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Preparation of Conductive Polyethylene Terephthalate Yarns by Deposition of Silver & Copper Nanoparticles

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Abstract

The assemblage of textiles and electronics in a single structure has led to the development of smart textiles for functional purposes and special products. Conductive yarn as a necessary component of smart textiles is being developed by a number of techniques. The objective of the current study was to impart conductivity to yarn by coating the silver and copper nanoparticles on the surface of multifilament polyester textile fibres. The surface morphology and electrical conductivity of the coated yarns were investigated. The wash ability of the conductive yarns developed was also studied. The yarns showed good retention of the nanoparticles, as proven by the very small loss of the conductivity of the material.

Key words: coatings, nanoparticles, technical yarn, smart textiles.

non-flexible electronic products. They consolidate a high level of intelligence and can be divided into three subgroups: active, passive and intelligent smart textiles [2]. Passive smart textiles are able to sense the environment based on sensors, while active smart textiles sense stimuli by integrating an actuator and sensing device. Intelligent smart textiles are able to sense, react and adapt their behaviour according to given circumstances.

The key component of smart textiles is conductive yarn. Conductive yarns are widely used for the development of sensors like piezo electrics and fibre optics because of cost-effectiveness, durability and ease of measurement [3, 4]. The recent trend in smart textiles is to seamlessly merge wearable computers into ordinary clothing, as the tactile properties of wearing materials are important for people. It would be uncomfortable to have wires and hard plastic cases against their bodies. Therefore electronic circuits are built entirely out of textiles to distribute data and power, as well as to perform touch sensing, keeping in mind the comfort of the wearer [5, 6]. Fabric-based sensing has been a large field of research in the biomedical and safety communities. Fabric sensors can be used for electrocardiogram (ECG) [2], electromyography (EMG), and electroencephalography (EEG) sensing [7]. Conductive yarns are also used in interactive club apparel (responding against the beat of music), garments with functional sleeves (to type pager messages, dial phone numbers and play music), apparel with integrated communication devices [8], functional textiles for home and offices to control lighting, temperature, etc. [9, 10]. Such

sensors are also used to detect arm action for improving golf or tennis swings [11].

The two general categories of conductive yarns include naturally conductive fibres and those specially treated to impart conductivity [12]. Naturally conductive yarns are spun directly from conductive materials including metals [13, 14]. Electrically conductive metals such as stainless steel, ferrous alloys, nickel, aluminum, copper, titanium, etc. are used for this purpose. Silk organza is a well-known highly conductive yarn with thin copper foil wrapped over silk fibre [5]. The other category of conductive yarns is produced by special treatments. Thermoplastic polymers (PE or PS) with conductive fillers are spun to produce conductive yarns [15], which can be accomplished either by metal coating, electrolysis deposition (ELD) of metal particles [14, 16] on the surface of yarns or by using the wet spinning process [17, 18]. Conductive polymeric materials are coated by in situ solution or vapour phase polymerisation to obtain conducting yarns [19]. PEDOT has received considerable attention for a vast range of application areas [20] such as, EMI shielding [21], heat generation [22], light emitting diodes and chemical sensors [23]. Other techniques include the physical padding of electrolyte solution on yarn [24] and printing by lithography [25]. Nano technology is the current state of the art field of study in textiles in which different sorts of nano particles are applied on textile materials to obtain specific properties [26, 27].

However, no study on the mechanical durability or wash ability of modified yarns has been made. The conductivity

Introduction

Smart textiles are related to a broad field of studies and products that induce the functionality and usefulness of common fabrics in specialised products. The assembly of textiles and electronics in a single structure, called e-textiles, is significant for the development of smart materials [1]. These materials have a tendency of accomplishing a wide spectrum of functions (sense, reactio, signal and power transmitting etc.), found in rigid and

Table 1. List of samples produced and their electrical conductivity results.

