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Reduction of Surface Resistance of Jute Fiber by Coating with PEDOT and Characterization of the Effect of Different Parameters on Resistance

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ABSTRACT

Poly (3, 4-ethylenedioxythiophene) (PEDOT) is a highly conductive polymer which is most widely used for surface coating purposes. In this study for reducing surface resistance oxidative chemical vapor deposition (OCVD) technique was used for coating PEDOT on jute fiber, which is composed of cellulosic natural fiber and ferric (III) chloride ($FeCl_3$) was used as an oxidant for surface modification before polymerization. The objective of this study was to reduce resistance of jute fiber and improve conductive behavior. The surface resistance of jute fiber was investigated by changing different polymerization parameters such as oxidant concentration, soaking time, drying time, reaction time and reaction temperature. Optimum resistance values were recorded for 15% oxidant concentration, 150 min soaking time, 30 min drying time, 75° C temperature and 30 min reaction time. The novelty of this work is to introduce jute fiber for conductive material formation through PEDOT deposition technique.

1. Introduction

Jute is a cellulosic natural bast fiber which was previously known as the golden fiber of Bangladesh due to its contribution as the most foreign currency earning sector and also for its golden color. Although from the last few decades' plastic industry has been dominating jute market due to its diversity, attractiveness, user-friendly and cheap production cost, plastic materials are not biodegradable like jute products [14]. Now-a-days production of biodegradable and eco-friendly materials getting more interest and researchers are more concern about sustainable development. So the golden fiber of Bangladesh is about to get its life back. One of the best example was reported

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in a previous work that, “Bangladeshi scientist Dr. Mubarak Ahmad Khan, presently working as scientific advisor of Bangladesh Jute Mills Corporation (BJMC) and former Chief Scientific Officer of Bangladesh Atomic Energy Commission invented a biodegradable and eco-friendly bag from jute cellulose” [14] which may start a new era of jute.

Although the first purpose of clothing was to cover human body, its functional and aesthetic purposes are getting interest day by day, so the concept of smart textiles or intelligent textiles are being popular in modern society. “Smart textiles, encompassing electronics combined with textiles also called textronics, have a very promising realm in science and technology nowadays because of commercial viability and public interests” [2]. Smart textiles can sense and react to environmental conditions or stimuli, from mechanical, thermal, magnetic, chemical, electrical, or other sources [3,4]. Normal electronic materials are not flexible and wearable but e-textiles are flexible, wearable and at the same time they possess conductive and sensing behaviors [23,24]. Like other commercially available textile fibers as PEDOT/PSS coated nylon, viscose and polyester fibers [5-8, 25], PEDOT coated jute fibers can be used as conductive fibers for smart textiles, heat generation, semiconductor applications and military applications. Untreated jute fiber has a huge resistance, approximately 300-1200 M ohm/cm [9]. So coating is used to reduce the resistance of jute fiber. For coating cellulosic fibers there are several conductive polymers such as, Polyaniline (PANI), polypyrrole (PPY), poly (3,4-ethylenedioxythiophene) (PEDOT), poly (3,4-ethylenedioxy thiophene), poly (styrenesulfonate) (PEDOT:PSS) etc. but among all PEDOT is most widely used, because it has high conductivity up to 300 S/cm and more environment-friendly than other conductive polymers [10-12]. So it may be a great scope to modify jute fiber with PEDOT for smart textile applications.

Several chemical vapor deposition (CVD) techniques are available for coating such as atmospheric pressure CVD, low pressure CVD, oxidative chemical vapor deposition (OCVD), ultra high volume CVD, liquid inject CVD, microwave plasma-assisted CVD etc. In this study OCVD technique has been used among all the techniques, because jute is a cellulosic fiber and cellulose do not react directly with 3,4-ethylenedioxythiophen (EDOT). An oxidation reaction is needed to initiate the polymerization reaction of EDOT monomer & jute fiber. For this reason, $FeCl_3$ was used as an oxidant. This study focused on the reduction of jute fiber resistance.

