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Optimization and Electrochemical Evaluation of PVA assisted Electrospun Polyindole Nanofibers for Supercapacitors

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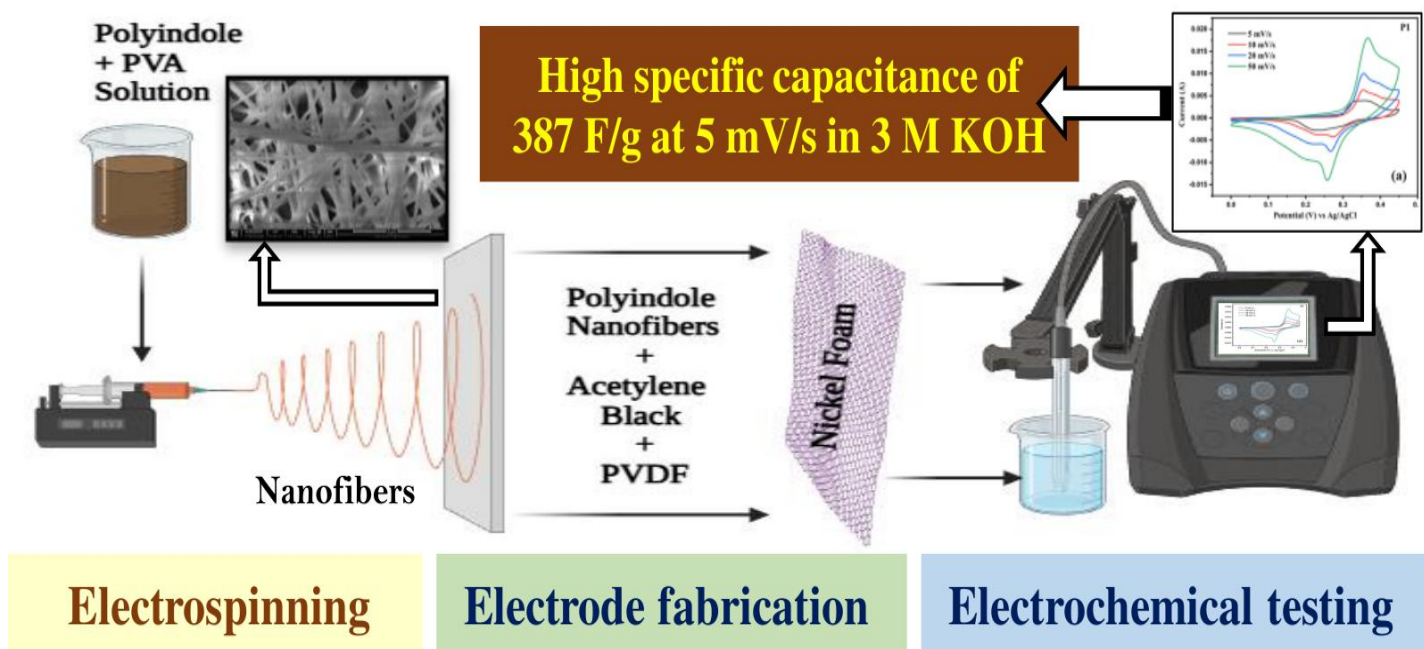
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Abstract

Conducting polymer nanostructures exhibit good theoretical capacitance but their 1D nanostructuring remains a challenge. In this study, polyindole nanofibers were successfully fabricated by electrospinning method using PVA as carrier polymer. For the investigation of its chemical, structural and morphological properties, XRD, FTIR and FESEM techniques were used. The electrochemical performance was evaluated by CV and GCD assessment in 3 M KOH electrolyte. XRD results confirmed the amorphous nature of polyindole whereas characteristic functional groups and vibrational modes were identified by FTIR. FESEM images revealed non-woven, bead free uniform nanofibers with average diameter ranging between 200-300 nm. Among the three electrodes, P2 exhibited highest specific capacitance of 387 F/g at scan rate of 5 mV/s and 356.7 F/g at 0.5 A/g from CV and GCD profiles respectively. Areal capacitance exhibited by P2 was found to be 1161 mF/cm² at 5 mV/s. Electrochemical study shows overall pseudocapacitive behavior of all three samples with significant super capacitance retained at higher scan rates and increased current densities, indicating good rate capability. Overall, these findings demonstrate the potential of as such fabricated electrospun polyindole nanofibers as a promising electrode material for supercapacitor applications.

Keywords: *Electrospinning, Polyindole, Nanofiber, CV, Supercapacitance, Supercapacitor*

Graphical abstract



1. Introduction

The demand for energy is rising day by day around the globe due to its different usage in fields like industry, transport, electronic instruments and electric vehicles. At the same time, renewable energy resources are not reliable to that extent [1]. To overcome this issue, energy storage systems like supercapacitors and batteries are good alternatives owing to their promising electrochemical properties. But although traditional batteries provide high energy density, they have constraints of slow charging time, low power density and limited cycle life [2,3]. On the other hand, supercapacitor offers higher power density, faster charging time and improved cycling stability making them promising alternative for next generation energy storage applications [3,4].

Pseudocapacitors, in particular, achieves improved energy density due to the presence of redox active electrodes evidencing the importance of choosing suitable active materials [2]. Conducting polymers have gained significant attention as active material for pseudocapacitor due to its fast redox kinetics, tunable conductivity, mechanical flexibility and predominant high theoretical capacitance [4–6]. Polyaniline (PANI), polypyrrole (PPy), polythiophene (PTh) and poly (3,4-ethylenedioxythiophene) (PEDOT) are widely used conducting polymers owing to their good electrical conductivity and easy processability [2,7]. However, despite having their advantages, many conducting polymers exhibit limited mechanical stability, minimal electrochemical cycle life and insufficient surface area particularly when conventional electrode fabrication methods or non-conductive binders are used [6,7]. Polyindole (PIn), a heterocyclic conducting polymer which is composed of fused benzene-pyrrole structure which integrates the characteristics of both polypyrrole and poly(p-phenylene) [8–10]. When compared with PANI and PPy, PIn demonstrated improved thermal and environmental stability, high redox activity, slower degradation rates indicating its high suitability for sustainable energy storage as its fused ring aromatic structure play a role in improved overall electrochemical behavior [5,10].

But relatively low intrinsic conductivity issue along with rigid polymer chains of PIn create challenges during synthesis and nanostructuring [11]. However, these challenges can be addressed through various approaches such as adaptation of better synthesis methods [12], development of binary or ternary composites [12–15], and application of novel electrode fabrication techniques which yield materials with high surface area and enhance electrochemical performance which is known to be morphology dependent as unfavorable morphology can

alter desired performance [13,16,17]. Hence to overcome these limitations, a simple and scalable electrospinning technique was used for controlled fabrication of PIn nanofibers with high surface area and thereby improving their conductivity and overall electrochemical performance. These electrospun nanofibers exhibit high porosity and large surface to volume ratios making them a suitable choice for high performance supercapacitor electrodes [11,18]. The electrospinning of polyindole is quite challenging due to its rigid backbone, its low solubility and insufficient chain entanglement which is necessary for fiber formation. To resolve this issue, some carrier polymers like PVA, PEO, PVP and PAN are incorporated with conducting polymers to support uniform and bid free electrospinning. Nevertheless, these carrier polymers can affect the electrical conductivity therefore their minimum incorporation must be optimized [11,19]. PVA is proven suitable carrier polymer due to its electrospinnability and stability, in contrast PVP and PAN have certain limitations [20]. Despite possessing enhanced surface area and doping-dedoping efficiency compared to bulk PIn, research regarding application of electrospun PIn nanofibers in energy storage remains limited [11,17,19–22].

