

ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL

**DEVELOPMENT OF TEXTILE BASED TEMPERATURE SENSOR FOR
WEARABLE ELECTRONICS**



Ph.D. THESIS

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Department of Textile Engineering

Textile Engineering Programme

SEPTEMBER 2021

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SEPTEMBER 2021

İSTANBUL TEKNİK ÜNİVERSİTESİ ★ LİSANSÜSTÜ EĞİTİM ENSTİTÜSÜ

**GİYİLEBİLİR ELEKTRONİKLER İÇİN TEKSTİL TABANLI SICAKLIK
SENSÖRÜ GELİŞTİRİLMESİ**

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EYLÜL 2021

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Date of Submission : 04/09/2021

Date of Defense : 30/09/2021





To my grandmother Zeliş,



FOREWORD

I would like to express my deepest gratitude to my research advisor, Assoc. Prof. Dr. Senem KURŞUN BAHADIR, for the opportunity to work on her research project and for her valuable advices, supervision, dynamic guidance, and continuous support throughout my PhD. She was incredibly encouraging, positive, motivating, and guiding as she taught me a variety of research techniques, from idea generation to data analysis and writing, all of which are essential to become a scientific researcher.

I would like to express my special thanks to my thesis committee members, Assoc. Prof. Dr. Umut Kıvanç ŞAHİN and Prof. Dr. Güralp ÖZKOÇ, for their time and support; their valuable comments aided me in improving the quality of my work throughout my thesis study.

I would like to thank Assoc. Prof. Dr. Ersin SAYAR for his support and encouraging me. His scientific contribution is very valuable to my PhD research.

I would like to thank to Prof. Dr. Vladan KONCAR at ENSAIT for accepting me to laboratory during my research period in France, for his guidance and support.

I also want to give my sincere thanks to my best friends and lab-mates; Beyza BOZALI, Esma Nur NEBİPAŞAGİL, and Hatice AÇIKGÖZ TUFAN. Their friendship is one of the most valuable things in my life. In difficult times, we always found a way to create a positive moment. I also want to thank my lab-mates Meltem TEKÇİN and Pelin ALTAY for their helpful presence.

Eventually, I would like to express my gratitude and endless love to my parents; Meryem, Serdar and Arda ARMAN and husband Muzaffer KUZUBAŞOĞLU. I am grateful for their continuous encouragement, support and motivation over the years.

September 2021

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ABBREVIATIONS

AFM	: Atomic Force Microscope
ANN	: Artificial Neural Network
BR	: Bayesian regularization
CIE	: Commission Internationale de l'Éclairage; International Commission on Illumination
CIJ	: Continuous inkjet
CNT	: Carbon Nanotube
CVD	: Chemical Vapor Deposition
COOH	: Carboxylic Acid
DOD	: Drop-on-demand
EG	: Ethylene Glycol
E-Textile	: Electronic Textile
FG	: Functionalized Graphene
FGO	: Functionalized Graphene Oxide
GNP	: Graphene Nanoplatelets
GNW	: Graphene Nanowalls
FTIR	: Fourier Transform Infrared Spectroscopy
LM	: Levenberg–Marquardt algorithm
MLP	: Multilayer Perceptron
MSE	: Mean-square Error
MWCNT	: Multi-walled Carbon Nanotube
NTC	: Negative Temperature Coefficient
Oh	: Ohnesorge
PANI	: Polyaniline
PDMS	: Polydimethylsiloxane
PEDOT:PSS	: Poly(3,4-ethylenedioxythiophene) polystyrene sulfonate
PEN	: Polyethylene naphthalate
PES	: Polyether sulfone
PET	: Polyethylene terephthalate
PI	: Polyimide
PTC	: Positive Temperature Coefficient

PTFE	: Polytetrafluoroethylene
PU	: Polyurethane
PVDF	: Polyvinylidene fluoride
R	: Correlation Coefficient
Re	: Reynolds
rGO	: Reduced Graphene Oxide
RH	: Relative Humidity
RTD	: Resistance temperature detectors
TCR	: Temperature Coefficient of Resistance
SEBS	: Styrene Ethylbutylene Styrene
SEM	: Scanning Electron Microscope
Sa	: Arithmetic-mean Height
SDBS	: Sodium Dodecylbenzenesulfonate
SDS	: Sodium dodecyl sulfate
SLE	: Special Limits of Error
Sq	: Root-mean-square Height
SWCNT	: Single-walled Carbon Nanotube
Triton X-100	: 2-[4-(2,4,4-trimethylpentan-2-yl)phenoxy]ethanol
Tween 20 /80	: Polyoxyethylene-20(or 80)-sorbitan monolaurate
UV-Vis	: Ultraviolet - visible
We	: Weber
Z	: Dimensionless Number

SYMBOLS

A, B, C	: Steinhart coefficients
ΔR	: Resistance change
ΔT	: Temperature change
DL	: Difference in lightness/darkness value
DE	: Total colour difference value
S_{sens}	: Sensitivity value
θ	: Contact angle
ρ	: Density
η	: Viscosity
σ	: Surface tension
L	: Nozzle diameter
V	: Droplet velocity
β	: Thermal index (temperature constant)
α	: Difference in the Seebeck coefficient of thermoelements
T	: Transmittance
wt	: Weight
n	: Number of observations
y(k)	: Experimental data
t(k)	: Predicted data



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DEVELOPMENT OF TEXTILE-BASED TEMPERATURE SENSOR FOR WEARABLE ELECTRONICS

SUMMARY

Due to their compressibility, bendability, and compatibility with irregular and curvilinear surfaces, flexible and stretchable devices are attracting attention and have a wide range of applications. The increasing number of publications in this field demonstrates the growing popularity of flexible sensors. Flexible sensors provide mechanical robustness, biocompatibility, multifunctionality, and comfort when compared to conventional rigid sensors. For this reason, next-generation wearable technologies are expected to be driven by interest in flexible, stretchable, and soft devices.

Textiles, in addition to their protective and aesthetic functions, provide an exceptional flexible platform for providing sensing functions and comfort to the wearer with diverse range of fibers, yarns, and fabric structures.

New developments in printed electronics enable mass production of sensors using efficient printing processes by considerably minimizing costs and enhancing the potential of large-scale production.

In this thesis, at first, the capabilities of temperature sensors, their sensing method, and previous research that has been conducted on them are presented. Additionally, the techniques and uses of inkjet printing are examined in detail. A comprehensive explanation of inkjet printing technology and printing challenges are issued. Dispersion is required for the development of inks that include carbon nanotubes. Due to the hydrophobic nature of carbon nanotubes, they must be distributed using a combination of mechanical and chemical methods. Numerous methods, including ultrasonication, non-covalent and covalent alterations, were used to disperse nanotubes. The use of various types of carbon nanotubes in CNT ink formulations is also studied. The development of conductive inks formulations containing CNT, PEDOT:PSS and CNT/PEDOT:PSS with a proper evaluation guideline is studied. Moreover, the concepts and properties of functional materials, as well as the critical additives used during the printing process that can have a significant influence on the printing process of conductive inks are discussed. The physical, structural, morphological, and electrical properties of the materials are investigated using various techniques (UV-Vis, FTIR, optical profilometer, SEM, AFM, optic microscope, multimeter, etc.). With relevant to print quality, the textile basis material should be dependable, maintaining a level surface and good uniformity during the printing process. In order to create conductive material sensors for temperature measurement, the inkjet printing process was used, which has the advantage of reducing ink waste while also being a low-cost and simple method. Following the procurement of CNT-based inkjet suitable dispersion, a PEDOT:PSS/CNT composite ink and a PEDOT:PSS inkjet appropriate dispersion are manufactured for temperature sensing. Appropriate ink formulations have been developed to produce high-quality inkjet-printed sensors, which are typically characterized by low imperfection points throughout the surface of

