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Study of the Structural, Morphological, and Electrical Properties of Pyrrole/Activated Carbon Derived from Rice Husk on Cotton Fabric

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ABSTRACT

In this work, activated carbon (AC) and pyrrole were successfully deposited onto cotton fabric using simple dip and dry methods. First, the fabric was bleached. Next, a mixture of AC derived from rice husk, polyvinyl alcohol (PVA) as a binding agent, and sodium dodecylbenzene sulphonate (SDBS) as a surfactant was prepared. This mixture was applied to the fabric using a brush. After that, the AC treated fabric was submerged in a pyrrole solution of the desired molarity, followed by immersion in a ferric chloride ($FeCl_3$) solution for 2 h. Four samples were prepared, and structural, morphological, and electrical characterisation was performed using field emission scanning electron microscopy (FESEM), energy dispersive X ray spectroscopy (EDX), and Fourier transform infrared (FTIR) spectroscopy. Electrical characterisation was conducted using a four point probe method at room temperature for 2 min on 1×1 cm^2 samples. Among all the samples, PPy/AC/cotton fabric (2) exhibited the highest conductivity of 5.5 S/cm. The study also demonstrated that increasing the molarity of pyrrole and $FeCl_3$ initially enhanced the conductivity. However, when the molarities were increased to 0.3 M pyrrole and 0.6 M $FeCl_3$, as well as 0.4 M pyrrole and 0.8 M $FeCl_3$, the conductivity decreased, and the fabric became stiffer and more rigid.

1. Introduction

Many researchers have recently become interested in incorporating conductive polymers into Textile materials have attracted significant attention due to their numerous applications in energy storage, sensors, electrical devices, and medicinal domains [1]. Owing to its ease of polymerisation, biocompatibility, and thermal stability, polypyrrole (PPy) is the most commonly used conductive polymer on cotton fabric. Moreover, PPy treated fabrics exhibit excellent mechanical strength and high conductivity [2]. Various polymerisation methods can be employed to integrate pyrrole into cotton fabrics or fibres, including chemical polymerisation, vapour phase polymerisation, micro emulsion techniques, ultrasonication, and electrochemical polymerisation [3].

Among these methods, chemical polymerisation using ferric chloride ($FeCl_3$) as an oxidising agent offers several

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benefits, including low cost, ease of polymerisation, and minimal contamination risk. However, a drawback of polypyrrole after polymerisation is that it becomes insoluble and infusible, making it difficult to combine with other materials such as carbon, graphene, and other carbon based fillers. To overcome this issue, materials are added during polymerisation [4].

Rice husk (RH) is considered agricultural waste and contains approximately 37 wt% carbon. The main challenge of RH is its low conductivity due to its high silica content; however, after silica removal, it becomes ideal for use in supercapacitors and electrodes [5]. Activated carbon (AC) is insoluble in both organic solvents and water; therefore, various methods, such as electrodeposition, have been used to address this limitation. Historically, charcoal was used to dye fabric black by dipping, which is a primitive form of the screen printing technique [6]. Wang et al. [7] treated cotton fabric with carbon nanotubes (CNTs) and pyrrole via in situ polymerisation to produce wearable sensors with outstanding electrochemical, antibacterial, and electrothermal properties. Conductivity tests indicated a value of 4.5 S/cm when the pyrrole concentration was 0.2 mol/L, and a capacitance of 34.4 mF/cm^2 was recorded when used as a prototype supercapacitor electrode.

Cotton fabric treated with multi walled carbon nanotubes (MWCNTs) by a dip and dry method was subsequently coated with polypyrrole via potentiostatic deposition on the MWCNT/cotton fabric [8], resulting in a low sheet resistance of 6.0 ± 0.4 Ω/sq. In another study, polypyrrole was integrated with CNTs and cotton fibres to develop multi purpose wearable sensors [9]. Testing of supercapacitor electrodes resulted in a capacitance of 30 F/g. However, a significant challenge remains in the integration of AC into cotton fabric via the dip and dry method, followed by the combination of AC/cotton fabric with pyrrole through in situ polymerisation. While numerous studies have explored combining CNTs and other carbon forms with cotton fabric, there is limited research on AC treatment of cotton fabric, especially using AC derived from RH.

The main objective of this work is to combine AC and cotton fabric by the dip and dry method with the aid of polyvinyl alcohol (PVA) and to treat the AC/cotton fabric with pyrrole via in situ chemical polymerisation using $FeCl_3$. This study also aims to determine the optimum pyrrole–$FeCl_3$ molar ratio to achieve high conductivity for various applications in medical devices, smart textiles, and industry.

