


Research Article

Polyaniline Deposition on the Surface of Cotton Fibers: Structural Studies, Swelling Behavior, and Water Absorption Properties

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Polyaniline-modified natural fibers have been recognized as promising candidates for conductive clothes, UV protection, and electromagnetic interference shielding. Hence, the purpose of this study was to investigate the effect of surface deposition of cotton fibers using polyaniline via *in situ* polymerization, and preceding structural changes were further screened by FT-IR, UV-Vis, TGA, SEM/EDX, and conductivity in comparison with bare cotton fibers used as the control sample. Polyaniline was introduced on the surface of cotton fibers as a conductive form, which was confirmed by electrical conductivity ($1.54 \times 10^{-4} \text{ Scm}^{-1}$) equivalent to semiconductor materials. Detection of particular peaks at $\text{NK}\alpha$ 0.379 keV and $\text{ClK}\alpha$ 2.621 keV from EDX analysis revealed the introduction of nitrogen and chlorine, respectively. Polyaniline deposition on the cotton surface was successful to introduce hydrophobic environment to the system to enhance resistance to water absorption meaningfully.

1. Introduction

The inherent features of conducting polymers have attracted considerable attention of scientists and researchers. Polyaniline (PAni) is one among such polymers, which uses the least expensive monomer with the advantage of the ease of polymerization [1, 2]. Electronic structure of polyaniline can be altered by varying the ratio of oxidation-reduction states of amino functionalities, which includes fully reduced leucoemeraldine, completely oxidized pernigraniline, and partially oxidized emeraldine forms (Figure 1) [3]. This can be achieved by adding oxidizing or reducing agents under varied reaction media.

Polyaniline backbone is wealthy in amine and imine multifunctionalities comprised of benzoid ($-\text{B}-\text{NH}-\text{B}-\text{NH}-$)_n and quinoid ($-\text{B}-\text{N}=\text{Q}-\text{N}=\text{B}-$)_n repeating structures [4], which are responsible for the fascinating features of surface

chemistry. Sole polyaniline exists in the form of aggregated [5] powder which has limited surface area [6], poor processability, and weak mechanical strength [7]. These drawbacks hindered the scope, versatility, and multidisciplinary applications of polyaniline. Hence, different substrates and modifiers are required to obtain functionally active materials that display enhanced properties and performances. Natural fibers such as kenaf, cotton, jute, kapok, and banana [8–11] are suitable candidates used as a substrate phase to carry polyaniline.

Polyaniline salt has played a key role as a conductive filler in the production of electrically conductive fabrics such as cotton, coconut, nylon, and polyester [12, 13], which are used as antistatic coatings, pressure-sensing, electromagnetic interference shielding, and UV protection [14]. Electroconductive fabrics, particularly polyaniline-based cotton textiles, show considerable applications in wearable electronics,