the printing material. The sensor manufacturing process is then completed by including silver yarn, followed by the application of silver based conductive glue and an encapsulating operation. Spectrophotometer studies were conducted to determine the qualities of carbon nanotube printing when many print passes are used, as well as the color characteristics of the produced specimens. The properties of CNT based, PEDOT:PSS based and CNT/PEDOT:PSS composite based sensors are compared to investigate their temperature sensing performance. Hence, proper ink formulations with appropriate physical and chemical properties that typically affects homogeneous printing surface characteristics and sensing properties, were successfully developed by analysing their morphologies and printing parameters. It was determined whether the printed temperature sensors performed properly by subjecting them to a temperature range ranging from 25 to 50 degrees Celsius. Furthermore, wear and performance tests, such as durability against bending, folding, humidity, rubbing, washing, light, and human sweat, were carried out with the help of some characterization methodologies in order to investigate the sensor's reliability and durability under unfavorable situations. The sensor real time measurement using of a mannequin and human gloved hand are reported with discussions. As a result, during our proof-of-concept inquiry, our newly designed temperature sensor was placed to a mannequin's skin and human body on a gloved hand for temperature monitoring. Our developed wearable sensor provides highly accurate temperature monitoring.

Lastly, the application based on artificial intelligence for the modeling of wearable sensors in various temperature and humidity conditions is described. Artificial neural networks (ANN) are used to model wearable sensors in various temperature and humidity conditions. The relationship between temperature, humidity, and electrical resistance is presented with the use of ANN.

This innovative wearable temperature sensor development process is expected to aid development of smart wearable technologies. The developed sensor with its good mechanical properties and excellent sensing performance is believed to be useful for use in the textile products. Moreover, this developed sensor also offers the opportunity to be directly included in wearable smart systems in industrial production. In addition to the lack of standardized and consistent manufacturing techniques, there are unfortunately not yet any regular and comparable tests that can be used for the development and implementation of wearable e-textile sensors. Hence, this study will pave a way for development phases and implementation of wearable e-textile sensors, in particular, contribute to industrialization in this area.

To conclude, the developed textile-based sensor might be a solution instead of rigid device components for human body temperature monitoring and it can be directly utilized by sticking the sensor on various garment types while maintaining the user's comfort. Hence, it reveals a strong potential for use in wearable healthcare and biomedical applications.

GIYİLEBİLİR ELEKTRONİKLER İÇİN TEKSTİL TABANLI SICAKLIK SENSÖRÜ GELİŞTİRİLMESİ

ÖZET

Esneklikleri, bükülebilirlikleri ve düzgün olmayan ve eğrisel yüzeylerle uyumlu olmaları nedeniyle esnek ve gerilebilir cihazlar dikkat çekmekte ve geniş bir uygulama alanına sahiptir. Bu alandaki artan yayın sayısı, esnek sensörlerin artan popüleritesini göstermektedir. Geleneksel rijit sensörlere kıyasla mekanik sağlamlık, biyouyumluluk, çok işlevlilik ve konfor sağlayabilmektedirler. Yeni nesil giyilebilir teknolojilere ilgi, esnek, bükülebilir ve yumuşak cihazlara olan ilgiden kaynaklanmaktadır. Esnek sıcaklık sensörlerinin, soğuk zincir sisteminde gıda lojistiğinde, itfaiyecilik görevi yapan kişilerin fizyolojik parametrelerinin izlenmesinde ve vücut ısısı ölçümü gibi alanlarda kullanımı üzerine çok çeşitli araştırmalar devam etmektedir.

Tekstiller, koruyucu ve estetik işlevlerine ek olarak, çok çeşitli lifler, iplikler ve tekstil yapıları nedeniyle kullanıcıya giyilebilir elektronik uygulamaları için olağanüstü esnek ve rahat bir platform sağlar. Örneğin, kullanıcının vücut ısını izlemek için giysilere yerleştirilen klasik termokupllar, ciddi rahatsızlıklara ve hatta cilt tahrişlerine neden olabilmekteyken, bu dezavantajdan kaçınmak için tekstil bazlı sıcaklık sensörünün kullanımı ön plana çıkmaktadır.

Basılı elektronikteki yeni gelişmeler, maliyetleri önemli ölçüde azaltarak ve büyük ölçekli üretim potansiyelini artırarak verimli baskı süreçleri kullanan sensörlerin seri üretimini mümkün kılmaktadır. Alt tabaka üzerine iletken malzeme basmak için en basit ve en uygun maliyetli tekniklerden biri inkjet baskı tekniğidir. Fonksiyonel bir mürekkebin seçimindeki en önemli adım, onun akışkan özelliklerini belirlemektir. Özellikle yüzey gerilimi, viskozite ve yoğunluk, alt tabaka üzerinde mürekkebin birikmesinde önemli bir rol oynar.

Bu tezde, öncelikle sıcaklık sensörleri, çalışma presinsipleri ve literatürde sıcaklık sensörleri üzerinde yapılan araştırmalar sunulmuştur. Ayrıca inkjet baskı teknikleri ve kullanımları detaylı olarak incelenmiştir. Inkjet teknolojisi ve basılabilirlikleri hakkında kapsamlı bir literatür incelemesi yapılmıştır. Karbon nanotüp (CNT) içeren mürekkeplerin geliştirilmesi için dispersiyon işleminin yapılması gerektirir. Elektriksel iletkenlik, kimyasal kararlılık ve son derece yüksek mekanik dayanım özellikleri, son yıllarda karbon yapıların dramatik gelişimine önemli katkılarda bulunmuştur. Karbon nanotüplerin hidrofobik doğası gereği, mekanik ve kimyasal yöntemlerin bir kombinasyonu kullanılarak dispersiyon yapılabilir. Nanotüpleri dağıtmak için ultrasonikasyonun yanısıra kovalent olmayan değişiklikler ve kovalent değişiklikler dahil olmak üzere çok sayıda yöntem kullanılmıştır. CNT mürekkep formülasyonlarında çeşitli karbon nanotüplerin kullanımı incelenmiştir. Uygun bir değerlendirme yöntemi ile CNT, PEDOT:PSS ve CNT/PEDOT:PSS içeren iletken mürekkep formülasyonlarının geliştirilmesi incelenmiştir. Ayrıca, baskı işlemlerinin gerçekleşmesi için iletken mürekkeplerde kullanılan kritik katkı