2. Materials and Methods

2.1. Preparation of AC

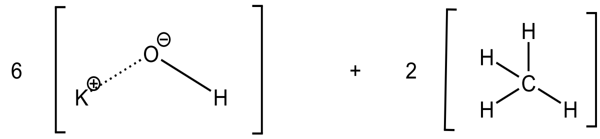
The chemicals used in this method were potassium hydroxide (KOH; molecular weight 56.11 g/mol), sodium hydroxide (NaOH; molecular weight 40.00 g/mol), and nitric acid (HNO_3; molecular weight 63.01 g/mol). First, 50 g of RH was cleaned with water, treated with HNO_3 for 24 h under stirring, rinsed with distilled water, and dried for 24 h at 30 °C. Next, the RH was treated with 500 mL of 1 M NaOH solution for 24 h to remove silica. The reddish brown solids were filtered and dried at 105 °C for 24 h. The RH was then carbonised at 400 °C for 4 h, during which it was converted into black charcoal. It was subsequently treated with NaOH two additional times for 20 min each to remove the remaining silica. All mixing was performed at room temperature. Finally, the rice husk charcoal (RHC) was filtered, washed with distilled water, and dried at 105 °C for 24 h, as shown in Figure 1.

2.2. Preparation of AC from RHC (1-hour activation)

Initially, RHC was treated with KOH in ration1:5 (RHC:KOH) by stirring for 30 min, followed by 15 min of ultrasonication. The sample was subsequently dried at 105 °C for 24 h. It was then activated at 850 °C under a nitrogen gas flow rate of 0.1 L/min for 1 h. Upon completion, the greyish sample was washed with HNO_3 and distilled water to maintain the pH of 6–7, filtered, and dried at 105 °C for 24 h, as shown in Figure 3.

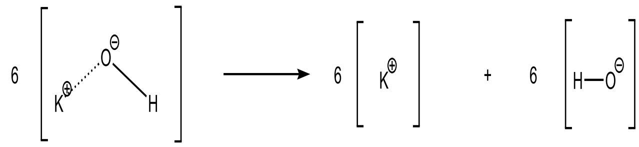
The steps for the formation of porosity in RHC after treatment with KOH are as follows.

1. Combination of KOH and charcoal: During this step, KOH reacts with the carbon in charcoal to produce potassium (K), hydrogen (H_2), and potassium carbonate (K_2CO_3).

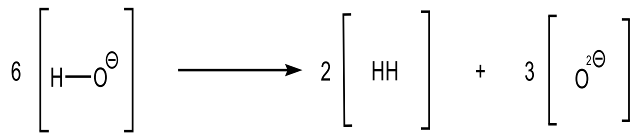


2. Transitional step: This step consists of three stages: dissociation, H_2 formation, and K_2CO_3 formation.

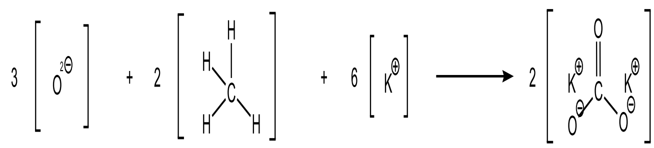
a. Dissociation: In the initial phase of the process, KOH dissociates into potassium ions (K^+) and hydroxide ions (OH^-).



b. Formation of H_2: Hydrogen is produced when the process proceeds, and the dissociated hydroxide ions react with the carbon.



c. Formation of K_2CO_3: Potassium carbonate is formed when KOH reacts continuously with carbon.



The pores are formed as gases such as H_2 and carbon monoxide are released during these reactions, resulting in gaps and holes in the carbon framework. The porous nature of carbon is critical for AC, as it provides a large surface area for adsorption. Furthermore, this process becomes more efficient at high temperatures.

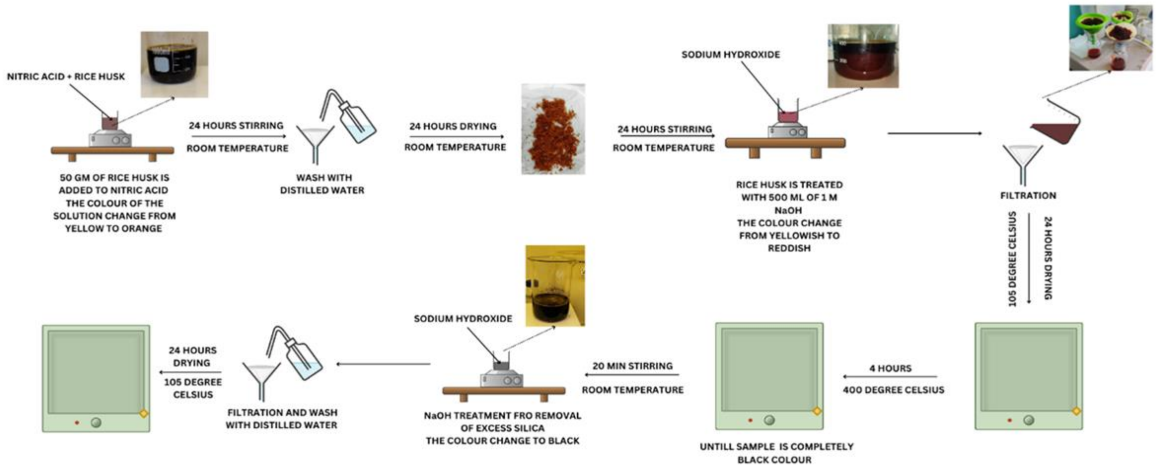


Fig. 1. Preparation of RHC.

