ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL

DEVELOPMENT OF STRETCHABLE CONDUCTIVE FABRIC THROUGH DIFFERENT METALS COATING APPROACH FOR E-TEXTILE APPLICATIONS

Ph.D. THESIS

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İSTANBUL TEKNİK ÜNİVERSİTESİ★LİSANSÜSTÜ EĞİTİM ENSTİTÜSÜ

E-TEKSTİL UYGULAMALARI İÇİN FARKLI METAL KAPLAMA YAKLAŞIMIYLA UZAYABİLİR İLETKEN KUMAŞ GELİŞTİRİLMESİ

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Zuhaib HASSAN, a Ph.D. student of ITU Graduate School student ID 503122807, successfully defended the thesis/dissertation entitled "DEVELOPMENT OF STRETCHABLE CONDUCTIVE FABRIC THROUGH DIFFERENT METALS COATING APPROACH FOR E-TEXTILE APPLICATIONS", which he prepared after fulfilling the requirements specified in the associated legislations, before the jury whose signatures are below.

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To my parents and spouse,

FOREWORD

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June 2022 Zuhaib HASSAN
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ABBREVIATIONS

Acronyms	: Description
ASTM	: American Society for Testing and Materials
AATCC	: American Association of Textile Colourists and Chemist
EMI	: Electromagnetic Interference
EDX	: Energy Dispersive X-ray Spectroscopy
ESD	: Electrostatic Discharge
FTIR	: Fourier Transformed Infrared
GSM	: Gram per square meter
ICP-AES	: Inductive Couple Plasma-Atomic Emission Spectroscopy
PET	: poly(ethylene terephthalate)
PDA	: Polydopamine
PPm	: Parts Per Million
SEM	: Scanning Electron Microscopy
XRD	: X-ray Diffractometry

SYMBOLS

ρV	: Volume Resistivity
ρs	: Surface Resistivity
Rs	: Surface Resistance
RV	: Volume Resistance
Ω.cm	: Resistivity
S/cm	: Conductivity
Wt.%	: Weight percentage
g/m^2	: Density
MHz	: Mega hertz
GHz	: Gega hertz

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DEVELOPMENT OF STRETCHABLE CONDUCTIVE FABRIC THROUGH DIFFERENT METALS COATING APPROACH FOR E-TEXTILE APPLICATIONS

SUMMARY

This study aims to manufacture and characterize various types of conductive (cotton knitted and polyester knitted) fabrics. The fabrics were prepared through the electro-less copper metal coating approach. The effect of copper electroleess plating on different types of fabric structures and on different crossections of yarns was also observed. The main response of prepared fabric was electrical conductivity, EMI shielding and their durability against washing and rubbing. The current research activity has multiple benefits considering extensive comparison with other studies. Previously electroless plating has been performed by different salts and reducing agents' combinations over different fibres. However, there were no study available to metallize the fabric structures having different GSM and different cross section of fibres. Secondly, we have also studied the effect of different fibrous cross-section (round, hollow round, W shape and octolobel) against copper electroless plating.

At first, nine-combed cotton knitted fabric samples with different yarn fineness and elastane percentage were selected in order to see effect of these parameters on electrical conductivity and physical properties of samples such as an increase in weight and thickness, impact of washing cycles and abrasion resistance on the electrical conductivity of the fabric sample. The surface morphology of all the knitted cotton fabric samples were also explored before and after the coating method via scanning electron microscope (SEM) and it showed a remarkably uniform deposition of copper on the fabric surface. the energy dispersive spectroscopy (SEM-EDX) was performed to determine the coated material content on the surface of the fabric after the metal coating process. The utility of conductive fabrics was analyzed for electromagnetic shielding ability over frequency range of 30 MHz to 1.5 GHz. The electrical conductivity and amount of metal deposition was found to be higher for the fabric samples having less GSM and higher cotton percentages in their structures. The results revealed that knitted cotton fabric of 5% elastane with the finer yarn count (Ne=40/1) showed lowest resistivity (3.24 Ω.cm) as compared to the other knitted cotton fabric of 10% elastane with a finer count (Ne=40/1) or 5% elastane with coarser (Ne=30/1). The increase in elastane content into fabric structure also influences the fabric stretchability.

The objectives of **second part of the study** were, to carry out research with the best performing three samples obtained from the first part of the study and three single jersey knitted cotton fabric samples were selected. The selected fabric samples with GSM (136, 154 and 176 GSM) out of nine fabric samples were used in this part. These fabric samples provided the lowest value of electrical resistivity coupled with high EMI shielding and more contents of metal particles. Thereafter, the selected

samples were **pre-treated** with laser to enhance the surface roughness, then electroless plating was performed in order to see the impact of roughness on copper deposition. This section of the research work addresses the development and characterization of conductive cotton fabrics treated with lasers in context of copper (Cu) metallization methodologies. The abrasion resistance, thickness, and durability of the laser-treated knitted cotton fabric samples were investigated. Additionally, samples exhibited exceptionally consistent deposition of Cu nanoparticles on the surface of cotton fabric when the surface morphology of the laser-treated surfaces was examined by employing the scanning electron microscope (SEM) both before and after the coating procedure. To assess the elemental analysis on the surface of the treated samples following the electroless metallization process, an energy dispersing spectroscopy (SEM-EDX) examination was performed. This section of the study indicated that fabric samples that had been laser-treated outperformed untreated fabric samples in terms of wear resistance. Abrasion resistance being one of the significant features in electric textile applications, laser-treated samples might thereby be the best options.

The **third part** of the study was the development and characterization of conductive textured and non-textured polyester fabrics with different cross-sections. The electroless copper plating method was selected to impart conductivity on fabric structures. The deposition of copper nanoparticles on textured and non-textured polyester fabrics was characterized by electrical conductivity, electron scanning microscopy (SEM), microscopic morphology, and energy dispersive X-ray spectroscopy (EDX). SEM images revealed a uniform copper nanoparticle coating of a thin film on textured and non-textured polyester fabrics. The properties of conductive textured polyester fabrics were compared in terms of electrical conductivity, wear resistance, thickness and durability with non-textured conductive polyester fabrics. Structural studies showed that the crystalline surface of the textured and non-textured polyester fabric structure is not affected by electroless metallization. Conductivity studies have shown that textured (lowest resistivity 2.18 Ω.cm) and non-textured (lowest resistivity 76.39 Ω.cm) polyester fabrics have good electrical conductivity. When the durability of conductive textured and non-textured polyester fabrics was examined against washing and rubbing fastness, the textured polyester fabrics showed good retention of copper nanoparticles by maintaining their electrical conductivity level after 250 abrasion cycles. Furthermore, resistivity analysis was also carried to study the effect of copper metallization and conductivity against different morphological structures of fibres. It was observed that there are lower values of electrical resistivity for each coated sample. The resistivity was found to be lowest for hollow round coated fibres (either textured or non-textured). The behaviour of metal deposition for hollow round fibers and electrical conductivity was further justified from the SEM analysis. The W shape fibers showed less amount of metal deposition and higher electrical resistivity values as compared to all. The final applications of developed copper plated fabrics are in the field of smart textiles, sensors, stretchable actuators, EMI shielded panels and stretchable electrodes.

Keywords: Conductive textiles, Electroless plating, Copper coating, Electromagnetic interference shielding, sensors and actuators, Stretchable conductive fabrics, Smart textiles, metal coatings, textured polyester fabrics, Metal coated, Cross-sectional fiber.

E-TEKSTİL UYGULAMALARI İÇİN FARKLI METAL KAPLAMA YAKLAŞIMIYLA UZAYABİLİR İLETKEN KUMAŞ GELİŞTİRİLMESİ

ÖZET

Bu tez çalışması kapsamında akımsız bakır kaplama yöntemi kullanılarak çeşitli iletken kumaş tiplerinin üretimi ve karakterizasyonu amaçlanmıştır. Bu amaçla farklı elastan oranlarına sahip pamuklu süprem kumaşlar ve farklı kesitlerde tekstüre ve tekstüre olmayan polyester örme kumaşlar kullanılmıştrır. Böylece , bakır kaplamanın farklı kumaş yapıları ve farklı iplik kesitleri üzerindeki etkisi incelenmiştir. Bu yöntemle kaplanan kumaşların elektriksel iletkenlik değerleri, EMI zırhlama, yıkama ve sürtünmeye karşı dayanıklılıkları test edilip ölçülmüştür. Mevcut çalışmanın, literatürdeki diğer çalışmalarla kapsamlı bir karşılaştırması yapıldığında öne çıkan iki özgün değeri bulunmaktadır. Bunlardan birincisi; akımsız kaplamının farklı gramaj değerlerine sahip kumaş yapıları üzerindeki eletro mekaniksel etkilerinin incelenmesidir.İkinci olarak ise farklı lifli kesitlerin (yuvarlak, içi boş yuvarlak, W shape ve octolobal) bakır akımsız kaplamaya karşı etkisinin incelenmesidir.

İlk olarak farklı iplik inceliği ve elastan yüzdesine sahip dokuz adet penye pamuklu örme kumaş numuneleri akımsız kaplama işlemi ile kaplanarak, kumaş ağırlığı , kalınlık ve elastan oranı gibi parametrelerin, numunelerin elektro mekanik özelliklerine etkisi incelenmiştir. Daha sonra farklı sayıdaki yıkama işlemlerinin ve aşınma gibi faktörlerin kumaş üzerine kaplanan bakır miktarına ve dolayısıyla kaplanmış numunelerin elektriksel iletkenliği ve fiziksel özelliklerine etkisi araştırılmıştır.

Akımsız kaplama işlemi öncesi kumaş numuneleri non-iyonik deterjanla 20°C sıcaklıkta 40 dakika süresince yıkanmıştır. Daha sonra numunelere 5g/l Tin (II) klorit ve 5 ml HCL solüsyonunda 25° Csıcaklıkta 10 dakika süresince 1 Ph değerinde sensitizasyon işlemi uygulanmıştır.Kumaş yüzeylerinin aktifleştirme işlemi ise kumaş numunelerini 10g/l gümüş nitrat ve 10 ml/l amonyum solüsyonunda 25°C sıcaklıkta 10 dakika daldırma işlemiyle gerçekleştirilmiştir. Bakır nanoparçacıkların kaplama işlemi ,kumaşların 10g/l Bakır(II) sülfat pentahidrat 45g/l potassium sodium tartrate ($KNaC_4H_4O_6 \cdot 4H_2O$),20ml/l formaldehid (HCHO) solüsyonu ve 10g/l sodium hidroksit solüsyonunda 30 C sıcaklta 20 dakika ve 11 pH değerinde işlem görmesiyle gerçekleştirilmiştir. Araştırmada kullanılan bütün kumaş numunelerine aynı reçete ve prosesler uygulanmış ve bakır nanoparçacıklarının akımsız yöntemle kumaş yüzeylerini homojen şekilde kaplaması sağlanmıştır.

Bakır kaplama işlemi sonrasında bütün örme kumaş numunelerinin yüzey morfolojisi taramalı elektron mikroskobu (SEM) ile kaplama uygulamasından önce ve sonra incelenmiştir. SEM incelemesi sonucu kumaş yüzeyinde düzgün bir bakır birikimi olduğu gözlemlenmiştir. Metal kaplama işleminden sonra kumaş yüzeyindeki kaplanmış malzeme içeriğini belirlemek için enerji dağıtıcı spektroskopisi (SEM-EDX) yapılmıştır. İletken kumaşların 30 MHz ile 1.5 GHz

frekans aralığında elektromanyetik kalkanlama özelliği analiz edilmiştir. Daha düşük kumaşa gramajına sahip olan ve elastan oranı daha düşük olan kumaş numunelerinde elektriksel iletkenlik ve metal biriktirme miktarı daha yüksek bulunmuştur. Sonuçlar, Ne=40/1 iplik numarasına sahip %5 elastanlı pamuklu örme kumaşın, Ne=40/1 iplik numarasına sahip %10 elastanlı pamuklu kumaşa ve Ne=30/1 iplik numarasına sahip %10 elastanlı pamuklu kumaşa göre daha az elektriksel direnç değeri (3.24 Ω.cm) gösterdiği bulunmuştur. Aynı numunelerin elektromanyetik kalkanlama testleri 30MHz ve 1.5GHz frekansları arasında gerçekleştirilerek elektromanyetik kalkanlama verimliliğinin düşük gramajlı kumaşlarda daha yüksek olduğu sonucuna varılmıştır.

Çalışmanın ikinci bölümünün kapsamını, kumaşların yüzey pürüzlülüğünün akımsız kaplamaya etkisinin incelenmesi oluşturmaktadır. Bu bağlamda, numuneler, yüzey pürüzlülüğünü arttırmak için lazer ile ön işleme tabi tutulmuş, ardından pürüzlülüğün kumaş yüzeyinde bakır birikimi miktarına etkisini görmek için akımsız kaplama işlemi uygulanmıştır..Bunun için çalışmanın ilk bölümünden elde edilen en iyi elektro mekaniksel performans gösteren üç süprem örme pamuklu kumaş numunesi seçilmiştir (136, 154 ve 176 gr/m^2 değerlerine sahip kumaşlar). Araştırmanın bu kısmı, bakır metalizasyon teknikleri açısından lazerle işlenmiş iletken pamuklu kumaşların üretimini ve karakterizasyonunu açıklamaktadır. Lazerle işlenmiş örme numunelerin fiziksel özellikleri kalınlık, dayanıklılık ve aşınma etkisi açısından incelenmiştir. Ayrıca, lazerle işlenmiş numunlerin yüzey topografyası, kaplama işleminden önce ve sonra bir taramalı elektron mikroskobu (SEM) kullanılarak analiz edilmiştir.Yapılan bu analizler, kumaşların yüzeyinde bakır metal nanoparçacıklarının dikkat çekici şekilde düzgün bir şekilde çökeldiğini göstermiştir. Akımsız metalizasyon yönteminden sonra numunenin yüzeyindeki element bileşimini belirlemek için bir enerji dağılım spektroskopisi (SEM-EDX) analizi yapılmıştır. Çalışmanın bu kısmı, lazerle yüzeyleri işlenmiş numunelerin, işlem görmemiş kumaş numunelerine kıyasla aşınmaya karşı daha iyi direnç gösterdiğini göstermiştir. Elektronik tekstil uygulamalarında aşınma direnci anahtar özelliklerden biri olduğundan, lazerle işlenmiş numuneler bu tür uygulamalar için ideal adaylar olabilirler.

Çalışmanın üçüncü bölümü, farklı kesit şekillerinde üretilmiş filamanlara sahip tekstüre işlemi uygulanmış ve tekstüre işlemi uygulanmamış polyester ipliklerle üretilen polyester kumaşların akımsız kaplama sonucu iletken hale getirilmesi ve bu parametrelerin numunelerin elektro mekaniksel özelliklerine etkisinin incelenmesini kapsamaktadır.. Bakır nanoparçacıkların tekstüre işlemi uygulanmış ve uygulanmamış numunler üzerindeki kaplama performansı, elektriksel iletkenlik değerlerinin ölçülmesi, SEM ve EDX analizi ile karakterize edilmiştir. SEM görüntüleri sonucu tüm polyester kumaşlar üzerinde ince bir filmin tek tip bakır nanoparçacık kaplamasını ortaya çıkardığı gözlemlenmiştir.Ayrıca, iletken numuneler elektriksel iletkenlik, aşınma direnci, kalınlık ve dayanıklılık açısından karşılaştırılmıştır. Yapısal çalışmalar polyester kumaş yapısının kristal yüzeyinin akımsız metalizasyondan etkilenmediğini göstermiştir. İletkenlik çalışmaları, tekstüre işlemi uygulanmış polyester ipliklerle örülen kumaşlarda (en düşük direnç 2.18 Ω.cm) ve tekstüre işlemi uygulanmayan polyester ipliklerle örülmüş kumaşlarda (en düşük direnç 76.39 Ω.cm) değerleri, polyester kumaşların iyi elektriksel iletkenliğe sahip olduğunu göstermiştir. Tekstüre işlemi sonucunda polyester iplikler daha hacimli (lifleri birbirine dolaştırılarak daha pürüzlü) bir iplik yapısı oluşturdukları için bu ipliklerle örülmüş kumaşların yüzeyleri de tekstüre olmayan polyester ipliklerle örülen kumaşlara göre daha pürüzlü kumaş yüzeyleri

oluşturmaktadır. Bunun sonucu olarak, bakır nanoparçacıklar kaplama işlemi sırasında tekstüre kumaşların pürüzlü yüzeylerinde daha fazla birikerek bu kumaşların elektriksel iletkenliğini artırmaktadır (dolayısıyla elektriksel direnç değerleri düşmektedir)..Elde edilen iletken tekstüreli ve tekstüresiz polyester kumaşların yıkama ve sürtünme haslıklarına karşı dayanıklılıkları incelendiğinde, tekstüre polyester kumaşların 250 aşınma döngüsünden sonra bile elektriksel iletkenlik seviyelerini koruduğu bulunmuştur.. Ayrıca, bakır kaplamının ve elektriksel iletkenliğin liflerin farklı morfolojik yapılarına karşı etkisini incelemek için farklı kesitlere (içi boş yuvarlak, yuvarlak, oktolobal, w-şekil) sahip tekstüre ve tekstüre olmayan ipliklerle örülmüş kumaşların bakır kaplama işleminden sonra elektriksel direnç ölçümleri de yapılmıştır. Elektriksel direnç değerlerinin, içi boş yuvarlak kaplanmış lifler (tekstüreli veya tekstüresiz) için en düşük olduğu bulunmuştur. W kesitli lifler, tümüne kıyasla daha az miktarda metal biriktirme ve dolayısıyla daha yüksek elektriksel direnç değerleri göstermiştir. SEM analizleri sonucunda içi boş yuvarlak liflerin kesit şeklinin özellikle tekstüre işleminden sonra yassılaştığı, bu formuyla liflerin üzerinde daha fazla bakır parçacıklarının birikmesine neden olduğu sonucuna varılmıştır. W kesitli liflerde ise lif kesitindeki düzensiz şekil nedeniyle daha az metal birikmesi oluşmuş ve dolayısıyla elektriksel iletkenlik değerleri diğer kesitlere sahip ipliklerle örülen kumaşlardan en düşük (elektriksel direnç değerleri en yüksek) elde edilmiştir. Bu gruptaki kumaşların elektromanyetik kalkanlama verimlilik sonuçları incelendiğinde en iyi değerin 600MHz ve 1500MHz frekansları arasında 73 dB ile içi boş yuvarlak tekstüre ipliklerle örülmüş kumaşların gösterdiği sonucuna varılmıştır.
Sonuç olarak e-tekstil uygulamalarında kullanılabilecek elektriksel iletkenlik değerlerine sahip olan iletken kumaşlar akımsız metalizasyon yöntemi ile geliştirilmiş ve bakır nanoparçacıklarla farklı yüzey özelliklerine ve esneklik değerlerine (uzama değerlerine) sahip kumaşlar homojen şekilde kaplanmıştır. Elde edilen elektriksel iletken kumaşların aşınmaya karşı dayanıklılıklarının yüksek olması elektronik tekstil uygulamalarında önemli bir avantaj olarak ortaya çıkmaktadır.

Anahtar Kelimeler: İletken tekstiller, Akımsız kaplama, Bakır kaplama, Elektromanyetik parazit koruması, sensörler ve aktüatörler, Gerilebilir iletken kumaşlar, Akıllı tekstiller, metal kaplamalar, dokulu polyester kumaşlar, Metal kaplı, Kesitsel elyaf.

1. INTRODUCTION

Research in the field of functional textiles has recently gained prominence, as a result of its use in a variety of sophisticated advanced materials (Ali et al, 2021) thus a wide range of functional textile materials including solar textiles (reflective and absorbing), shape-memory textile materials, moisture-permeable textiles, antibacterial and UV-protective textiles, conductive textiles, and color-changing fabrics have been produced (Ali et al, 2021). Conductive textiles, in particular, have become one of the most active and fast-growing areas due to their unique and novel implementation of wearable and flexible electronics, heat-generators, sensors and actuators, EMI shielding, etc (Ashayer-Soltani et al, 2016).. Textiles made from intrinsically naturally occurring conductive yarns and those specifically modified to induce conductivity are the two main methods of imparting electrical conductivity to textiles. Electrically conductive materials, such as metals, can be spun directly into naturally conductive yarns(Ahmad et al, 2017). The surface metallization (Ag, Ni, Al, Cu, etc.), chemical coatings, deposition of conducting fillers (carbon black or carbon nanotubes, etc.), and coating of conducting polymeric materials including polythiophene, polyaniline (Kim et al. 2006), and polypyrrole(Maity et al. 2014), etc, could all be utilized for imparting electrical conductive properties on textile surfaces (B. Kim et al, 2006) (Maity et al, 2014). However, there are limitations in each technique of metallization. Such as, the conductive textiles developed by integrating metallic wires lost their comfort characteristics (Liu et al, 2010). Textiles coated with conductive inks, conductive polymers, and chemical doping have tendency to loss their electrical conductivity due to rubbing and washing (Coessens et al, 2001). As a result, various types of textile fabrics are metalized in this research work by using electroless plating approach (Yamashita et al, 2014).

The electroless plating technique was selected to deposit the copper metal over the textile materials, and it is relatively cheaper compared to other available options for making the conductive fabric (Molina et al, 2015). The effect of different fibrous cross-section (round, hollow round, W shape and octolobel) against copper electro less plating has been investigated, which is the major novelty of the current research work. Secondly, the effect of different GSM of fabrics and the effect of pre-treatments against metal deposition was evaluated. Flexible and stretchable electrodes as well as sensors were fabricated using a conventional electroless plating method on knitted stretchable fabric samples.

Copper coated fabrics should have good electrical properties even in stretched form. Hence, the fabrics will be better replacement for the carbon rubber-based hard electrodes (use in electrotherapy, EMG, ECG etc.) (Keller and Kuhn, 2008).

1.1 Research Goal and Study Objectives

The metal coated stretchable fabrics have received considerable interest in the field of sensors and flexible electrodes. The main aim of study was to develop the electrically conductive stretchable sensors. The polyamide fiber is well known due to its stretch and recovery behavior. Polyamide with elastane adjust easily with the body's natural movements, but the original shape is recovered when it is allowed to relax. Fabrics containing spandex yarn have a wide application value, especially because of their increased extensibility, elasticity, the high degree of recovery, good dimensional stability and simple care (Marmaralı, 2003) (McIntyre et al, 2005) (Tezel and Kavuşturan, 2008).

At first, the poliamid knit fabrics with elasthane were selected and treated with plasma. Subsequently, the copper electroless plating was performed to make them electrically conductive. The fabrics were characterized and examined by the Scanning Electron Microscope (SEM). The changes in surface roughness and etching effect was observed. SEM analyses showed that the surface roughness increased due to the etching effect of the plasma treatment. The electrical conductivity was also examined. The fabrics remained poor to show the good electrical conductivity. Their minimum electrical resistivity was recorded in the range of 84,000 Ω.cm. The SEM analysis of copper deposition on the fiber surfaces were also examined. Where the fibers were showing insufficient coating. Actually, the reason is the polyamide fabric was denser, elastic and thick. The structures of the fabric materials have an impact on the conductivity as well. The structural characteristics of the polyamide fabric may be the cause of this outcome. The activation and plating stages of the plating procedure result in weak interconnectivity between the conductive regions of the fabric sample because the plating solution cannot penetrate deeply within the microstructure of the polyamide fabric.

Hence, it was decided to carry out further study on polyamide blended knitted fabrics. Knitted fabrics are widely used in apparel products for their extraordinary properties like elasticity, extension, higher strength, better solvent, flame and heat resistance and hand and wearing comfort for whole the year and seasons. Moreover, a knitted fabric has always elastic properties even without elastane.

The further study was aimed to manufacture and characterize various types of conductive (cotton knitted and polyester knitted) fabrics. The fabrics were prepared through the electro-less copper metal coating approach. The effect of copper electroless plating on different types of fabric structures and on different cross section of yarns was also observed. The main response of prepared fabric were electrical conductivity, EMI shielding and their durability against washing and rubbing.

At first (first part of the study), to produce conductive textile materials employing combed knitted cotton fabric with elastane and examine the impact of fabric characteristics on the electrical and physical features of fabrics, copper was deposited using an electroless plating technique. The characterisation of conductive knitted combed fabric samples and the best conductive cotton fabric for subsequent electronic textile applications are both important subjects covered in this study. In this study, nine different types of GSM fabrics were used (ranging from134 to 202 GSM). It was observed that the fabric with lower GSM (136, 154 and 176 GSM) provided the lower electrical resistivity.

In second step (second part of the study), three fabric samples having light GSM (136, 154 and 176 GSM) out of nine fabric samples as mentioned in **the first part of the study** were selected**.** These fabric samples provided the good results of electrical conductivity, EMI shielding and more contents of metal particles. In this work, Prior to the conductive coating procedure, the surfaces of all fabric sample were processed with an oxygen (O_2) laser to improve the surface roughness of fabric and enhance the durability of the substrates for the metal-coated fabrics. The fabric substrate was then coated with copper particles that were deposited using electroless methods. Owing to their excellent penetration across the surface of the substrates that have been laser-treated, the base of Cu nanoparticles offers a homogeneous metal deposition. After electroless plating, the effectiveness of each laser-treated fabric sample was evaluated in terms of its physical characteristics and electrical conductivity. The purpose of deposition the copper over knitted fabric was to find the suitable application in the field of stretchable sensors, electrodes and panels.

Furthermore, the developed conductive fabrics are quite flexible and stretchable, so fulfill the demand of comfort. Moreover, electrodes also provide good washing fastness and avoid from cracks regarding stretching and rubbing action.

Third step (third part of the study) was to perform the same electroless plating recipe on polyester fabric. In fact, for hosiery, apparel, and textile applications, the cotton (among natural fibers) and polyester (among synthetic fibers) are most commonly used. They are also blended in different ratio to use them in number of applications. So, the same recipe of electroless plating was applied on the synthetic fiber polyester. The polyester fibers (textured and non-textured) were selected for this purpose. The copper nanoparticles were deposited over the polyester fabric surfaces via electroless metal coating. The base of copper nanoparticles is sufficiently dense, homogeneous and uniform metal coating throughout the textured and non-textured polyester fabric surfaces. The thickness and electrical resistivity deviations are avoided due to the redox potential of copper nanoparticles in the electroless metallization method. The current study focuses on the advancement of electrically conductive of polyester fabrics without losing their physical and comfort characteristics. The performance of all the textured and non-textured polyester fabric samples after the electroless metallization was observed by different tools like electrical conductivity, thickness, SEM images, EDX analysis, microscopic morphology, rubbing fastness and durability. The research work was divided into following **four parts**.

1.1.1 Copper electroless plating over cotton knitted fabric (9 different GSM)

- Pretreatment of substrate (cotton and elastane) fabric with sodium hydroxide
- Electroless deposition of copper metallic particles over the substrate
- Examine the effect of nine different GSM fabrics (light and heavy weight GSM) on metal deposition and electrical properties
- Morphological characterization of coated fabrics scanning electron microscope (SEM) and EDX
- Confirmation and count of metal particles deposition by X-ray diffraction (XRD) and ICP techniques
- Estimation of EMI shielding and electrical conductivity
- Examination of durability of performance against washing and abrasion resistance
- Development of stretchable conductive sensors for e-textile applications

1.1.2 Copper electroless plating over cotton knitted fabric (3 different GSM) with laser pre-treatment

- To investigate the effect of laser treatment on copper plating deposition.
- Electro less deposition of copper metallic particles over the substrate
- Investigate the effect of three different GSM (light weight GSM) fabrics on metal deposition and electrical properties.
- Morphological characterization of coated fabrics scanning electron microscope (SEM) and EDX
- Confirmation and count of metal particles deposition by X-ray diffraction (XRD) and ICP techniques
- Estimation of EMI shielding and electrical conductivity
- Examination of durability of performance against washing, and abrasion resistance

1.1.3 Electroless plating of copper over textured non-textured polyester fabric having different correctional yarns

- To investigate the effect of textured and non-textured polyester fabrics on metal deposition and electrical properties.

- To investigate the effect of metal deposition on various correctional structures in terms of electrical conductivity.
- Electro less deposition of copper metallic particles over the substrate polyester
- Morphological characterization of coated fabrics scanning electron microscope (SEM) and EDX
- Confirmation and count of metal particles deposition by X-ray diffraction (XRD) and ICP techniques
- Assessment of EMI shielding and electrical conductivity
- Examination of durability of performance against washing, abrasion resistance
- Development of stretchable conductive sensors for e-textile applications

1.1.4 Comparative study regarding electrical and functional properties among all previous parts (study1, study2, and study3) and also with literature

- To investigate the effect of pre-treatment on metal deposition and electrical properties.
- To compare the results regarding amount of metal contents
- To compare the results about EMI shielding and electrical resistivity
- To compare the washing, rubbing and other durable properties

2. LITERATURE REVIEW

Textiles are metalized for a variety of reasons, including electrical conductance, electromagnetic shielding behaviour, antimicrobial properties, radar and UV reflection, anti-stabbing, superior antistatic properties, insulation, and an aesthetic and bright metallic effect. This chapter examines the literature for conductive textile materials, basic materials used to develop conductive textile materials, characteristics of metal-coated textile materials, methods of preparation, the importance of in-situ metallic deposition, electroless plating, and metal-coated textile material implementations.

2.1 Electrically Conductive Metalized Fabrics

Textile metallization is a procedure that improves and expands the functional characteristics of textile (Keller and Kuhn, 2008). Metallized textile materials show a range of advantages as compared to the fabrics constructed exclusively of polymers and thin metal wires. The features of metallized fabrics over non-metallized fabrics comprise chemical resistance, resistance to extreme weather conditions such as soil, sunlight, and smog as well as the reduced sorption of hydrophilic compounds. Thin metal coating, on either side, can make flexible and light-weight textiles when compared to textiles made from metallic sheets and thin metal wire (Jarach et al, 2020) (Cui et al., 2015).

Additional feature is its ability to develop vibrant, shining, and decorative textiles in the domain of textile fashion design (Tseghai et al, 2021) (Dolez, 2021a). It is also possible to minimize the static charge by employing metallization technique. Static charge is generally found on the surface of polymer sheets and synthetic textile materials. The presence of these charges attracts dust particulate matter, and also have damaging effects towards equipment and electronic chip (Jarach et al, 2020) (Cui et al, 2015). In the area of smart textiles, there is also a considerable advantage.

Metal coated textiles are valuable owing to their unique electromagnetic shielding and electrical conductivity properties. Electrically conductive stretchable textiles offer a novel framework for the development of textiles sensors for clinical applications such as electrocardiogram sensing (Arquilla et al, 2020) (Cesarelli et al, 2021), electromyography (EMG) (Stoppa and Chiolerio, 2014), and electroencephalography (EEG) (Tseghai et al, 2020). Because of their antimicrobial properties, these textiles are frequently used in surgical gowns and bandages as well (Jarach et al, 2020) (Cui et al, 2015). They can even be embedded into textile materials for health monitoring, sport-action detectors and to monitor field-effect transistors due to their interesting feature of flexibility. Metal-coated fabrics have also found their implications as electrodes in energy harvesting systems (Dolez, 2021a) (Satharasinghe et al, 2020) and can be used to replace carbon-based polymeric electrodes, among other things.

2.2 Different Materials for Metallization

Materials are categorized as conductors, insulators, or semiconductors, based on their electrical properties. Figure 2.1 illustrates the range of electrical resistivity for various materials.

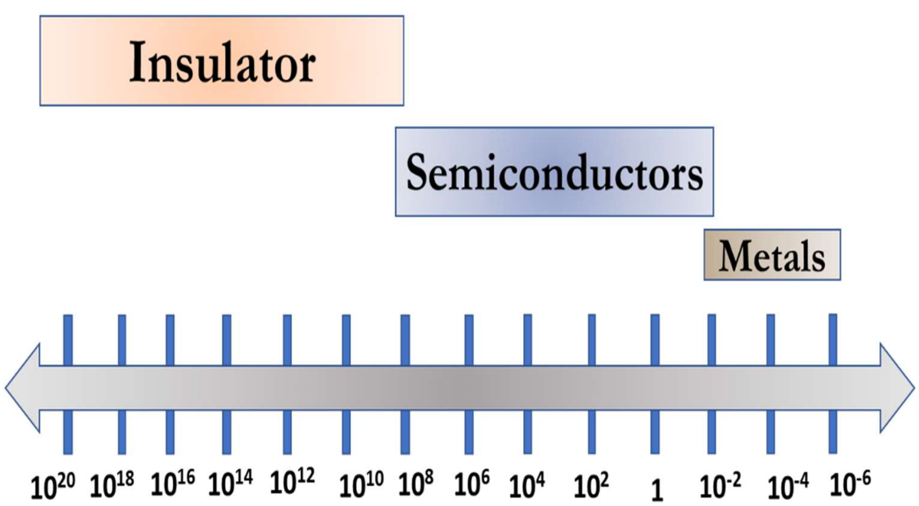


Figure 2.1 : Range of electrical resistivity for materials (Makvandi et al, 2021).