maddelerinin yanı sıra fonksiyonel malzemelerin özellikleri de önem arz etmektedir. Malzemelerin fiziksel, morfolojik, yapısal ve elektriksel karakterizasyonları (UV-Vis, FTIR, optik profilometre, SEM, AFM, optik mikroskop, multimetre vs.) yapılmıştır. Baskı kalitesi ile ilgili olarak, kullanılacak tekstil malzemesi baskı işlemi sırasında düz bir yüzey ve iyi bir homojenlik sağlamalıdır. Sıcaklık ölçümü için iletken malzeme sensörleri oluşturmak amacıyla, mürekkep israfını azaltmanın yanı sıra düşük maliyetli ve basit bir baskı tekniği olan inkjet baskı işlemi kullanılmıştır. Araştırma laboratuvarımızda sıcaklığa duyarlı CNT, PEDOT: PSS ve CNT/PEDOT kompozit mürekkeplerinin her biri için kapsamlı bir değerlendirme yapılmıştır. Tipik olarak baskı malzemesinin yüzeyi boyunca düşük kusur noktaları ile karakterize edilen, yüksek kaliteli mürekkep püskürtmeli baskılı sensörler üretmek için uygun mürekkep formülasyonları geliştirilmiştir. Sensör üretim süreci daha sonra gümüş iplik ve gümüş esaslı iletken yapıştırıcı dahil edilerek tamamlanmakta olup, ardından bir yüzey kaplama işlemi ile sonlandırılmıştır. Çok sayıda baskı geçişi kullanıldığında karbon nanotüp baskı kalitesinin yanı sıra üretilen numunelerin renk özelliklerini belirlemek için spektrofotometre çalışmaları da yapılmıştır. CNT tabanlı, PEDOT:PSS tabanlı ve CNT/PEDOT:PSS kompozit tabanlı sensörlerin özellikleri, sıcaklık algılama performanslarını incelemek üzere karşılaştırılmıştır. Böylelikle, baskı yüzeyi özelliklerini ve algılama özelliklerini etkileyen uygun fiziksel ve kimyasal özelliklere sahip uygun mürekkep formülasyonları, morfolojileri ve baskı parametreleri açısından analiz edilerek başarıyla geliştirilmiştir. Basılı sıcaklık sensörlerinin 25°C ile 50°C arasında değişen bir sıcaklık aralığına tabi tutularak düzgün çalışıp çalışmadığı belirlenmiştir. Ayrıca, sensörün olumsuz koşullar altında güvenilirliğini ve dayanıklılığını araştırmak için bükülmeye, katlanmaya, neme, sürtünmeye, yıkamaya, ışığa ve insan terine karşı dayanıklılık gibi performans testleri bazı karakterizasyon metodolojileri yardımıyla gerçekleştirilmiştir. Sensörün gerçek zamanlı performans testleri, manken ve eldivenli insan eli üzerinde gerçekleştirilerek rapor edilmiştir. Sonuç olarak, kavram kanıtı araştırmamız sırasında, yeni tasarlanmış sıcaklık sensörümüz, sıcaklık izleme için bir mankenin vücuduna ve insan vücudunda eldivenli bir el üzerinde yerleştirilmiştir. Geliştirilmiş giyilebilir sensörümüz, son derece hassas sıcaklık izleme sağlamaktadır.

Son olarak, tez çalışmasında çeşitli sıcaklık ve nem koşullarında giyilebilir sensörlerin modellenmesi için yapay zekaya dayalı uygulama yapılmıştır. Son yıllarda, yapay zekâ alanı, yapay zekanın çok çeşitli araştırma alanlarında uygulanabilmesi nedeniyle farklı disiplinlerdeki araştırmacılar tarafından büyük ilgi görmektedir. Yapay sinir ağları (YSA), biyolojik sinir ağlarının organizasyonu ve/veya işlevsel özelliğinden esinlenen bir modeldir. Yapay sinir ağları, çeşitli sıcaklık ve nem koşullarında giyilebilir sensörleri modellemek için kullanılmıştır. Elektrik direnci bir ağ çıkış değişkeni iken nem ve sıcaklık ağ giriş değişkenleridir. Elde edilen çıktıların hedef değerlerle tutarlılığını anlamak için YSA modelinin tahmin performansını analizi gerçekleştirilmiştir. Oluşturulan modelinin çıktısı, ortalama kare hata ve regresyon parametreleri kullanılarak değerlendirilmiştir. Sıcaklık, nem ve elektriksel direnç arasındaki ilişki YSA kullanılarak elde edilmiştir. Sonuç olarak, geliştirilen YSA modeli nem ve sıcaklık açısından sensörün elektriksel direnç değerini yeterli doğrulukla tahmin etme yeteneğine sahiptir. Bunun yanında, geliştirilen YSA modeli ile deneysel süreç de kısaltılabilecektir.

Bu yenilikçi giyilebilir sıcaklık sensörü geliştirme sürecinin, akıllı giyilebilir teknolojilerin geliştirilmesine yardımcı olması beklenmektedir. İyi mekanik özelliklere ve mükemmel algılama performansına sahip geliştirilen bu sıcaklık

sensörünün, tekstil ürünleri açısından faydalı olacağı öngörülmektedir. Ayrıca, geliştirilen bu sensör, endüstriyel üretimde giyilebilir akıllı sistemlere doğrudan dahil edilebilme olanağı da sunmaktadır. Standartlaştırılmış ve tutarlı üretim tekniği eksikliğine ek olarak, giyilebilir e-tekstil sensörlerinin geliştirilmesi ve uygulanması için kullanılabilecek düzenli ve karşılaştırılabilir testler maalesef henüz bulunmamaktadır. Dolayısıyla bu çalışma, özellikle giyilebilir e-tekstil sensörlerin geliştirme aşamalarının önünü açacak ve bu alanda sanayileşmeye katkı sağlayacaktır.

Sonuç olarak, geliştirilen tekstil tabanlı sensör, insan vücut sıcaklığının izlenmesi için rijit cihaz bileşenleri yerine bir çözüm olabilir ve sensör çeşitli giysi tiplerine yapıştırılarak kullanıcının konforunu da etkilemeden doğrudan kullanılabilir. Yüksek doğrulukla geliştirilen sensör ile özellikle vücut sıcaklığının sürekli takibi gereken riskli durumlarda kişinin konforunu koruyabilecektir. Bu önerinin sağlam gelişimi, akıllı giyilebilir sistemlerin yaygın olarak kabul edilmesini kesinlikle teşvik edecektir. Tekstil platformunun kullanımı, yumuşaklık, yüksek yüzey alanı, hafiflik ve iyi esneklik gibi birçok tercih edilen özellik sağladığından geliştirilen tekstil bazlı sıcaklık sensörü giyilebilir sağlık uygulamalarında kullanım için güçlü bir potansiyel ortaya koymaktadır.

Bu doğrultuda tezde, inkjet baskı kullanılarak oluşturulan giyilebilir tekstil tabanlı sıcaklık sensörleri için yeni bir konsept ortaya konmuştur. Bunların sonucu olarak, tez çalışmasında geliştirilen sensör giyilebilir sağlık ve biyomedikal uygulamalarında kullanım için güçlü bir potansiyel göstermektedir.



1. INTRODUCTION

Technological interest and media attention for wearable smart devices have recently increased dramatically. Wearable innovation has seen a huge increase in the number of products developed and investment in financial support for entrepreneurs. The growing popularity of wearable and flexible devices, from an innovative perspective, can be attributed to their potential applications as being portable, flexible, and stretchable human-interactive sensors, actuators, displays, and energy storage devices (Afroj et al., 2019; Baeg & Lee, 2020).

The devices that people wear can be highly useful in providing up-to-date and accurate information about people's activities and behaviors. In the medical field, there are multiple types of highly valued vital signs that should be monitored in the medical literature. Body temperature, heart rate, brain activity, and muscle motion are all considered important indicators of human health, and they should be monitored on a continuous basis, particularly in patients (Dias & Paulo Silva Cunha, 2018; Mukhopadhyay, 2015). Such a wearable system could potentially reduce the pressure on the healthcare system caused by issues such as an aging society's rapid growth and provide a person's healthiness regardless of time or location (Rodgers et al., 2014).