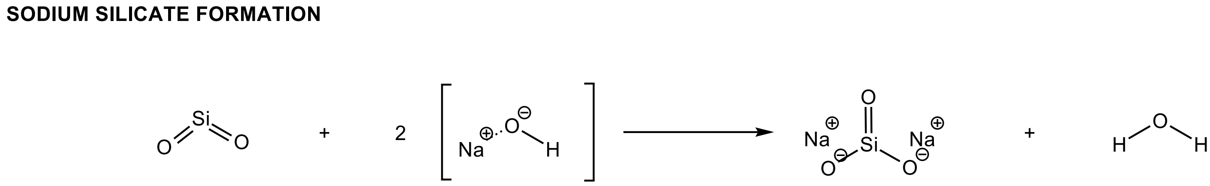


Fig. 2. Formation of sodium silicate.

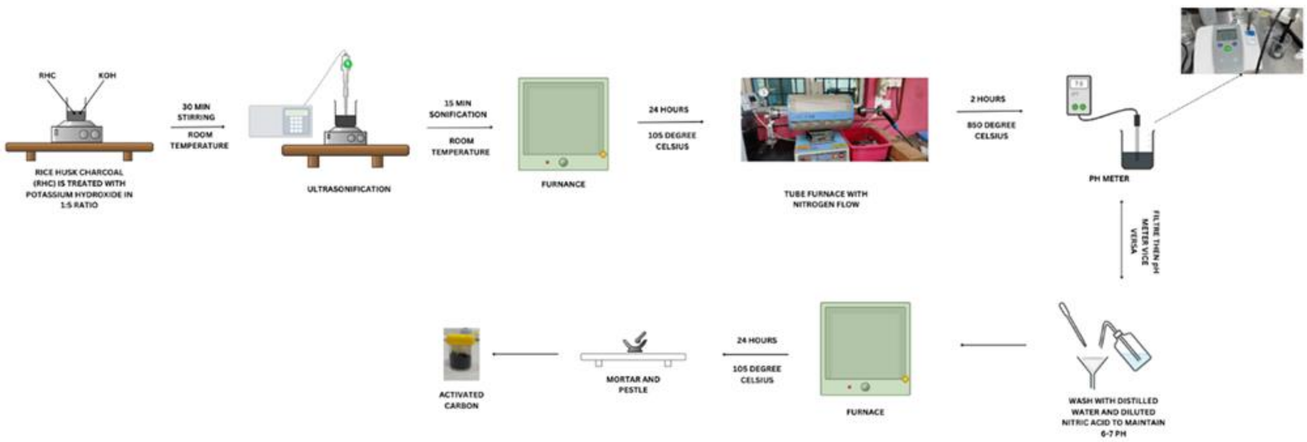


Fig. 3. Preparation of AC from RHC with 1-h activation.

2.3. Preparation of Cotton Fabric

The chemicals used for the preparation of cotton fabric include unbleached cotton fabric (20 × 20 cm^2), Triton X-100 (molecular weight 250.38 g/mol), sodium carbonate (molecular weight 105.9884 g/mol), technical grade calcium hypochlorite (molecular weight 142.9828 g/mol), and artificial vinegar (molecular weight 60.05 g/mol).

• Scouring of cotton fabric

First, 1 mL of Triton X-100 and 5% sodium carbonate (based on the fabric weight of 2.5 g) was added to 25 mL of distilled water. The solution was heated to 60 °C and stirred for 5 min. Subsequently, the fabric was submerged in the solution for 1 h. Afterwards, the fabric was washed thoroughly with distilled water and dried overnight at room temperature, as shown in Figure 4.

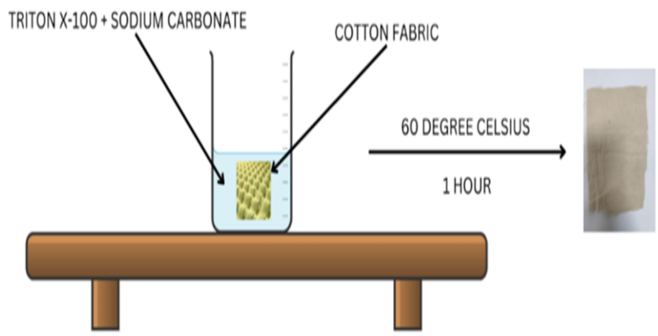


Fig. 4. Scouring of cotton fabric.

• Bleaching of scoured fabric

Initially, 3 wt% calcium hypochlorite was added to 25 mL of distilled water at room temperature and stirred until the calcium hypochlorite particles were completely dissolved. The scoured fabric was immersed in this solution for 20 min. After that, the bleached fabric was submerged in 60 mL of vinegar solution for 20 min, followed by thorough washing with distilled water and drying overnight at room temperature, as presented in Figure 5.