Sr. No.	Sample ID	Nanoparticles	Number of dips	Electrical conductivity, Siemens/cm	
				Before washing	After washing
1	P1	Ag	50	1.94×10^{-5}	1.84×10^{-5}
2	P2	Ag	100	4.80×10^{-4}	4.04×10^{-4}
3	P3	Ag	150	1.10×10^{-3}	0.96×10^{-3}
4	P4	Cu	50	3.24×10^{-5}	3.16×10^{-5}
5	P5	Cu	100	2.84×10^{-3}	2.65×10^{-3}
6	P6	Cu	150	3.58×10^{-3}	3.01×10^{-3}

of conducting polymers depends greatly on doping. This doping level changes dramatically by washing in water or detergent, leading to a rapid drop in conductivity. Besides the wash ability of this type of conductive yarns is poor. Hence their application in some fields such as conductive circuits is also limited. To date, how to effectively modify a cotton surface with controllable conductivity and high durability remains a great challenge [16]. A key issue in the fabrication of functional nanostructures is the 'exact' location of nanometer-sized objects [28]. A number of techniques have been used for making conductive yarns. However, to the best of our knowledge, no study has introduced the coating of silver and copper NPs on multifilament polyester textile yarns. Attempts were made in the present study to investigate the different physical and chemical properties of silver and copper coated yarns. The wash ability of the conductive yarns developed was also investigated in this study.

■ Experimental

Multifilament polyester yarn (ICI Private Limited, Pakistan) with 48 filaments and a linear density of 16.65 tex was used as a substrate to produce conductive yarns. Two different types of nanoparticles (Ag and Cu) were deposited on these. The synthesized nanoparticles had a size range of 10-15 nm, which was determined using a Malvern Zetasizer (Malvern Instruments Ltd., United Kingdom), nano series based on the dynamic light scattering principle of the brownian motion of particles. The chemicals used for the synthesis and deposition of nanoparticles had 99.99% purity and were obtained from Merck Co. Ltd., Pakistan.

The multifilament polyester yarn was treated with 10 wt% aqueous NaOH solution at room temperature for 10 min and rinsed with distilled water. The deposition of nanoparticles of silver was

carried out according to the reaction mechanism described by Chao-Hua Xue et al [29] with little modifications. For instance, aqua ammonia (28 wt%) was added drop-wise into a 0.3M aqueous solution of AgNO_3 , and stirred continuously until a transparent solution of $[\text{Ag}(\text{NH}_3)_2]^+$ was obtained. Alkali treated polyester yarn was dipped in this solution for 3 minutes and dried at 100 °C for 3 minutes. The dip and dry process was repeated a number of times to deposit the maximum concentration of $[\text{Ag}(\text{NH}_3)_2]^+$ on the yarn. The resulting yarn was transferred into 0.1M glucose stock solution, and the remaining $[\text{Ag}(\text{NH}_3)_2]^+$ solution was also poured into glucose solution to maximise the deposition of silver on the yarn. The reaction was allowed to proceed for 15 min. Finally the yarn was rinsed with water and dried in air. Three different samples (P1-P3) were prepared by varying the number of dips as shown in **Table 1**, with same concentration of AgNO_3 , glucose and aqueous NaOH solution.

Likewise, copper sulphate (CuSO_4) was used as the base material to synthesize Cu nanoparticles along with sodium hydrosulphite. The same pre-treatment technique was adopted to prepare the substrate for the deposition of Cu nanoparticles by reducing copper (II) according to the mechanism described by Yi-Hsuan Chou [30]. The alkali treated yarn was dipped and dried several times in the copper sulphate (CuSO_4) solution at 100 °C, with a time interval of 3 minutes. The copper sulphate (CuSO_4) treated substrate was then transferred to the sodium hydrosulphite solution for 20 minutes. We did not dip the yarn in a hot solution of copper sulphate, the actually solution being at room temperature. The sample after reduction was dried in an oven for 3 minutes at 100 °C. Three different conductive yarn samples (P4-P6) were prepared by the deposition of Cu nanoparticles as given in **Table 1**. The concentration of the copper sulphate

(CuSO_4), reducing agent (glucose) and aqueous solution of NaOH was constant for all samples.

As it was an initial study, we focused on the conductivity of nanoparticle coated yarns and their stability, where time is indeed a crucial factor. To overcome this constraint, the recipe could be optimised in further research.

We used a much less amount of copper sulphate salt in the solution with constant stirring, which meant we had very fine particles mixed homogeneously in the solution. As particles are very fine, they will cover more surface area over the yarn, but due to less concentration of copper sulphate in the solution, it is impossible to coat a maximum amount of particles in one step. Therefore we increased the number of dips to cover more and more surface area of the yarn. Again this is the reason that we adopted drying – dipping – drying – reduction, because we gain a maximum surface area covered evenly with fine particles. On the other hand many procedures are available to prepare the particles and then attach them over the surface, but there are some problems concerned with them such as the accumulation of particles before applying in the solution and an uneven distribution of particles over the surface.