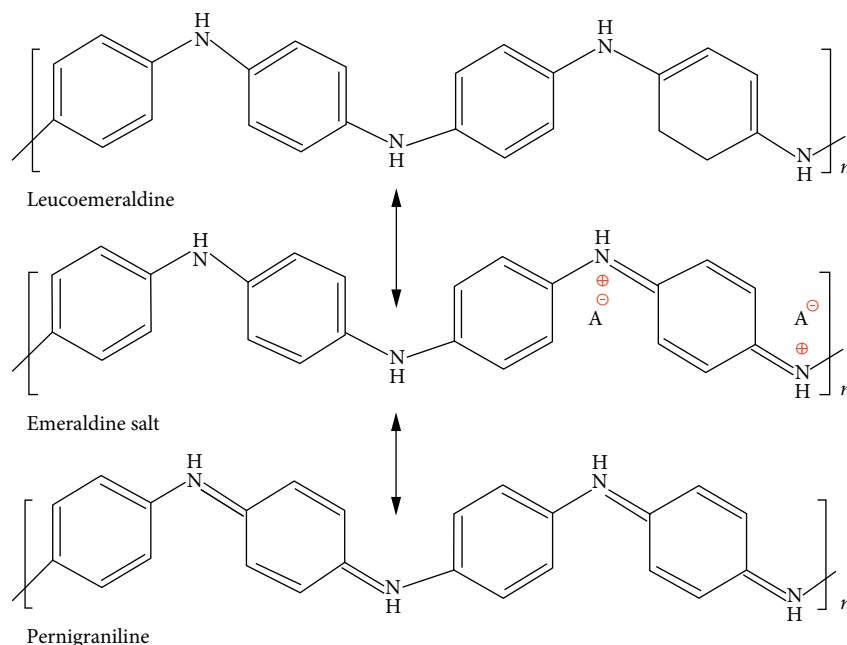


FIGURE 1: Molecular design of oxidation states of polyaniline, where A^- denotes anionic species.

sensory devices, heating utensils, recovery of precious metals, and environmental remediations [7, 15]. Among many substrates, cotton (*Gossypium hirsutum*) fiber belongs to the *Gossypieae* tribe, which grows comfortably in the USA, South America, India, Egypt, Sudan, South Africa, and Ethiopia [16]. It has a soft and crystalline fibrous structure, which possesses a fairly strong mechanical property and low density [17].

Polyaniline deposition can be done by *in situ* oxidative polymerization [18–20]. It is termed as nondestructive technique which does not require removal of template materials. Moreover, *in situ* oxidative polymerization is a proven method to avoid aggregate formation in order to maximize the surface area, which is known to be a suitable technique for mass production at reasonable costs [21]. However, application of natural fibers as a substrate phase has certain shortcomings such as high moisture regain when exposed to water results swelling [22]. Consequently, *in situ* deposition of polyaniline on the surface of natural fibers is supposed to introduce hydrophobic polymer matrix by reducing water absorption features of the fibers [23].

Therefore, the present study is primarily targeted to investigate the influence of polyaniline deposition on the surface of cotton fibers by *in situ* oxidative polymerization. Besides, the effect of surface modification using polyaniline on structural features of cotton fibers was investigated by Fourier transform-infrared (FT-IR) spectroscopy, UV-Visible (UV-Vis) spectroscopy, thermogravimetric analysis (TGA), scanning electron microscope (SEM) coupled with energy dispersive X-ray (EDX) spectroscopy, and electrical conductivity.

2. Materials and Methods

2.1. Chemicals and Materials. The following reagents were of analytical grade and used without further purification: aniline ($\text{C}_6\text{H}_7\text{N}$), ferric chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$),

hydrochloric acid (HCl), chloroform (CHCl_3), acetone ($\text{C}_3\text{H}_6\text{O}$), and ethanol ($\text{C}_2\text{H}_5\text{OH}$) purchased from Merck Chemical Co. (Germany), through a local agency. Cotton (*Gossypium hirsutum*) fiber was obtained from the local market of Mekelle, Northern Ethiopia. It was washed with double distilled water, followed by acetone and ethanol, and finally dried in air at room temperature.

2.2. Chemical Deposition of Polyaniline on the Surface of Cotton Fibers. Chemical deposition of polyaniline on the surface of cotton fibers was carried out using the following procedure. 10 gm of cotton fiber was soaked in 0.2 M HCl solution on stirring. 150 mL of 0.5 M aniline was added into the flask containing acidified fiber on subsequent stirring. Equimolar of ferric chloride hexahydrate solution was added into the mixture dropwise while stirring [8, 24], and the polymerization process was allowed for 12 hours at room temperature. Polyaniline-deposited cotton fiber was filtered and washed with dilute HCl solution followed by double-distilled water repeatedly until the filtrate became colorless. It was oven-dried at 50°C .