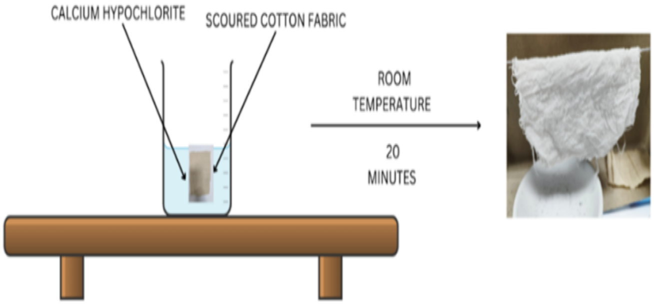


Fig. 5. Bleaching of scoured fabric.

2.4. Preparation of AC/Bleached Cotton Fabric

The chemicals used in this method were sodium dodecylbenzene sulphonate (SDBS; molecular weight 348.48 g/mol), polyvinyl alcohol (PVA; average molecular weight not specified), and AC from RH. First, 1 g of PVA was added to 15 mL of distilled water and stirred for 2 h at 90 °C. Next, 0.5 g of SDBS was added to the solution at room temperature and stirred until completely dissolved. Finally, 3 g of AC powder was added to the solution and stirred for 24 h at room temperature until no bubbles remained on the surface of the solution. The solution was applied evenly to the 20 × 20 cm^2 cotton fabric using a paintbrush. Upon completion, the fabric was dried overnight at room temperature. Subsequently, the fabric was cured using a hot iron press and stored in a sealed container for further processing (Fig. 6).

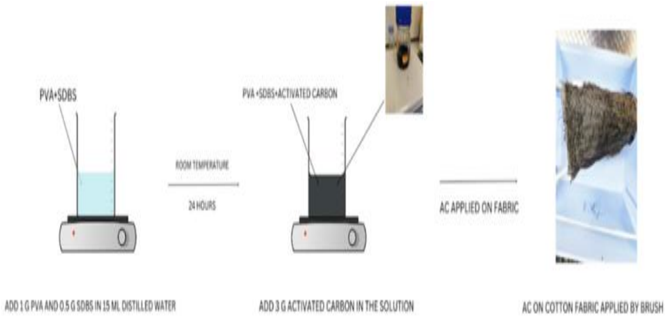


Fig. 6. Preparation of AC solution.

2.5. *Preparation of AC/Cotton Fabric/Pyrrole*

The chemicals used in this method are pyrrole (molecular weight 67.09 g/mol), $FeCl_3$ (molecular weight 162.21 g/mol) and AC/cotton fabric. In this research, four samples were prepared with different molarities of pyrrole and $FeCl_3$, as tabulated in Table 1.

Table 1. Sample preparation with different molarities.

SL No.	Sample	Pyrrole (M)	$FeCl_3$ (M)	Time (h)	Temp. (°C)
1	PPy/AC/Cotton (1)	0.1	0.2	2	25
2	PPy/AC/Cotton (2)	0.2	0.4	2	25
3	PPy/AC/Cotton (3)	0.3	0.6	2	25
4	PPy/AC/Cotton (4)	0.4	0.8	2	25

A 20 × 20 cm^2 of AC-treated fabric was cut into four samples, each measuring 4 × 4 cm^2. These samples were then treated with different molarities of pyrrole and $FeCl_3$, maintaining a pyrrole–$FeCl_3$ ratio of 1:2. The molarities used were 1:2, 2:4, 3:6, and 4:8. First, 0.2 M $FeCl_3$ was dissolved in 25 mL of distilled water at room temperature with stirring. Next, 0.1 M pyrrole was applied evenly to the AC/cotton fabric using a dropper and left to dry for 5 min. The pyrrole/AC/cotton fabric was then immersed in the $FeCl_3$ solution for 2 h for polymerisation at room temperature. After polymerisation, the fabric was thoroughly washed with distilled water to remove any unreacted pyrrole and $FeCl_3$ from the surface of the fabric, and then dried overnight at room temperature, as shown in Figure 7. This process was repeated to produce another three samples with different molarities, as detailed in Table 1.

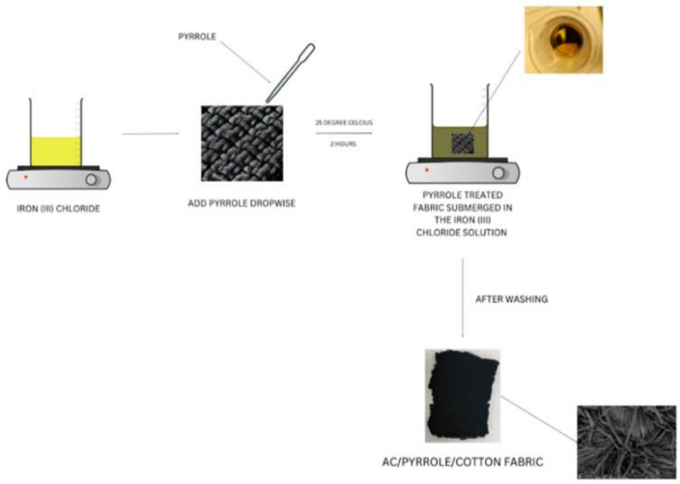


Fig. 7. Preparation of AC/pyrrole/cotton fabric.