When compared to PIn polymer powder, the electrospun PIn nanofiber membranes reported by Zhijiang and co-workers [23] exhibited higher specific surface area, improved electronic conductivity and better doping/dedoping characteristics highlighting importance of nanofiber morphology. Similarly, Khaled *et al.* [19] and Gergin *et al.* [24] used polyacrylonitrile (PAN) as a carrier polymer in dimethylformamide (DMF) solution in order to electrospun PIn by producing nanofibers with higher surface area and enhanced electronic conductivity. As a result, electrospun PIn nanofibers with different carrier polymers stand out as a fiber polymer electrode for energy storage devices [23] and sensors [24].

From overall reported data, it was found that PIn exhibits specific capacitance in the range from 21.80 to 343.20 F/g [11,21,25–30]. In case of freestanding electrospun PIn and PIn/CNT electrodes specific capacitance was significantly increased up to 238 F/g and 476 F/g respectively for energy density of 1.0 A/g. where PIn:PEO blend was used [11]. In another reported study [21] using PIn:PEG blend, a specific capacitance of 198 F/g at 2 mV/s was obtained, which highlights the crucial influence of carrier polymer on the overall electrochemical behaviour. But despite all favorable electrochemical properties shown by PIn, research on its morphology-controlled nanostructures, particularly electrospun nanofiber remains very limited [1,5,10].

In previously reported studies, most of the research was focused around bulk PIn or alternatively emphasis was given to its composite materials. Addition of conductive materials like carbon black, graphene and metal oxides is well known for improved electrode performance, where sometimes complex fabrication methods were also used to achieve that. For pure PIn or PIn based structures, morphological changes have also found useful for better results but they come with some limitations like low electroactive surface area, poor intrinsic conductivity and limited ion and electron transport [11,31]. Hence, in-depth study of the structural, morphological and electrochemical properties of electrospun PIn nanofibers is essential to evaluate their suitability to serve as high performance supercapacitor electrodes [32,33].

The present study aims to fabricate electrospun polyindole nanofibers with PVA using electrospinning technique and evaluate their electrochemical performance for supercapacitor electrode applications. Nanofibers were prepared using different PVA:PIn ratios to study the effect of structure and morphology on electrochemical properties.

Field-emission scanning electron microscopy (FE-SEM) was employed for morphological insights of prepared nanofibers, while their structural composition and functional groups were identified through X-ray Diffraction (XRD) and Fourier-transform infrared spectroscopy (FTIR). EDX analysis was used to confirm elemental composition. The fabricated electrodes were tested in 3 M KOH electrolyte for electrochemical assessment, where specific capacitance was calculated using both cyclic voltammetry and galvanostatic charge-discharge (GCD) techniques. As a result, these electrodes of electrospun PIn/PVA nanofibers exhibited high specific capacitance in the range of 200-400 F/g surpassing the performance of pure polyindole electrodes reported in many earlier studies.

This work highlights the role of nanostructuring and optimized carrier-to-conducting polymer ratios, which yielded ultrafine nanofibers (~200-300 nm) compared to the ~640 nm fibers in the only reported study on PVA-assisted polyindole nanofiber by Mike *et al.* [17], where stainless steel yarn was used as substrate and the study primarily investigates the effect of carbon black addition in Polyindole based system. This result was accompanied by our high specific capacitance of 297 mF cm⁻² compared to documented 5.40 to 8.79 mF cm⁻² at same scan rate of 50 mV/s. However, to the best of our knowledge, no comprehensive study has yet been reported that systematically investigates the electrospun polyindole nanofibers using PVA as a carrier

polymer integrated with porous nickel-foam electrodes for supercapacitor applications. The present study bridges the existing gaps in nanostructured polyindole research by providing insights for compositional optimization while investigating the electrochemical performance of PVA-based electrospun polyindole nanofibers for energy storage applications.

2. Experimental work

2.1. Materials

Acetone (99.5%), ethanol (99.7–100%), ferric chloride (98%), indole (99%), poly(vinyl alcohol), N,N-dimethylformamide (DMF), N-methyl-2-pyrrolidone (NMP), acetylene black, and poly(vinylidene fluoride) (PVDF) were of analytical grade and used as received without further purification. All solutions used in the synthesis were prepared using double-distilled (DD) water to maintain purity. PVA has been used for electrospinning without any further modification.

2.2. Synthesis of Polyindole (PIn)

Polyindole (PIn) was synthesized via the oxidative polymerization method using indole as the monomer and ferric chloride (FeCl_3) as the oxidizing agent. A 1 M solution of indole was prepared in an aqueous medium and kept for continuous stirring for several minutes to ensure homogeneity. Subsequently, a 1 M solution of FeCl_3 was prepared separately and then added dropwise to the monomer solution kept under continuous stirring. Then reaction mixture was stirred at for 4 hours at room temperature to facilitate polymerization. Further keeping it undisturbed overnight, the mixture exhibited a dark brown colour, indicating the successful formation of PIn. In next stage, obtained product was filtered and washed thoroughly with DI water and acetone in order to remove any residual impurities. Finally, the resultant product of PIn was kept at 70°C for 3 hours for drying in hot air oven and lastly crushed it into a fine powder using a mortar and pestle [34]. This synthesis procedure is illustrated in Fig. 1.

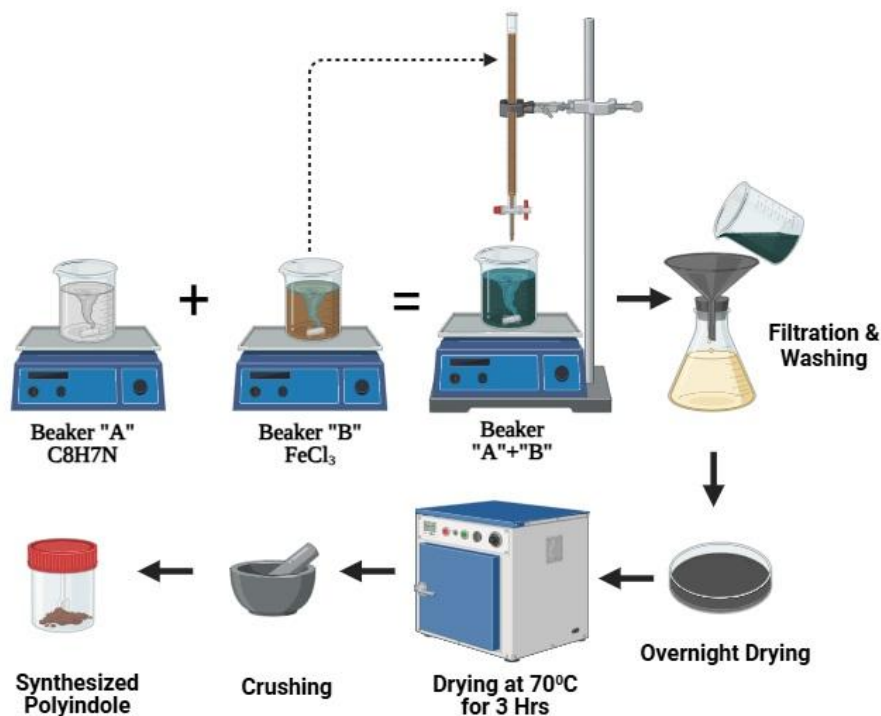


Fig. 1 : Schematic illustration of synthesis of Polyindole.