2.3. Character Studies. Bare cotton fibers and polyaniline-deposited cotton fibers were characterized using Fourier transform-infrared (FT-IR) spectrometer (IRAffinity-1S, Shimadzu) via KBr disk technique in a spectral range of $4000\text{--}400\text{ cm}^{-1}$. Both samples were subjected to thermal analysis on the simultaneous thermal analyzer (DSC-TGA SDT-Q600) with the following parameters: a continuous nitrogen flow (20 ml/min) and a heating rate of $40^\circ\text{C}/\text{min}$ from 25°C to 600°C . The surface morphology of bare and polyaniline-deposited cotton fibers was observed using a highly customizable scanning electron microscope (SEM) (JSM-IT300LV, JEOL, USA) coupled with a port for

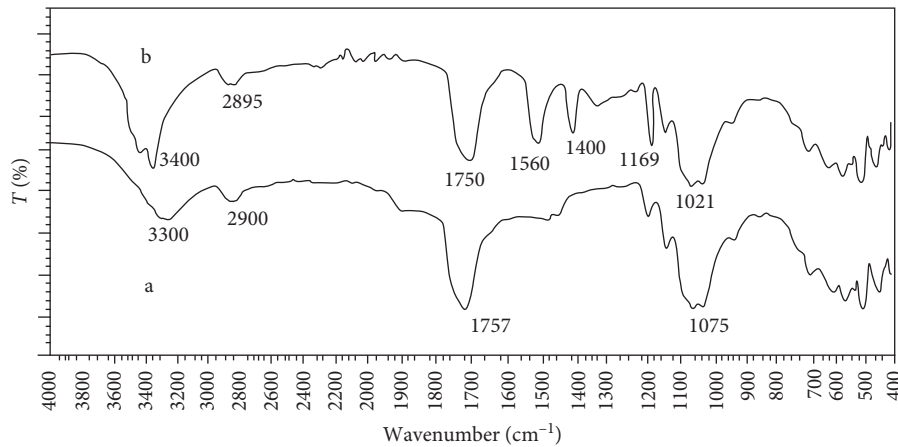


FIGURE 2: FT-IR spectra of (a) bare cotton fibers and (b) polyaniline-deposited cotton fibers.

analytical attachment of energy dispersive X-ray (EDX) spectrometry. The samples were gold-coated by the sputtering technique to avoid charging and observed under different magnifications.

2.4. Washability Test for Polyaniline. Polyaniline-deposited cotton fiber immersed in chloroform and deionized water (as control) is exposed to Mesdan Autowash with a speed of 40 rpm at 60°C for 90 minutes wash time. The concentration of extracted polyaniline was then analyzed using PerkinElmer Lambda 9 UV-Visible spectrophotometer in the wavelength region of 200–800 nm [25].

2.5. Water Absorption. Water absorption studies were performed according to ASTM D 570-99 method [26] with minor modifications. 1 g of bare and polyaniline-deposited cotton fiber specimens were prepared for water absorption experiment. Each specimen was fully immersed and soaked in double-distilled water for 12 hours at room temperature. At the end of the immersion, the samples were taken out from the water, the surface water was taken off using tissue, and wet weight values were measured and reported in weight percent.

2.6. Swelling Behavior. Swelling behavior of cotton and polyaniline-deposited cotton fiber was measured using an optical microscope which is well fitted with a high-resolution Leica camera. It also had image analysis software (Mesdan video analyzer) which allowed the measurement of fiber diameter at various points [25]. The presented diameter was an average of measurements taken at ten different locations along the fiber length.

2.7. Electrical Conductivity Tests. Electrical conductivity of polyaniline-deposited cotton fibers was measured using a portable conductometer device (Hanna Instruments HI 8033) by dissolving in chloroform.

3. Results and Discussion

3.1. Fourier Transform-Infrared (FT-IR) Spectroscopy. Weak bands at 2900 and 2895 cm^{-1} (Figure 2) correspond to CH symmetric stretching vibration of hydrocarbons derived from cellulose of cotton fibers. The intense peak at 1757 and 1750 cm^{-1} is recognized as C=O stretching vibration of carbonyl groups of cellulose [27] derived from cotton fibers. It is noticeable that there is a slight shift toward lower wavelength and diminished peak intensity as a result of polyaniline deposition, which might be attributed to weaker penetration capacity of infrared energy [28]. Appearance of new bands at 1400 and 1560 cm^{-1} represents C-N stretching vibration of benzoid (N-B-N) and C=N stretching mode of quinoid (N=Q=N) ring, respectively, which confirmed the deposition of polyaniline in the partially oxidized form, emeraldine salt. The peak at 1169 cm^{-1} is characteristic of polyaniline conductivity, which is a measure of electron delocalization [7]. The common band around 1021 and 1075 cm^{-1} belongs to C-O β -glycosidic linkage of glucose [29] in cellulose.