Commercial wearable devices, such as watches, jackets, and bracelets, have become popular as additional accessories to supplement the functions of smartphones and tablets (Figure 1.1) (Baeg & Lee, 2020; Dias & Paulo Silva Cunha, 2018). A highly targeted progress of high ultra-lightweight electronic materials is bringing true "wearable" IT devices such as input and output data processing and information storage that can be attached to or placed into clothing. Systems of these devices may be placed within fibers, woven fabrics, or clothing, that merge nanoscience, material technologies, and IT (Baeg & Lee, 2020).

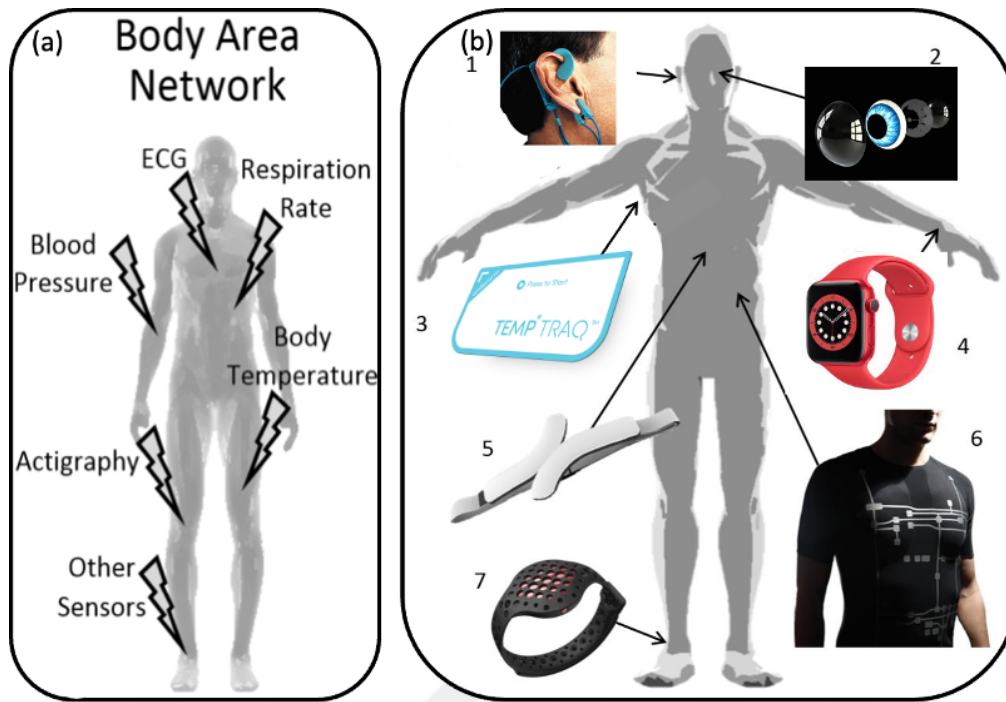


Figure 1.1 : Sensors are placed around the human body to monitor vital signs (Dias & Paulo Silva Cunha, 2018). (b) A diagram illustrating the common types of wearable health devices (1) Philips ear clip sensor; (2) Mojo Lens; (3) TempTraQ thermometer; (4) Apple Watch Series 6; (5) QardioCore; (6) Vital Jacket® (7) Moov (activity tracker) (Apple Watch Series 6 - Apple (TR), n.d.; Biodevices – Soluções de Engenharia Biomédica, n.d.; M1194A Pediatric and Adult Ear Sensor | Pulse Oximetry | Philips, n.d.; Mojo Lens | The World's First True Smart Contact Lens, n.d.; Moov™ Fitness Coach, n.d.; Qardio - State of the Heart Technology, n.d.; TempTraQ | Wireless Baby Thermometer | Temperature Monitor | TempTraq, n.d.; Dias & Paulo Silva Cunha, 2018).

1.1 Research Aim

Overall, the aim of this work is to create significant progress in the field of wearable temperature sensor networks through the implementation of a textile platform. The thesis will focus on creating a flexible, soft and thin textile-based sensor for the application of temperature measurement system for humans.

1.2 Objectives

The primary objective of this thesis is to address the need for wearable flexible, thin, and soft temperature sensors by exploring affordable new possibilities, using prior knowledge of other fabrication techniques as a starting point. In order to achieve this goal, the thesis includes a comparison of various temperature-sensitive inks that are presented throughout. It is not only an overview of the development of functional

materials using the inkjet printing technique to obtain textile-based sensors, but it is also an investigation of the implementation on reliability properties using various characterization techniques, which form the structure of this thesis. The following are the detailed objectives of this thesis:

- To overcome the drawbacks that traditional temperature sensors have, such as a lack of flexibility, rigid structures, and conformal form factors, so that the development of flexible, wearable temperature sensors for use in a variety of situations is focused.
- To generate knowledge in the formulation of several functional materials for temperature-sensitive applications, as well as to provide appropriate requirements for their implementation as an inkjet ink.
- To characterize and assess the developed sensors for a specific measurement scenario relevant to temperature sensor applications.
- To develop printed sensors that are resistant to mechanical stress and environmental conditions encountered while wearing the sensor.

1.3 Motivation for the Thesis

Starting with the idea of developing wearable temperature sensors, the properties of textile surfaces and printed electronic devices for the production of low-cost, light, soft, sensitive, and high-accuracy sensors are comprehensively studied in my PhD work. The core of the thesis focuses on the development of printed temperature sensors, including the production of temperature sensitive conductive inks. The printing technique is capable of depositing functional materials effectively and thereby providing a decrease in the amount of required material and waste. Owing to their attractive and unique electronic properties compared to other materials, nanomaterials are potential materials for upcoming printed electronic applications and, furthermore, large-scale production capability. The utilization of printing techniques for the deposition of nanomaterials is expected to provide an innovative platform for the development of functional electronic devices relying on nanomaterials. For this reason, the various nanomaterial-based ink formulations for ink-jet printing in order to have textile-based flexible sensor structure have been explored in this thesis.

One of the main features of the project is to develop low-cost, flexible, and wearable systems. Most temperature sensors available on the market are made using expensive rigid materials and high energy-consuming manufacturing processes. The production process has been carried out using techniques of production that are relatively low-cost and have low material consumption. The methodology used is also ideal for manufacturing processes on a large scale, potentially resulting in a decrease in production costs. The thesis study demonstrated here targets to fulfill the existing technological deficit in the area of flexible temperature sensors by creating low-cost sensors on textiles with inkjet printing technology.

With regard to aim of the research, although inkjet printing process enables fast implementation and design application with mask-less and noncontact operations, there are some critical issues that should be overcome. For instance, , the use of printing technology brings challenges in the manufacture of sensor devices, including the formulation of ink and the selection of substrates. Moreover, the desire to enable direct contact with the human body to obtain specific measurements while preserving the wearer's ease and comfort is a challenging issue for sensors. In this respect, the thesis work described herein targets to fill the current technological gap in the field of flexible temperature sensors by using inkjet printing technology to develop low-cost and sensitive temperature sensors on a textile structure.

1.4 Novelty Statement

The novelties of this thesis are:

1. An optimized formulation of functional inks for textile-based wearable temperature sensors.
2. A method of low-cost inkjet printing sensor patterns on textile fabric for temperature measurement.
3. A detailed analysis of the suitability of CNT, PEDOT:PSS and their composite inks as temperature sensitive-materials on textile fabric.
4. An evaluation and comparison of differently designed sensor patterns and fabricated sensors.
5. A methodology for textile-based wearable temperature sensor performance evaluation by using different measurement techniques.