2.3. Preparation of Polyindole Nanofibers

Electrospun polyindole (PIn) nanofibers were fabricated using electrospinning technique (E-Spin Nanotech) where PVA assisted as carrier polymer for proper electrospinning. Firstly, a 7wt% PVA solution was prepared by dissolving PVA into DI water under continuous stirring until it gets transparent. Separately, polyindole powder was dispersed in dimethylformamide (DMF) to obtain uniform mixture. Then the blend of PIn/DMF were gradually introduced into PVA solution with varying PVA:PIn mass ratios of 1:0.5, 1:1 and 1:1.5 named as P1, P2 and P3 respectively. These blends were kept under constant stirring to get electro spinnable polymer mixtures followed by 10-minute ultrasonication to ensure complete homogenization. Finally, the resultant solutions were loaded into a syringe with 21G gauge needle and electrospun under optimized conditions: an applied voltage of 20-25 kV, flow rate of 1 mL/h, and needle tip to collector drum distance of 12 cm. The nanofibers were collected on an aluminium foil mounted on rotating drum collector operating at 1200 rpm to ensure uniform fiber deposition throughout the foil. Post electrospinning, for stabilization of as-spun nanofiber mats, overnight thermal treatment was given in hot air oven at 70⁰ C for improving their structural integrity and evaporation of residual solvent. Finally free standing non-woven electrospun polyindole nanofibers were obtained suitable for further characterization and electrochemical assessment.

2.4. Electrode fabrication

Owing to its high conductivity and porous structure, $1 \times 1 \text{ cm}^2$ nickel foam was employed as current collector for fabrication of working electrodes. The nickel foam substrates were thoroughly cleaned by ultrasonication in 1 M HCl and acetone, followed by rinsing with DD water and drying at 60°C for removing any contaminants prior use. For the fabrication of electrodes, active material, acetylene black and PVDF were combined in 80:10:10 weight ratio respectively, using NMP as solvent as per the standard procedure. Approximately 3 mg of the dry composite material was used to prepare the slurry and homogeneously coated on pretreated nickel foam using doctor blade method. As-prepared electrodes were then dried in a vacuum oven for 12 hours at 60°C for ensuring proper adhesion of active material to nickel foam and complete solvent evaporation. These electrodes were used as working electrodes in electrochemical characterization [35].

Since the acetylene black and PVDF ratio, and hence the total mass loading were kept constant for all three electrodes (P1-P3), the differences in their electrochemical performance can be primarily attributed to the variation in the PVA:PIIn ratio and the resulting changes in fiber characteristics.

2.5. Characterization techniques and measurements

Multiple analytical techniques were used for systematic investigation of synthesized nanofibers for their structural, chemical and morphological characteristics. The crystalline and structural study was done using X-ray diffraction (Bruker D8 Advance) with Cu-K α radiation ($\lambda = 1.789 \text{ \AA}$) operating at scan rate of $2\theta = 10\text{--}80^\circ$ and a scan speed of $10^\circ/\text{min}$. Fourier Transform Infrared (FTIR) Spectroscopy (Perkin Elmer) was used to identify the molecular structure and the functional groups of the active materials. The morphological observations were studied using field emission scanning electron microscopy (FESEM) coupled with energy dispersive X-ray spectroscopy (EDX), which provided deep insights into chemical composition and surface characteristics of electrospun nanofibers.

Electrochemical measurements, such as cyclic voltammetry (CV) and galvanostatic charging/discharge (GCD) were carried out on CHI660F electrochemical workstation. Electrochemical measurements were carried out by using the three electrodes system with an active material coated nickel foam as a working electrode (WE), Ag/AgCl as a reference electrode (RE), and platinum wire (Pt) serving as a counter electrode (CE) in 3 M KOH electrolyte solution.

3. Results and discussion

3.1. Structural analysis of electrospun PIn nanofibers

3.1.(a) X-ray Diffraction (XRD) Analysis

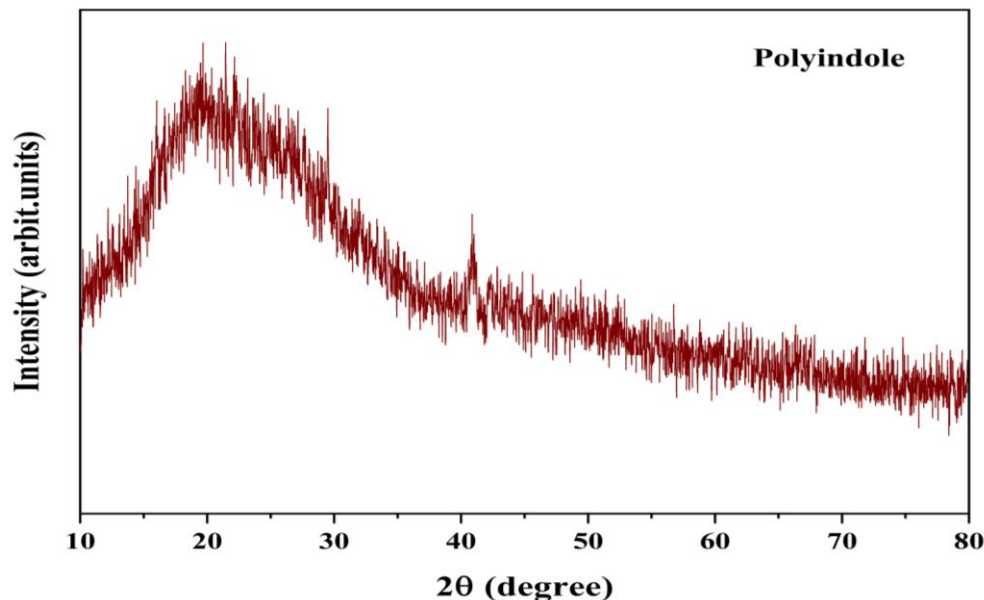


Fig.2 : a) XRD patterns of Pure Polyindole.

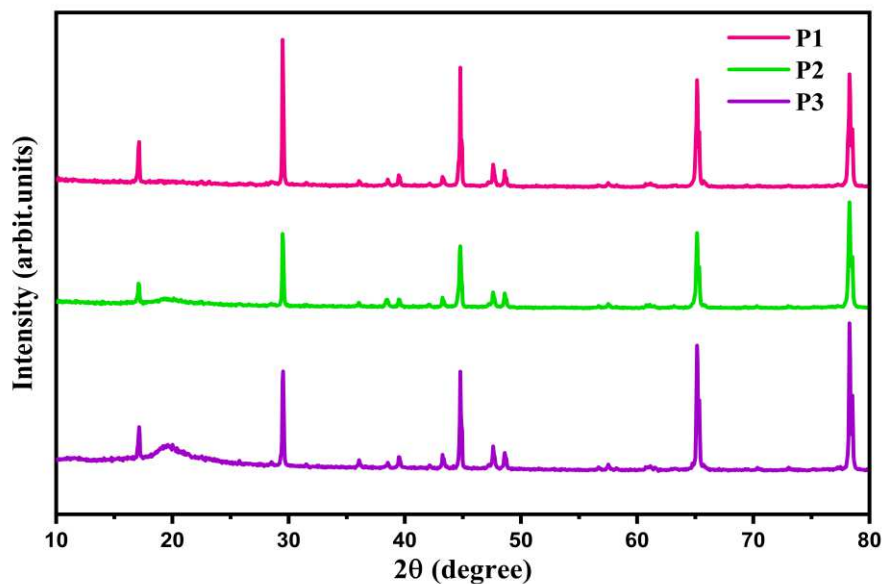


Fig.2 : b) XRD patterns of P1, P2 and P3.

