/CNT/PMN, MEABR, BPV, and sequential MEABR + BPV evaluated under both peak and minimum current density conditions during real textile wastewater treatment. The table summarizes the quantitative performance metrics for each configuration, facilitating direct comparison of organic and dye pollutant removal in single-electrode, multi-electrode, biophotovoltaic, and integrated hybrid systems
- 19 Dye decolorization and COD removal efficiencies for various biofilm reactor 318
 configurations including Biofilm/CC, Biofilm/PANI/CNT/PMN, MEABR, BPV, and sequential MEABR + BPV evaluated under both peak and minimum current density conditions during real textile wastewater treatment. The table summarizes the quantitative performance metrics for each configuration, facilitating direct comparison of organic and dye pollutant removal in single-electrode, multi-electrode, biophotovoltaic, and integrated hybrid systems

List of symbols

R, R_{ct}, R_s	Resistance, charge transfer resistance, solution resistance (Ω)
I, I_{peak}	Current, peak current (A, mA, μ A)
V, E	Voltage, potential (V, mV)
J	Current density (mA/cm ²)
η	Overpotential (V)
t	Time (seconds, minutes)
A	Electrode area (cm ²)
C, C_{dl}	Capacitance, double-layer capacitance (F)
Z	Impedance (Ω)
Q	Electric charge (Coulombs)
n	Number of electrons transferred (unitless)
D	Diffusion coefficient (cm ² /s)
θ	Contact angle (degrees)
σ	Conductivity
λ	Wavelength
ρ	Density
ϕ	Work function or phase angle

List of Terminology used

BES	Bioelectrochemical System
MFC	Microbial Fuel Cell
MEC	Microbial Electrolysis Cell
MES	Microbial Electro Synthesis
PEM	Proton Exchange Membrane
Substrate	It can be the substrate present in wastewater or the substrate/base material used for electrode preparation
PMN	Polyester Microfiber Nonwoven (acts as substrate for electrode fabrication)
Electroactive microbe	An electroactive microbe is a type of microorganism capable of transferring electrons to or from an electrode, effectively "interfacing" with electrical systems. These microbes can naturally generate electric currents through their metabolic processes
Exoelectrogen/Electricigen	An exoelectrogen is a type of electroactive microorganism that can transfer electrons to an external electrode during its metabolic processes
PANI	Polyaniline (PANI) is an electrically conducting polymer
MEABR	Multi Electrode Assembly Bioelectrochemical Reactor
BPV	Bio-Photo-Voltaic cell
EET	Extracellular Electron Transfer
COD	Chemical Oxygen Demand (mg/L)
TOC	Total Organic Carbon (mg/L)
16S rRNA	Ribosomal RNA gene marker for microbial identification
EPS	Extracellular Polymeric Substances (biofilm matrix components)
Biofilm	Microbial layer on electrode surfaces
CV	Cyclic Voltammetry
DPV	Differential Pulse Voltammetry
EIS	Electrochemical Impedance Spectroscopy
CA	Chronoamperometry
CP	Chronopotentiometry

FRA	Frequency Response Analysis
FESEM	Field Emission Scanning Electron Microscopy
EDX	Energy Dispersive X-ray Spectroscopy
XRD	X-ray Diffraction
FTIR	Fourier Transform Infrared Spectroscopy
ToF-SIMS	Time-of-Flight Secondary Ion Mass Spectrometry
CNT	Carbon Nanotubes
PC	Polycarbonate
Au	Gold
TiO ₂	Titanium Dioxide
rGO	Reduced Graphene Oxide
GO	Graphene Oxide
MOF	Metal-Organic Framework
CB	Carbon Black