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Surface Deposition of Metals on Textile Structures

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ABSTRACT

The present work deals with the development of electrically conductive, EMI shielded, multifunctional fabrics. At first, the conductive textiles were developed by in-situ deposition of copper particles. The scanning electron microscope, and X-ray diffraction techniques were employed to study the morphology of deposited copper particles. The utility of conductive fabrics was analysed for electromagnetic shielding effectivity over frequency range of 30 MHz to 1.5 GHz. The electromagnetic interference shielding was found to increase with increase in number of dips, which was attributed to increased reflection of EM waves due to dense, uniform, and percolated network of conductive copper particles on the surface. Furthermore, the role of deposited copper particles on antibacterial properties was examined against pathogenic bacteria such as Staphylococcus aureus and Escherichia coli. At the end, the durability of fabrics was examined against washing. The fabrics showed good retention of the copper particles, proved by scanning electron microscopic microstructures and small loss in the conductivity of the material after washing.

The objective of second part was to develop multifunctional plain-woven conductive fabrics with acceptable electrical conductivity by in-situ deposition of silver particles. The effect of silver nitrate concentration and number of dips was investigated for change in electrical conductivity, EMI shielding, and antimicrobial properties of coated fabrics. SEM analysis were employed to study the morphology of deposited silver particles. The EMI shielding was found to increase with increase in concentration of silver particles. Furthermore, silver particles were also deposited on stretchable knitted fabrics by adopting the same method of in-situ deposition. The purpose of this study deals with the development of stretchable conductive fabrics for possible applications in electrotherapy. The performance of silver-coated fabrics was evaluated based on number of properties such as electrical conductivity at normal and stretching state of fabrics, antibacterial, and durability. Furthermore, the conductive fabrics were subjected to various repeated extensions and change in electrical resistivity was examined to simulate the performance of electrodes under various movements of human body. With increase in extension till 80%, very small change in electrical resistivity was observed and after 90% extension, the electrical resistivity was found to increase significantly. The resistivity was found to remain constant for repeated extensions of over 100 cycles and also there was insignificant change in electrical resistivity when constant current was applied over prolonged time. The utility of silver-coated fabrics can be expected as flexible textile electrodes in transcutaneous electrical nerve stimulation electrotherapy applications.

The third part of study, proposed a simple way of surface metallization of cotton fabrics by electroless plating using a shorter route than is conventional. The fabric surface was activated by deposition of silver and copper nanoparticles, and then a thin layer of copper was coated using electroless plating. The performance of coated fabrics was compared in terms of electrical conductivity, electromagnetic interference (EMI) shielding, Joule heating and antibacterial properties. The samples prepared by electroless plating of copper showed greater performance for the fabric first deposited with silver particles than those first deposited with copper particles. Samples of copper electroless plating over silver had surface resistivity of 20 Ω, EMI shielding of 75.53 dB and Joule heating of 119 °C by using DC input varied from 5–10 V and at constant current 1 A (the experiment was carried out up to 10 watt). Moreover, samples with modified electroless plating showed better attachment of the metal layer and therefore longer durability

The objective of fourth part was to make electrically conductive multifunctional fabrics and their further use as electrodes for the development of triboelectric generator (TrEG). The conductive fabrics were made by the coating of thin copper layer and then electroplating of silver layer. The surface structure, electrical conductivity and antibacterial properties of coated fabrics were examined to know their multifunctional properties. Later, the energy harvesting performance of conductive fabric electrodes was studied by combining with oppositely charged triboelectric materials such as silicon rubber and rabbit fur. The fabricated TrEG was found to produce 21 V and 3.5 μA current under the stretching action whereas 33 V and 6 μA current under the pressing action. Furthermore, its energy harvesting performance was investigated under the mechanical actions of human body, it generated about 10 V from elbow movements and about 40 V from foot movements.

Keywords: Conductive textiles, copper particles, silver particles, stretchable conductive fabrics, surface metallization, electromagnetic interference shielding, antibacterial properties, smart textiles, TENs electrodes, electrotherapy, electroless plating, metal coatings, sensors and actuators, copper plating, energy harvesting, triboelectric generator

ABSTRAKT

Tato disertační práce se zabývá vývojem elektricky vodivých, multifunkčních textilií chránících proti elektromagnetickému záření v širokém rozsahu frekvencí (EMI). V první části práce byly připraveny vodivé textilie pomocí in-situ povrchové depozice částic mědi. Pro studium morfologie nanesených částic mědi byl použit skenovací elektronový mikroskop a rentgenové difrakční techniky. Funkčnost vodivých tkanin byla analyzována hodnocením účinnosti elektromagnetického stínění v kmitočtovém rozsahu 30 MHz až 1,5 GHz. Bylo zjištěno, že stínění elektromagnetického rušení se zvyšuje se zvyšujícím se počtem dílčích nánosů, což přímo souvisí se zvýšeným odrazem elektromagnetického záření jako důsledek tvorby husté (za perkolačním prahem) rovnoměrnější povrchové vrstvy vodivých částic mědi. Dále byla zkoumán vliv povrchové vrstvy částic mědi na antibakteriální odolnost proti patogenním bakteriím, jako je Staphylococcus aureus a Escherichia coli. Textilie s povrchovou vrstvou částic mědi vykazovaly dobrou odolnost v praní, což bylo prokázáno jak mikroskopickým zkoumáním mikrostruktury, tak malou ztrátou elektrické vodivosti po praní.
Cílem druhé části disertační práce bylo vyvinout multifunkční tkané vodivé textilie v plátnové vazbě s přijatelnou elektrickou vodivostí pomocí in-situ nanášení částic stříbra. Byl zkoumán vliv koncentrace soli stříbra a počtu dílčích nánosů na změnu elektrické vodivosti, elektromagnetického stínění (EMI) a antimikrobiálních vlastností těchto multifunkčních tkanin. Pro studium morfologie uložených částic stříbra byla použita SEM analýza. Bylo zjištěno, že elektromagnetické stínění se zvyšuje se zvyšováním obsahu částic stříbra.
Stříbrné částice byly také nanášeny na elastické pleteniny pomocí stejného způsobu in-situ nanášení. Cílem byla příprava elastických (vratně deformovatelných) vodivých pletenin pro možné aplikace v elektroterapii. Výhodnost těchto pletenin byla hodnocena na základě řady vlastností, jako je elektrická vodivost v normálním a deformovaném stavu, antibakteriální schopnosti a trvanlivost. Do 80% ní tahové deformace byla pozorována velmi malá změna elektrické vodivosti. Po 90% ní tahové deformaci bylo zjištěno, že se elektrický odpor významně zvyšuje. Bylo simulováno chování vodivých pletenin (elektrod) při různých pohybech lidského těla pomocí změn elektrického odporu při opakovaných cyklech prodlužování a odlehčování. Bylo zjištěno, že elektrický odpor zůstává konstantní při více než 100 cyklech prodlužování a odlehčování. Dlouhodobé působení konstantního elektrického proudu nevýznamně změnilo elektrický odpor elastických vodivých pletenin. Tyto elastické vodivé pleteniny textilií bude možno použít jako flexibilní textilní elektrody v elektroterapii transkutánní elektrické stimulace nervů.
Třetí část disertační práce je zaměřena na návrh jednoduchého způsobu povrchového ukládání částic kovů na bavlněné tkaniny bez použití elektrického pole zkráceným. Povrch tkaniny byl aktivován nanesením nanočástic stříbra a mědi a následně byla vytvořena tenká vrstva částic mědi bez elektrickým pokovováním. Funkčnost povrchově pokovených tkanin byla hodnocena pomocí elektrické vodivosti, elektromagnetického stínění, teploty odporového ohřevu a bakteriální odolnosti. Textilie, kde byla provedena aktivace nanesením částic stříbra s následným bezproudovým pokovováním mědí, vykazovaly vyšší účinnost ve srovnání s textiliemi, kde byla provedena aktivace nanesením částic mědi s následným bezproudovým pokovováním mědí. Textilie aktivované nanesením částic stříbra s elektrolytickým pokovením mědi měly povrchový odpor 20 Ω, elektromagnetického stínění 75,53 dB a teplotu 119 ° C při ohřevu pomocí stejnosměrného napětí 10 V při konstantním proudu 1 A (10 wattů). Kromě toho tyto textilie vykazovaly delší trvanlivost efektů.
Cílem čtvrté části disertační práce bylo připravit elektricky vodivé multifunkční textilie využitelné jako elektrody pro triboelektrický generátor (TrEG). Vodivé textilie byly vyrobeny depozicí částic mědi a následnou galvanizací stříbrem. Pro posouzení multifunkčních vlastností byla zkoumána struktura povrchu, elektrická vodivost a antibakteriální vlastnosti. Byla

studována účinnost sběru energie vodivých textilních elektrod kombinací s opačně nabitými triboelektrickými materiály, jako je silikonový kaučuk a králičí kožešina. Bylo zjištěno, že takto připravený TrEG produkuje napětí 21 V a proud 3,5 μA při protažení. Při stlačení je produkováno napětí 33 V a proud 6 μA. Dále byla zkoumána energetická účinnost tohoto triboelektrického generátoru při mechanickém působení lidského těla. V důsledku pohybů loktů se generuje napětí asi 10 V a v důsledku pohybů nohou napětí asi 40 V.

Klíčová slova: Vodivé textilie, částice mědi, stříbra částice, elastomerní vodivé textilie, povrchová metalizace, stínění elektromagnetického rušení, antibakteriální vlastnosti, inteligentní textilie, elektrody TEN, elektroterapie, elektrolytické pokovování, kovové povlaky, senzory a akční členy, pokovování mědi, sběr energie, triboelektrický generátor.

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"Seek knowledge from the cradle to the grave"

Hazrat Muhammad P.B.U.H

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LIST OF ABBREVIATIONS

Acronyms	Description
ASTM	American Society for Testing and Materials
CPC	Courses per centimetre
DC	Direct Current
ECG	Electrocardiography
E. coli	Escherichia Coli
EDTA	Ethylenediaminetetraacetic Acid
EDX	Energy Dispersive X-ray Spectroscopy
EMG	Electromyography
EMI	Electromagnetic Interference
EPI	Ends per Inch
ICP-AES	Inductive Couple Plasma-Atomic Emission Spectroscopy
MRSA	Methicillin-resistant Staphyllococcus aureus
NPs	Nanoparticles
PAN	Poly(acrylonitrile)
PET	poly(ethylene terephthalate)
PPI	Picks per Inch
SEM	Scanning Electron Microscopy
TENS	Transcutaneous Electrical Nerve Stimulation
TrEG	Triboelectric Generator
WPC	Wales per centimetre
XRD	X-ray Diffractometry

LIST OF SYMBOLS

ρV	Volume Resistivity
ρs	Surface Resistivity
Rs	Surface Resistance
RV	Volume Resistance

1 INTRODUCTION

In recent years, research on functional textiles gained significant importance due to their utilization in different advanced materials [1]. A variety of functional textiles e.g. solar textiles (reflecting or absorbing), color-changing textiles, shape-memory textiles, waterproof and moisture permeable textiles, anti-bacterial textiles, UV protected textiles and electrically conductive textiles have been developed [1,2]. Among them, the electrically conductive textiles are becoming most dynamic and fast-growing sectors due to their novel applications in flexible and wearable electronics, sensors and actuators, electromagnetic interference (EMI) shielding, heat generators, etc. [3]. There are various approaches to render the textiles with electrical conductivity such as fabrics made by naturally conductive yarns and those specially treated to impart conductivity. The naturally conductive yarns can be spun directly from electrically conductive materials like metals [4, 5]. While special treatments like chemical coating, surface metallization (Cu, Al, Ni, Ag, etc.), deposition of conductive fillers (carbon black, carbon nanotubes, etc.), coating of conductive polymers (polyaniline, polypyrrole, polytiophene, etc.) can be attempted to impart electrical conductivity on textile surfaces [5, 6]. However, the conductive textiles made by blending of metal wires during yarn or fabric formation lead to deterioration of their comfort properties and were easily affected by washing and abrasion [7]. They cannot be widely used for personal protective clothing because of their prickliness, heavy weight, cost inefficiency, poor flexibility, and poor scratch resistance. The fabrics made by a coating of conductive polymers, inks, and chemical doping were found not durable to washing and were susceptible to cracking and rubbing [8-10]. Therefore, the textile surface metallization was investigated in detail in this research work as unique method to provide multifunctional properties such as electrical conductivity, EMI shielding, ohmic heating and anti-bacterial properties [10].

2 THESIS SIGNIFICANCE, SCOPE AND OBJECTIVES

The main aim of thesis is to investigate preparation properties and selected applications of multifunctional textiles having required electrical conductivity, EMI shielding, ohmic heating capability, and anti-bacterial properties by different surface metallization methods. To develop the conductive fabrics for novel applications (electrotherapy, energy harvesting) a suitable amount of deposited metal is required. This thesis is focused on the metallization of two different kinds of fabrics i.e. 100 percent cotton woven fabric and knitted stretchable fabric composed up of nylon, spandex and cotton.

At first in-situ deposition of copper particles was done on plain woven cotton fabric and studied their electrical and functional properties.

In second step silver particles were deposited on cotton fabric and knitted fabric. The purpose of deposition the silver particles over knitted fabric was to find the suitable application in the field of electrotherapy for TENs machine. Carbon rubber electrodes with gel or metal plates covered with nonconductive dry (when is wet it is conductive) fabric have been used in transcutaneous electrical nerve stimulation (TENS) applications [11]. However, in the case of general electrotherapy there are a lot of problems concerned with the surface electrode placed on the skin or needle electrode inserted into the living body [12]. Despite of these facts these type of electrodes are still in use for TENS application. In this thesis, I have selected special substrate (composed of cotton, nylon and spandex) and report a special technique for the deposition of silver nanoparticles onto fibres and within fabric structures. Furthermore, the developed conductive electrodes are quite flexible and stretchable, so fulfil the demand of comfort. Changes in electrical conductivity with repeated extension were investigated to improve the properties of fabric based conductive electrode materials to be adaptable for human body movement during electrotherapy treatment. Moreover, electrodes also provide good washing fastness and avoid from cracks regarding stretching, have good drape, softness and hand feel. Fabrics are developed by direct growing very fine conductive nanoparticles of silver on it. Electrodes have markedly reduced the incidence of contact dermatitis, and can also be easily applied over a wounded or injured skin (cuts, scrapes, scratches, and punctured skin) because developed fabrics have antibacterial and hygienic properties due to silver nanoparticles.

Third step was to perform the electroless plating on previously copper and silver particles coated woven fabrics. Generally, electro less plating is performed after a number of

steps (1) pre-treatment, (2) sensitization of the surface, (3) activation of the surface (4) copper plating. The aim was to eliminate the sensitization step by coating of copper or silver particles over the surface of fabric before electroless plating of any metals. The surface activation by coating of silver or copper particles was expected to provide more uniform and stable base for further electroless plating. This further resulted into more even deposition of metals during electroless plating and thus fewer variations in electrical conductivity (surface and volume) across the substrate. Compared to conventional electroless plating, this work was focused on environmental friendly chemicals free from palladium, stannous and formaldehyde [16-19].

In fourth step, the cost effective and complete procedure to develop TrEG (Triboelectric generators) is described. we performed the silver plating on stretchable knitted fabrics composed up of nylon, spandex and cotton. Then the suitable application of plated fabrics in the field of energy harvesting was find. The energy harvesting device (Triboelectric generators) was developed by using the silver-plated fabrics. This soft, stretchable and fully flexible TrEG is totally different from previously developed TrEG by researchers[15-19]. The silicon rubber and rabbit fur as a dielectric media to develop charge were here proposed. After making the TrEG, it was tested against energy generation performance in different ways. The voltages and current generation was measured under different stretching and pressing conditions. So, end results were feasible regarding energy harvesting from mechanical motion of human body also.

The research activities were divided into following four parts.

2.1 Deposition of copper particles on cotton

- In-situ deposition of copper particles on cotton woven fabric by sequential dipping in copper sulphate and then sodium hydrosulphite solutions.
- Characterization of morphology of coated fabrics by EDX, scanning electron microscope (SEM), and X-ray diffraction (XRD) techniques.
- Evaluation of electrical conductivity, EMI shielding and antibacterial properties of coated fabrics.
- Examination of durability of performance against washing and oxidation over prolonged storage.

2.2 Deposition of silver particles on textiles

- In-situ deposition of silver particles on cotton woven fabric by sequential dipping in silver nitrate salt solution and then glucose stock solutions and study its functional properties.
- In-situ deposition of silver particles on stretchable knitted fabric nylon/cotton/spandex by sequential dipping in silver nitrate salt solution and then glucose stock solutions.
- Modify the structure of stretchable knitted fabric nylon/cotton/spandex to attach more particles.
- Study of utility of silver-coated knitted fabric for applications in electrotherapy.
- Examination of change in electrical resistivity to simulate the performance of electrodes under various movements of human body.

2.3 Electroless plating of copper over previously coated fabrics

- Developing of short route electroless plating for metal deposition on textiles.
- Elimination of the activation steps used in conventional electro less plating by prior deposition of copper or silver particles.
- Evaluating the ohmic heating at different voltages and time intervals.
- Study the effect of weight gain percentage of metal contents against electrical conductivity.

2.4 Plating of silver on stretchable knitted fabric

- Develop the conductive fabric based fully comfortable stretchable electrodes.
- Fabrication of triboelectric generator by using silicon rubber and rabbit fur in combination with plated conductive fabric electrodes.
- Study of the energy harvesting performance of triboelectric generator under the mechanical stretching and pressing actions of human body movements (i.e. elbow and foot).

3 LITERATURE REVIEW

Textiles are metallized for different purposes including electrical conductivity, electromagnetic shielding, antibacterial properties, UV and radar reflectivity, better antistatic properties, insulation, anti-stabbing, for decorative and shiny metallic appearance. In this chapter, literature is reviewed for electrically conductive textiles, common materials used for making conductive textiles, properties of metal coated textiles, preparation methods, significance of in-situ deposition of metal particles, electroless plating and applications of metal coated textiles.

3.1 Electrically conductive metalized fabrics

The metallization of textile is the process which adds to and enhance the functional properties of textile [17]. Metallized textiles have numerous advantages over fabrics made entirely of thin metal wires and polymers. The advantages over no metallized fabrics include resistance to sever weather conditions like sunlight, smog and soil, resistance to chemicals and lower the sorption of hydrophilic materials. Thick and dense coating of metal is done in the field of protective textile to enhance the anti-ballistic and anti-stabbing properties. On the other hand by thing metal coating we can achieve the flexible and light in weight fabrics as compared to fabrics produced by thin metal wires and metallic sheets [21, 22].

Another advantage is in the field of textile fashion designing to produce the bright, shining and decorative textiles [23, 20]. By using metallization, it is possible to reduce the static charge. The static charge usually produces on the surface of synthetic fabrics and polymer sheets. These charges cause the attraction of dust particles and significantly put the electronic chips and devices in risk of damage [21, 22]. Another significant advantage is in the field of smart textile. Where metal coated textiles are important due their novel property of electrical conductivity and electromagnetic shielding. Electrically conductive flexible textile materials are new approach for the production of fabric sensors which can be used for certain medical application like electromyography (EMG), electroencephalography (EEG) and electrocardiogram sensing. These textiles also use in bandages and surgical gowns due to antibacterial effect. Moreover, due to their novel property of flexibility they can be integrated into textiles to detect field effect transistors, health monitoring, sports action detectors. Metal coated textiles can be used as electrodes in energy harvesting devices and in the replacement of carbon based polymer electrodes etc.

3.2 Different materials for metallization

From point of view of electrical conductivity, the materials are classified as conductors, semiconductors, and insulators. Electrical conductivity is the reciprocal of electrical resistivity, represented by letter ρ (rho). The electrical resistivity is mostly measured in SI unit represented as ohm-meter (Ω·m). While the SI unit of electrical conductivity is siemens per metre (S/m). In case of electrically conductive textiles, the resistivity can be measured in two ways. The one is measuring the surface resistivity and second is volume resistivity. The most common unit for volume resistivity (according to ASTM D257) are Ohm centimetre (Ω cm) or Ohm millimetre (Ω mm). However, the physical unit for surface resistivity is ohm (Ω), which often mixed with the standard (SI derived) unit for resistance. In order to differentiate between these two confusing units (unit of electrical resistance and electrical surface resistivity), the surface resistivity now often expressed in Ohm/square (Ω/square). The range of electrical resistivity for different materials is classified in Figure 1.

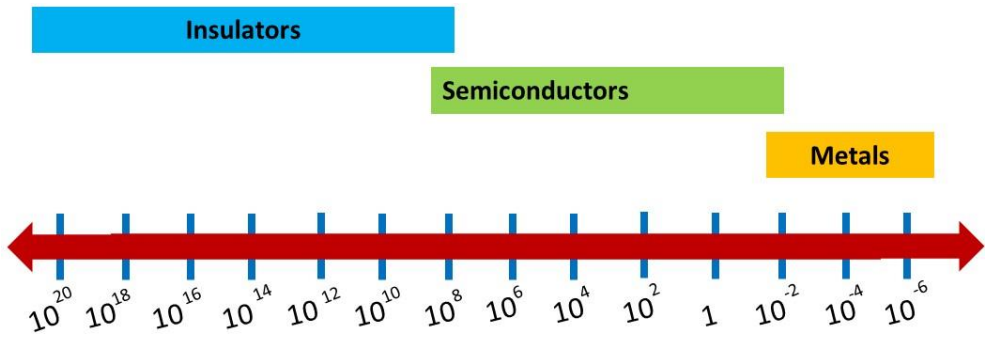


Figure 1: Range of electrical resistivity (Ω cm) for materials [21].

In general, for making conductive textiles we mostly prefer the class of conductors. Good conductive materials are metals, carbon and intrinsically conductive polymers [22]. Initially, metals have been weaved into textile structures just for decorative purpose. It was 1960s when first time textile was metallized by vapor deposition technique for attaining the electrical conductivity. To achieve the electrical conductivity metals can be coated in pure form (only one element) as well as in the form of alloys (compounds by using different combinations). The most commonly used metals for high electrical conductivity are silver, copper, gold, aluminum, zinc, cobalt, palladium, platinum, and nickel [24, 27]. Among all conductive metals silver is the most conductive and is most demanding due to high oxidation resistance. Copper comes next to silver regarding conductivity but its conductive strength is almost very near to silver. Copper is most demanding due to its availability and low cost.

However, the surface of Cu film especially of nano size is susceptible to oxygen and gets oxidized spontaneously.

To enhance the conductivity and avoid from oxidation the trend of co-metal deposition has led to a new generation of metal coating. This composite deposition is popular for more than 50 different alloys and among them mostly are based on silver, copper, cobalt and nickel. The most famous combinations are Cu-P, Ni-B, Cu-Ni, Ag-Cu, Cd-Cu, Ni-P, Cu, Cu-P, Co-P, Ni-Cu-P and Ni-Sn-P [24]. These copper-based alloys give high electrical conductivity. Silver copper alloy has almost the same electrical conductivity as ordinary high-conductive copper and give better resistant to oxidation as compared to use copper alone. However, the alloys of copper and silver with beryllium, tin, cadmium, chromium and bronzes have a higher electrical resistivity (almost about 20 to 85%) than pure copper and silver but are more resistant to corrosion and wear [25]. The order of electrical resistivity and conductivity of different materials at 20 °C is given in Table 1.

Table 1: Electrical conductivity of materials at standard temperature.

Material	**ρ (Ω m) at 20 °C**	**σ (S/m) at 20 °C**	**Reference**
Graphene	10^{-8}	10^{8}	[26]
Silver	1.59×10^{-8}	6.30×10^{7}	[27]
Copper	1.68×10^{-8}	5.96×10^{7}	[28]
Gold	2.44×10^{-8}	4.10×10^{7}	[27]
Aluminium	2.44×10^{-8}	3.50×10^{7}	[27]
Tungsten	5.60×10^{-8}	1.79×10^{7}	[28]
Zinc	5.90×10^{-8}	1.69×10^{7}	[29]
Nickle	6.99×10^{-8}	1.43×10^{7}	[28]
Iron	9.71×10^{-8}	10^{7}	[27]
Tin	1.09×10^{-7}	9.17×10^{6}	[28]
Lead	2.20×10^{-7}	4.55×10^{6}	[27]
Mercury	9.80×10^{-7}	1.02×10^{6}	
Graphite	2.50×10^{-6}	2×10^{5}	
Carbon black	5.00×10^{-4}	1.25×10^{3}	[27]
Conductive polymers	10^{-3} to 10^{-1}	10^{1} to 4.6×10^{3}	[30]
Sea water	2.00×10^{-1}	4.80	[29]
Drinking water	2.00×10^{1}	5.00×10^{-2}	[30]

Semiconductors	6.40×10^{2}	1.56×10^{-3}	[27]
Wood	10^{4}	10^{-4}	[30]
Glass	10^{11}	10^{-11}	[27]
Hard rubber	10^{13}	10^{-14}	[30]
Air	1.30×10^{16}	3×10^{-15}	[30]
Diamond	10^{12}	10^{-13}	[27]
PET	10^{13}	10^{-13}	[31]
Teflon	10^{23}	10^{-23}	

3.3 Properties of electrically conductivity textiles

3.3.1 Electromagnetic interference shielding

Nowadays, electromagnetic interference (EMI) is the fourth kind of public space pollution. EMI consists of unwanted radiated signals causing unacceptable degradation of system or equipment performance. These problems if left unattended can cause severe damage for commercial and scientific electronic instruments, safety operation of many electronic devices, antenna systems and military electronic devices [32]. Most of the common and main reason for electromagnetic interference is the development of electrostatic discharge (ESD). It is a common phenomenon when high frequency signals transmitted out from one electronic device and cause the malfunctioning of nearby equipment. Electrostatic discharge (ESD) can be easily observed even by a non-technological person. The common signs of ESD are bumping of mobile phone near the television, click heard on audio systems when a light is switched on, distorted television reception in the form of flashes on the screen. Electromagnetic radiations are also hazarding for human being regarding health and cause many problems such as nervousness, headache, symptoms of languidness, insomnia on exposure to electromagnetic waves. As a result, the research on electromagnetic shielding materials has increased dramatically. The Electromagnetic shielding can be defined as to prevent the propagation of electrical and magnetic waves from one place to another by using either a conductive or magnetic material. The simple theory for the prevention of EMI is to filter all the incoming and outgoing interferences. Shielding effectiveness is the ratio of imposing energy to the remaining/residual energy. During the shielding mechanism, the electromagnetic radiations are absorbed and reflected. The residual is defined as part of the remaining energy that is neither reflected nor absorbed by the shielding material but it is emerged out from the shield. The effect of EMI shielding can be accomplished by minimizing the signals passing through a system

either by absorption or reflection of waves. The reflection of radiation from the shield is possible if the shield has mobile charge carriers [3]. From these aspects, flexible conductive textiles have gained the popularity due to their satisfactory electrical conductivity, electromagnetic interference shielding effectiveness, electrostatic dissipation, breathability, and light weight. In a variety of manufactured forms such as fabric tape or foam gaskets, they are an important kind of materials for preventing electromagnetic interference [33]. For instance, these fabrics could prevent the possible explosions of guided aircraft due to electromagnetic interference.

In order to render the textiles with electrical conductivity and electromagnetic shielding, there are various approaches such as surface metallization, coating of carbon materials, coating of conductive polymers, etc. Among them, the textile surface metallization is a kind of unique method which can provide multifunctional properties such as electrical conductivity, electromagnetic interference shielding, anti-static and anti-bacterial properties, UV radiation screen, and radar reflectivity. The commonly used metal coating techniques are metal foil and laminates, conductive paints and lacquers. However, metallic fabric produced in such traditional manners consists of defects, such as stiffness, poor air permeability and heavy in weight. The sputter coating, vacuum deposition, flame and arc spraying, and electroless plating are some of the novel methods of surface metallization which can overcome the limitations of traditional methods [34].

The electroless plating has advantages such as coherent metal deposition, excellent conductivity and shielding effectiveness, and applicability to complex shaped materials [35]. In previous research, fabrics plated with Cu showed more effective EMI shielding than those plated with Ni and Cu–Ni. Each metal coated fabrics showed EMISEs of 68–35 dB for copper, 37–32 dB for nickel, and 46–32 dB for copper–nickel, respectively at the frequency of 100 MHz–1.8 GHz [36]. In another research, the ultrasonic-assisted electroless Ag plating of polyethylene terephthalate fabrics showed the SE of more than 32 dB at frequency ranging from 0.01 MHz to 18 GHz [33, 36]. However, the plating of Ag is costly than the plating of copper. Due to novel properties such as flexibility and stretch ability the metallized textile are also emerging as a material of choice for EMI shielding of sensitive equipment, particularly in aerospace and defense system. Electrically conductive textiles are widely used as a shielding cover in wide-ranging products for the computer, general electronic, automotive industries, medical applications, telecommunications, aerospace and defense applications [37]. Instead of fabric form, the metal-coated textiles are tailored to produce the ready-to-use adhesive tapes, panels, bags, curtains and so on [38]. Metal particles or electroless plated fabrics are quite

flexible and comfortable, can be used in undergarments for personal protection. These garments are most in-demand for pregnant women who used to work in front of computer and in the electromagnetic radiation environments during the period of pregnancy. Electrically conductive fabrics could also serve to reduce the effects of complex electromagnetic environment on guided aircraft for its improvement of the survival ability. Undergarments which are even made from the metallized fabric can provide personal shielding against the low-frequency electromagnetic radiation. Targeted markets include pregnant women who operate computers and those who work around high voltage lines and microwave environments [39].

3.3.2 Heating performance of electrically conductive fabrics

Nowadays, flexible electrically conductive fabrics are widely used in heating systems of various technical applications. Heat producing textile is new field of interest for electronic researchers. The main advantages of using conductive textiles for heat producing system are due to their unique ability to bend, flexibility and compatibility with other irregular geometries Conductive yarns and fabrics are better replacement of heat producing electronic chips and hard wire being incorporated into the textiles [40]. Ohmic heating (takes its name from Ohm's law) is also known as resistive heating or joule heating. The principle of ohmic heating is that heat generates by flowing current through conductive textile which resists the flow of electric charges. The applied voltages and opposed current by resistance in a circuit based on a basic principle of Ohm's law as illustrated in Figure 2 [41].

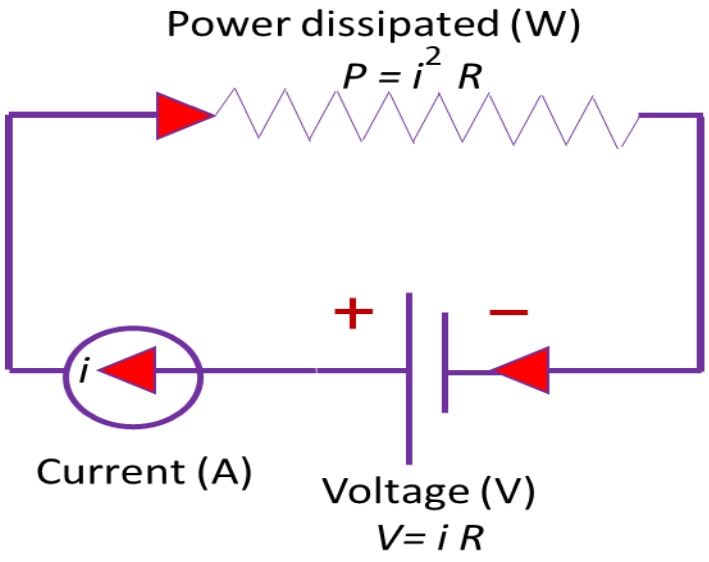


Figure 2: Illustration circuit based on a basic principle of Ohm's law [41].

where P is generated power, i is the current flowing through conductive material, V is voltage and R represents the resistance of material.

By cutting the conductive textiles into various shapes and sizes the resistors of different specific values can be produced. The conductive strips (cut from conductive textile) can be placed or stitched as parallel or in series with wearable garments. During the flow of electric current from these yarns or strips, the amount of heat will produce. Usually, the conductive fabrics have range of resistance value from a few ohms to many kilo-ohms. The theory is that the conductive strips should be thin enough to produce more heat. However, in the same time, the overflow of electric current should me manage to avoid from breakage of circuit. Every square inch of textile will dissipate heat in terms of several watts of power depending on the resistivity level of used conductive strips. However, to produce more heating the high voltages should be applied but fabric strips must be stronger against heavy charges. In this situation plain weave, twill or single jersey garments with heavy GSM about more than 150 g/m^2 can be chosen. Other most important factor to produce resistive heating fabrics is the selection of fibers. The fabric composed up of natural fibers like cotton or wool are more suitable than man made synthetic fibers. Moreover, the attached battery (current source) should be light in weight but it must produce enough voltages [42].

In order to obtain higher power dissipation, the resistor can be scaled up in the fabric area However, in the case of high-power dissipation the high level of power supply is mainly required. Textile-based electrical heating systems should be wrapped with a flexible surface or garments. Hence, produce the efficient heating performance due to heat delivery by conduction and convection mechanisms. These garments are more significant to regulate the human body temperature during the outdoor activities like skiing, snowboarding and hiking, etc.

In modern living style, the textile-based heated panels and curtains should replace the use of radiators and conventional heaters. Heat generating textiles can be incorporated into various household items such as carpet, blankets, seating, bedding and towels. In addition to household items heating gloves and jackets also cover the big market. The main aim for using the heating system in fabrics is to maintain the human body temperature. The normal temperature of human body is around 37°C, below which severe health condition hypothermia can occurred. Therefore, textile heaters are necessary in various applications and conditions. These textile heaters can be applied on various parts of human body. However, the flexibility of the heating system is necessary to shape or wrapped according to irregular body parts.

Heat producing conductive textiles are classified according to the type of conductive material used in them. The heat-producing electrically conductive textiles are produced by coating of conductive polymers, inks, metals or carbon-based materials. Metal-based textile heaters used in Dorman® - Seat Heater Pads is shown in Figure 3.