Fig. 2 presents the X-ray diffraction (XRD) patterns of a) pure polyindole (PIn) and the three compositions b) P1, P2 and P3 recorded over a 2θ range of 10° – 80° . Fig. 2 (a) reveals a broad, diffused characteristic peak centered around $2\theta \approx 20^\circ$ – 25° , indicating its predominantly amorphous or semi-crystalline nature, which is typical for conducting polymers with disordered chain structures confirming successful formation of PIn [36]. The absence of multiple sharp diffraction peaks and the appearance of a broad reflection are characteristic

features of polymeric materials, confirming the successful polymerization of the indole monomer [6,37]. The broad hump indicates poor long-range molecular ordering, indicative of an amorphous structure that enhances ionic mobility. Ion transport kinetics are generally faster in amorphous regions, which in addition to limited crystallinity are advantageous for electrochemical applications [38].

Fig. 2 (b) displays the XRD patterns of electrospun nanofiber mats (P1 to P3) deposited on aluminium foil. The diffractograms are dominated by sharp peaks at $2\theta = 44.73^\circ$, 65.13° , and 78.22° , corresponding to the (200), (220), and (311) planes of metallic Al (PCPDFWIN #040787), along with weaker reflections near 31.96° and 39.52° indexed to γ - Al_2O_3 (PCPDFWIN #100425), which can be attributed to a thin, thermally formed alumina layer on the foil or partial surface oxidation during processing [39,40]. A broad amorphous halo appearing in the lower 2θ range becomes more prominent with increasing polyindole content, confirming the amorphous nature of the electrospun nanofibers. The results of elemental composition from EDX also supports this structural observation of XRD. The interchain separation (R) of polyindole was estimated from the broad amorphous hump observed at $2\theta = 21^\circ$ using the following empirical relation by Klug and Alexander [41].

$$R = \frac{5\lambda}{8\sin\theta} \quad \dots \quad (1)$$

where $\lambda = 0.154$ nm is the wavelength of Cu $K\alpha$ radiation and θ is the Bragg's angle.

By using above formula, the value of the interchain separation was estimated to be approximately 0.528 nm, which is found to be consistent with previously reported values for amorphous conducting polymers [26,42]. This moderate interchain spacing reveals the loosely packed polymer chain structure providing sufficient free space to facilitate ion transport, which is one of key factors contributing to enhanced performance for energy storage applications.

3.1.(b) Fourier Transform Infrared (FTIR) Analysis

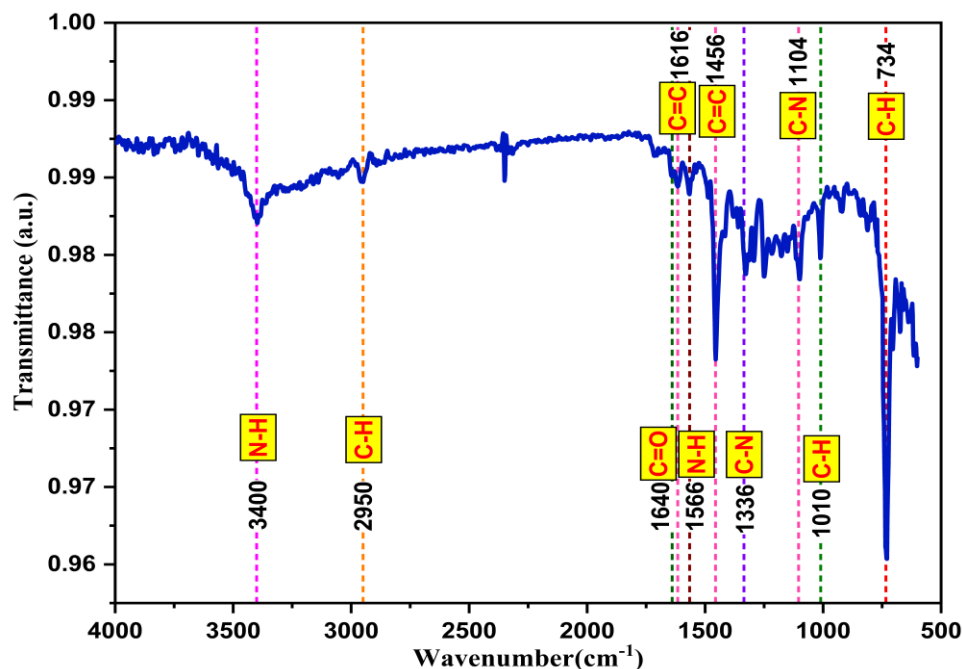


Fig.3 : FTIR spectra of Pure Polyindole.

Fig. 3 depicts the FTIR spectra of pure polyindole. This spectrum exhibits several characteristic bands of pure polyindole that confirms the successful formation of polymer along with retention of its aromatic structure. Two strong peaks appearing at 734 and 1456 cm⁻¹ corresponds to the C-H out-of-plane bending and C=C stretching vibrations of the benzene ring in the indole unit. From this it is observed that the aromatic ring remained intact and is not directly involved in the process of polymerization [43–47]. One distinct absorption band appearing at 1104 cm⁻¹ is assigned to the C-N stretching vibration within the polymer backbone reflecting the presence of aromatic amine linkages thereby confirming the formation of a conjugated polymer network [46]. Another two peaks observed at 1336 and 1010 cm⁻¹ are associated with the stretching of pyrrole ring and C-N bond vibration, which further supports the successful incorporation of indole units into the chain of polymer [47]. The band appearing near 2950 cm⁻¹ can be allocated to the asymmetric stretching vibration of C-H bonds in aromatic ring [48]. Further, the absorption bands at 1566 and 3400 cm⁻¹ are ascribed to deformation vibrations and N-H stretching which indicates that the nitrogen atoms are not the active sites of polymerization and instead the polymerization likely progressed through the indole monomer [34]. Additionally, the C=C and C=O stretching vibrations can be corresponded to the peaks observed at 1616 and 1640 cm⁻¹, indicating the presence of some conjugated structures with minor oxidative contributions [34,43]. Collectively, all these spectral features validate the successful synthesis of polyindole polymer preserving its

aromatic integrity. These all assignments are collectively summarized in table 1 along with respective bonds and vibrational modes.

Table 1 : FTIR spectral assignments of polyindole with corresponding functional groups and literature references.