Furthermore, the peak at approximately 3400 cm^{-1} belongs to N-H stretching vibration of the amino group. This shows the existence of polyaniline [30] within the fiber structures, which proved inclusion of polyaniline as a result of *in situ* chemical polymerization. The other band at 3300 cm^{-1} in the bare cotton fibers (Figure 2(a)) represents O-H stretching vibrations which confirmed the presence of hydrogen bonding within the cellulose of cotton fibers [31].

3.2. Washability Tests via UV-Visible Spectroscopy. The filtrate after washing in chloroform was visually identified, and a greenish solution alongside faded cotton fibers was observed. From UV-Vis absorption spectra of chloroform filtrate solution, three distinct bands are visualized. The strong absorption peak at the maximum wavelength (λ_{max}) of 300 nm (Figure 3(b)) represents π - π^* transition from benzoid (N-B-N) ring of the polyaniline backbone. The other peak which appears at 425 nm is the feature of polaron- π^* transitions. One final band at 610 nm is assigned to π -polaronic transitions [32, 33]. These evidences proved that

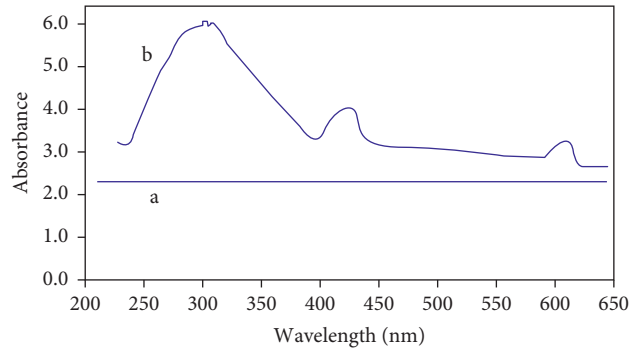


FIGURE 3: UV-Vis spectra of polyaniline-deposited cotton fibers in [(a) deionized water and (b) chloroform], respectively.

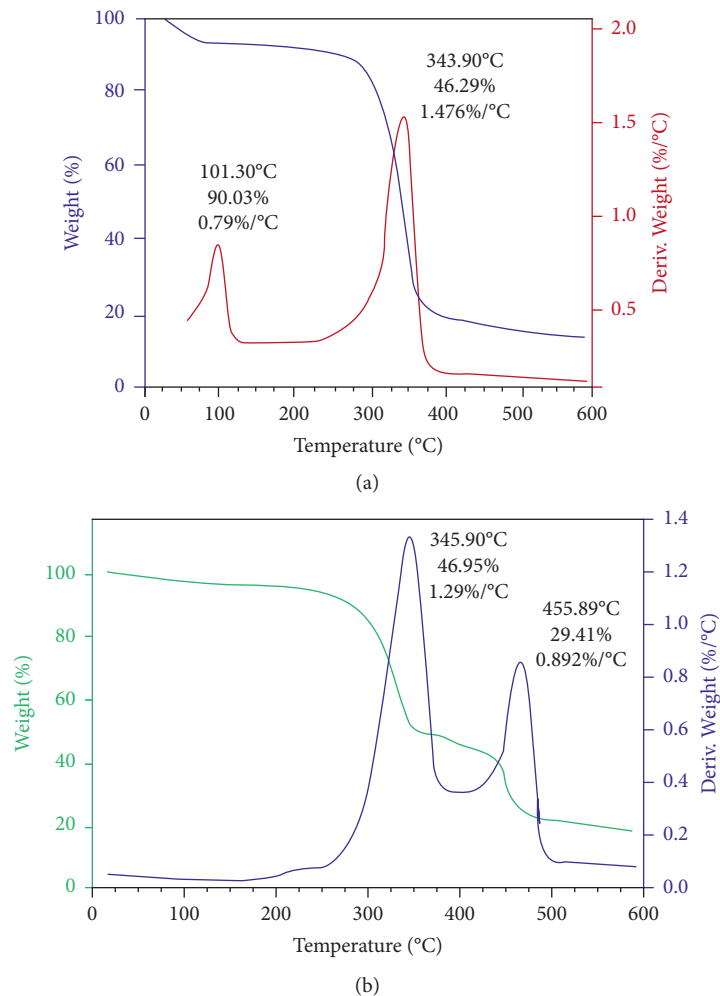


FIGURE 4: TGA thermogram of (a) bare cotton fibers and (b) polyaniline-deposited cotton fibers.