6. Application of artificial intelligence to textile-based sensor networks to model the behavior of developed sensors against temperature and humidity.
7. The demonstration of inkjet-printed textile-based body temperature sensors with high sensitivity and operability in the 32-42 °C temperature range.

1.5 Structure of the Thesis

Six distinct sections comprise the structure of this thesis dissertation. Chapters 2 reviews temperature sensors, printing technology, temperature sensitive materials, textile-based sensors as well as sensor design aspects. Chapter 3 introduces brief information about the experimental set-up, methods, equipments, characterization and fabrication techniques used in this thesis. Chapter 4 discusses the potential of materials for formulation of functional inks for sensor applications, as well as the fabrication and characterization of printed wearable temperature sensors and their performance analysis. Moreover, a detailed discussion on results are given in this chapter as well. In chapter 5, artificial neural network architecture was introduced to model the CNT printed sensor structure's behaviour in different humidity and temperature environments. In chapter 6, thesis results and achievements are summarized, and the thesis's result is stated. This chapter further presents future development ideas and recommendations. The content of the chapters are summarized as follows:

In Chapter 2, the main topic is the analysis of textile-based wearable temperature sensors, the type and principle of temperature sensors and their design aspects, and also related works in the literature. This chapter follows the attainment of two different objectives: wearable sensor applications and the establishment of a temperature sensor guideline. The fundamentals of textile-based sensors and the requirements and properties of a temperature sensor are described in detail. Moreover, Chapter 2 covers printing technologies for sensor applications. Various printing processes, such as screen, gravure, and flexographic printing are discussed, then the inkjet printing process in detail, paying special attention to the concepts and principles required to fully comprehend the technique, as well as the printing characteristics that define inkjet printability (viscosity, surface tension, contact angle, particle size, z dimensionless number, volatility, resolution, and surface roughness) is explained. Chapter 2 also introduces temperature-sensitive inks for temperature sensors. The formulation guidelines for conductive inkjet inks are given in detail, with examples of carbon

nanotube and PEDOT:PSS ink. In this regard, the most important properties of functional materials, as well as the fundamentals and main parameters of inkjet inks, are explained.

In Chapter 3, the first section of the chapter describes the equipments used to manufacture the ink formulations and temperature sensors developed in this thesis. And then, this chapter summarizes the concepts and principles discussed in this thesis, along with the techniques used to characterize the ink materials and sensors and their methodology.

In Chapter 4, firstly the fabrication of temperature-sensitive inkjet inks is explained. At this section, our laboratory's achievement of three distinct ink formulation goals are given: the development of a proper evaluation guideline for CNT inks and the formulation of PEDOT: PSS and CNT/PEDOT composite inks. Moreover, the concepts and properties of functional materials, as well as the critical additives used during the printing process that can have a significant influence on the fabrication of the printed sensors are discussed. Secondly, the development of a textile-based temperature sensor is also explained. The explanation of fundamental design information is based on data gathered from a literature review. Additionally, the ink deposition technique used in inkjet printing and an environmentally friendly method for encapsulating inkjet-printed structures are demonstrated. Moreover, the performance of the printed temperature sensors was evaluated by exposing them to a range of temperatures from room temperature to 50 °C. Investigation on the effect of serpentine patterns on resistance and TCR value was discussed. Moreover, some performance tests such as durability against bending, folding, perspiration, washing, light, abrasion, humidity etc. with characterization methodologies to investigate the sensor's reliability, sensitivity, and sensor real time measurement are reported with discussions.

In Chapter 5, the application based on artificial intelligence for the modeling of wearable sensors in various temperature and humidity conditions is described. Artificial neural networks (ANN) are used to model wearable sensors in various temperature and humidity conditions. The relationship between temperature, humidity, and electrical resistance is presented with the use of ANN.

In Chapter 6, the results and achievements of the research presented in this thesis are summarized, and the final conclusion of this thesis is presented. This final chapter further includes recommendations for future research and development.





2. STATE OF THE ART

2.1 Temperature Sensors

Temperature sensors can be used to measure body temperature from the mouth, skin, eardrum, under the arm, and rectum, and any rise in temperature suggests a problem with the patient's immune system (G. M. N. Islam et al., 2020).

In this section, the types of temperature sensors and their sensing mechanisms are given in detail. Then, some published studies on flexible sensors are summarized.

2.1.1 Sensing mechanism of temperature sensors

Many physical stimuli, such as resistance, volumetric expansion, vapor pressure, and spectral properties, are temperature-dependent. For this reason, the physical stimuli associated with temperature are used in the development of temperature measurement systems. The measurement result, i.e. temperature value, is derived from a measurement model based on a measurement principle, that is, from a mathematical relationship between the data contained in a measurement (Childs, 2001a; Dramićanin, 2018). Resistance temperature detectors, thermocouples, and thermistors are the most common types of temperature sensors.

2.1.1.1 Resistance temperature sensors

Resistance temperature detectors (RTD) utilize temperature and electrical resistance adjustments in conductors and semiconductors to detect changes in temperature. However, considerations such as cost, temperature coefficient of resistance (TCR), resistance to oxidation, and manufacturing restrictions limit the choice. Copper, gold, nickel, platinum and silver are the most commonly used materials. The properties of these materials are shown in Table 2.1 (Childs, 2001c). For example, platinum may not be preferred due to its low TCR value and high cost, but it has excellent thermal properties and is resistant to oxidation (P. Liu et al., 2009). Nickel is used because of its high TCR value and low cost (Husain & Kennon, 2013). Copper's disadvantages include lower resistance than other metals such as nickel and silver, and

susceptibility to corrosion. Copper can be used in a controlled environment and in application areas where a linear temperature property is required (Hagart-Alexander, 2003). Gold has higher electrical conductivity and is more flexible compared to copper (C.-Y. Lee et al., 2008). Apart from these, conductive polymers and carbon-based materials are widely used for temperature sensor applications (Bali et al., 2016; Dinh et al., 2016; J. Yang et al., 2015).

Table 2.1 : Resistance temperature coefficient and resistivity values of various materials at 20 °C.

Material	Resistance Temperature Coefficient (TCR) (C ⁻¹)	Resistivity (Ωm)
Nickel	6.0 x 10 ⁻³	59 x 10 ⁻⁸
Silver	4.0 x 10 ⁻³	1.6 x 10 ⁻⁸
Copper	3.9 x 10 ⁻³	1.7 x 10 ⁻⁸
Platinum	3.85 x 10 ⁻³	11 x 10 ⁻⁸
Gold	3.4 x 10 ⁻³	2.4 x 10 ⁻⁸

The TCR value is positive when the resistance increases with an increase in temperature, and negative when the resistance decreases with an increase in temperature (Chu et al., 2015). The TCR can be calculated with the following equation:

$$TCR = \frac{R_b - R_a}{R_a(\Delta T)} \quad (2.1)$$

$\Delta T = T_b - T_a$ is the change in temperature of the sensor, T_a is the initial temperature of the sensor, T_b is the current temperature of the sensor, R_a is the sensor's initial resistance, and R_b is its resistance at a certain temperature. The sensitivity of the temperature sensor is calculated by the formula below (Ali et al., 2019).

$$S_{sens} = \frac{\Delta R}{R_a} \quad (2.2)$$

Here, S_{sens} sensitivity value and $\Delta R = R_b - R_a$ show the resistance change (Ali et al., 2019).

The use of RTDs in industrial applications is becoming increasingly dominant compared to thermocouples or other types of thermal sensors, due to their high accuracy and repeatability.