Wavenumber (cm ⁻¹)	Vibrational Mode / Bond	Reference
734, 1456	C–H (aromatic indole ring), C=C bond in the benzene ring of indole	[43–47]
1104	Stretching of C–N bond	[46]
1616	C=C stretching	[43–45]
1640	C=O stretching	[34,49]
1336, 1010	Pyrrole ring stretching and C–N vibration	[47]
2950	C–H stretching	[48]
1566, 3400	N–H stretching and deformation	[50,51]

3.2. Morphological Analysis

3.2. (a) Field Emission Scanning Electron Microscopy (FESEM)

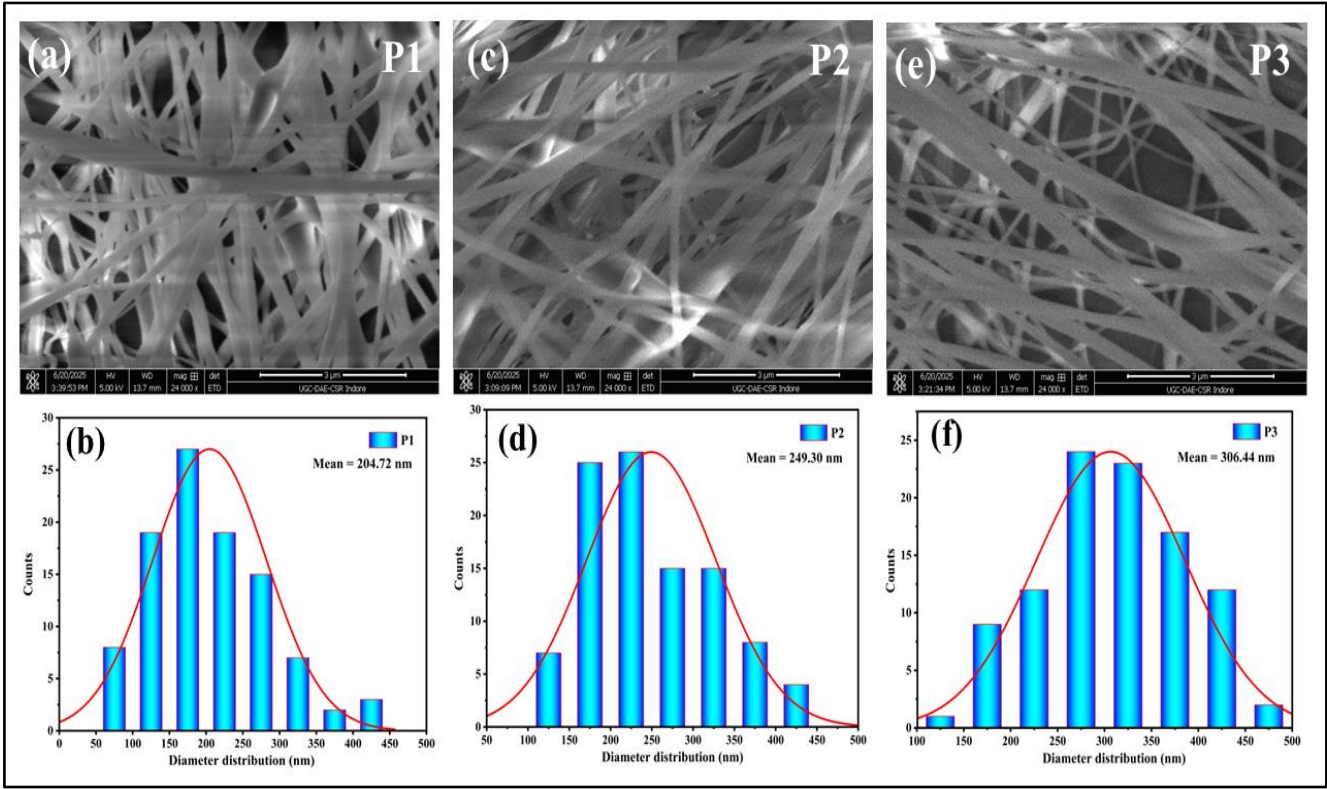


Fig. 4 : FESEM images and average diameter distribution of samples P1 (a) & (b), P2 (c) & (d) and P3 (e) & (f).

The field emission scanning electron microscopy (FESEM) was employed for investigation of the surface morphology of the electrospun PIn nanofibers. The FESEM images revealed a uniform, interconnected

network of nanofibers with a smooth surface and a relatively bead-free morphology, indicating successful electrospinning. The porous network formed by the nanofibers of polyindole is clearly observed, exhibiting smooth surfaces, uniform round morphology, and random orientation throughout the structure [31]. ImageJ software was used to determine the average fiber diameter, which was found to be in the range of ~200 to 300 nm, depending on the PVA:PIIn ratio used. The nanofibers shows non-woven structure providing enlarged surface area which is highly beneficial for supercapacitor applications as it provides a large surface area for electrochemical interactions and facilitates rapid ion diffusion throughout the electrode matrix [52–55]. As the concentration of polyindole in PVA solution increases, greater variation in fiber thickness is observed leading to broader fiber diameter distribution. On the other hand, a decrease in diameter of nanofibers is an increase in specific surface area which provides more electroactive sites for charge storage [56]. At higher polyindole incorporation, a slight increase in fiber diameter is observed with visible surface roughness. This could happen due to increased solution viscosity and more polymer chain entanglement during the electrospinning [11,57]. The 3D interconnected non-woven bead free nanofibrous network obtained is expected to improve both the electrolyte accessibility and charge transport mechanism by producing extra charge storage sites, thereby contributing to enhanced supercapacitor performance.

3.2.(b) Energy Dispersive X-ray (EDX) Analysis

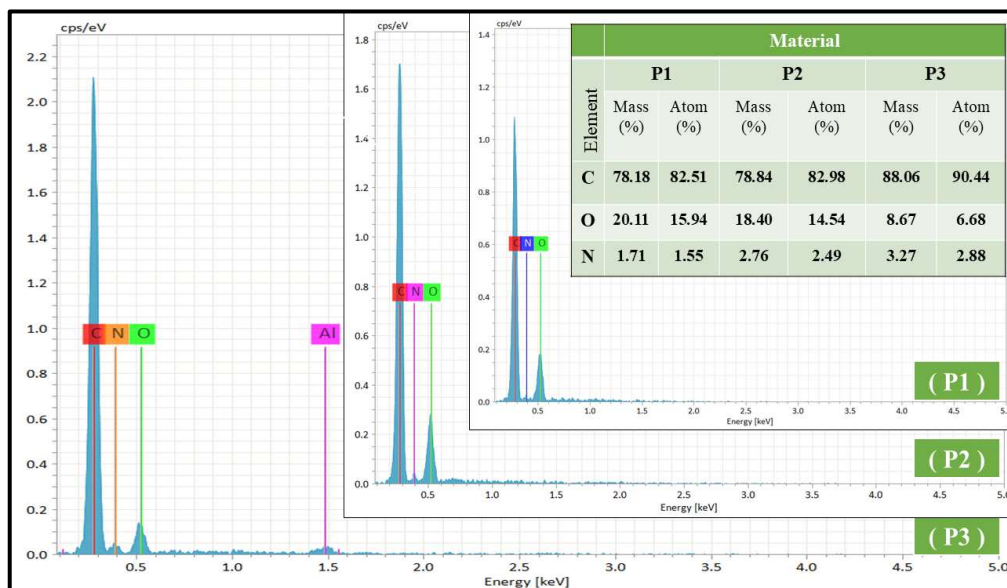


Fig. 5 : Elemental composition of the synthesized P1, P2 & P3 obtained from EDX analysis.

Energy Dispersive X-ray (EDX) analysis was used in addition to FESEM to confirm the elemental composition of the electrospun nanofibers which further explores uniform distribution of elements throughout the material under study. The EDX spectra and its respective elemental composition (Table inset, Fig. 5) shows the presence of desired elements namely carbon (C), nitrogen (N) and oxygen (O) in all three samples since they are part of polyindole itself. It contains a strong carbon peak which is expected from polyindole backbone, while nitrogen is originated from intact indole ring indicating successful polymerization [58,59].