As it is known that conductivity is exactly equal to the inverse of resistivity, so the less will be the resistance of a fiber, the more will be the conductivity. If a fiber possesses less resistance it will be applicable for smart textile applications. Xue et al. [21] reported that uniformly coated textile fiber shows satisfactory performance for conductive fiber sensors and actuators. So the objective of this study was to modify jute fiber surface and improve its conductive behavior for e-textile applications for transportation of energy and data [22].

2. Materials and Methods

2.1 Materials

In this work, jute fiber fibers (172 tex, z-twist, 158 TPM) were collected from “The Crescent Jute Mills Co. Ltd” Khulna. Detergent, wetting agent, sequestering agent, stabilizer, caustic soda and hydrogen peroxide was used for pretreatment purpose. Ferric (III) chloride ($Fecl_3$) (Sigma-Aldrich, 98%), methanol (CH_3OH) (Aldrich, 99%) EDOT monomer (CLEVOUS1M V2) was used for oxidative chemical vapor deposition reaction.

2.2 Pretreatment

As jute fiber contains both cellulose and lignin, the first step of this work was to remove the lignin. The jute fibers were cut into 15cm length and pretreated with 2g/L detergent, 3g/L wetting agent, 2g/L sequestering agent, 3g/L stabilizer, 4 g/L caustic soda and 14 g/L H_2O_2. This pretreatment was carried out for 1hour at M:L=1:10, 70º C temperature and p^h value of 11. In this way most of the lignin contents appears to be decreased.

2.3 Oxidation by $FeCl_3$

The pretreated jute fibers were submerged into 5-20 weight % of ferric (III) Chloride ($FeCl_3$), stirred gently and kept for soaking at room temperature for 10 min to 24 hours. This solution of oxidant was prepared by diluting $FeCl_3$ into methanol (CH_3OH). After oxidation drying time was maintained from 5 to 60 min and drying temperature was room temperature of the lab.

2.4 Deposition of PEDOT onto jute

In this work for deposition of PEDOT upon jute, oxidative chemical vapor deposition (OCVD) method was used which is one of the most widely used deposition technique for conductive polymers. At first the oxidized jute fibers were inserted into a tubular vacuum deposition glass tube. In another separate flask EDOT monomer was kept and there was a connection between the reaction chamber and the flask of EDOT monomer. For this reaction a carrier gas was needed and nitrogen gas was used for this purpose. To vaporize the EDOT monomer 70°C temperature was maintained in the flask.