In general, the conductor class for developing conductive textile materials has been recommended. For this purpose, carbon, metals, and inherently conductive polymeric materials are good conductive mediums (Rytlewski and Jagodzin, 2016). Metals were conventionally woven into textile constructions primarily for aesthetic reasons. The first time a textile was metallized using a vapor deposition technique to provide electrical properties was in the 1960s. Metals can be deposited in a pure state (only one metal) or in the form of alloys to obtain electrical conductivity (Sarif Ullah Patwary, 2015). Aluminium, silver, gold, copper, nickel, platinum, cobalt and palladium are by far the most frequently utilized metals for enhanced electrical conductivity (Satharasinghe et al, 2020) (Liu et al, 2021). Silver has been the most conductive of all conductive metals (Ahmad et al, 2020), as well as the most demanding due to its inertness to oxidation Copper is second mostly to silver in terms of conductivity, yet its conductive strength is practically similar to silver. Copper is in high demand due to being cost-effective and easily available (S. Liu et al, 2016) (Tseghai et al, 2020). Nonetheless, the surface of Cu coatings, particularly those of nanoscale, is vulnerable to oxygen and spontaneously oxidizes. The development of co-metal deposition has led to a new generation of metal coatings to improve conductivity and prevent oxidation. This composite deposition has been used to produce over 50 distinct alloys, the majority of which are made of copper, nickel, silver, and cobalt. The most well-known combinations are Cu, Ag-Cu, Ni-B, Cd-Cu, Cu-Ni, Ni-P, Cu-P, Co-P, Ni-Sn-P, and Ni-Cu-P (Bai et al, 2020) (Wang et al, 2020) (Zhong et al, 2022). Enhanced electrical conductivity is provided by these copper-based alloys. When compared to using copper alone, the silver-copper alloy has nearly the same electrical-conductive values as ordinary high-conductive copper and provides higher oxidation resistance. Copper and silver alloys with chromium, beryllium, cadmium, bronze and tin on either side, have greater electrical resistivity (nearly 20 to 80 % of total) than pure silver and copper metals but exhibit more resistance to wear and corrosion (Stoppa and Chiolerio, 2014). Table 2.1 shows the order of electrical conductivity and resistivity of various materials at 20 °C.

Table 2.1 : 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}	(Alemour et al., 2018b)
Silver	1.59×10^{-8}	6.30×10^{7}	(Serway, 1998)
Copper	1.68×10^{-8}	5.96×10^{7}	(Giancoli, 2009)
Gold	2.44×10^{-8}	4.10×10^{7}	(Serway, 1998)
Aluminium	2.44×10^{-8}	3.50×10^{7}	(Serway, 1998)
Tungsten	5.60×10^{-8}	1.79×10^{7}	(Giancoli, 2009)
Zinc	5.90×10^{-8}	1.69×10^{7}	(Cleaning, 2005)
Nickle	6.99×10^{-8}	1.43×10^{7}	(Giancoli, 2009)
Iron	9.71×10^{-8}	10^{7}	(Serway, 1998)
Tin	1.09×10^{-7}	9.17×10^{6}	(Giancoli, 2009)
Lead	2.20×10^{-7}	4.55×10^{6}	(Serway, 1998)
Mercury	9.80×10^{-7}	1.02×10^{6}	(Serway, 1998)
Graphite	2.50×10^{-6}	2×10^{5}	(Cleaning, 2005)
Carbon black	5.00×10^{-4}	1.25×10^{3}	(Serway, 1998)
Conductive polymers	10^{-3} to 10^{-1}	10^{1} to 4.6×10^{3}	(Electrical, 2009)
Sea water	2.00×10^{-1}	4.80	(Cleaning, 2005)
Drinking water	2.00×10^{1}	5.00×10^{-2}	(Electrical, 2009)
Semiconductors	6.40×10^{2}	1.56×10^{-3}	(Serway, 1998)
Wood	10^{4}	10^{-4}	(Electrical, 2009)
Glass	10^{11}	10^{-11}	(Serway, 1998)
Hard rubber	10^{13}	10^{-14}	(Electrical, 2009)
Air	1.30×10^{16}	3×10^{-15}	(Electrical, 2009)
Diamond	10^{12}	10^{-13}	(Serway, 1998)
PET	10^{13}	10^{-13}	(Muthukumar and Thilagavathi, 2012)
Teflon	10^{23}	10^{-23}	(Electrical, 2009)

2.3 Different Correctional Shapes of Fibers and their Effects on Functional Properties

One of the most essential morphological characteristics of fibres is its cross-sectional geometry. Normally fibers are produced in round fiber cross section through spinneret. However, a circular cross section is not always preferable in fibre applications. Melt-spun fibres having noncircular cross sections were first developed in the 1960s. Noncircular fibres have been produced since then to improve the functionality and appearances of synthetic fibres. At present, most commonly produced cross-sectional fibers are trilobal, circular, hollow circular, octalobal, solids, scalloped oval and tetrakelion etc as shown in Figure 2.2 (Hearle, 2014).

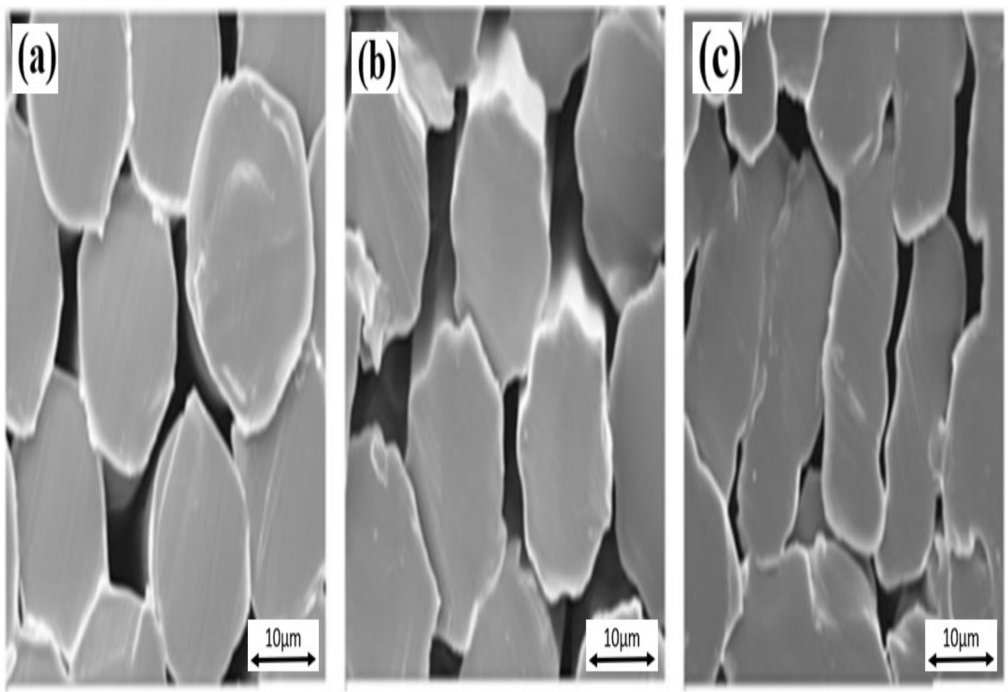


Figure 2.2 : SEM images of the single filament: (a) round, (b) octolobal and (c) W-channel [16].

Variations in the shape of spinneret hole can easily affect the cross section of a synthetic fibre formed by the melt-spinning technique (Hosseini et al, 2011). The coefficient of friction, bulkiness, handling, bending stiffness, softness, comfort, lustre, pilling, and performance of fibres with a noncircular cross-sectional shape differ from those of fibres with a circular cross-sectional shape as shown in Figure 2.3(Şardağ and Özdemir, 2011)

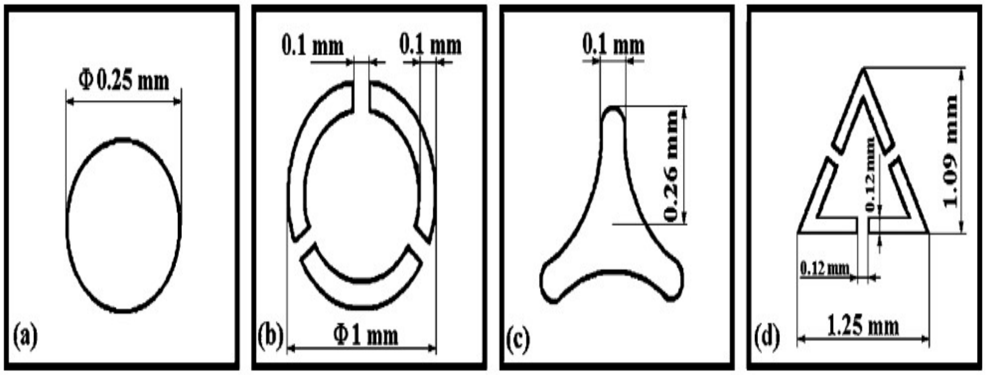


Figure 2.3 : Cross-sectional shapes of the nozzle holes in the spinnerets: (a) round, (b) hollow and round, (c) trilobal, and (d) hollow-trilobal (Cesarelli et al, 2021).

Simple, noncircular fibres are hollow fibres made in various geometries such as trilobals, circles, squares, and triangles.

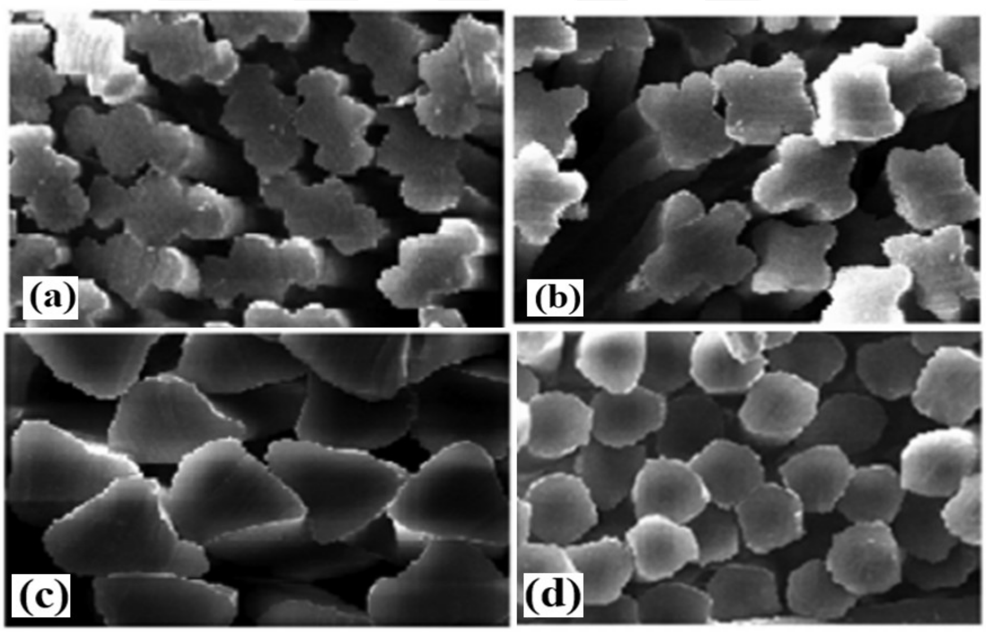


Figure 2.4 : Scanning electron micrographs of different cross-sectional shapes of fibers: (a) scalloped oval (b) tetrakelion (c) trilobal, and (d) circular (Stoppa and Chiolerio, 2014).

The difference among round and hollow fibre spinning methods is related to an additional geometrical variable (inner radius) (Oh et al, 1998). Hollow fibres are warmer, stiffer, and bulkier, due to the air inclusion, high bending resistant, and provide an increased opaque appearance due to diffused light reflection when compared to ordinary fibres with the same linear density. The majority of hollow fibre publications included the hollow-round polypropylene fibres. In fact, variations in fibre size, shape and density bring a change in number of functional properties in yarns and made fabrics with them. Also, the fibre shape, size and cross-section affect the thermal conductivity, electrical conductivity, cohesion and bulkiness. As a result, such geometrical parameters influence not just early phases in processing of mechanical staple fibres, but also for the later phases, also in yarn state, when the fibres are considerably more linearized (Oh et al, 1998).

Because of the round orifices of the spinneret, regular melt-spun fibres have circular sections. However, surface and mechanical attributes such as moisture regain, lustre, crease recovery, friction, abrasion resistance, bulk, pilling, flexural rigidity, dyeing, resilience, covering power, tenacity, can all be adjusted by modifying filament cross section. By modifying the cross section to a trilobal form, the initial attempt at non-circular cross - section was to emulate the gloss of silk filaments. Many alternative cross sections have already been developed since then for a variety of applications. Some cross-sectional shapes of the fibers are shown in Figure 2.5 (Bueno et al, 2004).

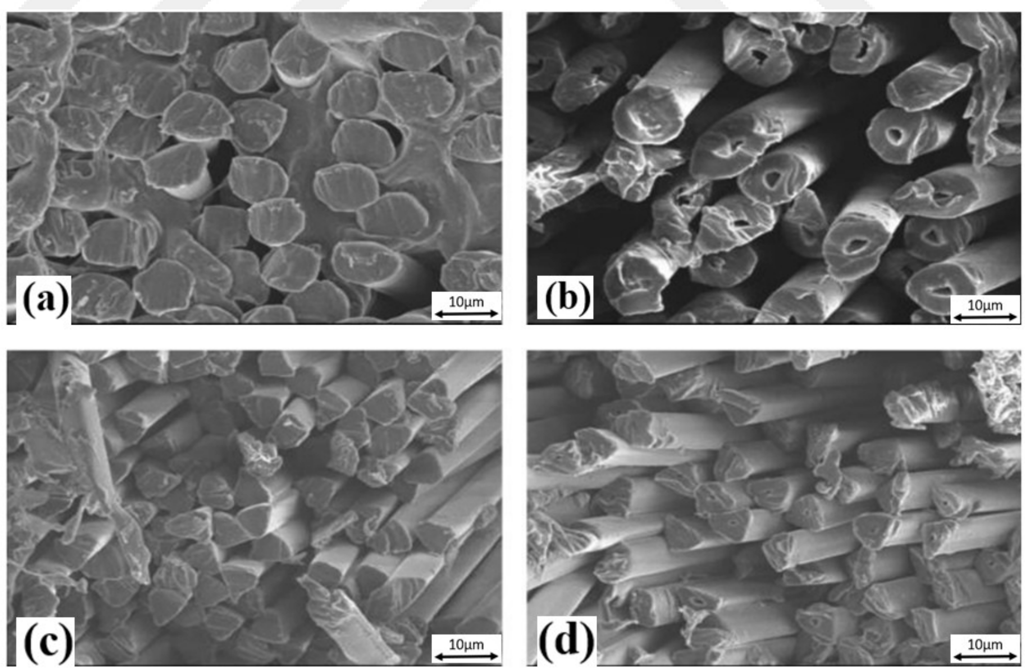


Figure 2.5 : Cross-sectional shapes of the fibers (a) round, (b) hollow-round, (c) trilobal, and (d) hollow-trilobal (Tseghai et al, 2021).

The majority of filament cross section research has been on its impacts on fibre characteristic (Bueno et al, 2004), with only a few studies extending to its impact on fabric properties (Oh et al, 1998). Air-jet texturing allows for the development of yarns with a variety of characteristics. One of these options is to change the filaments cross section of the feeder yarn. Changing the cross section of filaments has a direct impact on the yarn structure because it alters the flexural rigidity of filaments as well as the drag forces being acted on them. Modifications in the yarn structure arise from changing the cross section of the filaments in air-jet texturing as shown in Figure 2.6. In terms of overall cross-sectional area, the W-channel is the most unique. As a result, it produced the most peculiar yarn structure: uneven yarn, bulky, with a lot of open loops. Although the cross-sectional form of the filaments has a direct effect on light fastness and abrasion resistance, all of the cross sections employed abrasion resistance and light fastness and tests yielded good results. Changing the cross section of the filaments had the greatest impact on the air permeability of the textiles. The air permeability of the W-channel is the lowest, while that of the octolobal is the highest (Bueno et al, 2004).

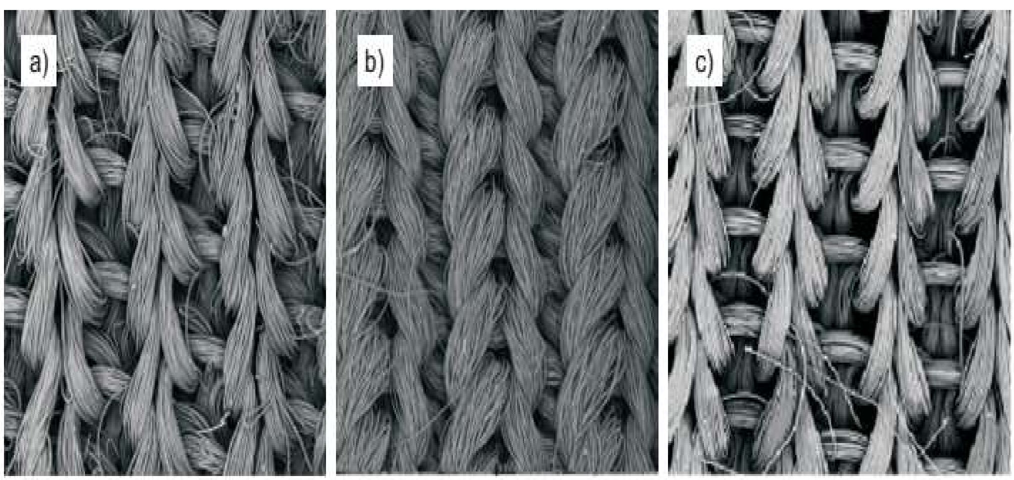


Figure 2.6 : SEM photos of: (a) air-jet textured, (b) false-twist textured, and (c) non-textured fabrics respectively (Tseghai et al, 2021).

2.4 Texturing of Synthetic Yarns

Synthetic fibers have become more desirable as natural resources are depleted and worldwide consumption for textile products rises. Because synthetic yarns lack the aesthetic and handling characteristics of fibres, special techniques must be used to combine the better properties of synthetic fibers, such as, uniformity, high strength, and stretch, with the traits that are distinctive to natural fibres (Eskin, 2003). Texturing has been one of the techniques that results in a bulky and crimped structure, as well as a natural appearance, high elastic extensible, warmth, bulkiness, softness, and stretch (Stojanović et al, 2018) (Abbasi et al, 2020).The elastic extensible property, crimp grade, and bulkiness of the textured yarns are primarily assessed. The curling of the elementary filaments and significantly increased air gap between particular filaments generate the relatively high bulkiness of textured fibers. The primary goal of texturing the filament yarn is to produce a bulky structure, which in itself is beneficial since the spaces in the structure provide effective insulation and modify the density of material. Dispersed light reflections result from an unorganized sometimes less organised yarn surface, giving it a desired matte impression. The sponge-like texture is softer than that of the lean twisted 'flat' yarn, and the coiled or crimped filament structure provides the structure a lower effective elastic modulus than that of a flat yarn (Özçelik et al, 2007). Texturing yarn can be accomplished in a number of ways. Air-jet (Mutlu and Demir, 2021) (Huang et al, 2019), false-twist texturing (Foster et al, 2021) (Taghavi et al, 2022), and steam-jet (Foster et al, 2022) (Kim and Kim, 2018), are some of the existing processes used in the industry. Air-jet texturing has been a well filament yarn processing method which has been around for almost half a century (Hearle, 2014). Unlike other texturing techniques, air-jet texturing can be used on any filament yarn. To be heat-set, all of the aforementioned texturing methods necessitate thermoplastic yarns. Non-thermoplastic yarns, such as rayon, cannot be used because of the air jet. Furthermore, it is a good method for developing a yarn structure similar to that of staple yarns. This is a significant concession to the preferences of the end user (Wilson and Kollu, 1987). The filament yarn typically textured through overfeeding it in to a high-pressure air jet to generate a looped and also more natural yarn texture mostly in air-jet texturing technique. The amount of bulkiness is determined by the input and jet-take out rates. Air flow texturing of continuous polyester filaments allows for the fabrication of a wide variety of yarn count (50 to 60,000 dtex), which seem to be impossible to achieve with other texturing methods. One of the key benefits of the procedure, the potential to generate an infinite variety of mixed yarns, has yet to be completely implemented (Wilson and Kollu, 1987). Figure 2.7 depicts a continuous filament yarn that has been air-jet textured.

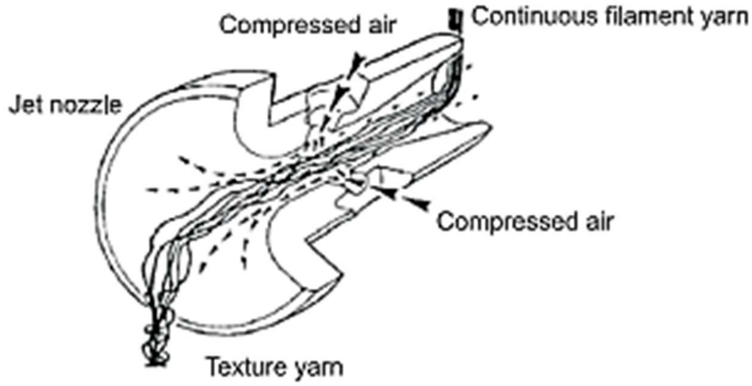


Figure 2.7 : Diagram of a continuous filament yarn being air-jet textured (Makvandi et al, 2021).

Thermoplastic filaments have false-twist texture. The fibres are twisted, heated, and then cooled in the twisted state, followed by untwisted in false-twist texturing. Due to its increased texturing rates and convenience, false-twist texturing is a widely used commercial method for producing textured filament yarn (Eskin, 2003). In air-jet texturing, shape and amount of the loops are affected by both feed yarn properties and process parameters. Increasing overfeed and number of effect component increase the looped structure (Figures 2.8-2.10).

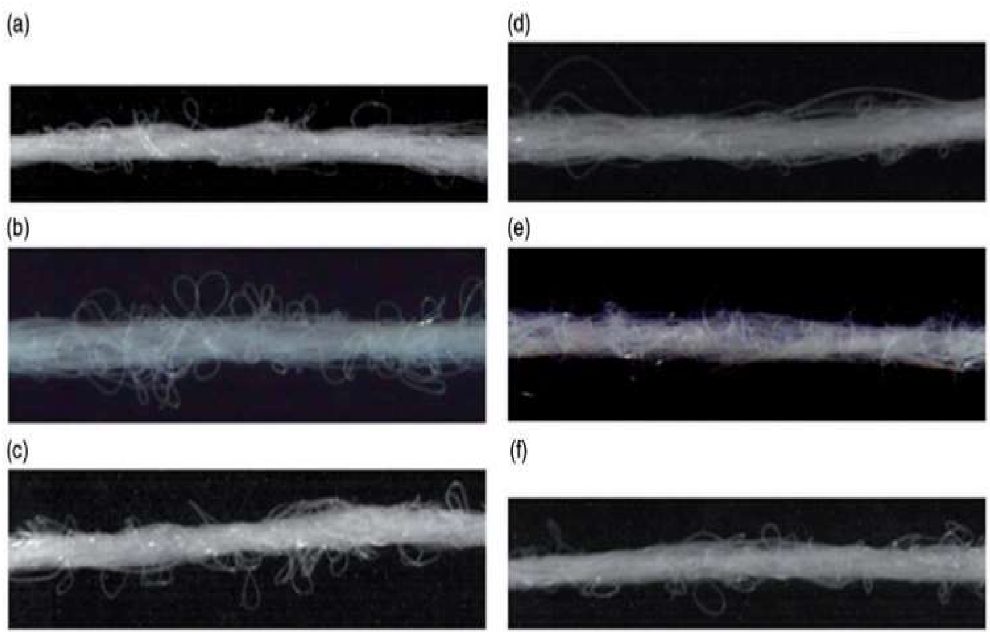


Figure 2.8 : Optical microscopy images of the air-jet textured yarns: (a) Round-l, (b) Round-2, (c) Round-3, (d) Round-4. (e) Round-5 and (f) Round-6.

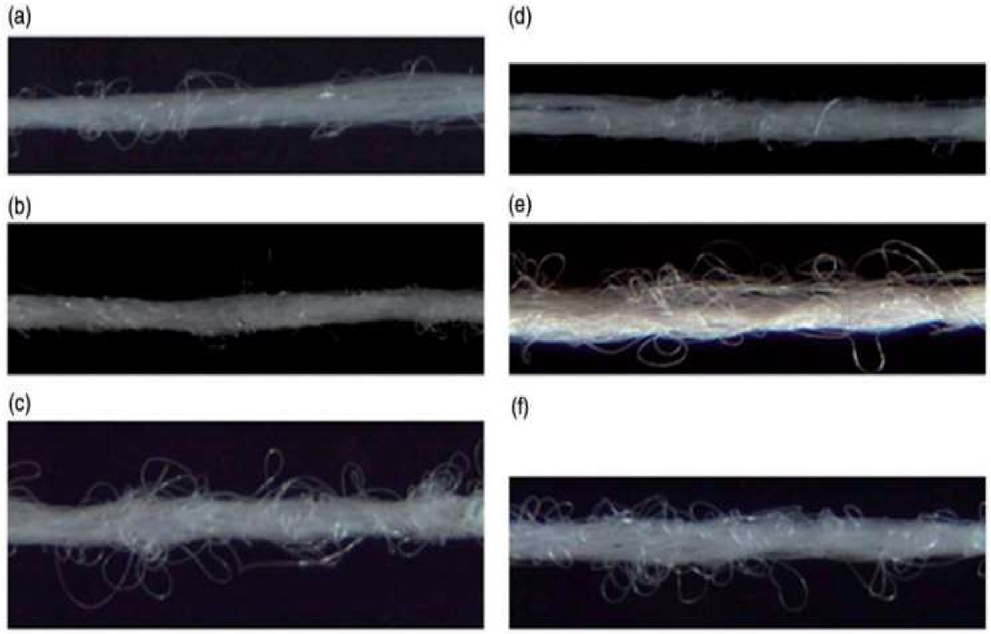


Figure 2.9 : Optical microscopy images of the air-jet textured yarns (a) Octo-1, (b) Octo-2, (c) Octo-3, (d) Octo-4, (e) Octo-5 and (f) Octo-6.

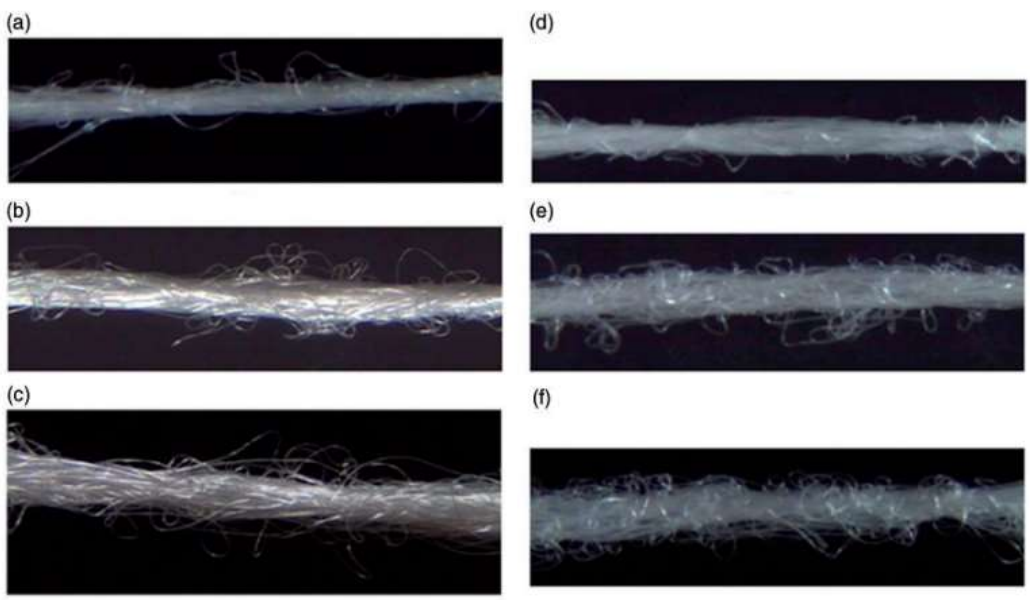


Figure 2.10 : Optical microscopy images of the air-jet textured yarns: (a) W-1, (b) W-2, (c) W-3, (d) W-4, (e) W-5 and (f) W-6.

2.5 Electromagnetic Interference Shielding

Recently, electromagnetic interference (EMI) has become the fourth type of pollution in public areas. EMI is termed as hazardous and unnecessary emitted signals cause undesirable degradation of equipment and system (Tamim et al, 2021) (Wang et al, 2021). If left untreated, these issues can lead to serious damage to scientific and commercial electronics, as well as antenna systems, the secure operation of various electronic equipment, and military electronics. (Wu et al, 2007). The formation of electrostatic discharge is the most prevalent and primary cause of electromagnetic interference (ESD). This is a common phenomenon whereby the high-frequency radiations are emitted from one electronic equipment and cause nearby devices to malfunction. Even a non-technical person can easily detect electrostatic discharge (ESD). ESD symptoms include cell phone bumps against the television, sound system bangs whenever a light is turned on, and distorted television transmission appearing as flashes on the display screen. Electromagnetic radiations are also harmful to human health and can trigger a range of issues such as anxiousness, hypertension, languidness sensations, and insomnia when subjected to them. Consequently, there has been a massive increase in research into electromagnetic shielding (EMI) materials (Palanisamy et al, 2021) (Duan and Lu, 2021) (Zhong et al, 2020). Electromagnetic shielding is described as the use of a conductive or magnetic material to restrict the transmission of magnetic and electrical radiation from one point to the other. Filtering all inbound and outbound external interference is the basic premise for EMI protection. The ratio of imposed energy to residual energy is termed as the shielding performance. Electromagnetic radiations are absorbed and reflected during the shielding process. The residual can be explained as the fraction of the residual energy that is not absorbed or reflected through the shielding material and rather radiates out of it. EMI shielding is achieved by reducing the number of signals flowing through the systems, either via reflection of absorption of waves. The shielding material is able to reflect radiations only if it contains free electrons. Stretchable conductive textile materials have acquired considerable attention owing to their high electrical conductivity, effective electromagnetic interference shielding (EMI), electrostatic dissipating behaviour, low weight and breathability. They are considered a significant material for minimizing electromagnetic interference in the form of various manufactured products including foam gaskets or fabric tape (Biermaier et al, 2021). These materials, for example, could protect the guided aircraft from exploding owing to electromagnetic interference.

The coatings of conductive polymeric materials, textile surface metallization, coatings of carbon-based materials, and other techniques have been used to provide textiles with electrical conductivity and electromagnetic shielding properties. Surface metallization is one of the most distinct processes that is capable of providing multifunctional characteristics including electromagnetic interference shielding (EMI), conductive behaviour, antibacterial and anti-static effects, ultraviolet rays screening, as well as radar reflectivity. Conductive paints, metallic laminates and foil, and lacquers are all common metal coating processes. Metallic textiles developed through these conventional techniques, on the other hand, has flaws such as hardness, low air permeability, and a high weight. The electroless plating, sputter coatings, vacuum deposition, and flame or arcs spray coating, are examples of emerging surface metallization technologies that potentially overcome the shortcomings of conventional approaches (Yip et al, 2009).

Coherent metallic deposition, high conductivity, effective shielding, and adaptability to complex geometric shape substrate are all benefits of electroless plating (An et al, 2018). Textiles plated with copper had better EMI shielding than those coated with nickel or copper-nickel, according to earlier studies. At 100 MHz–1.8 GHz, each metal-coated fabric had EMISEs of 37–32 dB for nickel, 68–35 dB for copper, and 46–32 dB for copper-nickel, respectively. (Han et al, 2001). The SE of ultrasonic-assisted electroless plating of silver on polyethylene terephthalate textiles was more than 32 dB in another study (Biermaier et al, 2021) (Liu et al, 2021), with frequency varying from 0.01 MHz to 18 GHz. However, silver plating is more expensive than copper plating. Metalized textile materials are also developing as a best material for EMI shielding of sophisticated equipment, notably for defence purposes and in aerospace, owing to novel features such as stretchability and flexibility. Conductive textiles materials are utilized as a shielding cover in a variety of products for the aerospace, defence industries, computers, automotive, electronics, healthcare, and telecommunications (Coatings et al, 2017). Metal-coated textiles are customized to develop ready-to-use bags, adhesive tapes, curtains, panels, and other products rather than just fabric (Alemour et al, 2018). Metal electroless plated materials are very comfortable and flexible, and therefore can be utilized as personal-protection undergarments. These clothing are particularly popular amongst pregnant women who needs to work next to the computers and in areas with high levels of electromagnetic radiation during their pregnancies. Electrically conductive materials might also assist guided aircraft to survive in a more sophisticated electromagnetic environment by reducing the effects of the surrounding environment. Personal shielding from low-frequency electromagnetic waves can be achieved by wearing undergarments composed of metalized fabric. Pregnant women who use computers, as well as those who work near high voltage power lines or microwaves, are among the target customers (Serway, 1998).