The elemental mapping shows homogeneous distribution of elements, suggesting that the polyindole is uniformly distributed across the nanofiber matrix. No other extra peaks are observed, which validates the purity of the prepared materials. As the polyindole concentration increases in PVA solution, corresponding gradual increase is observed in carbon content from 78.18 wt% for P1 to 88.06 wt% for P3. This result demonstrates the practical incorporation of high amounts of polyindole into nanofibrous structures using electrospinning. The similar trend is exhibited by nitrogen also as it rises from 1.71 wt% to 3.27 wt% with increasing polyindole loading. In converse, the percentage of oxygen content is decreased with increasing amounts of polyindole, which may be attributed to reduced PVA proportion for relative ratios with which the three blends were prepared. These results support the morphological observations and are expected to facilitate consistent electrochemical behavior across entire electrode surface.

3.3 Electrochemical Performance

3.3. (a) Cyclic Voltammetry (CV) Analysis

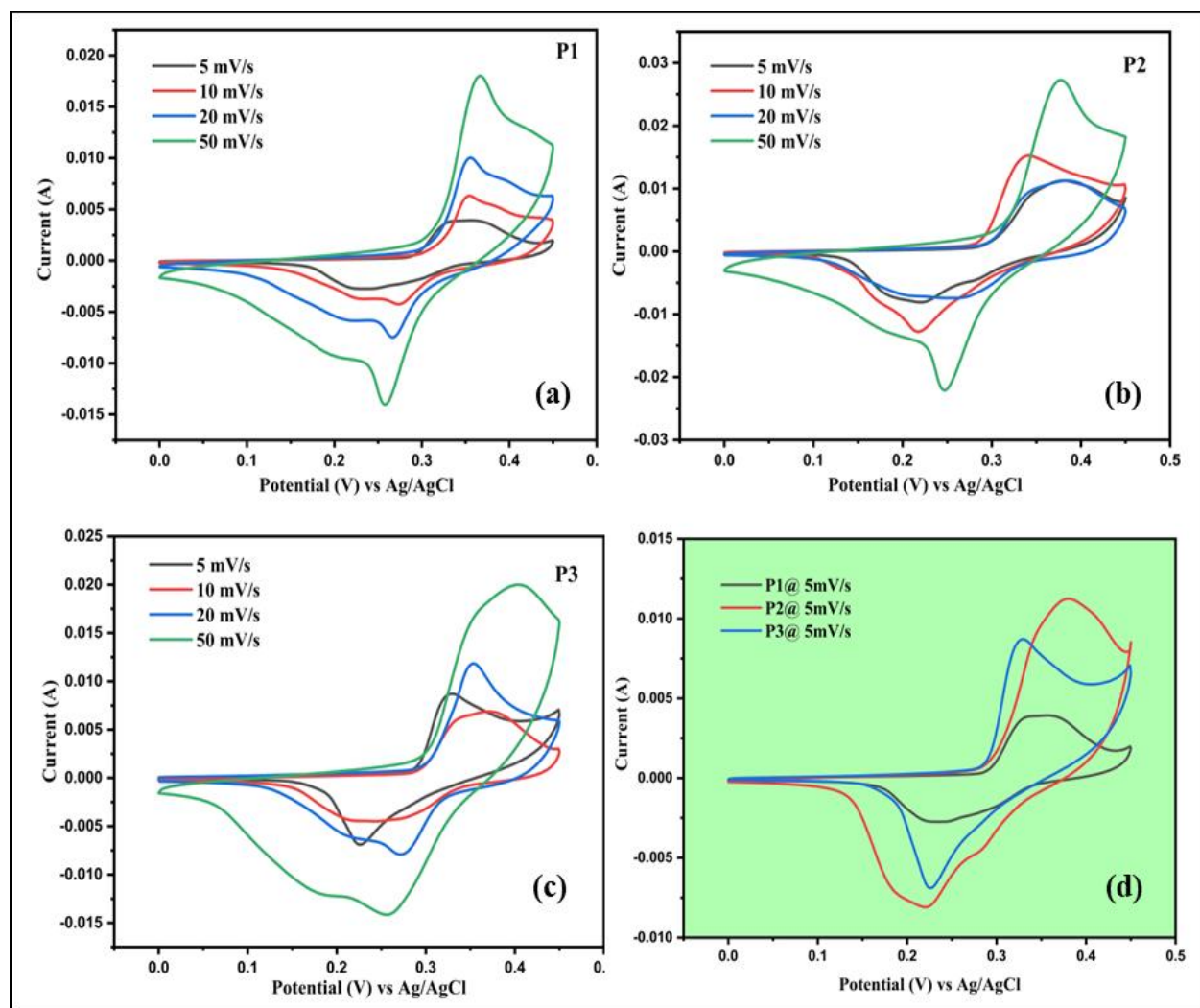


Fig. 6 : Cyclic voltammetry (CV) curves of three electrodes recorded in a three-electrode configuration using 3 M KOH electrolyte: (a) P1, (b) P2, and (c) P3 measured at different scan rates (5–50 mV/s), and (d) comparative CV responses of P1, P2, and P3 at a scan rate of 5 mV/s.

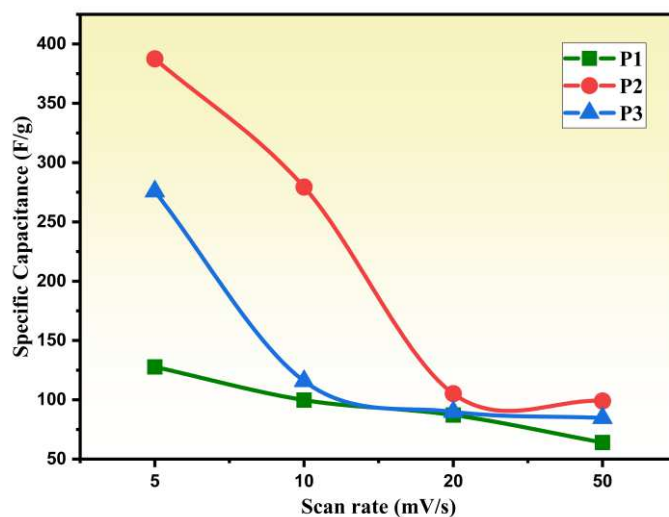


Fig. 7 : Variation of specific capacitance as a function of scan rate for P1, P2, and P3 electrodes.

CV measurements were performed in 0 to 0.45 V potential window from with different scan rates ranging from 5 to 50 mV/s. The specific capacitance (C_s , F/g) was calculated from the enclosed area of CV curves and through the GCD technique based on the following equations [33,60].

$$C_s = \frac{\int_{V_i}^{V_f} I(V) dV}{2ms\Delta V} \quad \dots\dots (2)$$

$$C_s = \frac{I\Delta t}{m\Delta V} \quad \dots\dots (3)$$

where $\int I(V)*dV$ is the area under cyclic voltammogram, m is the mass of electrode material coated on substrate (g), s is the employed scan rate (mV/s), ΔV is the applied potential window (V), I is discharge current whereas Δt is the discharge time.

Similarly, for comparative analysis, the areal specific capacitance ($F\text{ cm}^{-2}$) was calculated by normalizing the capacitance with respect to the geometric area (1 cm^2) of the electrode rather than its composite mass (3 mg) using equations (2) and (3) for CV and GCD measurements, respectively.