In samples P1-P6 developed, organic-inorganic binder, supplied by CHT Bezema, Pakistan, was applied (15 g/l), which is specifically used for the binding of metal particles on textiles. The purpose of the binding agent was to fix the nanoparticles on the surface of yarn and to improve the adhesion of nanoparticles on the surface of polyester yarn. The samples were cured at a temperature of 100 °C.

Characterisation

Characterisation of these conductive yarns was performed at a number of levels, including the characterisation of nanoparticles as well as investigation of the conductivity of the yarns developed and their durability.

Surface characterisation of conductive yarn

Scanning electron microscopy (SEM-Quanta FEI 250, Quanta, USA) of the conductive yarn samples was done to observe the morphology of Ag and Cu nanoparticles deposited on the surface of polyester yarn.

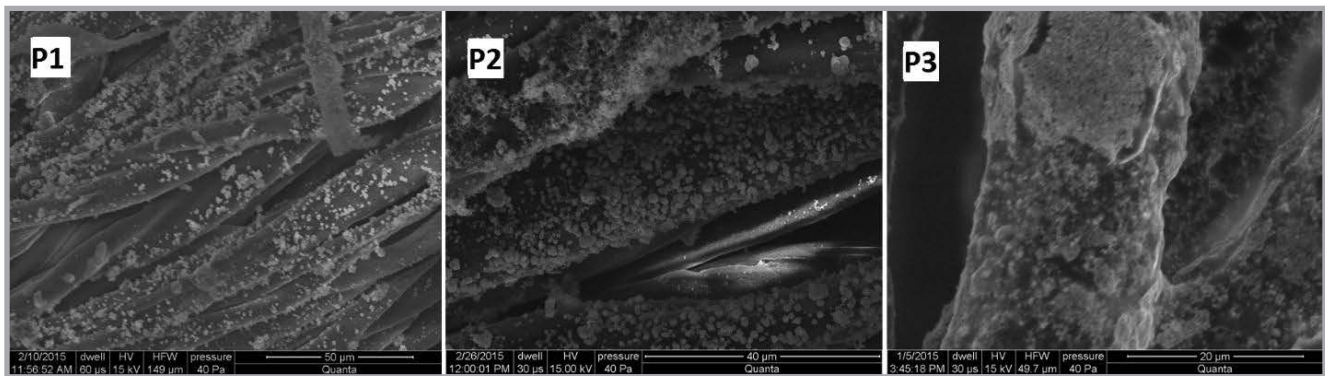


Figure 1. SEM images of Ag nanoparticle coated conductive yarns.

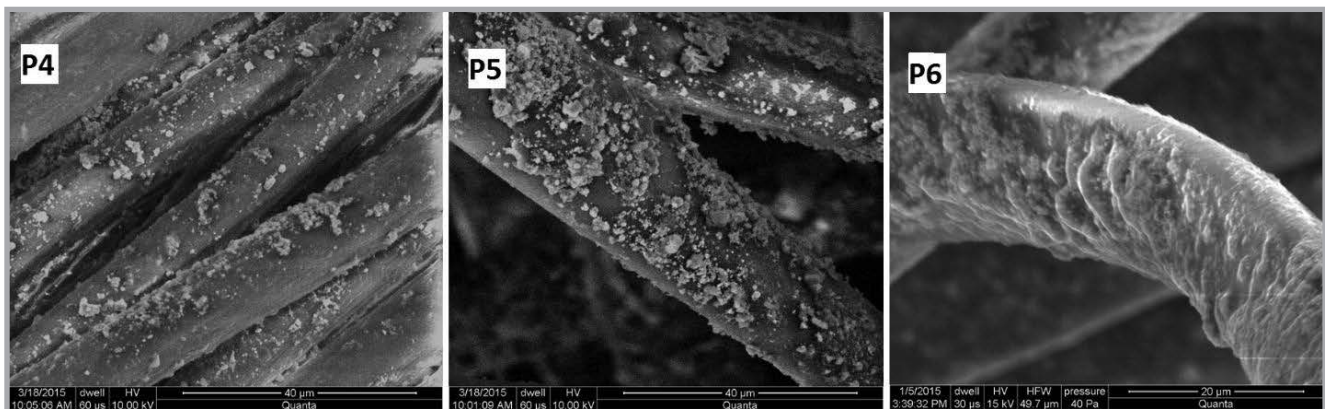


Figure 2. SEM images of Cu nanoparticle coated conductive yarns.