2.6 Preparation Methods of Electrically Conductive Metalized Fabrics

Textile metallization processes have a long history and have developed with the passing years. Tiny wires or flattened strips of metals were woven into textiles in ancient times to give aesthetic and ornamental features. It was not until twentieth century that these technologies were updated. Between the two world wars, enormous advances in metallization occurred, with metal strips attached to both sides of cellulosic materials. The produced materials, on either side, were weighty and hard (McIntyre, 2005). Following WWII, technology underwent a significant transformation. Textile metallization began with new surface coating treatments. Conductive paints, metal sheets, laminates, and lacquers are all common metal coating processes. Metallic fabric made through these conventional method, on the other hand, has flaws such as rigidity, low air permeability, and is weighty (Yip et al, 2009). Sputter coating, flame and arc spraying, vacuum deposition, and electroless plating are examples of emerging surface metallization approaches that can address the shortcomings of conventional procedures (Şardağ and Özdemir, 2012). Some researchers choose to sensitize the surface first with vacuum deposition followed by sputtering, with subsequent application of silver plating to the surface of conductive material. This method is superior for achieving surface conductivity, but volume conductivity via materials is extremely challenging in this instance. In terms of volume conductivity, coherent metal deposition, durability, and adaptability to complex structured materials, electroless plating over solution sensitised textiles is preferred. Researchers have used electroless copper plating, a form of the non-electrolytic methodology for the deposition on the fabric from solution (Oh et al, 1998). According to a study, copper electroless deposition is based on a catalysed redox reaction between formaldehyde based reducing agents dissolved in alkaline medium and metal ions at elevated temperature (Vaškelis et al, 2007). Due to the production of toxic gaseous products during the process of plating process, this technique is unsafe and frequently fails, particularly on an industrial level. Researchers attempted to fix the problem by replacing formaldehyde with alternative reducing agents and oxidation accelerators notably nickel sulphate and sodium hypophosphite (Ghosh, 2019). Despite the fact that researchers have experimented with various kinds of electroless plating, each strategy has its own number of restrictions. Due to the low reduction potential of copper and the fact that copper is not a better catalyst for hypophosphite oxidation, electroless plating with other reducing agents such as sodium hypophosphite has limitations in terms of fixation and defective reduction. They have been utilizing HCl to perform electroless plating in acidic media, which changes the reducing agent's reduction potential and causes the oxidation of the reducing agent to generate H+ ions (Root et al, 2018). Electroless plating with cobalt, nickel, lead, palladium, and stannous, (which requires pre-treatment) is not suitable for hygienic purposes because these metals are toxic and inflammatory to the skin (Eskin, 2003). Some research documented the traditional procedure of initiating electroless plating with palladium metal, which is often used as a catalyst site. (Xuejiao, 2009). Due to the rising cost of palladium, this technique of plating is getting increasingly expensive. As a result, a cost-effective and efficient activation mechanism was required. Additionally, prior to copper plating, people have used two-step techniques: first, sensitizing, then activation, and finally, plating. As a result, more study is needed for the effective preparation of copper-coated fabric in a secure environment. (Coessens et al, 2001).

2.7 Electroless Plating

The terminology "electroless plating (ELP)" refers to the chemical reduction process of depositing a homogeneous film or coating of metals on a substrate without the use of an external electrical power source (Yu et al, 2015). Because of this method's ability to self-catalyse or autocatalyze, no electric power source is needed in general. ELP was done in the 19th century by reducing silver with aldehyde reducing agents. Following that, during the twentieth century, considerable progress was made in this domain. When scientists began to incorporate various metals and alloys for plating without the use of an external electrical source. Since, the process has been extensively researched and applied in a variety of fields, including automotive, aerospace, biomedical, electronics, and then even textile metallization.

Electroless deposition metallization of textiles resulted in a fine, homogenous, and dense coating of metal on the surface. The role of the reducing agent in this technique, as well as its selection, is critical. There are two forms of electroless plating. Displacement deposition electroless plating is the first type, while autocatalytic deposition electroless plating is the second. Active metal-coated fabric is soaked in the solution of metallic salt including less active metallic ions (M1+) during displacement deposition. Consequently, the following reaction occurs.

$$M1^{+} + e^{-} \rightarrow M1 \qquad (2.1)$$

The metal M1 is coated over the surface of substrate and develop a uniform dense film. In this reaction, various combinations such as Cu/Fe, Ag/Zn, Au/Ag, and Au/Ni can be used. Cu/Zn, Ag/Zn, Au/Ag, Au/Ni, Cu/Al, Pt/Ni, Pt/Fe, Pt/Co (Xue et al, 2012) are few examples of this reaction (Sharaf et al, 2015).

Less active metals are not capable of performing autocatalytic deposition during the autocatalytic mechanism. The choice of reducing agent is critical in this metal deposition method (Yu et al, 2013). When choosing a reducing agent, it should have a good standard reducing power in comparison to the metals being reduced (silver and copper). For example, the reduction potentials of several key reducing agents, such as formaldehyde (-1.30 V), and hypophosphite (-1.57 V) and are more useful than the reduction potentials of silver +0.80V and copper +0.34V (Inoue et al, 2015). Another chief characteristic of reducing agents is their ability to self-catalyze reactions, which can facilitate the deposition of the maximum amount of copper on the target metal (fabric coated with Cu particles) (Kobayashib, 1994). Once this reaction is initiated, metal deposition occurs too quickly, not only on the surface of the substrate but also throughout the entire volume.

The following are some of the most significant advantages of electroless plating (Rytlewski, 2016).

- It is simple to use, facile, flexible, and adaptable to a wide range of materials.

- Electroless plating, unlike other processes including chemical vapour deposition, physical vapor deposition, and electroplating, is not harmful to human health (Guo, 2013).
- It is also time and cost effective
- It enables metal layer coatings to have a uniform thickness.

2.8 Research on Different Fibers Structure with Metal Coating for Electrical Properties

Yahya Atwa et al developed a more mechanically flexible and a much lighter coating by depositing random networks of solution-synthesized nanowires of silver on cotton filaments, nylon, and polyester (Atwa, 2015). The polyol technique was used to synthesise silver nanowires in solution at low temperatures, and dip-coating was used to deposit them onto threads.

The nanowires concentration in the coating solution and the numbers of dipping cycles were used to adjust the density of the nanowire film being deposited nanowire film. The microstructure of polyester, nylon, and threads after three dips in the coating solution can be seen in Figure 11, and their resistivity was measured at 15 cm, 12 cm, and 11 cm, respectively. Table 2 shows the relationship between electrical resistivity and metal content for nylon threads, showing that as the amount of metal concentrations increased, the electrical resistivity decreased (Atwa, 2015). They also tested the resilience of coated nylon threads by bending them through various cycles. When compared to available commercial conductive thread, the silver-coated nylon thread showed practically constant resistivity. Figure 12 illustrates this trend.

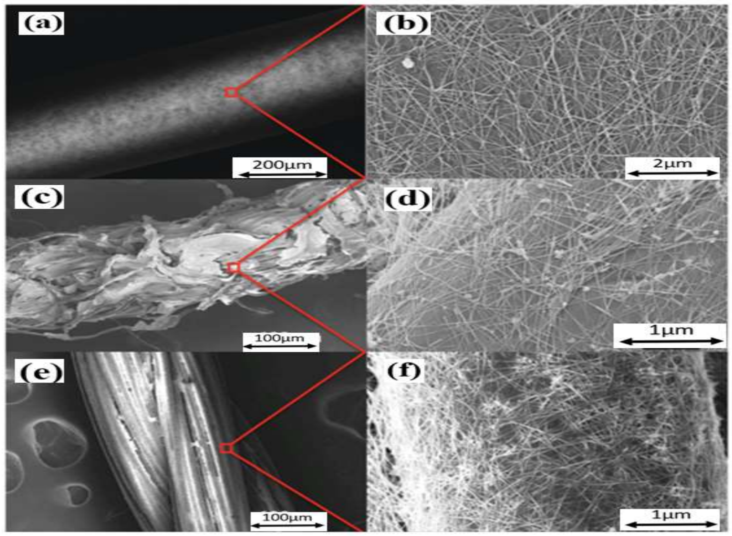


Figure 2.11 : SEM images of silver nanowires coated (a, b) nylon (b, c) cotton and (c, d) polyester fabrics (Atwa, 2015).

Table 2.2 : Electrical resistivity for silver-coated nylon textile.

Metal coating (mg m^{-1})	Volume resistivity (Ω cm)
.24	12.0
0.52	2.5
1.07	0.8

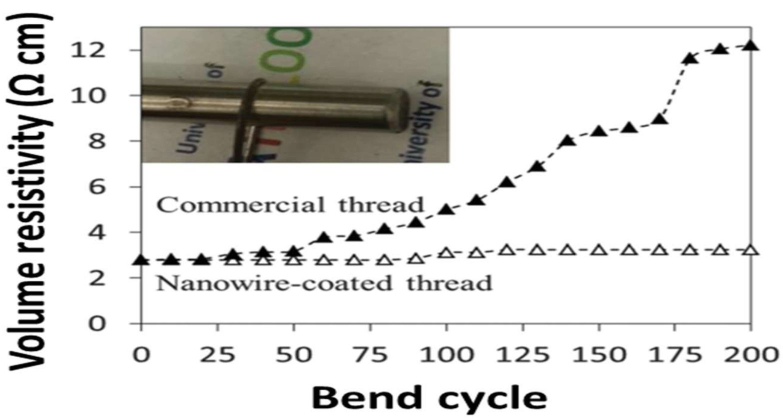


Figure 2.12 : Comparative analysis of electrical volume resistivity of commercially available thread with silver nanowires coated thread at varying number of bending cycles (Atwa, 2015).

Another presented plasma sputtering as an alternate technology for depositing silver on textiles, which enabled simultaneous cleaning and deposition. The authors compared the developed plasma sputter textiles to electroless coated textiles (see Figure 2.13). The smooth coatings provided excellent adherence to polyester fabrics, but the resulting electrical conductivity remained minimal (range in kΩ) primarily to the line-of-sight shadowing of the approaching sputtering atoms (Ghosh, 2019).

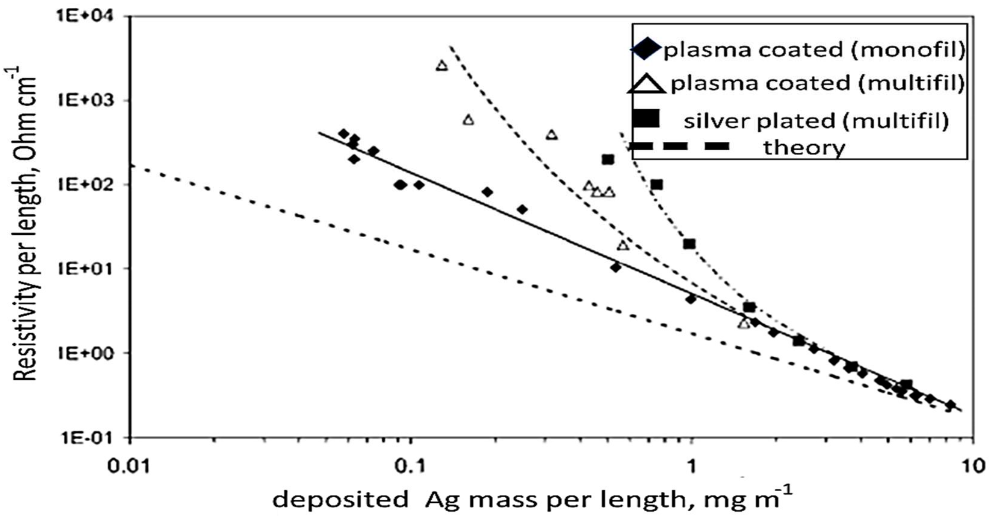


Figure 2.13 : Electrical resistivity as function of the amount of silver deposition (Hegemann et al, 2013).

Another study used surface initiated atomic transfer radical polymerization to generate polyelectrolyte brushes on cotton surfaces. The electroless depositing of metallic particles onto the brush-modified cotton threads was then carried out. The conductivity of the threads increased as plating time increased, reaching 1 S/cm after 1 hour. Subsequently, the conductive yarns were tested for durability under several stretching and bending cycles, with increased conductivity (0.28 S/cm) when strained and decreased conductivity (0.04 S/cm) when the stress was relieved. Up to 30 cycles, this procedure was reported to be entirely reversible. As illustrated in Figure 2.14, the durability of copper-coated textile was also examined over time. Due to copper's sensitivity to oxidation, a considerable loss in electrical conductivity has been observed (Kim et al, 2006b).

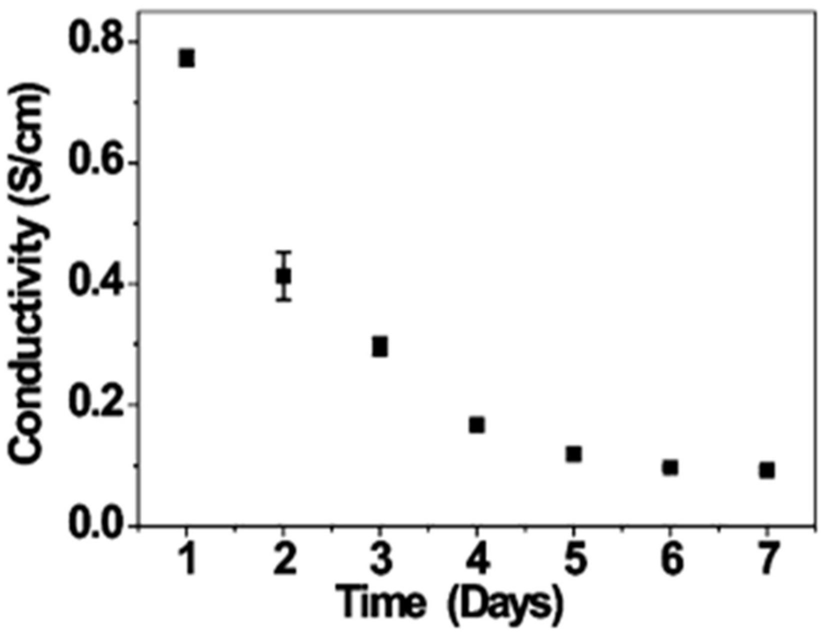


Figure 2.14 : Decline in electrical conductivity values with the passage of days (Liu et al., 2010).

A group of researchers examined the multiple functionalities of single-metal procedures with Ag or Cu and combined metal procedures with Cu/Ag. Cu/Ag combined treatment has a relatively low surface resistivity of 25.17/square in terms of surface resistivity. Even after hydrophobic coating, the surface resistivity was estimated to be constant at roughly 184.38/square. In all samples of single metal nanoparticles treatments, however, no conductivity was seen, irrespective of the type of metal nanoparticles employed or if the hydrophobic coating was applied on surface. The difference in the add-on of metallic particles and the difference in the exposed position of metals was responsible for this behaviour (Zhong et al., 2021).

Other researchers focused at the electromagnetic shielding properties of conductive knitted textiles composed of hybrid yarns with 50 m diameter metal fibres including stainless steel, copper, and silver. The core-spun yarn spinning equipment has been used to generate the hybrid yarns. The effects of wales density, radiation frequency, metal type, and geometry on electromagnetic shielding effectiveness (EMSE) were addressed. The geometric features of the fabric were observed to have a substantial impact on shielding performance among all experimental parameters, with Milano type knitted fabrics providing shielding effectiveness exceeding 20 dB. The Milano, lactose, cardigan, and composite textiles outperformed the rib and weft composite textiles in terms of shielding (see Figure 2.15) (Campos et al, 2018).

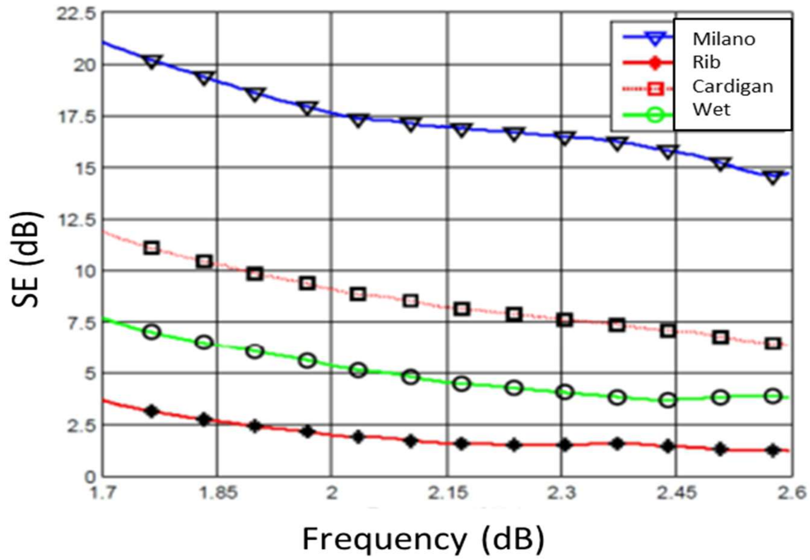


Figure 2.15 : EMI shielding performance of different fabric samples knitted with the combination of stainless steel, copper, and silver (Abdulla et al, 2017).

Copper plating onto a plasma-treated PET substrate has been used by Xuqing et al. to create robust stretchable conductive PET textiles. Figure 2.16 illustrates that the surface electrical resistivity of PET fabrics declines as plating time is increased, reaching as low as 2/square after 1 hour. This phenomenon was influenced by the presence of more copper nanoparticles, which resulted in the formation of ever more continuous Cu films (Xue et al, 2012).

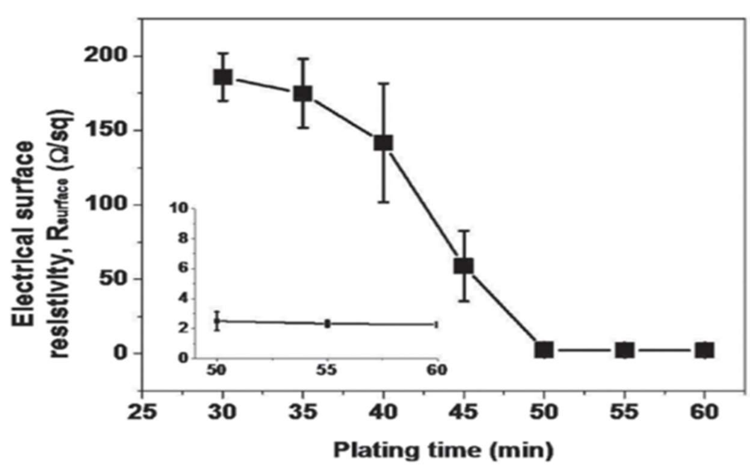


Figure 2.16 : Decline in electrical resistivity with the increase in electroless plating time (Liu et al, 2016).

Hao et al. established a unique two-step process for producing silver-plated cotton textiles with outstanding electrical conductivity and wash fastness. Through in-situ polymerization of dopamine, a polydopamine (PDA) coating was first coated on cotton fabric surface. The conductive fabrics were then produced by immersing the preceding fabrics in a silver-ammonia ($[Ag(NH_3)_2]OH$) solution and then reducing them in a glucose solution. (Liu et al, 2016). The catechol groups of polydopamine reduced the Ag ions in silver nitrate ($AgNO_3$) solution, and Ag nanoparticles were coupled with polydopamine to establish covalent bonds with the cotton substrate. While keeping the dopamine concentration constant, the surface resistivity decreases when the concentration of silver-ammonia ($[Ag(NH_3)_2]OH$) solution increases from 0.1 to 0.25 mol/L. As can be seen in Figure 2.17, when the concentration of silver-ammonia is varied between 0.2 to 0.25 mol/L and also the dopamine concentration is varied between 1 to 6 g/L, the surface resistivity decreases or increases moderately. At 0.2 mol/L silver-ammonia concentration and 6 g/L dopamine concentration, the surface resistivity was 0.12 0.02/square (Sharaf et al, 2015).

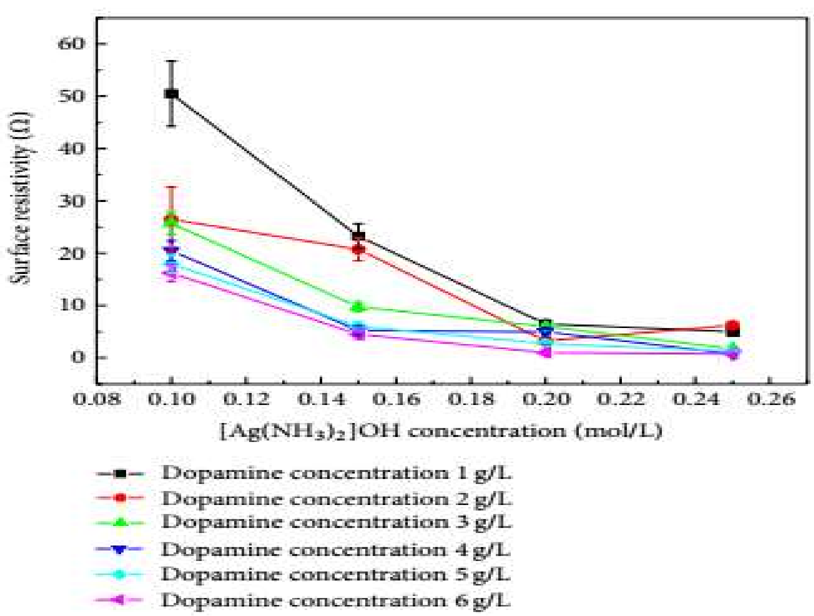


Figure 2.17 : The electrical resistivity behavior of Ag-treated cotton fabric samples pre-treated with varying concentrations of poly-dopamine film (Liu et al, 2016)**.**

A study was focussed the fabrication of electrically conductive poly-aniline coated polyester fabrics. The polyester fabric was alkaline activated before being immersed in an acidic aqueous bath of aniline monomer. The oxidizing solution was then utilised to activate the polymerization process. Before the ant test, the developed product was thoroughly washed and dried. Fourie-transformed infrared spectroscopy (FTIR) was used to investigate functional groups on the surface of the polyaniline coated fabric. Scanning electron microscopy has been used to examine the morphological characteristics of the produced conductive polyaniline. The electrical conductivity was noted about 100 S/Cm as shown in Figure 2.18 (Razaghi, 2018).

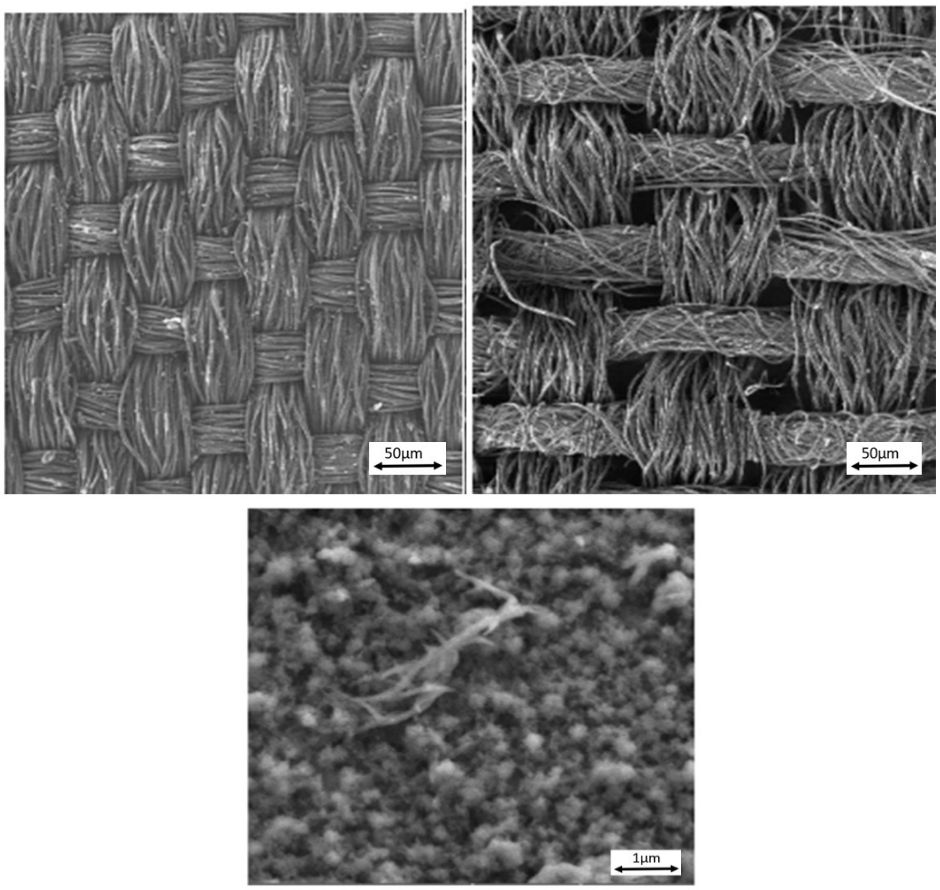


Figure 2.18 : polyester fabrics (a) untreated (b) treated (c) aniline coated.

To develop electrically conductive textiles, another investigation was conducted on polyester/viscose blend fabrics. The samples were initially exposed to microwave radiation for various periods of time, and then the optimized treated fabrics (8 minutes) was chosen for treatment as shown in Figure 2.19. Using a dispersion agent, stability, washing performance, and physical characteristics of the fabric, the physical properties and morphology of Cu nanoparticles with various percentages were measured. Scanning electron microscopy was used to photograph the surface morphology specimens.

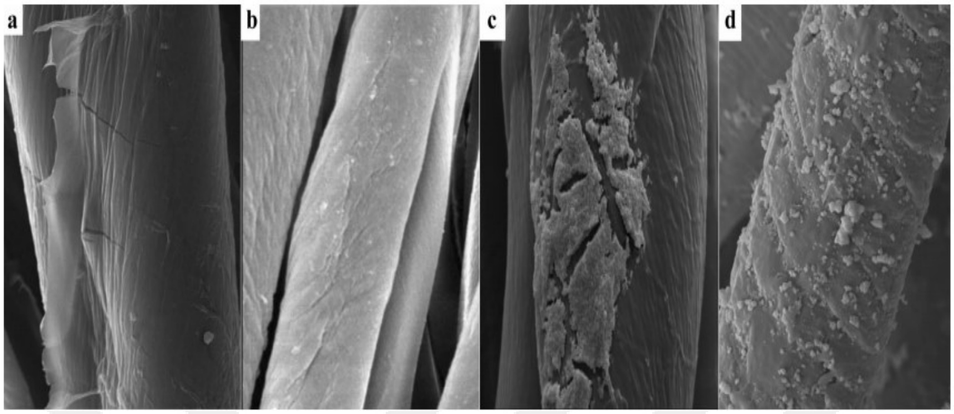


Figure 2.19 : Image of SEM sample of the fabric under 8-min microwave treatment: a Cu 3% o.w.f (5000x), b Cu 3% o.w.f (2000x), c Cu 7% o.w.f (5000x) d Cu 9% o.w.f (5000x).

The electrical conductivity parameters of the specimens were then measured and assessed according to AATCC 2005-76 standard testing protocol. In fact, the results indicated that optimum electrical resistivity, values were obtained for the samples having more microwave radiation time.

Table 2.3: Electrical resistivity and conductivity of Cu particles coated fabrics pretreated with different time of radiation time.

Run	**Factor**		**Responses**	
	Irridaiation Time (min)	**Go-nanopartilce (%)**	**R (Ω. cm)**	**(S/cm)**
Control 1	0	6	74652	0.0015
Control 2	8	0	Unlimited	Unlimited
1	8	6	6550	0.07943
2	8	6	6512	0.07995
3	8	6	6490	0.07894
4	4.8	4.2	32399	0.0345
5	4.8	7.8	3595	0.3851
6	12	6	9768	0.1199
7	9.2	8.9	507	0.2925
8	9.2	3.1	23970	0.01195

2.9 Research Question/ Gap

After a comprehensive literature review, research gaps were identified. Following research gaps were defined in accordance with the current data.

- The electroless plating method was chosen to fulfil this aim since the process allows the coating of non-conductive textile materials, and it is relatively cheaper compared to other available options for making the conductive fabric.
- Previously electro less plating has been performing by different salts and reducing agents' combinations over different fibres. However, there is huge gap in literature to study the effect of electroless plating on different surfaces and different GSM.
- One more major novelty of present study is that, we have also studied the effect of different fibrous cross-section (round, hollow round, W shape and octolobel) against copper electro less plating.
- In the same way, in literature huge gap regarding the coating of metals on same substrate but having different surface pre-treatments.
- Electroless plating is used to deposit a coating of a metal on a substrate without using an external power source, such as electricity. Unlike electroplating, usage of direct electric current is not required (M, 1997).
- Conventional electroless plating technique was employed for knitted stretchable fabrics to fabricate the stretchable and flexible sensors as well as electrodes. Therefore, it is preferable to coat the metal particles, nano- and microwires with binder (Adamczyk et al, 2004). Additionally, if a binder is utilised to adhere particles to the surface of fabric, its searchability is compromised.
- Cu-coated fabrics should have better electrical performance even in stretched form. Thus, the fabrics will be a good alternative to the harsh electrodes made of carbon rubber (use in EMG, ECG, electrotherapy etc.)(Keller and Kuhn, 2008).
- In literature plating mostly have been performed on solid round fibers. But present study reveals that the hollow fibers had higher amorphous orientation than the full fiber. The difference between the spinning processes of hollow

fibers and round fibers is due to an additional dimensional variable (inner radius), hollow fibers are bulkier, warmer because of the inclusion of air. Hence, we achieved maximum entrapped metal particles inside and on the surface of fibers. Which in turn provide the maximum threshold to conduct the electrical current.

- Numerous studies have documented the use of formaldehyde-based reducing agents to develop the Cu plating. The formaldehyde are easily available, less costly, and more economical (Brenner, 1946).
- Some more studies have been reported to use other famous reducing agents $NaBH_4$. Sodium hydrosulphide and glucose. The supplementary active ingredient sodium borohydride facilitates quick reduction. NaBH4 has a high concentration of H+ ions and produces metal ions quickly, which promotes cluster formation and uneven coating. (Ali et al, 2018). Moreover, the coat of sodium hydrosulphite is uneven and glucose is weak reducing agent (have less reduction potential). Hence, the formaldehyde having the reduction potential same like $NaBH_4$ and sodium hydrosulphide but is better to use (M, 1997).
- Additionally, the low pH that the formaldehyde-based reducing agent offers is favourable for improved nucleation of metal ions, that ultimately generates small metal particles and prevent aggregation (Kobayashib, 1994).
- The present method is also more economical regarding the selection of copper salt and reducing agent (Dolez, 2021b).
- The electroless plating is more durable as compared to, in-situ deposition of nanoparticles (facilitate thin layer coating) and also electro less plating is economical as compared to other costly methods such as chemical vapour deposition as well as physical vapour deposition etc (Hasan, 2018).
- Purpose for electro-less deposition is to provide the durable and even deposition. Conversely, carbon-based compounds, conductive inks, and conductive polymers offer insufficient binding and reduced electrical conductivity.
- Copper electroless deposition is also better regarding comfort properties (drape, thickness and stiffness).

- For Cu plating over lead, stannous, and cobalt, a considerable research has been performed. It is not suitable for hygienic uses. Tin (Sn) is dangerous and irritating to the skin, and people who already have skin conditions may be more vulnerable to the negative effects of these substances (Mackison et al, 1981). The existing Cu deposition demonstrated that fabric could be utilised for eco-sustainable purposes which include electrodes for (TENs) machines, west for military uses where electromagnetic shielding is required, and hygienic west in the situation that sliders are injured.
- In order to choose the right reducing agent, it must have a higher standard reduction potential than the metals (in this case, copper) that are being reduced. For example, the reduction potential of several key reducing agents such as hypophosphite (-1.57 V) and formaldehyde (-1.30 V) might become useful because they are greater than the reduction potential of Cu (+0.34V). (Huang et al, 2007). Another crucial characteristic of reducing agents is their capacity for self-catalytic reactions, which can aid in depositing the greatest amount of copper on the target metal (fabric activated using Ag)(Zenglin et al, 2003).

3. METHODOLOGY

3.1 Materials

Two types of fabrics were used for metallization. One was combed cotton knitted single jersey (used for study one and study two) and second was plain polyester fabric (used in study three). There were nine different samples of combed cotton fabrics (single jersey) with different counts, fabric weight (GSM) values and elastane percentage, there are given in Table 3.1, the structure and schematic of cotton knitted fabric sample is shown in Figure 3.1.

Table 3.1 : Specifications of the knitted combed cotton fabrics.

Number of samples	Code of uncoated sample	Code of coated sample	Fabric density g/m2	Elastane (%)	Denier	Count (Ne)
1	S1	C1	250.72	10	20	40
2	S2	C2	270.65	10	30	40
3	S3	C3	290.64	10	40	40
4	S4	C4	136.71	5	20	40
5	S5	C5	154.51	5	30	40
6	S6	C6	170.42	5	40	40
7	S7	C7	176	5	20	30
8	S8	C8	171.22	5	30	30
9	S9	C9	202.70	5	40	30

Figure 3.1 : Surface structure of cotton-elastane knitted (single jersy) fabric (a) original picture (b) schematic.

Second type of fabric was single jersey knitted fabric with areal density (136 g/m^2). The eight types of polyesters (textured and non-textured) fabric samples with different cross-sections (round, w shape, octolobal, and hollow round) are investigated in this work are reported in Table 3.2. The structure and schematic of polyester knitted fabric sample is shown in Figure 3.2.

Table 3.2 : Description of the polyester fabric.