3. Results and Discussion

Figs. 8 (a-d) demonstrate the FESEM images of all the specimens. Figure 8(a) shows bleached cotton fabric with a smooth surface and a 1/1 plain weave, whereas Figure 8(b) presents cotton fabric treated with AC. The latter shows the development of a more porous surface structure, indicating the successful deposition of AC.

Additionally, the samples in Figure 8 (c) (PPy/AC/cotton fabric (1)), Figure 8(d) (PPy/AC/cotton fabric (2)), Figure 8(e) (PPy/AC/cotton fabric (3)), and Figure 8(f) (PPy/AC/cotton fabric (4)) exhibit a rougher surface morphology [10], indicating the presence of polypyrrole when compared to Figure 8(a), which shows bleached cotton fabric.

Furthermore, upon comparing Figures 8(c–f), it can be observed that increasing the molarity of pyrrole and $FeCl_3$ results in a gradual thickening of the surface layer. To further support this observation, an energy dispersive X ray spectroscopy (EDX) analysis was conducted to determine the material composition, which mainly consisted of carbon, oxygen, and nitrogen, as presented in Figure 9. In Figure 9(a), bleached cotton fabric exhibits weight percentages of 72.7% carbon and 27.3% oxygen. Figure 9(b) indicates weight percentages of 66.5% oxygen, 25.0% carbon, 2.8% silicon, and 0.4% potassium for the AC treated cotton fabric. In Figure 9©, the weight percentages for PPy/AC/cotton fabric (1) are 41.4% oxygen, 25.2% nitrogen, 16.5% carbon, 0.1% silicon, and 0.2% iron. Figure 9(d), representing PPy/AC/cotton fabric (2), indicates weight percentages of 33.9% oxygen, 28.9% nitrogen, 14.4% carbon, 0.6% silicon, and 1.9% iron. The weight percentages for PPy/AC/cotton fabric (3) are 27.7% oxygen, 26.6% nitrogen, 12.5% carbon, 0.1% silicon, and 4.8% iron, as shown in Figure 9(e). Finally, Figure 9(f) for PPy/AC/cotton fabric (4) shows weight percentages of 32.0% oxygen, 29.5% nitrogen, 13.8% carbon, 1.9% silicon, and 2.0% iron.

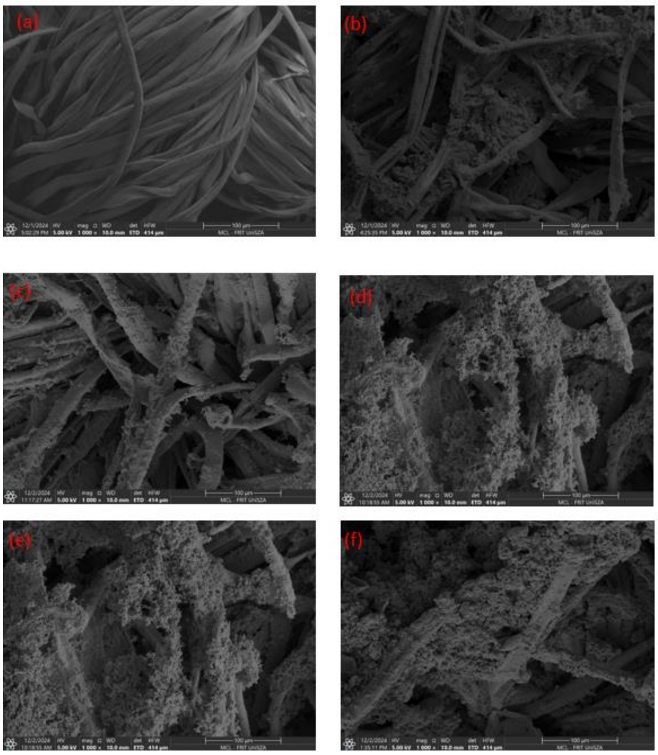


Fig. 8. Field emission scanning electron microscopy (FESEM) images at 1,000× magnification for (a) bleached cotton fabric, (b) AC-treated cotton fabric, (c) PPy/AC/cotton fabric (1), (d) PPy/AC/cotton fabric (2), (e) PPy/AC/cotton fabric (3), and (f) PPy/AC/cotton fabric (4).

