

Development of an electrically conductive cotton yarn coated with polypyrrole polymer

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Abstract:-Conductive materials serve as the basis for smart textiles, an area which has attracted more research and innovation in the last decade. This research sought to utilize the readily available cotton yarn in Zimbabwe and a conductive polymer, polypyrrole, to develop conductive cotton composite. The conductive cotton yarn was developed through in-situ oxidative polymerisation of pyrrole in the presence of yarn, using both the solution and vapour phase coating techniques with ferric chloride as an oxidant. Resistance of the conductive yarn was measured using the two probe method according to test standard AATCC-76 1995 to determine its conductivity. Mass specific resistance values and uniformity in coating thickness of the two differently coated yarns were compared and examined for evaluation. The optimum concentration of oxidant was 1 mole/litre, where the least value of 0.10kilo-ohms electrical resistance was obtained on a 72tex rotor spun yarn. Vapour phase coating technique was faster and gave better uniformity in coating thickness.

Keywords:-conductive cotton yarn, electrical resistance, polypyrrole,

I. INTRODUCTION

The cotton fibre

Cotton fibre is readily available in Zimbabwe. Its market has been more restricted to simple applications such as clothing and other domestic uses. The properties of the cotton fibre such as, good strength, flexibility and breathability would make it possible to be engineered to behave in a predetermined way [1]. If cotton yarns were conductive they would be ideal to use in electrical and electronic textiles in countries such as Zimbabwe where cotton is an abundant crop. Poor electro conductivity has been the chief limitation in the use of cotton yarns as electrical conductors. The development of conductive yarns is critical for various applications in wearable electronics, such as wearable displays, solar cells, actuators, data managing devices, biomedical sensors, electro static discharge, electromagnetic interference protection and radio frequency interference protection, where these yarns can be engineered to be used to complete the circuits[1]. Development of a conductive cotton yarn will enable a paradigm shift in the applications of the cotton from domestic uses to advanced uses such as electronic automotive and medical textiles just to mention a few. The traditional applications of cotton yarn e.g. in clothing and other household goods remain strong. In order to qualify for electronic textiles, yarns should be conductive. To make the cotton yarns conductive they should be coated with polymers whose coating yield conductivity values range from 10^{-3} to 150 Siemens/cm(S/cm). This covers the semi-conductor and conductor range, depending on the efficiency of the polymerisation process and the reagents used[1, 2]. Intensive studies on conductive polymers show that conductive polymers such as polyaniline (PAni), polypyrrole (PPy), and polythiophene(PThi) have attracted a lot of interest by many researchers[2-4]. PAni and PPy have some advantages, which include unique electrochemical properties, good conductivity, easy synthesis, and also have thermal, environmental, and chemical stability[2, 3] Conductivity values of insulator materials are about

10^{-12} S/cm, the values of conductive polymers are in the range 10^{-5} to 10^2 S/cm².

Physical and chemical properties of cotton

Cotton is a relatively strong fibre with strength of about 3-5 N/tex.[5, 6]. It is a cellulosic fibre which may be described chemically as poly (1,4 β -D)-anhydroglucopyranose [5]. The cotton fibre structure is comprised of multiple individual cotton fibrils, which are in turn composed of multiple micro fibrils, bundled together. The micro fibrils are made of poly-D-glucose chains, usually arranged in crystalline, or partially crystalline, domains [5, 7]. This structure allows the fibres to absorb large amounts of water, or other polar solvents. In addition to structural advantage in chemical uptake, mercerization process can also be carried out in cotton yarns to improve moisture, or oxidant uptake [5]. Flexibility, breathability, good processability, availability among others are properties which make cotton best choice for conductive polymer coating[3]. Cotton shows a linear decrease in log Rs (resistance) with relative humidity from a specific resistance of 10^{11} ohm g/cm² at 10% relative humidity to 10^6 ohm g/cm² at 80% relative humidity. Static electrification becomes a serious problem for cotton only for values greater than 10^{10} ohm g/cm² (about 30% relative humidity) [7]. However, the resistance is high enough for cotton to be an effective insulator. Poor conductivity has been the limiting factor