Sample	Type	Yarn fineness	Cross-section type
1	Polyester	150/160 textured	Hollow round shape
2	Polyester	150/160 textured	Round shape
3	Polyester	150/160 textured	Octolobal shape
4	Polyester	150/160 textured	W shape
5	Polyester	FDY non-textured	Hollow round shape
6	Polyester	FDY non-textured	Round shape
7	Polyester	FDY non-textured	Octolobal shape
8	Polyester	FDY non-textured	W shape

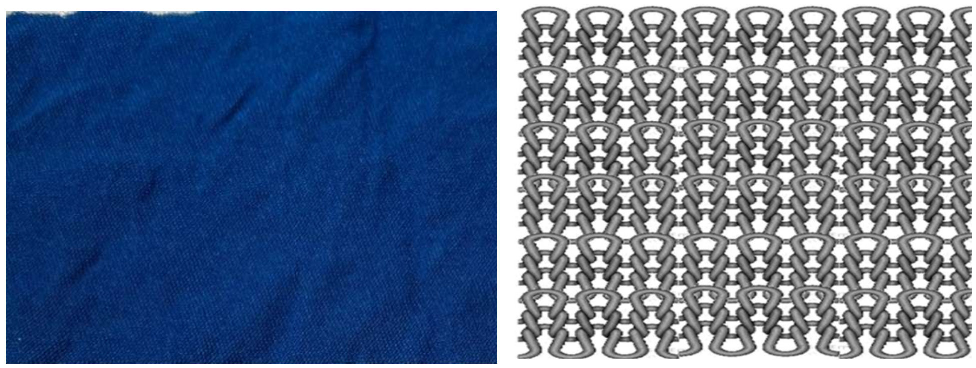


Figure 3.2 : Surface structure of polyester fabric (a) original picture (b) schematic.

The chemicals used for metallization were of analytical grade. Stannous chloride ($SnCl_2$) was acquired from Merck KGaA (Germany) and the HCl solution (37%) was supplied from Riedel-de Haen sigma Aldrich. AgNO3 was purchased from Merck and Ammonia solution (25%) was acquired from Merck KGaA (Germany). Copper (II) sulfate pentahydrate was purchased from Carlo Erba reagents (France), potassium sodium tartrate (KNaC4H4O6•4H2O) and formaldehyde (HCHO) was acquired from Merck, sodium hydroxide (NaOH) was supplied from Riedel-de Haen sigma Aldrich.

3.2 Methods

The experimental section is divided into three parts.

- First part, is the deposition of Cu by electroless plating over cotton knitted fabric (9 different GSM)
- Second part, is the copper electroless plating over cotton knitted fabric (3 different GSM) with laser pre-treatment
- Third part is the Cu electro-less plating on electroless plating of copper over textured and non-textured polyester fabric having different correctional yarns

3.2.1 Deposition of copper by electroless plating over cotton knitted fabric (9 different GSM)

First of all, we washed the fabric samples with 3% non-ionic detergent at 20 ^{o}C for 40 minutes to remove the oil, dust or impurities from the surface of the fabrics that might have been randomly scattered during the manufacturing process. The machine used for the washing process was SDL Atlas solution and the standard used for the washing process was 15ISO 5A 1984.

The electroless plating process consists of three steps, which were carried out in this study. These are sensitization, activization, and the plating process. All three steps were performed by an electric automatic controlled temperature heater IKA C-MAG HS7 and IKA ETS-D5. After the washing process, samples were rinsed with the solution (Distilled water plus some drops of HCl) for about 5 minutes. During the **sensitization** process, washed samples were subjected to the surface sensitizer, and we used a mixture of 5g/l of Tin (II) chloride ($SnCl_2$) and 5ml/l of HCl solution at 25 °C for 10 minutes and pH 1.

After the sensitization process, the second step is **activization**. The primary purpose of the activization process is to activate the samples for the plating process. Without the proper activization operation, we cannot achieve the required goal of copper coating on the fabric surface. In the activization process, we used a mixture of 10g/l of silver nitrate and 10ml/l of Ammonia solution at 25 °C for 20 minutes. After the activization process, samples were rinsed with the solution (Distilled water plus some drops of HCl). The next and foremost step is the **plating process**. Plating bath is the most important step in the electroless plating process and is composed of 10g/l of Copper (II) sulfate pentahydrate, 45g/l of potassium sodium tartrate ($KNaC_4H_4O_6 \cdot 4H_2O$) (Complexing agent), 20ml/l of formaldehyde (HCHO) solution, and 10g/l of sodium hydroxide (NaOH). This solution was used at 30 °C for 20 minutes and pH 11. The primary purpose of the complexing agents is to reduce the formation of copper to copper hydroxides ($Cu(OH)_2$) during the process.

Having completed the plating process, the next step was the **post-treatment process**. At this stage, copper plated fabric samples were rinsed with distilled water at 40 °C for 20 minutes by an electric automatic controlled temperature heater IKA C-MAG HS7 and IKA ETS-D5. Thereafter, clean copper plated fabrics were dried in Thermo Scientific Heraeus heating and drying oven machine at 80 °C for 30 minutes and were spread at room temperature with standard conditions for 24 hours until fully dry. In the electroless copper plating process, copper metal was deposited over the fabric sample and we used formaldehyde solution as a reducing agent in the plating bath. The schematic for the deposition of copper electroless plating over cotton knitted fabric is shown in Figure 3.3.

Table 3.3 : DOE for copper plating over different GSM knitted cotton fabrics.

Number of samples	Code of uncoated sample	Code of coated sample	Fabric density g/m2	Elastane (%)	Denier	Count (Ne)
1	S1	C1	250.72	10	20	40
2	S2	C2	270.65	10	30	40
3	S3	C3	290.64	10	40	40
4	S4	C4	136.71	5	20	40
5	S5	C5	154.51	5	30	40
6	S6	C6	170.42	5	40	40
7	S7	C7	176	5	20	30
8	S8	C8	171.22	5	30	30
9	S9	C9	202.70	5	40	30
9	S9	C9	202.70	5	40	30

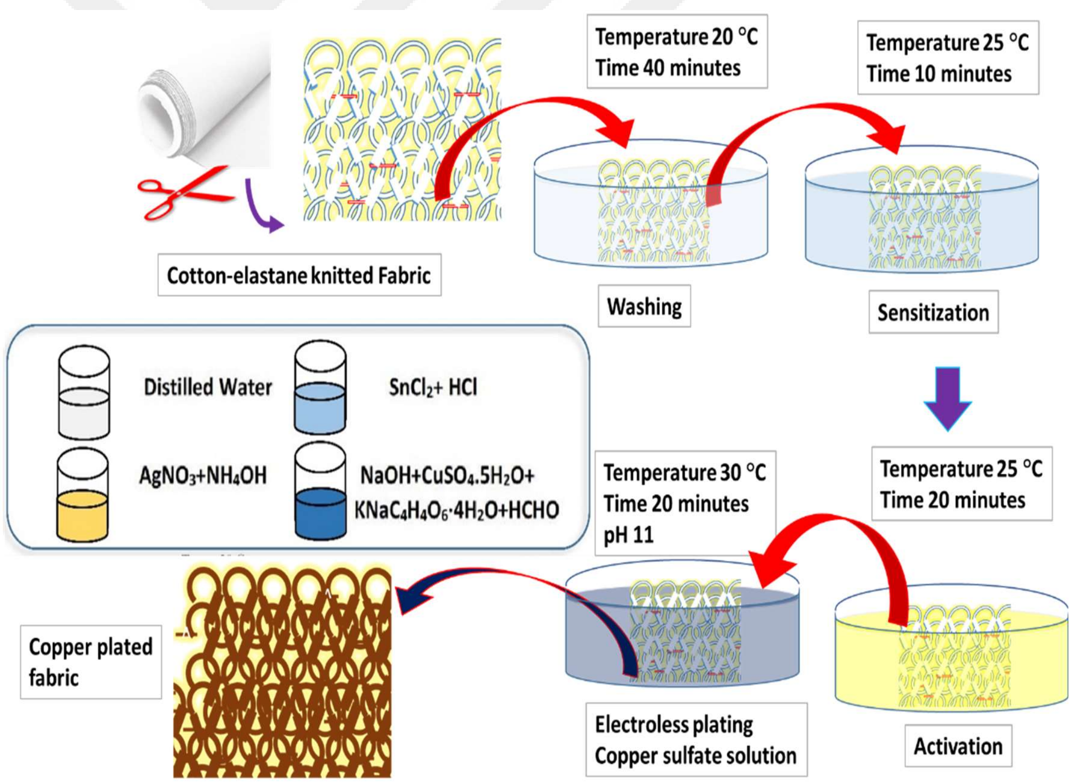


Figure 3.3 : Schematic illustration of the process of conductive fabrics via electroless plating process.

3.2.2 Copper electroless plating over cotton knitted fabric (3 different GSM) with laser pre-treatment

Three single jersey knitted cotton fabric samples (S4, S5 and S6) were selected from design of experiments of previous **study 1** as shown in Table 6**.** We selected three fabric samples (S4, S5 and S6) having light GSM (136, 154 and 176 GSM) out of nine fabric samples as mentioned in **previous study 1.** These fabric samples provided the good results of electrical conductivity, EMI shielding and more contents of metal particles.

In study 2, we pretreated the samples (S4, S5 and S6) with laser to enhance the surface roughness, then electroless plating was applied. In fact, we want to see the impact of roughness against copper deposition. Hence, the fabric samples S4, S5 and S6 were treated with CO_2 laser before conductive coating. After coating their code were termed as LS4, LS5 and LS6. Subsequently, the laser treated samples were subjected the electroless plating of copper. Now their sample codes were LC4, LC5 and LC6. The design of experiments is given below in Table 3.4.

Table 3.4 : Design of experiments for copper plating over laser pre-treated knitted cotton samples.

Code of Untreated sample	Code of laser treated sample	Code of copper treated sample	Fabric density g/m2
S4	LS4	LC4	**136.71**
S5	LS5	LC5	**154.51**
S6	LS6	LC6	**170.42**

Laser treatment

The fabrics were laser treated (20 watt power) to observe effect on the properties of conductive fabrics. First of all, the samples were washed with 4 percent detergent at 25 ^{o}C for 40 minutes to remove grease, lint or any other impurity from the surface of the substrate. SDL atlas solution washing machine was used for washing purposes and 15ISO 5A 1984 standard was used for washing.

All fabric sample surfaces were treated with CO_2 laser before conductive coating process in order to increase the surface roughness of the fabrics, thereby increasing the durability of the metal-coated fabric substrate. GS1490 100watt Golden Sign Laser Cutting Machine for a laser treatment was used. Firstly, the required pattern was made on the software and then installed in the laser machine via USB. During the laser treatment process, a speed of 100 mm/second and 20 watts per sample for 30 seconds was used. The distance between the laser point and the surface of the fabric remained 9 mm. The image of the laser machine and the laser-treated area is shown in the Figure 3.4.

Figure 3.4 : Golden sign laser cutting machine GS1490 and laser-treated area.

The electroless metallization normally consists of three stages that were applied in this work. These are processes of sensitization, activation and plating (Alemour et al, 2018a) (Bai et al, 2020). These three processes (sensitization, activation, and plating) were carried out in a beaker placed over the IKA C-MAG HS7 electric automatic heater. After washing, all samples were kept at room temperature to make them completely dry. Subsequently, sensitization, activation and plating were performed by using the same recipe (as used for study 1) mentioned above. Figure 3.4 represents the complete step-by-step conductive plating process including laser treatment of samples.

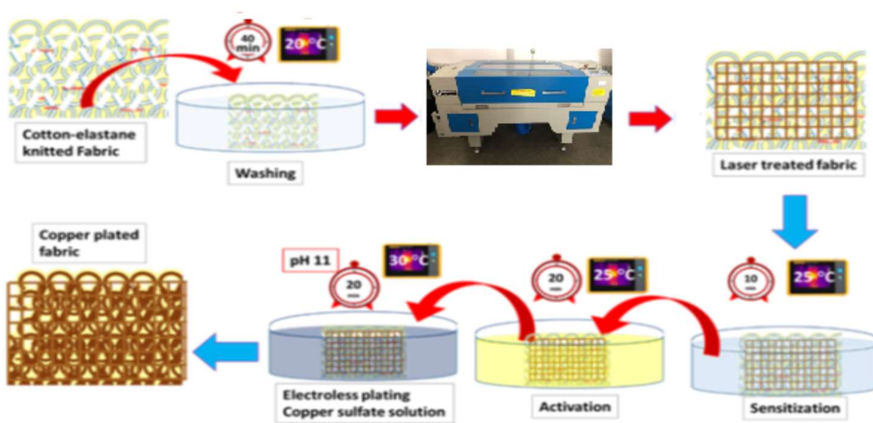


Figure 3.5 : Schematic representation of the process of laser-treated conductive fabrics by electroless copper plating.

3.2.3 Copper electro-less plating over textured and non-textured polyester fabric having different correctional yarns

Four different polyester fabric composed up of different fibrous cross sections (hollow, W shape, octalobel, and hollow-round) were selected. Each fabric was further subjected a texturing process. The False twist texturimg was done on four polyester fabrics (hollow, W shape, octalobel, and hollow-round) before use. Hence, we achieved total eight samples 4-textured (hollow round, round, octolobal and W shape) and 4–nontextured (hollow round, round, octolobal and W shape). The design of experiments is given in Table 8 below. The samples were termed into names as eight samples 4-textured (T-HR, TR, T-O and T-W) and 4–nontextured (NT-HR, NTR, NT-O and NT-W). Hence, in the same way after copper plating these samples were named as 4-textured coated (T-HR-coated, TR-coated, T-O-coated and T-W-coated) and 4–nontextured (NT-HR-coated, NTR-coated, NT-O-coated and NT-W-coated).

All the textured and non-textured substrates were washed at 30 °C for 40 minutes before the process with the SDL atlas machine (standard 15ISO 5A 1984) and then subjected to the first step sensitization, activization, and then plating process. The first step is sensitization. Here, Sn^{+2} ions are absorbed on the non-conductive polyester fabric samples from the solution of the tin chloride. The purpose of the sensitization is to open the pores of the fabric samples for the activization process. After the sensitization process, the next step was the activization. The activatization is an essential step for the metallization process. The primary aim of the activization is to trigger the surface of the fabric for the metallization process. Uniform metal deposition on the fabric surfaces is not possible without adequate activization. After activization, the next and the main step of the metallization is the plating process. In this process, the activated surface of the substrate is dipped into the metallized solution and becomes metallized after a certain amount of time. The best plating process gives the best metal coating to the substrate.

The copper metallization process is shown in Figure 3.6. Here in the plating bath, methanal is used as a reducing agent. The task of trying to reduce electroless metallization is very critical, as it helps to reduce cupric ions (Cu^{2+}) to copper metal (Cu) without any change in cupric ions. Here, the Rochelle salt was used as a complexing agent. Complexing agents generally decrease the copper generation to copper hydroxides ($Cu(OH)_2$). To make uniform and best quality deposition in the electroless metallization bath, the complexing agent is the critical property. Complexing agents are used to increasing the life of the plating bath and its stability. The plating rate of the electroless plating bath can be increased by adding the complexing agent's quantity.

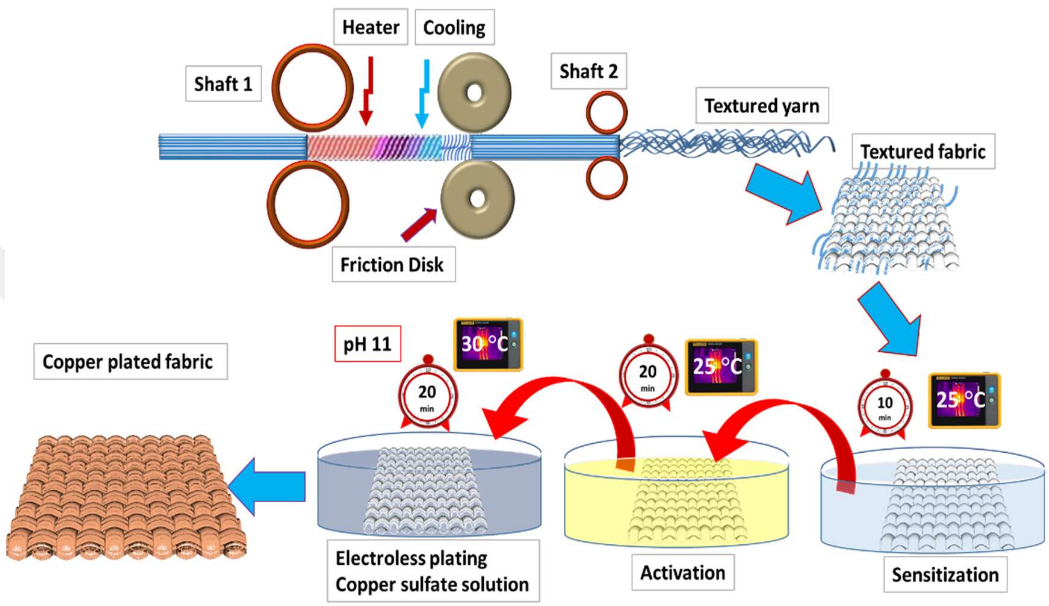


Figure 3.6 : Schematic representation of copper particles deposition on knitted fabric.

Table 3.5 : The DOE for the copper electro less plating over different cross-sectional textured non-textured polyester fabrics.

No of Samples	Fabric type	Cross-section type	Sample Code before copper coating	Sample Code after copper coating
1	Polyester textured	Hollow round shape	T-HR	T-HR-coated
2	Polyester textured	Round shape	T-R	T-R-coated
3	Polyester textured	Octolobal shape	T-O	T-O-coated
4	Polyester textured	W shape	T-W	T-W-coated
5	Polyester non-textured	Hollow round shape	NT-HR	NT-HR-coated
6	Polyester non-textured	Round shape	NT-R	NT-R-coated
7	Polyester non-textured	Octolobal shape	NT-O	NT-O-coated
8	Polyester non-textured	W shape	NT-W	NT-W-coated

3.3 Characterizations

3.3.1 Surface morphology testing

The surface morphology of coatings was studied using a scanning electron microscope (SEM). The Tescan VEGA III TS5130 SEM device applied a 10 kV accelerated voltage. The elemental percentages by weight was determined using EDX analysis. The XRD investigation was carried out using a diffractometer with a Cu Ka1 radiation (5 1.5406 A) power requirement (40 kV/30 mA) and a typical X-ray tube. The X - ray diffraction patterns were recorded in the 10–80° range with a 0.02° step size.

3.3.2 Electrical conductivity testing

The surface and volume resistivity of the samples were investigated employing concentric electrodes (as shown in Figure 26) using 100 V Dc power source, setting temperature at T = 21°C and a humidity levels of RH = 54 % in an air-conditioned room, according to ASTM D257-07. Prior to the experiment, samples were put in an air-conditioned room for 24 hours. The volume resistivity of a sample is determined by placing a voltage potential from across the opposite ends of testing samples and detecting the current that flows through it. The volume resistivity, V [mm], was derived using the following equation:

$$\rho_v = R_v\left(\frac{S}{t}\right) \tag{3.1}$$

here RV [Ω] indicate the volume resistivity measurement, t shows the fabric thickness [mm], S is the electrode surface area [mm^2] (D2/4), and D2 is the outer ring electrode inner diameter [mm].

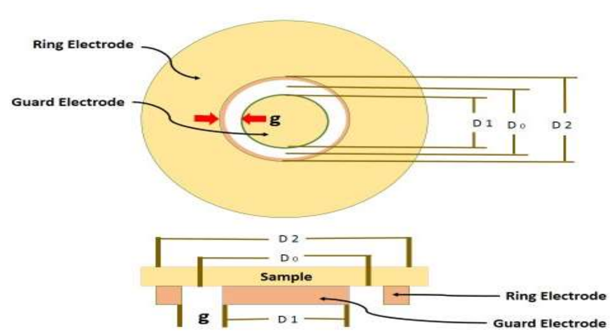


Figure 3.7 : Schematic representation and dimensions of concentric electrodes employed for volume and surface resistance measurement, where D1 = 50.4 mm, D2 = 69 mm.

3.3.3 Electromagnetic shielding testing

It was examined at a frequency ranging from 30 MHz to 1.5 GHz using the coaxial transmission line technique and the insertion loss principle followed by the standard testing protocol ASTM D4935-10. The measuring set-up is comprised of a sample holder. The output and input of sample holder is linked to the network-analyzer. The sample was held in a shielding effective test fixture (Electro-Metrics, Inc., model EM-2107A). The electromagnetic impulses were generated and received using a network analyzer (Rohde & Schwarz ZN3). As shown in equation 3, the ratio of transmitted to incident power of electromagnetic radiation was determined to describe the efficacy of EMI shielding (SE) in dB.

$$SE\ (dB) = 10\ log \frac{P_t}{P_i} \tag{3.2}$$

here, Pi and Pt and are the power density (W/m^2) in the absence of samples (incident) and with the samples (transmitted), respectively.

3.3.4 Durability testing

The durability to washing of fabrics coated with metals was investigated in order to have a better understanding of their performance in service. The metal coated textiles were stirred vigorously in 5 g/L solution of standard detergent using a liquor ratio of 50:1 following ISO 105-C01 standard method. For half an hour, the sample was washed at 40 °C at a stirring rate of 800 rpm. Prior to experimentation, the washed samples were dried and maintained for 24hrs in a standard environment (25ºC temp, and 65% humidity). Later, the effectiveness was confirmed using electrical conductivity measurements and scanning electron microscopy (SEM) examinations of metallic particles over the surface of fabric.

3.3.5 Rubbing test

The abrasion tests for all of the knitted cotton fabrics was performed by Martindale James H. Heal & Co., Ltd. The parameter set for the knitted cotton fabric was 9 kPa, and the standard used for abrasion tests was D4966-12.

3.3.6 Microscopic

The microscopic analysis of all of the coated and uncoated fabric samples was carried out using a Motic SMZ-140-143-FBGG Stereo Zoom Microscope.

3.3.7 Inductive coupled plasma

The Perkin Elmer Optima 3000 XL inductively coupled plasma-optical emission spectrometer was employed. A cyclone spray chamber, an excentric nebulizer, and a basic one-piece extended torch equipped to the quartz injector tube were included in the instrument. The specimens were dried at 60 ℃ for for 48 hrs, cut, followed by weighing to determine the exact metal content of textiles. 0.5 g of dried specimen were placed in microwave oven digestion vessels and processed using 8 mL of 7 M HNO3. The specimen digestion was performed using a closed-vessel microwave digestion process MLS 1200 Mega (manufactured by Milestone) with temperature regulation. The following was the temperature schedule: 5 minutes at 145 °C (250 W), 15 minutes at 175 °C (300 W), and 20 minutes at 200 °C (power 350 W). The resultant concentrations were cooled before being filled to a capacity of 10 mL of 7 M HNO3 and examined by ICP-OES. Following the same method, the IAEA-V-9 reference material for copper trace element measurement was digested.

3.3.8 Weight gain

The following equation 4 was used to evaluate the weight gain percentage during electroless plating.

$$w = \frac{m - m_0}{m_0} \times 100 \quad (3.3)$$

Here, m indicates final mass, m_0 denotes original mass of substrate while w indicates the total weight gain percentage.

4. RESULTS AND DISCUSSION (1ST STUDY)

Deposition of copper by electroless plating over cotton knitted fabric (9 different GSM)

4.1 Microscopic Analysis

By microscopic examination, we investigated the fabric surface morphology of the knitted cotton fabric before and after the coating process that how it affects by the electroless plating process. By comparing the appearance of coated and uncoated knitted cotton fabric shown in Figure 2.5, surface of the coated fabric was found to be covered by bright copper coating. The results were suggested to be caused by the copper metallic materials used in the plating process. The copper coatings seemed to be very smooth in the observation. No peeled-off path was observed. Furthermore, as illustrated in Figure 4.1, plating is uniformly over the fabric surface in the form of an orbicular shape. It has the appearance of crocodile skin. Furthermore, the SEM analysis in Figure 2.6 can be attributed to the deposition being denser and heavier. The S1 to S9 are the fabric samples without coating while C1 to C9 are their corresponding coated samples. The C1 to C9 samples surfaces are clearer after coating. As under the light microscope they are clearer due to the proper reflection of coating surface. Hence, it reinforces the claim of proper even coating. Furthermore, the samples C4, C5 and C6 are showing more dense coating as compared to all other samples. The reason is these samples were fabricated with light GSM of fabrics that 136, 154 and 170 g/m^2. These fabric samples were having maximum amount of metal and lower electrical resistivity.

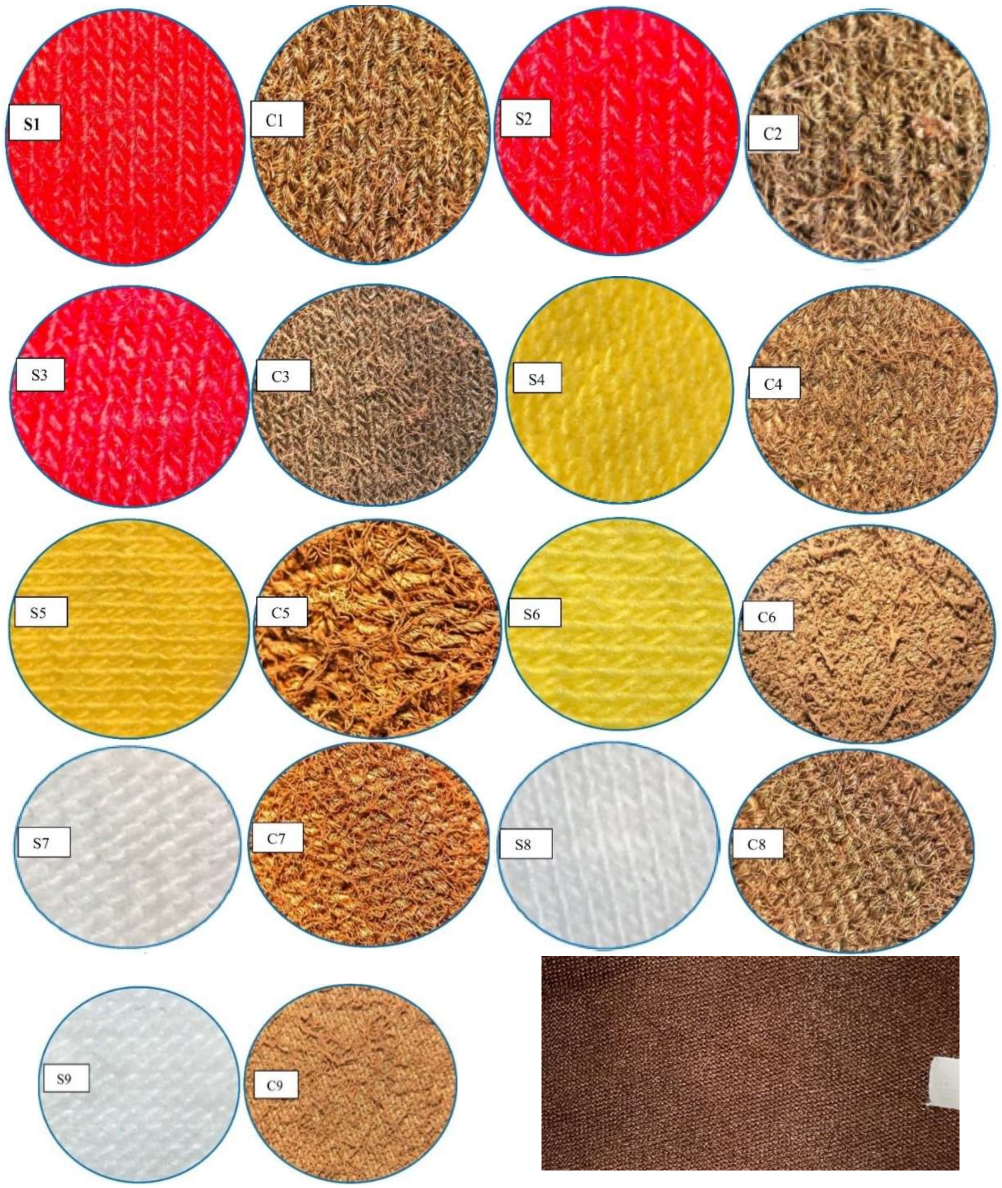


Figure 4.1 : Microscopic morphology of all the uncoated and coated Knitted cotton fabric.

4.2 Scanning Electron Microscope

Images of the knitted cotton fabric treated with 20 minutes of copper plating solution are shown in Figure 4.2 and Figure. 4.3. By comparing the appearance of coated and uncoated knitted cotton fabric shown in Figure 28, surface of the coated fabric was found to be covered by bright copper coating. Copper deposition was observed to be denser and uniform. After activation, the fibre surface was plated with a thin coating of silver. It was discovered that the sample prepared by subsequent electroless-plating over it might form a thicker layer of Cu. SEM images of the coated-knitted cotton fabrics at a specific point showing the coated material onto the surface of the fabric with a close view are shown in Figure. 4.2. Where, all the SEM images of coated fabric showed that there is a good coating over the surface of the cotton fabrics. The images have been obtained at 500x magnification of both coated and uncoated samples. It has been observed by their 5kx magnification that plating materials is properly deposited on every single yarn of the fabric samples and they have chains that are interlinked with eacother.

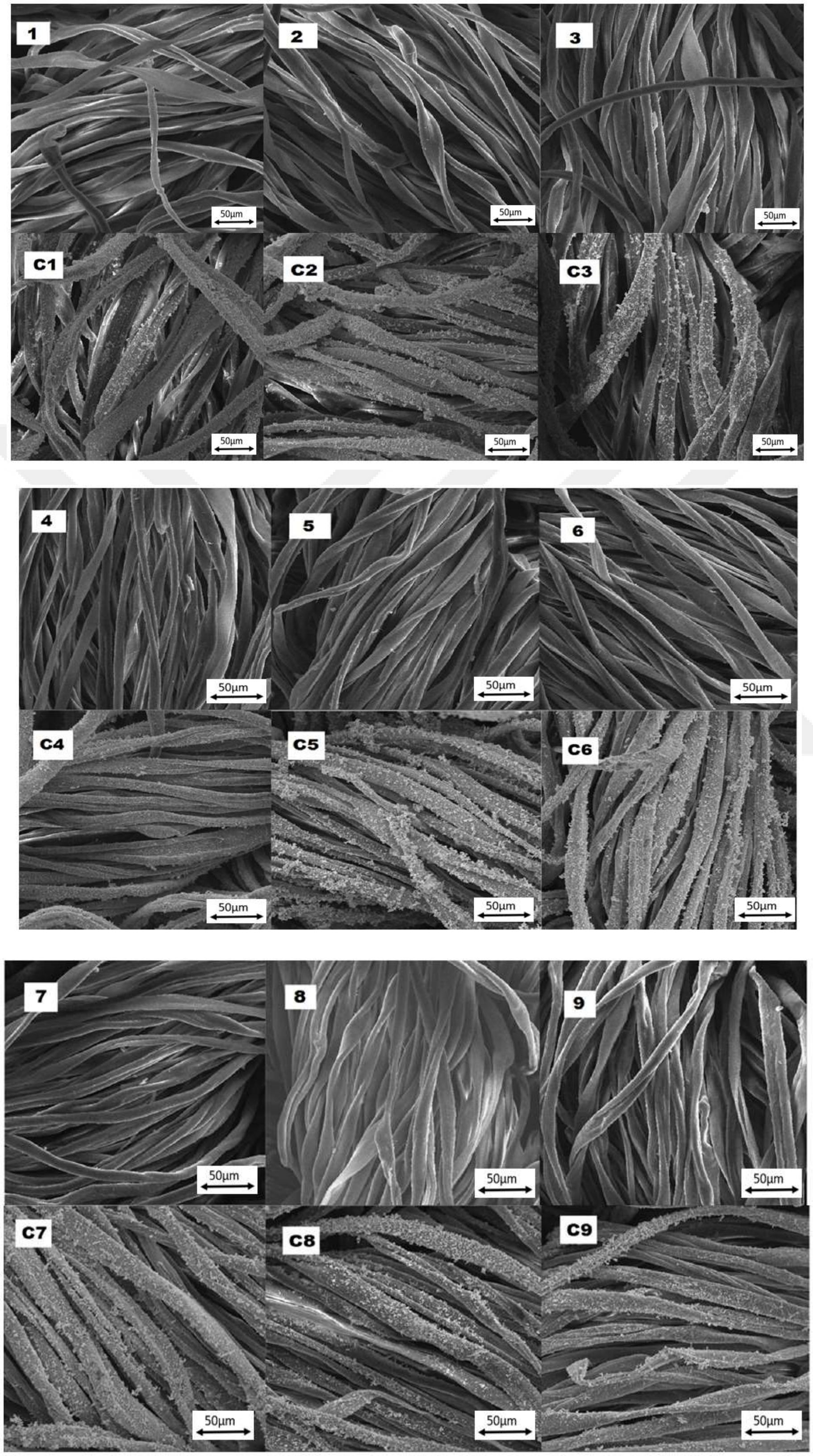


Figure 4.2 : SEM images of the all the coated and uncoated nine samples at 500x magnification.