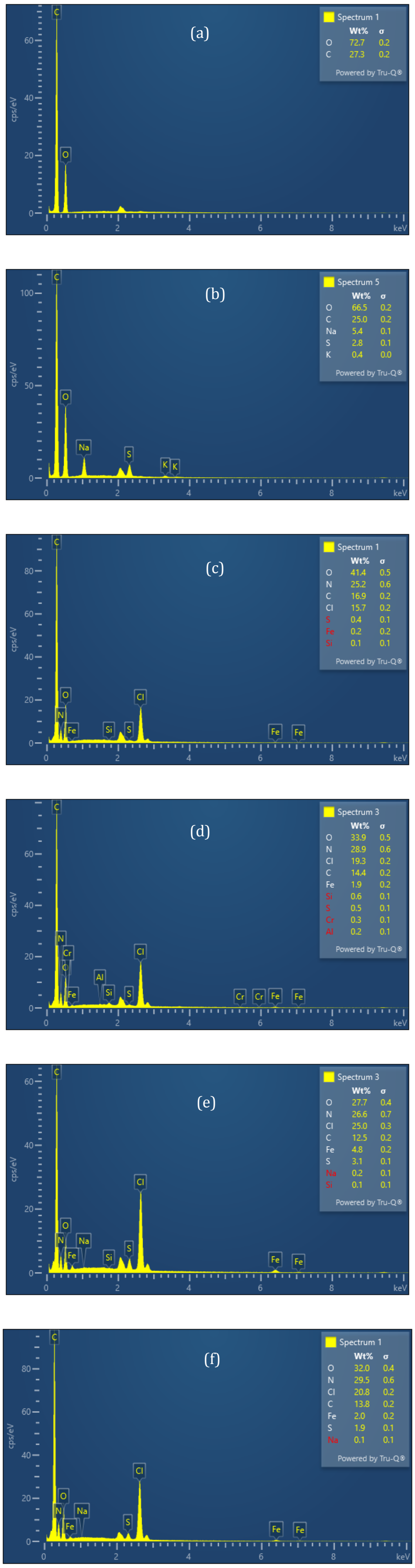


Fig. 9. EDX analysis of (a) bleached cotton fabric, (b) AC-treated cotton fabric, (c) PPy/AC/cotton fabric (1), (d) PPy/AC/cotton fabric (2), (e) PPy/AC/cotton fabric (3), and (f) PPy/AC/cotton fabric (4).

The elevated oxygen content is attributed to the inherent cellulose composition of the material, the oxygen containing functional groups present in the AC, and possible contributions from moisture or binder components. Cotton fibre consists primarily of cellulose, whose molecular structure includes hydroxyl (–OH) functional groups. These groups can chemically react to form other functional groups, such as carboxyl (–COOH) and carbonyl (–C=O) groups [11]. Furthermore, the EDX technique is semi quantitative and tends to underestimate lighter elements such as carbon, which may explain the relatively low measured carbon content. Minor discrepancies in the total elemental composition can be attributed to instrument limitations and data normalisation effects.

Figs. 10(a–f) illustrate the FTIR spectra of bleached cotton fabric, AC treated cotton fabric, PPy/AC/cotton fabric (1), PPy/AC/cotton fabric (2), PPy/AC/cotton fabric (3), and PPy/AC/cotton fabric (4), respectively. The broad peaks at 3342 cm^{-1} and 2909 cm^{-1} in Figure 10(a) indicate O–H stretching vibrations associated with hydrogen bonding and C–H stretching vibrations, respectively. The peak at 1030 cm^{-1} corresponds to overlapping bands attributed to cellulose functional groups, namely C–C, C–O, and C–O–C stretching vibrations [12]. In the spectrum of the AC treated cotton fabric (Figure 10(b)), a similar peak appears at 3331 cm^{-1}, while the peak at 2920 cm^{-1} represents the stretching vibration of the C–H bond.

In the spectra of the PPy/AC treated fabrics shown in Figures 10(c–f), broad tailing bands were observed between 4000 and 2000 cm^{-1}, which can be attributed to electronic transitions and overlapping vibrational modes. The peak at ~1500 cm^{-1} corresponds to the C=C stretching vibration, indicating π conjugated bonds responsible for conductivity through delocalised π electron movement, and also reflecting the aromatic structure of AC [13]. Moreover, the band between 1200 and 1100 cm^{-1} indicates the presence of C–N stretching vibrations, associated with oxidised polypyrrole structures. Furthermore, the band at 1030 cm^{-1} reflects the cellulose functional groups of cotton. Peaks between 700 and 600 cm^{-1} are attributed to C–H out of plane deformation vibrations [14–16].

The characteristic bands observed in the ranges of 1530–1550 cm^{-1} (C=C stretching vibration of quinoid rings) and 1450–1500 cm^{-1} (C–N/C=N stretching vibration of benzenoid rings) further confirm the formation of the polypyrrole (PPy) phase.

Table 2 presents the electrical conductivity of all samples in this study, as measured using the four-point probe test. Additionally, this method was employed for characterisation, with calculations performed using Equations (1), (2), and (3).