inhibiting use of cotton in electronic textiles. If used as an insulator in dry conditions, cotton like any other textiles tends to have static charge build up which is undesirable [1, 2, 8]. Applications of poorly-conductive cotton yarns have been therefore more restricted to simple applications such as clothing and fashion industry, [1, 2, 9]. Cotton can be made conductive by combining it with a conductive polymer. Use of conductive polymers together with textile materials has some advantages, such as flexibility which provides ease in processability. Though metal coating gives better conductivity values, polypyrrole polymer coating was adopted for this study. Other commonly used conducting polymers include polyaniline, polythiophene and their derivatives. Polypyrrole was chosen because of its easy accessibility and ease of management during the coating process.

The Polypyrrole Polymer

Polypyrrole is a conducting material whose properties can be modified through chemical synthesis and composite processing. The polymer's conductivity can be varied over several orders of magnitude [10]. Thin film deposition, ease of synthesis, environmental stability, commercial availability and availability of reagents required for polymerisation, are some key advantages associated with polypyrrole [10]. The physical properties of the conducting polymer are highly influenced by the method of preparation, the characteristics of other additives in the reaction mixture, and the reaction conditions [11]

Physical properties of polypyrrole

Bulk quantities of PPy can be obtained as fine powder using the oxidative polymerization of the pyrrole monomer by selected transition metal ions in water or various other solvents [1, 2, 12]. When aqueous ferric chloride is used a very rapid reaction with pyrrole takes place resulting in a black powder (polypyrrole) which is insoluble in all common solvents [11]. PPy films thinner than 1 mm have different spectral properties depending on the conditions of synthesis and degree of PPy oxidation as shown in Figs 1 and 2.

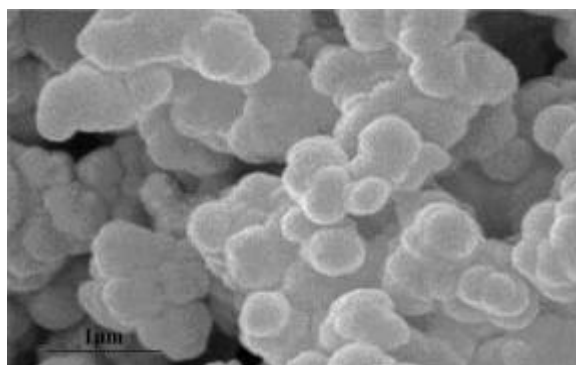


Fig. 1: Scanning electron micrograph of pure PPy in aqueous media. Reaction conditions: ($\text{FeCl}_3 = 48 \text{ g/L}$, pyrrole monomer $14.9 \times 10^{-2} \text{ mol/L}$, volume of solution 100 mL, reaction time 5 hours at room temperature [11, 13].

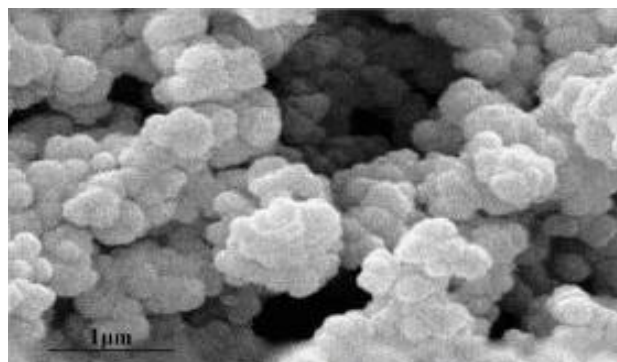


Fig 2: Scanning electron micrograph of pure PPy in non-aqueous media (methanol). Reaction conditions: ($\text{FeCl}_3 = 48 \text{ g/L}$, pyrrole monomer $14.9 \times 10^{-2} \text{ mol/L}$, volume of solution 100 mL, reaction time 5 hours at room temperature) [11, 13]. The films have good adhesion to the substrate. Thickness of over 10 mm however, are relatively easily peeled off. The adhesion depends on factors such as the nature, coarseness, hydrophobicity of the substrate surface as well as the solvent used [11, 12]. The stability of the doped PPy films in air is relatively high as their degradation occurs only above 150 - 300 °C (depending on the dopant anion). Thermal degradation of PPy starts with the loss or decomposition of dopant [4, 13, 14].