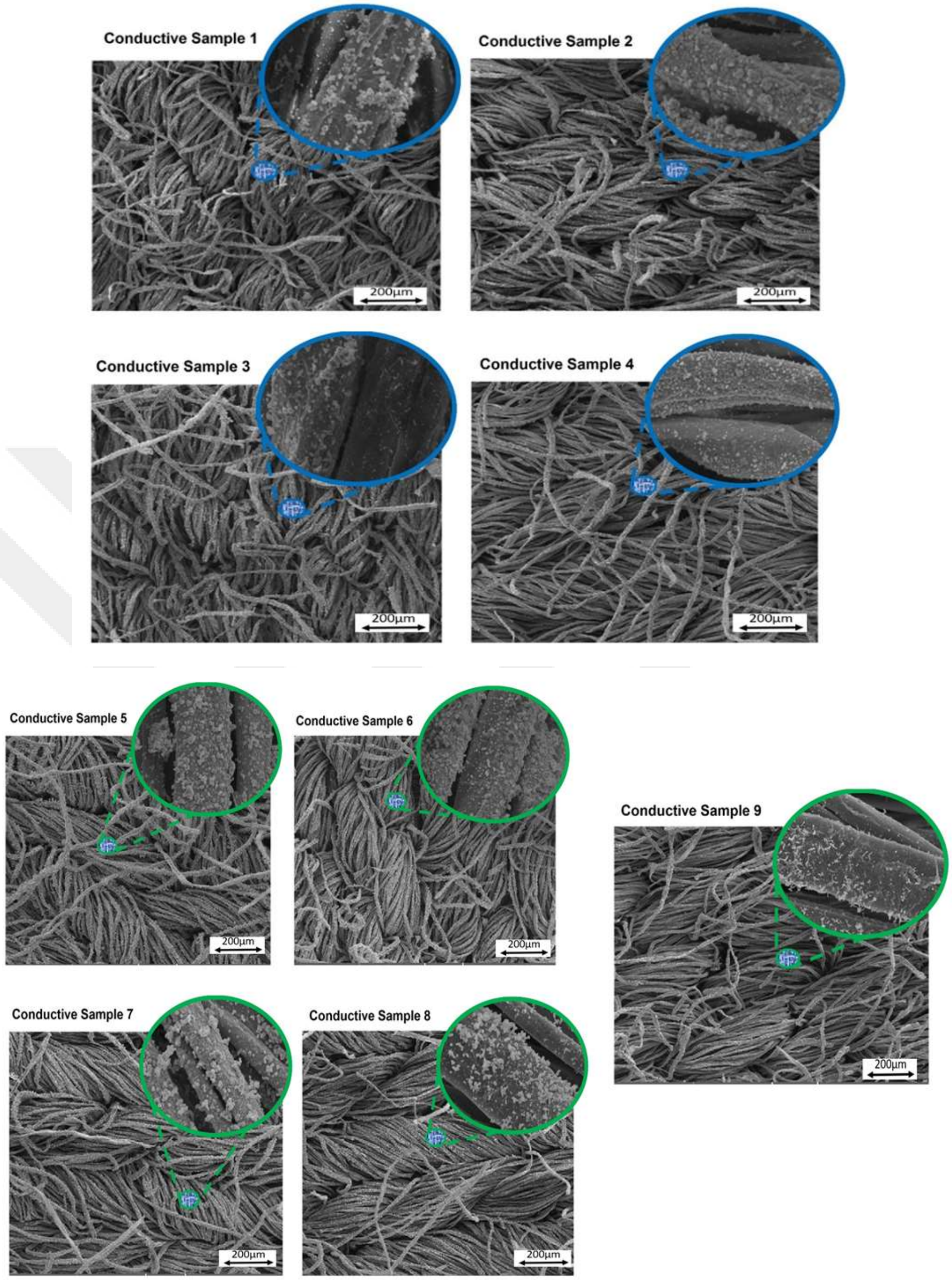


Figure 4.3 : SEM images of all the conductive nine samples at 100x magnification and their close view at a magnification of 5kx.

4.3 Energy-Dispersive X-Ray Spectroscopy (EDX)

The surface structures of the samples with copper plating are observed by EDX. The EDX technique have been used for the specific point on the surface of the fabric to check the chemical composition on that specific area. In the Figure 30, it showed that copper, silver, carbon and oxygen were deposited on the surface of the sample. It means that copper is well deposited on the surface of the samples.

Furthermore, the amount of copper and other elements is given in weight percentage in given Table 4.1. We can se that the amount of copper deposition is more for the samples having less electrical resistivity is very less electrical resistivity. These are the fabric samples with less GSM and good electrical conductive. We can say that the amount of metal deposition have direct relation with electrical conductivity.

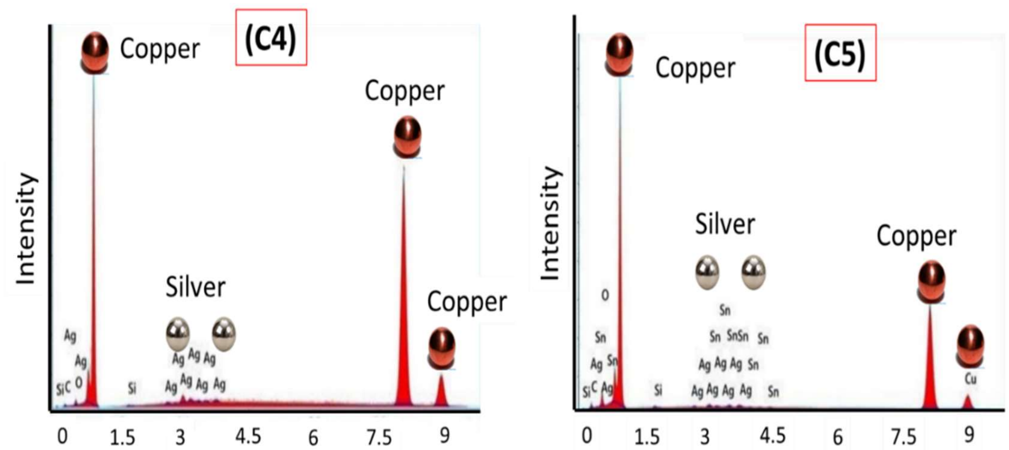


Figure 4.4 : Elemental peaks of copper and silver metals appeared on C4 (136 GSM) and C5 (154 GSM).

Table 4.1 : EDX analysis of conductive fabrics.

Resistivity (Ω.cm)	**Code of sample**	**Wt. % C**	**Wt. % O**	**Wt. % Ag**	**Wt. % Ca**	**Wt. % Cu**
21.64	C1	52.67	32.21	3.62	0.85	10.65
77.56	C2	51.89	32.57	3.97	2.39	9.18
93.81	C3	54.86	31.91	2.24	2.17	8.82
3.24	C4	45.06	31.83	2.33	0.94	19.84
5.34	C5	47.48	32.24	2.58	1.25	16.45
8.90	C6	50.84	33.63	2.63	0.83	12.07
16.14	C7	51.85	32.61	3.61	0.85	11.08
30.45	C8	52.98	33.41	3.01	1.13	9.47
54.88	C9	51.87	33.91	2.62	1.17	10.43

4.4 Electrical Volume Resistivity

The electrical resistance values of all the samples for this study are reported in Table 10. The Results showed that samples C4, C5 and C6 have very less electrical resistivity meaning excellent conductivity values as compared to other samples. The reason is that, the samples C4, C5 and C6 were composed up with lower GSM (136, 154 and 170). It means the lower GSM was more convenient for metal deposition. The deposition over the low GSM fabrics gave the even deposition of copper particles over the surface. The behaviour can be further supported by SEM analysis. Where a greater number of particles are deposited over the low GSM fabrics. Furthermore, EDX analysis is also supporting the fact. Secondly, the samples C4, C5 and C6 having less amount of elastane (5%) and more cotton (95%). The cotton fiber due to heterogeneity in nature and having more tendency for copper (positive ions) provide more deposition of metal. Hence the electrical resistivity will be low for the fabric samples having more amount of cotton (95%, C4, C5, C6, C7, C8, C9) as compared to less cotton (90%, C1, C2 and C3). The effect of low electrical resistivity with low GSM and more copper deposition is also clear from the results of ICP. Where the low GSM fabrics are showing more tendency for the deposition of copper in ppm.

Hence, the lower GSM resulted in lower electrical resistivity of coated fabrics. This trend could be due to the formation of percolated network by creation of continuous connectivity between the small sized copper particles.

Table 4.2 : Electrical resistivity values of copper coated textile with different GSM.

Sample code	**Fabric density g/m2**	**Resistivity (Ω. cm)**	**St.Dev**	**Elastane (%)**	**Denier**	**Count (Ne)**	**Amount of copper (ppm)**
C1	250.72	67.64	1.126	10	20	40	119,000
C2	270.65	77.56	3.796	10	30	40	117,000
C3	290.64	93.81	3.226	10	40	40	111,000
C4	**136.71**	**3.24**	0.046	5	20	40	160,000
C5	**154.51**	**5.34**	0.014	5	30	40	158,000
C6	**170.42**	**8.90**	0.115	5	40	40	153,000
C7	176	16.14	0.255	5	20	30	145,000
C8	171.22	30.45	0.630	5	30	30	132,000
C9	202.70	54.88	3.631	5	40	30	125,000

4.5 EMI Shielding Effectiveness

Figure 4.5 depicts the results of EMI shielding effectiveness for the coated fabrics C4, C5 and C6. The fabric samples C4, C5 and C6 were selected for further experiments because these samples gave good electrical conductivity as compared to others. The electromagnetic shielding effectiveness was found to more with less GSM fabric samples (C4, C5 and C6). The sample C6 (170 GSM) showed the lowest electromagnetic shielding effectiveness of about 35 dB in frequency range of 600 MHz–1500 MHz. The electromagnetic shielding effectiveness of 40 dB and 50 dB was exhibited by samples (C5 and C4) respectively. The maximum shielding effectiveness in this range was attributed to increase multiple internal reflections of electromagnetic radiations due to higher electrical conductivity. The smallest electrical resistivity i.e., 3.2 cm for sample C4 significantly affected the effectiveness of the EMI shielding. Increasing copper concentration (160, 000) in the samples increased their ability to protect. Due to the homogeneous and compact packing of Cu particles on the surface of fabric, this phenomenon was attributed to enhanced electrical conductivity behaviour. From SEM images given in figure 28, which indicate an extensive network of Cu particles, help to further clarify this.

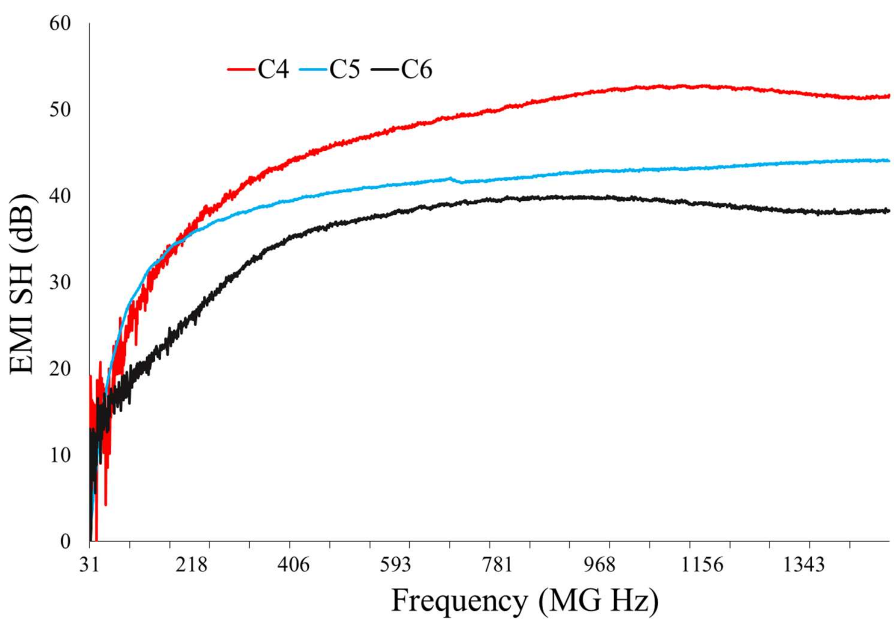


Figure 4.5 : EMI SH effectiveness for the conductive fabrics.

4.6 XRD Analysis

The phase composition of plated copper was determined using XRD analysis on the prepared plated samples. The X - ray diffraction patterns of copper plating on silver activated fabric is shown in Figure 4.6 over the 2 range of 20 to 80 degrees with a 0.02-degree step. The exact indexing of all the observed diffraction peaks to their structures reveals the purity of the phase for the deposited copper over silver. Four new major silver peaks were identified at 2θ values of 38.1, 44.3, 64.5, and 77.5, which were ascribed to the diffraction peaks of the (1 1 1), (2 0 0), (2 2 0), and (3 1 1) planes (silver) with cubic structure documented in the International Center for Diffraction Data, respectively (JCPDS data number 04-0783 card)(M. Shateri-Khalilabad, 2017)[37]. Other impurity like AgO, on the other hand, did not show any distinct peaks. We also discovered all of the diffraction peaks related to the Cu structure in the same sample. The diffraction peaks at 2θ of 43.3 o, 50.5 o, and 74.2 o, respectively, indicated the (1 1 1), (2 0 0), and (2 2 0) planes for copper [50]. The strong peak indicated the crystalline structure of copper particles. As a result, no impurity peaks were observed, with the exception of the peak of copper oxide Cu_2O phase at 2θ of 38o [51].

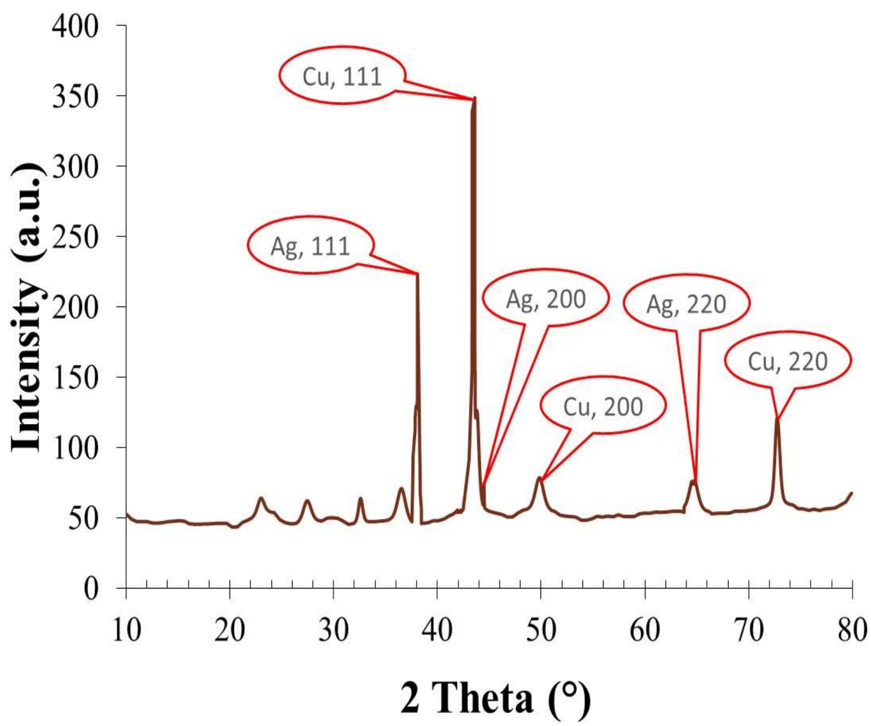


Figure 4.6 : XRD Pattern for metals coated fabric.

4.7 Washing Cycles

We performed the washing cycles of the conductive fabric to checked its resistivity and measured the weight. Usually, this method used to investigate the washing durability property of the conductive fabrics. We checked the washing durability up to 5 washing cycles. The results showed that after five washing cycles, the sample C4, C5 and C6 lost fewer conductivity values as compared to other conductive samples. Furthermore, the comparison of increase in electrical resistivity with decrease in weight of all coated samples is also shown in Figure 4.7. It can be seen that there is increase in electrical resistivity values and decrease in weight of all coated samples. The results also showed that after five washing cycles, the sample C4, C5 and C6 lost fewer decrease in weight values as compared to other conductive samples.

The behaviour can be attributed the fact that samples C4, C5 and C6 were already having very less electrical resistivity (containing more amount of metal) and there is still retention of coating even after sever washing.

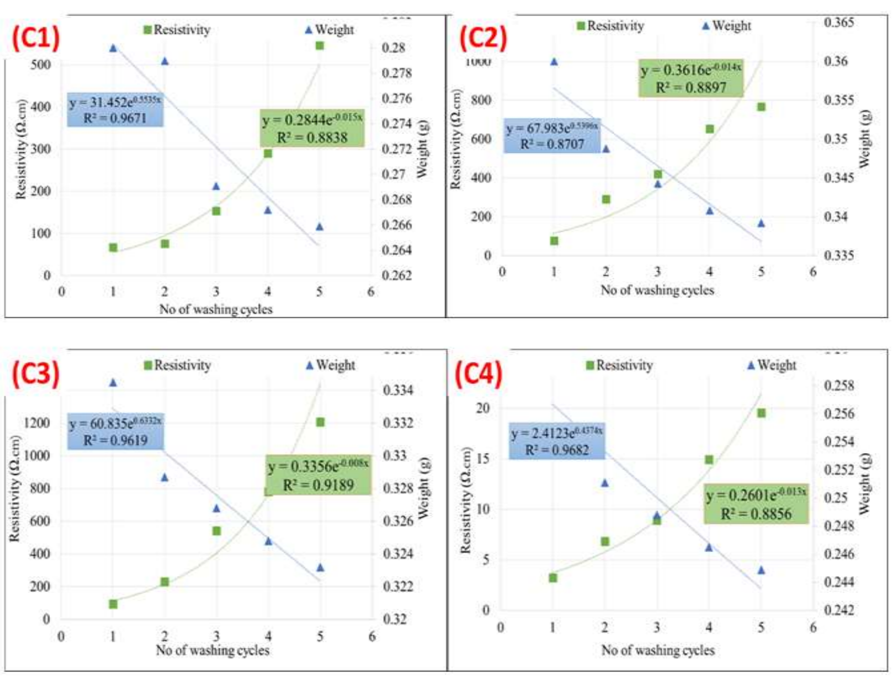


Figure 4.7 : Increase in electrical resistivity with decrease in weight of all coated samples.

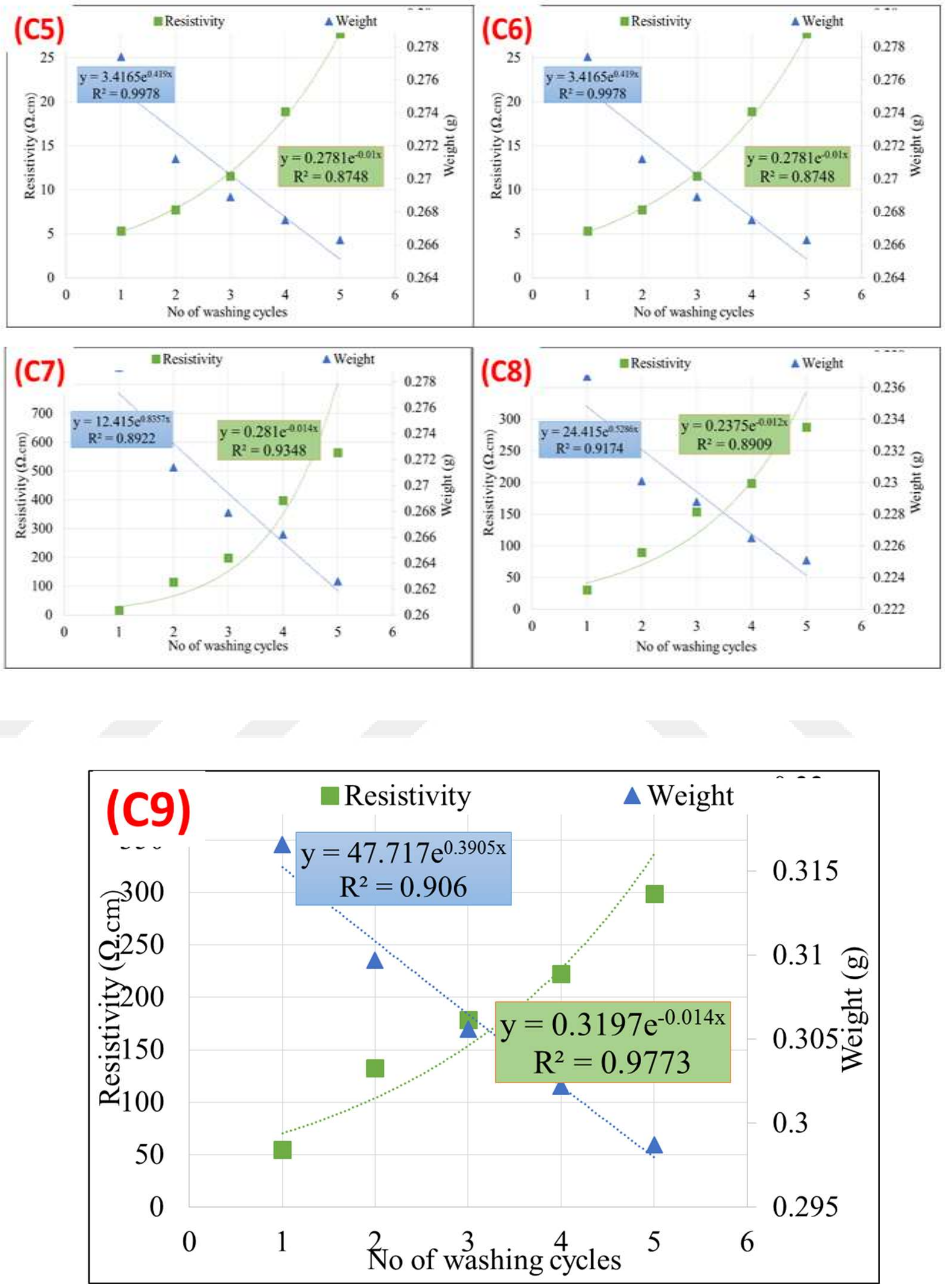


Figure 4.7 (continued) : Increase in electrical resistivity with decrease in weight of all coated samples.

4.8 Abrasion Cycles

It is an essential test and used to measure a fabric's ability to withstand abrasion. We checked the abrasion resistance up to 250 cycles.

The increase in electrical resistivity and decrease in weight with increase in number of abrasion cycles of all coated samples is also shown in Figure 4.8. The results showed that sample number C1, sample number C2 and sample number C3 showed less abrasion resistance and lost their conductivity after reaching 60 abrasion cycles and it is likely to showed no rubbing fastness whereas sample number C4, sample number C5 and sample number C6 showed excellent abrasion resistance even after reaching 250 abrasion cycles. Samples number C7, sample number 8 and sample number 9 lost their conductivity values after reaching 150 cycles of abrasion and these samples are much better than sample C1, sample C2 and sample C3. Furthermore, the results also showed that sample number C1, sample number C2 and sample number C3 showed insignificant decrease in weight and showed good rubbing fastness whereas sample number C4, sample number C5 and sample number C6 showed excellent abrasion resistance even after reaching 250 abrasion cycles.

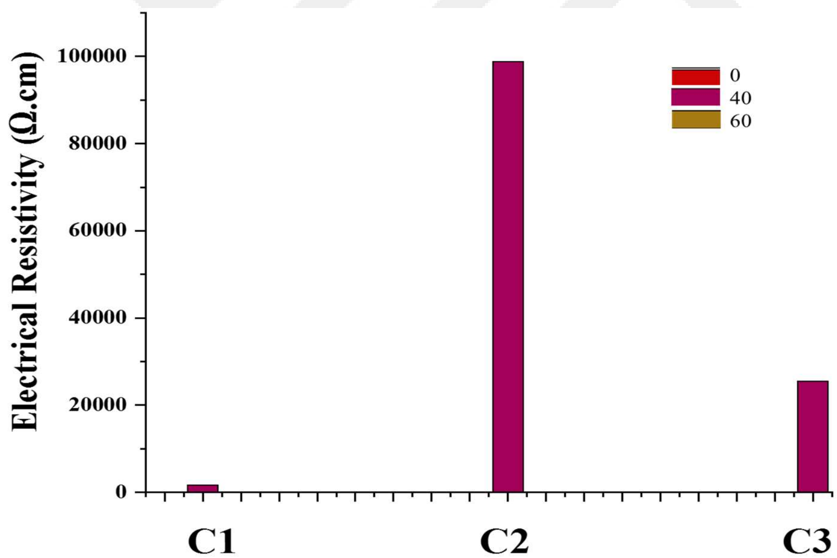
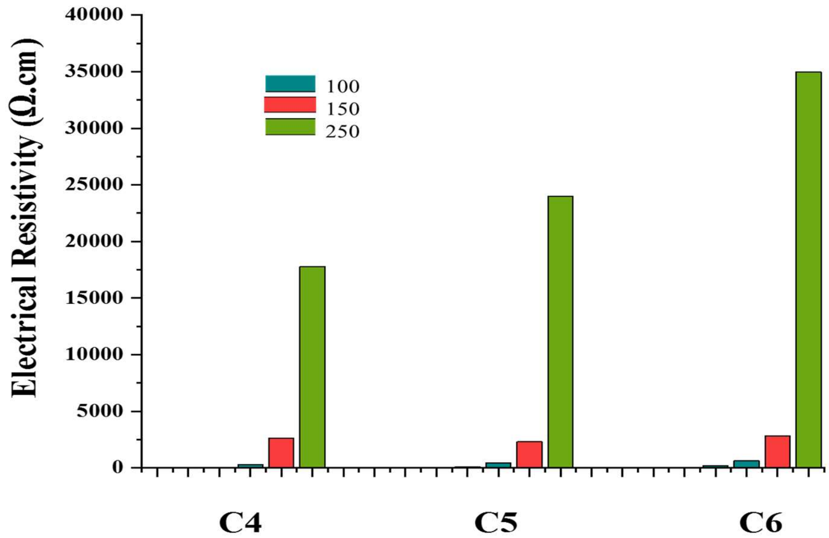


Figure 4.8 : Decrease in weight and increase in electrical resistivity with number of abrasion cycles for all coated samples.



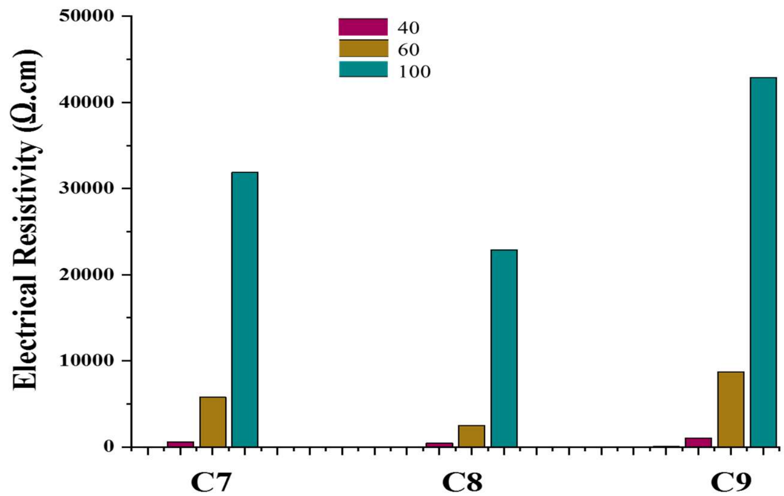
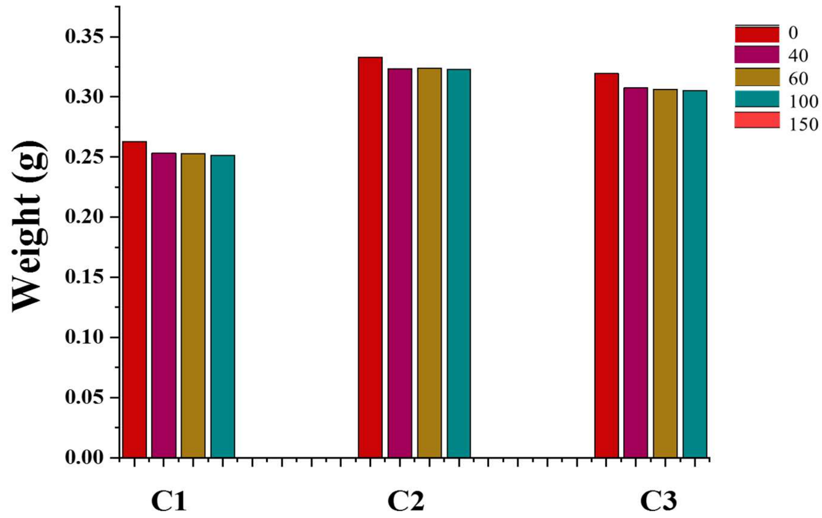


Figure 4.8 (continued) : Decrease in weight and increase in electrical resistivity with number of abrasion cycles for all coated samples.



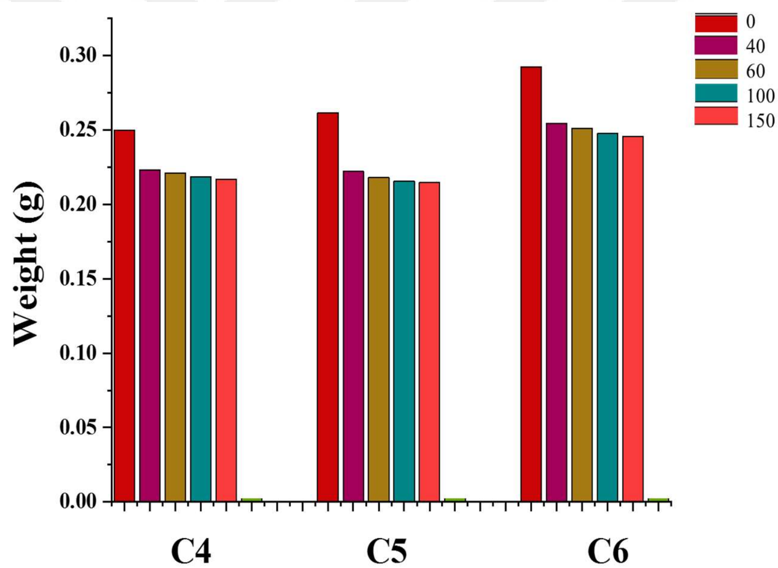


Figure 4.8 (continued) : Decrease in weight and increase in electrical resistivity with number of abrasion cycles for all coated samples.

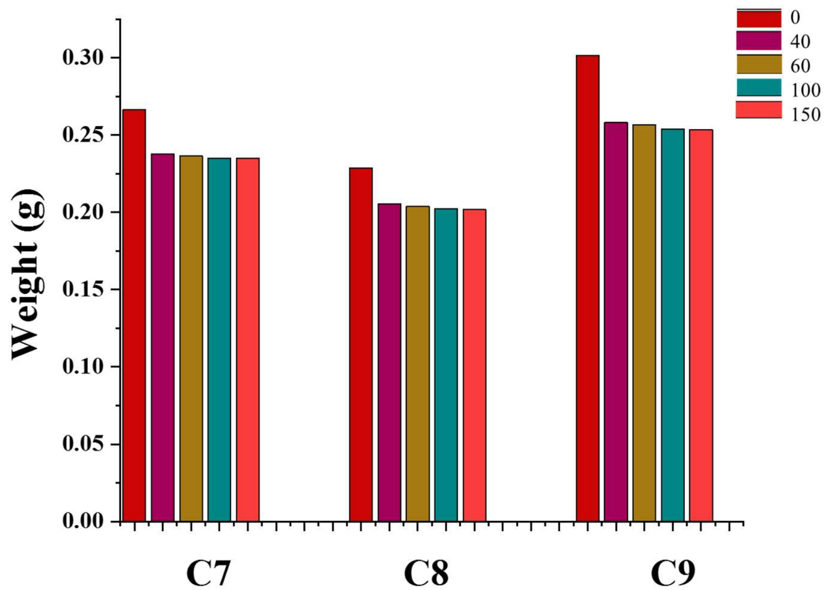


Figure 4.8 (continued) : Decrease in weight and increase in electrical resistivity with number of abrasion cycles for all coated samples.

5. RESULTS AND DISCUSSION (2ND STUDY)

Copper electroless plating over cotton knitted fabric (3 different GSM) with laser pre-treatment

In this chapter the results will explain about samples (S4, S5 and S6). Three single jersey knitted cotton fabric samples (S4, S5 and S6) were selected from design of experiments of previous **study 1** as shown in Table 6. We selected three fabric samples (S4, S5 and S6) having light GSM (136, 154 and 176 GSM) out of nine fabric samples as mentioned in previous study 1. These fabric samples provided the lowest value of electrical resistivity coupled with high EMI shielding and more contents of metal particles.

In study 2, we pre-treated the samples (S4, S5 and S6) with laser to enhance the surface roughness, then electroless plating was applied. In fact, we want to see the impact of roughness against copper deposition. Hence, the fabric samples S4, S5 and S6 were treated with CO_2 laser before conductive coating. After coating their code were termed as LS4, LS5 and LS6. Subsequently, the laser treated samples were subjected the electroless plating of copper. Now their sample codes were LC4, LC5 and LC6. The design of experiments is given below in Table 5.1.

Table 5.1 : Design of experiments for the metal coating laser treated knitted fabrics.

Code of Untreated sample	Code of laser treated sample	Code of copper treated sample	Fabric density g/m2
S4	LS4	LC4	136.71
S5	LS5	LC5	154.51
S6	LS6	LC6	170.42

5.1 Results of Microscopic Analysis

The microspic analysis was done to investigate the fabric surface morphology of the knitted cotton fabric before and after laser treatment, and also after the metal coating over laser pre-treated fabrics samples. The process show that how the surfaces will affect by the electroless plating process.