polyaniline was deposited either by weak chemical intact or via simple physical means. No absorption peak was observed in the deionized water filtrate solution (Figure 3(a)). This is an evidence that polyaniline is insoluble in water and in other polar media. Results of this study are in good agreement with prior works by the authors on sisal fibers [25].

3.3. Thermogravimetry Analysis (TGA). The upper stage of the thermogram of bare cotton fibers (Figure 4(a)) which extends up to 101°C is due to the evolution of moisture content within the fiber structure. The second step at approximately 344°C shows the breakage of the glycosidic link and bond cleavage of C-H, C-C, and C-O from the cellulose of cotton fibers [34].

TABLE 1: Weight loss of bare cotton fibers and polyaniline-deposited cotton fibers as a function of temperature.

Sample	Temperature (°C)	Weight loss (%)	Process	Reference
20 mg of bare cotton fiber	60–120	9.42	Dehydration of water/moisture	[31]
	275–400	53.71	Decomposition of organic matter	
	400–600	—	—	
20 mg of polyaniline-deposited cotton fiber	60–130	—	—	[36]
	275–375	53.05	Decomposition of organic matter	
	380–600	23.64	Degradation of the polyaniline backbone	

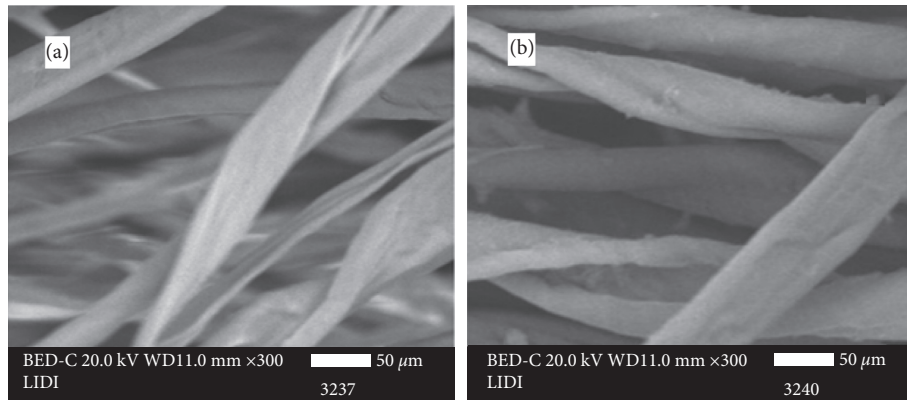


FIGURE 5: SEM micrographs of (a) bare cotton fibers and (b) polyaniline-deposited cotton fibers.