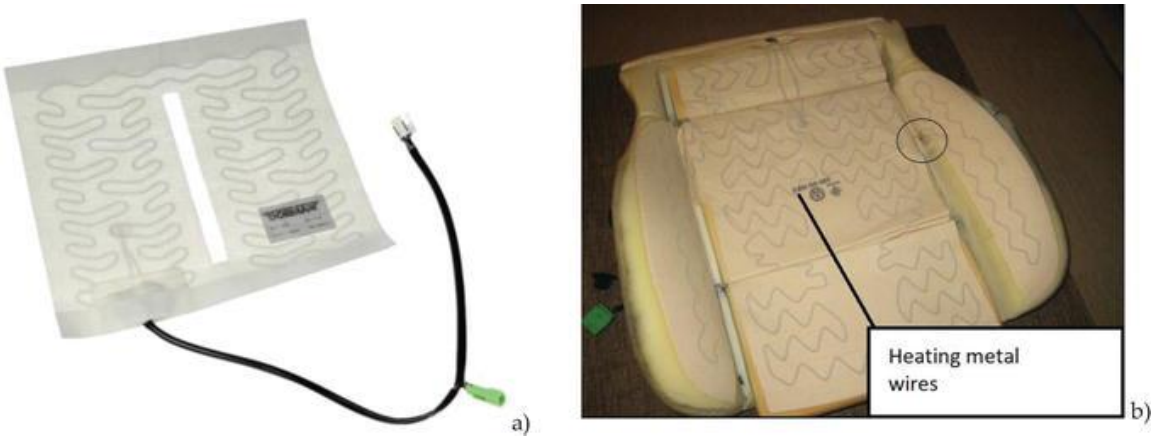


Figure 3: Textile-based car seat heaters[43].

Metal coated textiles are more suitable for human body due to unique properties. The silver or copper-coated electrodes in electrotherapy of various human parts (muscles, tendons and ligaments) will also provide antibacterial effect in addition to heating. Several research studies reported on textile-based resistive heating system. The Haitronic HPS605D power supply was used to provide DC voltage (from 0 V to 60 V) to each heating element, A range of voltages (from 0 to 60 V) with 1 A of constant current was applied to each conductive fabric. The maximum heat generated was recorded by FLIR C2 thermal camera. The type of material, conductive coating and burn out temperature are listed in Table 2 [44].

Table 2: Heat generated by different materials during burning *[44]*.

Material/Substrate	**Type of coating**	**Temperature °C**
Woven polyester fabric	Nickel-Copper-Nickel	110 °C
Polyester nonwoven	Nickel on Copper plated	120 °C
Polyester /nylon 6 fabric	Nickel-Chromium	105 °C
Polyester/nylon/cotton	Nickel-Copper-Chromium	195 °C

3.3.3 Antibacterial

Hygiene has acquired importance in the field of protective textiles. In the field of medical and high-tech applications, textile consumers are looking for antibacterial fabrics.

Microorganism growth is dangerous for both living and non-living matters. Microorganisms have severed effect on textile raw materials, wet processing chemicals, rolls or bulk materials in storage room. Some critical effects of microbial attack are unpleasant smell from socks and undergarments, spread of stains, degradation of textile fibres and even some allergic diseases. Famous species of micro-organisms are yeast, mould, fungus, mildew. There are both good and bad types of microorganisms are part of our everyday lives.

Some natural fibres are better food and living place for microbes due to their inherent natural properties of hydrophilicity and their porous structure. Micro-organisms like the moist and warm environment in socks and developed the rapid colonies. The pathogenic microbes have adverse effect on human skin and cause severe infection. In addition, the staining, bad odour and loss of the functional properties of textile substrates are the results of microbial attack. Antimicrobial agents used to kill or inhibit the growth of bacteria. Agents that kill bacteria are called bactericidal, while the agents that inhibit their growth are known as bacteriostatic. Usually, antimicrobial agents are classified into two groups one is natural anti-microbial agents and second is synthetic antimicrobial agents. Effective natural antimicrobial agents are chitosan, clove, turmeric, tulsi, neem, pomegranate and Aloe Vera. While among synthetic agents the Antimicrobial dyes, Quaternary ammonium compounds, Triclosan (2, 4, 4'-trichloro-2' hydroxydiphenyl ether), Regenerable N-halamine Polyhexamethylene biguanides (PHMB), and peroxyacids, Metals and metal salts such as copper, silver, zinc, and nanoparticles of noble metals and metal oxides. Microbes are always present on human body even on clean skin. The normal range of microorganisms on human hand are between 100-1000 microbes/cm^2. Textiles, having feature approximates to human body provide an excellent medium for growth, adherence, propagation and transfer, of infection caused by microbial species.

The antibacterial property of coated fabrics can be attributed to the combination of chemical and physical interactions of bacteria with particles. Nanoparticles can incorporate into the cell via endocytosis mechanisms. Afterward, the cellular uptake of ions increased as ionic species were subsequently released within the cells by nanoparticle dissolution [45]. This resulted in high intracellular concentration gained within the cell for further massive oxidative stress.

Micrometric metal are did not cause cell damage as compared to highly biocidal nanoparticles at the same mass [46]. A summary of the mechanisms associated with the antimicrobial behavior of metal nanoparticles are shown displayed in Figure 4.

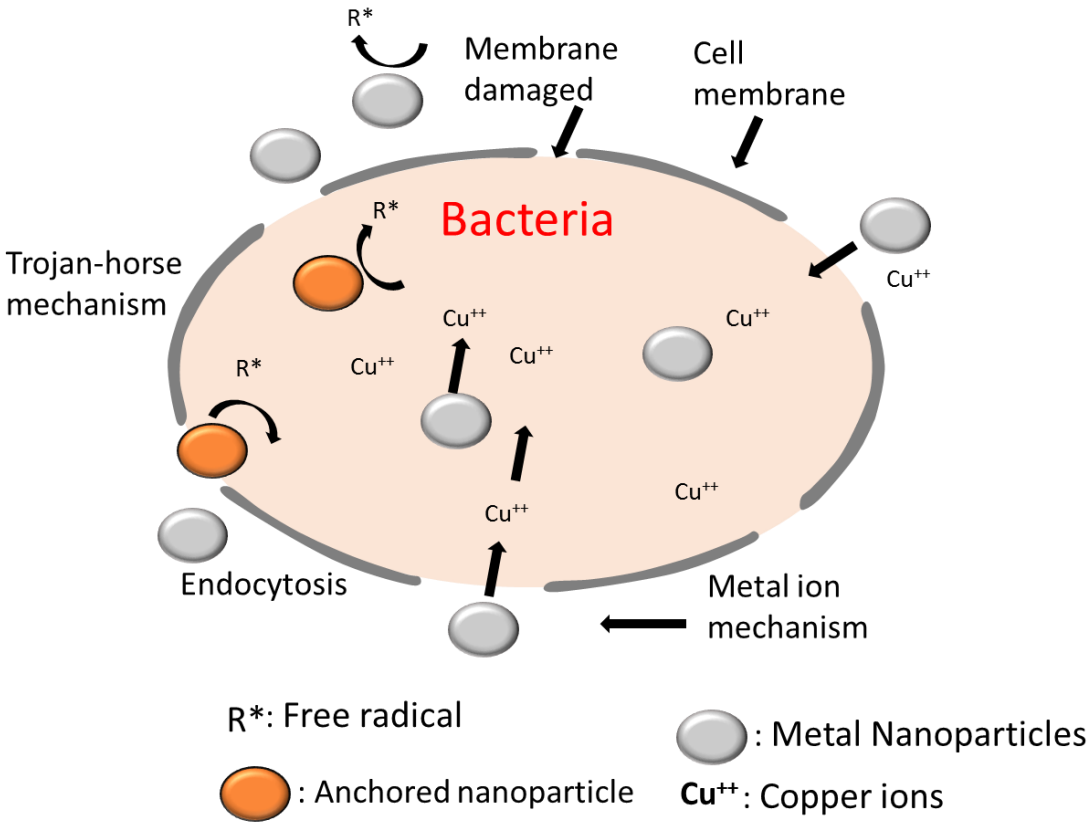


Figure 4: Brief description of mechanisms associated with the antimicrobial behavior of metal nanoparticles, catalyzed radical formation, release of metal ions and Trojan-horse effect" due to endocytosis processes[47].

The unique properties of antimicrobial effect of nanoparticles arise from a variety of aspects, including the similar size of nanoparticles and biomolecules such polynucleic acids and proteins [48]. The antibacterial behavior of copper nanoparticles could be attributed due to the chemical interactions, physical interactions or combination of both:

(a) Chemical interactions can occur between:

(i) Cell membrane components and Cu^{++} ions , (ii) Cu^{++} ions and components in the interior of cell (transportation of Cu^{++} ions into the cell), (iii) Cell membrane components and H_2O_2, generated due to the presence of Cu-NPs, (iv) Chemical species generated due to Cu nanoparticles and components in the interior of cell, (v) Cu nanoparticles are able to form organic complexes with sulfur-, nitrogen- or oxygen-containing functional groups present in the microorganism. This may result in defects in the conformational structure of nucleic acids and proteins, besides changes in oxidative phosphorylation and osmotic balance. Finally,

micro-organisms exposed to toxic doses of some metal particles upregulate genes involved in the elimination of ROS generating oxidative stress [49].

(b) Physical interactions can be as follows:

(i) Physical blockage of the transport channels of cell membranes by Cu nanoparticles, (ii) Physical damage to the membrane components by Cu-NPs due to abrasion, (iii) Penetration of Cu-NPs particles through cell membrane to interact with interior of the cell, (iv) direct interaction between Cu-NPs and bacterial cell membrane components through electrostatic effect.

(c) A combination of the physical and chemical interaction as described above.

These results stress the complex behaviour of antimicrobial Cu nanoparticles as either the particle itself or their ions can participate in the biocide mechanisms. Anyway, independent of that, in the end, metal nanoparticles are the active biocidal agent.

Copper is most essential for life; therefore, all cells possess a sufficient amount of inherent copper. Hence, to keep intracellular copper at safe levels all cells possess copper homeostatic mechanisms. However, under different environmental conditions external copper cause to imbalance the homeostatic system and leads to overload the intracellular copper. The condition becomes too severe and approaches to the toxic level. The toxic effect of copper has a wide range of mechanisms and till now it is unknown to describe with certainty which mechanism is active against a particular bacteria. The redox property of copper is the well-known and most occurring mechanism resulting in lethal oxidative damage to cells. All different mechanisms are still under research. to various extents. In fact, under different environmental and growth conditions the behaviour of copper is different on cells. The summary of all major mechanisms is closed in Figure 5. Unknown pathways support to enter the copper in cells. The cytoplasm of cell has the ability to reduce the copper to Cu^{+}, which in turn participate in Fenton type reactions and produce highly reactive hydroxyl radicals. This leads to reacts non specifically with proteins, lipids, nucleic acid and free amino acids. The anaerobic conditions make the copper-glutathione complexes, which behaves as copper-donors for metalloenzymes. The dominant toxicity mechanism is referred as the displacement of iron from iron-sulfur cluster proteins by Cu^{+}[50].

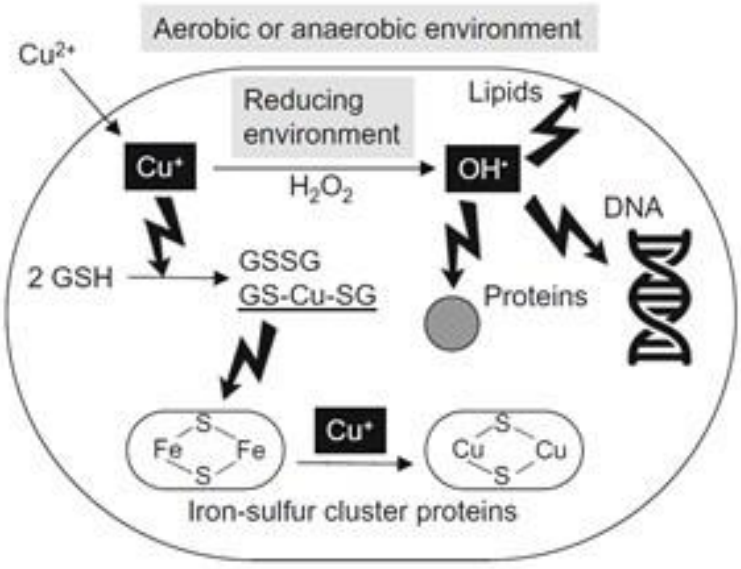


Figure 5. Mechanisms of copper toxicity[50]

3.4 Preparation methods of electrically conductive metalized fabrics

Metallization methods of textile has a long history and have been modified with the passage of time. In ancient times, thin wires or flat strips of metals used to weave into the fabrics to add aesthetic and decorative properties. These technologies were not changed until the twentieth century. The tremendous revolutions in metallization came between the world wars, where the strips of metal were bonded both side of cellulosic fabrics. However, the developed structures were heavy in weight and hard [19]. After the Second World War, the technologies were radically changed. The metallization of textile started with new coating techniques. The commonly used metal coating techniques are metal foil and laminates, conductive paints and lacquers. However, metallic fabric produced in such traditional manners consists of defects, such as stiffness, poor air permeability and heavy in weight [34]. The sputter coating, vacuum deposition, flame and arc spraying, and electroless plating are some of the novel methods of surface metallization which can overcome the limitations of traditional methods [34, 51]. Some researchers selected the sensitization of surface firstly with vacuum deposition and sputtering, secondly applied silver plating on the surface of conductive material. This technique is better for achieving surface conductivity but in this case, it is very difficult to achieve volume conductivity through material. Among these techniques, electroless plating over solution sensitized fabrics is considered better regarding volume conductivity, durability, coherent metal deposition and applicability to complex shaped materials. Electroless copper

plating is a type of non-electrolytic method of deposition from solution on fabrics and has been applied by researchers [52]. As reported in a study, copper electroless deposition method uses a catalytic redox reaction between metal ions and dissolved reduction agent of formaldehyde in alkaline medium at high temperature. This technique is risky due to the formation of hazardous gaseous product during the plating process and badly fails especially for industrial scale. The researches had tried to solve this problem by substituting formaldehyde with other reducing agents coupled with oxidation accelerator such as sodium hypophosphite and nickel sulphate. Except that researchers have been tried different types of electroless playing but each technique was having some limitations. As electroless plating by substituting with other reducing agents like sodium hypophosphite arise the limitations regarding fixation and improper reduction due to low reduction potential and also copper is not a good catalyst for oxidation of hypophosphite [53]. They have been applying electroless plating in acidic media by using HCl, this, in turn, change the reduction potential of reducing agent and also oxidation of reducing agent produce H^+ ions. Electroless plating over nickel, palladium, stannous, cobalt and lead (needs pre-treatment steps) cannot be used for hygienic applications, as these metals are hazardous and irritant to skin [54]. Some studies described the conventional process about the use of palladium metal which is commonly employed as the catalyst sites to initiate the electroless plating [55]. Now a day, this method of plating is becoming costly due to increase in the cost of the palladium. So, there was need to develop a cost-effective and reliable activation process. Furthermore, people has been using two-step methodologies before copper plating first is sensitizing and second is activation then plating. Therefore, further research is necessary to effectively prepare copper coated fabric in safe conditions [6].

3.5 Research on electrically conductive metalized fabrics

A number of techniques have been devoted to plating of copper using formaldehyde as reducing agents, however, this process may release hazardous gases during their operation [3]. In another research, the ultrasonic-assisted electroless silver plating of polyethylene terephthalate fabrics showed the SE of more than 32 dB at frequency ranging from 0.01 MHz to 18 GHz [56]. However, the plating of silver is costly than plating of copper. Therefore, further research is necessary to effectively modify the textile surface with controllable conductivity and high durability in safe conditions. Several papers have studied the electroless copper plating solutions using sodium hypophosphite as reducing agent due to its low pH, low cost, and relative safety features [3]. Those researches focused mainly on the effect of additives,

on the properties of the deposits and the application of electroless copper plating to fabrication of printed circuit boards.

In previous research, fabrics plated with Cu showed more effective EMI shielding than those plated with Ni and Cu–Ni [36]. Each metal coated fabrics showed EMI SEs of 35 dB for copper, 37–32 dB for nickel, and 46–32 dB for copper-nickel, respectively at the frequency of 100 MHz–1.8 GHz [33]. Deng *et a*l. deposited the aluminium films on textile structure. It was observed that after the 30 minutes of coating time, the substrate showed the lowest electrical resistivity about 21 Ω cm than coating for 20 minutes 5.3×10^2 Ω cm [57]. Roya et al. coated silver particles over the cotton substrate. The fabric was first pre-treated with poly(diallydimethylammonium chloride) solution. After that silver nitrate was used to develop the silver particles by reduction method and obtained lowest surface resistivity about 0.2 Ω/square [58]. Chao et al. describe the effect of silver particles coating on simple cotton fabric and found lowest electrical resistivity about 37 Ω/square. They also studied the antimicrobial property of developed fabrics and found the clear zone of inhibition against gram-positive and gram-negative bacteria [59]. In another study, polyester fabrics were coated with copper and silver particles to make the electrically conductive, antibacterial and hydrophobic textiles. The fabrics showed a higher antibacterial efficiency against the gram-positive bacteria as compared to gram-negative bacteria. They studied the electrical resistivity about 184 ohm/square [60]. Hui et al. [7] combined silver nanowires with Cupro fabrics (it is viscose prepared via dissolution in Schweitzer reagent) for dissolution in using a dipping–drying method to prepare electrically conductive fabrics. The silver nanowires were first adhered to and then absorbed by microfibers to form electrically conductive fibers. The electrically conductive fabric had low resistivity and good stretchability as well as excellent flexibility. Xue et al. produced Ag particles on cotton fabric by reducing the $[Ag\ (NH_3)_2]^+$ complex with glucose. Ag particles formed a dense coating around the fibres. Dan et al. describe a facile way to make the copper silver complex coating. The fabric was pretreated by carboxylic styrene butadiene with catalyst PdCl, then activated with silver and copper plating was performed. They achieved electrical resistivity about 60 mΩ/square and EMI SE about 70 dB [61]. In another study a cotton fabric was plated with copper where different concentrations of copper sulphate were studied with different intervals of time. They achieved very less electrical resistivity with EMI shielding about 12 dB [36]. Wei et al. examined the surface morphology, electrical properties and wetting performance of textile coated by silver metal. The electromagnetic shielding efficiency was found to increase with increase in the coating of metal (Figure 6).

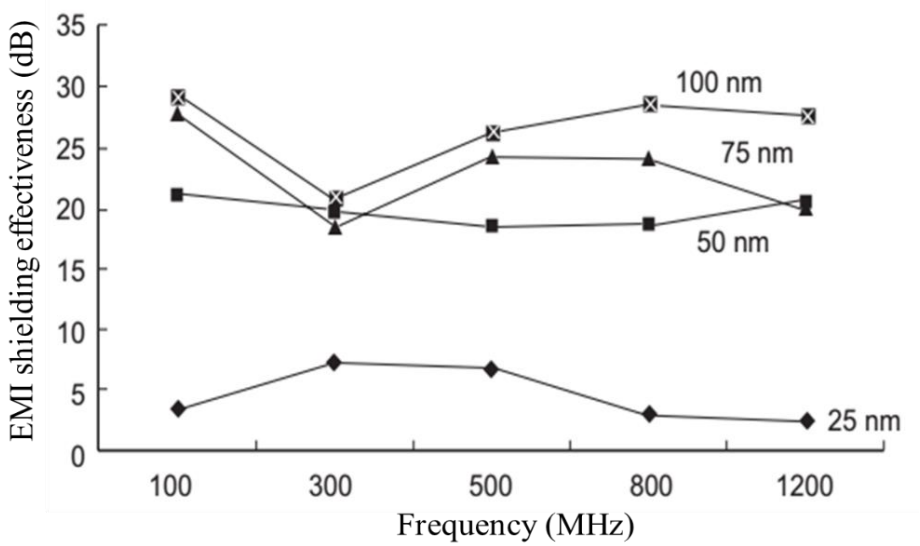


Figure 6. Increase in EMI SH with increase in thickness of coating [61].

In another work, highly electrically conductive and super amphiphobic cotton fabrics were fabricated by a solution-dipping method which involved $(NH_4)_2PdCl_4$-catalyzed electroless deposition of copper. The as-prepared fabrics had a sheet resistivity of ~ 0.33 Ω/square and showed excellent electromagnetic interference shielding with electrothermal heating ability. Furthermore, the decrease in electrical resistivity was observed with increase in plating time (Figure 7).

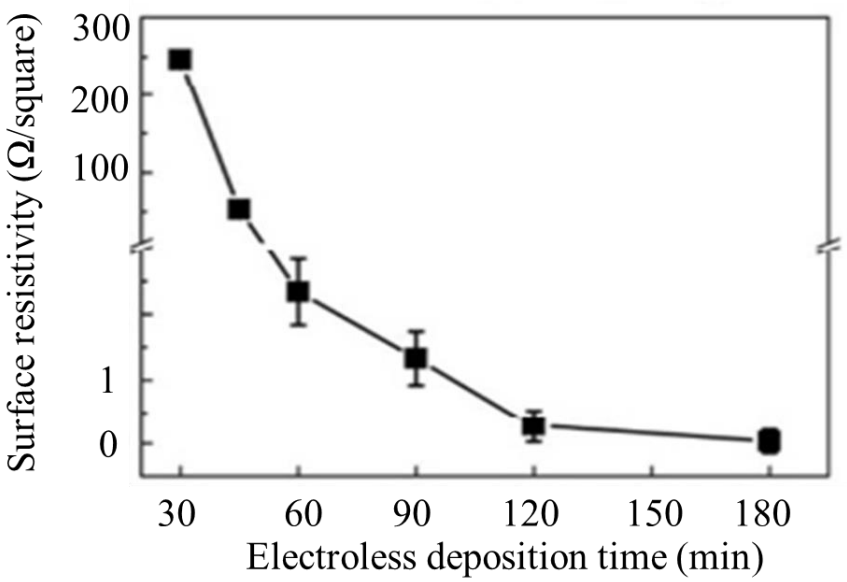


Figure 7. Decrease in surface resistivity with increase in electroless plating time[62]**.**

The researchers also developed fluorinated, copper plated, florinated- poly-allylamine hydrochloride (PAH) treated (F/Cu/PAH) fabrics for durable EMI shielding performance under corrosive environments. The average EMI SE of the Cu/PAH-coated and F/Cu/PAH-coated cotton fabrics was reported ~20.9 and~20.8 dB, respectively. The F-POSS/POTS layer endowed the resultant cotton fabrics with a durable EMI shielding performance as the average EMI SE of the F/Cu/PAH-coated cotton fabrics slightly decreased to ~17.4 and ~18.0 dB after immersion in aqueous H_2SO_4 solution (pH 1) and KOH solution (pH 14) for 100 hours (Figure 8a). On the other hand, the Cu/PAH coated cotton fabrics without the super amphiphobic layer almost completely lose their EMI shielding performance after immersion in aqueous H_2SO_4 and KOH solutions for 2 hours (Figure 8b) [62].

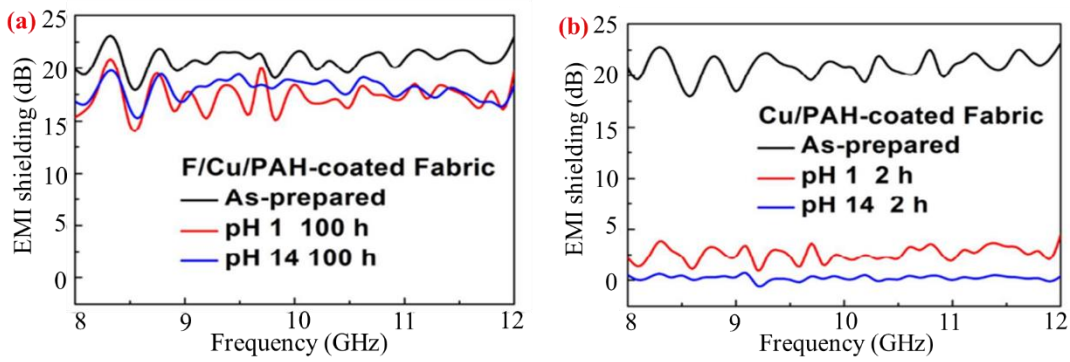


Figure 8: (a) EMI SH of F/Cu/PAH coated textile before and after treatment with H_2SO_4 at pH 1 and with KOH solution pH 14 for 100 hours. (b) EMI SH of F/Cu/PAH coated textile before and after treatment with H_2SO_4 at pH 1 and with KOH solution pH 14 for 2 hours and F/Cu/PAH coated textile before and after treatment with H_2SO_4 at pH 1 and with KOH solution pH 14 for 100 hours [62]

Yahya Atwa et al. demonstrated much lower weight and mechanically flexible coating by deposition of random networks of solution-synthesized silver nanowires on nylon, polyester, and cotton threads. Silver nanowires were synthesized in solution at relatively low temperatures by the polyol method and their deposition around threads was achieved through dip-coating. The density of the deposited nanowire film was varied through the concentration of the nanowires in the coating solution and through the number of dipping steps. Figure 9 shows the microstructure of nylon, cotton and polyester threads after 3 times dipping in solution, and their resistivity was measured around 12 Ω cm, 11 Ω cm and 15 Ω cm, respectively. The dependence of resistivity against metal contents is tabulated in Table 3 for the nylon threads, where it showed that increase in the amount of metal content resulted in decrease of electrical resistivity [63]. Furthermore, they performed different bending cycles to check the durability of coated

nylon threads. The resistivity of silver-coated nylon thread was almost constant as compared to commercially available conductive thread. This behaviour can be seen in Figure 10.

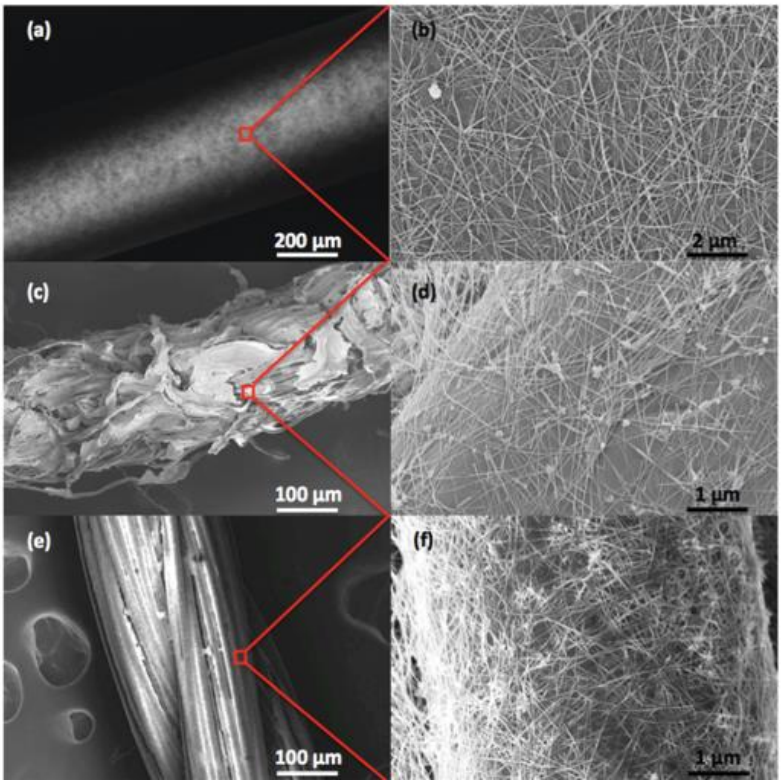


Figure 9. SEM images of silver nanowires coated (a, b) nylon (b, c) cotton and (c, d) polyester fabrics [63].

Table 3: Electrical resistivity for silver-coated nylon thread [63].

Metal coating (mg m^{-1})	**Volume resistivity (Ω cm)**
0.24	12.0
0.52	2.5
1.07	0.8

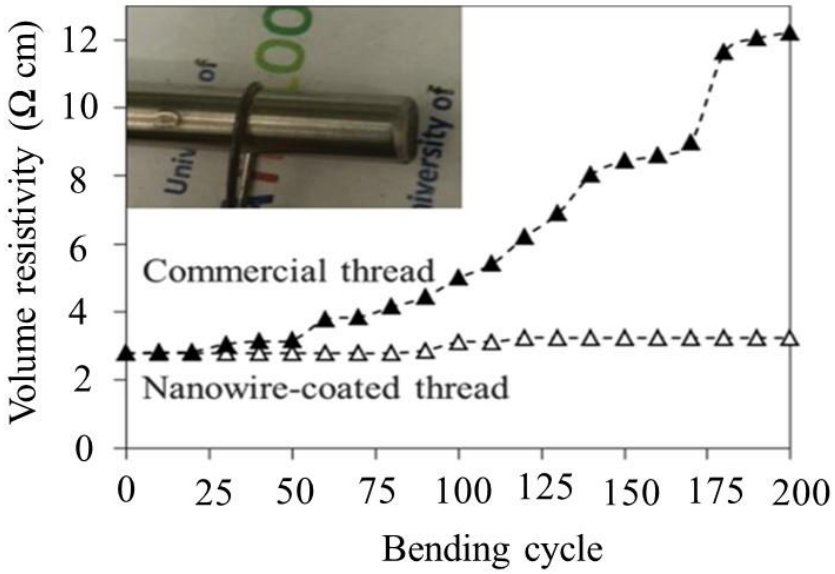


Figure 10. Comparison of electrical volume resistivity between silver nanowires coated thread and commercially available thread at different number of bending cycles [63].

An alternative technique to deposit Ag on textiles was proposed by Hegemann et al. using plasma sputtering which allowed cleaning and deposition in a one-step process. The authors compared the plasma sputter fabrics with electroless plated fabrics (see Figure 11). Excellent adhesion on polyester textile was obtained with smooth coatings, however, the resultant electrical conductivity was limited (ranging in kΩ) due to line of sight shadowing of the incoming sputtered atoms [64].

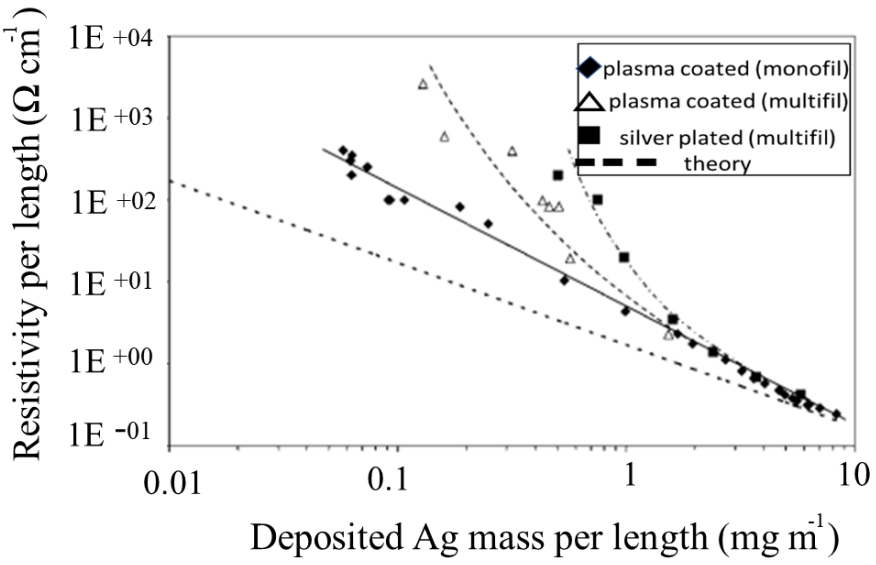


Figure 11. Electrical resistivity as function of the amount of silver deposition [64].

In another research, polyelectrolyte brushes were first grown on cotton surfaces by surface initiated atomic transfer radical polymerization. Then, the electroless deposition of metal particles was subsequently performed onto the brush-modified cotton yarns. The conductivity of the yarns was found to increase with increase in plating time and reached~1 S/cm at 60 min. Later, the durability under multiple bending and stretching cycles was investigated and the conductive yarns exhibited higher conductivity (0.28 S/cm) when stretched, and lower conductivity (0.04 S/cm) when the stress released. This process was found fully reversible up to 30 cycles. The durability of copper-coated textile was also investigated with the passage of days as shown in Figure 12. The significant effect of reduction in electrical conductivity was observed due to the susceptibility of copper towards the oxidation [5].

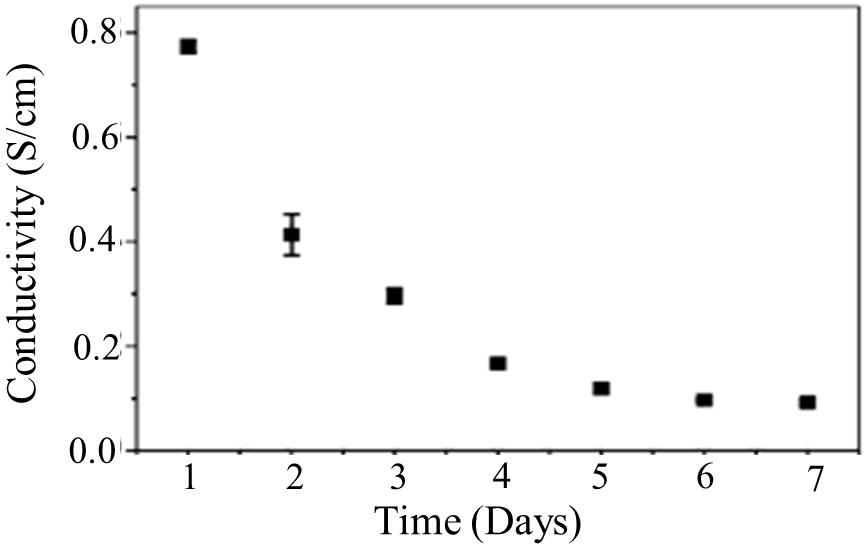


Figure 12. Decrease in electrical conductivity with the passage of days [5].

The single metal treatment with Cu or Ag, and the combined metal treatment of Cu/Ag were compared for the multi functionality by Hyae et al. With regard to surface resistivity, Cu/Ag combined treatment showed the lowest surface resistivity of 25.17 Ω/square. Interestingly, the surface resistivity was found stable around 184.38 Ω/square even after hydrophobic coating. On the other hand, no conductivity was observed in all cases of single treatments of metal nanoparticles regardless of the type of metal nanoparticles used and whether or not the surface hydrophobic coating was applied. This behaviour was attributed to

the difference in the add-on of the metal particles and the difference of exposed area of metals [60].