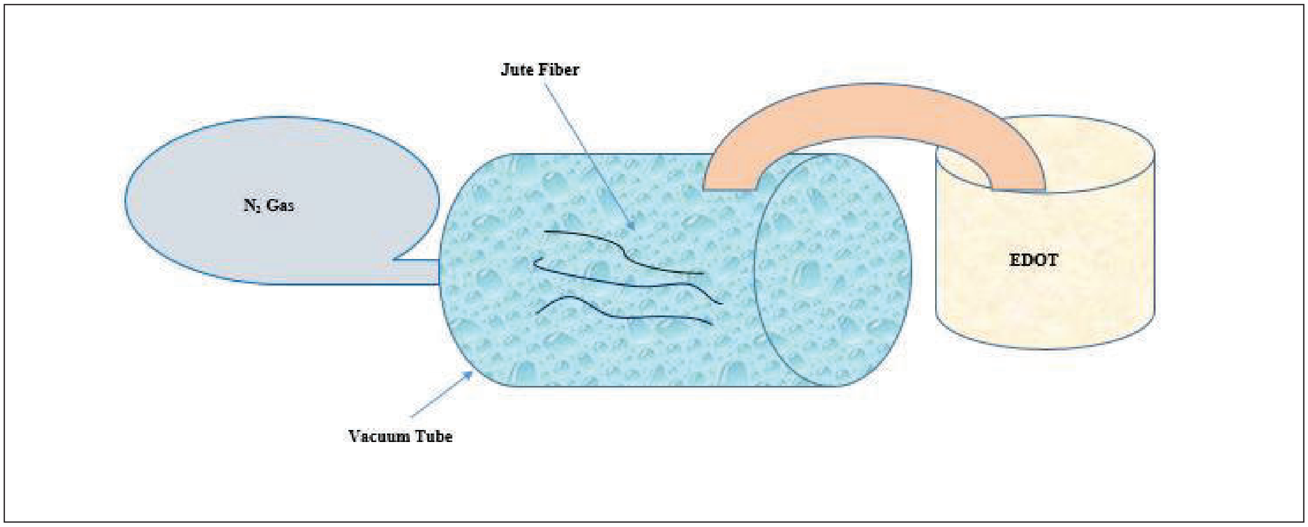


Figure 1: Schematic diagram of PEDOT deposition.

The vaporized EDOT monomer was carried by the nitrogen gas to the reaction tube where the polymerization reaction occurred at 75°C temperature. The reaction times varied from 10 min to 120 min. To remove the unreacted monomers and byproducts, the coated jute fibers were again treated with $FeCl_3$ solution at room temperature. Then the treated jute fibers were dried at room temperature.

2.5 ATR-FTIR spectroscopy

The deposition of PEDOT on jute fiber was investigated in ATR mode by SHIMADZU IRSpirit FT-IR spectrometer with QATR-S attachment. Number of scan was 32, band resolution was 4 cm-1 and Range of spectra was 600 to 2000 cm-1.

2.6 Characterization of modified jute fiber

At first the resistance of untreated jute fibers was measured with Fluke 177 True-RMS digital multi-meter (Resistance range: up to 50M Ω, Accuracy: +/- 0.9+1). Then the resistance of different treated jute fiber samples was measured by changing different polymerization parameters such as oxidant concentration, soaking time, drying time, reaction time and reaction temperature. During the assessment of one parameter all other parameters were kept constant. Then the measured values of resistance were tabulated and analyzed.

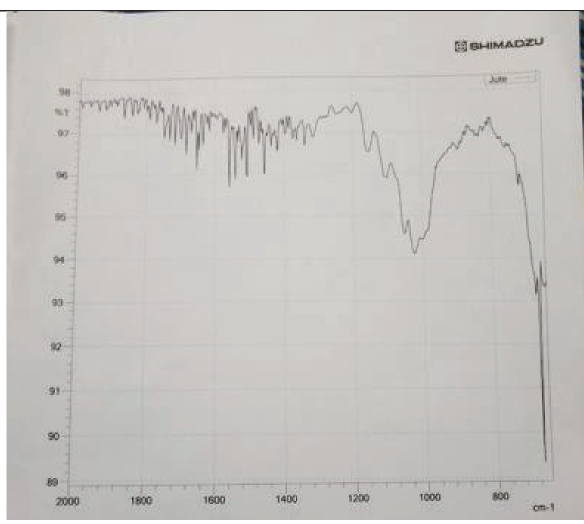
3. Results and Discussion

As jute is a cellulosic natural fiber, it contains huge amount of hydroxyl (OH^{-}) group in its structure, so it has a strong interaction with $FeCl_3$. For the initiation of polymerization reaction of jute with EDOT vapor, $FeCl_3$ was used in pretreatment reaction. As previously reported that $FeCl_3$ is absorbed efficiently by cellulosic materials when it is pretreated by it before polymerization reaction [15,17]. Jute fiber structure is very suitable for polymerization of conductive polymers. Since $FeCl_3$ treated jute fibers shows a strong interaction with EDOT vapor, after polymerization reaction it acts like an integrated parts of jute fiber. As PEDOT is a highly conductive polymer, PEDOT coated jute fiber shows a lower resistance that means a good conductive behavior. As shown in Figure 2, oxidized jute fibers look deep brown in color and after coating the color of fiber turns into darkish blue as a layer of PEDOT forms upon it.