As shown in Fig. 6, all three electrodes displayed distinct and nearly symmetric redox peaks arising from characteristic pseudocapacitive behavior associated with the reversible doping-dedoping processes of polyindole backbone. Among the three electrodes, P2 exhibited the highest current response and the largest enclosed CV area demonstrating superior charge storage capability compared to other two electrodes. This performance is associated to the optimized PIN:PVA composition, which improves both electronic conductivity and ion diffusion. As the scan rate increased from 5 to 50 mV/s, the CV profiles retained their shape indicating efficient ion transport and good rate capability of electrodes. Fig. 7 represents the variation of specific capacitance with scan rate, revealing a gradual decrease at higher scan rates due to restricted ion diffusion as fewer ions can reach the active sites [19]. At 5 mV/s, P2 achieves the maximum specific capacitance of 387 F/g followed by P3 (276 F/g) and P1 (128 F/g). Even at high scan rate of 50 mV/s, these three electrodes retained 25.5%, 30.7%, and 50.1% of their initial capacitance, respectively. The superior capacitance with stable retention of P2 is attributed to its better uniform nanofibrous morphology which provides an enlarged surface area and facilitates charge transport [4,31]. Following the same trend, P2 delivered areal capacitance of 1161 mF/cm^{-2} at 5 mV/s while P1 achieved reasonable 384 mF/cm^{-2} and 828

mF/cm⁻² by P3. When a higher scan rate of 50 mV/s was employed P2 exhibited remarkable areal capacitance of 297 mF cm⁻².

There are multiple factors contributing to the high specific capacitance achieved in this study. At first the addition of acetylene black during electrode fabrication works as conductive filler which improved the electron mobility as conductive additives like CNT and carbon black are known to enhance capacitance by assisting in electron-ion transfer [11,14,21,61]. Secondly, the homogeneous nanostructure obtained by electrospinning provides larger surface contributes to the shorter ion diffusion pathways which expedite rapid charge transfer resulting in improved performance [4,11,21,29]. Although P3 contains more Polyindole content, it exhibits lower capacitance than P2 due to increased fiber diameter and reduced surface area, resulting in higher resistance and lower conductivity highlighting the importance of nanostructuring [10]. In contrast, P1, despite its higher PVA content, maintains reasonable capacitance owing to the intrinsic redox activity of polyindole and finer fiber dimensions that favor enhanced electrical conductivity [10,17,21,27].

3.3.(b) Galvanostatic Charge Discharge (GCD)

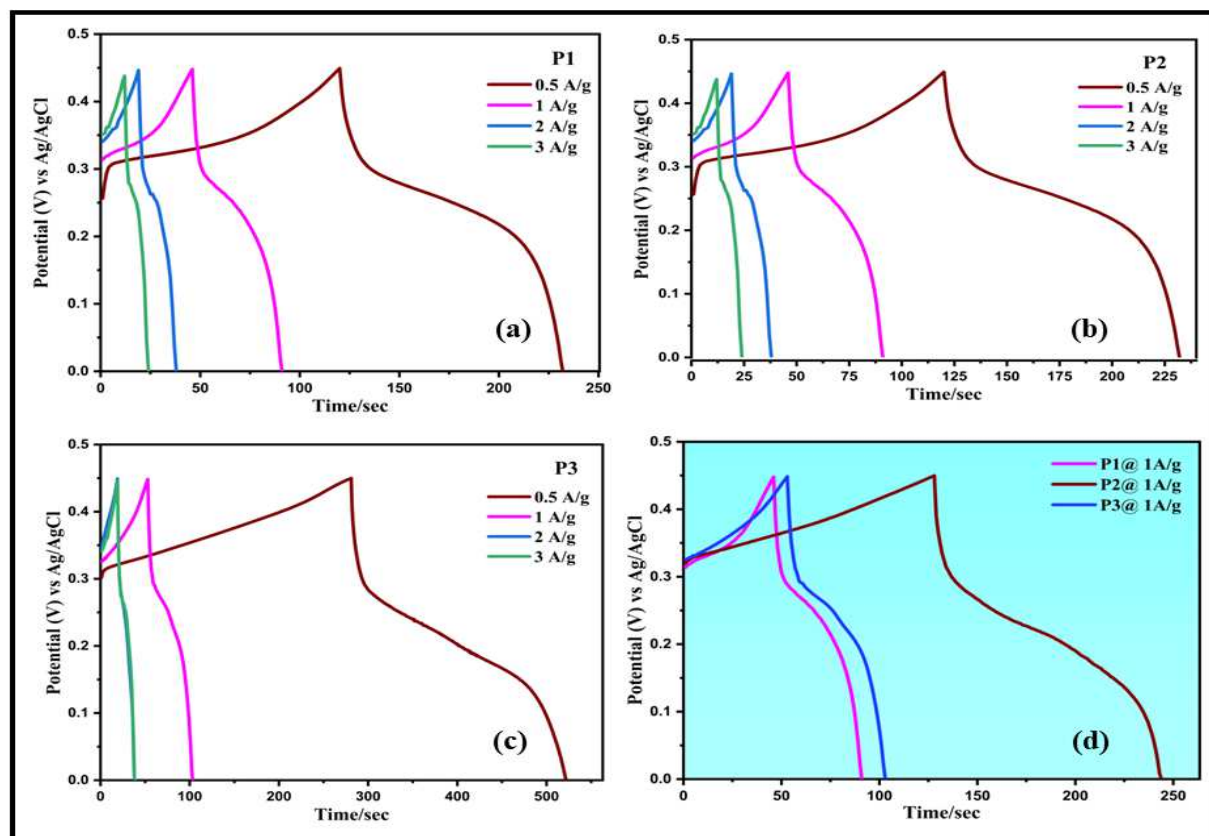


Fig. 8 : Galvanostatic charge–discharge (GCD) profiles of three electrodes: (a) P1, (b) P2, and (c) P3 measured at different current densities, and (d) comparative GCD curves of P1, P2, and P3 at a current density of 1 A/g.

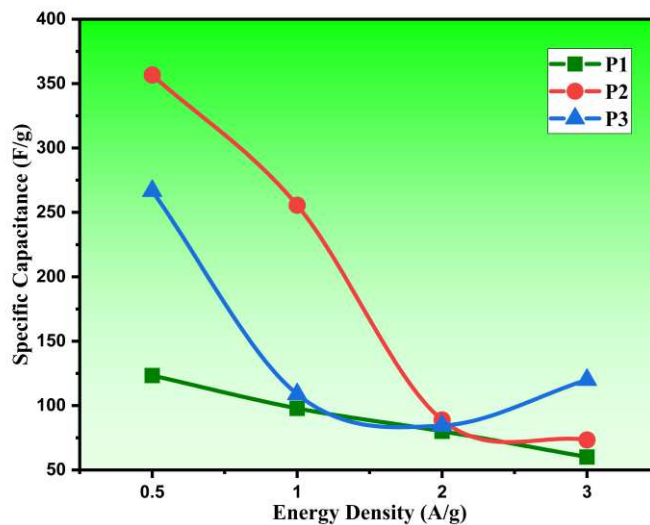


Fig. 9 : Variation of specific capacitance as a function of current density for P1, P2, and P3 electrodes.