Another research reported the electromagnetic shielding effectiveness of conductive knitted fabrics made up of hybrid yarns containing 50 μm diameter metal fibres such as copper, silver and stainless steel. The hybrid yarns were produced with core-spun yarn spinning machine. The variation in electromagnetic shielding effectiveness (EMSE) with the factors, such as radiant frequency, metal type, wales density and geometry were discussed. Among all experimental factors, the geometry of the fabric was found to affect shielding effectiveness significantly where Milano type knitted fabrics provided shielding effectiveness above 20 dB. The Milano and cardigan composite fabrics gave better shielding performances than rib and weft composite fabrics (See Figure 13) [65].

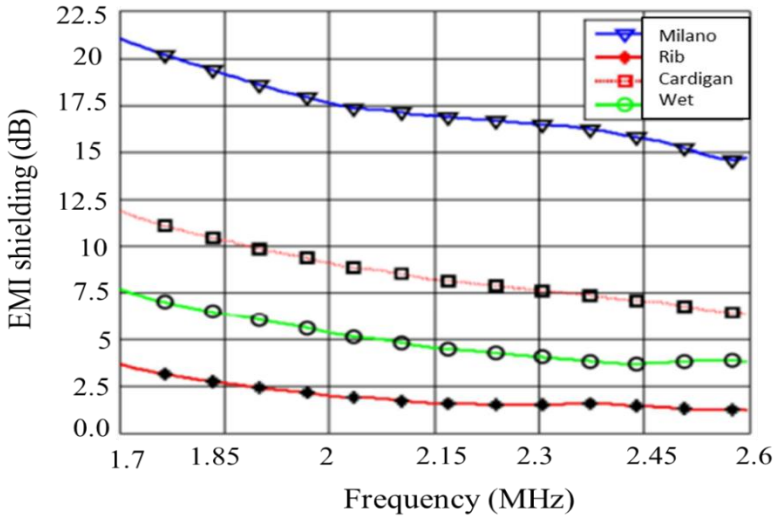


Figure 13. EMI SH behavior of different fabrics knitted with the combination of copper, silver and stainless steel [65].

Sharaf et al. produced conductive fabrics with the combined coating of polyaniline/CuO (PANI/CuO) where CuO nanoparticles were formed on cotton fabric via ultrasound-assisted template method. First sample was obtained by exposing polyaniline treated sample to CuO nanoparticles. The second sample was prepared by CuO treated cotton fabric undergoing polymerization reaction by aniline monomer. The third sample was obtained by treating cotton samples with PANI/CuO nano composite. The different sequence of treatments were found to affect the variation in the weight percent of CuO deposited on fabric, and therefore different electrical conductivity results as shown in Figure 14 [66].

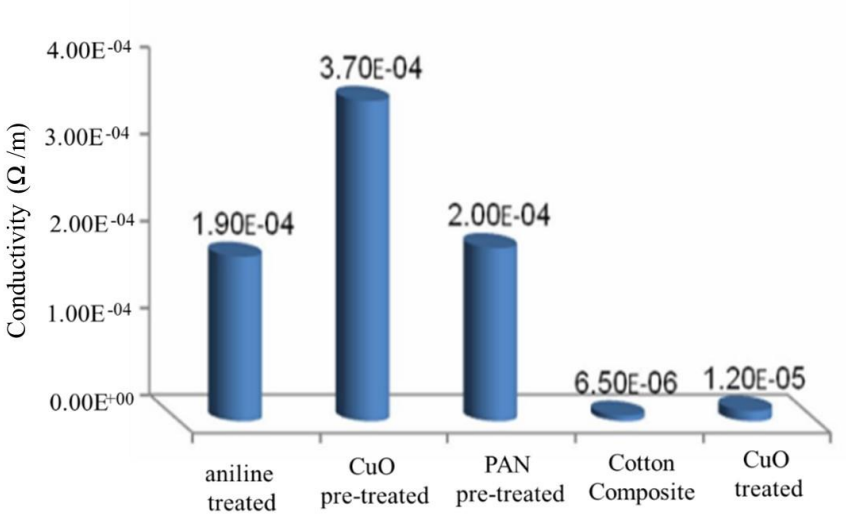


Figure 14. Electrical conductivity values of the cotton fabrics treated with different conductive coatings [66]

Xuqing et al. designed the durable flexible conductive PET fabrics by copper plating onto plasma treated PET substrate. From Figure 15, the electrical surface resistivity of PET fabrics was found to decrease with increase in plating time, reaching as low as 2 Ω/square at 60 min. This behavior was attributed to increased quantity of copper nanoparticles which formed more continuous copper films [67].

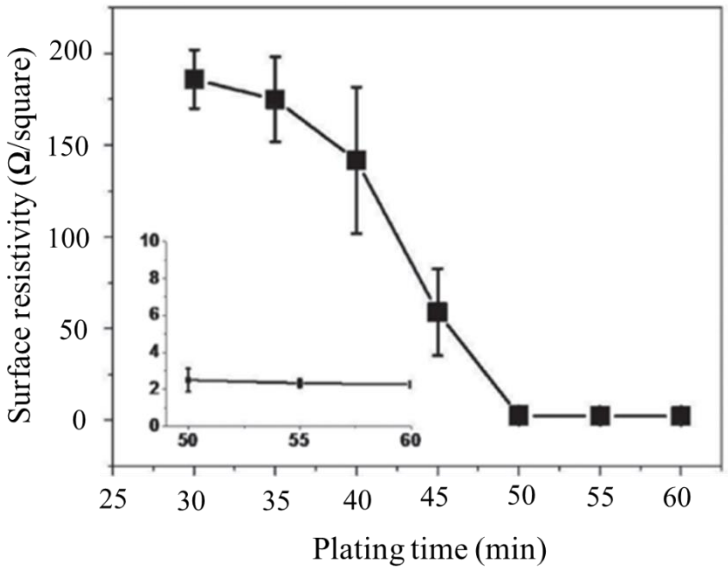


Figure 15. Decrease in electrical resistivity with increasing electroless plating time [67].

Hao et al. demonstrated a novel two-step method for fabricating silver-plated cotton fabrics with high electrical conductivity and excellent washing fastness. First, polydopamine (PDA) film was coated on the surface of cotton fabrics by in situ polymerization of dopamine. Subsequently, the conductive fabrics were prepared by dipping the previous fabrics in silver-ammonia ($[Ag(NH_3)_2]OH$) solution followed by reduction in glucose solution [68].The silver ions in silver nitrate solution were reduced by the catechol groups of polydopamine, and silver nanoparticles were combined with polydopamine to form covalent bond on the surface of cotton fabrics. The surface resistivity was found to decrease with increase of concentrations of silver-ammonia ($[Ag(NH_3)_2]OH$) solution from 0.1 to 0.25 mol/L when concentration of dopamine is a constant value. The surface resistivity has slight decreasing or increasing when the concentration of silver-ammonia varies from 0.2 to 0.25 mol/L and the concentration of dopamine varies from 1 to 6 g/L as shown in Figure 16. The surface resistivity of 0.12 ± 0.02 Ω/square. was measured at 0.2 mol/L concentration of silver-ammonia and 6 g/L concentration of dopamine [68].

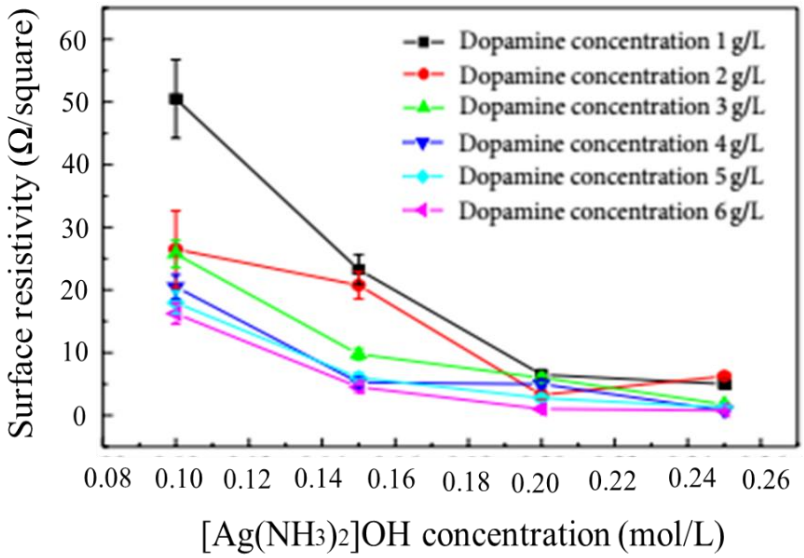


Figure 16. The electrical resistivity of silver-coated cotton fabrics pretreated with different concentrations of poly-dopamine film [68].

Tunakova et al. studied an effect of metal content, a placement of conductive yarn geometry against EMI shielding. It was confirmed in this study that electromagnetic shielding effectiveness increases with increasing metal fibre contents. Sample with the highest content of metal fiber reached the highest electromagnetic shielding effectiveness—50.5 dB for frequency 1,500 MHz, whereas sample containing the lowest portion of conductive component

displayed the lowest electromagnetic SE (13.16 dB for frequency 1,500 MHz [37]. The knitted structure containing 1% of metal fibre shown SE lower than 1dB, while knitted fabric made of hybrid yarn containing 20% of metal fibre shown SE about 10 dB. Figure 17 and 18 displayed the effect of the number of fabric layers (n) on the SE of woven fabrics, where SE increased linearly with the increase in the number of fabric layers [37].

Table 4: Electromagnetic shielding effectiveness of different structures of fabrics with different conductive components [37].

Material	EMI SE (dB)
Woven fabrics made of 100% cotton	0
Hybrid knitted fabric – 80% PP/20 % SS	10
Hybrid woven fabric-- 80% PP/20 % SS	14
Hybrid woven fabric-- 25% PP/75 % SS	50
Polypyrrole coated woven PES fabric	20
Woven fabrics made of 100% carbon	57
Copper coated PES woven fabrics	67
100% aluminium foil (30 g/m^2)	80

Here: ss is stainless steel, PP is polypropylene

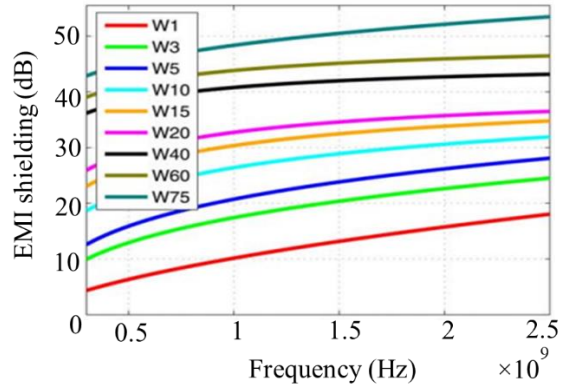


Figure 17. EMI SH of fabrics composed up of different metal contents [37]

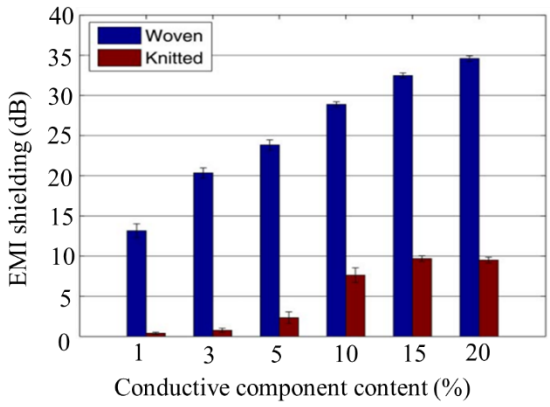


Figure 18. Comparison of EMI SH between woven and knitted structure fabrics [37]

3.6 Applications of electrically conductive metalized fabrics

3.6.1 Electrodes for TENs machine use for electrotherapy

Recently, electrotherapy (by using electric pulses) is increasingly being used in physiotherapy and rehabilitation to reduce pain, enhance healing, and improve the patient's mobility [69]. Transcutaneous electrical nerve stimulation (TENS) is a simple, non-invasive analgesic technique which is used widely in health-care settings by physiotherapists, nurses and midwives [70]. TENS is mainly used for the symptomatic management of acute and non-malignant chronic pain [71]. Transcutaneous electrical nerve stimulation (TENS) therapy is normally applied with conductive hydrogel electrodes to the treatment zone where body nerves are stimulated by electrical current [72]. Transcutaneous electrical nerve stimulation (TENS) treatment is rarely associated with negative side effects and has been reported to be effective in patients with peripheral neuropathic pain [73], e.g., patients with diabetic neuropathy [74], and patients with pain of differing origin but less effective in patients with central neuropathic pain [75].

Treatments through TENS are based on different action mechanisms, but the most commonly used one is gate control theory. The working principle of this theory can be seen in Figure 19. Gate control system is thought as responsible from activities of inhibitor interneurons which is placed in substantia gelatinosa. According to this theory, large a-alpha and a-beta nerves (responsible for pain) are stimulated by electrical stimulation. Thus, pain signals (run through the small nerves) that transmit pain to the brain are decreased. As a result, to perceive and differentiate the pain is prevented. Furthermore, small a-delta and C nerves open the spinal gate system and activate opioid system. So, endorphine hormone is secreted and patients feel better [32, 33].

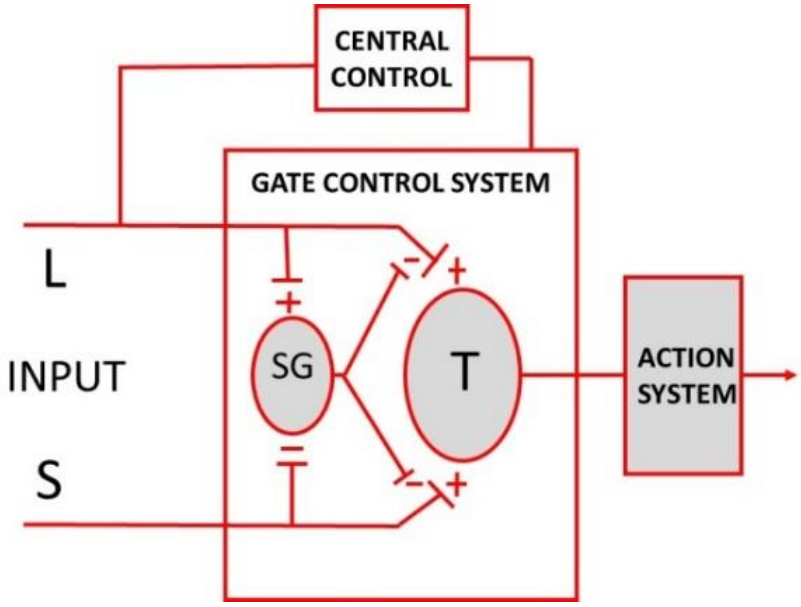


Figure 19. Schematic diagram of the gate control theory of pain mechanisms. L, the large diameter fibers; S, the small-diameter fibers; T, transmission cells; SG, substantia gelatinosa; +, excitation; -, inhibition [78].

In most cases, the widely used type of electrode for TENS applications is self-adhesive hydrogel electrode. There are a lot of disadvantages associated with self-adhesive electrodes because of their viscous structure like a feeling of discomfort, creating redness, rashes, sweat, unable to be washed, hard enough not compatible with body structure, less life and being not hygienic [11]. In some cases, the patient is recommended to use TENS for a long time, in this situation patients should be encouraged to use TENS whenever the pain is present. This is the situation for ongoing chronic pain, this may mean that patient use TENS over the entire day. Johnson et al. [79] reported a study of long-term users of TENS where 75% used TENS on a daily basis and 30% reported using TENS for more than 49 hours a week. When TENS is used continuously in this way it is seriously advised to the patient to monitor skin condition under the electrodes on a regular basis. So, they need to stop the operation of TENS over the skin after intervals. Hence their treatment is affected. Some patients report post-stimulation analgesia although the duration of this effect varies widely, lasting anywhere between 18 hours [80]. Due to long term application of TENS patients may experience skin irritation, skin maceration, swelling, reddening beneath or around the electrodes. This is commonly due to dermatitis at the site of contact with the electrodes resulting from the constituents of electrodes, electrode gel or adhesive tape [81]. It is also crucial that patients need to wash their body parts

after the application of gel electrodes. Therefore, the interest in manufacturing pad electrode materials with improved conductive and physical properties has grown.

3.6.2 Energy Harvesting (Tribo-electric nanogenerators)

The concept of energy harvesting is related to energy scavenging, where ambient energy present in the environment is converted into electrical energy. The basic principle is same as to the principle of large- scale renewable energy generation such as wind turbines, but the amount of energy produces in this manner (energy harvesting by using textile source) is much smaller. This can produce about tens of microwatts to a few watts. Even then, energy harvesting is very important as it is potential alternative to or batteries. Actually, batteries are source of providing less energy for daily use smart devices but they need to recharge and also problematic in case of dispose. Batteries are also not fit for smart textile applications as they need to remove before washing the textiles also they are hard and heavy in weight [82]. In these situations, there was strong need to develop portable, flexible, stretchable, soft and long-life energy harvesting devices which can convert working environment motion into power. The wearable energy harvesting device Triboelectric generators (TrEG) were implemented first time in 2012 to convert mechanical energy to electricity [83]. Furthermore, the utility of these generators is very important regarding low power wearable devices, which includes various field of applications such as medical device, sensors and activity monitoring sensors to fashion accessories. Triboelectric generators are able to harvest energy with various motions such as mechanical, wind, water movements or even with the motion of human source [84]. Previously, a lot of triboelectric generators has been made by the combination of variety of materials and technologies like vacuum-based, foil based [85], iterative fiber-drawing process [86], and film casting method so on[87]. Film casting TrEG was made by laminating the stretchable conductive fabric with polydimethylsiloxane (PDMS), while inner layer was again made of non-stretchable polyurethane (PU) composite [87]. Researchers have been developing several TrEG with special techniques but they were in drawbacks due to manufacturing methods, requiring special processing techniques for example vacuum-based manufacturing[88]. Most of them were limited to generate energy based on rubbing and pressing based, but not stretchable [53-55]. Several conductive fiber based TrEG have also been reported [90]. The another study contains turboelectric generators made from carbon nanotubes coated fabric but it was not stretchable [91]. Recently, stretchable TrEG were fabricated using serpentine-patterned electrodes and a wavy-structured Kapton film but they were limited in stretchability up to 22% due to the conventional planar structure design [92].

3.7 Comparative performance of copper and silver

Among famous noble metals the copper and silver are found in native or "elemental" form at the earth's surface. The both metals are lustrous, delightful, and good resistance to corrosion. They are ductile, soft and capable of drawn into thin wires. Therefore, easily found into nanowires and particles. Furthermore, nanocoating of these metals do not affect the drape and bending property of materials. Copper was the predominant early metal, especially after the technique of smelting it from its ores and trade made it widely available. It is still the third most important industrial metal, after iron and aluminium [47].

Copper is predominant early metal often described as a "red" metal having density 8.94 g/cm^3 with atomic number 29 and atomic weight 63.57. Copper having valences +1 (cuprous) and +2 (cupric), with abundantly found in (cupric) +2. Its naturally-occurring isotopes have mass numbers 63 (69%) and 65 (31%). Generally, cupric salts are easily produced by auto-oxidation of cuprous salt as shown in Equation 1.

$$2\,Cu^{+} \rightarrow Cu + Cu^{++} \tag{1}$$

While silver is quite resistant to corrosion but is easily tarnished by materials containing sulphur, such as eggs, rubber and mustard. Silver has excellent resistance against oxidation. However, tarnished silver contains only black Ag_2S. The tarnish can be removed easily by rubbing Ag_2S against sodium bicarbonate, $NaHCO_3$, paste [93, 94]. Generally, each metal has the capability of corroding in different environmental conditions. Surface corrosion is negligible but most dangerous to thin layer of coating. Usually, measured in the loss of thickness per year. This type of corrosion is predominately for copper coated substrates with general corrosion rate 0.02mm/year. This type of corrosion can be avoided by coating the thick layer of copper on the material. Pitting is another type of corrosion can be occurred on protective layer, where copper coating is damaged by cyclic bending of textile or substrate. Furthermore, pitting is assisted by surface contaminations having copper alloys or sulphide pollution on copper coated textiles. Moreover, another factor reinforced corrosion is the cracking of coated copper layer on surface. Copper-nickel and copper silver alloys are the best to avoid from this type of corrosion. Erosion corrosion is one more type of corrosion forms on copper alloys and is sufficient to damage it. It is mostly happened in the case of nanoparticles coting and more beneficial to provide antibacterial properties.

Both metals (copper and silver) kill a broad spectrum of bacteria. The word broad spectrum represents all families of bacterial strains including Gram-positive and Gram-

negative bacteria. Silver metal even in nano form is nontoxic until silver ions are released. Silver ions are released in wet conditions and cause to damage the cell wall. Silver antimicrobial activity is more effective in moist environment at slightly higher temperature than room temperature. In contrast to this copper is able to show antimicrobial behavior at all temperatures and humidity level. This distinction makes the copper coating more important since the antimicrobial activity must be possible for a typical room temperature at hospital. This kind of unique behavior of copper is due to its two ionic states. Although, copper can penetrate integument in humans and animals. Experiments were done against inflammatory response created by therapeutic effect [95]. Another experiment was done to measure the skin-identical complex, bis(glycinato) copper(II). The experiment was done on cat skin [96]. Electron micrographs of skin revealed the copper stained over the exposed area. Furthermore, 47ppm copper was found by atomic absorption analysis. Electrical conductivity of silver is higher than all other metals. The copper comes next to silver and then gold and aluminium. The reason for higher electrical conductivity of silver, copper and gold is due to their unique electron configuration. The full d-shell orbital of first three elements, where S1 in outer most shell make them most conductive. According to quantum free electron theory, the conductivity in metals depends on Fermi sphere surface area. The electrical conductivity is higher for higher surface area of fermi sphere. The fermi surface area for silver metal is higher than all [97, 98]. The comparison between electrical resistivity, EMI shielding and antibacterial properties of copper and silver nanoparticles coated fabrics was done in a single study. In all properties, both fabrics (copper and silver nanoparticles coated) showed the insignificant difference [47].

3.8 In-situ deposition of particles

In-situ deposition of particles has been using in vast area of applications including technical textiles, bio-medical engineering, environmental protection, electronics, and advanced materials etc. While discussing about the textiles, the technology has focused on producing the (different shapes and size) nanostructures over the fabrics for improvement in different functional properties like moisture management in textiles, oil and dirt repellence, water repellence, improving dyeability, comfort level, flame retardancy and now also in smart textiles to produce electrically conductive fabrics etc. [89, 90]. The term in-situ deposition elaborate the deposition of the metal in an appropriate position. This procedure refers the deposition or directly growth of particles during their formation in the bath. Substrate or fabric is simply dipped into the salt solution and ions tends towards the fabric. Subsequently, these ions are directly reduced on the surface of fabric and convert to metal particles. In previous

studies, metal particles were developed in solution by sol-gel method and then were coated on substrates. This method has various drawbacks regarding, agglomeration, clusters, uneven coating and also needs for binder. Furthermore, enveloped metal particles coated fabrics cannot provide proper threshold to conduct electric current. In situ deposition is fast, simple and efficient method for depositing metal particles. In-situ deposition method is efficient to evenly and controlled distribution of particles over the substrate. In situ coating of particles tend to be thin and less dense deposition than other costly and heavy metal depositing techniques such as chemical and physical vapor deposition. The usefulness of in-situ deposition of metal particles is limited to situations [91, 92].

3.9 Electroless plating

The term "electroless plating (ELP)" can be defined as the deposition of continuous film or coating of metals over the substrate by chemical reduction method, without an external current source[103]. In general, no current source is required because of self-catalyzing or autocatalytic ability of this method. During the 19 century, ELP was done by reduction of silver using aldehyde agents. Afterward, the tremendous progress started in this field during the 20 century. When researchers started to combine different metals and alloys for plating without external current source. Since then, the technique has been studied widely and emerge in different fields such as, aerospace, electronics, automotive, biomedical and even in textile metallization.

Textile metallization by electroless deposition produced a thin, uniform and compact layer of metal over the surface. During this procedure role of reducing agent and its selection is very important. Electroless plating is of two types. Type one is displacement deposition electroless plating and type two is autocatalytic deposition electroless plating. During the displacement deposition, an active metal-coated fabric is immersed into metal salt solution containing less active metallic ions ($M1^+$). As a result, the following reaction takes place.

$$M1^+ + e^- \rightarrow M1 \qquad (2)$$

The metal *M1* is deposited on the substrate and forms a continuous compact film. Different combination can be adopted in this reaction Ag/Zn, Au/Ag, Au/Ni, Cu/Fe. There are many examples of this reaction, Ag/Zn, Au/Ni, Au/Ag, Cu/Zn, Cu/Al,, Pt/Fe, Pd/Ni, Pt/Co [104].

During the autocatalytic process, less active metals are unable to perform the autocatalytic deposition. In this procedure of metal deposition selection of reducing agent is

very important. For selection of proper reducing agent, it should have high standard reduction potential as compared to the metals (copper and silver) being reduced. For instance, the reduction potentials of some important reducing agents like hypophosphite (-1.57V), formaldehyde (-1.30 V) can be useful as they are higher compared to the reduction potentials of copper +0.34V and silver +0.80V [105]. The another important property of reducing agent is self catalytic reaction ability which can help in deposition of maximum copper on target metal (fabric coated with copper particles) [106]. Once initiated this reaction the deposition of metal starts too quickly not only on the surface of substrate but also in whole volume.

Some major advantages of electroless plating are given below [19].

- Very easy to apply, flexible and is relatively suitable for all types of materials.
- Electroless plating is not health hazardous like other techniques such as physical vapor deposition, chemical vapor deposition and electroplating.
- It also save the cost and time of plating.
- It allows the uniform thickness of metal layer coating.

3.10 Research question/Gap

Research gaps were found after an extensive literature review shown above. According to the knowledge following research gaps were identified.

In-situ deposition of metal particles on woven and stretchable fabric for produce the flexible, stretchable sensors and electrodes (was here selected). Hence better replacement of coating the particles and nano/microwires of metals with binder [102]. Furthermore, searchability of fabric is also affected if binder is used to attach particles over the fabric surface.

Copper and silver particles coated fabrics should have good anti-bacterial properties and ohmic heating. Due to antibacterial effect, resistive heating, flexibility and searchability the fabrics will be better replacement for the carbon rubber-based hard electrodes (use in electrotherapy) [11].

Developed stretchable electrodes would be easily applied for long time over the skin of patient, and avoid from discomfort, redness, rashes and sweat, which were the frequent problems occur by self-adhesive carbon electrodes [12, 79].

In addition, fabric based electrodes can be washed and reused, while adhesive carbon based electrodes cannot use for more than three times [81]. Furthermore, it is compulsory and irritant to apply adhesive gel each time over the skin to attach the rubber electrodes.

Formaldehyde based reducing agents have been reported in many studies to develop the copper and silver particles. Formaldehyde has severed disadvantages on health such as carcinogen and irritant to skin, so cannot use for electrotherapy or in other medical applications [107]. Some more studies are available for another famous reducing agent $NaBH_4$. Sodium borohydride is extra active agent and give fast reduction. $NaBH_4$ provide a lot of H^+ ions and fast nucleation of metal ions, which tends to the formation of clusters and uneven coating [47]. Moreover, the coat of $NaBH_4$ is too high than sodium hydrosulphite and reduction potential is almost the same. Hence, glucose and sodium hydrosulphite are safe to use and better replacement of formaldehyde and sodium borohydride [108].

Moreover, glucose and sodium hydrosulphite reducing agents provide low pH, low cost and relative safety. Low pH is attractive for better nucleation of metallic ions, which tends to produce small size metal particles and avoid from aggregation [109]. While in basic pH there is need of alkali and complexing agents, which cause Metal hydroxide/oxide ions and produce uneven coating with large size agglomerated particles [101,102].

In situ deposition of particles provide thin layer of coating and resultant fabric will be light in weight as compared to heavy metal coating techniques such as physical vapour deposition and chemical vapour deposition etc.

Furthermore, by dip-dry-reduction method, we are directly growing/reducing the particles over the fabric surface. Therefore, eliminated the problems of cluster and agglomeration which are more common in solution or sol-gel method [112].

Purpose for in-situ deposition is to provide the better base for further electroless/electroplating of metals. So, in this way we have eliminated a number of pre-treatments, activation and sensitization steps, which are normally required to proceed further metal plating. Whereas conductive inks, conductive polymers and carbon-based materials provide in inadequate base and are not suitable form hygienic point for the development of electrodes (use in medical applications).

Surface activation with nanoparticles is more stable and provide better base for further electroless plating. Because nanoparticles cover more surface area and also penetrate into the fabric structure properly, to provide the volume conductivity. Furthermore, we are using copper and silver nanoparticles. Who has positive redox potential than copper which is going to be deposit. Actually, if we do use any base particles which has negative redox potential than copper depositing. Then the host element will replace by copper. So, the base will not give the

homogeneous ground and will cause for the thickness variation for copper plating, in turn, electrical resistivity will also affect [113].

Copper electroless deposition on nano copper and silver particles is also better regarding comfort properties (drape, thickness and stiffness).

After deposition of silver and copper particles the fabric is ready for copper electroless plating. This method of plating is focused on less cost and environmental friendly (palladium free, stannous free and formaldehyde free) copper plating over cotton fabric [113].

A lot of worked has been done for copper plating over, stannous, cobalt and lead. Which cannot be used for hygienic applications. As tin (Sn) is hazardous and irritant to skin and persons with existing skin disorders may be more susceptible to the effects of these agents[14]. In existing methods copper and silver particles base for the deposition of further copper plating also confirmed the antibacterial activity of developed fabrics. Fabrics can be used for environmental friendly applications like electrodes for (TENs) machine, west for military application where we need electromagnetic shielding and hygienic west in case of injury of sliders.

During electroless plating by conventional method we need to use hydrochloric acid with stannous chloride which in turn effect the strength of cotton fabric. Instead of it, we are performing the electroless plating in alkaline bath using sodium hydroxide. Therefore, a study is describing more favourable deposition of copper in alkaline pH [13].

For selection of proper reducing agent, it should have high standard reduction potential as compared to the metals (copper and silver) being reduced. For instance, the reduction potentials of some important reducing agents like hypophosphite (-1.57V), formaldehyde (-1.30 V) can be useful as they are higher compared to the reduction potentials of copper +0.34V and silver +0.80V [105]. The another important property of reducing agent is self catalytic reaction ability which can help in deposition of maximum copper on target metal (fabric coated with copper particles) [106].

Researchers have been using formaldehyde and hypophosphite as a reducing agent during electroplating, which has serious disadvantages. Formaldehyde has severed disadvantages when use for electroplating. It is carcinogenic, irritant to skin, give harmful vapor etc. While using hypophosphite is not proper for copper, because copper is not a good catalyst for the oxidation of hypophosphite. So, in result give not proper plating over copper.

So to overcome these issues glyoxalic acid appeared as a best alternative, provide more stable, higher deposition with very less environmental pollution [114,115].

Electroless plating method by using glyoxalic acid is more environmentally friendly and not give off harmful vapours.

The main advantage of performing the electroless plating over already copper and silver particle coated fabric is to achieve very low electrical resistivity with high EMI shielding because silver and copper particles coated base is already conductive.

4. METHODOLOGY

Two types of fabrics were used for metallization. One is plain-woven standard bleached cotton fabrics with weave structure (EPI × PPI = 28 × 23, warp and weft count = 23s Ne, GSM = 150 g m^{-2}) having thickness 0.35 mm were used. Calculated volume porosity is 72%. The woven fabric was purchased from Licolor, a, s. Figure 20 is showing the macroscopic image of plain-woven cotton fabric taken by ProgRes CT3 macroscope.

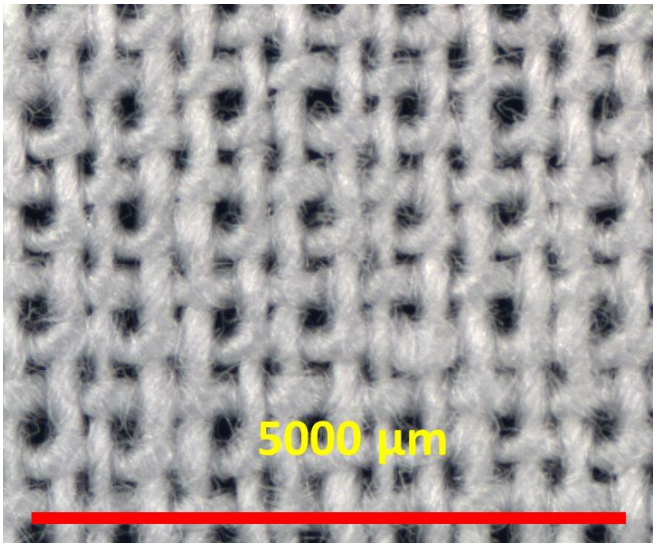


Figure 20: Microscopic image of plain-woven cotton fabric

The second fabric is a special type of knitted textile mostly used for medical therapy. This knitted fabric is composed up of cotton-Nylon 66–Lycra (80:15:5) with areal density - GSM 110 g/m^2 and construction of WPC × CPC = 10 × 9 (26 Tex, 20 Denier, 70 Denier), having thickness about 0.85 mm. The knit type of the substrate was 1×1 laid-in full knit selected on the basis of being more stable compact structure. Normally, the loop structure and nature (inherent extension) of material will give the extension due to available yarn length in loop. Moreover, Lycra will also assist in extension as well as to recover back to the original dimensions, as the recovery of Lycra is excellent. This type of knitted fabric is composed up of one loop of main cotton yarn, second loop of air covered nylon yarn and inlaid spandex as a plating yarn. The Figure 21 (a-c) are used to further elaborate the knitted structure. The fabric was obtained from Research and development laboratory, Interloop, Faisalabad.