The resistivity (ρ, Ω·cm) was calculated as described in [17].

$$\rho = \frac{V}{I} \times \frac{\pi}{ln(2)} \times t \quad (1)$$

Where ρ is the resistivity, V is the voltage, I is the current, t is the thickness of the sample, π is a constant (3.14), and ln(2) is the natural logarithm of 2 (≈ 0.632).

Conductivity (σ, s/cm) was obtained using Eq. 2:

$$\sigma = \frac{1}{\rho} \quad (2)$$

Where σ is the conductivity and ρ is the resistivity.

Resistance (Ω) was determined using Eq. 3:

$$Resistance = \frac{V}{I} \quad (3)$$

Where V is the voltage and I is the current in the equation.

Cotton is a widely used textile material; however, it is inherently insulating. To impart electrical conductivity, pyrrole (PPy) and activated carbon (AC) derived from rice husk (RH) were incorporated into the fabric in varying ratios. The negligible conductivity measured for bleached cotton is attributed to instrumental limitations when attempting to record electrical responses from highly insulating materials. In such cases, the measured signal is dominated by instrumental noise, resulting in a small negative value.

After calculating conductivity using Eq. (2), PPy/AC/cotton fabric (2) exhibits the highest conductivity of 5.5 S/cm. The second-highest conductivity is 1.5 S/cm for PPy/AC/cotton fabric (3), followed by 1.5 S/cm for PPy/AC/cotton fabric (1). The next value is 1.4 S/cm for PPy/AC/cotton fabric (4). The lowest value, –0.3 S/cm, corresponds to bleached cotton fabric, confirming its insulating nature. Overall, the results indicate that using 0.2 M pyrrole and 0.4 M $FeCl_3$ produces the most favourable conductivity, signifying an optimised PPy deposition. As the molarity increases beyond this point, the conductivity decreases, which can be attributed to the formation of a thicker, less uniform PPy/AC layer that increases interfacial resistance and limits charge transport.

The spacing between the probes was maintained at the standard fixed spacing of the four point probe apparatus (approximately 1.0 mm). Since no repeated measurements were conducted, the experimental uncertainty could not be directly estimated. Nevertheless, the measurement uncertainty can be inferred from the instrument specified uncertainties, including errors in voltage and current readings, probe positioning, and sample geometry. Incorporating repeated measurements in future work would enable a more robust statistical assessment of measurement errors.

Future studies will involve the fabrication of a graphene based AC composite extracted from RH, which shows significant potential for supercapacitor applications. Graphene based supercapacitors are considered among the most reliable energy storage materials due to their exceptional thermal stability, rapid charge–discharge characteristics, and long cycle life [18].