For the charge-discharge study, the GCD profiles (Fig. 8) were recorded at different current densities (0.5, 1, 2, and 3 A/g) with same potential range from 0 to 0.45V as the specific capacitance was calculated from both the CV and GCD techniques. The nearly symmetric GCD curves of all three samples are slightly curved having nonlinear slopes which is characteristic of pseudocapacitive behaviour, where the reversible faradaic redox reactions occur within the polyindole matrix. The noticeable small IR drop at the beginning of every discharge cycle is caused by the inherent low electrical conductivity of both polyindole and PVA[4,62]. Electrode P2 delivered the highest specific capacitance of 356.7 F/g at 0.5 A/g current density which is in agreement with its superior current response observed in CV. While P1 and P2 showed notable specific capacitance of 123.3 F/g and 266.7 F/g respectively at 0.5 A/g. Fig. 9 Displays the variation of specific capacitance as a function of current density, where the specific capacitance of 60 F/g at 3 A/g by P1 sample was found to be lowest value in present electrochemical assessment. The trend exhibited by these GCD profiles is consistent with the CV results, which highlights the positive influence of relatively increased polyindole content on overall electrode performance. Overall, the congruence between CV and GCD analysis underlines the need to optimize conducting polymer to carrier ratios in order to achieve high capacitance, fast ion diffusion and robust rate capability, with P2 exhibiting the most balanced performance among the studied electrodes. This performance is compared with earlier reported nanofiber materials with different carrier polymers and is summarized in table 2.

Table 2 : Specific Capacitance of PIn Nanofibers showing influence of carrier polymer.

Nanofiber electrode material	Carrier polymer	Specific Capacitance	Current density/ Scan rate	Reference
PIn	PEO	238 F/g	1 A/g	[11]
PIn/CNT	PEO	521 F/g	5 mV/s	[11]
PIn	PAN	1810 mF/g	5 mV/s	[19]
PIn	PEG	198 F/g	2 mV/s	[21]
PIn/CNT	BC	552.6 F/g	10 mA cm ⁻²	[22]
PIn	PVA	8.79 mF cm ⁻²	50 mV/s	[17]
PIn	PVA	387 F/g	5 mV/s	Present work
		1161 mF/ cm ⁻²	5 mV/s	
		297 mF cm ⁻²	50 mV/s	

3.3.(c) Electrochemical Impedance Spectroscopy (EIS)

Electrochemical Impedance Spectroscopy (EIS) was performed to evaluate the electrical and ion-transport characteristics at the electrode-electrolyte interface in 3 M KOH electrolyte at an OCP of 0.24456 V over the frequency range of 100 kHz to 0.1 Hz. Nyquist plots in Fig.10 depicts the response from electrochemical system by plotting the imaginary component Z'' against the real component Z' . They exhibit a low frequency response which gradually shifts towards a nearly linear trends since, at low frequencies, ions begin to penetrate deeper into the porous nanofiber structure of the electrode. Consequently, the influence ion diffusion increases alongside the capacitive behavior of charge storage, and the Nyquist plot appears almost like a straight line [63].

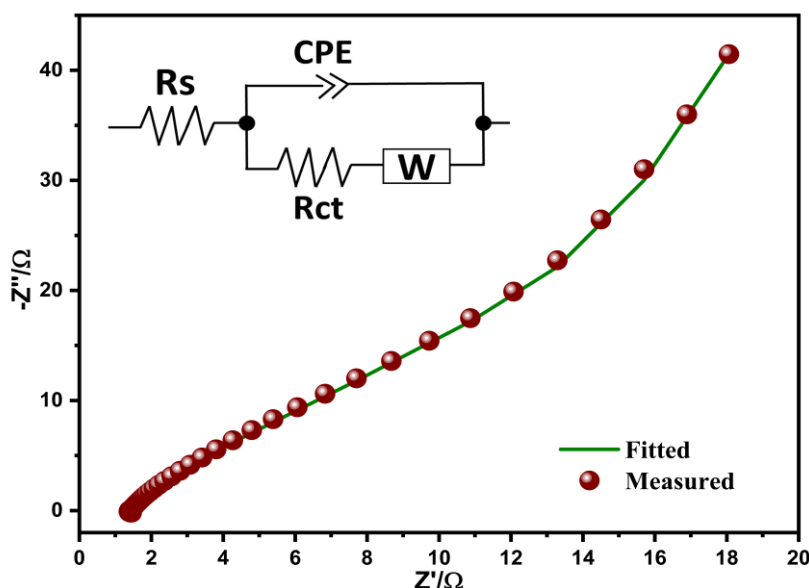


Fig. 10 : Nyquist plots for P2 sample (Insect shows its equivalent circuit)

The equivalent electrical circuit was fitted using ZView 4 software for extraction of different parameters such as R_s and R_{ct} etc. reported onwards. The comparatively low series resistance ($R_s = 1.406 \Omega$) indicates low ohmic resistance and favorable electrical connection within the electrode/electrolyte system whereas the determined R_{ct} value of 15.59Ω represents the resistance encountered during charge transfer processes [64]. The CPE exponent value of 0.863 indicates a predominantly non ideal capacitive behaviour with a slight deviation from ideal one which is reasonable due to porous electrode structure [65,66]. In the EIS model, the finite-length Warburg element represents the impedance arising from ion diffusion for which $W_o-R = 69.15 \Omega$, $W_o-T = 0.047593$, and $W_o-P = 0.9036$ values are obtained which contributes to low frequency response[67]. Overall, the low series resistance, predominantly capacitive behavior, and the observed effect of ion diffusion indicate a favorable interaction between the electrode and electrolyte. Therefore, EIS results demonstrate that these PIn nanofibrous electrodes possesses the electrochemical characteristics required for supercapacitor applications.

Conclusions

In summary, bead free, non-woven electrospun PIn nanofibers were successfully fabricated using electrospinning with optimized PVA:PIn ratios. The characteristic polymeric functional groups were verified by FTIR after XRD confirming the successful formation of PIn. FESEM images displayed uniform nanofibrous network with average fiber diameters ranging between 200 to 300 nm. During this study, P2 electrode showed remarkable performance delivering a specific capacitance of 387 F/g at 5 mV/s from CV measurements. Calculation of specific capacitance from nearly symmetric GCD curves demonstrates its congruence with CV results achieving 356.7 F/g at 0.5 A/g. EIS study further showed low resistance values of R_s and R_{ct} . This improved electrochemical performance is attributed to the uniform nanofiber morphology, optimized polymer compositions, better electrolyte accessibility and the addition of acetylene black which improved electrical conductivity. These findings add up to the fact that enlarging the surface area by nanostructuring the material enhances electrolyte accessibility and with incorporating conductive additives improves ion transport kinetics. Therefore, the as fabricated electrospun PIn nanofibers promises high performance and competitive alternative as flexible electrode material for future energy storage applications.

Declaration of Generative AI and AI-assisted technologies in the writing process

During the preparation of this manuscript, author(s) used ChatGPT in order to correct the complex grammatical errors and improve sentence structure. After usage, the authors reviewed and edited the content as needed and take full responsibility for the content of the published work.

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Author Contributions

A. P. Bangar: Conceptualization, Methodology, Investigation, Formal analysis, Data curation, Validation, Writing & editing. A. P. Pariskar: Data curation, Formal analysis, Resources, Validation. A. S. Daryapurkar: Resources, Methodology, Investigation, Characterization support. Gunjan Verma: Investigation, Characterization support, Writing – review & editing. S. A. Waghuley: Supervision, Conceptualization, Methodology, Validation.

Data Availability

The data presented in this study are available upon request from the corresponding author.

Declarations

Conflict of Interest

The authors declare that there is no known conflict of financial or personal interests that could have influenced the work reported in this study.

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