Figure 5.1 is showing the surfaces of knitted cotton fabric before and after laser treatment. It can be observed that the fabric samples gained slight roughness after the laser treatment. By comparing the appearance of coated and uncoated knitted cotton fabric shown in Figure 5.1 (a) and (b), surface of the coated fabric were found to be covered by bright copper coating. The results were suggested to be caused by the copper metallic materials used in the plating process. The copper coatings seemed to be very smooth in the observation. No peeled-off path was observed. Furthermore, it is clearly shown in the Figure 4.8 (c), that plating is clearly formed over the fabric like has an orbicular form. Similar to a crocodile-skin surface. Moreover, as the the deposition was denser and heavy this can also be attributed from the SEM analysis given in Figure 4.8. The sample (a) S4 without laser treatment and (b) with laser treatment are the fabric samples without meal coating while (c) is the fabric sample LC4 after copper deposition. The LC4 sample surfaces are clearer after coating. As under the light microscope they are clearer due to the proper reflection of coating surface. Hence, it reinforces the claim of proper even coating. The fabric LC4 samples were having maximum amount of metal and lower electrical resistivity.

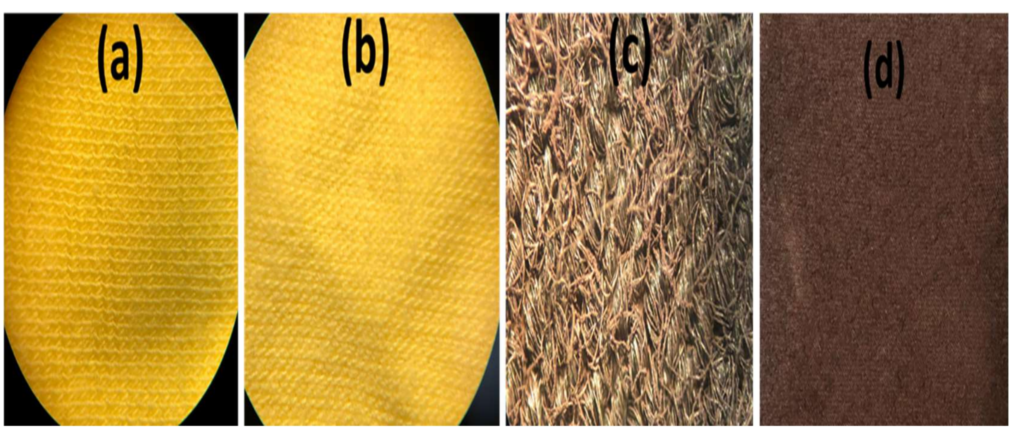


Figure 5.1 : The surfaces of knitted cotton fabric (a) before, (b) after laser treatment, (c) after copper plating and (d) original copper deposited sample.

5.2 Scanning Electron Microscope

SEM analysis of all samples was performed before and after the electroless plating process. Images of all laser samples treated with copper plating solution for 20 minutes are shown in Figure 4.8 and Figure 5.1. The SEM images indicate the deposition of copper nanoparticles of all the samples. The metal deposition was homogeneous and evenly covered over the entire surface of the laser-treated samples. By comparing the appearance of all coated and uncoated laser-treated cotton fabric samples shown in Figure 4.8, the vibrant copper metal coating was found on the surface of the coated laser-treated fabric samples. The analysis indicates that all dynamic copper coatings on the laser-treated fabric samples are due to copper metallic materials that were used in the electroless plating process. No peeled-off pattern was observed on the surface. The view of the coated and uncoated areas on the surface of the fabric by a scanning electron microscope (SEM) is shown in Figure 5.2 and close-up view of the SEM image on the surface of laser-treated fabric sample are shown in Figure 5.2. According to the SEM images, the surface of laser-treated cotton fabrics covered in a massive metal of copper nanoparticles. SEM images of all samples were obtained with a 500x magnification. In addition, 100x and 2.5Kx magnification SEM images of all substrates showed that the plating materials are deposited adequately on all surfaces of the fabric.

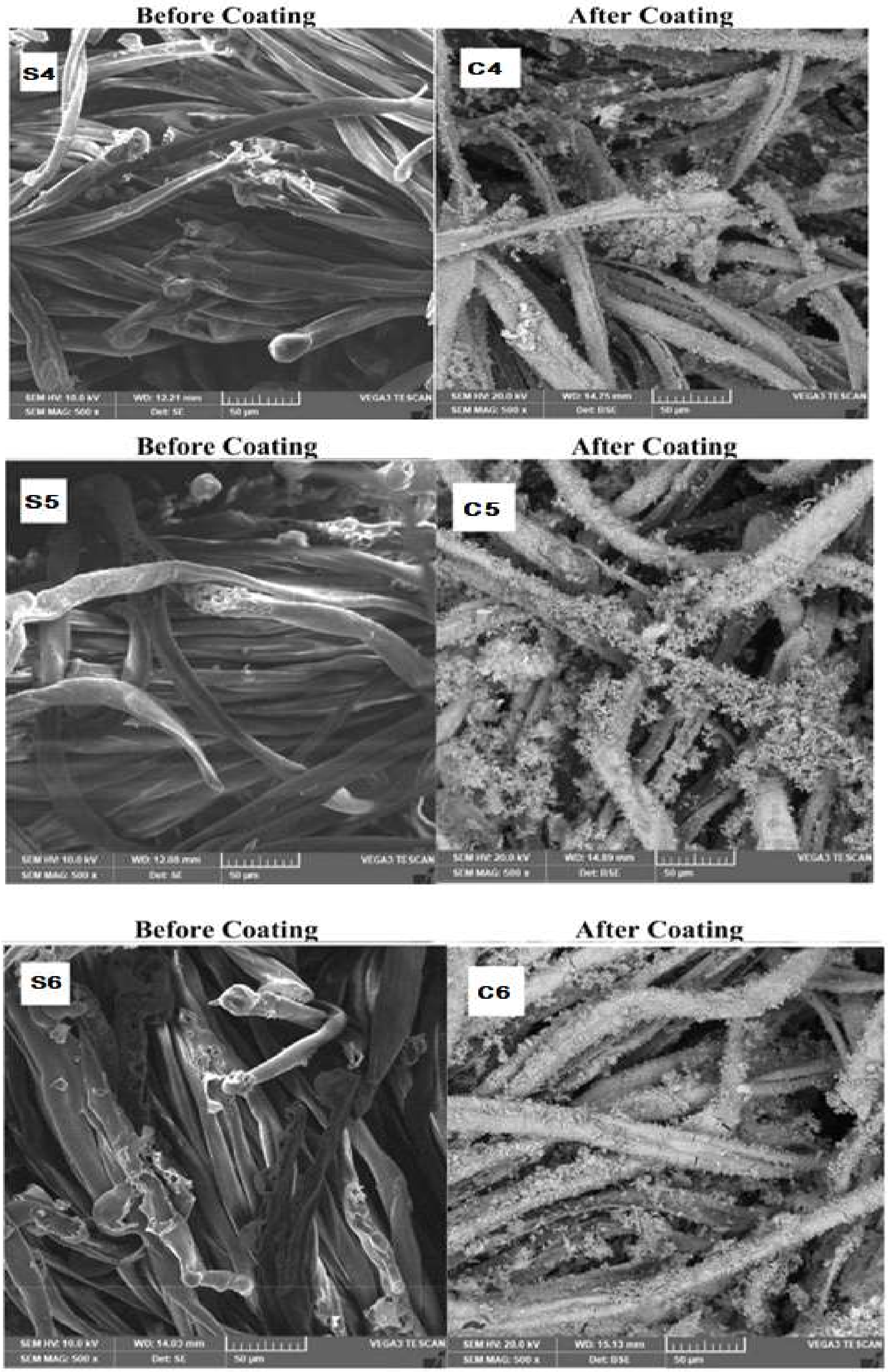


Figure 5.2 : SEM images of laser treated cotton samples (left) and Cu-coated cotton (right) at 500x magnification.

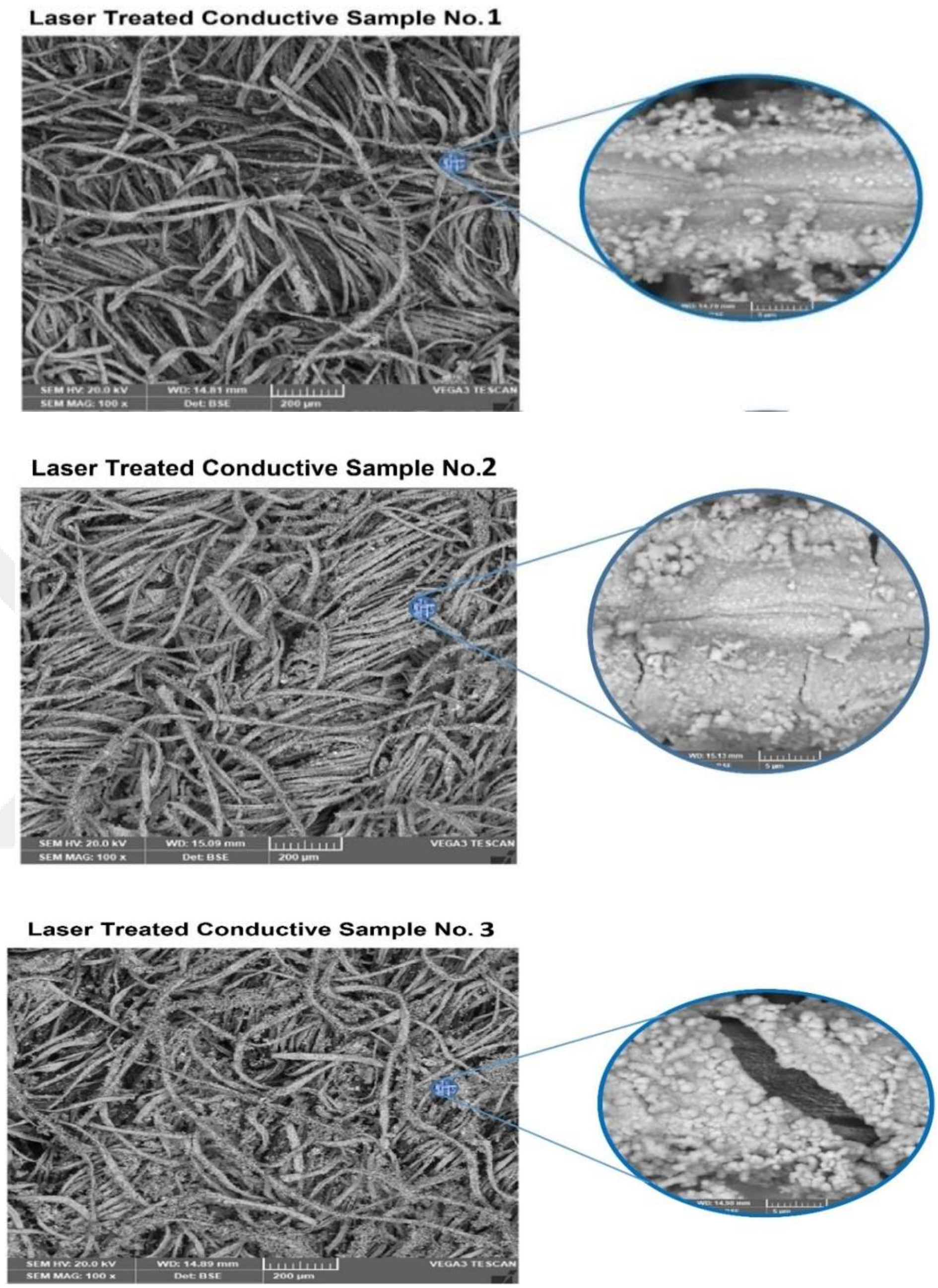


Figure 5.3 : SEM images of all the laser-treated conductive fabrics at 100x magnification and their close view at a magnification of 2.5kx.

5.3 Energy-Dispersive X-ray Spectroscopy EDX

Elemental analysis of all laser-treated copper metal-plated fabrics was evaluated by energy-dispersive X-ray spectroscopy analysis. In all the results, the copper and silver peaks were clearly visible, as shown in Figure 5.4. Following copper metal plating on the surface of the samples, the stiffness of the cotton fabrics increased.

The laser-treated samples showed relatively high levels of rigor due to the structure of the samples obtained after the laser process.

For a particular point on the surface of the knitted cotton fabric sample, the SEM-EDX technique was used to analyze the chemical components in that particular area and their corrosponding resuls are summerised in Table 12. The results showed that copper, silver, carbon, and oxygen were deposited on the surface of all the samples and that copper was at a high concentration (more than 20) while in the case of non-treated metalized copper plating, the copper concentration was up to very less (Ahmad et al, 2020). It means that the copper is entirely employed on the surface of the substrates and higher in copper plated laser-treated fabric samples.

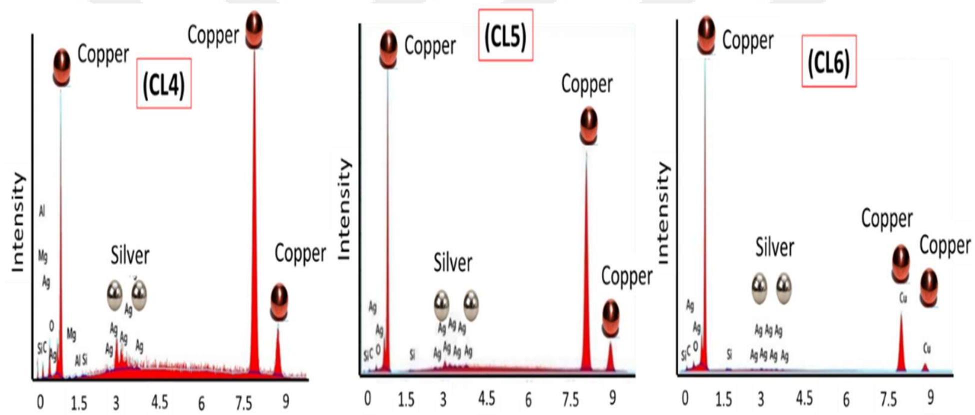


Figure 5.4 : EDX images of all the laser treated conductive samples.

Table 5.2 : EDX analysis of deposited copper on laser pretreated fabrics.

Resistivity (Ω.cm)	**Sample code**	**C Wt. %**	**O Wt. %**	**Ag Wt. %**	**Ca Wt. %**	**Cu Wt. %**
2.456	CL4	46.16	28.84	3.32	0.97	20.71
4.738	CL5	47.45	30.27	3.89	1.25	17.14
6.628	CL6	47.31	31.01	3.62	1.87	16.19

5.4 Electrical Volume Resistivity

The electrical conductivity results for all laser-treated knitted cotton fabric samples for this study are shown in the Table 5.3. The data indicate that all laser-treated fabric samples showed less resistivity (good conductivity) values (2.45 Ω. cm), which are appropriate for electronic textile applications. The lowest electrical resistivity values were recorded for sample LC4 coupled with high metal contents. The reason is that sample LC4 is the same sample C4 (having less resistivity in study

1). The sample was pretreated with laser and roughness appeared over the surface. Subsequently, metal deposition showed the bright copper coating. The copper coatings seemed to be very smooth and constructed the continuous threshold to conduct the electrical current. Moreover, as the the deposition was denser and heavy this can also be attributed from the SEM analysis given in Figure 5.3.

Table 5.3 : Electrical resistivity results for all laser-treated knitted cotton fabric.

Code of Untreated sample	Code of laser treated sample	Code of copper treated sample	Fabric density g/m^2	Resistivity (Ω. cm)	ICP (ppm)
S4	LS4	LC4	136.71	2.456	172,000
S5	LS5	LC5	154.51	4.738	159,000
S6	LS6	LC6	170.42	6.628	156.000

5.5 EMI Shielding Effectiveness

Figure 5.5 depicts the results of EMI shielding effectiveness for the samples LC4, LC5 and LC6. The fabric samples LC4, LC5 and LC6 showed better EMI SH values as compared to **previous study 1** samples (C4, C5 and C6)**.** The reason is that, laser treatment gave the roughness over the surface and attract more metal particles. The more deposition of metal over the surface results into higher electrical conductivity. Which in turn increases the EMI shielding of all laser pretreated metal coated samples.

The sample CL6 (170 GSM) showed the least EMI shielding effectiveness of about 43 dB in frequency ranging from 600 MHz to 1500 MHz. The electromagnetic shielding effectiveness of 52 dB and 65 dB was exhibited by samples (LC5 and LC4) respectively. The maximum shielding effectiveness in this range was attributed to increase multiple internal reflections of electromagnetic radiations due to higher electrical conductivity. The smallest electrical resistivity i.e., 2.4 cm for sample CL4 significantly affected the effectiveness of the EMI shielding. Increasing copper concentration (172,000) in the samples increased their ability to protect. Due to the homogeneous and compact packing of Cu particles on the surface of fabric, this phenomenon was attributed to enhanced electrical conductivity behavior. From SEM images shown in figure 5.3, which indicate an extensive network of Cu particles, help to further clarify this.

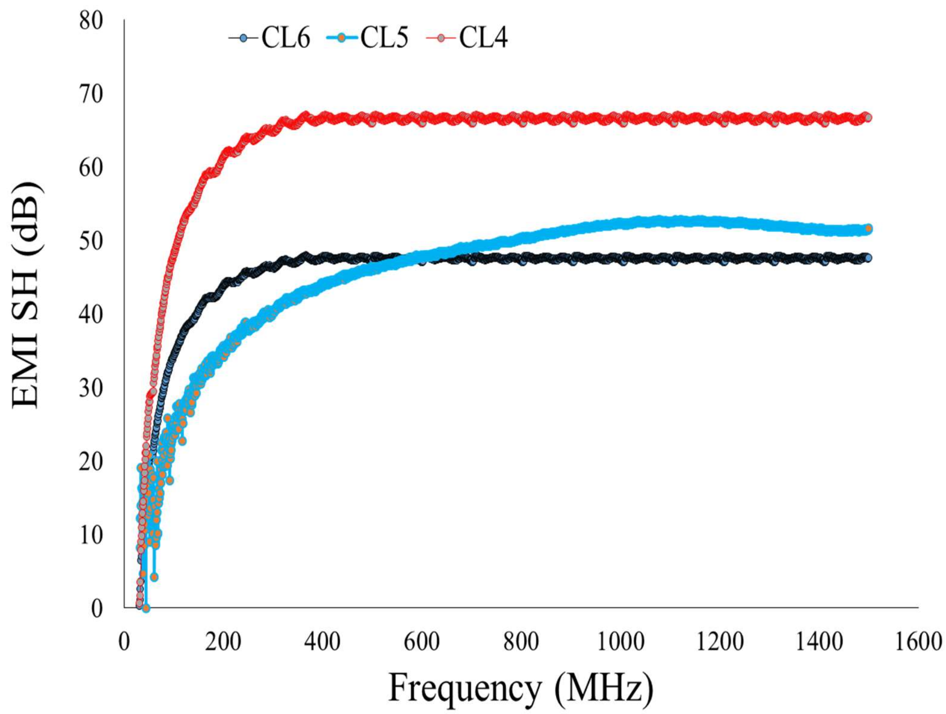


Figure 5.5 : Electromagnetic shielding results for all laser-treated knitted cotton fabric.

5.6 The durability of the knitted cotton fabric

Since excess consumption of copper metal particles can have harmful effects on the human body the durability of the laser-treated copper-plated fabric is of great importance (Muthukumar and Thilagavathi, 2012). A standard washing method was performed to examine the durability of the samples and the results are shown in Figure 5.7. The durability of all samples up to 5 wash cycles was observed in this work. The results showed that all samples expressed good conductivity values even after completing the five washing cycles.

As from the results of the washing cycles of laser-treated fabric samples, the resistance increased after the five washing cycles. As shown in the previous studies (Ravandi and Valizadeh, 2011) the possible reason of this is oxidation of the copper metal which can decrease the conductivity of the fabric surface when the samples are placed in the air.

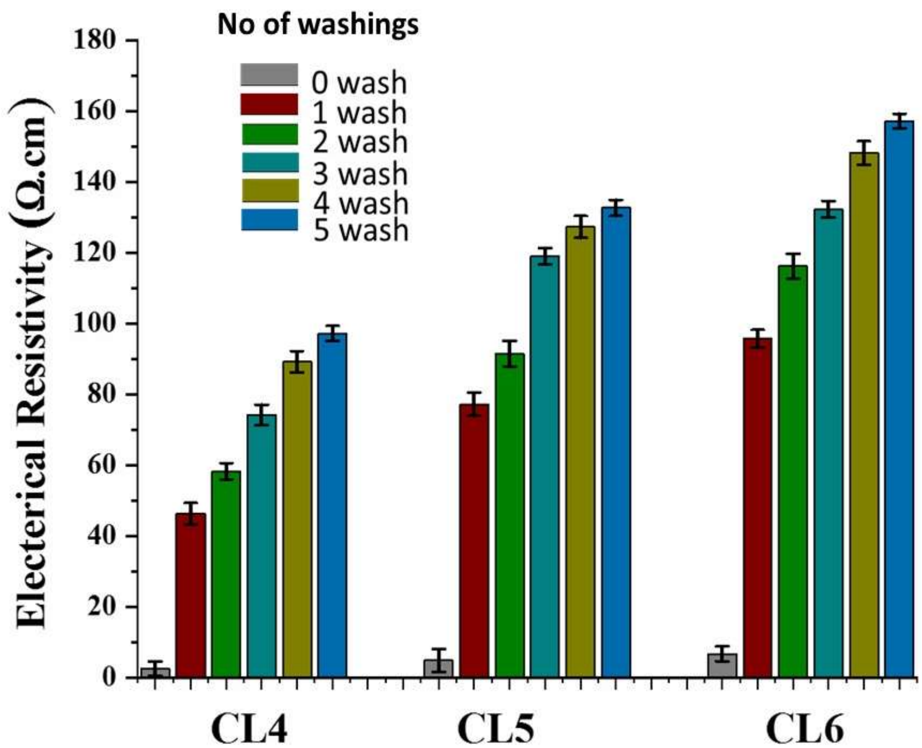


Figure 5.6 : Washing cycles of the laser-treated samples.

5.7 Wear Resistance

It is an important test and used to investigate the ability of the fabric to withstand abrasion. Abrasion resistance of all the laser-treated samples up to 250 abrasion cycles was observed in this study. The results revealed that the samples which have finer fabric structure showed high abrasion resistance and the samples which have denser fabric structure showed low abrasion resistance. The abrasion resistance depends on the fabric structure of the fabric samples and metal coating types. After the Cu-coating process, copper metals firstly accommodate on the outer layer and then inner layer of the fabric samples. Usually, the outer layer of the fabric sample is bigger as compared to inner layer due to coating process. However, when we checked the abrasion resistance of the fabric samples, then the weight of the fabric decreases because the outer layer of the metal-coated is decreasing and hence conductivity decreases. The results showed that the samples, which are laser-treated are more conductive as compared to the fabric samples that we performed in our previous work (Ahmad et al, 2020).

All the abrasion resistance cycle results with respect to weight and resistivity are shown in figure 5.8 and figure 5.9. Figure 5.8 shows the abrasion cycle graph w.r.t weight of the fabric samples. Sample LC4 and Sample LC3 were shown to have lost 10% of weight, while Sample LC2 had lost 20% of the weight. Figure 5.9 shows the graph of the abrasion cycles w.r.t electrical resistivity of the laser samples.

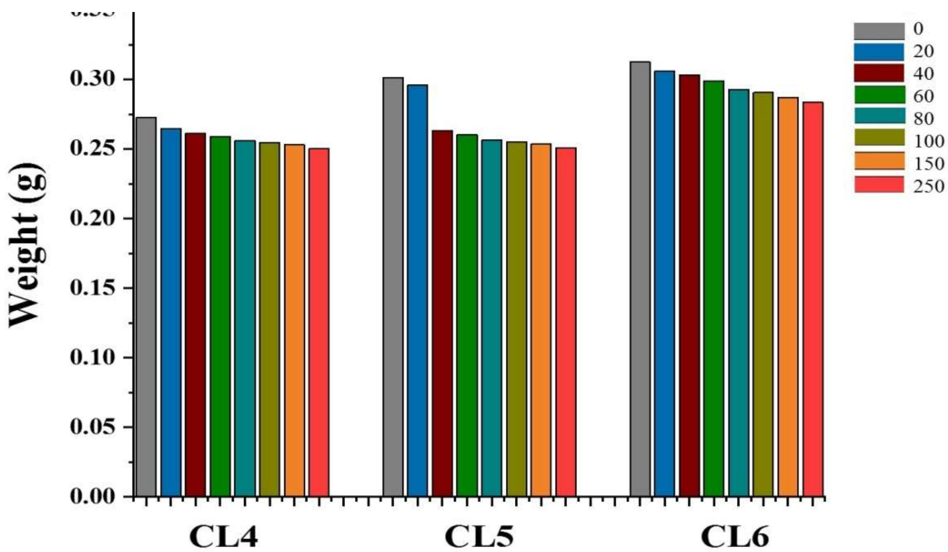


Figure 5.7 : Abrasion cycles of laser-treated sample w.r.t weight.

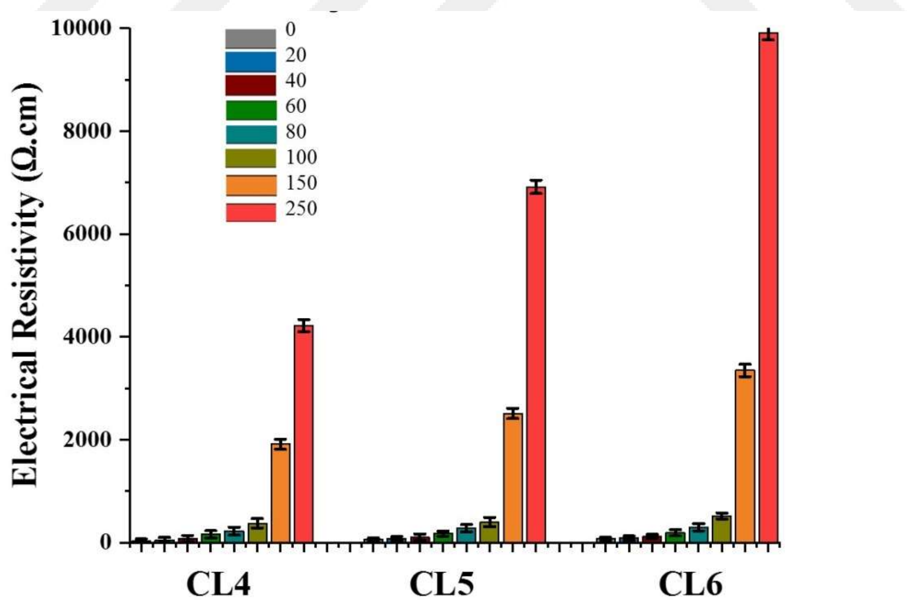


Figure 5.8 : Abrasion cycles of laser-treated sample w.r.t electrical resistance.

5.8 Mechanism of Electroless Plating of Copper

The possible steps involved in the electroless plating of copper over silver activated cotton fabric fabric has been shown in Figure 5.10.

Different parameters including complexing agent concentration, metal slat concentration, temperature, reducing agent concentration, and pH can regulate reactions occuring during the electroless plating phase. As the concentration of copper sulphate increases, the deposition rate of Cu could be enhnaced due to the higher mass transfer of Cu ions to the surface of electrons and thus the faster rate of electron transfer. However, due to the low stability of the solution bath, the rate of deposition can decrease dramatically when the Cu^{2+} ions concentration increases. This is due to a reduced effect of the complexing agent, resulting in unfavorable reactions in solution and electrolyte breakdown. Copper plating can produce a rough surface if the metal salt concentration is very high (Glenn, 1990). While selecting a reducing agent, the standard reduction potential of reducing agent should be high in comparison to the metals being reduced (copper and silver). For example, the reduction potentials of several key reducing agents, such as hypophosphite (-1.57 V) and formaldehyde (-1.30V), are more useful than the reduction potentials of silver +0.80V and copper +0.34V (Huang et al, 2007). Another essential attribute of reducing agents is their ability to self-catalyze reactions, which can facilitate the deposition of the maximum amount of copper on the target metal (silver activated fabric)(Zenglin et al, 2003). It is critical to provide anodic oxidation to the substrate metal for preventing copper from being replaced by electroless plated copper. The reducing agent formaldehyde, which is widely known for exhibiting anoidic oxidation (HCHO), has been used as a viable alternative to other reducng agents in this study. Hence, the generation of H^+ ions in aqueous solution of reducing agent causes an acidic pH of the solution.

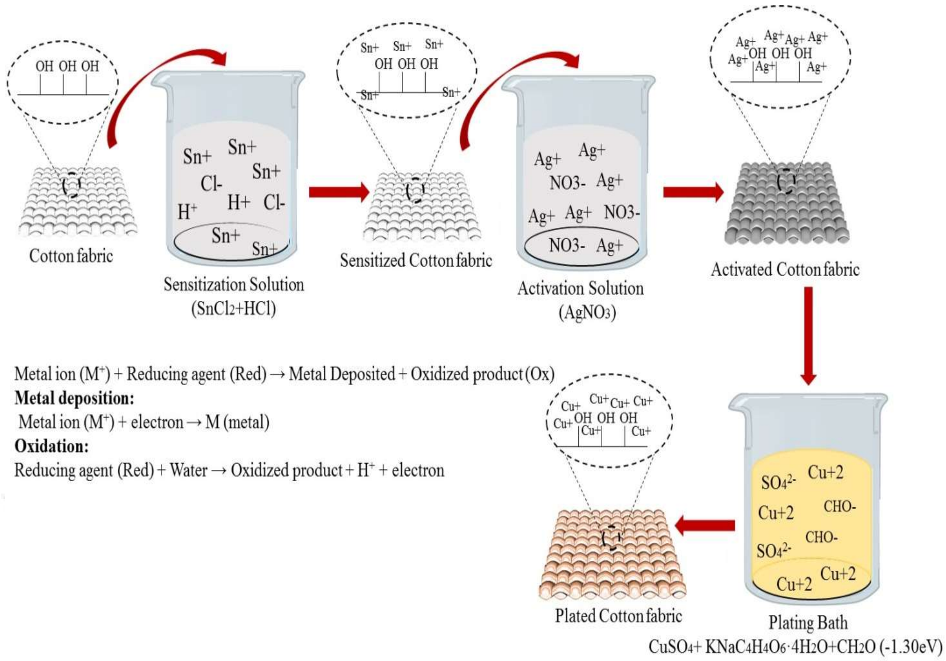


Figure 5.9 : Chemical mechanism for the attachment of copper and other ions with fabric.

The complexing agent $KNaC_4H_4O_6·4H_2O$ was employed to regulate the pH and to prevent the metal salt and reducing agent from reacting spontaneously by forming complexes with the free coordination points present in metal salt $(KNaC_4H_4O_6·4H_2O)^{-2}$. When no complexing agent is used in the solution, the potential of producing copper hydroxide or copper hydride in the solution increases (Kobayashib, 1994). The rate of reaction is determined by the concentration of the complexing agent. By using the complexing agent, the bath stability can be enhanced even further. $Cu(OH)_2$ precipitation occurs in the bath even in the absence of a reducing agent if the complxing agent concentration is lower than the concentration of Cu ions. Cu_2O formation is more likely when there is more complexing agent present. The complexing agent concentration should be chosen so that there is an adequate number of free metal ions accessible for reduction (Xie, 2002)(Kwang-Lung, 2002). When a complexing agent has a low stability constant, it results in the production of increased metal ions in comparison to the metal complex, lowering the plating rate. The amount of complexing agent used in water at equilibrium with complex and free ions can be observed in the equation 5.1.

$$M^{+z} + mL^{n-1} \rightarrow M\,L^{z-mn} \tag{5.1}$$

The stability constant can be estimated from equation (5.2)

$$\text{Stability constant} = \frac{[M\,L^{z-mn}\,]}{M^{+Z}+[mL^{n-1}]} \quad (5.2)$$

M is the metal ion, m is the number ligand or complexing agents, L is the Ligand, Ln-1 represents whole complexing agent, and z-mn shows the oxidation number.

Protons or hydroxyl ions are produced during the deposition process, changing the pH of the solution. The reactions (8) and (9) indicate, for example, that oxidation of a reducing agent can create H^+ and so lower the pH (Barker, 1993). Above 11 was found to be the best acceptable pH.

Moreover, a previous investigation revealed that when the concentrations of Cu ions and complexing agent in solution were equivalent, the precipitation of $Cu(OH)_2$ still occurred as the OH- increased. As a result, the pH had a significant impact on bath stability in electroless plating (Inoue et al, 2015).

Electroless plating bath composition is difficult to determine since it can result in a much more complex electrochemical reaction route (Ohno, 1991). Understanding of the mixed potential theory (anodic and cathodic half reactions) can be useful in the electroless plating method. The anodic reactions are associated with reducing agent oxidation (see reaction (8)), meanwhile the cathodic reactions are associated with metal reduction (see reaction (9). The selection of reducing agent is critical because it is responsible for anodic oxidation. Yu et al (Ohno, 1991) documented the anodic oxidation of formaldehyde on Cu in a study.