Chemical properties of polypyrrole

Polypyrrole (PPy) is a conducting polymer that has attracted special and wide research focus because of its low resistivity (good electrical conductivity), redox properties, fewer toxicological problems and environmental stability[1, 2]. In most cases, PPy has been considered as a one-dimensional polymer having a poly-conjugated backbone. This polymer is called a *p*-doped conjugated polymer because of the alternating single and double bonds in the polymer chain made up of five membered heterocyclic rings, '*p*' means that it is formed through oxidative polymerisation. As a conjugated conducting polymer however, the brittleness, infusible and insoluble nature of PPy owing to strong inter-chain bonding tends to limit its practical uses[12, 14]. Its other disadvantage is the instability to atmosphere in its non-conductive state[4]. In its conducting state, the main advantage is that PPy is inert to the atmosphere. Its radical changes spontaneously to positively charged PPy when it is dipped in an acidic aqueous solution and it can change when bonded to a hydroxyl ion if dipped in an alkaline solution[15]. PPy can be deposited onto insulative substrates such as cotton yarn through, chemical polymerization yielding polypyrrole-substrate composites[11]. The process ability and mechanical properties of this polymer can also be improved either by blending PPy with other polymers or by forming copolymers of PPy. Thus PPy-based composites may provide the cotton yarns with electrical properties similar to metals or semi-conductors. The conducting polypyrrole can be used in light weight electronics such as rechargeable batteries, electro-optical devices and in the medical field for controlled release of drugs[9, 11, 13, 14, 16].

This study meant to develop a conductive cotton yarn coated with polypyrrole, that will enable a paradigm shift in the uses of cotton from traditional uses to advanced applications such as electronic textiles. To achieve this there was need to ensure that yarn is prepared, as well as the pyrrole for in-situ polymerisation using ferric chloride as an oxidant. Polypyrrole was found suitable to use to coat the cotton yarn using solution polymerization and vapour phase polymerization methods. It was then necessary to compare the electrical resistance of uncoated yarn and the yarn coated with polypyrrole, and analyse of the effect of oxidant concentration using different solvents, as well as the effect of oxidant concentrations at different pyrrole monomer concentrations on the electrical resistance of the coated yarns were made.

II. METHODOLOGY

Experiments were conducted using the solution and vapour phase polymerisation coating methods[1].

Materials

Materials used were 100% Cotton, 72 tex, rotor spun yarn; 99% ferric chloride (FeCl_3); 97% pyrrole monomer as it does not require further purification; ethanol ($\text{C}_2\text{H}_5\text{OH}$); sodium hydroxide (NaOH); nitrogen gas (N_2); hydrogen peroxide (H_2O_2); and distilled water [1].

Preparation of yarn for polymerisation

The cotton yarns were initially mercerised and bleached to remove impurities and increase solvent monomer uptake. After that electrical resistance tests were conducted on the uncoated yarn

Mercerization and bleaching of the cotton yarn

A 72 tex cotton yarn was mercerized using NaOH in order to remove impurities, and increase solvent and monomer uptake of cotton yarn due to fibre swelling. It was then bleached using the following concentrations[2]: 35% hydrogen peroxide, 5% wetting agent, 0.5% stabilizer, in order to improve whiteness and easier colour change observation during coating[2]. Bleaching was conducted at a '*bleach to yarn ratio*' of 1:100 based on mass of yarns. The yarn mass ranged between 0.00093g/10cm for a 1cm specimen, 0.0093g/10cm for a 10cm specimen and 0.0186g for a 20cm specimen. The bleaching process was carried out at 95-100°C for 30minutes. The bleached cotton yarns were mercerised using 20% sodium hydroxide at 30 ° for one hour[2]. Yarns were rinsed and dried.