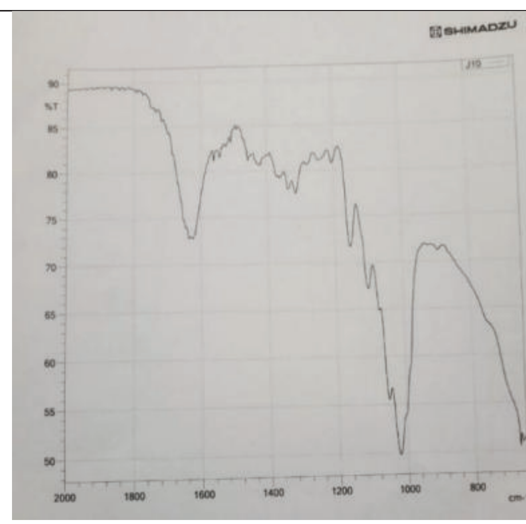
Figure 2: (a) Oxidized jute fiber (b) Coated jute fiber.

3.1 FTIR analysis

PEDOT deposition on jute fiber was investigated by ATR-FTIR analysis which is shown in Figure 3. FTIR spectra of pure jute fiber is illustrated in Figure 3(a), which is showing a strong peak at 1040 cm-^{1} and some weak peaks at 1060, 1120, 1160 cm-^{1}. FTIR spectra of PEDOT coated jute fiber is illustrated in Figure 3(b), which is showing similar peaks as pure jute but there is a strong peak visible at 1640 cm-^{1} which indicates the absorption of PEDOT as the peaks of PEDOT are found mainly at the range of 1045 to 1700 cm-^{1} although some PEDOT peaks are found above 3000 cm-^{1} [5, 6, 26]. So from the FTIR spectra it can be concluded that enough PEDOT has been absorbed by jute fiber.



(a)



(b)

Figure 3: FTIR spectra of (a) Pure jute fiber (b) PEDOT coated jute fiber.

3.2 Effect of $FeCl_3$ concentration on resistance of jute fiber

The extent of polymerization reaction of jute fiber depends on the quality of surface modification. For starting the polymerization reaction, the surface of the jute fibers needed to be modified sufficiently and the surface modification depends on oxidant concentration. Recent study has recommended that, for deposition of PEDOT in vapor phase polymerization technique, the concentration of $FeCl_3$ has a potential influence on conductivity and surface morphology [16,19,20]. Figure 4 shows prepared oxidant solutions with different $FeCl_3$ concentrations. In this study 5%, 10%, 15% and 20% solution of $FeCl_3$ has been used. In a previous work of PEDOT deposition on natural fiber, researchers found 3-15% as optimum $Fecl_3$ concentration [5].

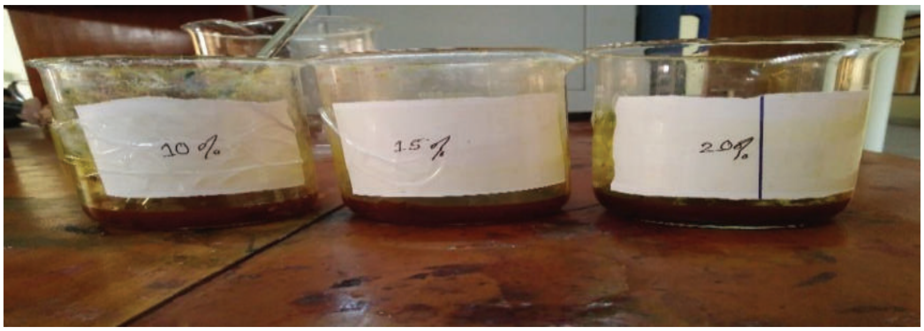


Figure 4: Prepared oxidant solutions with different concentrations

During the measurement process, the value of resistance for the untreated jute fiber was showing nothing, it might be due to the resistance value was beyond the maximum range of the multi-meter. The resistance values for 5% $FeCl_3$ solution and for 10% $FeCl_3$ solution is shown in Figure 5. For 5% $FeCl_3$ solution the resistance values were recorded from 0.74 kΩ to 1.51 kΩ. For 10% $FeCl_3$ solution the resistance values were recorded from 0.62 kΩ to 1.16 kΩ.