Anodic reactions

$$2HCHO + 2OH = 2CHO^- + H_2O + H_2 + 2e \quad (5.3)$$

Cathodic reaction

$$KNaC_4H_4O_6{\cdot}4H_2O \rightleftharpoons K^+ + Na^+ + C_4H_4O_6^- + 2OH^- + 2H^+ \quad (5.4)$$

$$Cu\,(KNaC_4H_4O_6{\cdot}4H_2O) + 2e \rightleftharpoons Cu + (KNaC_4H_4O_6{\cdot}4H_2O)^{-2} \quad (5.5)$$

The formaldehyde oxidaton is crucial because it promotes cupric-ion reduction through intermediates. The autocatalysis theory states that reducing cupric ions refreshes the electroless Cu surface, resulting in additional catalytic sites and hence accelerated oxidation of reducing agent oxidation (see Figure 5.11 and reaction) (11).

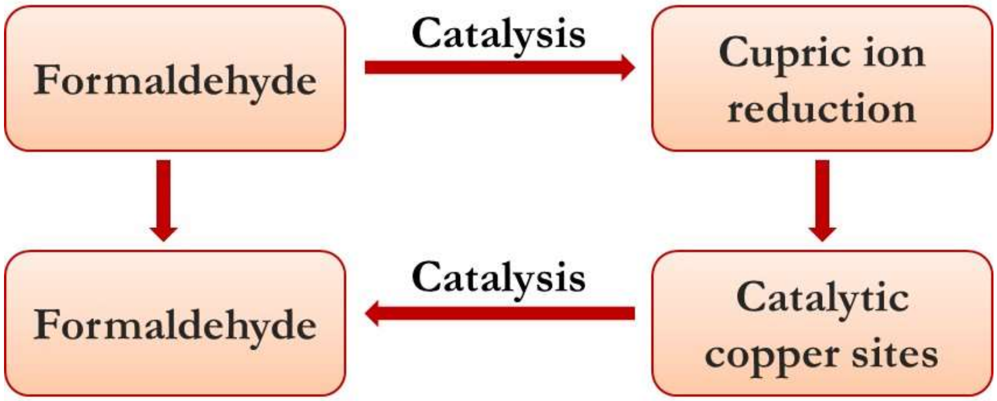


Figure 5.10 : Autocatalysis mechanism involved during electroless plating of copper.

Overall reaction

$Cu\ (KNaC_4H_4O_6 \cdot 4H_2O) + 2HCHO + 4OH^- = Cu + 2CHO^- + 2H_2O + H_2 + KNaC_4H_4O_6 \cdot 4H_2O^{-2}$

6. RESULTS AND DISCUSSION (3RD STUDY)

Electroless plating of copper over textured non-textured polyester fabric having different correctional yarns

In this chapter the results will explain about **third study,** which was performed by using the same electroless plating recipe on polyester fabrics. In fact, for hosiery, apparel, and textile applications, the cotton (among natural fibers) and polyester (among synthetic fibers) are most commonly used. They are also blended in different ratio to use them in number of applications. So, we decided to apply same recipe of electroless plating on the synthetic fiber polyester. The polyester fibers (textured and non-textured) were selected for this purpose. Four different polyester fabric composed up of different fibrous cross sections (hollow, W shape, octalobel, and hollow-round) were selected. Each fabric was further subjected a texturing process. The False twist texturimg was done on four polyester fabrics (hollow, W shape, octalobel, and hollow-round) before use. Hence, we achieved total eight samples 4-textured (hollow round, round, octolobal and W shape) and 4–nontextured (hollow round, round, octolobal and W shape). The design of experiments is given in Table 8 in section **3.2.3** of materials and method. The samples were termed into names as eight samples 4-textured (T-HR, TR, T-O and T-W) and 4–nontextured (NT-HR, NTR, NT-O and NT-W). Hence, in the same way after copper plating these samples were named as 4-textured coated (T-HR-coated, TR-coated, T-O-coated and T-W-coated) and 4–nontextured (NT-HR-coated, NTR-coated, NT-O-coated and NT-W-coated).

6.1 Microscopy Morphology

Microscopic analysis of all the polyester (textured and non-textured) fabric samples having different cross-sections were observed before and after the coating. The images showed that the all the substrate including textured and non-textured polyester fabric coated uniformly and coating solution penetrated into the pores of the fabric structure. The microscopical images of these analyses are reported in Figure 6.1 and Figure 6.2. By comparing the appearance of coated and uncoated knitted cotton fabric, the surface of the coated fabric was found to be covered by bright copper coating. The results were suggested to be caused by the copper metallic materials used in the plating process. The copper coatings seemed to be very smooth in the observation. No peeled-off path was observed.

Among the textured coated and un-coated samples. The T-HR, T-R, T-O and T-W are the fabric samples without metal deposition, while the T-HR-coated, T-R-coated, T-O-coated and T-W-coated are their corresponding metal deposited samples. Furthermore, among all metal deposited samples the amount of copper is density coated over the textured hollow round coated (T-HR-coated) and textured round coated (T-R-coated) samples. The results can be further reinforced with ICP, and EDX values. Where more amount of metal deposition is found in case of textured hollow round coated (T-HR-coated) and textured round coated (T-R-coated) samples. Furthermore, the SEM analysis can be attributed to the deposition being denser and heavier. The surfaces of metal deposited textured samples are clearer after coating. As under the light microscope they are clearer due to the proper reflection of coating surface. Hence, it reinforces the claim of proper even coating.

Furthermore, among the non-textured coated and un-coated samples as shown in Figure 6.1. The NT-HR, NT-R, NT-O and NT-W are the fabric samples without metal deposition, while the NT-HR-coated, NT-R-coated, NT-O-coated and NT-W-coated are their corresponding metal deposited samples. The overall metal deposition is less as compared to textured samples. It means the texturing of fabric surface has prominent effect for the deposition of more metal. However, among all non-textured samples, the amount of copper is again more over the surface of hollow round coated (NT-HR-coated) and non-textured round coated (NT-R-coated) samples. It means the fibrous cross section have also significant effect against metal deposition. The hollow round fibers may gained more metal deposition due the two reasons. As, (1) plain round structure (provide plain surface to deposit even metal) and second reason is (2) the hollow inner channels may adsorb more copper ions from solution. In fact, the plain surface is more necessary for even metal deposition, because electrical threshold always works with continuous and even network of deposited metals (Ali et al, 2018).

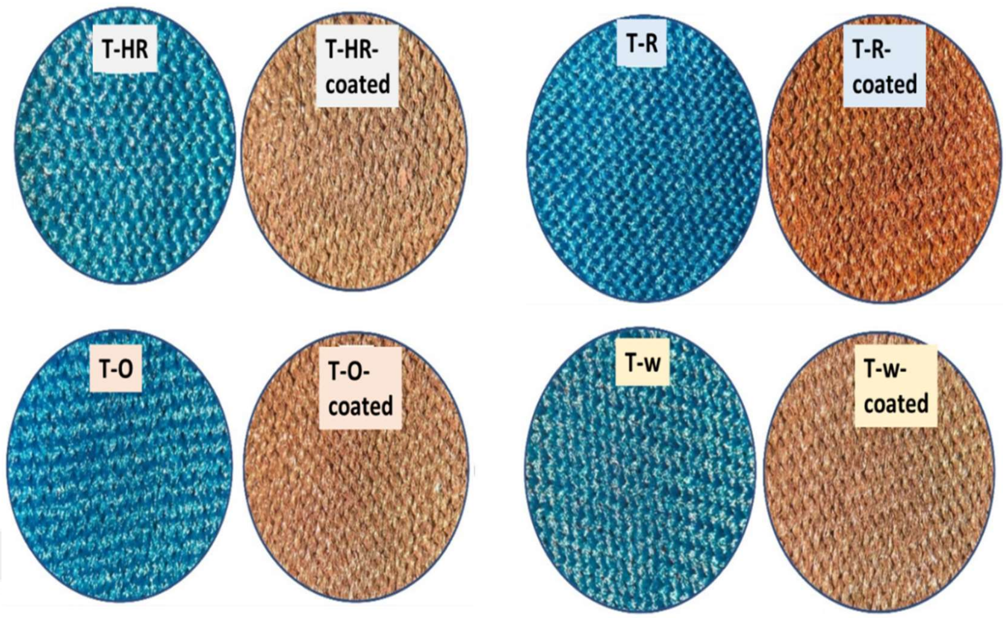


Figure 6.1 : Microscopy morphology of coated and uncoated textured polyester fabrics.

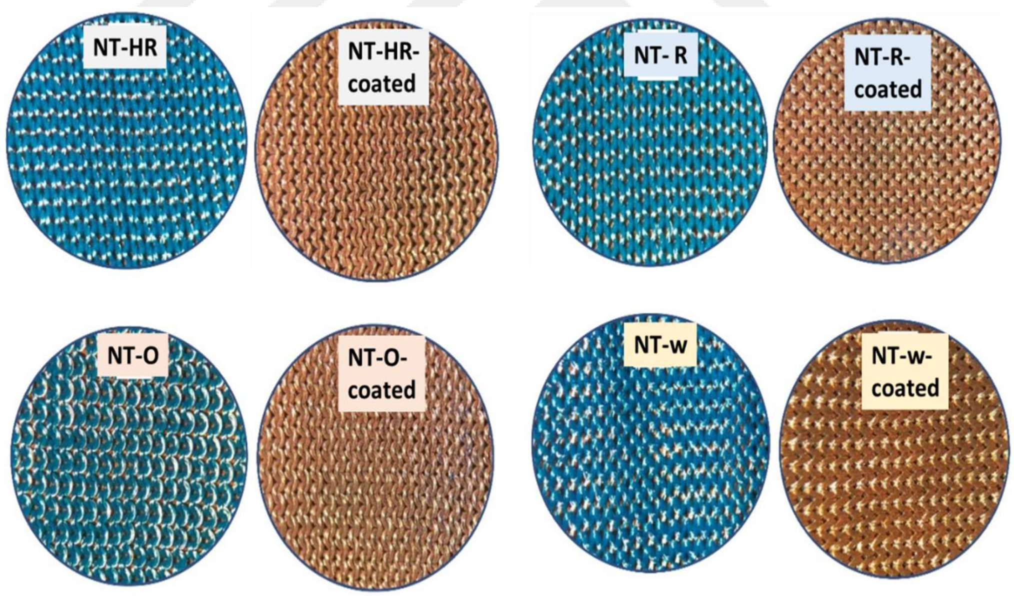


Figure 6.2 : Microscopy morphology of coated and uncoated non-textured polyester fabrics.

6.2 Scanning Electron Microscopy (SEM)

Before and after the metal coating process, the SEM analysis was carried out on all samples of the polyester (textured and non-textured). SEM images in Figure 6.3 and 6.3 shows structures and surface morphology of all the textured and non-textured Polyester fabric samples treated with and without copper metallization. The SEM images demonstrate the deposition of copper nanoparticles on the surface of all the polyester (textured and non-textured) samples. The copper nanoparticle deposition was amalgamated and equally covered to the whole surface of the textured and non-textured polyester fabric substrates.

It was shown from the appearance of all uncoated and coated textured and non-textured polyester fabric substrates that the glossy copper metal coating covered the surface of the conductively coated fabric samples. The study found that all the bright copper coating is due to copper nanoparticle materials used in the electroless plating. The SEM image showed no desquamate path on the fabric substrate. Figure 6.3 shows a close view of the surface of the coated fabric. It was illustrated that a tremendous copper metal coating covers the surface of cotton fabrics in all SEM images. SEM images of all the samples have been obtained at 100x magnification. Furthermore, 100x and 2Kx magnification SEM images of all the samples are reported in Figure 6.3. Their SEM images have observed that plating materials are appropriately deposited all over the fabric surfaces.

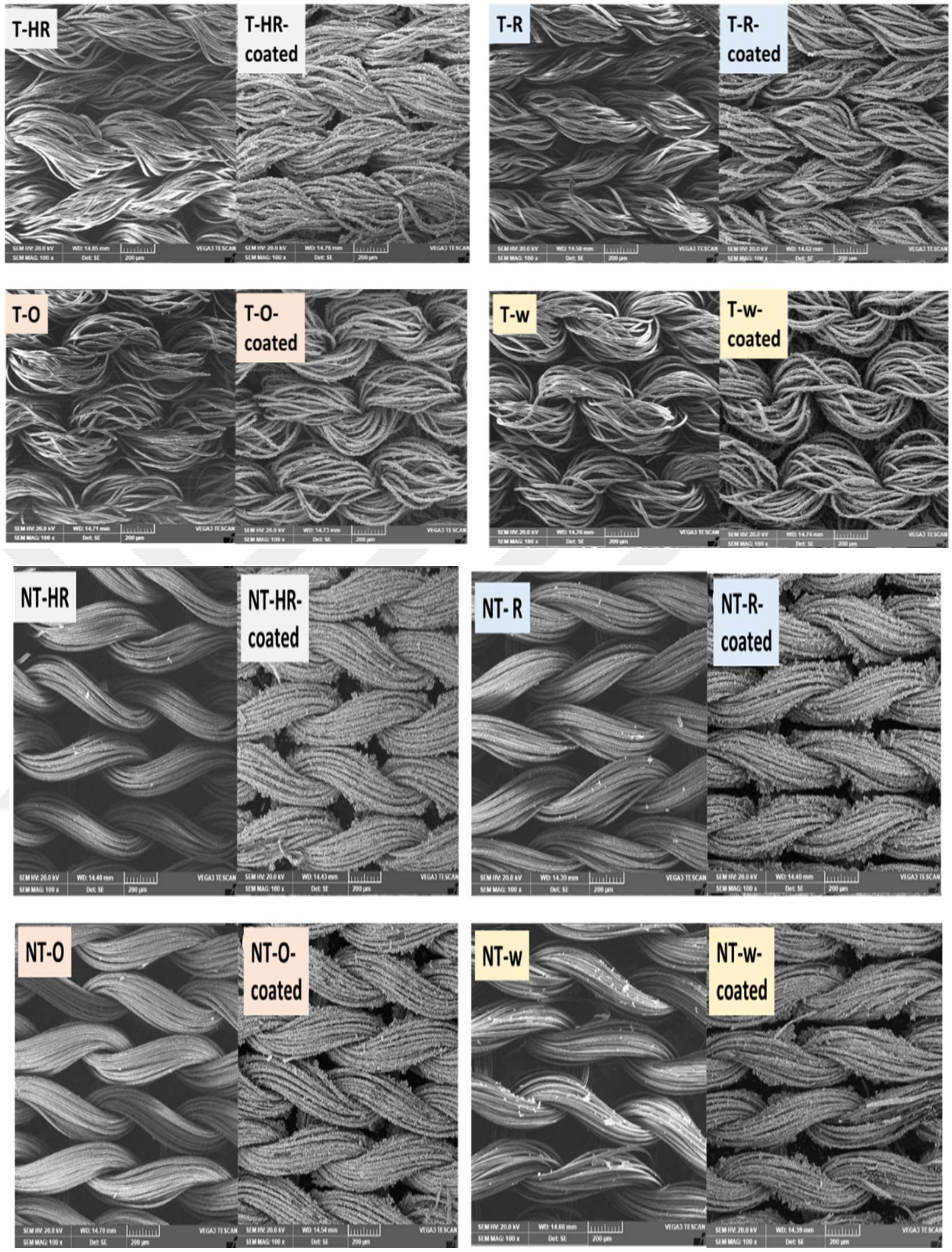


Figure 6.3 : SEM Images of Polyester (Textured and Nontextured) fabric before coating (left side) and after coating (right side) at 100x magnification.

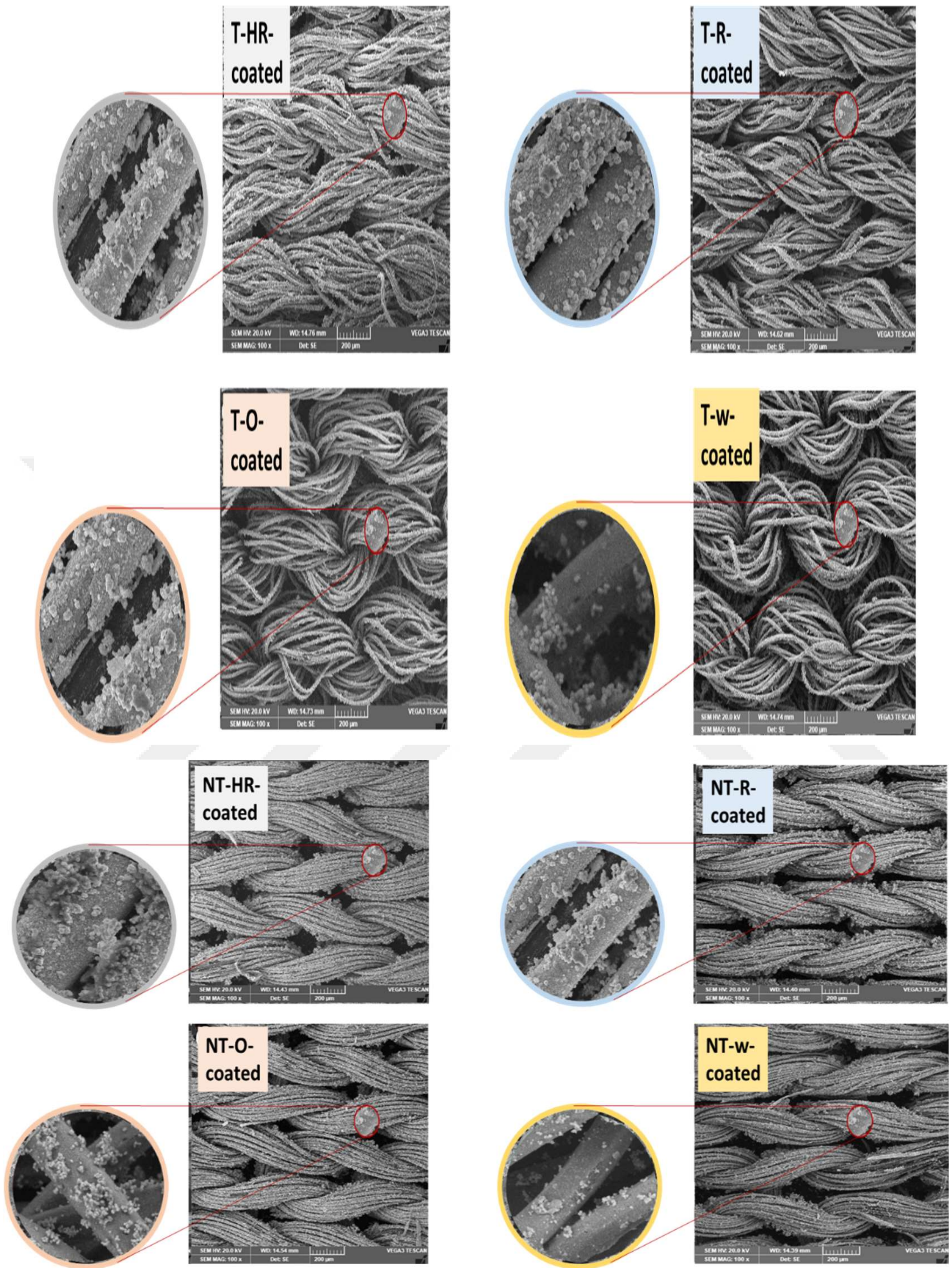


Figure 6.4 : SEM Images of Polyester (Textured and Nontextured) fabric at 100x magnification and their close view at a magnification of 2Kx.

Furthermore, SEM analysis was also carried to study the effect of copper metallization against different morphological structures of fibres. Figure 6.5 is showing the deposition of metal over non-textured uncoated and coated (hollow round, round, octolobal and W shape) samples. Likewise, the deposition of metal over textured uncoated and coated (hollow round, round, octolobal and W shape) samples.

At first, while observing the cross-sectional views of all fibers, it was observed that there is significant amount of metal deposition for each coated sample. The deposition was found most dense for hollow round coated fibers (either textured or non-textured). The reason is that hollow round fibers showed maximum amount of copper solution uptake because of voids or cavities in their longitudinal structure. Secondly, there is also compact coating over the round and octolobal fibers. The fibers are cylindrical in shape hence provide planner structure with compact arrangement. Therefore, even deposition of metal was found. While the W shape fibers showed less amount of metal deposition as compared to all. The reason is that these fibers were having irregular surface as well as cross sectional area.

Furthermore, among the non-textured coated and un-coated samples as shown in Figure 6.5. The NT-HR, NT-R, NT-O and NT-W are the fabric samples without metal deposition, while the NT-HR-coated, NT-R-coated, NT-O-coated and NT-W-coated are their corresponding metal deposited samples. The overall metal deposition is less as compared to textured samples. It means the texturing of fabric surface has prominent effect for the deposition of more metal. However, among all non-textured samples, the amount of copper is again more over the surface of hollow round coated (NT-HR-coated) and non-textured round coated (NT-R-coated) samples. It means the fibrous cross section have also significant effect against metal deposition. The hollow round fibers may gained more metal deposition due the two reasons. As, (1) plain round structure (provide plain surface to deposit even metal) and second reason is (2) the hollow inner channels may adsorb more copper ions from solution. In fact, the plain surface is more necessary for even metal deposition, because electrical threshold always works with continuous and even network of deposited metals (Ali et al, 2018).

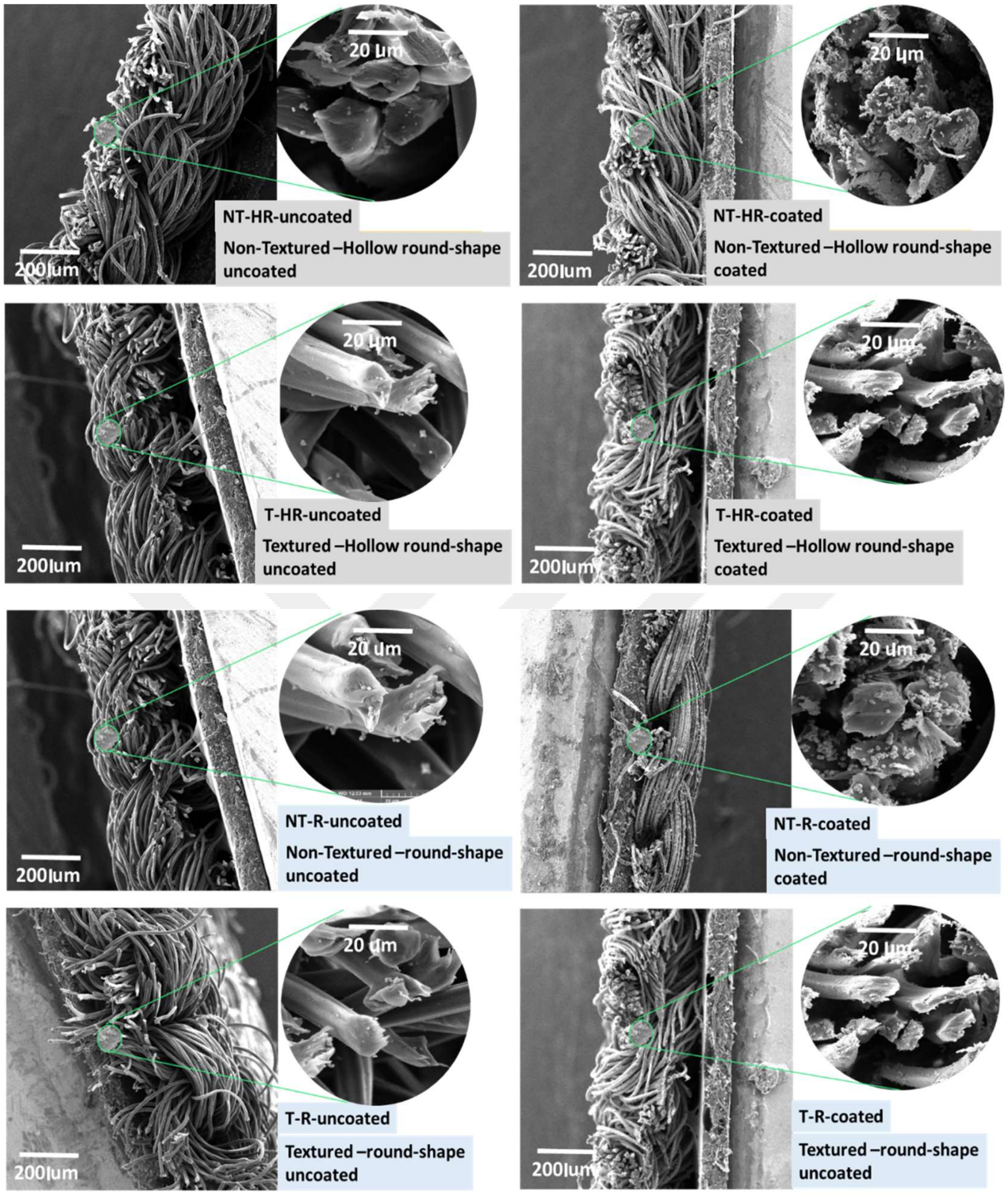


Figure 6.5 : SEM images of all textured and non-textured with different correctional shape.

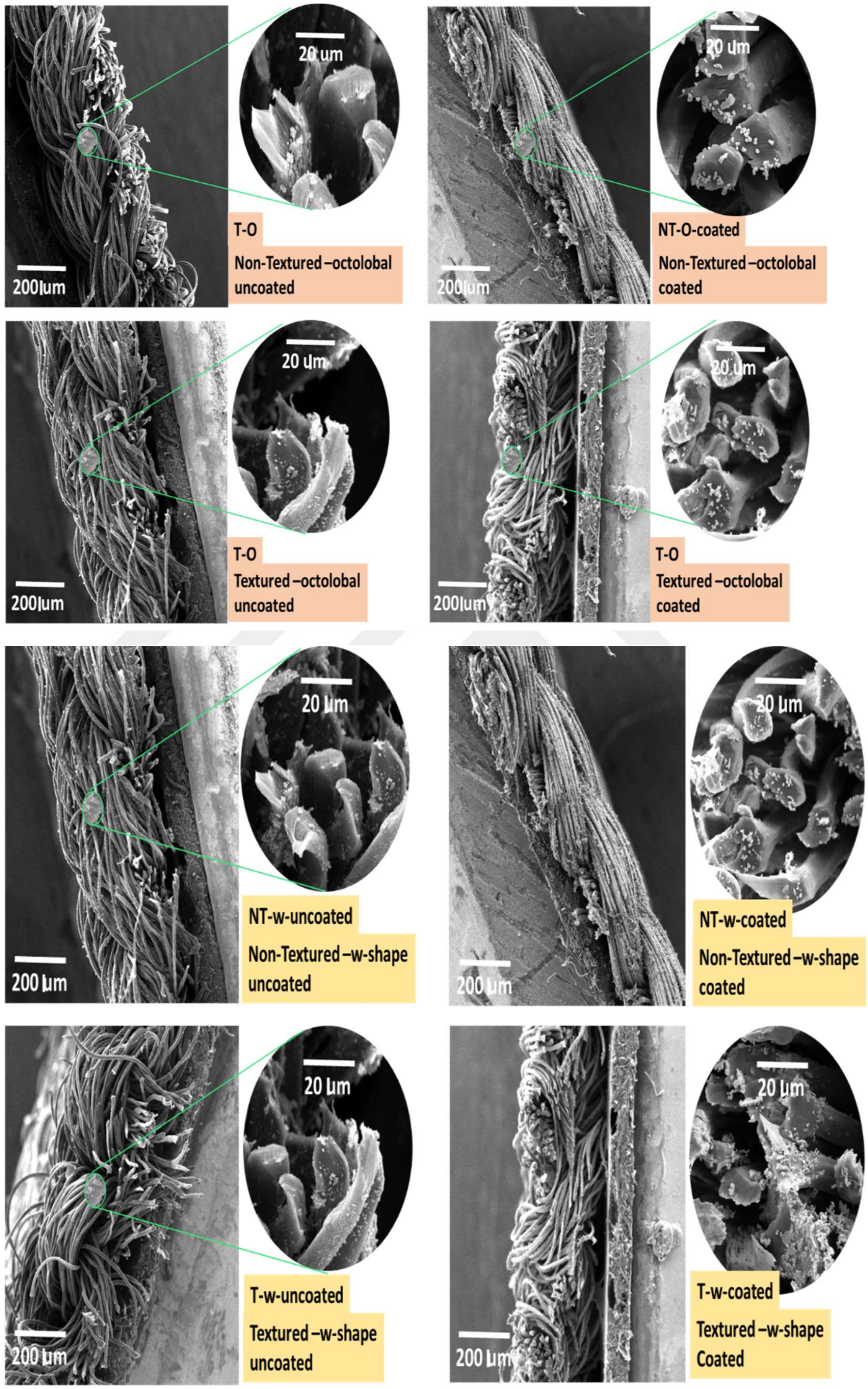


Figure 6.5 (continued) : SEM images of all textured and non-textured with different correctional shape.

6.3 Energy Dispersive X-ray Spectroscopy Analysis

The EDX is used to analyze the coated samples elementally. All polyester (texture and non-texture) fabrics were elementally analyzed in this study.

In the graphs, copper and silver peaks were clearly visible in all the results, as shown in Figure 6.6. After the copper metallization on the surface of the fabric samples, the stiffness of polyester (textured and non-textured) fabric increased was observed in this study. The structure of the sample showed significantly higher rigor in the textured polyester substrates.

EDX observes the surface structures of all metallic copper plated substrate. SEM-EDX spectrum was used to identify chemical composition within the specific area for a certain point on the surface of the samples of the polyester (texted and non-texted) fabrics. The results revealed that the principal compositions deposited in all samples on the surface of copper and silver and also showed that copper metal content reaches up to 22% (Pawar et al, 2009). This ensures the copper nanoparticles are deposited precisely on the substrate's surface. All research results are shown in Table 6.1.

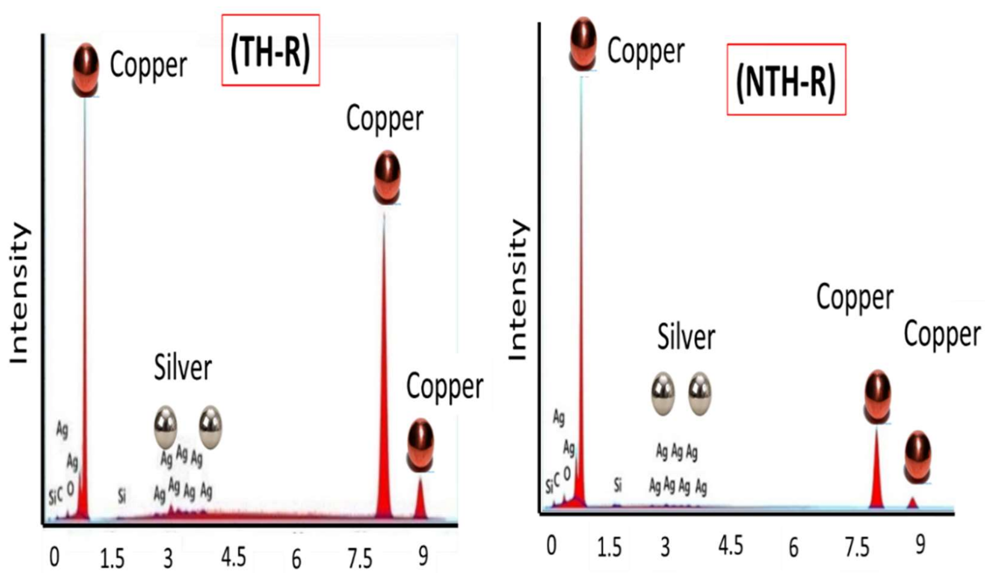


Figure 6.6 : EDX images of all the textured and non-textured Polyester fabric samples.

Table 6.1 : EDX analysis of all textured non-textured coated samples.

Resistivity (Ω.cm)	**Code of sample**	**Wt. % C**	**Wt. % O**	**Wt. % Ag**	**Wt. % Ca**	**Wt. % Cu**
2.16	T-HR-coated	44.01	29.86	2.35	1.97	21.81
3.08	T-R-coated	47.42	28.24	2.38	1.51	20.45
21.65	T-O-coated	49.83	33.63	2.75	1.71	12.08
54.87	T-W-coated	50.69	34.07	3.11	1.05	11.08
76.39	NT-HR-coated	48.97	35.51	3.92	0.95	10.65
82.54	NT-R-coated	51.04	33.5	3.84	1.17	10.45
113.76	NT-O-coated	49.75	34.65	3.61	0.91	11.08
129.62	NT-W-coated	46.84	37.53	3.84	2.61	9.18

6.4 Electrical Resistivity

The concentric electrodes strategy was used to achieve the resistivity of all textured and non-textured polyester textiles. Electrical resistivity of all the polyester (textured and non-textured) fabric samples having different cross-sections were observed before and after the coating. The electrical conductivity results for all fabric samples are shown in the Table 6.2. The results revealed that all fabric samples showed good electrical conductivity (less resistivity) values (2.16 Ω. cm), which are appropriate for all conductive textile applications. The resistivity is according to the flexible electrical electrodes. Where even deposition and lowest resistivity is required. The deposition can be confirmed form microscopic and SEM analysis. Where all the substrate including textured and non-textured polyester fabric coated uniformly and coating solution penetrated into the pores of the fabric structure. Among textured and non-textured comparison there is clear difference electrical resistivity values. All textured samples showed low electrical resistivity values as compared to non-textured samples. It is due the fact that texturing creates the roughness over the surface and results more copper deposition, which in turn results more conductivity (low resistivity).