Measuring electrical resistance of uncoated yarn

Electrical resistance tests of uncoated yarn were carried out using the mega-ohm meter according to test method AATCC-76 1995 and also according to the two probe method (Fig 3).

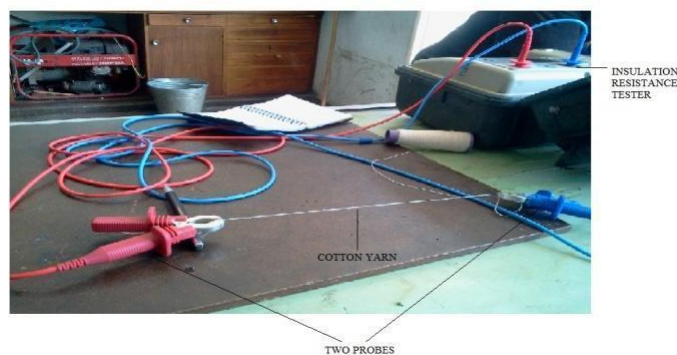


Fig 3: Setup for electrical resistance tests

A voltage of 250V was applied between two parallel probes (crocodile clips) holding the yarns at a distance of 20 cm, 10 cm and 1 cm apart. Resistance was recorded and used for comparison and analysis against that of coated yarns. Ten recordings were taken for each sample and used to calculate the average resistance. Tests were carried out at room temperature on samples that had been left to lie also at room temperature for 24 hours. The mass specific resistance was conducted using the mathematical expression [17]:

$$Rs = \frac{RNT}{l10^5} (1)$$

where R = resistance in ohms, l = distance between the ends of the specimen (cm), N = number of ends of yarn or fibre, and T = linear density of yarn or fibre in textile (g/1000m)

Coating yarn using vapour and solution phase methods.

Vapour and chemical (solution) phase polymerization methods were used in order to analyse the significant drop in resistance of yarns as a result of the use of each method. Water and ethanol solvents were used to dissolve Iron Chloride (FeCl_3). Polypyrrole coated yarns, treated with oxidant solutions prior to polymerisation, were analysed in order to determine the effect of solvents and their concentrations on conductivity.

Vapour phase polymerisation

The Vapour phase experimental set up was designed in such a way that the coating process was batch wise. Initially the yarns were pre-treated in oxidant solution as a separate process. The yarns were then taken to the polymerisation chamber, where they were held stationary and tensioned using corks (Fig 4).

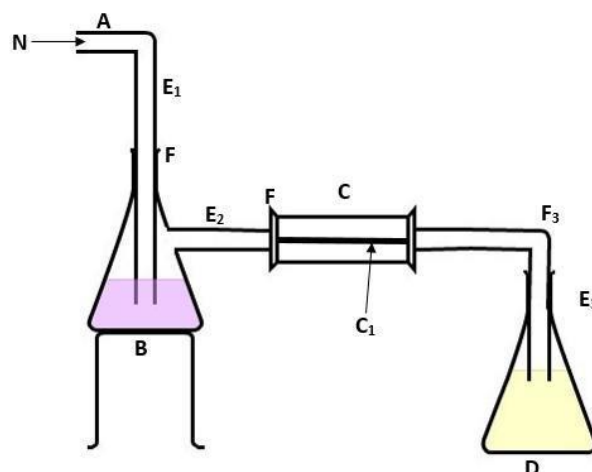


Fig 4: Schematic diagram of the vapour phase experimental set up [8]

A is the nitrogen gas, B is the conical flask with pyrrole monomer inside, C is the polymerisation column, the yarns, D is the conical flask with distilled water, E_1 to E_3 are plastic tubes and F_1 to F_3 are corks. The yarns were held at tension by corks F_2 and F_3 and were suspended within the polymerisation column.