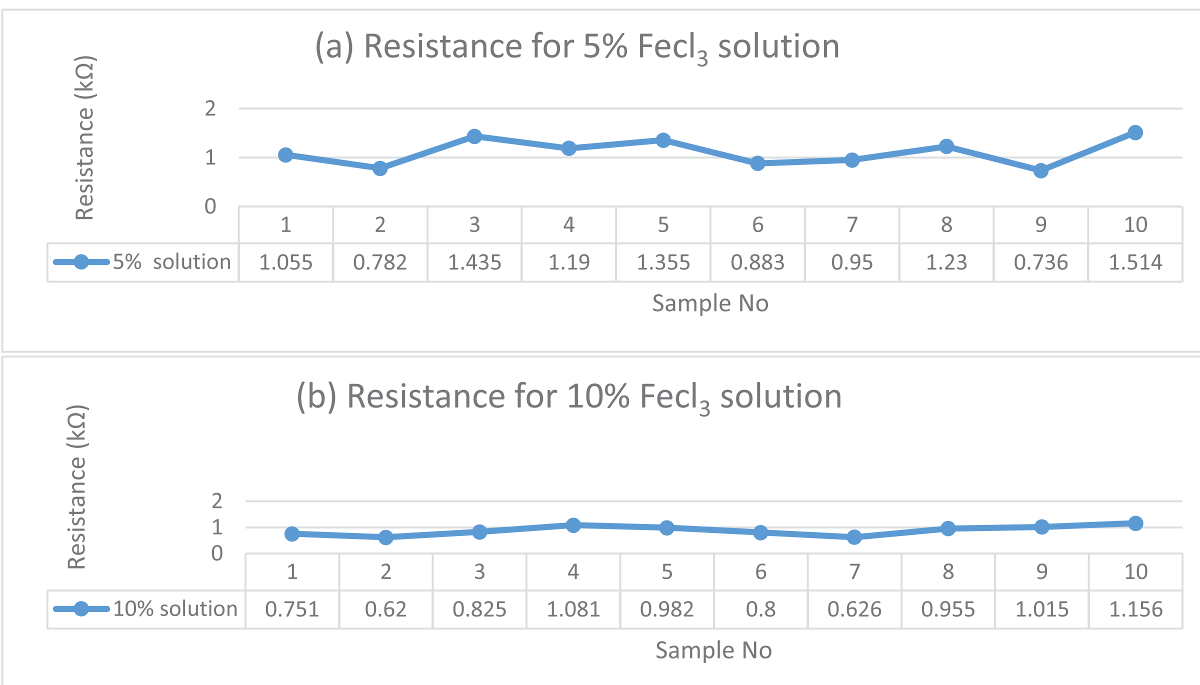


Figure 5: Effect of FeCl3 concentration on resistance (a) for 5% solution (b) for 10% solution.

The resistance values for 15% $FeCl_3$ solution and for 20% $FeCl_3$ solution is shown in Figure 6. For 15% $FeCl_3$ solution the resistance values were recorded from 0.46 kΩ to 1.05 kΩ. For 20% $FeCl_3$ solution the resistance values were recorded from 0.50 kΩ to 3.03 kΩ.