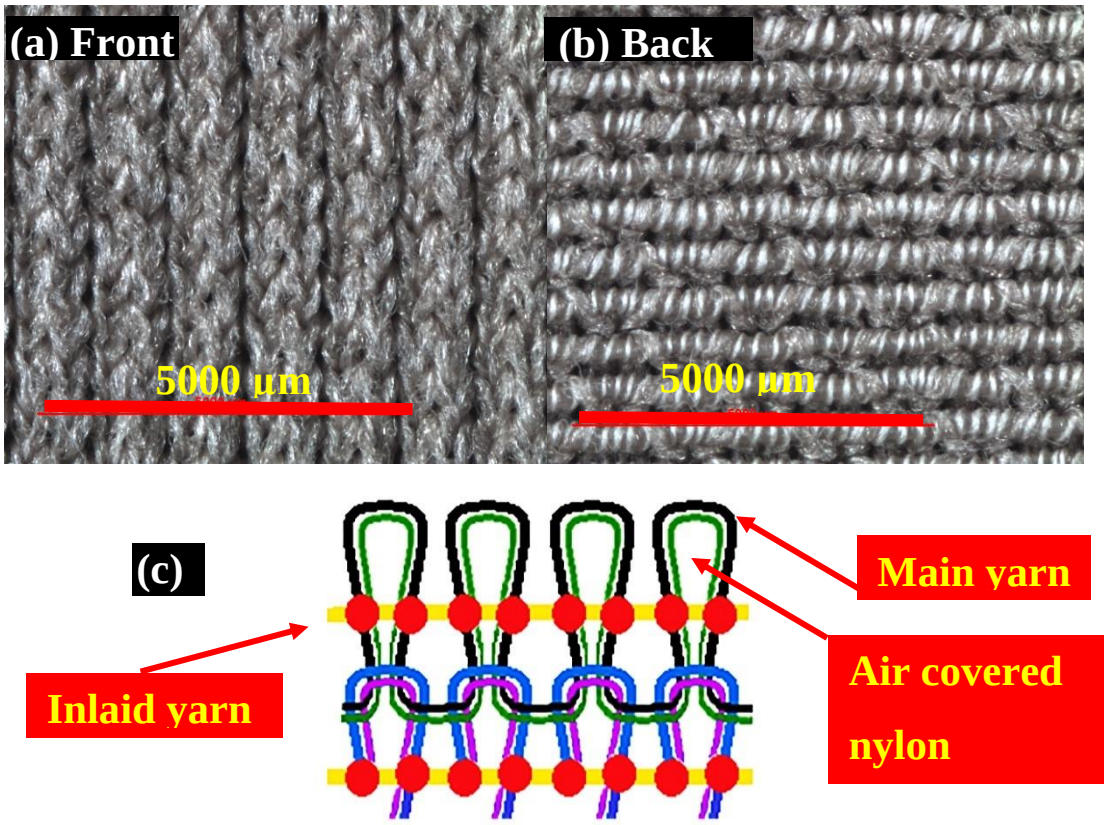


Figure 21.Surface structure of knitted fabric (a) front side, (b) back side (c) schematic

The chemicals used for metallization were of reagent grade from Sigma Aldrich. The rabbit fur (specie) was obtained from veterinary science department of rabbit farm, during the hot season from Pakistan (no rabbit was killed to obtained the fur). The silicon rubber was purchased from Wacker, Germany.

The experimental part is composed up of four parts.

- First part, is the development of copper particles on plain woven cotton fabric.
- Second part, is the deposition of silver particles over plain woven cotton fabric and deposition of silver particles on stretchable knitted fabric (to develop the electrodes for TENs machine for electrotherapy.
- Third part is related to further copper electro-less plating on previous two developed (Cu particles coated woven fabrics and Ag particles coated woven fabrics) fabrics.
- Part fourth is the silver electroplating over knitted fabric (to develop the energy harvesting device).

4.1 Deposition of copper particles on textiles

These experiments are done to incorporate the copper particles over the plain woven cotton fabrics. Copper sulfate was used as the base material along with sodium hydrosulfite as reducing agent for in situ deposition of copper particles on cotton fabrics. At first, different concentrations of copper sulfate from 30 g/L, 20 g/L and 10 g/L were prepared by dissolution in distilled water. Then, the cotton fabric was dipped 10 times in the solution and dried at 100 °C for 3 minutes. This procedure of dipping and drying was continuously carried out up to 150 dips. The dwell time of fabric in solution was about 30 seconds against each dip. Subsequently, (after each 10 number of dips) the treated fabrics were transferred to the 30 g/L sodium hydrosulfite solution. Hence we made 15 samples against each copper sulfate concentration. The concentration of sodium hydrosulfite was determined based on the concentration of copper sulfate. It should be always higher or at least equal to copper sulfate concentration. The reduction was continued for the duration of 20 minutes. The duration of reduction treatment was determined based on change in color of cotton. The cotton fibers changed their color from blue to blackish gray after 15 minutes of reaction with reducing agent. So 20 minutes of reduction treatment was allowed to complete the reaction. Schematic for the deposition of copper particles is shown in Figure 22.

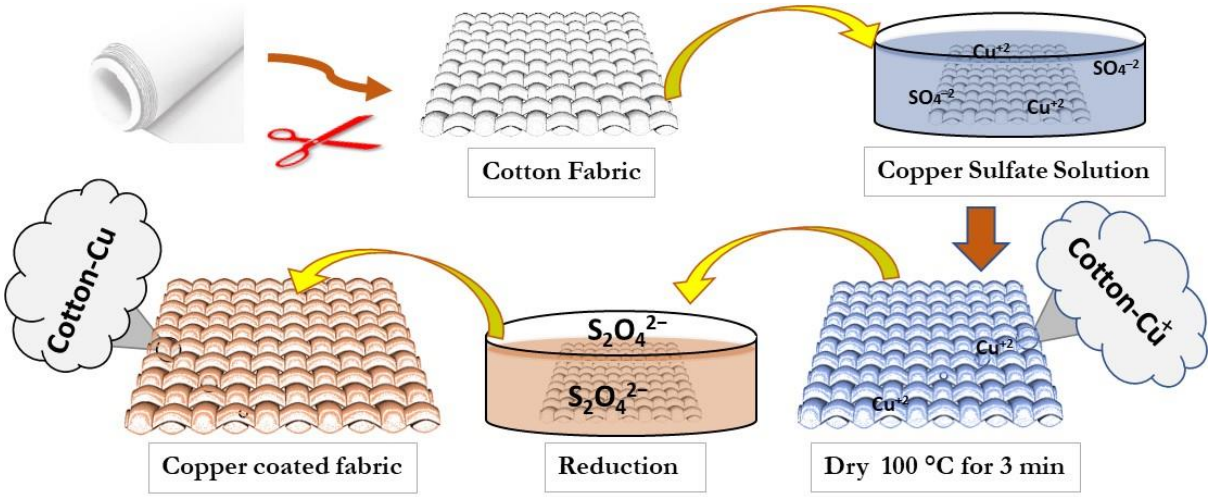


Figure 22. Schematic for the deposition of copper particles

Amount of coated copper was evaluated by weighting or original and treated fabric.

4.2 Deposition of silver particles on textiles

Silver particles were deposited on plain-woven cotton fabric and also on stretchable knitted fabric.

4.2.1 Silver particles on cotton woven fabric. The cotton fabrics were dip in 10 wt % aqueous sodium hydroxide (NaOH) solution at room temperature for 12 then washed with distilled water. At first, three different concentrations of silver salt ($AgNO_3$) from 51 g/L, 34 g/L and 17 g/L were prepared by dissolution in distilled water. Then, the aqua ammonia (28 wt %) was added dropwise into aqueous solutions of silver nitrate ($AgNO_3$) and stirred continuously until a transparent solution of $[Ag(NH_3)_2]^+$ was obtained. The cotton fabric was dipped in this solution for 30 seconds and dried at 100 °C for 3 minutes. To deposit the maximum concentration of $[Ag(NH_3)_2]^+$ on the fabric the (dip and dry) process was repeated a number of times. Hence, this procedure of dipping and drying was continuously carried out up to 150 dips. Subsequently, the treated fabrics were transferred to 18 g/L of glucose (reducing agent) solution. The reaction of reduction was allowed to proceed for 15 minutes. Finally, the fabrics were rinsed with distilled water and dried in air. Schematic for the deposition of silver particles is shown in Figure 23.

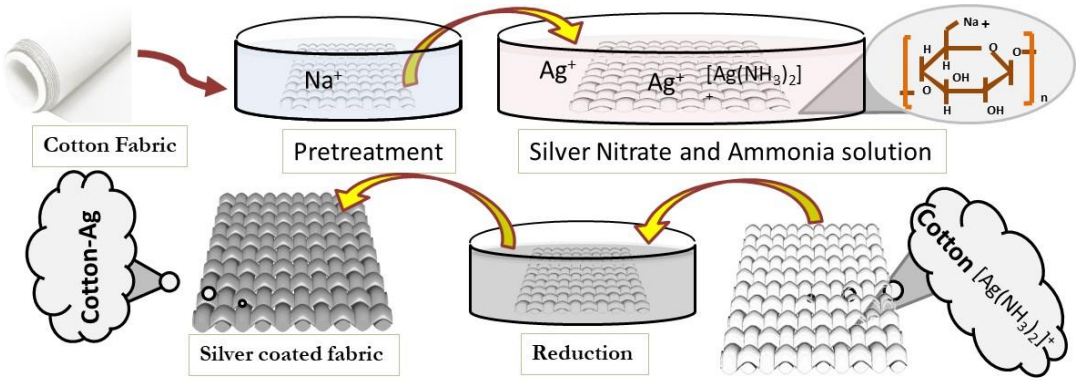


Figure 23: Schematic for the deposition of silver particles

Amount of coated silver was evaluated by weighting or original and treated fabric.

4.2.2 Silver particles on blended knitted fabric. Pre-treatment of fabric via local oxidation was carried out by dipping fabric into 1 % (w/w) $KMnO_4$ and 10 g/l sodium chloride (NaCl) for 25 min in an ultrasonic bath. This treatment was done to enhance the carbonyl groups on the nylon 66 fibers. The fabric was rinsed thoroughly with distilled water and dried at 60 °C. The fabric was then treated with 12 wt % aqueous NaOH solution at room temperature

for 10 min and rinsed with distilled water. Meanwhile, three different concentrated solutions (170 g/L, 85 g/L and 42.5 g/L) of silver nitrate ($AgNO_3$) were prepared and the aqua ammonia (28 wt %) was added dropwise to get the transparent solution of $(Ag(NH_3)_2)^+$. Later, alkali treated fabrics were dipped in each solution for 10 minutes and dried at 90 °C for 10 minutes. The dipping and drying process was repeated for 10 times to deposit the maximum concentration of $(Ag(NH_3)_2)^+$and Ag^+ ions on the fabrics. Towards the end, the dipped fabrics were immersed into 54 g/L glucose stock solution, and the reaction was allowed to progress for 15 min. The silver coated fabrics were then rinsed with distilled water, dried in air and post cured at 100 °C in the oven for 10 minutes.

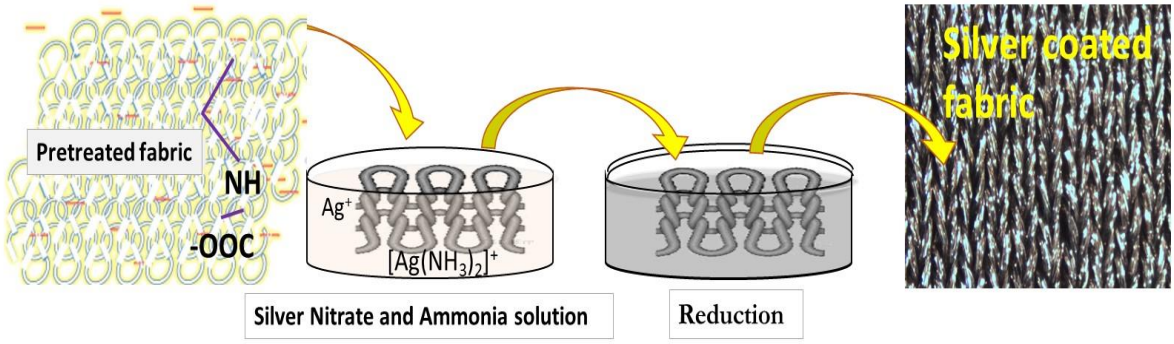


Figure 24: Schematic for the deposition of silver particles on knitted fabric

Amount of coated silver was evaluated by weighting or original and treated fabric.

4.3 Electroless plating of copper over previously coated fabrics

Based on previous experiments, the surface activation of fabric was carried out using 10 g/L of copper sulphate for deposition of copper particles and 17 g/L of silver nitrate for deposition of silver particles. Then, the copper and silver particles coated fabric samples were immersed in the electroless copper plating bath at room temperature for different intervals of time. The bath composed of 30 g/L $CuSO_4$-$5H_2O$, 20 g/L glyoxylic acid as reducing agent (50% aqueous solution), EDTA as complexing agent and 12 g/L sodium hydroxide. The concentration of EDTA was adjusted according to the concentration of copper sulphate in the ratio of 1:1 to avoid the formation of complexes with substrate metal (Ag or Cu). After plating the fabric was cleaned with deionized water and dried in oven at 85°C. The schematic mechanism of copper plating over silver and copper particles coated fabrics can be seen in Figures 25.

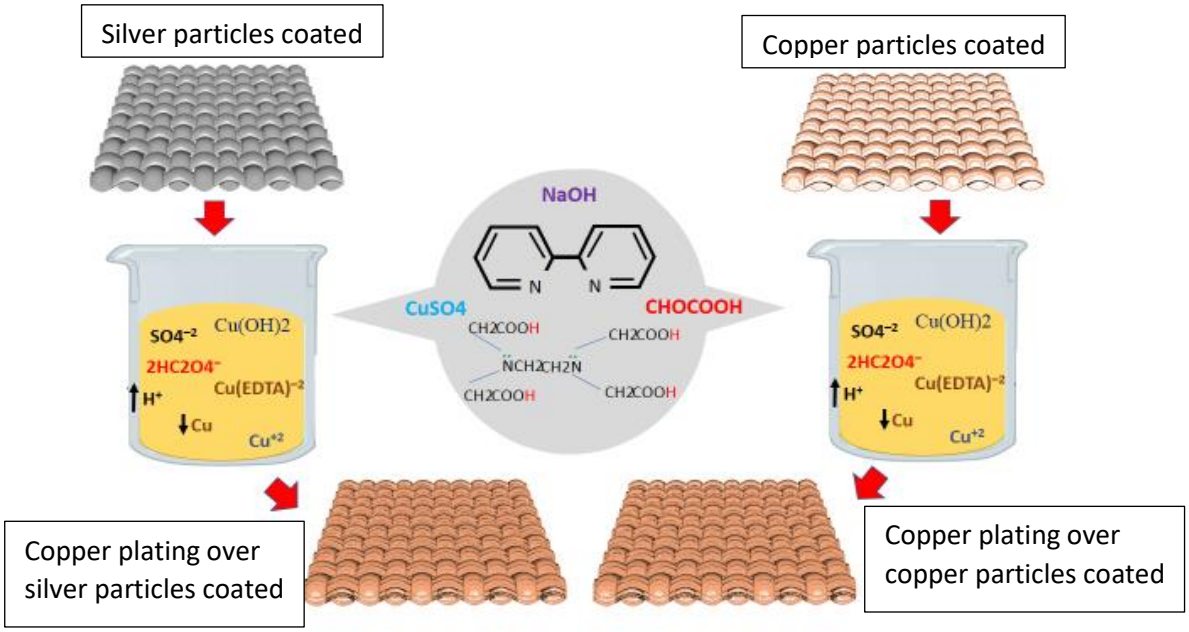


Figure 25: Copper electroless plating over silver and copper particles coated fabrics

Amount of coated metals was evaluated by weighting or original and treated fabric.

4.4 Plating of silver over stretchable fabrics for the development of TrNG

Cotton-Nylon–spandex (80:15:5) stretch fabrics were used as a substrate to produce the conductive fabrics. The conductive fabrics were made by the deposition of copper nanoparticles followed by silver electroplating. At first, the fabrics were treated with 15 wt % aqueous NaOH solution at room temperature for 10 min and rinsed with distilled water. Then 10 g/L of copper sulphate was prepared by dissolution in distilled water, blue colour was appeared. The alkali treated fabric was dip and dry several times in solution at 60°C, with a time interval of 30 minutes. The treated fabric was then transferred to the 30 g/L sodium hydrosulphite solution. The reduction was continued for the duration of 20 minutes. It is necessary, fabric should be conductive and act as a catalyst to locate conductive material on it. The purpose of silver electroplating was to produce compact layer of metal on substrate. Copper nanoparticles covered fabric was used as a substrate. To perform the electroplating, 5 gram of Silver nitrate ($AgNO_3$) was dissolved in 1 liter of distilled water. Electrolytic power source was rated at 10V/1-2 Amp. A constant current was maintained throughout the complete electroplating. Anode was connected with silver rod acting as source of silver metal while cathode was connected with conductive fabric (Cu-NPs coated) to deposit the silver metal. It was made sure that solution continuously circulate the electroplating bath. The whole setup is also shown in Figure 26.

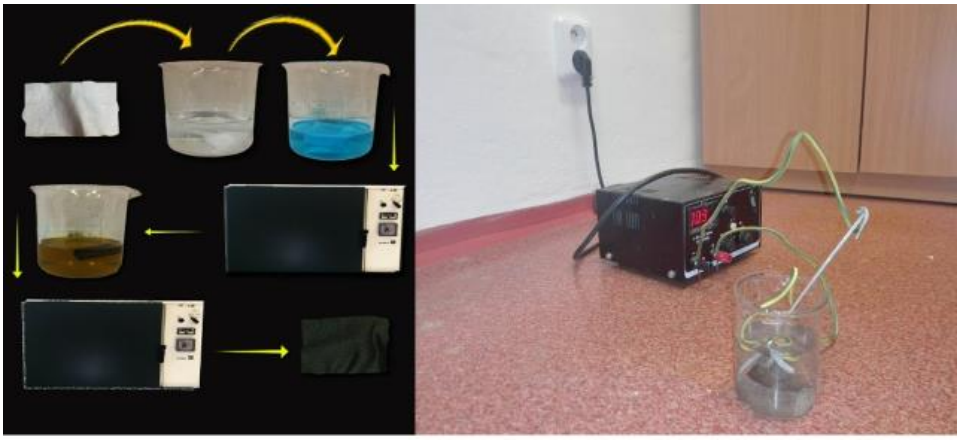


Figure 26: Set up for (a) CuNPs deposition (b) For silver plating

The displacement reaction will occur here

$$Cu + 2AgNO_3 \rightarrow Cu(NO_3)_2 + 2Ag \quad (3)$$

For fabrication of triboelectric generator, the rabbit fur and silicon rubber were employed as two triboelectric layers due to their high stretch ability, softness and different electron affinities. Rabbit fur is positive dielectric while the silicon rubber is negative dielectric. They were placed facing one another as illustrated in Figure 27. Furthermore, the silver-plated fabrics were used as electrodes around these triboelectric layers. The developed TrEG was designed to generate the energy under both stretching and pressing modes.

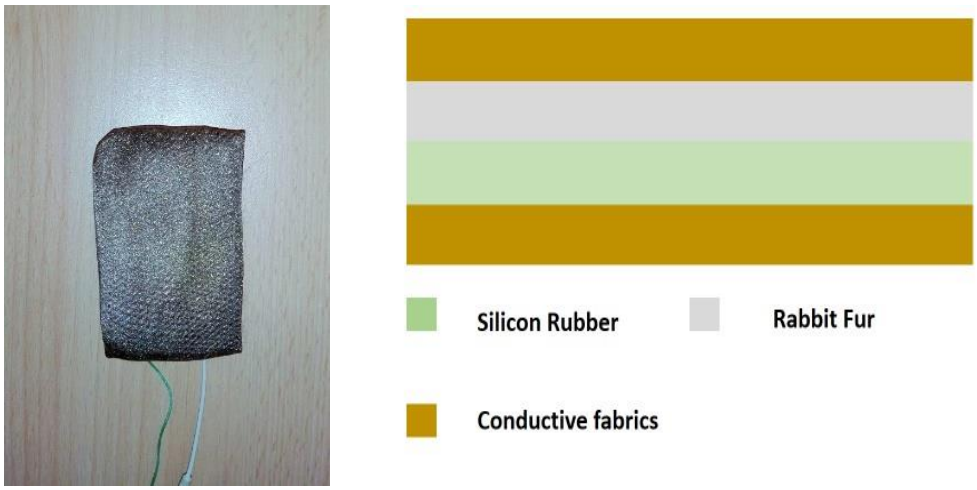


Figure 27: Composition of developed triboelectric generator

4.5 Surface morphology testing

Scanning electron microscope (SEM) was used to observe the surface structure of coatings. An accelerated voltage of 10 kV was applied by Tescan VEGA III TS5130 SEM apparatus. EDX analysis was performed to measure the elemental percentage by weight. XRD analysis was performed with a diffractometer equipped with a conventional X-ray tube Cu Ka1 radiation (1.54 A˚) power condition (40 kV/30 mA). The XRD pattern was measured in the 2θ range 10–80° with the step size of 0.02°.

4.6 Electrical conductivity testing

Volume and surface resistivity of the sample set was measured according to the standard ASTM D257-07, under the 100 V DC power supply, using concentric electrodes (see Figure 20, pressure 2.3 kPa was applied), at the temperature T = 21°C and the relative humidity RH = 54 % using an air-conditioned room. Samples were placed to the air-conditioned room 24 h prior to testing. Volume resistance is measured by applying a voltage potential across the opposite sides of the sample and measuring the resultant current through the sample. Volume resistivity, ρ_V (Ω mm) was calculated from the Equation 4:

$$\rho_v = R_v \left(\frac{S}{t}\right) \tag{4}$$

where R_V [Ω] is the reading of volume resistance, t is the thickness of the fabric (mm), S is the surface area of the electrode (mm^2) ($\pi D_2^2/4$), D_2 is the inner diameter of the outer ring electrode (mm).

Surface resistance is measured by applying a voltage potential between two electrodes of specified configuration which are in contact with the same side of the tested material. Surface resistivity ρ_S (Ω/square) was calculated from the Equation 5 [116].

$$\rho_s = R_s \left(\frac{\pi D_0}{g}\right) \tag{5}$$

where, R_S (Ω) is the reading of surface resistance, D_0 is ($D_2 - D_1$)/2, D_1 is the outer diameter of the centre electrode (mm), D_2 is the inner diameter of the outer ring electrode (mm), g is the distance between D_1 and D_2 (mm).

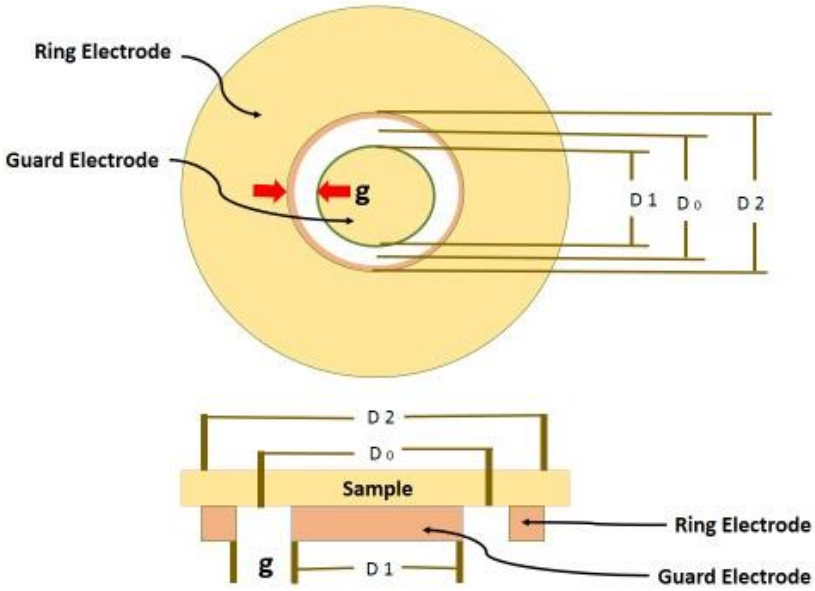


Figure 28: Scheme and dimensions of concentric electrodes used for measurement of surface and volume resistance, where D1 = 50.4 mm, D2 = 69 mm

4.7 Electromagnetic shielding testing

It was measured by coaxial transmission line method using insertion loss principle according to standard ASTM D4935-10 over frequency range of 30 MHz to 1.5 GHz. The measurement set-up consisted of a sample holder with its input and output connected to the network analyzer. A shielding effectiveness test fixture (Electro-Metrics, Inc., model EM-2107A) was used to hold the sample. The network analyzer (Rohde & Schwarz ZN3) was used to generate and receive the electromagnetic signals. The ratio between transmitted to incident power of the electromagnetic waves was calculated to express the effectiveness of EMI shielding (*SE*) in dB as depicted in Equation 6.

$$SE\ (dB) = 10\ log \frac{P_t}{P_i} \tag{6}$$

Where, P_t and P_i is power density (W/m^2) measured in presence of sample (transmitted), and without the sample (incident) respectively.

4.8 Antibacterial testing

The zone of inhibition test (AATCC 147) was conducted to confirm the antibacterial property of metal-coated fabrics [117]. The zone of inhibition is a clear area of interrupted growth underneath and along sides of the test material and indicates the bioactivity of specimen. It is a qualitative test for the bacteriostatic activity by the diffusion of antibacterial agent through agar. The bacterial strains of Gram-negative Escherichia coli (CCM 3954) and

Gram-positive Staphylococcus aureus (CCM 3953) used in this study were obtained from the Czech Collection of Microorganisms, Masaryk University Brno, Czech Republic. Bacterial suspensions were always prepared fresh by growing a single colony overnight at 37 °C in a nutrient broth. All agar plates were freshly prepared before the antibacterial tests.

4.9 Weight Gain

During the electroless plating the weight gain percentage was examined according to the following Equation 7.

$$w = \frac{m - m_0}{m_0} \times 100 \quad (7)$$

where *m* is final mass, m_0 is original mass of substrate and *w* is the total weight gain percentage.

4.10 Heating performance of conductive fabrics

The heating performance was observed for all conductive fabric samples by FLIR thermo-camera. Change in temperature on the fabric surface was measured by applying the voltage difference across its ends. Different voltages were applied at different interval of times and amount of heat generated is related to I^2 as given in equation 8.

$$P = I^2 R \quad (8)$$

where *P* denotes the total power dissipation, R denotes the resistance of the operating heater and *I* denotes the current passing through conductive samples.

4.11 Tensile properties

The TIRA test machine was used to measure the tensile properties stretchable fabrics as per ISO 13934-1 standard. The fabric strips were cut according to standard dimension (20 cm in length, 4 cm in width). The fabric strips were clamped between the jaws of tensile testing machine. The distance between the jaws was 10 cm. The experiment was rated with constant speed of 100mm/min.

4.12 Durability testing

The washing durability of metal coated fabrics was studied to have an idea of their activity in service. It was examined according to ISO 105-C01 by vigorously stirring the metal coated fabrics in 5 g/L standard detergent with the liquor ratio of 50:1. Sample was rinsed at 40 °C with stirring speed of 800 rpm for 30 minutes. After washing, all samples were dried and conditioned in a standard atmosphere (65 % humidity; 25 ^{o}C) for 24 hours before testing. Later,

the performance was verified based on measurement of electrical conductivity and SEM observation of metal particles on the fabric surface.

5 RESULTS AND DISCUSSIONS

5.1 Deposition of copper particles on textiles

5.1.1 Electrical conductivity

The effect of copper sulfate concentration and number of dips was investigated for electrical resistivity of copper-coated plain woven textiles. The development of electrical conductivity was verified by flow of electric current. It is clear from Figure 29 that the higher concentration of copper sulfate solution resulted in higher electrical resistivity of coated fabric samples. This behavior can be attributed to the formation of big sized copper particles at higher concentration of copper sulfate solution. Surprisingly, lower concentration 10 g/L of copper sulfate produced more conductive fabrics due to formation of percolated network by creation of continuous connectivity between the small sized copper particles. This can be further justified from SEM images shown in Figure 30 (a-b), where formation of more percolated network of smaller particles can be found in case of 10 g/L than 20 g/L copper sulfate pentahydrate concentration. Therefore, the action of agitation or ultrasonication for disrupting the nucleation of copper particles is good topic for further research in order to obtain more percolated network at higher copper sulfate concentration. Furthermore, the electrical resistivity was found to reduce with increase in number of dips for all concentrations of copper sulfate solution. This indicated more dense and uniform deposits of copper particles at higher number of dips. This can be further justified from SEM images shown in Figure 31. As lower concentration of copper sulfate provided acceptable results for electrical conductivity, so in further sections, detailed discussion is made on samples coated with 10 g/L of copper sulfate.

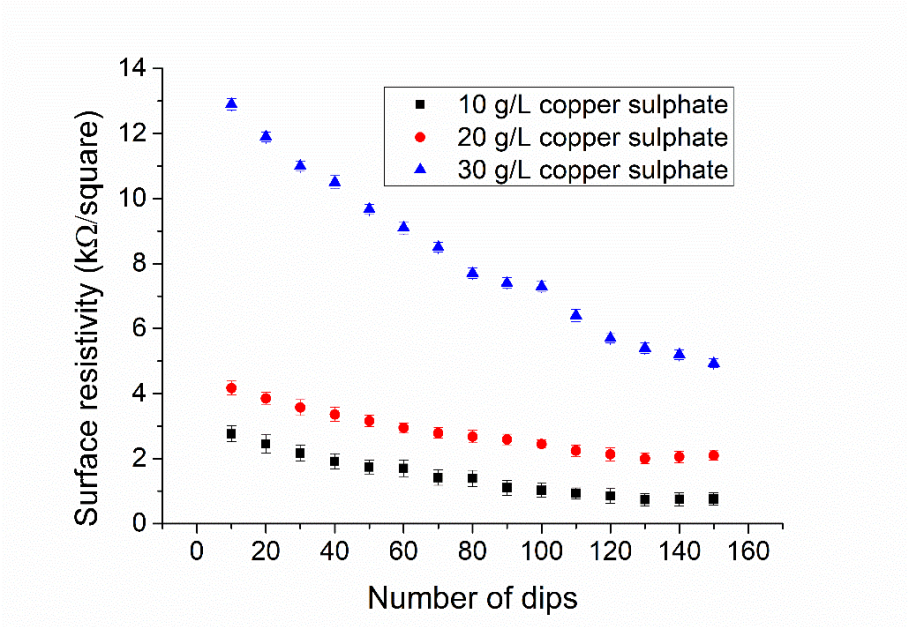
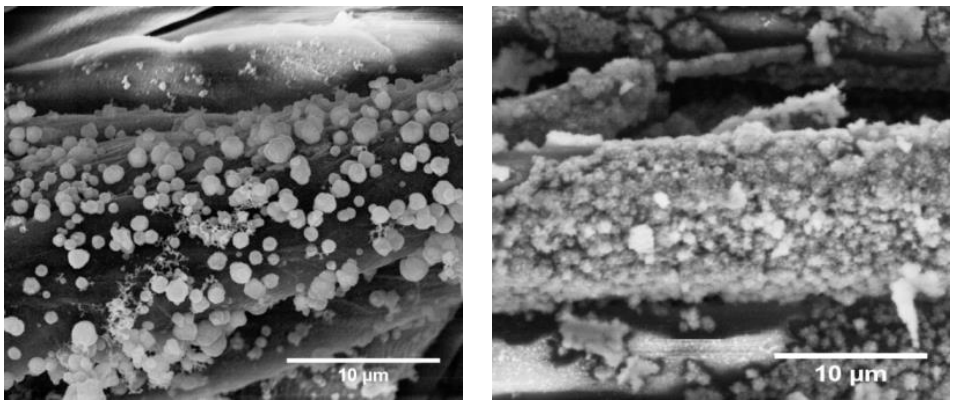


Figure 29: Effect of copper sulphate concentration and dipping on electrical resistivity (bars are limits of 95 % confidence interval)



(a) 20 g/L (b) 10 g/L

Figure 30: SEM image of copper coated fabrics for different copper sulfate concentration

5.1.2 SEM analysis

The scanning electron microscopy was employed to observe the deposition of copper on fabric surface. The SEM micrographs with inset images at a higher magnification shown in Figure 31 revealed the nano/micrometer scale of copper particles deposited on fabric surface. With increase in number of dips, the deposition of copper was found more uniform and denser. This further indicated the higher tendency of formation of percolated network of copper particles when number of dips increased. Table 5 shows the elemental composition of copper

coated fabrics determined by EDX analysis. It clearly showed the increase in contents of copper with higher number of dips.

Table 5: Elemental composition of copper coated cotton fabrics of 10 g/L copper sulfate

Wt. %	C	O	Si	Ca	Cu	Total
50 dips	53.06	38.83	2.33	0.94	4.84	100.00
100 dips	52.48	38.24	2.58	1.25	5.45	100.00
150 dips	52.84	37.63	2.63	0.83	6.08	100.00

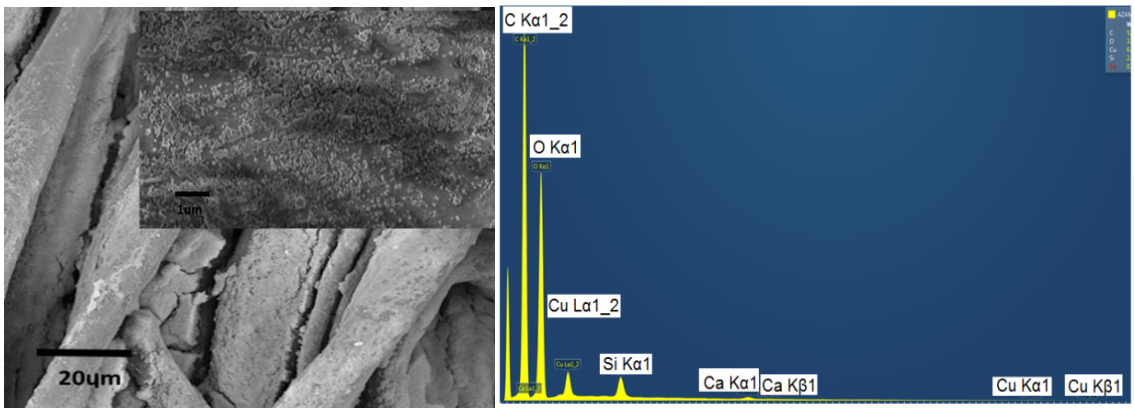


Figure 31: SEM image with EDX spectra for copper coated cotton fabrics at 150 dips

5.1.3 XRD analysis

The XRD analysis was carried out to know the phase composition of deposited copper particles. The XRD spectra of the cotton fibers dipped and dried in the copper solution prior to reduction treatment are not given because it will only show the peaks of cotton due to absence of crystal structure of copper. The XRD analysis was performed on copper coated cotton fibers after reduction step. Figure 32 shows the XRD patterns of samples for the 2θ range of 10 to 80 degrees with a step of 0.02 degree. The phase purity of the prepared copper particles can be clearly seen from perfect indexing of all the diffraction peaks to the copper structure. The diffraction peaks appeared at 2θ of 43.3 º, 50.5 º, and 74.2 º represented (1 1 1), (2 0 0) and (2 2 0) planes of copper, respectively [3]. The crystalline nature of copper particles was confirmed from the sharp peak, whereas the broadening of the peaks indicated the formation of nanoscale copper particles [118].