Looking more specifically, the RTD is actually a resistor element consisting of integrated components that are advantageous over many standard electronic

measurement techniques and suitable for measurement in most situations (Seleghim Jr, 2011).

In order to obtain the desired performance from the sensor, a number of issues, such as material, size, temperature range, accuracy, precision, configuration, resistance temperature coefficients, should be taken into account (Seleghim Jr, 2011).

RTDs generally have better tolerance than thermistors. Typical tolerance for RTDs is:

- Platinum: 0.01% to 0.03%
- Copper: 0.2%
- Nickel and nickel / iron: 0.5%

An RTD and a thermistor are similar in structure, except that they have better tolerance and overall low resistance (Ball, 2003).

2.1.1.2 Thermocouples

A thermocouple is a type of temperature sensor that consists of two different thermoelements (conductors, semiconductors, or a combination of both) (Figure 2.1) that allow the generation of a potential difference or voltage based on the thermoelectric phenomenon which is known as Seebeck. According to Seebeck, if heat energy is applied to the two different conductor ends, a thermal voltage (electromotive force, emf) occurs between the end points. The electromotive force causes the electrons to move from the hot end to the cold end. This force, which is formed in the opposite direction to the electron flow, is called the "Seebeck electromotive force" and the event is called the "Seebeck Thermoelectric Effect" (Childs, 2001b; Childs et al., 2000; Grafa et al., 2007). The rugged, low cost, and self-powered properties of thermocouples make them preferable for having a stable reference temperature. However, due to its continuous exposure to sudden environmental changes, its use as a wearable sensor is limited (Davidson et al., 2017).

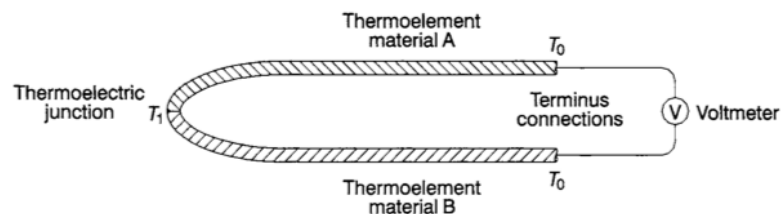


Figure 2.1 : Schematic of a thermocouple made of A and B thermocouple (Childs, 2001b).

The potential difference (V) is calculated as:

$$V = \alpha \times \Delta T \quad (2.3)$$

where α denotes the difference in the Seebeck coefficient of thermoelement A and B, and ΔT is the difference between the temperatures of hot and cold junctions. The Seebeck coefficient is usually measured in $\mu\text{V/K}$ (H. S. Lee, 2010).

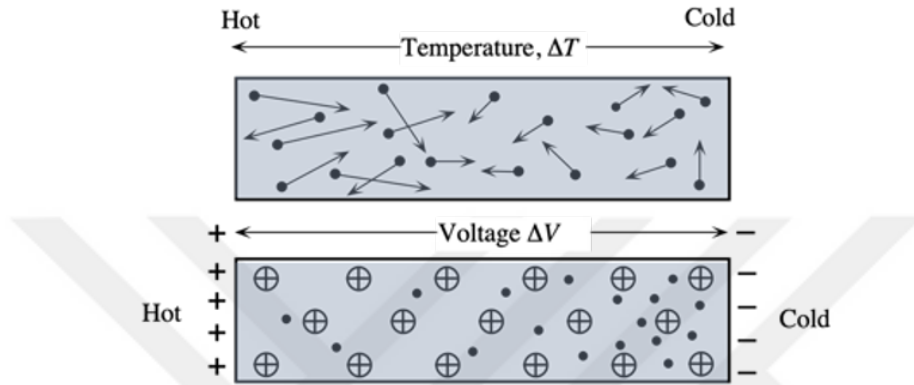


Figure 2.2 : Representation of the Seebeck effect (Kasap 2001).

When a conduction material undergoes a temperature change, electrons begin to move from one end of the wire to the other (Figure 2.2) (Chambers, 1977). Here, the direction of electron diffusion changes depending on the electrical properties of the material. When the electrons spread from the hot to the cold region, the positive metal ions remain exposed and if they accumulate electrons in the cold regions, a negative electromotive force (emf) will be generated. If electrons move from a cold to a hot region, the cold side is positive and the hot side, a positive emf is produced (Kasap 2001). This is known as the "Seebeck effect" (Seebeck, 1822).

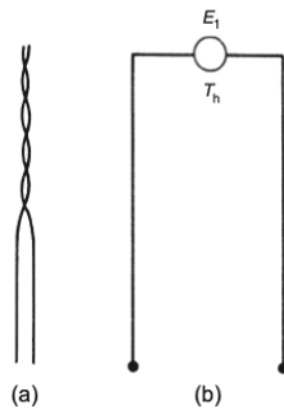


Figure 2.3 : (a) Thermocouple and (b) equivalent circuit (Morris & Langari, 2012).

Figure 2.3a shows the structure of the thermocouple, and Figure 2.3b shows the equivalent electrical circuit of the thermocouple. The emf generated at the point where the different wires are connected is a voltage source. The emf produced at the hot welding point is measured from the two open end points (or reference points) of the thermocouple (Morris & Langari, 2012).

The advantages of thermocouples are low cost, stable and fast, rapid response to temperature changes and working at high temperatures. The materials of thermocouple wires are not inert, and the thermoelectric voltage developed along the length of the thermocouple wire will prevent corrosion. The relationship between process temperature and thermocouple signal is not linear. Calibration of the thermocouple should be done by comparison with a nearby comparison thermostat while in use. Also, there is no linear relationship between the thermocouple and the process temperature signal. A separate thermocouple is also required to calibrate the thermocouple (Lanzetta & Gavignet, 2011).

2.1.1.3 Thermistors

Thermistors are ideal for detecting small temperature changes and are manufactured from materials that have a significant change in electrical resistance with temperature (Alan, 2001; K. A. Hu et al., 2011). Thermistors as thermally sensitive resistors can have a negative temperature coefficient (NTC) or a positive temperature coefficient (PTC) (Bentley, 2005; “Instrument Engineers’ Handbook: Process Measurement and Analysis,” 2013). The semiconductor-based thermistors exhibit NTC behavior which differentiates them from thermocouples based on the Seebeck effect (Wen et al., 1999).

The resistance-temperature relationship is not very linear. It can operate in the temperature range of -50°C to 200°C depending on the type of material used and the production method (S. Wang et al., 2003). The reference temperature for most applications is 25°C (Ibrahim, 2002).

The thermal index (temperature constant) β depends on the material used in the manufacture of the thermistor. The thermal index is another criterion that indicates the measurement capability of the temperature sensor connected to the resistance (M. M. Hussain & El-Atab, 2019). This parameter also depends on temperature and manufacturing tolerances. Values for NTC thermistors generally range from 2000k to 5000k (Ibrahim, 2002).

$$S = \frac{1}{R} \frac{dR}{dT} = - \frac{B}{T^2} (K^{-1}) \quad (2.4)$$

Generally speaking, its accuracy is 1 °C for standard types and ± 0.2 °C for high precision types (Regtien & Dertien, 2018).