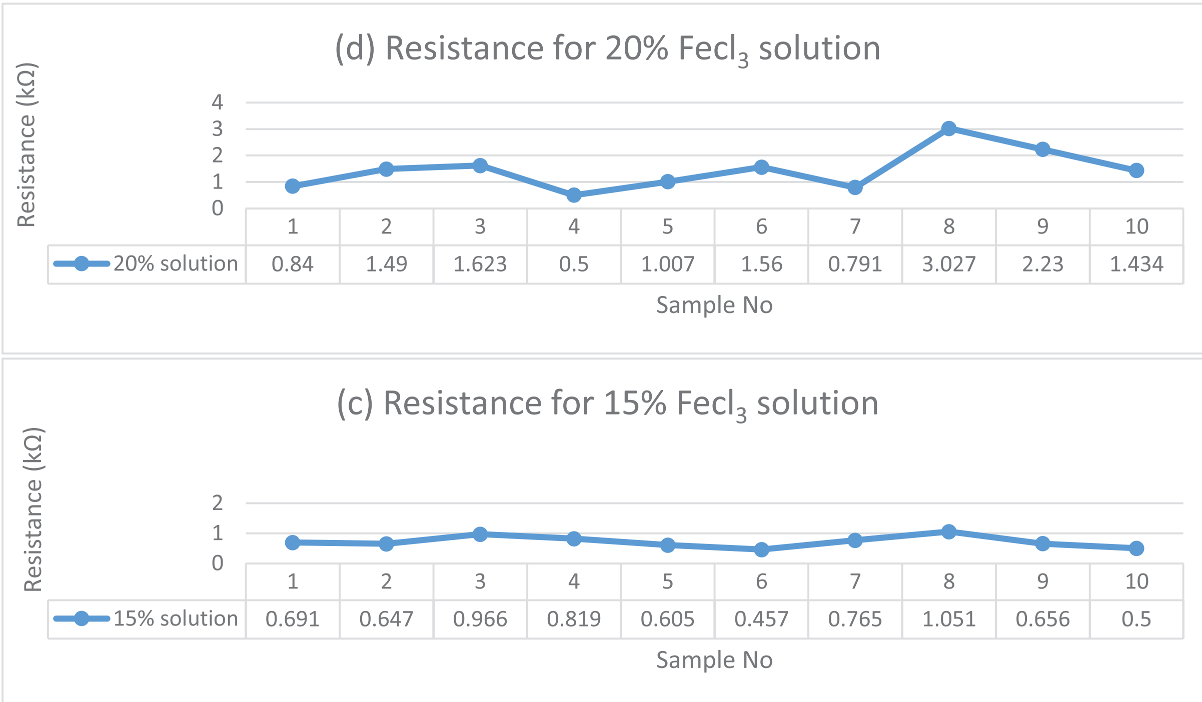


Figure 6: Effect of FeCl3 Concentration on resistance (c) for 15% solution (d) for 20% solution.

For 5 % $FeCl_3$ solution, the resistance measurement showed very high values of jute fibers and as the $FeCl_3$ concentration increased to 10% keeping the other polymerization parameters constant, the resistance values decreased. It might be because of low oxidation energy of $FeCl_3$ at 5 % concentration. With the increment of $FeCl_3$ concentration, the oxidation energy of jute fiber increased [5,6] as a result effectiveness of polymerization reaction increased and resistance decreased.