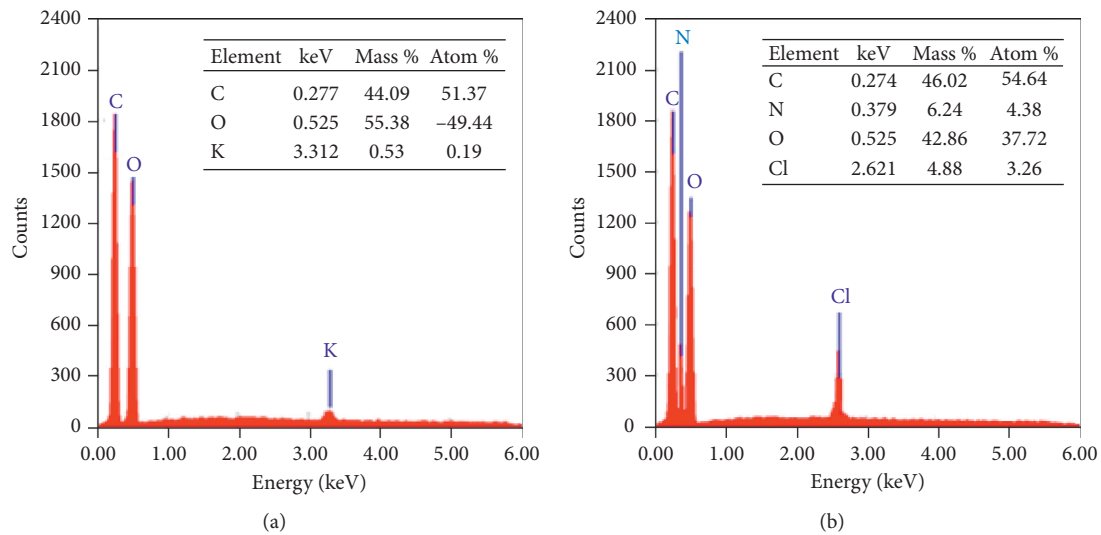


FIGURE 6: EDX spectra of (a) bare cotton fibers and (b) polyaniline-deposited cotton fibers.

In the case of polyaniline-deposited cotton fibers (Figure 4(b)), the first stage at 346°C represents the bond cleavage of C-H, C-C, and C-O from the cellulose of cotton fibers. The last stage beyond 456°C, which is thermally stable compared to the bare cotton fibers, represents a degradation of the polyaniline backbone (Table 1) [35].

3.4. Surface Morphology (SEM/EDX). Polyaniline deposition has shown considerable variations on the surface morphology of cotton fibers. At a glance of naked eye, *in situ* polymerization has completely changed white cotton fibers

to dark green suggesting that a conductive form of polyaniline was introduced [3]. The surface morphology of polyaniline-coated cotton fibers (Figure 5(b)) revealed that major section of the surface is full of roughness and fluffy fur or needle-like structures, while SEM micrograph of bare cotton fibers (Figure 5(a)) is smooth and clean. These morphological variations might be a result of polyaniline deposition on the surface of cotton fibers.

EDX spectrum (Figure 6(b)) showed the appearance of new peaks in $NK\alpha$ 0.379 keV and $ClK\alpha$ 2.621 keV [23], which proved that polyaniline was successfully deposited on the surface of cotton fibers. Detection of such peaks

TABLE 2: Water absorption, swelling behavior (in terms of diameter), and electrical properties of bare cotton fibers and polyaniline-deposited cotton fibers.

Sample	Fiber diameter (μm)	Electrical conductivity (Scm^{-1})			Water absorption (12 h) (%)		
		Cycle I	Cycle II	Cycle III	Cycle I	Cycle II	Cycle III
Bare cotton fibers	26.12	3.42×10^{-10}	3.25×10^{-10}	3.39×10^{-10}	26.3	25.9	25.82
Polyaniline-deposited cotton fibers	46.93	1.54×10^{-4}	7.01×10^{-7}	4.2×10^{-8}	3.4	14.6	16.5

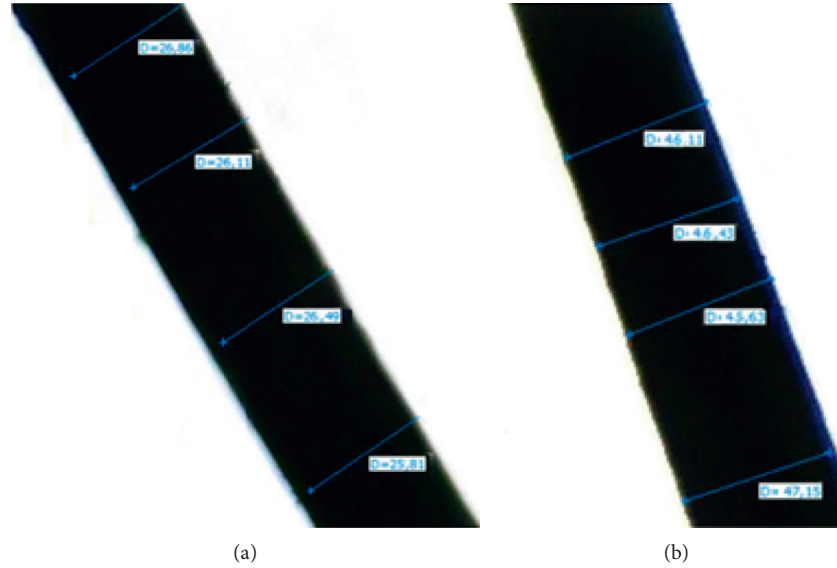


FIGURE 7: Image of optical microscope of (a) bare cotton fibers and (b) polyaniline-deposited cotton fibers.