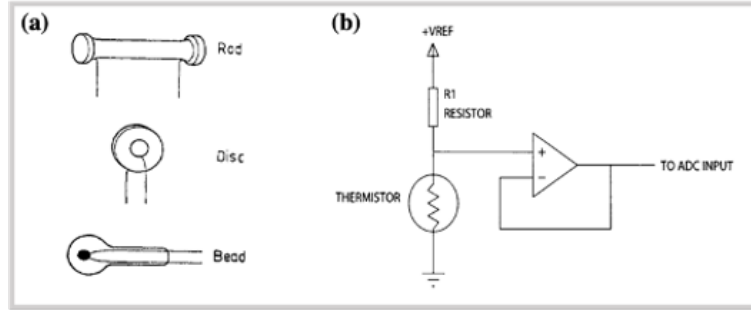


Figure 2.4 : (a) Thermistor constructions (Bentley, 1984) and (b) a thermistor circuit (Ball, 2003).

Table 2.2 : Temperature sensors: characteristics and typical applications (S. Wang et al., 2003).

Sensor	Signal output	Measurement range (°C)	Accuracy	Typical application possibilities
RTD	Resistance	-200 ~ 850	± 0.2 °C	Precise and stable measurements
Thermocouple	Voltage	-250 ~ 400	± 0.5 °C	Food and agricultural applications
Thermistor	Resistance	-50 ~ 200	$\pm 0.1\%$	Reference for data loggers

The most common types of thermistors are constructed in rod, disc, and bead form (Figure 2.4a). They are encapsulated in glass or plastic. The bead thermistor is the smallest in size, with a diameter of 0.15 mm to 1.25 mm (Godse & Bakshi, 2009). Figure 2.4b exhibits a typical circuit used to measure the temperature with a thermistor. The accuracy of this circuit depends on thermistor tolerance, resistance tolerance and reference accuracy (Ball, 2003). Table 2.2 presents examples of typical applications of temperature sensors mentioned above. (S. Wang et al., 2003).

2.1.2 Skin temperature measurement

The ability to sense and regulate body temperature is critical for human health (Lim et al., 2008). Body temperature assessment and evaluation have a strong influence on nursing care decisions, laboratory test orders, medical diagnosis, and treatment in clinical practice (Sund-Levander & Grodzinsky, 2012). Physiological abnormalities

and death will arise from a temperature change of $\pm 3.5^{\circ}\text{C}$ from the resting temperature of 37°C (Lim et al., 2008). Though temperature change provides vital health information, continuous body temperature monitoring and data processing is uncomfortable with current devices. These devices enable merely intermittent measurement of temperature (Tamura et al., 2018).

Despite the fact that skin temperature is varied and vulnerable to the surrounding environment, it can be a reliable predictor of core temperature change depending on the measurement site and level of activity (Tamura et al., 2018). The ideal location for temperature measurement would be determined by the kind of instrument provided and used, and even the measurement reasons. In medical practice, sublingual, axilla, and tympanic temperatures are frequently preferred, while in sports and exercise conditions, rectal, oesophagus, and gastrointestinal temperatures are most intensively chosen (Ilsley et al., 1983; Lim et al., 2008). Some areas, such as the upper thorax or the axilla, have minimal effects on ambient temperature, allowing skin temperature to be more effective (Tamura et al., 2018).

2.1.3 Flexible temperature sensor studies

Yang et al. demonstrated an ultrasensitive wearable temperature sensor prepared using graphene nanowalls (GNWs) in combination with polydimethylsiloxane (PDMS) exhibiting a TCR of $0.214^{\circ}\text{C}^{-1}$ (J. Yang et al., 2015). In another study, Wang et al. developed a PEDOT: PSS based temperature sensor exhibiting a high sensitivity of $-0.77\% \text{ }^{\circ}\text{C}^{-1}$ in the range of 25°C to 50°C (Y.-F. Wang et al., 2020). Soni et al. designed a skin-conformable GO/PEDOT: PSS based temperature sensor. This sensor includes an NTC behavior with approximately 80% resistance change and a sensitivity of $1.09\% /^{\circ}\text{C}$ with temperatures ranging from 25 to 100°C (Soni et al., 2020). Zhang et al. developed a hybrid sensor based on PEDOT: PSS/reduced graphene oxide aerogel that exhibits high performance in temperature-sensitive properties (F. Zhang et al., 2020). Wu et al. fabricated a flexible temperature sensor based on the flake graphite (FG)/ CNT/PDMS composite by the screen-printing method. When the mass ratio between FG and CNT is close to 4:1, the TCR value is mostly preserved at the same level as 0.028 K . FG/CNT/PDMS films also exhibited good linearity (L. Wu et al., 2019). The miniaturized flexible temperature sensor based on the MWCNT/polyvinylidene composite (PVDF) was developed by Sibinski et al. and the

TCR of the sensor was found to be $0.13\% \text{ K}^{-1}$ (Sibinski et al., 2010). In a further study, the temperature sensors were fabricated using graphene/ PEDOT: PSS ink with an inkjet technique and the printing was done on a skin-conformable polyurethane plaster (adhesive bandage). Graphene / PEDOT: PSS temperature sensors exhibited a high sensitivity of $0.0006^{\circ}\text{C}^{-1}$ with a negative temperature dependency (Vuorinen et al., 2016). Maskey et al. introduced a PEDOT:PSS/ functionalized graphene oxide–polyvinylidene fluoride (FGO–PVDF) based thermistor with a $1272.57 \Omega /^{\circ}\text{C}$ sensitivity, $0.395 \text{ }^{\circ}\text{C}^{-1}$ TCR, and a 40 s per $^{\circ}\text{C}$ response time from 10°C to 30°C for intelligent packaging systems along the food logistic chain (Maskey et al., 2020). Turkani et al. developed a CNT based NTC thermistor by using additive print manufacturing processes. A 53% resistance change was observed with a TCR of $-0.4 \text{ }^{\circ}\text{C}^{-1}$ in the range of -40°C to 100°C (Turkani et al., 2018). Zhu et al. demonstrated circuit design strategies that can enhance the robustness and accuracy of a CNT based flexible temperature sensor having a TCR of 14.35×10^{-3} (Zhu et al., 2018). Some of the reported temperature sensors having composite structures are shown in Table 2.3.

Table 2.3 : Several reported temperature sensors: materials, production methods, and performance.

Material	Method	Measurement Range ($^{\circ}\text{C}$)	TCR ($\%/^{\circ}\text{C}$)	Ref.
GO/PEDOT: PSS	Drop casting	25-100	1.09	(Soni et al., 2020)
PEDOT:PSS/rGO-aerogel	Freeze drying	30-50	1.69	(F. Zhang et al., 2020)
MWCNT/GNP	Vacuum filtration	30-100	0.138	(J. Huang et al., 2019)
MWCNT/ PVDF	Printing/ dip coating	20-120	0.13	(Sibinski et al., 2010)
Graphene-PEDOT: PSS/ PU	Inkjet printing	35-45	0.06	(Vuorinen et al., 2016)
rGO-PEDOT:PSS/ PU	Spin coating	30-80	1.34	(Trung et al., 2016)
PEDOT: PSS-CNT/ Flexible substrate	Mask printing	22-48	0.61	(Honda et al., 2014a)
PEDOT:PSS/ FGO–PVDF	Mask printing/ bar coating	10-30	0.395	(Maskey et al., 2020)
CNT/ PET	Screen/ gravure printing	-40-100	0.4	(Turkani et al., 2018)
FG/CNT/PDMS	Screen printing	40-80	2.8	(L. Wu et al., 2019)
SWCNT/ SEBS	Photolithography	16-55	1.435	(Zhu et al., 2018)

2.2 Printing Technologies for Sensor Fabrication

This section summarizes recent advancements in the literature on printing methods for sensor manufacturing. Several printing processes, such as screen, gravure, and flexographic printing are discussed.