Conductivity of yarn

After making sure of the deposition of nanoparticles on the surface of yarn by SEM, the conductivity of these yarns was determined. A digital multi-meter – Fluke 17B (FLUKE TECHNOLOGIES PVT. LTD., India) was used to measure the conductivity of these yarns. The DMM used to determine the conductivity was Fluke (17B). The Coated yarn was clipped between two crocodile connectors connected to the DMM. The connectors were one centimeter apart. The electrical resistance of the yarn(s) was noted down. After that the conductance was determined using the expression (Conductivity = 1/Resistance). The testing is performed at standard atmospheric conditions ASTM-1776.

Durability of conductivity

The washing fastness of these conductive yarns was observed in order to have an idea of their activity in service. The washing fastness was evaluated according to ISO 105-C01. A 5 g/l standard detergent was used at a liquor ratio of 50:1. The sample was rinsed for 30 minutes at 40 °C in the solution and then dried.

Results and discussion

The morphology of the silver and copper nanoparticles deposited on the surface of yarn was investigated using scanning electron microscopy (SEM). The SEM images confirm the deposition of nanoparticles on the surface of yarns. Meanwhile it can also be observed that the deposition of nanoparticles increases with an increase in the number of dips of the yarn in the solution, because sample P2 was produced by 100 dips, which almost perfectly covered it with nanoparticles, while sample P3 was produced by 150 dips, which totally covered it with nanoparticles, as can be observed from the SEM images (**Figure 1**). The loading of nanoparticles for this sample was estimated to be 6 percent by weight of fabric by weighing the yarn before and after deposition. **Figure 2** shows SEM images of Cu coated polyester conductive yarns. The results are almost similar to those of Ag coated yarns, where the covering nanoparticles increase with an increase in the number of dips. The highest loading of nanoparticles was also estimated to be 6 percent by weight of yarn.

Virgin polyester yarn is well-known to be an electrically insulating material [31]. In contrast, the Ag and Cu coated polyester yarns developed were supposed to be electrically conductive. The conductivity of the yarns was also expected to increase with increasing deposition of the nanoparticles. This hypothesis was confirmed by the electrical conductivity tests performed on the conductive yarn samples developed, shown in **Table 1**.

The three polyester conductive yarns produced by deposition of Ag nanoparticles (P1, P2 and P3) showed conductivity values of 1.94×10^{-5} , 4.80×10^{-4} and 1.09×10^{-3} Siemens/cm, respectively. The resulting trend justified our hypothesis that increasing the number of dips in the solution results in the deposition of a higher amount of nanoparticles on the surface of the yarn, and hence conductivity is increased. This significant difference in the conductivity of P1, P2 and P3 can be attributed to the deposition of a higher amount of nanoparticles on P3 yarn as compared to P1 and P2.

In the case of Cu NP coated conductive yarn, it is clear from the **Table 1** that the

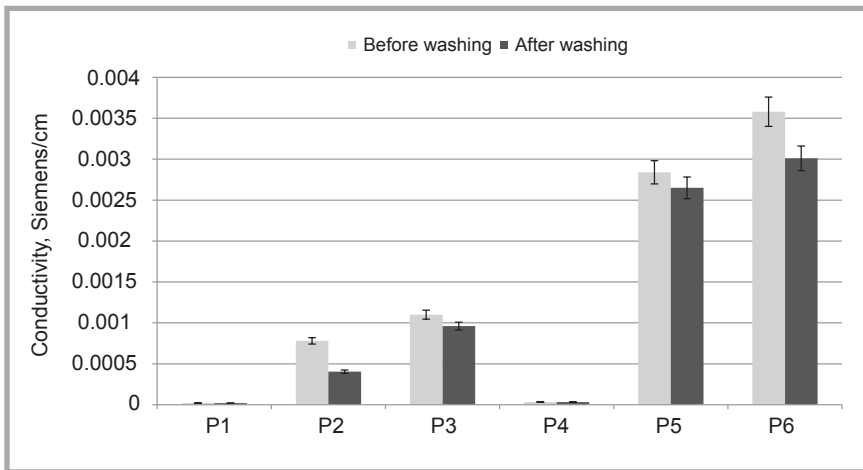


Figure 3. Electrical conductivity before and after washing.