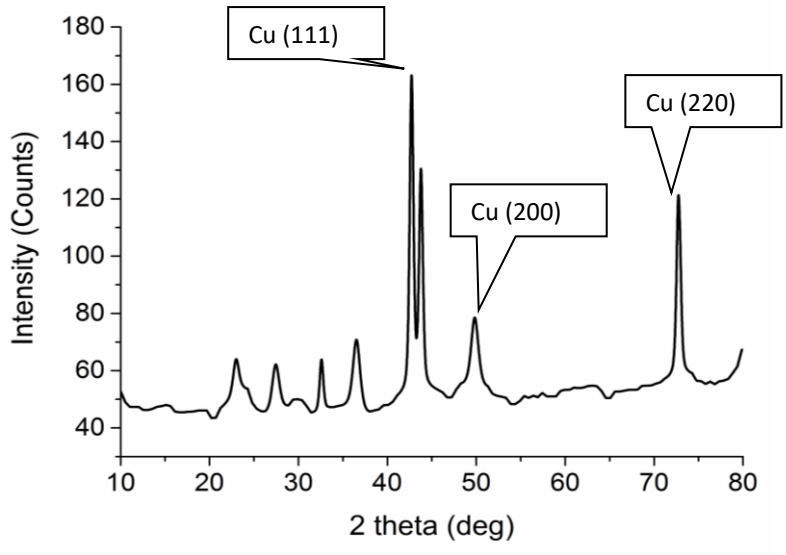
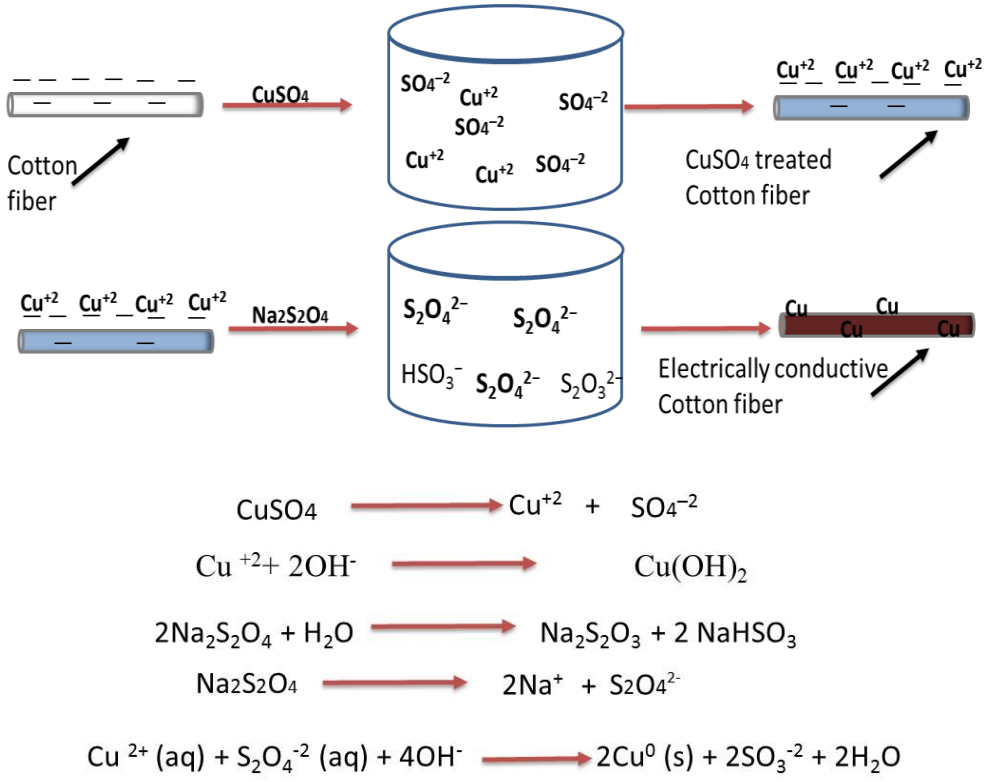


Figure 32:XRD patterns for copper coated cotton fabrics

5.1.4 Mechanism

The mechanism of attachment of copper on cotton fabric surface can be explained from the schematic diagram shown in Figure 33. The dissolution of $CuSO_4$ in water resulted into Cu^{+2} and SO_4^{-2} ions. Subsequently, copper (II) ions adsorbed on the surface of cotton fibers based on heterogeneity (voids or pores) of the cellulose phases in the fabric [119].

Due to enrichment of anionic cotton substrate, further uptake of copper ions continued. This resulted in formation of ionic bond between the copper (II) and negative groups available on cotton surface. In similar way, sodium hydrosulphite $Na_2S_2O_4$ dissolved in water to give reducing agent dithionite ion ($S_2O_4^{2-}$). Later, when cotton fabric was dipped in aqueous $Na_2S_2O_4$ solution, the redox reaction occurred between oxidizing Cu^{+2} ions and reducing $S_2O_4^{-2}$ ions. This ultimately reduced Cu^{2+} to Cu^{+}, and possibly to copper metal. Moreover, the presence of few sulphur ions produced insoluble and conductive copper sulphide layer [120]. The sulphur was not detected in EDX analysis given in Table 5 because it was present in small quantity and a few spots. The color of cotton changed from white to blue after treatment with copper sulfate solution, and then from blue to dark brown after treatment with reducing agent sodium hydrosulfite.



$$CuSO_4 \longrightarrow Cu^{+2} + SO_4^{-2}$$

$$Cu^{+2} + 2OH^- \longrightarrow Cu(OH)_2$$

$$2Na_2S_2O_4 + H_2O \longrightarrow Na_2S_2O_3 + 2\,NaHSO_3$$

$$Na_2S_2O_4 \longrightarrow 2Na^+ + S_2O_4^{2-}$$

$$Cu^{2+}(aq) + S_2O_4^{-2}(aq) + 4OH^- \longrightarrow 2Cu^0(s) + 2SO_3^{-2} + 2H_2O$$

Figure 33: Mechanism of copper deposition on cotton fabrics

5.1.5 Electromagnetic shielding of copper particles coated fabric

Figure 34 shows the results of shielding effectiveness for the cotton fabric samples coated from 10 g/L of copper sulphate solution after 50, 100 and 150 dips. The number of dips showed significant effect on shielding effectiveness. The samples showed increase in shielding effectiveness with more number of dips in copper sulphate solution. The sample produced from 50 dips revealed the lowest electromagnetic shielding effectiveness of about 6 dB in frequency range of 600 MHz–1.5 GHz. On the other hand, the sample produced from 100 and 150 dips exhibited the maximum shielding ability of 10 dB and 13 dB, respectively. This behavior was attributed to increased reflection of EM waves due to formation of dense, uniform and percolated network of conductive copper particles with higher number of dips [121]. This can be further explained by SEM images shown in Figure 31, where dense network of copper particles can be found for more number of dips.

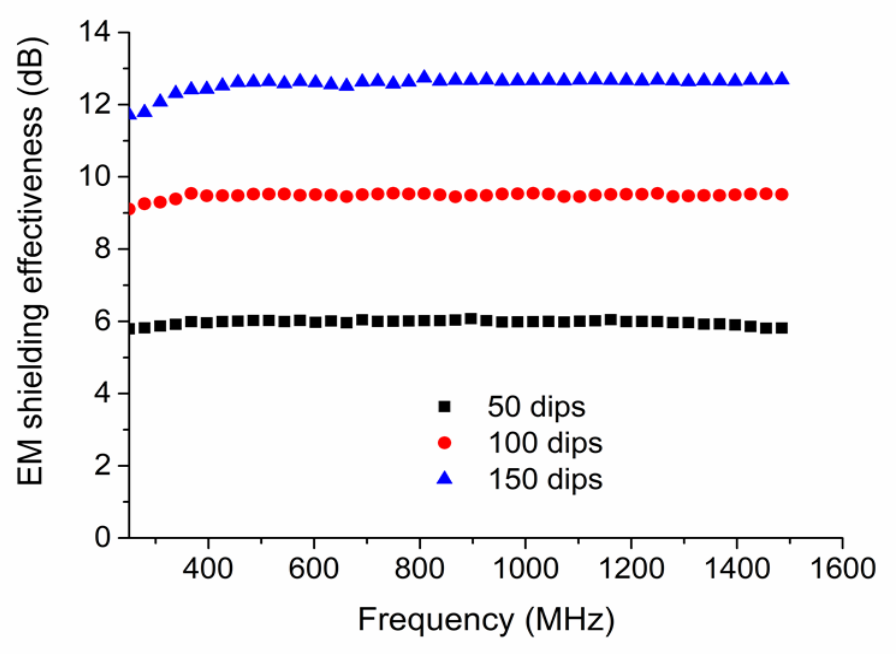


Figure 34:Shielding effectiveness of copper coated cotton fabrics

5.1.6 Antibacterial properties

The anti-bacterial property is important because EMI shielding fabrics are excellent media for microorganism growth, particularly when used in hospitals or working environments with unhealthy indoor air quality [122]. The anti-bacterial properties are studied in this work to find suitable applications of coated fabrics as personal protective clothing in hospitals. The antibacterial activity of copper coated fabrics was tested against Gram-negative Escherichia coli and Gram-positive Staphylococcus aureus. The test was repeated three times and the average value of zone of inhibition presented in Figure 35. The virgin cotton fabric without copper coating showed no antibacterial activity. However, the zone of inhibitions was evidenced against both type of bacteria Staphylococcus aureus and Escherichia coli after the copper coating. Furthermore, Staphylococcus aureus depicted the highest sensitivity as compared to Escherichia coli. The zone of inhibitions for Staphylococcus aureus increased from 9.5 to 15.5 mm, while for Escherichia coli it increased from 7.5 to 12 mm with increasing number of dips as shown in Figure 36. The antibacterial property of coated fabrics can be attributed to the combination of chemical and physical interactions of bacteria with copper particles[123,124].

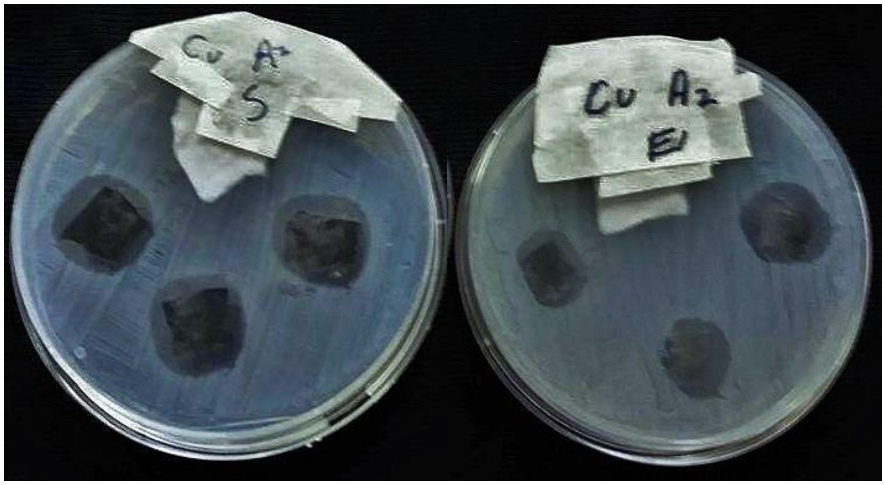


Figure 35: Zone of inhibition for copper coated cotton fabrics

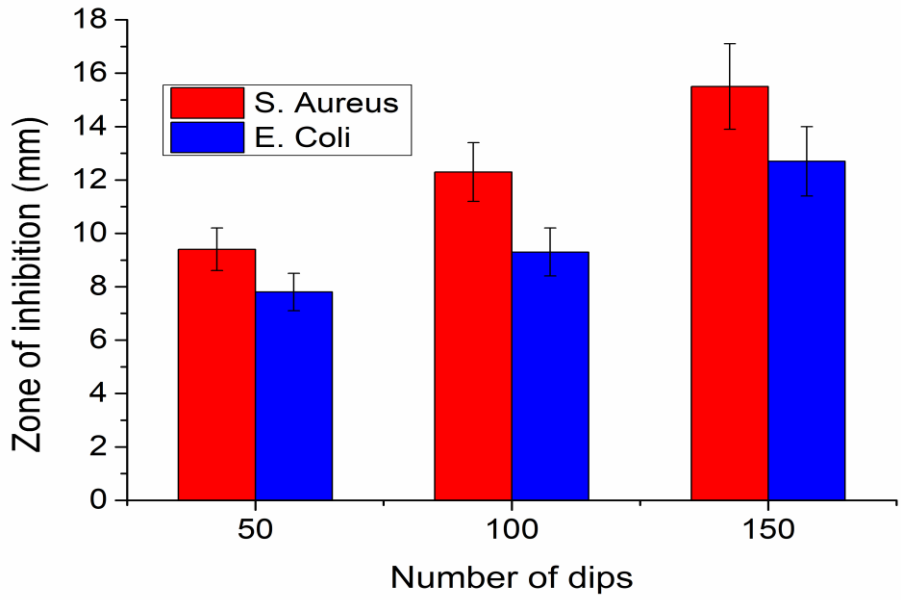


Figure 36: Values of inhibition zone against different number of dips. (bars are limits of 95 % confidence interval)

5.1.7 Durability

When the intake of copper exceeds the range of biological tolerance, it can cause adverse effects, including hemolysis, gastrointestinal distress, liver and kidney damage in humans [125]. Therefore, the removal of copper particles and their durability was verified against washing. The electrical resistivity of samples was measured before and after washing

as shown in Figure 37. There was no significant difference in change in conductivity of fabrics before and after washing. Therefore, similar EMI shielding properties can be expected for the fabrics after washing. Furthermore, no significant increase in the resistivity of the fabrics was found before and after washing. This indicated efficient working of firmly fix the copper particles on cotton fabric surface without deterioration of electrical conductivity. The SEM images shown in Figure 38 also confirmed the presence of copper particles on fabric surface after washing. This indicated strong attachment of copper particles with fabric surface and therefore reduced toxicity in routine applications.

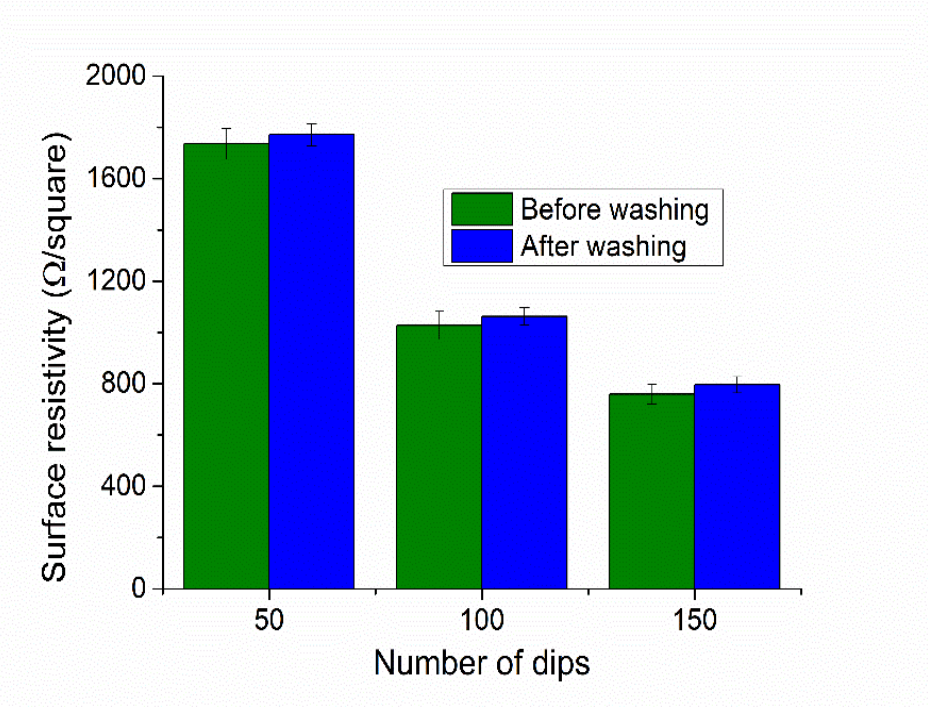
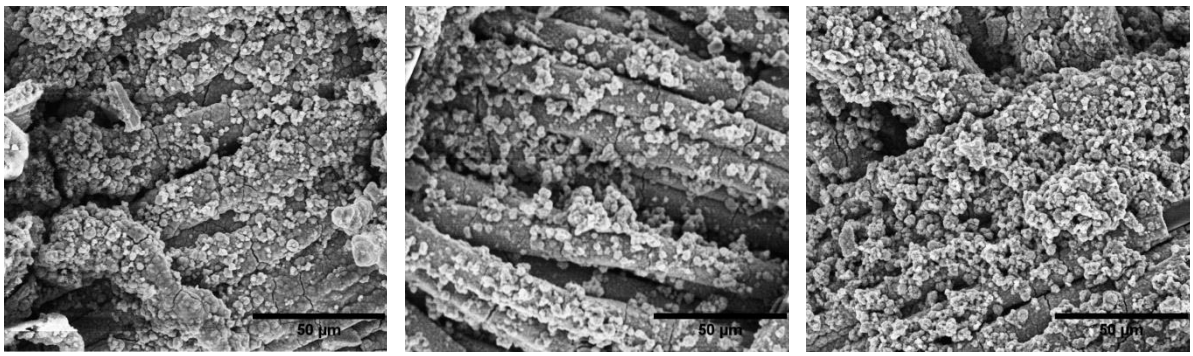


Figure 37: The electrical resistivity of samples before and after washing (bars are limits of 95 % confidence interval)



(a) 50 dips (b) 100 dips (c) 150 dips

Figure 38: SEM image of copper coated cotton fabrics after washing

5.1.8 Oxidation of copper-coated fabrics

Copper coated textile was suitable because of low cost and electrical properties. The electrical properties of copper are almost near to silver. But the universal problem regarding oxidation make the use of copper limited in some area of applications. The copper particles especially in range from nano to micro are most susceptible to oxidation and gets greenish-black (it is not colour of CuO but mixture of carbonate and hydroxide). That is why developed copper particles coated conductive fabrics were put in standard atmosphere (65% RH, 20 ± 2 °C) for a number of days. It was observed that up to 50 days the electrical resistivity remains almost the same. After 50 days the process of oxidation become expedite and we observed the reduction in electrical conductivity over time as shown in Figure 39. So, the developed electrodes from these fabrics were not remain suitable for the use in transcutaneous electrical nerve stimulation (TENs) device. Therefore, further research was aimed at coating of silver particles. Moreover, electroless copper plating was performed to enhance the ageing properties of copper coated textile.

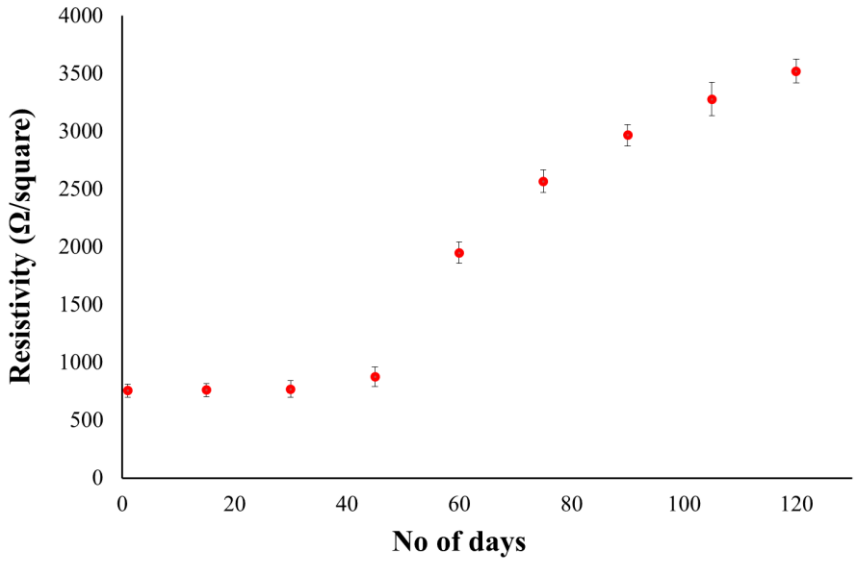


Figure 39: Reduction in electrical resistivity due to oxidation with the passage of days (bars are limits of 95 % confidence interval)

5.2 Deposition of silver particles on textiles

The results are composed up of two sections. At first, results are described for silver particles coated woven fabrics. Secondly, results for silver particles coated stretchable fabric electrodes.

5.2.1 Silver particles coated woven fabric

5.2.1.1 Electrical conductivity

The untreated cotton fabric is electrically insulating material. However, the silver coated cotton fabrics were supposed to be electrically conductive. The conductivity of the fabrics was expected to increase with increasing deposition of the nanoparticles. This hypothesis was confirmed by the electrical conductivity tests performed on the developed conductive fabric samples. It is clear from Figure 40 that the higher concentration of silver nitrate solution caused to increase electrical resistivity of coated fabric samples. This behaviour can be attributed to the formation of big sized silver particles at higher concentration of silver nitrate solution. The lower concentration (i.e. 17 g/L) of silver nitrate produced more conductive fabrics due to formation of percolated network by creation of continuous connectivity between the small sized silver particles. Furthermore, the electrical resistivity was found to reduce with increase in number of dips for all concentrations of silver nitrate solution. This indicated more dense and uniform deposits of silver particles at higher number of dips. This behavior can be further justified from SEM images shown in Figure 41. As lower concentration of silver nitrate provided acceptable results for electrical conductivity, so in further sections, detailed discussion is made on samples coated with 17 g/L of silver nitrate.

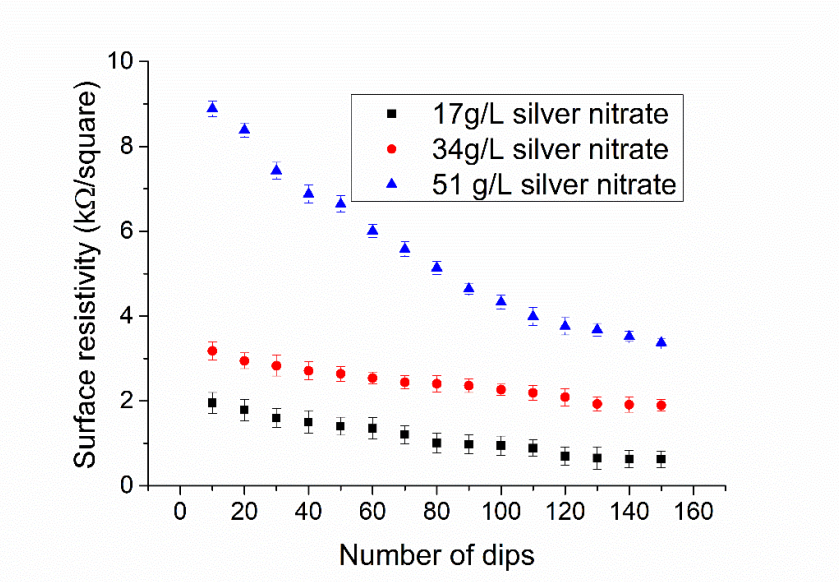


Figure 40: Effect of silver nitrate concentration and dipping on electrical resistivity (bars are limits of 95 % confidence interval)

5.2.1.2 SEM analysis of silver particles coated textile

The scanning electron microscopy was employed to observe the deposition of silver particles on fabric surface. The SEM images shown in Figure 41 depict the nanometer scale of silver particles deposited on fabric surface. With increase in number of dips, the deposition of silver was found more uniform and denser. This further indicated the higher tendency of formation of percolated network of silver particles when number of dips increased. The elemental compositions of silver coated fabrics determined by EDX analysis are shown in Table 6. It clearly showed the increase in contents of silver with higher number of dips.

Table 6: Elemental composition of silver coated cotton fabrics of 17 g/L silver nitrate

Wt. %	C	K	Cl	Ag	Total
50 dips	61.01	36.60	-	2.39	100.00
100 dips	51.46	43.07	0.28	5.19	100.00
150 dips	51.74	37.59	-	10.67	100.00

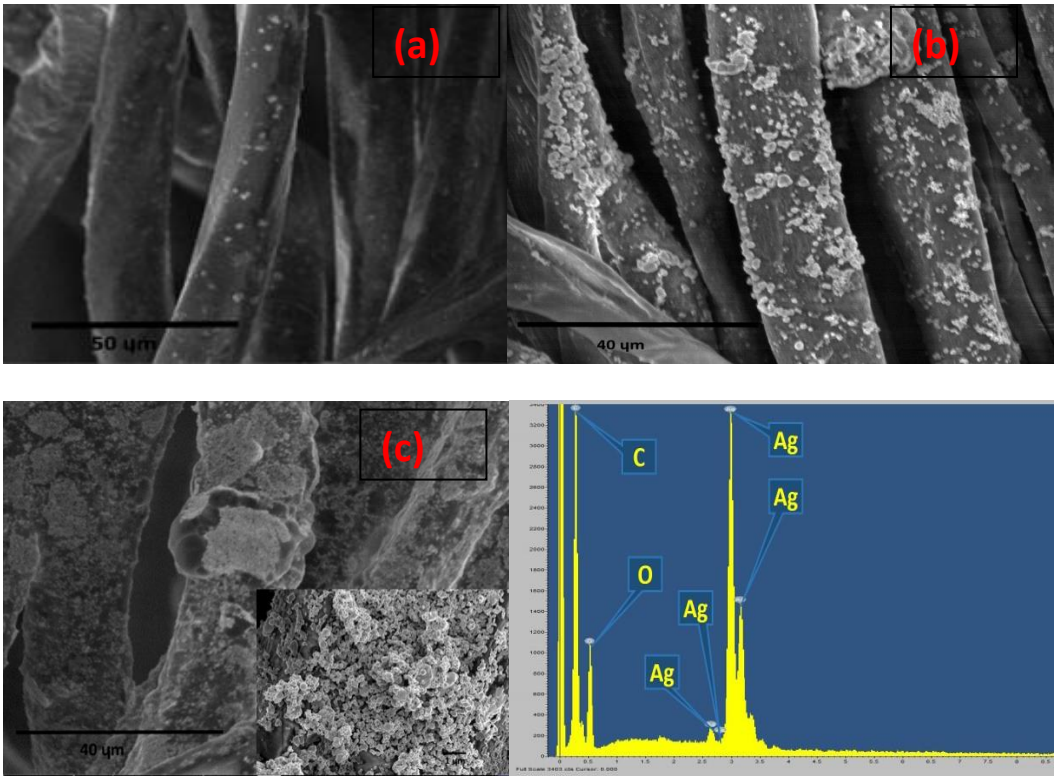


Figure 41: SEM image with EDS spectra for silver coated fabric (a) 50 (b) 100 (c) 150 dips

5.2.1.3 Mechanism

The mechanism of attachment of silver on cotton fabric surface can be explained from the schematic diagram shown in Figure 42. The dissolution of $AgNO_3$ in water resulted into Ag^+ and NO_3^- ions (see 40 (a). Subsequently, the cotton fibers absorbed the silver ions to some extent based on heterogeneity of the cellulose phases in the fabric [119]. Ammonia also forms the complex ion $[Ag(NH_3)_2]^+$ with Ag+ through the equilibrium reaction (see 40 (c)) and this ion could also act as the oxidizing agent to form Ag°(see 40 (d)). Due to enrichment of anionic cotton substrate, further uptake of silver ions and complex ion $[Ag(NH_3)_2]^+$ resulted in formation of a strong ionic bond between the Ag^+ and negative groups available on cotton surface. In similar way, the reducing agents could be any organic substance which further reduces the silver ions and complex ion $[Ag(NH_3)_2]^+$ into Ag° means possibly to silver metal (see 40 (d)). Each reaction is governed by its rate constant. It is further assumed that Ag° atoms are produced first and then they combine to form nanoparticles [126].

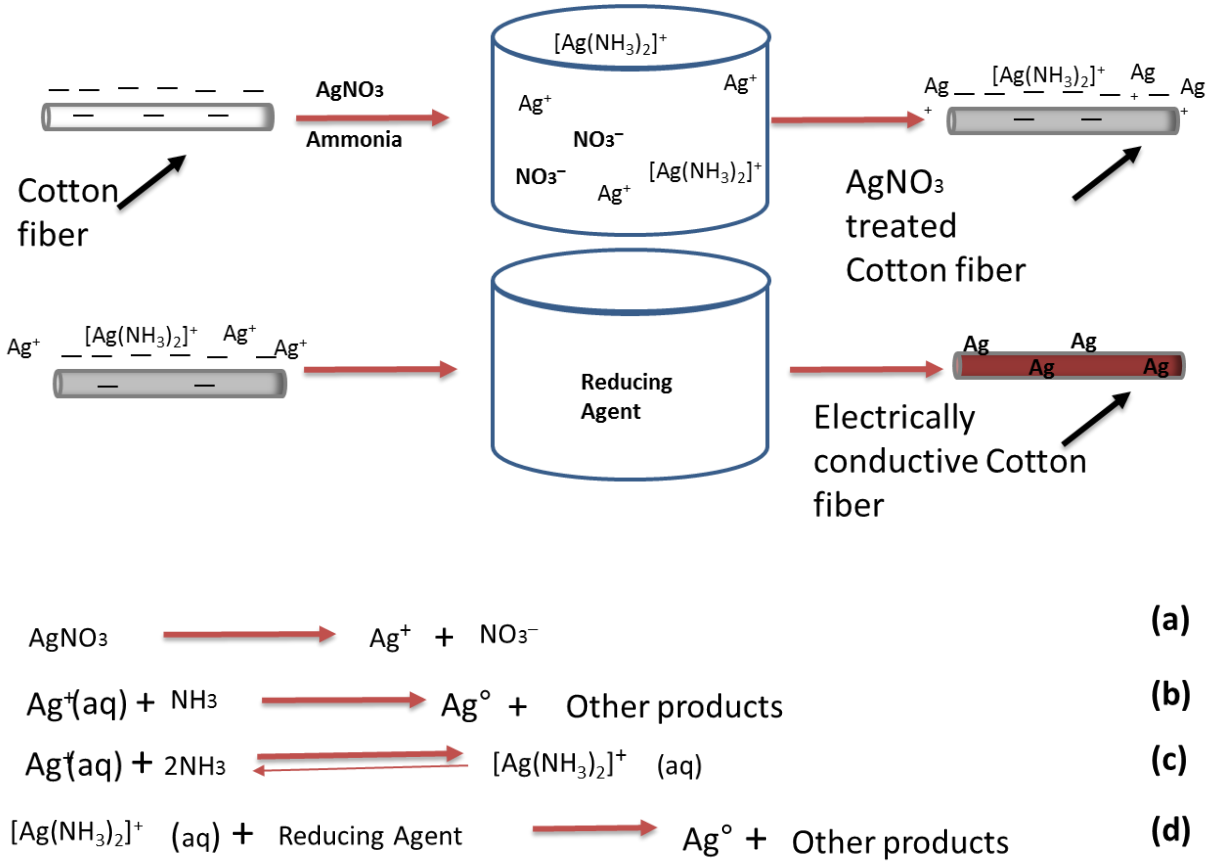


Figure 42: Mechanism of silver particle deposition on cotton fabrics

5.2.1.4 Electromagnetic shielding

Figure 43 shows the results of shielding effectiveness for the fabric samples coated with 17 g/L solution of silver nitrate after 50, 100 and 150 dips. The electromagnetic shielding effectiveness was found to increase with increase in number of dips. This behavior was attributed to higher electrical conductivity behavior of samples prepared from more number of dips due to uniform and dense packing of silver particles on the fabric surface. This can be further explained by SEM images shown in Figure 41, where dense network of silver particles can be found for more number of dips.

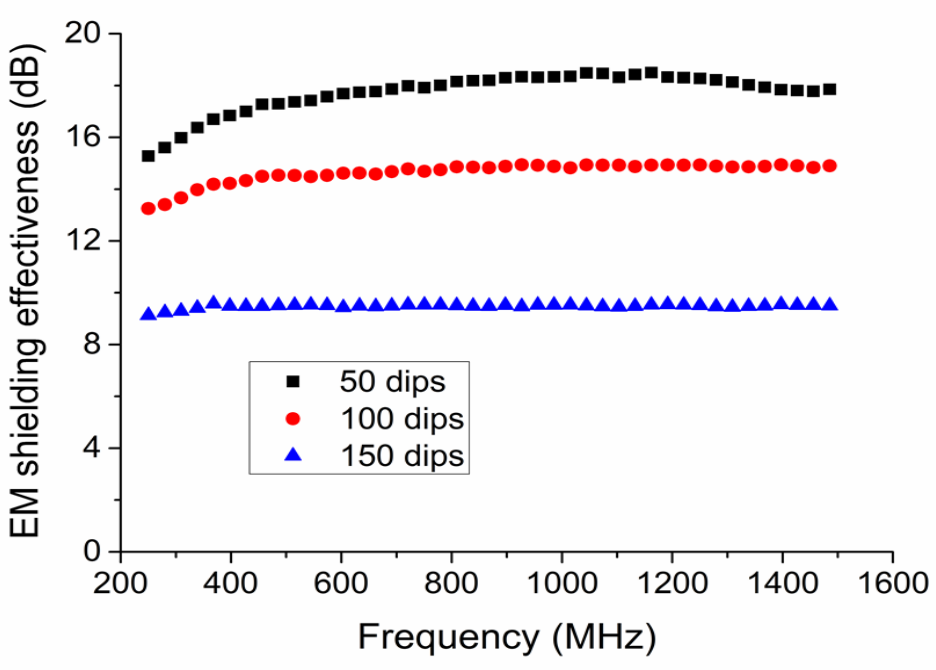


Figure 43: EMI shielding effectiveness of silver coated cotton fabrics

5.2.1.5 Antimicrobial properties

The antimicrobial activity of silver coated fabrics was tested against gram-negative *E.coli* and Gram-positive *S. aureus*. Figure 44 shows the zones of inhibition around fabric samples after 24 h of incubation in dark at 37 °C. The untreated fabric samples without silver coating showed no antimicrobial activity, however, the zone of inhibitions was evidenced against both type of bacteria *S. aureus* and *E. coli* after the silver coating.