Solutions of the initiator or oxidant in ethanol and water were also prepared at six different concentrations of FeCl_3 of 0.4, 0.6, 0.8, 1.0, 1.2 and 1.4 moles/litre. The cotton yarns were pre-treated with oxidant solutions for

one hour and then later inserted into the polymerisation column where they were held under tension. Cotton yarn substrates were then exposed to pyrrole vapour with nitrogen as the carrier gas in the polymerisation chamber, until the yarns turned black due to coating. The coating process for each yarn took approximately 2 to 5 seconds [5, 15].

Solution phase polymerisation

Solution phase was designed in such a way that the yarns were immersed in test tubes which were placed in beakers with ice-blocks to keep the temperature between 0°C and 5°C. The polymerisation beakers were placed in a card box and covered with black sheeting once pyrrole was added, to avoid exposure of pyrrole to light as it polymerises in light. Fig 5 shows the experimental setup of the solution coating method.

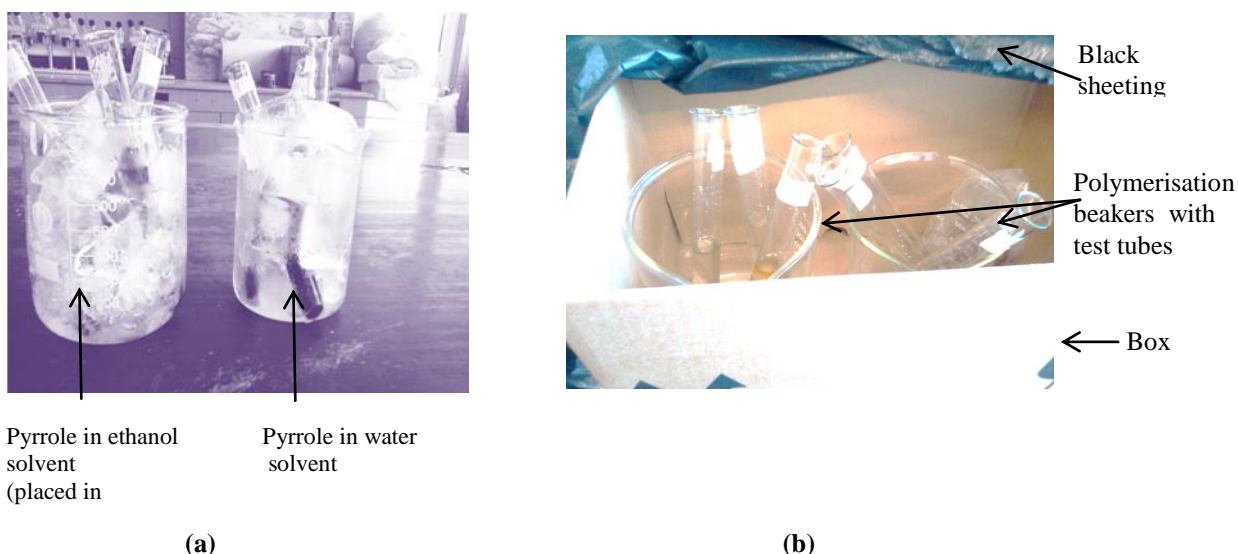


Fig 5 a) and b): Design and setup for solution phase coating

Required materials for solution phase polymerisation were:

Pyrrole of 3%, 5% and 10% concentration, FeCl₃ of concentrations of 0.4, 0.6, 0.8, 1.0, 1.2 and 1.4 moles per litre of solvent, as in vapour polymerisation, and material to liquor ratio of 1:100 for all solutions. Mass of yarns was determined using a balance with a precision of 0.0001g. Required amount of solution was calculated based on mass of yarns, and the amount of pyrrole monomer was calculated based on their pyrrole concentration required. The amount of FeCl₃ was calculated at the six varying concentrations (0.4moles/litre to 1.4moles/litre), for each of the solvents (water or ethanol). Cotton yarns were pre-treated in aqueous oxidant solutions and ethanol- oxidant solutions of varying concentrations for one hour. The pre-treated cotton yarns were then added to pre-cooled monomer solutions (5°C). The test tubes with pyrrole solution were placed in the cold beakers(Fig5). The polymerization process was carried out for 16 to 24 hours. Cotton yarns were washed in distilled water and dried under standard room conditions. They were then left to lie at room temperature for 24 before testing them for electrical resistance[10].