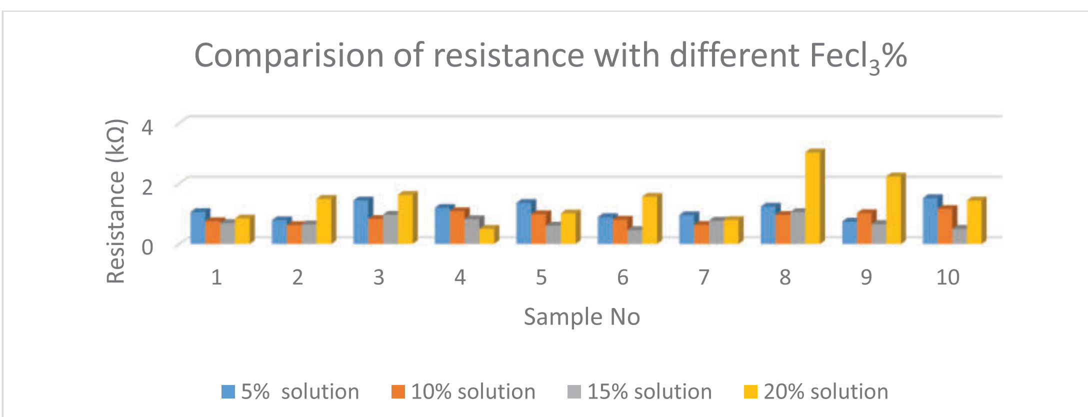


Figure 7: Comparison of jute fiber resistance with different FeCl3 concentration

Again resistance values more decreased after increasing the $FeCl_3$ concentration to 15%, this might be due to the same reason as before, however the resistance values again started to increase after increasing the $FeCl_3$ concentration to 20%. Figure 7 shows the comparison of jute fiber resistance with different $FeCl_3$ Concentrations. As reported in a previous work, if the $FeCl_3$ concentration further increases the resistance values will also be increased due to the formation of short chained polymer of PEDOT [18]. So it can be suggested that, if all other polymerization parameters kept constant then 15% concentration of $FeCl_3$ would be better for reduction of jute fiber resistance by coating with PEDOT.

3.3 Effect of jute fiber soaking time on resistance

Figure 8 is showing the effect of jute fiber soaking time on resistance. The soaking time has been checked for 30 min, 60 min, 90 min, 120 min, 150 min and finally for 1440 min (24 Hour). It is noticed from the figure that with the increment of soaking time of jute fiber in $FeCl_3$, resistance values were decreasing sequentially, this might be due to more oxidant absorption by jute fiber for longer soaking time. But the resistance value of jute fibers again started to increase after 24-hour soaking. It would be due to absorption of surplus oxidant solution and forming unconjugated polymer structure of PEDOT [13].

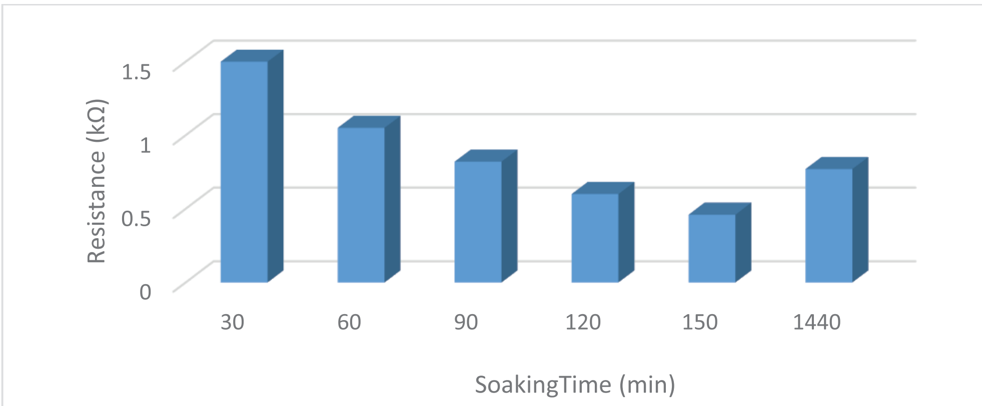


Figure 8: Effect of jute fiber soaking time on resistance.

3.4 Effect of drying time

The effect of jute fiber resistance on drying time is shown in Figure 9, drying effect was checked for 10 min, 15 min, 20 min, 25 min, 30 min, 35 min and finally for 40 min after oxidation. As the drying time increases, the resistance values of jute fiber decreases up to 30 min. But when drying time exceeds 30 min the resistance values started to increase. It would be due to the stabilization of chloride (Cl^-) active ions that might work for EDOT monomer oxidation [5]. So it can be concluded that better drying time of jute fiber after oxidation would be 30 min for optimum resistance value if all other parameters kept constant.