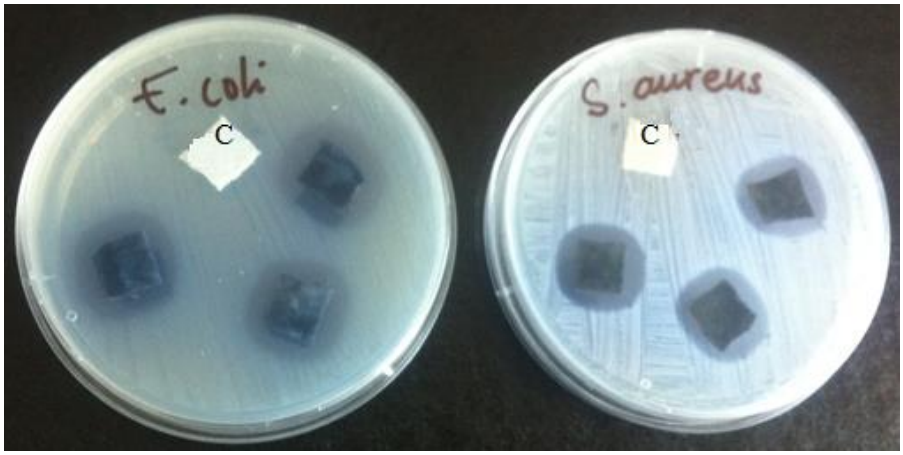


Figure 44: Antimicrobial property of silver coated cotton fabrics

5.2.1.6 Durability

The durability of conductive fabrics under washing has been a critical challenge. To investigate these properties (fabric samples coated from 17 g/L solution of silver nitrate after 50, 100 and 150 dips) were selected. These three samples were selected because they provided satisfactory results regarding electrical conductivity. The electrical resistivity of samples was measured before and after washing as shown in Figure 45. It is evident that there is only a slight change in the resistivity of these samples after washing. It means nanoparticles are attached firmly on the surface of fabrics without losing any conductivity. This was also verified by the SEM picture (Figure 46), which showed the presence of silver particles on fibre surface after washing.

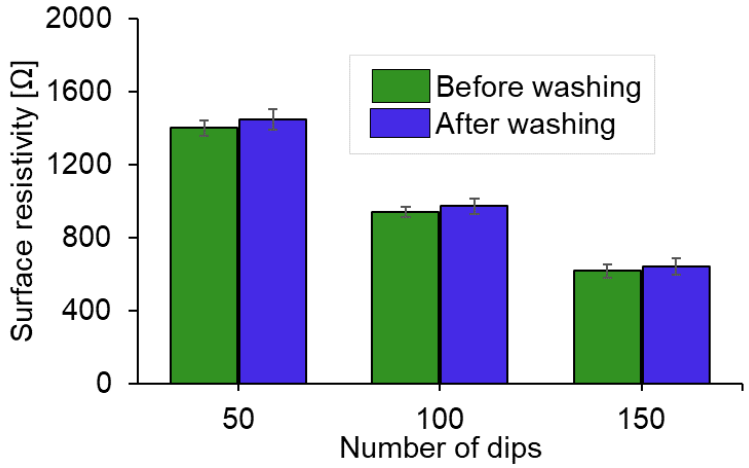


Figure 45: Electrical resistivity before and after washing (bars are limits of 95 % confidence interval)

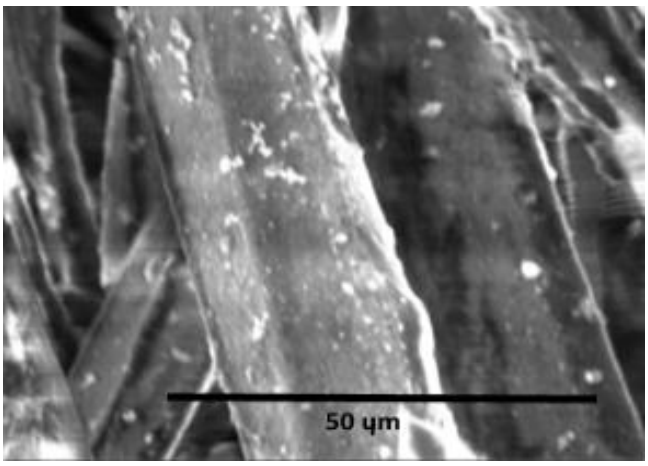


Figure 46: SEM image of silver coated cotton fabric after washing

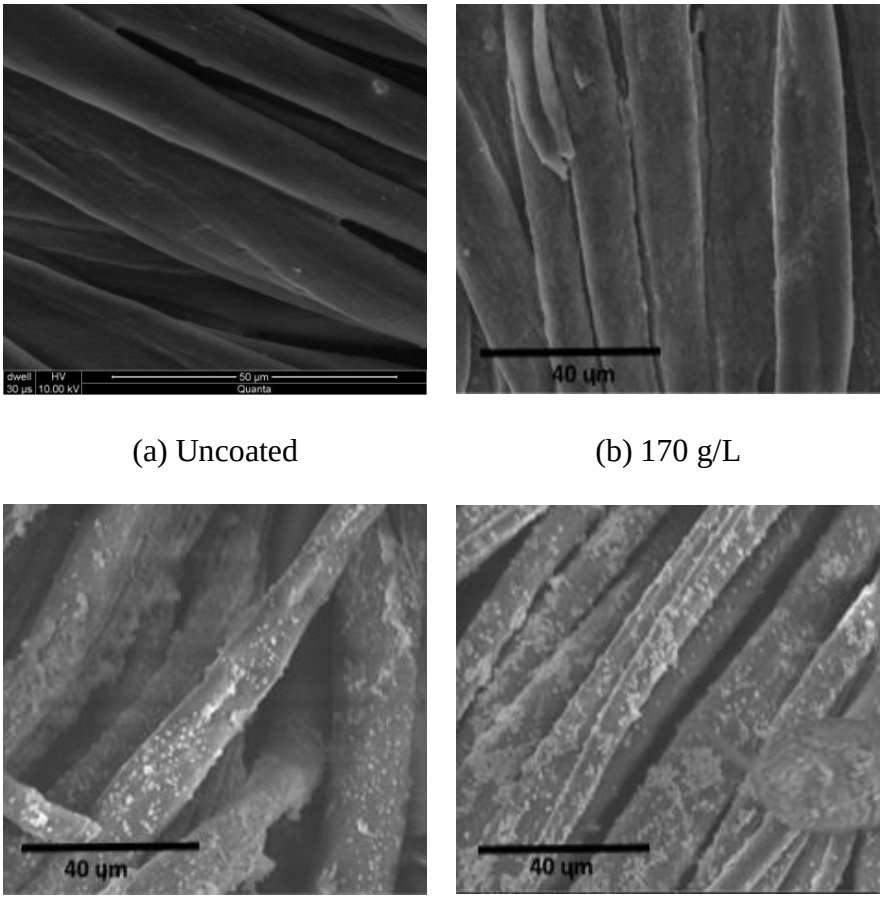
5.2.2 Silver particles coated knitted fabric

5.2.2.1 SEM analysis

It can be seen from Figure 47 that the deposited silver particles on knitted fabrics are in nanometer scale. The coating of silver particles was found to become more uniform and dense with decrease in the concentration of $AgNO_3$. The silver particles covered maximum surface area at 42.5g/L $AgNO_3$ concentration compared to 85 g/L or 170 g/L of concentrations. This further indicated the higher tendency of formation of percolated network of silver particles as $AgNO_3$ concentration decreased. The elemental composition for silver coated fabrics is shown in Table 7. The increase in contents of silver can be observed at lower concentration of $AgNO_3$. The thickness of fabric was increased to 0.93 mm from 0.85 mm after the deposition of silver particles.

Table 7: Elemental composition of silver coated knitted fabrics

Silver nitrate concentration (g/L)	**Weight (%)**		
	C	**O**	**Ag**
170	61.01	36.59	2.4
85	51.65	36.59	11.76
42.5	42.06	42.88	15.06



(a) Uncoated (b) 170 g/L

(c) 85 g/L (d) 42.5 g/L

Figure 47: SEM images of silver coated fabrics at different silver nitrate concentrations

5.2.2.2 XRD analysis

It was carried out to know the phase composition of deposited silver particles. Figure 48 shows the XRD patterns of samples for the 2θ range of 20 to 80 degrees with a step of 0.02 degree. The phase purity of the prepared silver particles can be clearly seen from perfect indexing of all the diffraction peaks to the silver structure. Compared to the untreated cotton fabric, four new peaks at 2θ values of 38.1, 44.3, 64.5 and 77.5 were detected for silver coated fabrics, which were respectively attributed to the diffraction peaks of the (1 1 1), (2 0 0), (2 2 0) and (3 1 1) planes of silver with cubic structure reported in the International Center for Diffraction Data (JCPDS data number 04-0783 card) [127]. Furthermore, no characteristic peaks were observed for other impurities such as AgO.

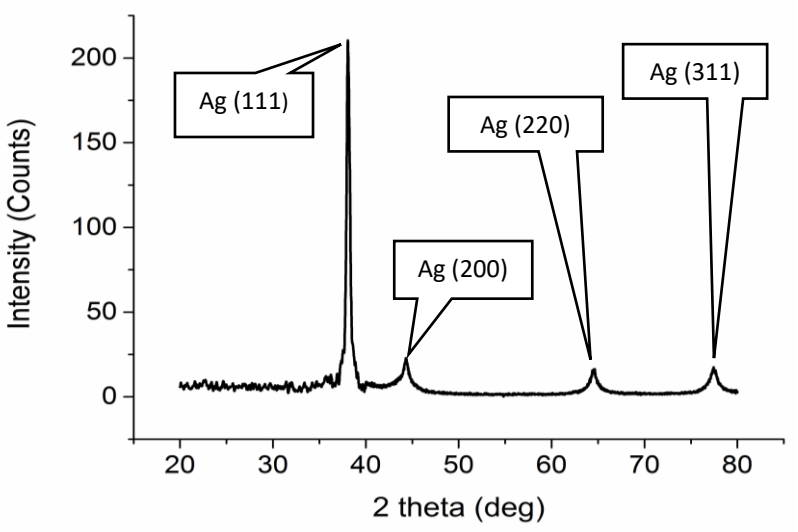


Figure 48: XRD patterns for silver coated fabric

5.2.2.3 Electrical conductivity

The electrical resistance is related to the electrically conductive channels paths. More electrically conductive channels (particles, fillers, conductive fibres etc.) mean lower electrical resistance [128]. In present work, the effect of different concentrations of silver nitrate on electrical conductivity of coated fabrics was studied. From Figure 49, the resistivity was found to increase with increase in the concentration of silver nitrate. This behaviour can further be justified from regression analysis (i.e., equation of line) between two parameters. The relationship between concentration of silver nitrate and volume resistivity is positive, as explained by the equation of line and higher R^2 (0.988) value. The fabrics exhibited higher electrically conductivity at lower silver nitrate concentration due to uniform and dense deposition of silver particles which enabled the formation of more conductive paths. Furthermore, the formation of percolated network at 42.5 g/L silver nitrate concentration was in agreement with previous discussion on Figure 47 of SEM microstructures of coated fabrics. The development of electrical conductivity was later verified by flow of electric current as shown in Figure 50. The straight line in Figure 49 is regression line obtained by minimizing of standard least squares criterion (sum of squared deviation of points from regression line). All parameters (slope and intercept) were statistically significant.

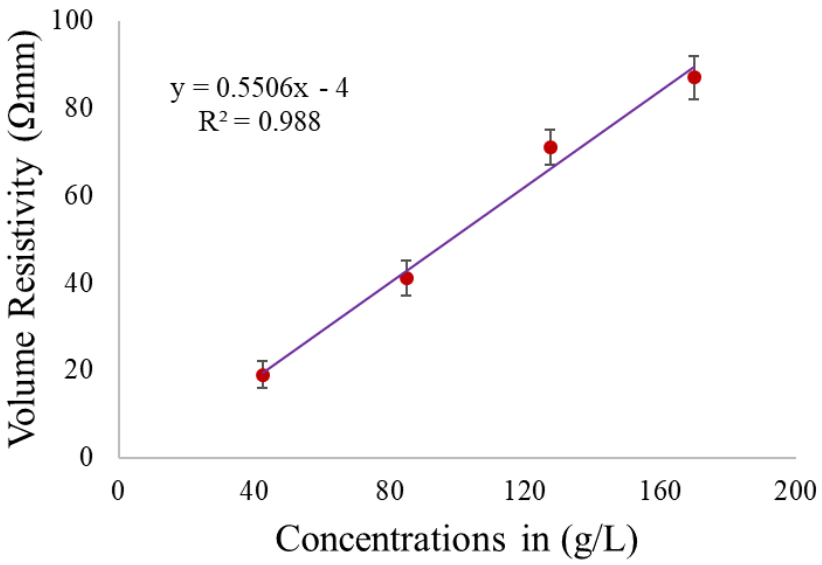


Figure 49: Effect of silver nitrate concentration on volume resistivity

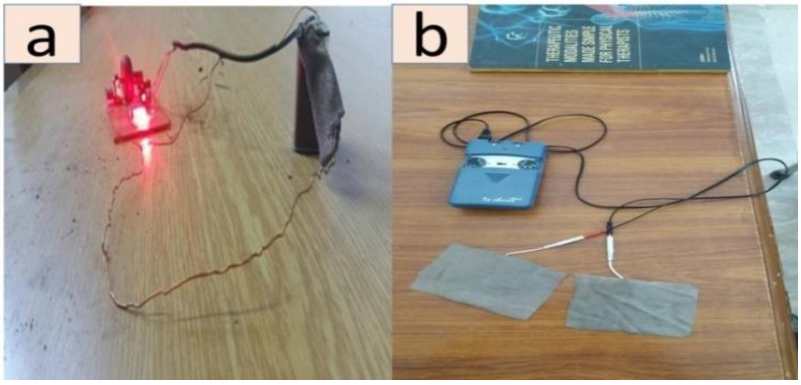


Figure 50: Flow of electricity in (a) silver coated fabrics (b) fabric electrodes with TENS

In order to simulate the performance of electrodes under various movements of human body, the conductive fabrics made from 42.5 g/L silver nitrate were subjected to various extensions and change in resistivity was examined. Figure 51 shows the electrical volume resistivity of silver coated fabrics as a function of different extensions ranging from 0 to 100 %. At 0% stretch (i.e., when the fabric was unstretched), the volume resistivity was 19 Ω mm. The volume resistivity was found to gradually increase with increase in extension, reaching 164 Ω mm at 70% stretch and 478 Ω mm at 80% stretch. The change in volume resistivity was so small that it was considered a constant value in the stretch range of 0–80%. Nevertheless, the volume resistivity was found to increase sharply beyond the 80% extension, where it reached to 2167 Ω mm at 90% stretch and then to 4340 Ω mm at 100% stretch. This behavior can be attributed to the rupture of conductive network due to increasing of mean interparticle

distance at higher extensions. This behavior of stretching can further elaborate by a recent study, researchers developed a stretchable electrically conductive fabric by coating of silver nanowires on cellulosic fabric. Initial electrical resistivity of fabric was around 0.0047 Ω. The resistivity was also measured as a function of stretching where maximum resistivity was measured about 0.0274 Ω at 200% stretching and then sharply to the highest value of 112.1649 Ω at 210% [7]. In another similar study, nylon/ spandex (95/5) electrically conductive knitted fabrics were used as a electrodes for TENs device. The fabrics were firstly coated with, poly-pyrrole, then for surface catalyzation, fabrics were dipped into a catalyzation solution containing $SnCl_2$, $PdCl_2$ and HCl. After that, furthermore plating was performed. It was noticed that there was insignificant decrease in electrical conductivity even up to 80|% stretch. However, due to low electrical conductivity the developed electrodes were not efficient for TENs device as compared to conventional electrodes [69].

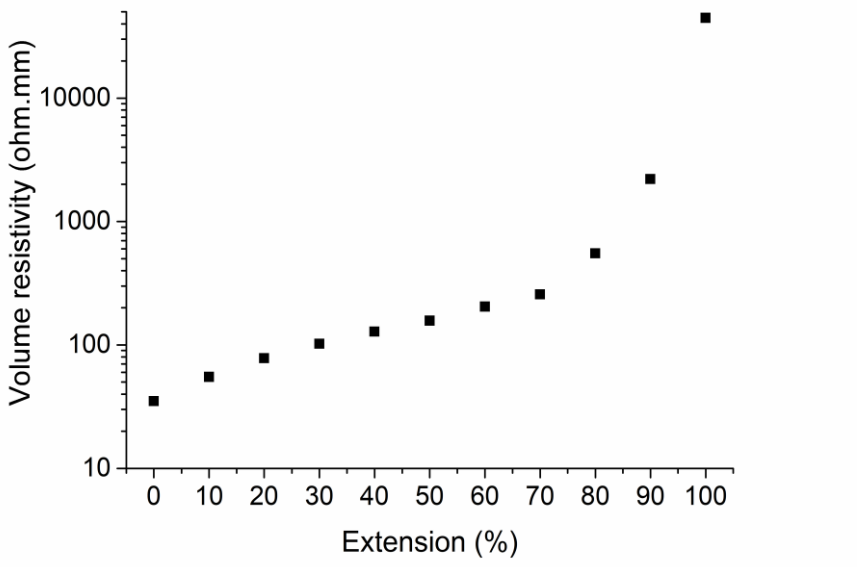


Figure 51: Effect of extensions on volume resistivity of silver coated knitted fabric

From previous discussions, the electrical resistivity of coated fabrics was found to change with increase of extensions. Therefore, further investigation on multiple usages of developed textile electrodes was studied under repeated stretching and relaxing tests. Figure 52 showed the effect of repeated 50% and 100% extensions on change in volume resistivity of coated fabrics. For 50% extension, the volume resistivity of coated fabrics did not change significantly under different cycles of stretching. However, at 100 % extension, the coated fabrics showed significant change in volume resistivity with increase of stretching cycles. This

behavior can be attributed to the damage of percolated networks of silver particles due to formation of cracks under repeated excessive extension.

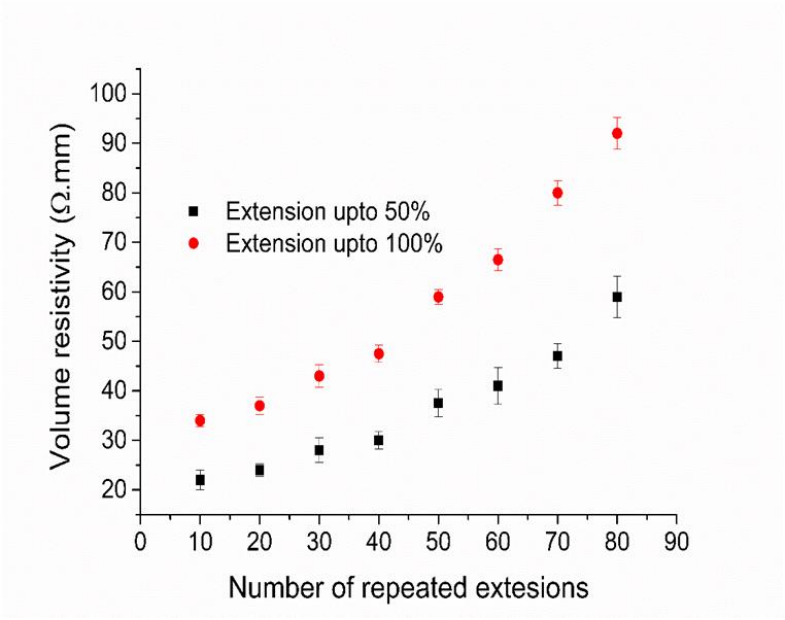


Figure 52: Change in resistivity with number of repeated extensions (bars are limits of 95 % confidence interval)

For electrotherapy applications over prolonged duration, the change in volume resistivity of coated fabrics was investigated with increase in time when constant current of 20 mA was applied. From Figure 53, it can be seen that the volume resistivity of coated fabrics remained unchanged with time over 13 min when the same current was applied. Therefore, consistent performance of developed electrodes can be expected for efficient electrotherapy operations over prolonged durations.

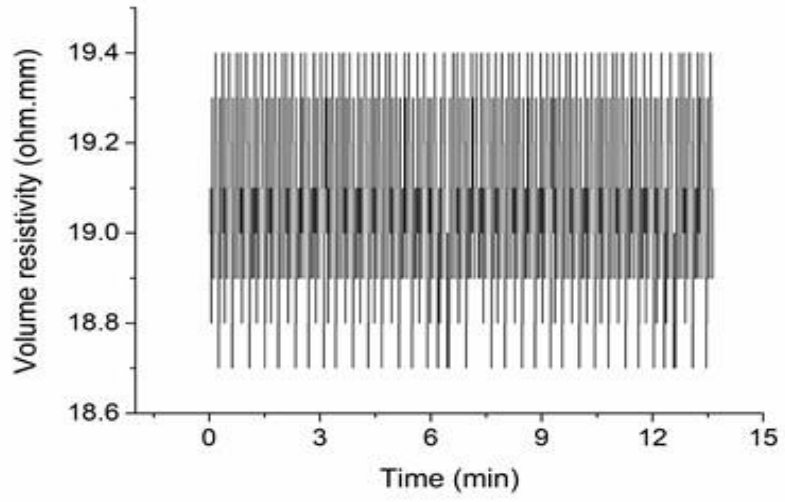


Figure 53: Change in resistivity over prolonged duration with constant current of 20 mA

5.2.2.4 Force extension curve analysis

TIRA tensile testing machine was used to evaluate force extension analysis of knitted fabrics. The force extension behavior was checked to observe the effect of silver coating on the stretch-ability of knitted fabrics. The test was performed before and after coating of silver particles. The fabric strips were cut according to standard dimension (20 cm in length, 4 cm in width). The fabric strips were clamped between the jaws of tensile testing machine. The experiment was rated with constant speed of 100mm/min. The force extension curves are shown in Figure 54. It is clear from the behavior that there is insignificant effect on the searchability of knitted structure after coating of particles. Very small increase in force was observed after the loading of particles. The load bearing capacity of fabrics was improved due to coating of silver particles on the surface. This behaviour was attributed to small size of silver particles which can easily penetrate in cellulose polymeric chains and act as cross linking agents or fillers for load bearing capacity [129].

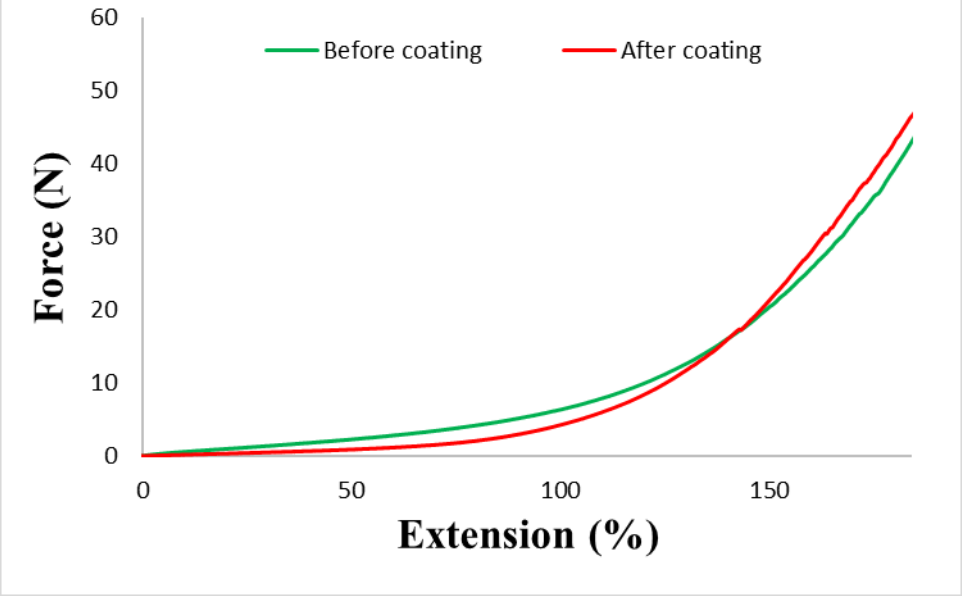


Figure 54: Force extension behavior of silver particles coated knitted fabrics

5.2.2.5 Antibacterial properties

The antibacterial activity of silver coated fabrics was tested against gram-negative *E.coli* and Gram-positive *S. aureus*. Figure 55 shows the zones of inhibition around fabric samples after 24 h of incubation in dark at 37 °C. It is clear that fabric sample without silver coating showed no antibacterial activity. However, the zone of inhibitions was evidenced against both type of bacteria Staphylococcus aureus and Escherichia coli after the silver coating. Further, Staphylococcus aureus depicted the highest sensitivity as compared to

Escherichia coli. The zone of inhibitions for Staphylococcus aureus increased from 8.4 to 13.2 mm while for Escherichia coli it increased from 6.2 to 10.7 mm. The antibacterial property of coated fabrics can be attributed to the combination of chemical and physical interactions of bacteria with particles. The silver particles incorporated into the cell and leads to further massive oxidative stress for antibacterial performance. However, the low efficiency of the antibacterial property in present work was related to low oxidative stress of larger size silver particles as compared to silver particles of below 100 nm in literature.

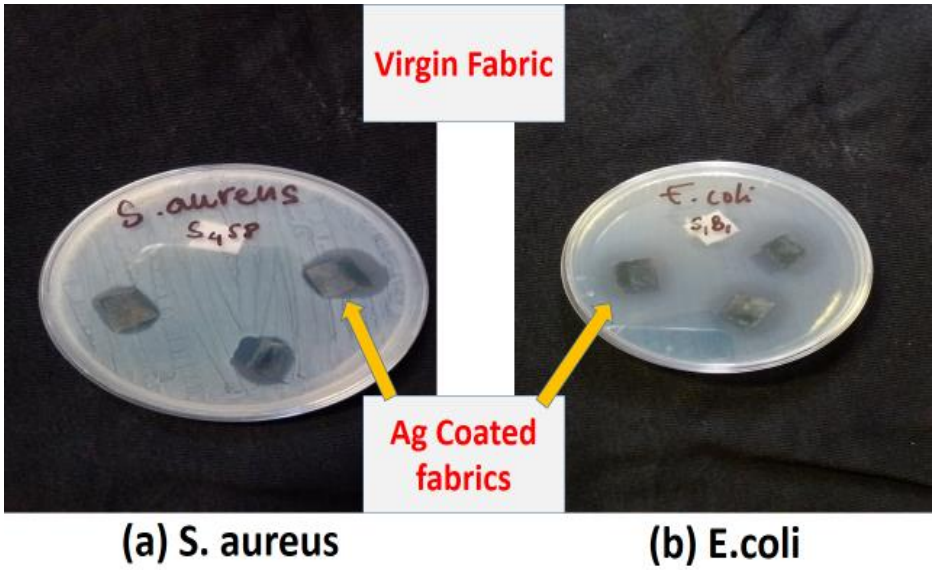


Figure 55: Antibacterial properties of silver coated fabrics

5.2.2.6 Durability of silver particles coated knitted fabric

The silver particles were attached to fabrics through a combination of both physical and chemical interactions. Further, the additional silver particles filled the spaces between fibers and stacked together to form the electrically conductive networks. Therefore, the durability of coated fabrics was studied to determine the chances of silver particles removal from the surface. At first, an adhesion test was performed with transparent tape. However, no visible particles were observed on the tape, which indicated strong bonding of silver particles with the fabric surface. In another test, the coated fabrics were washed according to method ISO 105-C01 standard and then change in electrical conductivity was measured (see Figure 56). From the results, the electrical resistivity of washed fabrics was found to increase in small percentage, which confirmed strong attachment of silver particles with fabric surface. Later, it was also verified from the SEM images shown in Figure 57, which depicted the presence of silver particles on fabric surface after the washing.

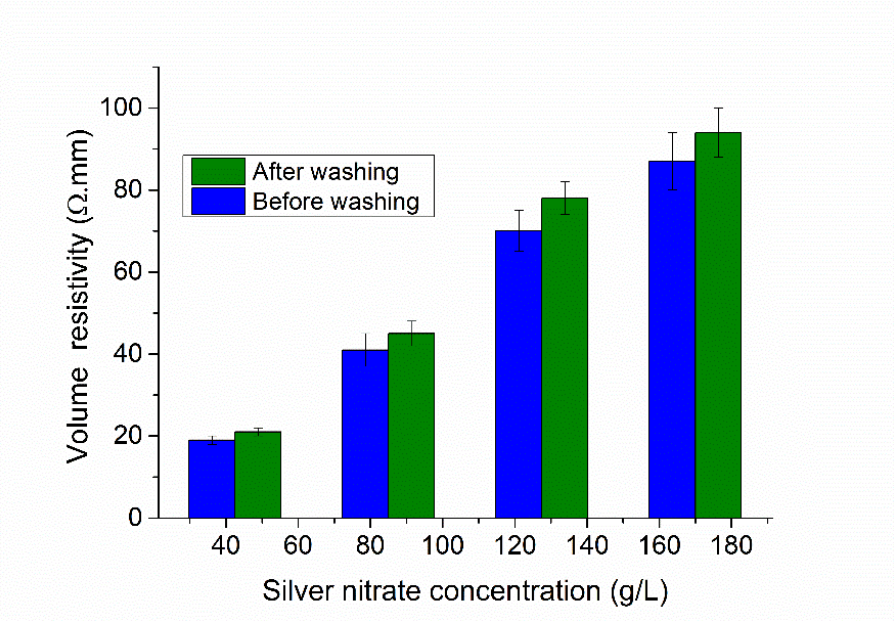


Figure 56: Electrical conductivity of silver coated fabrics before and after washing (bars are limits of 95 % confidence interval)

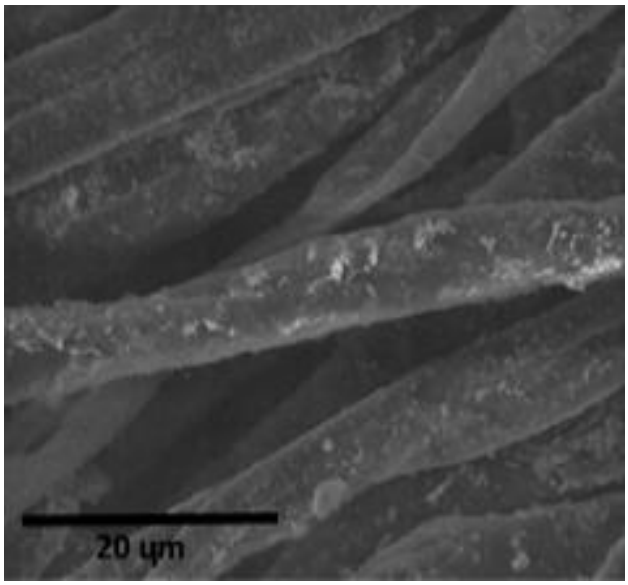


Figure 57: SEM image of silver coated fabric after washing

5.3 Electroless plating of copper over previously coated fabrics

The results are composed up of two sections. At first results are described for the electroless plated fabrics woven fabrics. Secondly, the results of triboelectric generators made from plated knitted fabrics.

5.3.1 Electroless plated fabrics

Based on previous experiments discussed in part 1 and part 2, the surface activation of fabric was carried out using 10 g/L of copper sulphate for deposition of copper particles and 17 g/L of silver nitrate for deposition of silver particles. Then, the copper and silver particles coated fabric samples were immersed in the electroless copper plating bath at room temperature for different intervals of time

5.3.2 Electrical conductivity

The results of electrical resistivity before and after electroless plating are shown in Table 8. It is clear that there is significant decrease in electrical resistivity after electroless plating. Compared to values of electrical conductivity reported in part 1 and part 2, the higher values of electrical conductivity in this work were attributed to more dense deposition of metal particles during electroless plating. The previous layer of deposited copper or silver particles was uniformly penetrated across the thickness of fabric and it allowed even coating of deposition of metal during subsequent electroless plating.

Table 8: Electrical resistivity results before and after copper plating on woven fabrics

Fabric samples	**Electrical resistivity (Ω/square)**	
	Before plating	**After plating**
Copper plating over copper particles coated woven fabric	759 ± 42	20 ± 2
Copper plating over silver particles coated woven fabric	620 ± 37	27 ± 1

5.3.3 Weight gain percentage

The weight gain percentage of the Cu-NPs coated cotton fabric, Ag-NPs coated cotton fabric and electroless plated cotton fabric was investigated. At first, the weight gain percentage was measured for all Ag-NPs coated fabric and Cu-NPs coated fabric samples. The effect of number of dipping cycles against weight gain percentage was measured for each concentration of copper sulfate (10, 20 and 30 g/L) and silver nitrate (17, 34 and 51 g/L).

It is clear from the Figure 58 that weight of cotton fabric was increased in both cases with increase in dipping cycles in respective salt solutions (copper sulfate and silver nitrate). It was observed that weight gain percentage against each concentration of copper sulfate (10, 20 and 30 g/L) and silver nitrate (17, 34 and 51 g/L) was almost the same.