confirmed the inclusion of chlorine (counter anion) as dopant and nitrogen from the polyaniline backbone, respectively [8, 37]. Existence of trace impurities such as potassium (Figure 6(a)) might be introduced from the reactants, solvents, and the fiber itself and was removed during *in situ* oxidative polymerization.

3.5. Water Absorption. Water absorption (equation (1)) data of bare and polyaniline-deposited cotton fibers calculated by weight difference are shown in Table 2. The experimental water absorption data of cotton fibers were 26.3% which abruptly dropped to 3.4% up on the subsequent polyaniline deposition [25, 38]. This incidence proved that the surface chemistry of cotton fibers is notably hydrophilic [17, 39], which is diminished up on *in situ* deposition of polyaniline thereby introducing hydrophobicity to the system [25, 39]. Water absorption features of polyaniline-deposited cotton fibers are increased on further washing by chloroform (compare values of cycle II and III).

$$\text{Water absorption (\%)} = \left(\frac{W_w - W_d}{W_d} \right) \times 100, \quad (1)$$

where W_d and W_w are the dry and wet weights of cotton fibers before and after soaking in the water system, respectively.

3.6. Swelling Behavior. *In situ* deposition of polyaniline on the surface of cotton fibers as a function of swelling (fiber diameter) recorded from optical microscope is summarized in Table 2. Thickness of cotton fibers (expressed in terms of fiber diameter) was increased by about 20 microns as a result of *in situ* deposition of polyaniline. The enhancement of fiber thickness is clearly visualized using markings from optical microscope (Figure 7). Even though the researchers are unable to fully support using other experimental evidences, data of swelling behavior show impregnation of polyaniline to the inner structures of cotton fibers [40].

3.7. Electrical Conductivity Studies. The electrical conductivity of bare cotton fibers is $3.42 \times 10^{-10} \text{ Scm}^{-1}$ while that of polyaniline-deposited cotton fibers was $1.54 \times 10^{-4} \text{ Scm}^{-1}$. Based on the level of conductivity, polyaniline-deposited cotton fibers lie in the semiconducting level, which is a clear evidence that polyaniline is deposited in its partially oxidized salt form [8, 41]. The level of electrical conductivity decreased significantly on further washing by chloroform (Table 2), which confirmed fade out of chemically deposited polyaniline via physical process. However, repeated washing could not nullify electrical conductivity which might be due to the existence of impregnated polyaniline within the fiber interstructures. This incidence is further supported by water absorption data (Table 2).

4. Conclusion

The main findings are summarized in the following manner:

- (i) Peaks from EDX spectra at $\text{NK}\alpha$ 0.379 keV and $\text{ClK}\alpha$ 2.621 keV proved inclusion of polyaniline in the salt form, emeraldine salt
- (ii) Introduction of polyaniline changed nonconducting cotton fibers ($3.42 \times 10^{-10} \text{ Scm}^{-1}$) to semiconductor materials ($1.54 \times 10^{-4} \text{ Scm}^{-1}$)
- (iii) Polyaniline deposition on cotton fibers reduced water absorption features by about 20% relative to the bare cotton fibers
- (iv) SEM microstructures confirmed formation of fluffy fur or needle like structures up on polyaniline deposition compared to smoother cotton fibers

Hence, polyaniline was successfully deposited on the surface of cotton fibers using iron (III) chloride as oxidant and dopant via *in situ* oxidative polymerization route which was verified via spectroscopic and microscopic instruments.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there are no conflicts of interest.

Acknowledgments

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