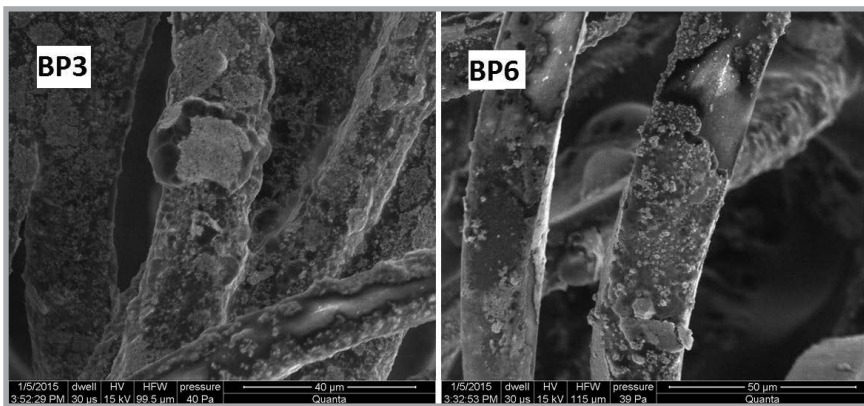


Figure 4. SEM images of conductive yarns after washing.

conductivity of sample P5 is higher than P4. The reason is that P5 was applied with a greater number of dips in the solution, meaning more nanoparticles were accumulated on the surface, which increased the conductivity. It also verifies that the conductivity increases with an increase in the number of dips. Moreover the conductivity of yarn P6 with 150 dips and P5 with 100 dips was found almost comparable. It means by increasing the number of dips from 100 to 150, the increase in conductivity is not that significant as compared to the increase observed between 50 and 100 dips. Therefore it can be concluded that 100 dips give optimum conductivity to the yarn, which is sufficient for getting the desired result while maintaining the economy.

The overall conductivity values explained the general trend of electrical conduction, which is higher for Cu as compared with Ag loaded yarn. The as-developed conductive yarns have the capability to be used in electrical application. A small LED was lit up using

the Ag and Cu nanoparticle coated conductive yarns.

Stability after washing

The key feature herein is that Cu/Ag nanoparticle coated yarns offer unique electrical stability under multiple bending and stretching. There was no standard testing performed to determine the bending and stretching. It is based on observation during electrical conductivity measurement. One critical challenge to these conductive yarns is their durability under washing and rubbing. To investigate these properties, the electrical conductivity of these yarns was investigated after washing. A standard washing test of these six samples was performed according to method ISO 105-C01. The conductivity was again checked for the washed samples and reported in *Table 1*.

It can be observed from *Figure 3*, that there is no significant decrease in the conductivity of the binder applied yarns before and after washing. It is evident that there is only a slight change in the

conductivity of these samples, even after severe washing. Likewise P4, P5 and P6 were the polyester yarn samples loaded with Cu nanoparticles, and showed only a slight decrease in electrical conductivity after washing. It means that the binder worked efficiently to keep the nanoparticles attached to the surface of the yarn and fix them firmly without losing any conductivity. This was also verified by SEM analysis before and after washing of the conductive yarn samples, as shown in *Figure 4*. A very small portion of the coating layer was delaminated, as shown in *Figure 4*. The results obtained for electrical conductivity showed the insignificant effect of this delamination caused by severe rubbing during the washing process. Therefore we conclude that our conductive yarn can withstand washing effects and can be a good alternative for current conductive yarns in the market.

Conclusions

Six different polyester conductive yarn samples were developed using two different nanoparticle types along with varying the number of dips. The successful application of Ag and Cu nanoparticles highlighted their potential application in the field of smart textiles. In addition, it was concluded that nanoparticle take-up can be improved by increasing the number of dips for better covering of the yarn surface. The application of a binder enhances the service life of the product with no significant change in the conductivity of the yarn after severe wash cycles.

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Declaration

The authors declare that there is no conflict of interest.

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