The maximum weight gain percentage for copper particles coating with 10, 20 and 30 g/L copper sulfate was 7.9 %, 7.5 % and 7.3 % respectively. While the maximum weight gain percentage for silver particles coating with 17, 34 and 51g/L silver nitrate was 9.9 %, 9.2 % and 8.9 % respectively. At lower concentration of each salt copper sulfate and silver nitrate the weight gain percentage was slightly higher as compared to higher concentrations. Overall, It was observed that weight gain percentage against each concentration of copper sulfate (10, 20 and 30 g/L) and silver nitrate (17, 34 and 51 g/L) was almost the same. In contrast to this, there was a huge difference between the electrical resistivity results. At lower concentration of each salt 10 g/L copper sulfate and 17 g/L silver nitrate fabric samples showed lowest electrical resistivity. The reason is that at higher concentration of salts there is more nucleation of ions inside the solution. These ions tend to decrease the total surface energy and eventually agglomerated. Hence the formation of big particles over the fabric surface and cause non-homogenous coverage. Secondly, by further increase in number of dipping cycles the heavy and non-homogeneous loosely held particles will erode back into the solution. This will cause uneven coating and remained fail to provide the conductive threshold. This behavior can be attributed to the formation of big sized copper particles at higher concentration of copper sulfate solution. Surprisingly, lower concentration 10 g/L of copper sulfate produced more conductive fabrics due to formation of percolated network by creation of continuous connectivity between the small sized copper particles. This can be further justified from SEM images shown in Figure 30 (a, b), where formation of more percolated network of smaller particles can be found in case of 10 g/L than 20 g/L copper sulfate concentration.

Therefore, the action of agitation or ultrasonication for disrupting the nucleation of copper particles is good topic for further research in order to obtain more percolated network at higher copper sulfate concentration.

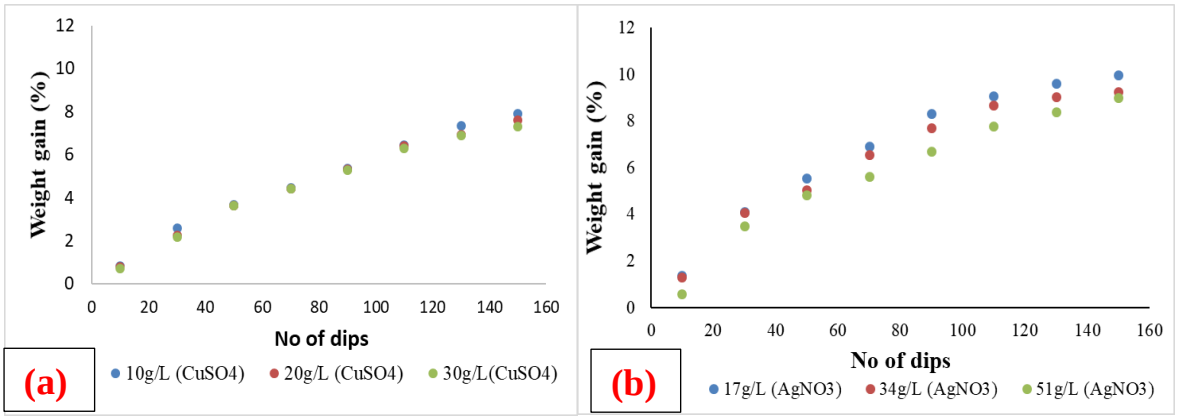


Figure 58: The weight gain percentage with increase in number of dipping cycles (a) Cu-NPs coated cotton fabric, (b) Ag-NPs coated cotton fabric

The percentage of fabric weight gain was also measured with electroless copper plating. The fabric weight gain was measured for copper plating over silver coated fabric and copper plating over copper coated fabric and their respective graphs are shown in Figures 59. The electrical resistivities were decreasing with increasing time of electroless plating for all the cases. The minimum resistivities and maximum weight gain percentage values were confirmed at 30 minutes of plating. From the trend lines it is clear that as we are increasing the time of electroless plating the percentage mass gain was going to increase and electrical resistivity was going to decrease. The mass gain percentage of copper plating over silver coated fabric is higher than mass gain percentage for copper plating over copper particles coated fabric.

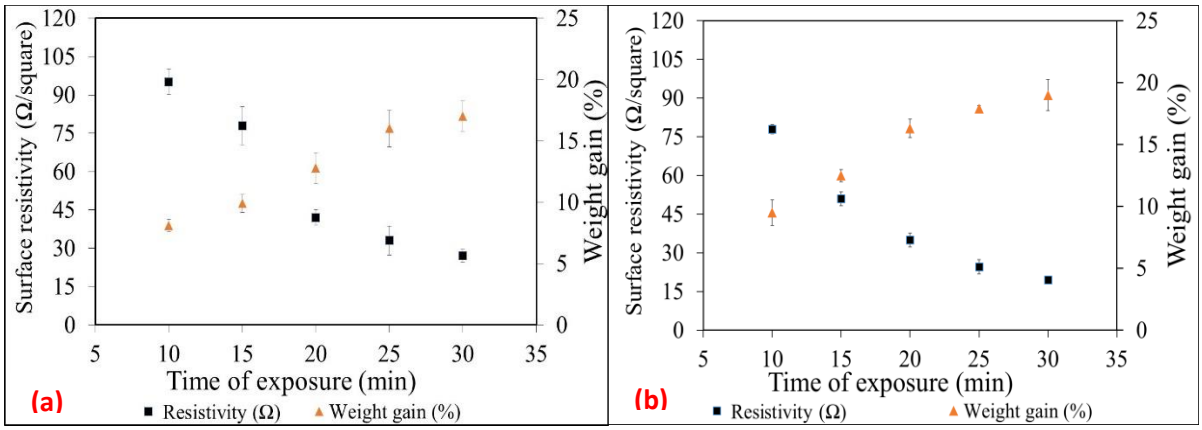


Figure 59: Mass gain percentage with time of copper plating over (a) Cu-NPs coated woven cotton fabric, (b) Ag-NPs coated woven cotton fabric (bars are limits of 95 % confidence interval)

5.3.4 EMI shielding

Before electroless plating, the EMI shielding values of copper and silver particles coated fabric were about 12.65 dB and 18 dB respectively (as reported in part 1 and part 2). After performing the electrolesss copper plating, the same fabrics showed higher values of EMI shielding. The EMI shielding for fabrics of copper plated over silver and copper plated over copper fabric was noticed about 75.53 dB and 66.41 dB, respectively. This behavior was attributed to their higher electrical conductivity values and therefore increased reflection of electromagnetic waves.

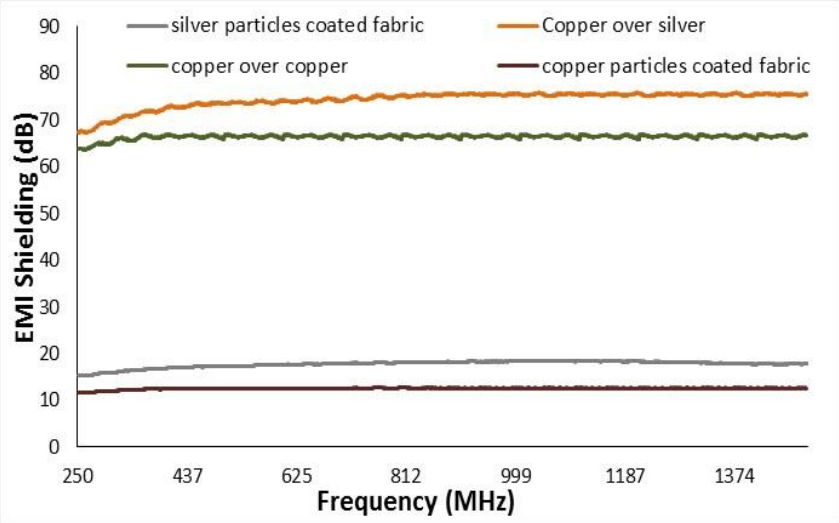


Figure 60: Electromagnetic shielding effectiveness of conductive fabrics

5.3.5 Heating performance

The heating performances of the Cu-NPs coated cotton fabric, Ag-NPs coated cotton fabric and electroless plated cotton fabric was investigated. Joule heating of conductive fabrics was measured by applying the voltage at the ends of fabric. The variation in surface temperature of fabrics were recorded (see Figure 61). Firstly, we applied fix voltage of 5 V with 0.9 to 1 A of current for 1 minute at the ends of eact fabric and recorded increase in temperature was studied. The maximum temperature observed for Ag-NPs (17 g/L) coated fabric and Cu-NPs (10 g/L) coated fabric was about 42.2 °C and 33.4°C respectively (see Figure 61 (a), (b)). While the temperature for copper plating over Ag-NPs coated fabric and copper plating over Cu-NPs coated fabric was recorded about 62.2°C and 68.2°C see Figure 61 (c), (d)). In second step the temperature was measured as a function of time up to 10 minutes with constant applied voltage of 5 V with 0.9 to 1 A of current. The maximum temperature (83 °C for copper plating over Ag-NPs coated fabric and 77°C for copper plating over Cu-NPs coated fabric) were obtained when the applied voltage was 5 V for 10 minutes, as shown in Figure 61 (e), (f)). The temperature on the fabric surface increased up to 83 °C and 77 °C within 1 min, then it slowly increased. In order to study the further stability of the surface heating of conductive fabric, the temperature was measured as a function of voltages with varying voltages from 5 to 10 V and current was maintained at 1 A. Hence the outcome (of different voltages DC input varied from 5–10 V and at constant current 1 A) was calculated in watt for plated fabric samples (copper plating over Ag-NPs coated fabric and copper plating over Cu-NPs coated fabric) and the steady state surface temperature was noted Figure 61 (g). The experiment was carried out up to 10 watt and the measured surface temperature

was stable throughout the duration of the experiment with a homogenous temperature distribution of 119 °C and 112 °C for (copper plating over Ag-NPs coated fabric and copper plating over Cu-NPs coated fabric). In a similar study cotton fabric were functionalized with carbon nanotubes (CNT), and completely loses the heating power of the conductive fabric after 220 s. While in the present study the stability of the conductive fabric is retained even after 60 min. In another study, Hamdani and co-workers tested the voltage effect on their functionalized conductive fabric made from silver coated polymeric yarn and reached a maximum surface temperature of 107 °C with 80 mm terminal separation and at 9 volt[130]. During the heating mechanism of conductive fabric system, the migration of charge carriers gets accelerated due to the applied electric potential. The heat is released once these charge carriers get collided in-elastically with phonons and defects present on the conductive materials. Since, the number of charge carriers get increased with increasing voltage, the surface temperature also starts increasing [131].

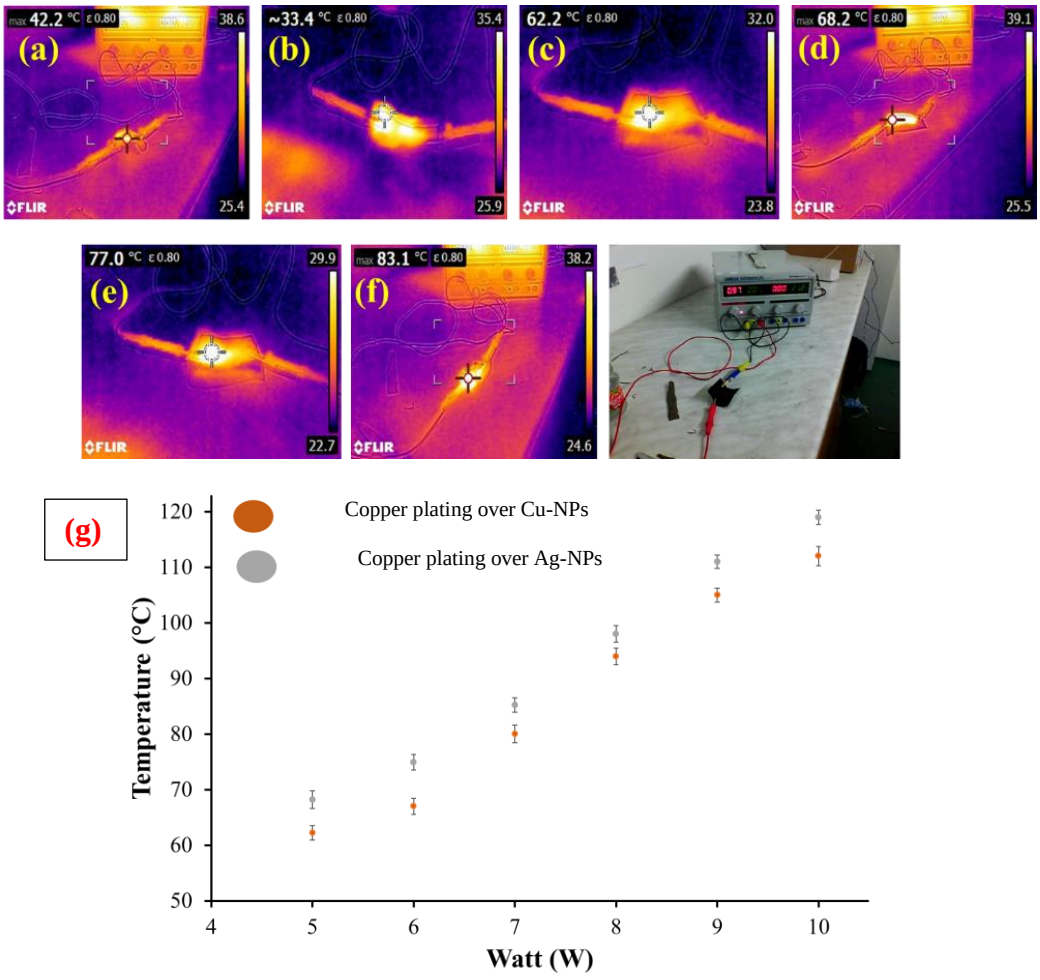


Figure 61: Surface temperature of copper plated fabrics samples: step one at 5 V and 1 min for (a) Cu-NPs coated fabric, (b) Ag-NPs coated fabric (c) electroless copper plating for Cu-NPs coated woven cotton fabric, (d) electroless plating for Ag-NPs coated woven cotton fabric respectively, step two at 5 volt and 10 minute (e), (f) and step three at 5-10 watt (g).

5.3.6 Mechanism of electroless plating of copper

Figure 62 explain the mechanism of electroless plating of copper on cotton fabrics activated with deposition of silver and copper particles. The reactions during the electroless plating stage can be controlled by different parameters such as concentration of metal salt, concentration of reducing agent, concentration of complexing agent, temperature and pH [132].With increase in concentration of copper sulphate, the deposition rate of copper can be increased due to increased mass transfer of copper ions to the electrode surface and therefore accelerated rate of electron transfer. However, on further increase in concentration of Cu^{2+} ions, the deposition rate can reduce significantly due to poor stability of solution bath. This can be attributed to less effect of complexing agent, and therefore adverse reactions in solution followed by electrolyte decomposition. At very high concentration of metal salt, the copper plating may result in rough surface [133]. For selection of proper reducing agent, it should have high standard reduction potential as compared to the metals (copper and silver) being reduced. For instance, the reduction potentials of some important reducing agents like hypophosphite (-1.57V), formaldehyde (-1.30 V) can be useful as they are higher compared to the reduction potentials of copper +0.34V and silver +0.80V [105]. The another important property of reducing agent is self catalytic reaction ability which can help in deposition of maximum copper on target metal (fabric coated with copper particles) [106]. This is important to provide anodic oxidation to the substrate metal in order to avoid the replacement of copper particles by electroless plated copper. The reducing agent formaldehyde is well known to show anoidic oxidation, but the use of formaldehyde is banned due to its toxicity. Therefore, the reducing agent glyoxylic acid (OCHCOOH) was used in present work as a potential alternative to formaldehyde. When copper sulphate is dissolved in water, it results in acidic pH due to formation of H^+ ions in solution.

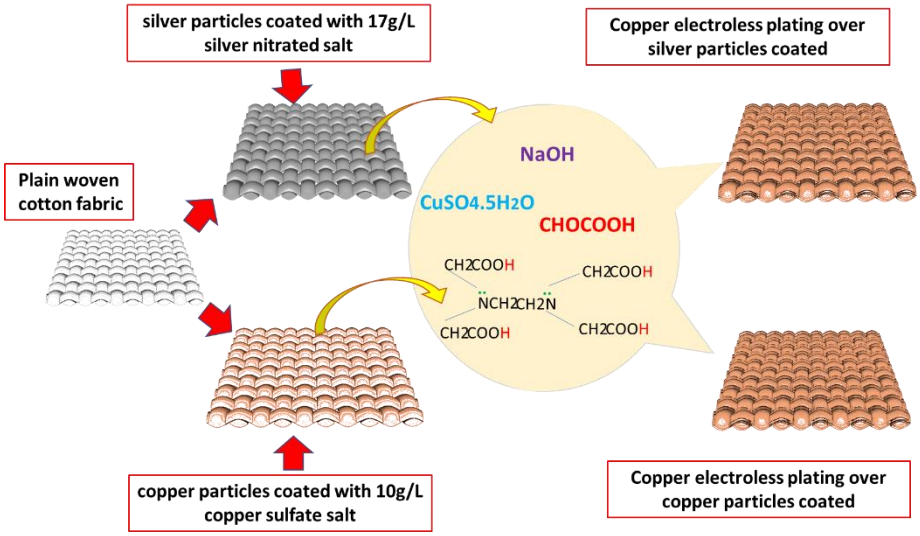


Figure 62: Schematic of copper electroless plating over silver and copper particle coated fabrics

The complexing agent EDTA was used to maintain pH and to avoid the spontaneous reaction between reducing agent and metal salt by formation of complex on free coordination sites available on metal salt $(Cu(EDTA))^{-2}$. The possibility of forming copper hydride or copper hydroxide in solution is increased when complexing agent not used in bath [109]. The concentration of complexing agent is important as it decides the rate of reaction. The bath stability is achieved further with the help of complexing agent. If the concentration of EDTA is less than Cu ion concentration, $Cu(OH)_2$ precipitates in the bath even without the presence of a reducing agent. At higher amount of EDTA, there are higher chances of forming Cu_2O. The concentration of complexing agent should be decided in such a way, that there should be sufficient amount of free metal ions available for reduction purpose [110,111]. If a complexing agent provide low stability constant then it yield a large amount of metal ions as compared to the metal complex and therefore causes the rate of plating to be lower. The amount of complexig agent during equilibrium with free and complex ions in water can be seen from reaction given in Equation 9.

$$M^{+z} + mL^{n-1} \rightarrow M\ L^{z-mn} \tag{9}$$

The stability constant can be estimated from Equation (10).

$$Stability\ constant = \frac{[M\ L^{z-mn}\ ]}{M^{+Z}+[mL^{n-1}]} \quad (10)$$

Where *M* metal ion, *m* is number of complexing agent or Ligand or coordination number, *L* is Ligand, L^{n-1} is complete complexing agent, *z-mn* is oxidation number.

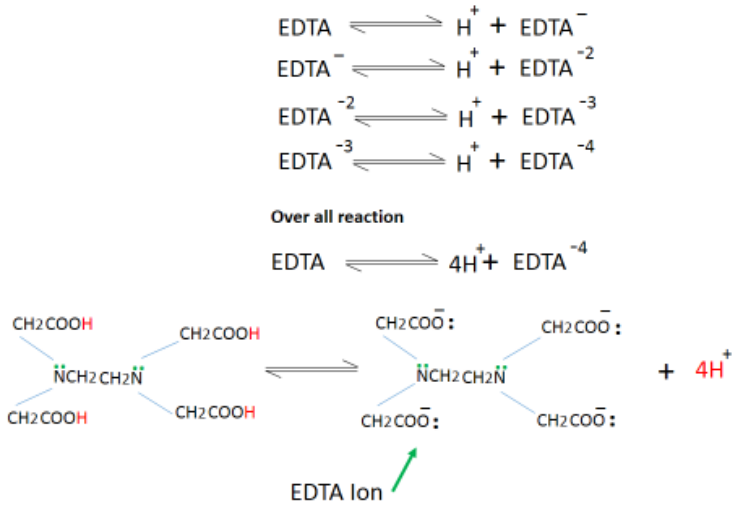
During the deposition process either proton or hydroxyl ions are generated, which in turn changes the pH of solution. For instance, the reaction (11) reaction (12) show that oxidation of reducing agent can produce H^+ and therefore lower the pH [13]. The most suitable pH was found above 11. Furthermore, in a previous study it was observed that when EDTA and copper ions concentration were identical in solution, the $Cu(OH)_2$ still precipitated with increasing the OH^-. Therefore, the pH played important role in controlling the bath stability role during electroless plating [134]. When there are dust particles in electroless plating bath, it may result in spontaneous reduction reactions with the metal nuclei. The stabilizer can be added into the bath to prevent this problem. The stabilizer acts as an inhibitor by adsorbing on the nuclei and shields them from spontaneous reduction reactions in the solution. Therefore, 40 ppm of 2,2' bipyridyl was added to the Cu-EDTA solution to act as stabilizer [135].

The composition of electroless plating bath can not be decided easily as they can lead to a more complex electrochemical reaction pathway [136]. During the electroless plating system, the knowledge of mixed potential theory (cathodic and anodic half reactions) can be beneficial. Here, reducing agent oxidation is concerned to the anodic reactions (see reaction (11)) whereas metal reduction is concerned to the cathodic reactions (see reaction (12)). The choice of reducing agent is important as it provides the anodic oxidation. In a study Yu *et al* reported the anodic oxidation of glyoxylic acid on Cu [136].

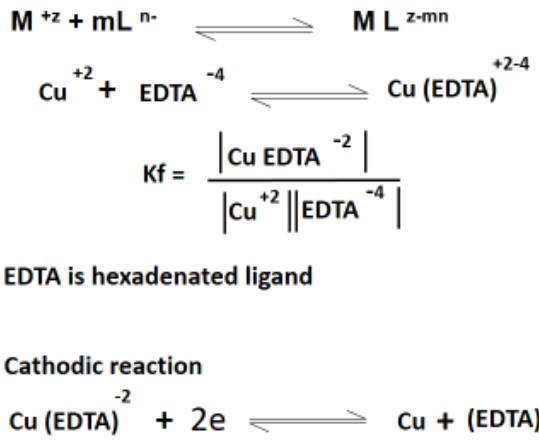
Anodic reactions

$$2OCHCOOH + 4OH^- = 2HC_2O_4^- + 2H_2O + H_2 + 2e \quad (11)$$

Cathodic reaction



(12)



(13)

The oxidation of glyoxylic acid is important because it enhances the reduction of cupric-ion through intermediates. The theory of autocatalysis addressed that the reduction of cupric ions can refresh the electroless Cu surface, thus providing more catalytic sites which in turn can accelerate the glyoxylic acid oxidation [115] (see Figure 63 and reaction (14)).

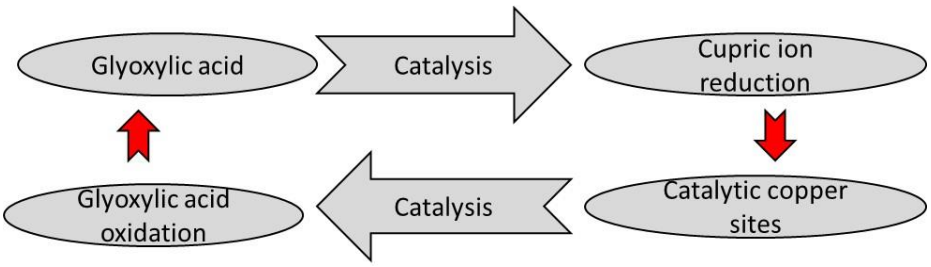


Figure 63: Autocatalysis mechanism involved during electroless plating of copper

Overall reaction:

$$Cu(EDTA)^{-2} + 2CHOCOOH + 4OH^{-} = Cu + 2HC_2O_4^{-} + 2H_2O + H_2\uparrow + EDTA^{4-}$$

Some possible side reactions:

$$2Cu^{+2} + CHOCOOH + 5OH^{-} = Cu_2O\downarrow + HC_2O_4^{-} + 3H_2O \quad (14)$$

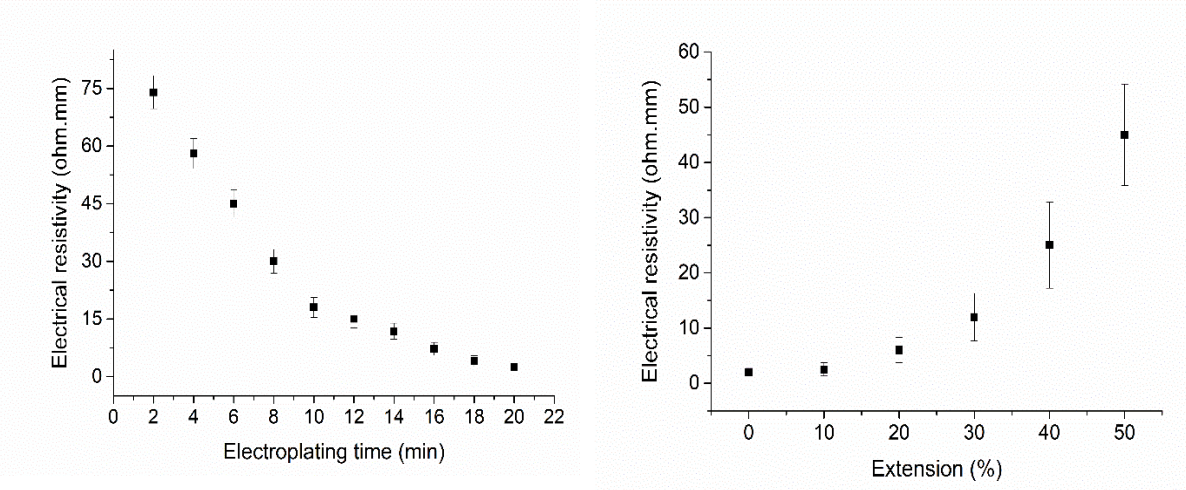
$$2CHOCOOH + 2OH^{-} = C_2O_4^{2-} + 2HOCH_2COOH + H_2O$$

$$Cu_2O + H_2O = Cu\downarrow + Cu^{+2} + 2OH^{-}$$

5.4. Triboelectric generators

5.4.1 Electrical conductivity of silver electroplated fabrics

The electrical volume resistivity of silver electroplated knitted fabrics was reported around 2 Ω mm. This behavior can be attributed to the formation of uniform and dense network of silver metal particles over the surface of fabric, which enabled the formation of more conductive paths. In order to simulate the performance of electrodes under various movements of human body (i.e. stretching on elbow), the electrical conductivity of silver plated fabrics was also examined at different stretch levels ranging from 0 to 50 %. From Figure 64 (b), it can be seen that the resistivity values of silver plated fabric without stretching was 2 Ω mm, however it increased to 45 Ω mm at 50 % stretch. Nevertheless, this change in electrical resistivity was very low and can be considered as constant value for successful operation of TrEG during the human activity.



(a) Electroplating time (b) Extension level

Figure 64: Electrical resistivity of silver electroplated fabrics (bars are limits of 95 % confidence interval)

The explanation on reduction in electrical conductivity of coated fabrics at higher extension can be given from the micro photos of stretch areas shown in Figure 65. The grid structure of fabrics showed no ruptures in coating at lower extension levels. However, the frame work of coating gradually started to break with increase of extension due to stretching of yarns in knitted loop structures. In present work, the 50 % extension was found as limiting stretching limit to prevent further reduction in electrical conductivity of coated fabrics

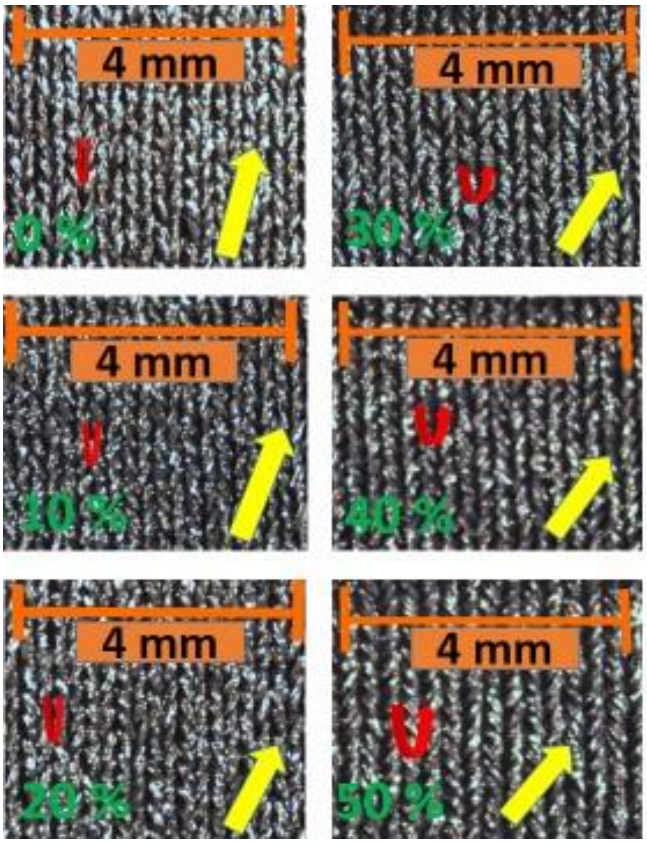
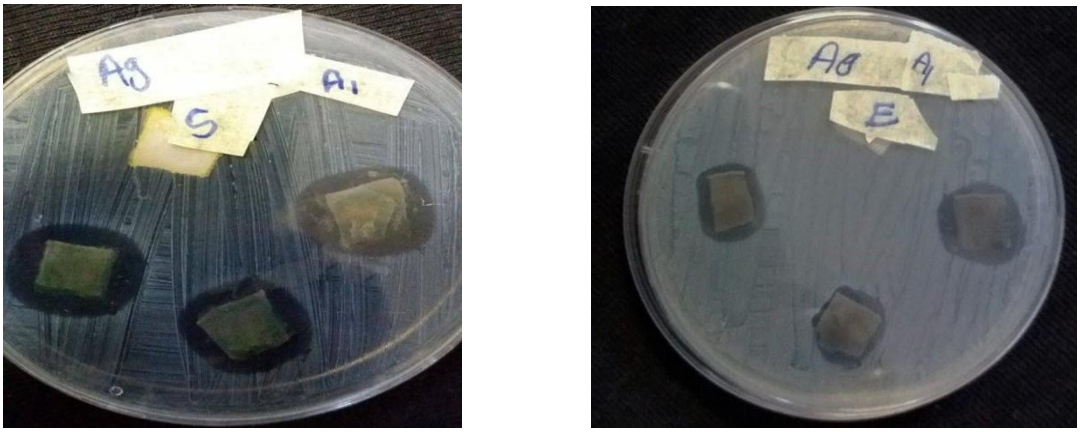


Figure 65: Photographs of silver electroplated fabrics at different stretch levels

5.4.2 Antibacterial properties of silver electroplated fabrics

The antibacterial property of conductive fabric electrodes is extremely important as triboelectric generators are excellent media for microorganism growth due to prolonged contact with human skin or unhealthy indoor air quality. The antibacterial activity of silver plated conductive fabrics was examined against Gram-negative Escherichia coli and Gram-positive Staphylococcus aureus. The fabric samples were kept for 24 h of incubation in dark at 37 °C and the zone of inhibition was observed. It can be clearly seen from Figure 66 that the zone of

inhibition was absent in case of uncoated virgin fabric samples (i.e. no antibacterial property), whereas the zone of inhibitions was evidenced against both type of bacteria Staphylococcus aureus and Escherichia coli after the silver electroplating. The zone of inhibitions of 17.3 mm and 12.5 mm were found in case of Staphylococcus aureus and Escherichia coli, respectively. This indicated higher sensitivity towards Staphylococcus aureus as compared to Escherichia coli. The combination of chemical and physical interactions of bacteria with silver particles resulted into the antibacterial property of coated fabrics. The action started with the incorporation of silver particles into the cell via endocytotic mechanisms, and then release of ionic species within the cells due to dissolution of particles [137]. Therefore, the antibacterial performance originated from the massive oxidative stress due to high intracellular concentration gained within the cell. These results suggested that the developed TrEG can be in contact with elbow or foot for longer time with additional benefits of antibacterial performance.

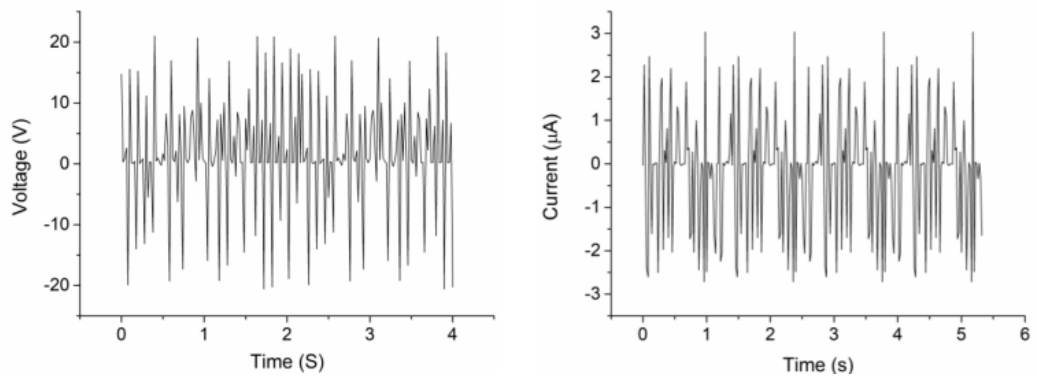


(a) Staphylococcus aureus (b) Escherichia coli

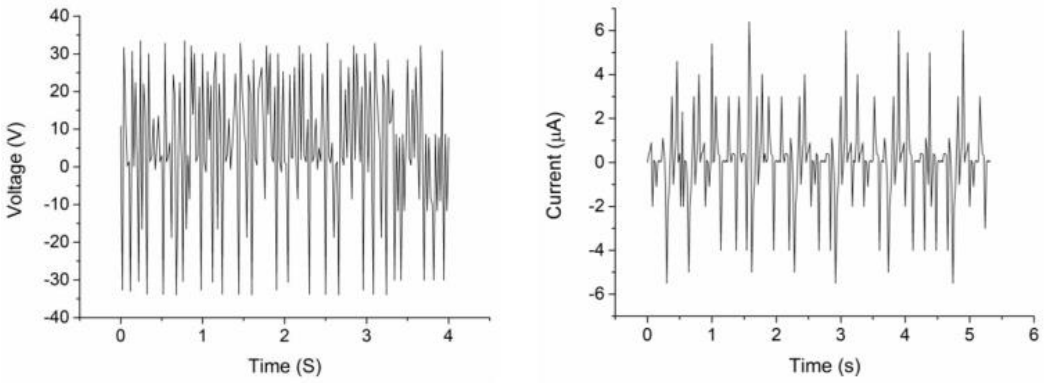
Figure 66: Antibacterial properties of silver electroplated fabrics

The performance of developed triboelectric generator was investigated under both stretching and pressing actions. When the two triboelectric layers were brought into contact, the AC current was produced and it was measured in terms of open circuit voltage *VOC* and short circuit current *ISC* using KEITHLEY TEKTRONIX 2450-SM, Source Meter SMU device. It can be seen from Figure 67 (a) that TrEG produced 21V and 3.5 μA current under the stretching state. However, when the pressing action was applied, it produced voltage of 33 V and current about 6 μA (see Figure 67 (b)). The different performance during stretching and

pressing can be attributed to the difference in friction, vertical contact separation and mechanical deformation of triboelectric layers.