III. RESULTS AND DISCUSSION

Electrical resistance of uncoated bleached cotton yarns

Electrical resistance of the uncoated yarn was measured after application of a very high voltage of 250V using the mega-ohmmeter. The yarn diameter was found to be 0.04mm, a value that is in agreement with that of Uster Technologies[18]. Table 1 shows the average results obtained per each length of specimen.

Table 1: Electrical resistance and mass of the uncoated 72 tex yarn.

Length of specimen (cm)	Resistance ($\Omega \times 10^9$)	Volume resistivity ($\Omega \cdot \text{cm}$)	Conductivity (S/cm)
20	24.2	3.04×10^7	3.28833×10^{-8}
10	12.6	1.58×10^7	6.31567×10^{-8}
1	0.96	1.21×10^6	8.28932×10^{-7}

The electrical resistance of uncoated yarns was in the giga-ohms range. Their conductivity, calculated as an inverse of volume resistivity was in the range of 10^{-8} to 10^{-7} qualifying these yarns as insulators or very poor conductors. A perfect insulator should have a conductance of less than or equal to 10^{-8} S/cm [17, 19]. Resistance, however decreased with decrease in specimen length. This is because, resistance is directly proportional to length, hence the shorter the material, the lower the resistance.

Vapour phase polymerisation (coating)

Vapour polymerisation technique did not result in uniformly coated yarns on the surface, therefore electrical resistance was not the same throughout the coated yarns because of variations in absorption of the oxidant solution, the different physical properties of the yarn such as crystalline and amorphous regions, and the rapid time of coating. At about 2 seconds colour change could be noted making it difficult to control the coating process and therefore resulting in variations. Thick and thin coated places were observed on the surface of the yarn causing the yarn to become brittle at some places. This affected the electrical resistance of the yarn. The effect of the oxidant concentration on the resultant polymer electrical resistance was determined.

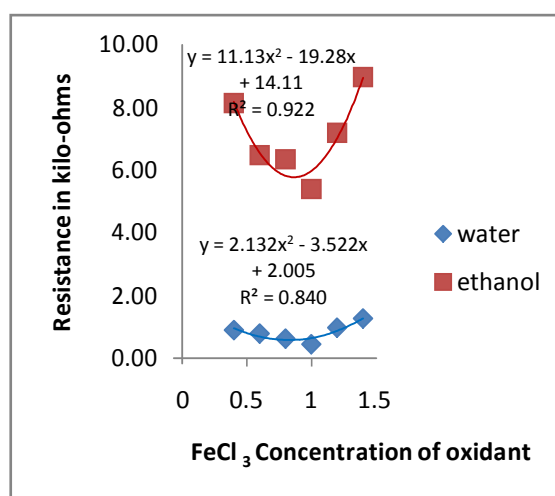


Fig 6: Effect of oxidant solvent and its concentration on electrical resistance of yarns

Effect of oxidant concentration on yarn's electrical resistance

Figs 6 shows how the concentration of oxidant and the type of solvent affect the electrical resistance of the yarn. In both cases, there is a drop in resistance as the FeCl_3 is increased. At 1 mole/litre both solvents show a point of saturation and the resistance is at its lowest. For the ethanol solvent there is greater variation (5.38 to 8.97 kilo ohms) in the 'electrical resistance' for different concentrations of FeCl_3 . These values show that the material lost their electrical resistance becoming more of conductors than an insulator. For a material to be an insulator its electrical resistance should more than 10^{-7} giga ohms (0.1 kilo ohm). The high value of the coefficient of determination R^2 (0.9228) means that the results are a true representation of the electrical resistance behaviour of coated yarns when ethanol was used an oxidant solvent. When water was used as an oxidant solvent, electrical resistance between of 0.88 and 1.24 kilo ohms was obtained, values lower than when ethanol was used. This indicates that the cotton yarn had become a better conductor than when ethanol was used. This could be attributed, to the fact that water being conductive itself also has an effect in aiding the cotton yarn to conduct electricity. Similarly to when ethanol was used, the high value of the coefficient of determination R^2 (0.8403) means that the results are a true representation of the electrical resistance behaviour of coated yarns when water was used an oxidant solvent. In both cases (ethanol and water) electrical resistance of the yarn decreased from 0 to 1 mole per litre concentration after which it started to increase again. This could be due to the fact that at 1 mole/litre a point of saturation was reached beyond which the FeCl_3 salts no longer had an effect on the yarns resistance. The other cause could be due to the fact that there was a possibility of over oxidation of the polymer which could result in irreversible loss of the electrochemical activity, hence reducing conductivity of the polymeric films. In this study it was noted that increasing concentration of the oxidizing agent improves the polymerisation rate and also the doping level of PPy increased the electrical conductivity of the PPy-cotton yarns [3].