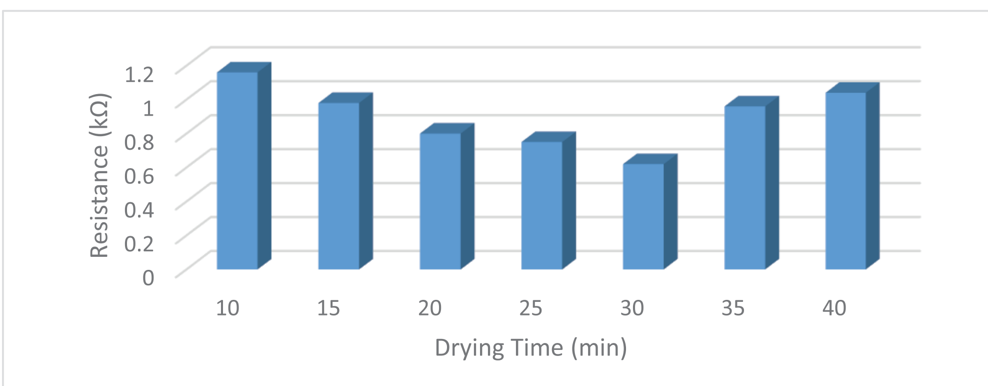


Figure 9: Effect of drying time on resitance.

3.5 Effect of reaction time

Figure 10 is showing the effect of reaction time on jute fiber resistance, which was checked for 10 min, 20 min, 30 min, 40 min and 50 min. As the reaction time increases from 10 min to 30 min, the resistance values decreases gradually but after 30 min the resistance values started to increase with increasing reaction time. The probable cause would be fulfilment of the polymerization reaction at 30 min and after 30 min due to exposer of PEDOT polymer at high temperature for longer time, its quality may decrease so resistance increases [5]. The continuous reaction of polymerization also depends on the presence of EDOT monomer and oxidant [1]. So the better time of reaction may be suggested as 30 min.

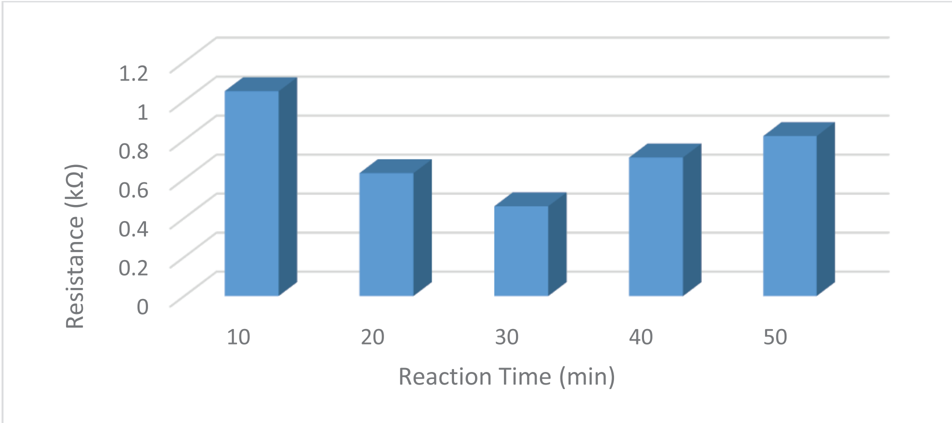


Figure 10: Effect of reaction time.

Conductive behavior of PEDOT coated material reduces due to several wash [26]. Short term durability was investigated by measuring resistance of coated jute fiber after one month of coating and seen around the same values of resistance but no long term investigation was conducted for the time more than one month and for after wash. Several studies have found that 3-15% oxidant concentration, 3-10 min drying time and 5-60 min reaction time is optimum for polymerization of EDOT [5,6], but these parameters may vary with variation of materials. From the above discussion it may be concluded that, optimum resistance values of jute fiber were recorded for 15% oxidant concentration, 150 min soaking time, 30 min drying time, 75° C temperature and 30 min reaction time.

4. Conclusions

The surface resistance of jute fiber has been reduced by polymerization reaction of EDOT using OCVD technique and the effect of different polymerization parameters has been studied here. Results shows that different parameters have a strong influence on jute fiber resistance. By controlling this parameters very lower resistance fibers are possible to produce which will be applicable for smart textile applications, heat generation, semi-conductor applications and other technical applications. As jute is a natural fiber material which is eco-friendly and easily biodegradable, its application in smart textile will play a positive impact on our earth and it will be better for a green and sustainable world.

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