Furthermore, resistivity analysis was also carried to study the effect of copper metallization and conductivity against different morphological structures of fibres.

At first, while among all the cross sectional fibers, it was observed that there are lower values of electrical resistivity for each coated sample. The resistivity was found lowest for hollow round coated fibers (either textured or non-textured). The reason is that hollow round fibers showed maximum amount of copper solution uptake because of voids or cavities in their longitudinal structure. Secondly, there is lower resistivity for the round and hollow round fibers. The fibers are cylindrical in shape hence provide planner structure with compact arrangement. Hence promote the electrical threshold and more conductivity. The behaviour of more metal deposition and electrical conductivity ca be further justified from the SEM analysis in Figure 6.5.

Figure 6.5 is showing the deposition of metal over non-textured uncoated and coated (hollow round, round, octolobal and W shape) samples. Likewise, the deposition of metal over textured uncoated and coated (hollow round, round, octolobal and W shape) samples. Therefore, even deposition of metal was found. While the W shape fibers showed less amount of metal deposition and higher electrical resistivity values as compared to all. The reason is that these fibers were having irregular surface as well as cross sectional area and low amount of metal deposition.

Table 6.2 : The electrical conductivity results for all fabric samples

No of Samples	Fabric type	Cross-section type	Sample Code before copper coating	Sample Code after copper coating	Resistivity (Ω.cm)	Amount of copper (ppm)
1	Polyester textured	Hollow round shape	T-HR	T-HR-coated	2.16	178,000
2	Polyester textured	Round shape	T-R	T-R-coated	3.08	176,000
3	Polyester textured	Octolobal shape	T-O	T-O-coated	21.65	163,000
4	Polyester textured	W shape	T-W	T-W-coated	54.87	160,000
5	Polyester non-textured	Hollow round shape	NT-HR	NT-HR-coated	76.39	159,000
6	Polyester non-textured	Round shape	NT-R	NT-R-coated	82.54	155,000
7	Polyester non-textured	Octolobal shape	NT-O	NT-O-coated	113.76	134,000
8	Polyester non-textured	W shape	NT-W	NT-W-coated	129.62	130,000

6.5 EMI Shielding

Figure 6.7 shows the results of shielding effectiveness for the fabric samples T-HR-coated and T-R-coated. The samples T-HR-coated and T-R-coated were selected because these samples showed lowest electrical resistivity values and higher amount of metal deposition, which in turn increases the EMI shielding of all metal coated samples. The fabric samples T-HR-coated and T-R-coated showed better EMI SH values.

The sample T-HR-coated showed the least EMI shielding effectiveness of about 73 dB in frequency ranging from 600 MHz to 1500 MHz. The electromagnetic shielding effectiveness of 62 dB was exhibited by samples T-R-coated. The maximum shielding effectiveness in this range was attributed to increase multiple internal reflections of electromagnetic radiations due to higher electrical conductivity. The smallest electrical resistivity i.e., 2.15 cm for sample

CL4 significantly affected the effectiveness of the EMI shielding. Increasing copper concentration (176, 000) in the samples increased their ability to protect. Due to the homogeneous and compact packing of Cu particles on the surface of fabric, this phenomenon was attributed to enhanced electrical conductivity behavior.

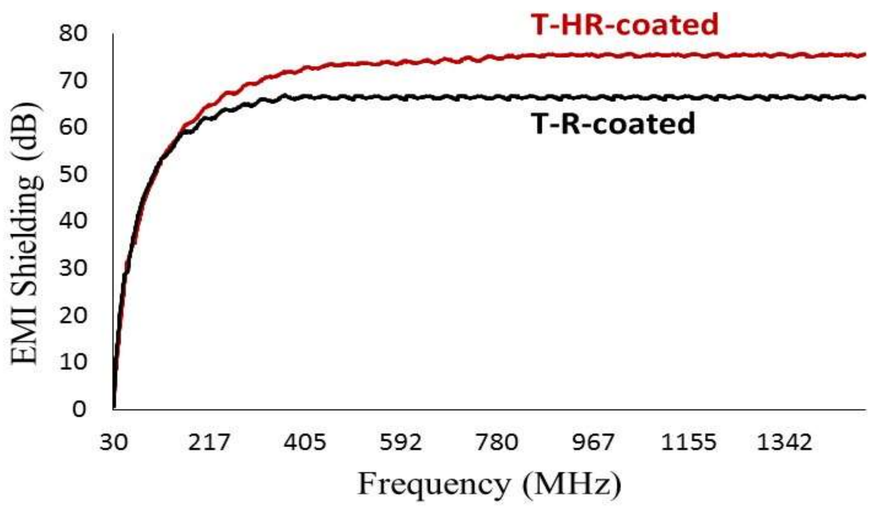


Figure 6.7 : EMI shielding of copper plated polyester fabrics.

6.6 Durability

We performed the washing cycles of the conductive fabric to checked its resistivity and measured the weight. Usually, this method used to investigate the washing durability property of the conductive fabrics. We checked the washing durability up to 5 washing cycles. The results showed that after five washing cycles, the sample T-HR-Coated lost fewer conductivity values as compared to other conductive samples. Furthermore, the comparison of increase in electrical resistivity with decrease in weight of all coated samples is also shown in Figure 6.8. It can be seen that there is increase in electrical resistivity values and decrease in weight of all coated samples. The results also showed that after five washing cycles.

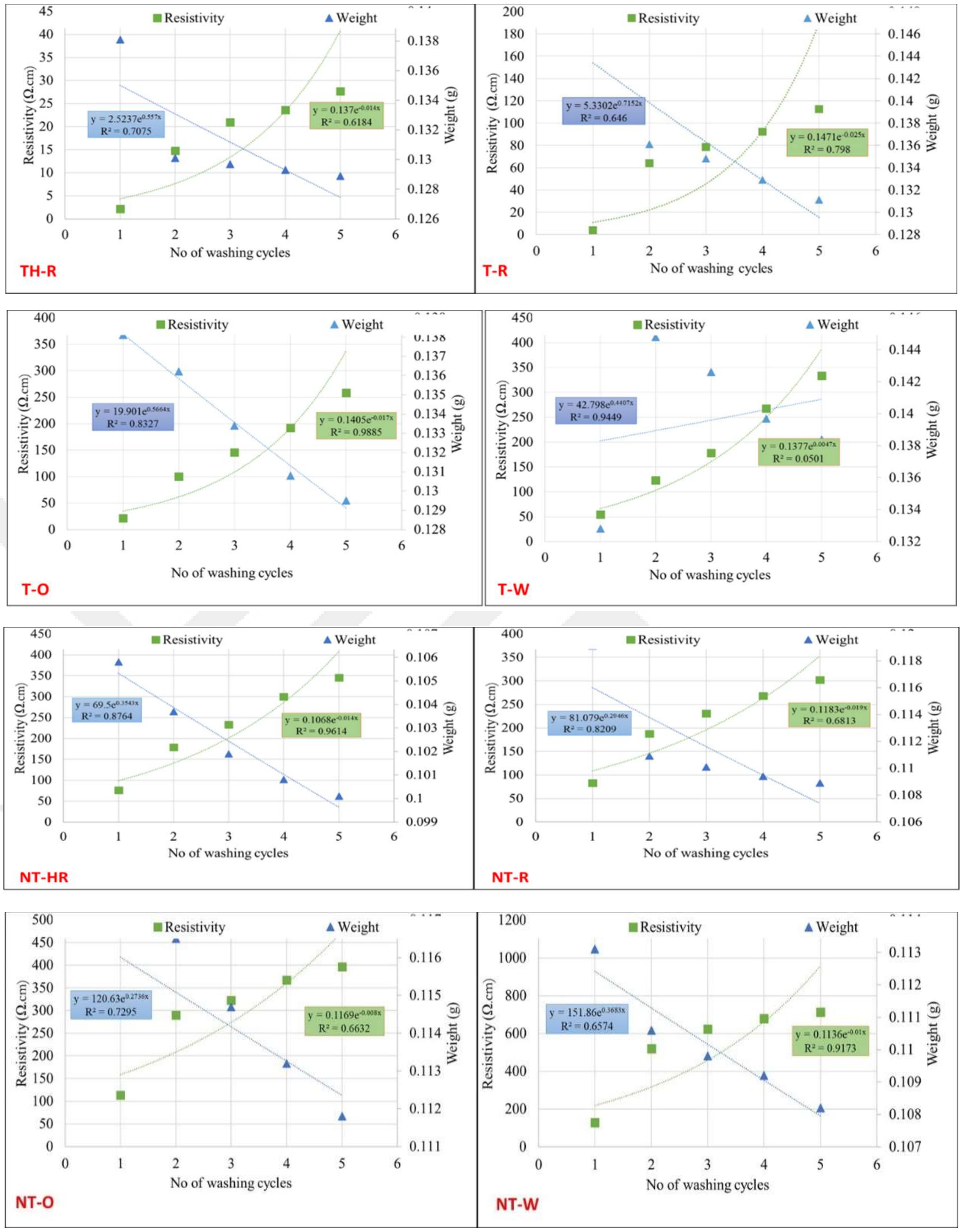


Figure 6.8 : Increase in electrical resistivity with decrease in weight of all coated samples.

6.7 Durability against Abrasion

The test is carried out to investigate the ability of the fabric to withstand against abrasion. Abrasion resistance of all the laser-treated samples up to 250 abrasion cycles was observed in this study. The results revealed that the samples which have more deposition of metal showed high abrasion resistance. The abrasion resistance depends on the fabric structure of the fabric samples and metal coating types. After the Cu-coating process, copper metals firstly accommodate on the outer layer and then inner layers of cross-sectional yarns. Usually, the outer layer of the fabric sample is bigger as compared to inner layer due to coating process. However, when we checked the abrasion resistance of the fabric samples, then the weight of the fabric decreases because the outer layer of the metal-coated is decreasing and hence conductivity decreases. The results showed that the samples, which were having more amount of metal deposition have been conductive (even after sever abrasion) as compared to the fabric non-textured samples (Ahmad et al, 2020).

All the abrasion resistance cycle results with respect to weight and resistivity are shown in Figure 6.9 and Figure 6.10. Figure 6.9 shows the abrasion cycle graph w.r.t weight of the fabric samples. Sample T-HR-Coated were shown to have lost 10% of weight, while Sample T-R-Coated had lost 20% of the weight. Figure 6.10 shows the graph of the abrasion cycles w.r.t electrical resistivity of the laser samples.

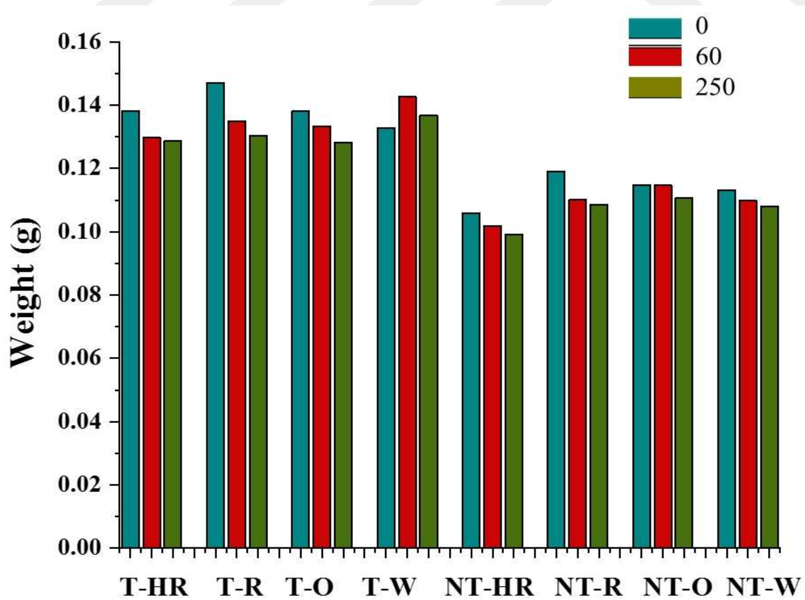


Figure 6.9 : Effect of abrasion cycles against weight.

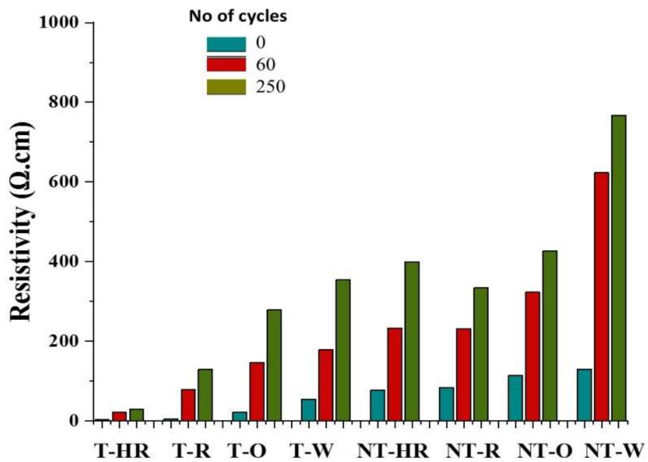


Figure 6.10 : Effect of abrasion cycles against electrical resistivity for all textured and non-textured samples.

6.8 Mechanism of Copper Electroless Plating over Polyester Fabrics

The deposition of copper metal was performed on the fabric sample in the electroless copper plating technique, and we employed formaldehyde as a reducing agent inside the plating solution. The reactions can be seen below.

Metal ion (M^+) + Reducing agent (Red) → Metal Deposited + Oxidized product (Ox)

There are two reactions in the electroless plating that are given below:

Metal deposition: Metal ion (M^+) + electron → M (metal) (6.1)

Oxidation: Reducing agent (Red) + Water → Oxidized product + H^+ + electron (6.2)

The electroless procedure, as shown in Figure 6.11, catalyses the reaction by using an appropriate reducing agent (formaldehyde (HCHO)) to supply the electron for the reduction reactions, and copper metal is deposited on the surface. For 20 minutes, we kept the plating bath at 30 ° C.

In first step, non-conductive substrates were immersed in a beaker consisting of a mixture of 5ml/l hydrogen chloride (HCl) and 5g/l stannous chloride (SnCl2) for 10 minutes at 25 °C, with pH maintained 1 throughout. In second step, Sn+2 ions are deposited on non-conductive substrates out of the stannous ion solution. After sensitization, the second phase was activation. The primary goal of the activation procedure is to prepare the substrate surface for plating. The necessary target for deposition of copper metal on the fabric surface cannot be attained without activation. For 20 min a t 25 °C, a mixture of 10ml/l ammonium hydroxide (NH4OH) and 10g/l silver nitrate ($AgNO_3$) was utilized in the activation step. The plating process was the next and most crucial phase. The plating bath, which contains a mixture of 45g/l Rochelle salt,10g/l copper sulphate, 10g/l caustic soda (NaOH), and 20ml/l methanol (CH_2O) solution is an important stage in the electroless plating technique. For the nonconductive substrate, the plating solution was employed for 20 min at 30 °C while maintaining a pH of 10. The rochelle salt has been used as a complexing agent in the plating bath. The primary purpose of complexing agents is to reduce copper formation to cupric hydroxide ($Cu(OH)_2$) and methanal, which are utilised as reducing agents throughout the copper metallization process. The reducing agent is labelled "Red," the oxidised product (Ox), and the metal ion (M+) in. Figure 6.11 shows how the reaction is catalysed by the application of a reducing agent (methanal in this case) that supplies the electrons for a reduction reaction and assists the deposition of copper metal on the laser-treated fabrics. During the plating phase, the temperature of the plating bath was maintained at 30°C for 20 minutes.

The completion of sensitization, activation and plating steps was followed by the washing and drying steps. During the washing phase, all conductive surfaces were washed thoroughly with distilled water for at 40 ° C for 20 min. All copper-plated fabric samples were then dried at 80°C in a heating oven for 20 min and the samples were spread for 24 h until totally dry, after which the characterisation of all prepared fabric samples was evaluated [36].

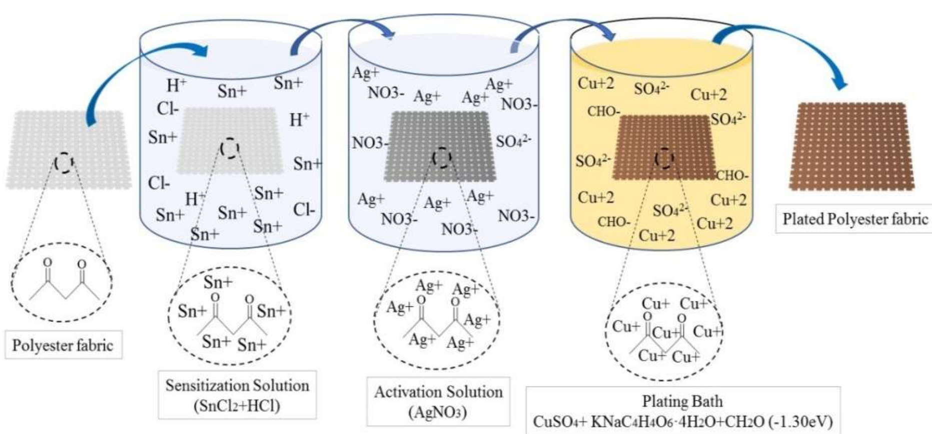


Figure 6.11 : Mechanism of copper electroless plating over polyester fabrics.

7. COMPARISON OF ALL EXISTING STUDIES AND WITH LITERATURE

7.1 Comparison Within the Study

In this chapter we described the comparative analysis regarding electrical properties of all previous work done.

First study was composed of the Deposition of copper by electroless plating over cotton knitted fabric (9 different GSM). Hence, nine different GSM were chosen and copper plating was performed. The resistivity values of sample were composed up with lower GSM (136) were very less as compared to other samples. Furthermore, EDX analysis is also supporting the fact. Secondly, the effect of low electrical resistivity (3.24 Ω. cm) with low GSM and more copper deposition (160,000 ppm) was also confirmed. While the said sample showed maximum EMI SH about 50 dB.

In second study, we selected the most suitable (lower GSM, and low electrical resistivity samples). We selected three fabric samples (S4, S5 and S6) having light GSM (136, 154 and 176 GSM) out of nine fabric samples as mentioned in previous study 1. These fabric samples provided the lowest value of electrical resistivity coupled with high EMI shielding and more contents of metal particles. In this study we pretreated the samples with laser to enhance the surface roughness, then electroless plating was applied. In fact, we want to see the impact of roughness against copper deposition. Hence, the fabric sample with GSM (136) again provided good electrical conductivity (lower resistivity) as compared to all other samples. In this time the sample GSM (136) provided the electrical resistivity (2.54 Ω. cm) lower than study 1. Because here it was pretreated with laser then copper deposition was performed.

The sample showed the EMI SH about 62 dB couples with 172,000 ppm of copper deposition.

The 3RD study was performed by using the same electroless plating recipe on polyester fabrics. The polyester fibers (textured and non-textured) were selected for this purpose. Four different polyester fabric composed up of different fibrous cross sections (hollow, W shape, octalobel, and hollow-round) were selected. Each fabric was further subjected a texturing process. The False twist texturimg was done on four polyester fabrics (hollow, W shape, octalobel, and hollow-round) before use. Hence, we achieved total eight samples 4-textured (hollow round, round, octolobal and W shape) and 4–nontextured (hollow round, round, octolobal and W shape). Microscopic analysis of all the polyester (textured and non-textured) fabric samples showed that the surface of the coated fabric was found to be covered by bright copper coating. Furthermore, among all metal deposited samples the amount of copper is densily coated over the textured hollow round coated (T-HR-coated) and textured round coated (T-R-coated) samples. The results were further reinforced with ICP, and EDX values. Where more amount of metal deposition (178,000 ppm) was found in case of textured hollow round coated (T-HR-coated) and textured round coated (T-R-coated) samples. It means the fibrous correction have also significant effect against metal deposition. The hollow round fibers showed the lowest electrical resistivity values about 2 (Ω.cm) coupled with high EMI shielding 75 dB. The comparison regarding the electrical resistivity, EMI shielding and amount of metal deposition the resuls are shown in Table 7.1.

Table 7.1 : The comparison regarding the electrical resistivity, EMI shielding and amount of metal deposition.

No of Samples	Fabric type	Cross-section type	Fabric GSM (g/m^2)	Sample Code after copper coating	Electrical Resistivity (Ω.cm)	Amount of copper (ppm)
1	Cotton knitted	solid	136	C4	3.24	160,000
2	Cotton knitted	solid	154	C5	5.34	158,000
3	Cotton knitted	solid	170	C6	8.90	153,000
4	Cotton knitted	solid	136	LC4	2.456	172,000
5	Cotton knitted	solid	154	LC5	4.738	159,000
6	Cotton knitted	solid	170	LC6	6.628	156.000
7	Polyester textured	Hollow round shape	136	T-HR-coated	2.16	178,000
8	Polyester textured	Round shape	136	T-R-coated	3.08	176,000
9	Polyester textured	Octolobal shape	136	T-O-coated	21.65	163,000
10	Polyester textured	W shape	T-W	T-W-coated	54.87	160,000
11	Polyester non-textured	Hollow round shape	NT-HR	NT-HR-coated	76.39	159,000
12	Polyester non-textured	Round shape	NT-R	NT-R-coated	82.54	155,000
13	Polyester non-textured	Octolobal shape	NT-O	NT-O-coated	113.76	134,000
14	Polyester non-textured	W shape	NT-W	NT-W-coated	129.62	130,000

Comparison with previous researches

The most key characteristics of metal coated fabrics are electromagnetic shielding and electrical conductivity. For metallization of textiles, researchers have used a variety of approaches and methodologies. There are numerous flaws in the methodologies they use, and the EMI shielding and electrical conductivity findings obtained are not particularly impressive. Electroless plating is used by Bharath et al to develop electrically conductive, EMI shielded textiles. He conducted all of the pretreatment procedures (pretreatment, sensitization, and activation) as well as a variety of coatings (Cu-Ni-Fe Alloy). In terms of electrical conductivity and EMI shielding, the results are relatively low. Under 75 dB of EMI SH [56], they achieved 465/square electrical resistivity.

There are quite similar studies available in literature; where coating of metal was done on cotton fabrics. Electromagnetic interference shielding effectiveness of copper plated fabrics (Chung, 2000) was examined after electroless plating of copper over cotton fabric. The fabricated substrate showed 5Ω/square having 12 dB of EM shielding.

Ali et al. tried to coat the cotton fabric by the deposition of copper nanoparticlrs by dip-reduction method. It was the particle coating by insitu deposition, where the remain to achive the 156 Ω/cm of elecgrical resistivity with maximum EMI shielding of 18 dB (Ali, Baheti, Militky, Khan, et al., 2018). The aslo tried to coat the copper/silver deposition on one structure of cotton textile by combine effect. The resistivity was lesser and EMI shielding was little improved (131, 22 dB). Copper Core Conductive Fabrics for smart Textiles applications was also performed by electroless deposition method (Ramachandran & Vigneswaran, 2009). They showed maximum 13 dB. Which is closer to our most poor develop sample (NT-W-coated) 129 Ω/cm of elecgrical resistivity with maximum EMI shielding of 24 dB.

Metallization of Polyester Fabric by Autocatalytic Copper Plating Process Using Glyoxylic Acid as a Reducing Agent (Qin & Guo, 2015) was done on textile structure. The achieved resistivity was almost 128 mΩ/square, with 50 dB. Also their results were close to commercialize develop sample RF Shielding Fabric Cotton (PW, n.d.), with 1Ω/Square with 55 dB. This work is much similar in results with our develop samples C4, LC4 and LC5.

The only studies having extraordinary results regarding electrical conductivity and higher EMI shielding are very less in literature. Preparation and Properties of Copper-Silver Complex Plating on PET Fabrics (D. Yu et al., 2015) was done. The combination of copper and silver with dense coating remain quite feasible to improve the electrical conductivity and EMI shielding. Furthermore, the applied the dual process of plating electroless/electroplating and achieved 60 mΩ/square with 70 dB EMI shielding. Their results are nearer to commercial sample Shieldex® Kassel Nylon (Https://Www.Vtechtextiles.Com/Wp-Content/Uploads/2018/08/1300101130-Kassel.Pdf, n.d.) showing 80 dB of EMI shielding. The provide results are also match our optimised well prepared samples Polyester textured Hollow round shape (electroless, 2.16 Ω/cm, 73dB), Polyester textured Round shape (electroless, 3.08 Ω/cm, 62dB).

Advantages of present work

The current research activity has multiple benefits considering extensive comparison with other studies. Previously electro less plating has been performing by different salts and reducing agents' combinations over different fibres. However, there was huge gap in literature to study the effect of electro lees plating on different surfaces and different GSM. Secondly, we have also studied the effect of different fibrous cross-section (round, hollow round, W shape and octolobel) against copper electro less plating. Furthermore, the coating of metals on same substrate but having different surface pre-treatments was also studied. Copper electroless plating fabrics is showing very high EMI shielding about 73 dB. Metallization has been done on pure synthetic fibres or hybrid fabrics by the researchers. Furthermore, our fabrics are completely copper-coated. The obtained textiles can potentially be used in medical applications for hygiene aspects. EMI shielded cotton with RF shielding (blended cotton, copper, and silver) are commercially available. (Afzali et al, 2010) Instead of using flexible metal coatings, these fabrics are produced by blending thin silver and copper wires thin with cotton fibres at yarn manufacture and weaving stage. Furthermore, when compared to our produced copper electroless plated textiles, the obtained EMI shielding (55dB) is lower (66-73dB). Shieldex produces higher EMI protected (80dB) textiles under kassel trade name (80dB comparable to present results of thesis of 66-75dB) (Vaškelis et al, 2007). Copper was applied to silver-plated nylon fibres to develop these materials. Current research has generated conductive textiles with low electrical resistivity, exceptional EMI shielding, and other functioning features. With thin and consistent metal deposition, an interconnected network of conductive nanoparticles was achieved. Electrical conductivity, EMI shielding, and durability are the main advantages of developed electrodes. Electrodes for in vivo applications (where conductive materials are connected to human skin) and electrotherapy as well as EMG and ECG electrodes, all demand these characteristics.

8. CONCLUSIONS AND RECOMMENDATIONS

At first, we selected nine-combed cotton knitted fabric samples with different yarn fineness and elastane percentage in order to see effect of these parameters on conductive and physical properties of samples such as an increase in weight and thickness, impact of washing cycles and abrasion resistance on the electrical conductivity of the fabric sample. Knitted combed cotton fabrics with 5% elastane and the fine count showed the excellent conductivity as compared to the other knitted cotton fabric with 10% elastane with a fine count or 5% elastane with coarser. The copper metal was successfully deposited on the surface of the knitted fabrics through the copper metal plating method. We performed all the necessary tests to check the characterization of all the samples. Samples C4, C5 and C6 which have a finer count and 5% elastane percentage showed excellent abrasion resistance compared to other fabric types and C7, C8 and C9 also showed good conductivity values as compared to samples C1, C2 and C3. Hence, this study proved that less the elastane percentage in the fabric structure, more the conductivity values and also depends on the structure of the fabric. The denser fabric structure is less conductive, and the finer fabric structure has shown excellent conductivity values. The possible reason for this result is due to the structural properties of knitted cotton fabric, which is thick and dense and the elastane percentage, thus plating solution cannot penetrate deep inside the structure of the knitted fabric surface that resulted in weak conductive areas within the structure. The electromagnetic shielding effectiveness was also found to more with less GSM fabric samples (C4, C5 and C6). The sample C6 (170 GSM) showed the lowest electromagnetic shielding effectiveness of about 35 dB in frequency range of 600 MHz–1500 MHz. The electromagnetic shielding effectiveness of 40 dB and 50 dB was exhibited by samples (C5 and C4) respectively. The maximum shielding effectiveness in this range was attributed to increase multiple internal reflections of electromagnetic radiations due to higher electrical conductivity. The lowest electrical resistivity (3.2 Ωcm) of sample C4 showed significant effect on shielding effectiveness. The samples showed increase in shielding effectiveness with more copper contents (160, 000 ppm). Thus, we have proceeded with knitted cotton fabric with 5% elastane and fine count as a suitable candidate for different E-Textile Applications like pressure sensors etc.

The part 2nd, was focussed, to carry out research on the best three samples obtained from study one. The selected fabric samples with GSM (136, 154 and 176 GSM) out of nine fabric samples (as mentioned in previous study 1) were used in this part. The selected samples were **pre-treated** with laser to enhance the surface roughness, then electroless plating was applied. The physical properties of the laser-treated knitted samples were examined in terms of thickness, durability, and abrasion impact. Furthermore, the surface topography of the laser-treated substrates was analyzed using a scanning electron microscope (SEM) before and after the coating process and samples demonstrated remarkably uniform deposition of copper metal nanoparticles on the surface of the fabric. Several types of experiments have been carried out to characterize samples of laser-treated knitted cotton fabric. Among them, the CL4 sample showed lowest resistivity (2.45 Ω.cm) coupled with high metal contents (172,000 ppm). While the associated EMI SH was about 65dB. Accordingly, this study confirmed that the lower the GSM values, the higher the conductivity values. These results also revealed that the structure of the fabric also affects the conductivity of the laser-treated substrate. The thicker fabric structure is less conductive, and the finer fabric structure has excellent electrical conductivity values. The laser-treated fabric samples showed good conductivity and higher wear resistance compared to non-laser-treated fabric samples. Rubbing fastness is very important for electronic textile applications. The fabrics showed good retention of the copper over the surface of fabrics even after sever, rubbing and washing as proven by electrical resistivity and weight lost.

The focus of 3rd study was on developing and characterizing conductive textured and non-textured polyester fabrics. We performed a list of tests to explored the effect of conductivity on textured and non-textured polyester fabrics and their cross-sections. Among them, the textured polyester fabric samples having hollow round and round shape cross-sections showed excellent conductivity while in the case of the non-textured polyester fabric samples, the samples which had hollow round and round cross-section types, showed good conductivity values. Furthermore, among all metal deposited samples the amount of copper is densily coated over the textured hollow round coated (T-HR-coated) and textured round coated (T-R-coated) samples. The results were further reinforced with ICP (178,000), and EDX values. Where more amount of metal deposition is found in case of textured hollow round coated (T-HR-coated) and textured round coated (T-R-coated) samples. Moreover, the overall metal deposition on non-textured samples was less as compared to textured samples. It means the texturing of fabric surface has prominent effect for the deposition of more metal. However, among all non-textured samples, the amount of copper is again more over the surface of hollow round coated (NT-HR-coated) and non-textured round coated (NT-R-coated) samples. It means the fibrous cross section have also significant effect against metal deposition. The hollow round fibers may gained more metal deposition due the two reasons. As, (1) plain round structure (provide plain surface to deposit even metal) and second reason is (2) the hollow inner channels may adsorb more copper ions from solution. In fact, the plain surface is more necessary for even metal deposition, because electrical threshold always works with continuous and even network of deposited metals (Ali et al, 2018).

Electrical resistivity of all the polyester (textured and non-textured) fabric samples having different cross-sections were observed before and after the coating. The results revealed that all fabric samples showed good electrical conductivity (less resistivity) values (2.16 Ω. cm), which are appropriate for all conductive textile applications. The resistivity was according to the flexible electrical electrodes. Where even deposition and lowest resistivity is required. Among textured and non-textured comparison there is clear difference electrical resistivity values. It is due the fact that texturing creates the roughness over the surface and results more copper deposition, which in turn results more conductivity (low resistivity). Moreover, among all the cross sectional fibers, the resistivity is lowest for hollow round coated fibers (either textured or non-textured). The reason is that hollow round fibers showed maximum amount of copper solution uptake because of voids or cavities in their longitudinal structure. Secondly, there is lower resistivity for the round and hollow round fibers. The fibers are cylindrical in shape hence provide planner structure with compact arrangement. Hence promote the electrical threshold and more conductivity. While the W shape fibers showed less amount of metal deposition and higher electrical resistivity values as compared to all. The reason is that these fibers were having irregular surface as well as cross sectional area and low amount of metal deposition. However, the textured polyester fabric samples showed overall good behaviour towards abrasion resistance, washing ability, and electrical conductivity values as compared to non-textured polyester fabric samples, which can be useful for electronic textile applications. Samples showed strong retentiveness of copper nanoparticles, demonstrated via the SEM microstructure, and a slight loss of electrical conductivity following washing. Thus, the scientific work that was presented described how the surfaces of various textiles were metallized. We developed conductive fabrics with exceptional electrical conductivity, EMI shielding, and antibacterial capabilities that are less rigid and flexible. Additionally, the electroless plating improved the electrical, washing, and rubbing qualities. The promising applications of developed textiles are as electrodes for medical devices, EMI shielding and strain sensors.

FUTURE WORK

- Different types of reducing agents and salts will be studied for metalization
- The simulation of conductivity and heat transfer mechanism will be studied on different structures of fabrics
- Examination of electrical parameters under various ageing environments (humidity, temperature etc).
- Tie-dyeing with the combination of electroless plating affects and their design effects of metallized fabric will be studied.
- Various combinations of metal coatings to enhance the electrical performances (electroplating, in situ deposition, and electroless plating)
- Metallization of aramid, wool, and glass fibres for various functional features.
- Copper oxidation prevention and increased performance durability (by applying binders, capping agents, semiconductors, and alloys)
- Effect of metal coating thickness on hardness, EMI shielding efficacy, hardness, and towards anti-stabbing action.

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