Solution phase polymerisation (coating)

The solution coating method was carried out for 24 hours to allow the yarns to be coated as uniformly as possible. Because of the difference in areas exposed as the reactions were carried out in test tubes, complete uniform coating was not achieved. For instance rolled yarns would coat more on the outer regions than the inner regions. Three different concentrations of pyrrole used in water and ethanol solvents were 3%, 5% and 10%. Pyrrole monomer was completely dissolved in ethanol and there was no coating of the yarns observed. Water formed emulsions with pyrrole, and as a result the pyrrole was oxidized by FeCl_3 upon immersing of oxidant treated yarns into the water-pyrrole solution, as the solution changed colour from pale yellow to black indicating polymerisation of pyrrole.

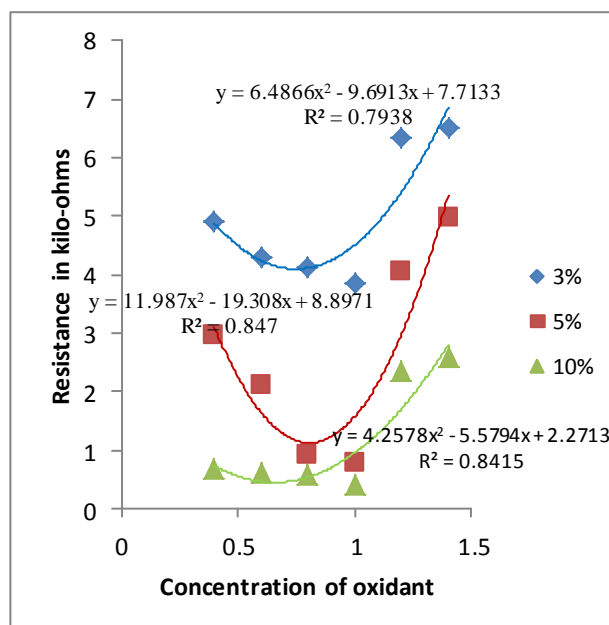


Fig 7: Resistance of yarns obtained at various oxidant concentrations and at three different pyrrole monomer concentrations.

The values of the coefficient of determination R^2 from Fig 7 are over 0.7, an indication that the results are a reasonable representation of the resistances obtained at varying concentrations. There were a lot of variations in the resistances of the solution coated yarns attributing to the low R^2 values. In addition to that, at 3% monomer concentration, there was minimal coating of the yarn with some grey patches observed. This is because at this concentration there were few polymeric molecules formed at the surface of the yarns. The value of R^2 , which is 0.79 is reasonably good although lower than other concentrations, and is as a result of non-uniform coating as compared to the other concentrations. Further observation, is that between 1 and 1.2 moles/litre, the sharp increase of resistance, as in the previous, is also a result of the resistance of the yarn being independent of salt content at high salt concentrations [13]. The electrical resistance value of between 0.395 kilo ohms and 2.59 kilo ohms at 10% of FeCl_3 concentration, between 0.9 kilo ohms and 4.99 kilo ohms at 5% FeCl_3 concentration, and between 4.133 kilo ohms and 6.52 kilo ohms at 3% monomer concentration qualifies the coated yarn as a conductor. Two competitive processes occur simultaneously: polymerisation in the yarn substrates and in the solution [20]. It was observed that the colour of the yarns changed from white to yellow as they were soaked in oxidant solutions. There was a gradual colour change from yellow to grey then finally black as the yarns were soaked in the pyrrole solution. This indicated an increase in polymer deposition as the concentration of the pyrrole increased. Low temperatures were maintained between 0 to 5°C, to lower the reaction rate of the polymerisation process. This was done so as to avoid generation of excess oligomers which could not be transported effectively into the fibrous sites of the yarn, a phenomenon which occurs due to higher rate of reaction. Polymer particles would form and accumulate in solution rather than in and on the yarns. At low reactant concentrations, there is no detectable conductivity, different threshold monomer concentrations have been obtained by different researchers [20].