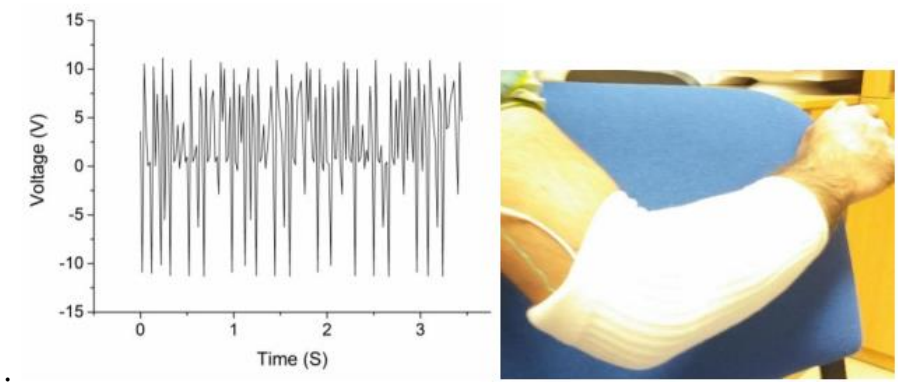
(a) Stretching action



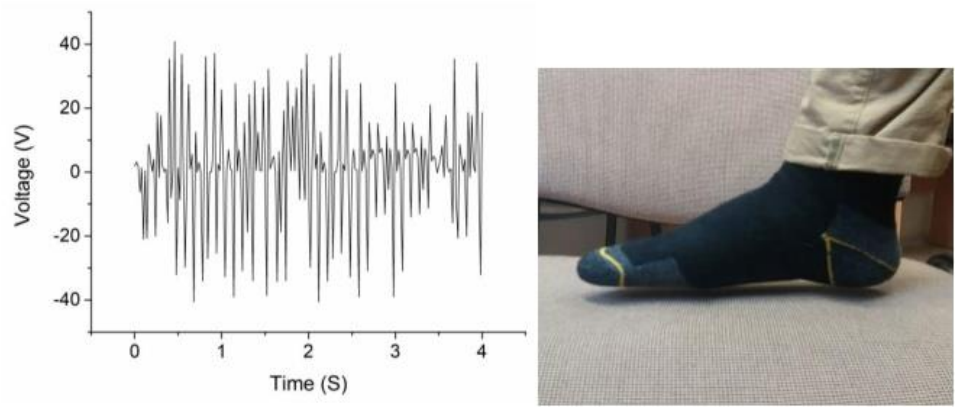
(b) Pressing action

Figure 67: Performance of TrEG under different mechanical movements

Furthermore, the utility of TrEG was investigated to harvest the energy from different daily human activities based on stretching and pressing motions. As major sources to create mechanical energy from human body are movements of elbow and foot, therefore TrEG was attached to those human body parts. The TrEG was found to generate about 10 V from elbow movements due to stretching (Figure 68 a), whereas it generated about 40 V from foot movements due to pressing motion (Figure 68 b).



(a) Stretching action from elbow



(b) Pressing action from foot

Figure 68: Output of voltage during human activities

Figure 69 shows the power generation mechanism of TrEG based on the coupling of triboelectrification and electrostatic induction. At the beginning when there is no rubbing action, no charge is transferred between the rabbit fur and silicone rubber (Figure 69 (a)). After the frictional contact between the rabbit fur and silicone rubber by rubbing, the TrEG get activated. This leads to the development of positive charge on the rabbit fur and negative charge on the silicone rubber. Subsequently, the electrons move from rabbit fur to silicon rubber through the silver-plated conductive fabric electrodes as shown in Figure 69 (b). Furthermore, when the external forces are removed, there is separation of rabbit fur and silicon rubber. This causes the flow of electrons from silicone rubber to rabbit fur via conductive fabric electrodes (Figure 69 (c)). In this way, the kinetic energy of stretching, pressing, and rubbing actions can be converted into electric energy by development of charges on the dielectric materials.

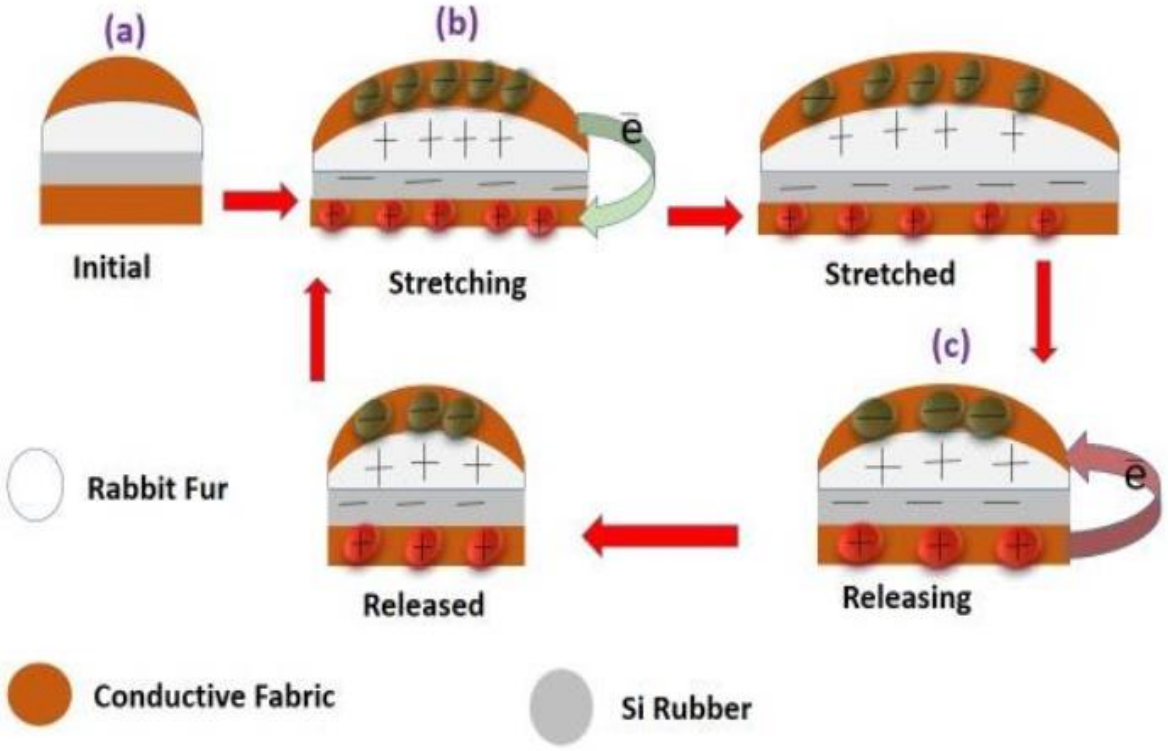


Figure 69: Illustration of the working mechanism of TrEG

5.5 Comparison with previous researches

Electrical conductivity and electromagnetic shielding are the most important properties of metal coated fabrics. Researchers have been using different techniques and methods for metallization of textile. There are a lot of defects regarding the methods they adopt and also obtained results are not so high for EMI SH and electrical conductivity. Bharath et al prepare the electrically conductive, EMI shielded fabrics by electroless plating. He performed all pretreatment steps (pretreatment, sensitization and activation) and done number of coatings (Cu-Ni-Fe Alloy). The results obtained are quite moderate for EMI shielding and electrical conductivity. They obtained 465 Ω/square electrical resistivity, with 75 dB of EMI SH [33]. Following table 9 is showing the comparison between current research and previous researches regarding EMI shielding and electrical resistivity and types of coatings.

Table 9: Comparison between current research and previous researches regarding electrical resistivity and EMI shielding

Conductive fabrics	**Method**	**Electrical resistivity**	**EMI SH (dB)**
Control plain woven	-	10^9	0
Control knitted	-	10^8	0
Copper particles coated woven fabric **(this thesis)**	Dip-Dry-Reduction	759 ±5 Ω/square	12.65
Silver particles coated woven fabric (this thesis)	Dip-Dry-Reduction	620 ±3 Ω/square	18
Silver particles coated knitted fabric use for TENs electrodes **(this thesis)**	Dip-Dry-Reduction	450 Ω/square	22
Copper plated on copper woven fabric **(this thesis)**	Electroless	27±5 Ω/square	66.41
Copper plated on silver woven fabric **(this thesis)**	Electroless	20 ±1 Ω/square	75.53
Silver plating over knitted fabric **(this thesis)**	Ag-plating	24 Ω/square	73
copper plating with all pretreatment steps [138]	Electroless	568 Ω/square	13
Shielding fabric by amorphous Cu-Ni-Fe alloy [33]	Electroless deposition	465 Ω/square	75
Electroless copper deposition on chemically grafting fabric [139]	Electroless	100 Ω/square	5.5
Metallization of polyester fabric by autocatalytic copper plating process [138]	Electroless	128 mΩ/square	40-50
Preparation and properties of copper-silver complex plating on PET fabrics [61]	Electroless/ electroplating	60 mΩ/square	70
Shieldex® Kassel nylon [140]	Standard (copper over silver plated)	0.03 Ω/square	80
Shieldex® Bremen-PW nylon [141]	Silver plated	0.3 Ω/square	35-60
RF Shielding Fabric cotton[142]	Cotton, copper, silver blended	1 Ω /square	55
Copper core conductive fabrics for smart textiles [143]	plating	-	13
Electromagnetic interference shielding effectiveness of copper plated fabrics [38]	plating	5 Ω /square	12
Copper plating over nickle [36]	Electroless plating		32-64

Weight gain percentage and thickness of all developed fabric samples was measured before and after metallizatiom. Furthermore, the results were compared with best studies available in literture. It is clear from table 10 that conductive fabric samples developed by deposition of copper and silver nanoprticles are showing very less increase in weight %. However, further copper electroless plating on previously Cu-NPs and Ag-NPs coated fabrics is showing sufficient increase in weight. Even though, this increase in weight is not so high and is comparable to different studies in literature. This is the benefit of our developed samples to achieve lowest electrical resistivity and high EMI shielding with sufficient amount of metal coating.

Table 10: Comparison between current research and previous researches regarding weight gain percentage

Conductive fabrics	**Method**	**Thickness**		**Weight % increase**
		Before coating	**After coating**	
Control plain woven	-	0.85	-	-
Control knitted	-	0.85	-	-
Copper particles coated woven fabric **(this thesis)**	Dip-Dry-Reduction	0.35	0.37	7.9
Silver particles coated woven fabric **(this thesis)**	Dip-Dry-Reduction	0.35	0.38	9.9
Silver particles coated knitted fabric use for TENs electrodes **(this thesis)**	Dip-Dry-Reduction	0.85	0.93	13
Copper plated on copper woven fabric **(this thesis)**	Electroless	0.37	0.41	17
Copper plated on silver woven fabric **(this thesis)**	Electroless	0.38	0.44	19
Silver plating over knitted fabric **(this thesis)**	Ag-plating	0.85	0.97	21
Copper particles coating [60]	Chemical reduction			30
Cu-NPs coated cotton [144]	Insitu-method	-	-	5.17
Silver particles coating [60]	Chemical reduction			5

Copper-silver particles combination [60]	Chemical reduction			31.2
Electroless nickel plating with all pretreament steps[145]	Electroless	0.270	0.277	18.15
Plating of Cu-Ni-P alloy onto cotton fabrics[146]	Electroless	0.43	0.69	18.47

After intensive comparison with different studies, the present research work has numerous advantages. At first, we developed pure silver and copper particles coated plain woven cotton fabrics, while according to our knowledge there are very less studies available for developing EMI shielded fabrics with copper and silver particles. Secondly, further copper electroless plating on previously Cu-NPs and Ag-NPs coated fabrics is showing very high EMI shielding about 75 dB. The researchers have been doing metallization on pure synthetic fibers or on blended fabrics. Moreover, our developed fabrics are purely copper and silver coated. The developed fabrics can be easily use in medical applications where hygienic properties are required. RF Shielding (copper, silver and cotton blended) fabrics are commercially available EMI shielded cotton fabrics [147]. These fabrics are developed by blending the thin copper and silver wires with cotton fibers during yarn manufacturing and weaving instead of flexible metal coating. Secondly, achieved EMI shielding (55dB) is less as compared to our developed copper electroless plated cotton fabrics (66-75dB). Shieldex is producing very high EMI shielded (80 dB) fabrics with trade name Kassel (80 dB comparable to current thesis results 66-75dB) [140]. These fabrics were produce by coating of copper over silver plated nylon fabrics. The porosity of this fabric is very low comparable with our products. The developed conductive fabrics of current research have extraordinary high EMI shielding, low elelctrical resistivity in addition to other functional properties (ohmic heating and anti-bacterial). Achieved interconnected network of conductive particles with thin and uniform metal deposition. The major benefits of developed electrodes are to provide adequate electrical conductivity, EMI shielding, antibacterial and resistive heating. All these properties are basically required to develop the electrodes for electrotherapy and in vivo applications (where conductive fabrics attached to human skin).

6. CONCLUSIONS

The part 1 of thesis was focused on the development of electrically conductive, multifunctional and durable cotton fabrics by in-situ deposition of copper particles. The copper particles were incorporated into the structure of cotton by sequential dipping in copper sulphate and then sodium hydrosulphite solutions. The amount of deposition of copper particles was controlled by several numbers of dips (i.e. 10 to 150) in copper sulphate. The concentration of copper sulphate was decided at 10 g/L based on acceptable level of achieved electrical conductivity. Further, the morphology of coated fabrics and copper particles was studied from SEM and XRD techniques. The utility of conductive fabrics was analysed for electromagnetic shielding ability over frequency range of 30 MHz to 1.5 GHz by coaxial transmission line method. The EMI shielding was found to increase with increase in number of dips, which was attributed to increased reflection of EM waves due to dense, uniform and percolated network of conductive copper particles on the surface. The sample produced from 50 dips revealed the lowest electromagnetic shielding effectiveness of about 6 dB in frequency range of 600 MHz–1.5 GHz. On the other hand, the sample produced from 100 and 150 dips exhibited the maximum shielding ability of 10 dB and 13 dB, respectively. For multifunctional behaviour, the copper coated cotton fabrics were further examined for antibacterial properties against pathogenic bacteria such as Staphylococcus aureus and Escherichia coli. The zone of inhibitions for Staphylococcus aureus increased from 9.5 to 15.5 mm, while for Escherichia coli it increased from 7.5 to 12 mm with increasing number of dips. Towards the end, the durability of coated fabrics was examined against washing. The fabrics showed good retention of the copper particles, proved by SEM microstructures and small loss in the conductivity of the material after washing.

The part 2 of thesis was focused on the development of electrically conductive fabrics by in-situ deposition of silver particles on the surface of cotton fabrics by sequential dipping in silver nitrate and then glucose stock solutions. The effect of silver nitrate concentration on electrical conductivity was investigated, and 17 g/L found to provide acceptable level of electrical conductivity values. Further, the amount of deposition of silver particles was controlled by several numbers of dips (i.e. 10 to 150) in silver nitrate solution. The higher number of dips produced dense network of silver particles, and therefore resulted in higher electrical conductivity. Later, the utility of conductive fabrics was analyzed for electromagnetic shielding ability over frequency range of 30 MHz to 1.5 GHz by coaxial transmission line method. The samples produced from higher number of dips provided higher EMI shielding due to increased reflection of EM waves. Moreover, the coated fabrics also showed promising

behavior towards antimicrobial properties. Towards the end, the durability of coated fabrics was examined against washing after application of binder on the fabric. The fabrics showed good retention of the silver particles, proved by SEM microstructures and small loss in the conductivity of the material after washing.

Furthermore, second section of part 2 was focused on the utility of silver coated stretchable fabrics as electrodes of TENS machine in electrotherapy applications. The sequential dipping in silver nitrate and then glucose stock solutions was employed for in-situ deposition of silver particles onto knitted fabric. The effect of silver nitrate concentration on performance of coated fabrics was investigated with respect to improvement of electrical conductivity, physiological comfort, and antibacterial properties. The dense and uniform deposition of silver particles was observed at lower (i.e. 42.5 g/L) concentration of silver nitrate and therefore resulted in higher electrical conductivity. Moreover, the coated knitted fabrics were subjected to repeated extension and change in electrical resistivity was examined. In the stretch range of 0–80 %, very small change in electrical resistance was observed, and then it changed significantly beyond 90 % of stretch. Moreover, the electrical resistivity of coated fabrics was found constant after repeated extension of several cycles and also when constant current was applied for prolonged time. Additionally, the coated fabrics also showed promising behavior towards antimicrobial properties. When the durability of coated fabrics was examined against washing, the fabrics showed good retention of the silver particles and small loss in the conductivity of the material.

The work in part 3 reported the significance of copper and silver coated woven fabrics (described in part 1 and part 2) for further deposition of metals during the electroless plating. The more even deposition of metals during electroless plating was obtained and thus fewer variations in electrical conductivity (surface and volume) across the substrate.

In part 4, silver plating was performed over knitted fabric. Plated fabric electrodes were used for the fabrication of TrEG self-powered device applications. The uniform and dense layer of metal was deposited on the fabrics. Then, the silicon rubber and rabbit fur were used as triboelectric materials in combination with plated conductive fabric electrodes and their energy performance was investigated under the mechanical stretching and pressing actions of human body movements (i.e. elbow and foot).

In this way, the presented research work described the various methods for surface metallization of fabrics. We produced more porous, less stiff and flexible conductive fabrics having extraordinary electrical conductivity and EMI shielding with antibacterial properties. The developed samples achieved interconnected network of conductive particles with thin and

uniform metal deposition. Moreover, the step for further electroless plating over the particles coated fabrics enhanced the rubbing, washing and electrical properties. The procedure of electroless plating was short routed (reduced a number of steps) free from hazardous fumes, less costly and simple. The promising applications of developed fabrics are EMI shielding, strain sensors and as electrodes for electrotherapy and energy harvesting.

7 CHAPTER: FUTURE WORK

- Investigation of electrical properties under different aging conditions (temperature, humidity, etc).
- Enhancement in electrical properties with different combinations of metal coating (in situ deposition, electroless plating, electroplating)
- Metallization of wool, glass and aramid fibers for different functional properties.
- Study of different triboelectric materials for energy harvesting
- Prevention of copper oxidation and improvement in durability of performance (by using capping agents, gums, binders, semiconductors, and alloys)
- Impact of thickness of metal coating on EMI shielding effectiveness, hardness and against anti-stabbing effect

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8. LIST OF PUBLICATION

8.1 Publications in Impact Factor Journals

[1] **Azam Ali**, Nhung. H.A Nguyen, Vijay Baheti, Munir Ashraf, Jiri Militky, Tariq Mansoor and S. Ahmad, "Electrical conductivity and physiological comfort of silver coated cotton fabrics," **Journal of textile institute.** vol. 109, pp. 1–9, 2017.

[2] **Azam Ali,** Vijay Baheti, Jiri Militky, Muhammad Zaman Khan, Veronika Tunakova, and Salman Naeem, "Copper coated multifunctional cotton fabrics," **Journal of industrial textile.** vol. 48, pp. 448–464, 2017.

[3] **Azam Ali**, Vijay Baheti, Jiri Militky, Muhammad Zaman Khan, "Utility of silver-coated fabrics as electrodes in electrotherapy applications," **Journal of applied polymer science**. vol. 135, pp. 46357, 2018.

[4] **Azam Ali**, Vijay Baheti, Jiri Militky, Muhammad Zaman Khan, Sayed Qamer Zia Gilani, "Comparative Performance of Copper and Silver Coated Stretchable Fabrics" **Fibers and Polymers**. vol. 19, 2018.

[5] **Azam Ali**, Vijay Baheti, Jiri Militky, Muhammad Usman Javaid, "Enhancement in ageing and functional properties of copper-coated fabrics by subsequent electroplating," **Applied physics A**, vol. 124, pp. 651, 2018.

[6] **Azam Ali,** Vijay Baheti and Jiri Militky, "Energy Harvesting Performance of Silver Electroplated Fabrics" **Journal of materials chemistry and physics of solids**, Vol. 231, pp. 33-40. 2019.

[7] **Azam Ali**, Vijay Baheti, Jiri Militky, Muhammad Zaman Khan, "Copper electroless plating of cotton fabrics after surface activation with deposition of silver and copper nanoparticles, copper and stannous chloride" **Journal of Physics and chemistry of solids**, Vol. 137, 2020.

[8] **Azam Ali**, Vijay Baheti, Jiri Militky, Muhammad Zaman Khan, and Guocheng Zhu, "Metal Coating on Ultrafine Polyester Non-woven Fabrics and Their Ageing Properties" **Fibers and Polymers**, Vol.20, pp.1347-1359, 2019.

[9] **Azam Ali**, Vijay Baheti, Jiri Militky, Muhammad Zaman Khan, "Development of electrically conductive composites based on recycled resources" **Journal of Textile Institute**, DOI: 10.1080/00405000.2019.1644121

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[12] Hafz Faisal Siddique, Adnan Ahmed Mazari, Antonin Havelka, Tariq Mansoor, **Azam Ali,** Musaddaq Azeem1, "Development of V-Shaped Compression Socks on Conventional Socks Knitting Machine," **Autex Research Journal** J. 2018.

[13] Tariq Mansoor, Hafiz Faisal Siddique, **Azam Ali** and Antonin Havelka, "Wrinkle free plaited knitted fabrics without pre-heat setting," **Journal of textile institute.** vol. 109, 2018.

[14] Sheraz Ahmad, M. Ashraf, **Azam Ali**, K. Shaker, M. Umair, A. Afzal, Y. Nawab, and A. Rasheed, "Preparation of conductive polyethylene terephthalate yarns by deposition of silver & copper nanoparticles," **Fibres and Textile Eastern Europe**. vol. 25, pp. 25–30, 2017.

[15] Salman Naeem, Vijay Baheti, Jiri Militky, and **Azam Ali**, “Multifunctional polylactic acid composites filled with activated carbon particles obtained from acrylic fibrous wastes,” **Polymer. Composites**. vol. 40, 2017.
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[17] Muhammad Zaman Khan, Vijay Baheti, Munir Ashraf, Tanveer Hussain, **Azam Ali,** Amjed Javid, Abdur Rehman" Development of UV Protective, Superhydrophobic and Antibacterial Textiles Using ZnO and TiO2 Nanoparticles,” **Fibers and Polymersˇ**., 2018. vol 19, PAGE 1647
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8.2 Publications in International Conferences

[1] **Azam Ali**, Electrically conductive textile sensors made by silver and copper nanoparticles” Oral presentation at the “International Conference on Advances in Functional Materials in UCLA (AAAFM-UCLA), 2019 at the University of California, Los Angeles, USA
[2] **Azam Ali**, Blanka Tomkova, Vijay Baheti, Jiri Militky, Musaddaq Azeem, Study the functional properties of silver nanoparticles coated fabric
91st, Textile Institute World Conference, Leeds UK, 2018
[3] **Azam Ali**, Vijay Baheti, Abdul Jabbar, Sundaramoorthy Palanisamy, Jiri Militky, Effect of jute fibre treatment on moisture regain and mechanical performance of composite materials AUTEX 2017, Greece
[4] **Azam Ali**, VeronikaTunakova, Vijay Baheti, Jiri Militky. Preparation of Conductive Yarns by Deposition of Silver & Copper Nanoparticles
95th International Conference on Innovative Engineering Technologies (ICIET) Rawalpindi, Pakistan, 29th-30th May, 2017
[5] **Azam Ali**, Vijay Baheti, Jiri Militky. Metallized Textile with Copper and Silver particles, STRUTEX 2018
[6] **Azam Ali**, Vijay Baheti, Jiri Militky. Copper and silver coated textile for smart applications, 47th Textile Research Symposium, 17 – 19 June 2019, Liberec

[7] Muhammad Zaman Khan, Vijay Baheti, Jiri Militky, **Azam Ali**, Sajid Faheem, Development of Multifunctional Polyester fabrics, functionalize with TiO2 nano particles AUTEX 2018, Turkey, ID no 3194

[8] Hafiz Faial Siddique, Adnan Mazari, Antonin Havelka, **Azam Ali**, Improve Hydrophobic Analysis of Nanofilament polyester fabric AUTEX 2018, Turkey

[9] Musaddaq Azeem, Blanka Tomkova, Jakub Wiener, **Azam Ali**, Thermal and Tactile Comfort of Nanofilament Fabric
91st, Textile Institute World Conference, Leeds UK, 2018

[10] Abdul Jabbar, **Azam Ali**, Muhammad Usman Javaid, Jiri Militky, Investigation of mechanical and thermomechanical properties of nanocellulose coated jute/green epoxy composites AUTEX 2017, Greece

[11] Sundaramoorthy Palanisamy, Veronika Tunakova, **Azam Ali**, Jiri Militky, Study on effect of moisture content and electromagnetic shielding effectiveness of cotton knitted fabric treated with various liquid media. 9th Central European Conference 2017

[12] Sundaramoorthy Palanisamy, Veronika Tunakova, **Azam Ali**, Jiri Militky, Daniel Karthik Study on textile comfort properties of polypropylene blended stainless-steel woven fabric for the application of electromagnetic shielding effectiveness
AUTEX 2017, Greece

8.3 Book Chapters

[1] **Azam Ali,** Vijay Baheti, Jiri Militky "Metal coated multifunctional fabrics" Recent trends in fibrous material sciences, volume 5, page 392, ISBN 978-80-7494-493-2 (2019)

[2] Salman Naeem, Sayed Qammar Zia, Vijay Baheti, Jiri Militky, **Azam Ali** "Electrical Conductivity of PLA Films Reinforced with Carbon Nano Particles from Waste Acrylic Fibers" Advances in Natural Fibre Composites, Springer (2018)

[3] Abdul Jabbar, Jiri Militky, **Azam Ali** "Investigation of Mechanical and Thermomechanical Properties of Nanocellulose Coated Jute/Green Epoxy Composites" Advances in Natural Fibre Composites pp 175-194, Springer (2018)

[4] Vijay baheti, Jiri militky, **Azam Ali** "Production of activated carbon particles at optimized carbonization conditions, Recent trends in fibrous material sciences volume 5, page 372, ISBN 978-80-7494-493-2 (2019)

Citations

Article 1: Azam Ali, Nhung. H.A Nguyen, Vijay Baheti, Munir Ashraf, Jiri Militky, Tariq Mansoor and S. Ahmad, "Electrical conductivity and physiological comfort of silver coated cotton fabrics," **Journal of textile institute.** vol. 109, pp. 1–9, 2017.

[No of citations = 15]

Article 2: Azam Ali, Vijay Baheti, Jiri Militky, Muhammad Zaman Khan, Veronika Tunakova, and Salman Naeem, "Copper coated multifunctional cotton fabrics," **Journal of industrial textile.** vol. 48, pp. 448–464, 2017.

[No of citations = 14]

Article 3: Azam Ali, Vijay Baheti, Jiri Militky, Muhammad Zaman Khan, "Utility of silver-coated fabrics as electrodes in electrotherapy applications," **Journal of applied polymer science**. vol. 135, pp. 46357, 2018.

[No of citations = 10]

Article 4: Azam Ali, Vijay Baheti, Jiri Militky, Muhammad Zaman Khan, Sayed Qamer Zia Gilani, "Comparative Performance of Copper and Silver Coated Stretchable Fabrics" **Fibers and Polymers**. vol. 19, 2018.

[No of citations = 9]

Article 5: Azam Ali, Vijay Baheti, Jiri Militky, Muhammad Usman Javaid, "Enhancement in ageing and functional properties of copper-coated fabrics by subsequent electroplating," **Applied physics A**, vol. 124, pp. 651, 2018.

[No of citations = 4]

Article 6: Azam Ali, Vijay Baheti and Jiri Militky, "Energy Harvesting Performance of Silver Electroplated Fabrics" **Journal of materials chemistry and physics of solids**, Vol. 231, pp. 33-40. 2019.

[No of citations = 2]

Resume

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Vision and skills

2 years of industrial experience in wet-processing department of textile. An enthusiastic, adaptive and fast-learning individual with a broad and acute interest in the synthesis mechanisms of nanomaterials (by sol gel process, insitu deposition, vacuum and arc spraying, electroless and electroplating method). Analytical and surface characterization, development of functional and multifunctional textiles, smart textiles, hydrophobic textiles, fabric-based electrodes for electrotherapy and energy harvesting. Making carbon and graphite particles by carbonization of waste materials and making the composites by hand layup, vacuum bagging and resin transfer method. I particularly enjoy collaborating with scientists of different disciplines to develop robust skills and solve new challenges attain professional excellence in dynamic and challenging organization by actively exercising my unique skills and abilities and bringing to bear my integrity.

Education

Ph.D. (Textiles & Materials Engineering) 2015-continue Technical University of Liberec, Czech Republic **Dissertation topic:** Surface deposition of metals on textile structures **Characterization tools:** XRD, FTIR, SEM, EDX, ICP-AES, Electrical and thermal conductivity, EMI shielding Antibacterial test, Air and Water vapor permeability, Ageing **Major subjects**: Nanotechnology, Advance materials, Analytical techniques, Smart materials, Metrology, Textile raw materials and chemistry, Statistical analysis, Heat and mass transfer. Science for making composites. **Research Area:** Nano-particles preparation. Analytical characterization. Electrically conductive polymers and textiles, conductive composites, Development of flexible electrodes for electrotherapy, medical devices and energy harvesting (Tribo-electrification and Piezo-electricity method)
M.Sc. (Textiles Engineering) 2012-2014 National Textile University, Faisal Abad, Pakistan **Dissertation topic:** Development of natural fiber hydrophobic composites **Characterization tools:** SEM, EDX, Tensile testing, Impact testing, ageing **Major subjects**: Technical textile, Medical textile, Research methodology, Non-woven, High-performance fibers, Analytical techniques, protective textiles, nanotechnology, Smart textile, composites fabrication
B.Sc. (Textiles Engineering) 2006-2010 The University of faisalabad, Faisal Abad, Pakistan **Dissertation topic:** To study the effect of catalyst concentration and curing conditions on resin finish **Characterization tools:** SEM, EDX, Tensile testing, Impact testing, crease recovery **Major subjects**: Nano materials finishing, Applied chemistry, Polymer chemistry, Textile Dyeing, Textile printing, Textile finishing, Textile raw materials, Applied mathematics, Textile testing, Applied physics, Applied chemistry, Electrical engineering, Electronics, Hydraulic pressure, Thermodynamics etc.
F.Sc. GC University Faisalabad, Pakistan **Main Subjects:** Physics, Chemistry, Math

Summary: Experience: Textile Products Specialist (Textile Engineering)

2 years of industrial experience in wet-processing department of textile.

6 years of experience as a Researcher.

PROFESSIONAL EXPERIENCE:

Proficiencies:

Organization: Sitara Textile Mills [6 months]
Designation: Training engineer

Organization: Arif Textile Mills [2½ year]
Designation: Assistant Manager
Time dedicated: September 2010~ January, 2012

- Textile pretreatments
- Dyeing
- Finishing
- Trouble shooting and mechanical maintenance supervision

Organization: National Textile Research Center (NTRC), NTU, Pakistan [2 Years]

Designation: Research Associate
Time dedicated: 2013 ~ September, 2015

- Preparation of
  - Nanoparticles of Copper, Silver, Zinc and Magnesium, Magnesium hydroxide
  - Micro particles of carbon and graphite from waste materials
  - Multifunctional textiles (hydrophobic, oil and water repellency)
  - Flame retardant fabrics (by making and imparting magnesium oxide and magnesium hydroxide nanoparticles)
  - Antibacterial fabrics
- Development of
  - Electrically conductive yarns and textiles by imparting metal particles
  - Making electrically conductive polymers and stretchable fabrics by imparting carbon based fillers
  - Stretchable electrodes
- Development of
  - Electrically conductive green composites
  - Hydrophobic natural fiber reinforced composites
  - Shape memory composites
- Carbonizing acrylic and Kevlar waste materials to make electrically conductive fillers
- Working on ageing properties of composites

Additional Expertise In Subjects

- Smart textile
- Composites
- Carbonization to make carbon fibers
- Heat and mass transfer through Porous Media
- Clothing Comfort
- Textile Fibrous material Properties
- Textile processing
- Statistical Data analysis (Regression Analysis using MINITAB)
- Sound knowledge of data analysis using Software: MINITAB, ORIGIN Pro, MS Word, MS Excel and MS Power Point, Python (Basics only), Irfan View, ImageJ, Chem sketch window, Blender Animation.

RESEARCH SCORE

- Research gate score **20.46**
- h-index **7**

AWARD and MEMBERSHIPS:

- Best poster award INTERNATIONAL PH.D. STUDENTS DAY, within workshop for PhD students. 12 November, 2019, Liberec, Czech Republic
- Membership of Pakistan Engineering Council

LANGUAGE PROFICIENCY:

URDU: Native Speaker
ENGLISH: First Language (CEFR Level C2), IELTS: Academics clear
HINDI : Speak well

GERMAN and CZECH: Learning at my own

INTERNATIONAL TOURS:

- 10 days stay in University of California Los Angles, USA
- 10 days stay in Greece for international conference AUTEX
- 10 days stay in Leeds for 91st, Textile Institute World Conference, Leeds UK
- Small tours regarding research project in France and Sweden
- Small tours regarding research project in Poland

INTERESTS AND ACTIVITIES:

- Scientific Novelties
- Habitual to play football and cricket
- Exploring advance ideas in Textile materials and Finishes.
- Event organizer for conferences