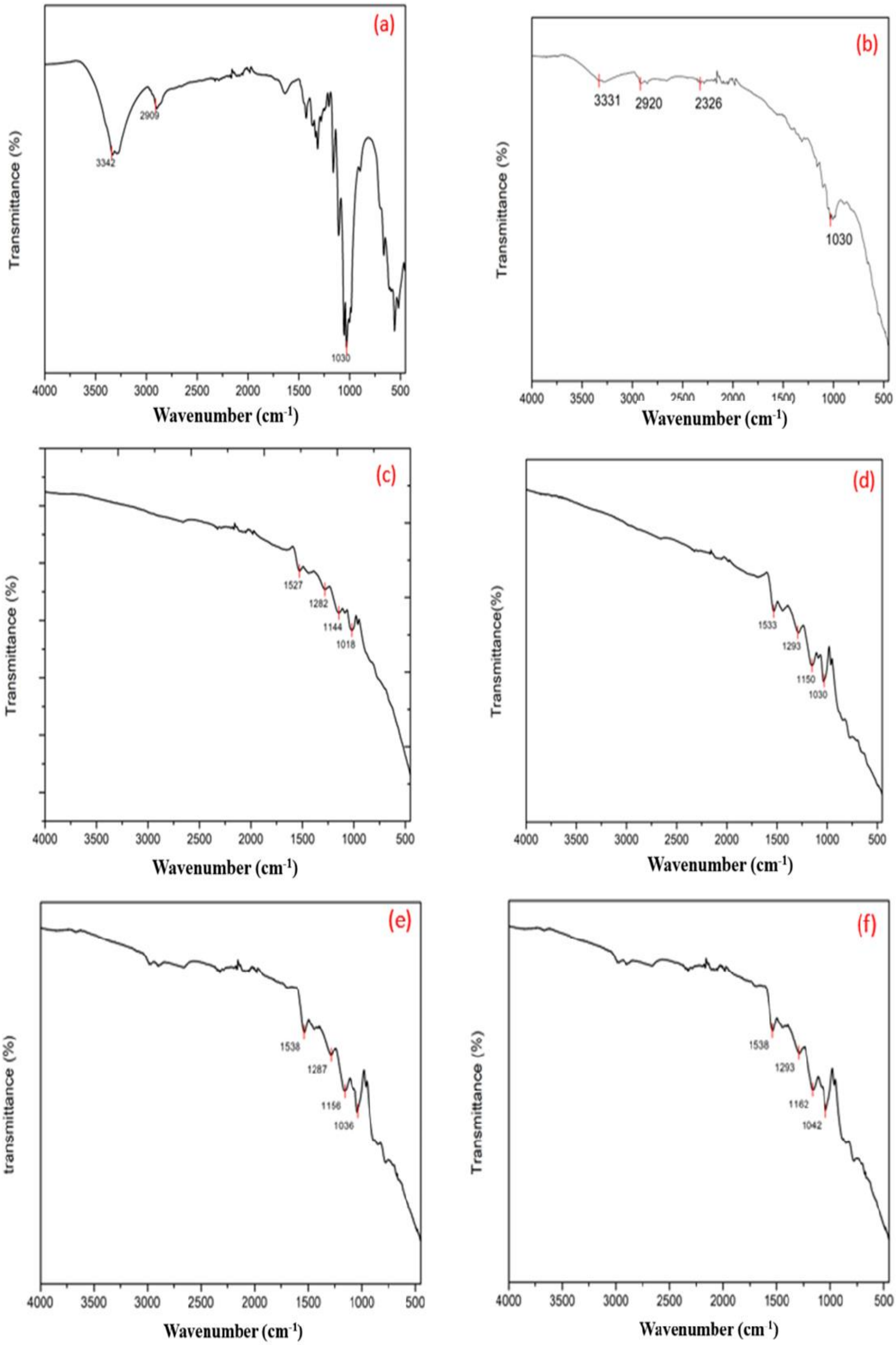


Fig. 10. FTIR spectra of (a) bleached cotton fabric, (b) AC-treated cotton fabric, (c) PPy/AC/cotton fabric (1), (d) PPy/AC/cotton fabric (2), (e) PPy/AC/cotton fabric (3), and (f) PPy/AC/cotton fabric (4).

Table 2. Results of conductivity evaluation using the four-point probe test.

Sample	Sample Size (cm^2)	Thickness (mm)	Resistance (Ω)	Resistivity (Ω·cm)	Conductivity (S/cm)
Bleached cotton fabric	1 × 1	0.2	29.43	2.667	Negligible
AC-treated cotton fabric	1 × 1	0.5	54.73	12.403	0.081
PPy/AC/cotton fabric (1)	1 × 1	0.4	3.69	0.669	1.495
PPy/AC/cotton fabric (2)	1 × 1	0.5	0.80	0.181	5.516
PPy/AC/cotton fabric (3)	1 × 1	0.6	2.38	0.647	1.545
PPy/AC/cotton fabric (4)	1 × 1	0.7	2.21	0.701	1.426

3. Conclusions

The objective of this research was to fabricate cotton fabric incorporating activated carbon (AC) derived from rice husk (RH) and polypyrrole (PPy) for various advanced textile applications. However, the available literature on integrating AC with cotton fabric followed by post treatment with pyrrole to enhance electrical conductivity is highly limited. In this study, it was observed that the use of polyvinyl alcohol (PVA) and sodium dodecylbenzene sulphonate (SDBS) enabled effective attachment of AC particles to the cotton fabric through a dip and dry method. Subsequent analyses confirmed the successful incorporation of both AC and PPy.

Surface characterisation was performed using FESEM (1,000×) to assess morphological changes, EDX to determine elemental composition, and FTIR to identify the functional groups present in the treated fabrics. The most critical assessment was the electrical conductivity measurement, conducted using a four point probe system, in which PPy/AC/cotton fabric (2) exhibited the highest conductivity among all samples.

Based on the results, increasing the molarity of pyrrole and $FeCl_3$ beyond 0.2 M and 0.4 M, respectively, led to a gradual decrease in conductivity. Visual inspection further revealed that higher molarities produced a thicker, stiffer, and more brittle surface layer, which hindered charge transport. These observations indicate that the use of PVA and SDBS is effective for facilitating AC attachment to cotton fabric, and that 0.2 M pyrrole and 0.4 M $FeCl_3$ represent the optimal concentrations for achieving high conductivity while maintaining acceptable fabric flexibility.

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Conflicts of Interest

The authors declare no conflicts of interest.

Authors Contribution Statement

Conceptualisation and study design: Manash Jyoti Das, Mohd Zaki Mohd Yusoff, Suraya Ahmad Kamil, Ali H. Jawad
Data collection and experimentation: Manash Jyoti Das, Mohd Zaki Mohd Yusoff, Suraya Ahmad Kamil, Ali H. Jawad,
Data analysis and interpretation: Manash Jyoti Das, Mohd Zaki Mohd Yusoff, Muhammad Syarifuddin Yahya, Syahril Amin Hashim
Manuscript writing and editing: Manash Jyoti Das, Mohd Zaki Mohd Yusoff
Supervision and project administration: Mohd Zaki Mohd Yusoff, Suraya Ahmad Kamil, Ali H. Jawad

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