In this study, 3% was taken as the threshold concentration. The surface resistance decreased with increase in monomer concentration from 3% to 5%. There was a further decrease at concentration of 10% as resistance values of 0.18 kilo-ohms close to those of the vapour coating technique were obtained. The percolation theory [20] describes the phenomenon of current conduction in composite materials. It allows the evaluation of the magnitude of conductivity in relation to content of conductive component related to the mass of the entire composite [20]. According to the percolation theory [20], creation of conducting paths resulting from direct contact between PPy molecules in the non-conductive cotton yarn results in significant drop of the yarn's resistivity. Increase of the monomer concentration means increase of conduction paths due to increase of PPy molecules within and on the yarn surface, therefore further reduction of resistivity. This explains the further reduced resistance of yarns at 10% concentration of monomer. There has to be a maintained optimum ratio between the doping, oxidizing and monomer reagents to ensure creation of conduction paths and this varies depending on the reagents used. FeCl₃ has been found to be a good chemical oxidant and water is the best solvent for chemical polymerisation with respect to desirable conductivity characteristics.

IV. CONCLUSION AND RECOMMENDATIONS

Conclusion

Electrically conductive cotton yarns were successfully developed as evidenced by the reduction in electrical resistance from the giga-ohms range of uncoated yarns to the kilo-ohms range of PPy coated yarns and the achievement of conductivity in the range of 10⁰S/cm. However, these yarns cannot effectively be used for powering as the resistance is still too high for that application. These yarns can be used in sensors particularly, piezo resistive sensors as variable resistors, electromagnetic interference shielding and can act as variable resistors due to the fact that when stretched, resistance of yarns changes. It was observed that physical properties of yarns such as hydrophilicity, surface morphology, shape factor, yarn twist and linear density influence the thickness of the coating, penetration of the PPy into the yarn interior and continuity of the coating along the yarn axis hence conductivity obtained. These reasons also account for lack of uniformity in the resistance values obtained along the yarns. The cost of producing conductive yarns is initially high since 100ml of pyrrole cost about USD \$130. However, from this little amount, several coatings can be achieved using both the vapour and solution methods. In this study, several coatings were carried out with only 50ml of pyrrole for all the experiments (10 per sample of each FeCl₃ concentration). The vapour method is however more economical than the solution as the unreacted pyrrole is obtained in its undiluted state.

Recommendations

More conductive yarns, than these obtained in this study can be developed from combining polymer coating and incorporation of carbon nanotubes into the yarns. Although this work was carried out in a two-step process for vapour phase polymerisation, there is risk of inhalation of toxic pyrrole vapours. As a result, it is recommended that for large scale production of conductive yarns, a continuous process be used. This process eliminates the need to manually insert the pre-treated yarns into the polymerisation column as was done for this study. The yarns can be drawn in through reed-like equipment into the polymerisation chamber. The polymerisation chamber has many vapour inlets so as to promote uniform coating of the yarns. There has to be an automated system to ensure let off and take up of yarns from the polymerisation chamber. The durability, of the conductive yarn to repeated laundering remains a question without a definite answer. However, it is recommended that, for a yarn which is used in applications where there is need for repeated laundering and abrasion, solution coating can be the best method to use as there is better penetration of PPy molecules into the fibre matrices of the yarn.

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