Figure 2.5 illustrates three different printing techniques, each of which can be explained as a "positive" or "negative" contact printing technique, as well as a "non-contact" printing technique. Contact printing is the first of two methods, because the substrate is physically touched by the print master. Positive contact printing (commonly referred to as relief printing) involves using a master image to make positive-contact prints. Screen printing is an example of negative contact printing in which a mesh is placed over the substrate. The term "non-contact printing" refers to the absence of physical contact between the master and the substrate. Inkjet printing is an example of non-contact printing (Beedasy & Smith, 2020).

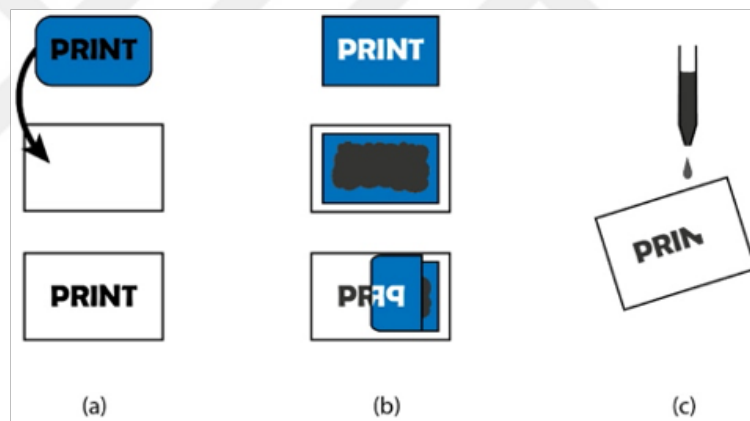


Figure 2.5 : Diagram of the three printing techniques. (a) shows the positive contact printing similar to stamping, (b) exhibits the negative contact printing similar to screen printing, and (c) shows non-contact printing similar to inkjet printing (Beedasy & Smith, 2020).

Techniques such as screen printing, spray printing, micro-transfer printing, gravure printing, laser printing, manual coating, and lithography are all used to print with promising results. The properties of some conductive inks and printing techniques are given in Table 2.4. Screen printing is a proven printing technique that utilizes a screen as a stencil and layers of ink on the printing surface. On the positive side, the pattern is of great quality; nevertheless, it is no longer extensively utilized because of its low resolution, difficult design, inefficient ink consumption, and inadequate control of ink thickness. With proper modifications and optimizations, photolithography may be

used to print the designs on flexible and easily-damaged substrates. Nevertheless, it is a slow, inefficient technique that necessitates the application of a mask before casting. Moreover, the equipment also requires a clean room, requires hazardous chemical solutions for etching, and uses more material (Salim & Lim, 2017). The compatibility of inkjet printing with different substrates, and the precise and fast transfer of ink on a substratum without contact with the surface, makes the inkjet printing technique very promising among the other techniques, including spin-coating, dip-coating, or screen printing (De Gans et al., 2004).

Table 2.4 : Properties of several previously developed conductive inks and their fabrication with different technologies.

Material	Solvent	Process	Viscosity	Resistance / (or resistivity)	Ref.
SWCNT	water	Inkjet printing	-	12 Ω .cm	(Simmons et al., 2007)
SWCNT	water	Inkjet printing	-	132 Ω /sq	(Tortorich et al., 2014)
SWCNT	water	Ink pen	-	20 k Ω /cm	(Han et al., 2014)
MWNT-(CO ₂ H) _n	water	Inkjet printing	-	40 k Ω /sq	(Kordás et al., 2006)
MWCNT	ethanol	Screen printing	2.09 Pa.s	0-5-13 Ω /sq	(Menon et al., 2017)
PEDOT:PSS	-	spray coating	8-25 mPa.s	10.8±1.2 k Ω /sq	(Isoniemi et al., 2015)
Graphene/PEDOT:PSS	DMSO/EG	inkjet printing	-	-	(Seekaew et al., 2014)
PEDOT:PSS/SWCNT-COOH	water/Ethanol	inkjet printing	-	10 k Ω /sq	(Mustonen et al., 2007)
Graphene/PEDOT:PSS	ethanol/diethylene glycol	inkjet printing	7.2 mPa.s	~9 k Ω	(Isoniemi et al., 2015; Vuorinen et al., 2016)
Graphene platelets/PANI	ethanol	screen printing	125 mPa.s	8.4 S/cm	(Xu et al., 2013)

2.2.1 Screen printing

Screen printing is a method of printing in which ink is passed through a mesh screen (Figure 2.6). Printing occurs when ink is imprinted or deposited onto the screen and then pushed by the squeegee blade through the screen through an image-specific open mesh to create a print (Weng et al., 2010).

Silver, gold, platinum, and metal carbon-based conductive inks are available for screen printing. Printing inks with certain complicated rheological properties necessitate the use of both conductors as well as suitable solvents and organic binders. Hence, while retaining the rheological characteristics of the ink, a balance between the conductive solids content and liquid solvent content must be achieved while exceeding the conductivity percolation threshold (Weng et al., 2010).

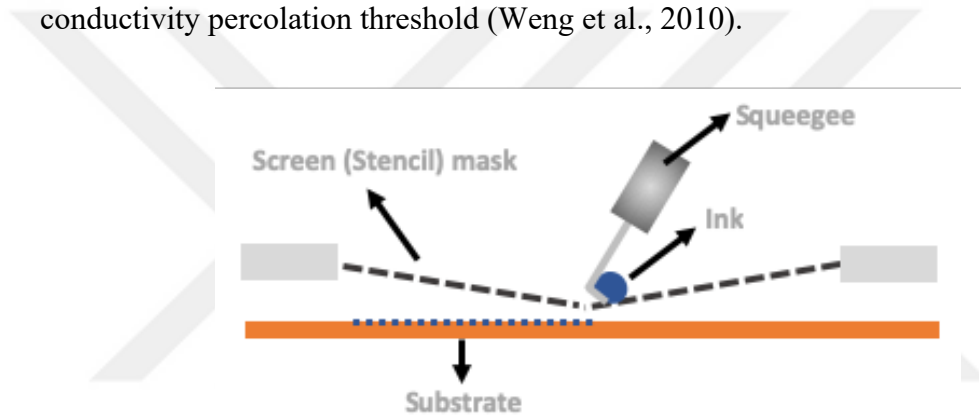


Figure 2.6 : Screen printing. Adapted from (Khan et al., 2015).

2.2.2 Gravure printing

The transfer of functional inks by direct contact between the engraved structures and the substrate is called gravure printing (Khan et al., 2015). The high printing pressure and adhesion forces between ink and substrate assist in transferring ink from the carved cells to the substrate. Figure 2.7 shows the gravure printing process (Weng et al., 2010). To remove excess ink from the rotating cylinder, a doctor blade is utilized (Khan et al., 2015). Along with the great throughput and resolution that gravure provides, it also has the ability to transfer varying amounts of material to different parts of the substrate. The thickness of the printed layers is regulated by the depth and density of the cells. In gravure printing, ink from the cylinder is transferred to the printing substrate at high pressure (3 MPa). To enable effective loading and transmission of the ink, the viscosity is generally between 0.05 and 0.2 Pa.s (Weng et al., 2010). Due to gravure printing's difficulty in generating homogeneous structures

with sharp edge pattern lines, its application is limited to the production of such layers in electrical devices (Khan et al., 2015).

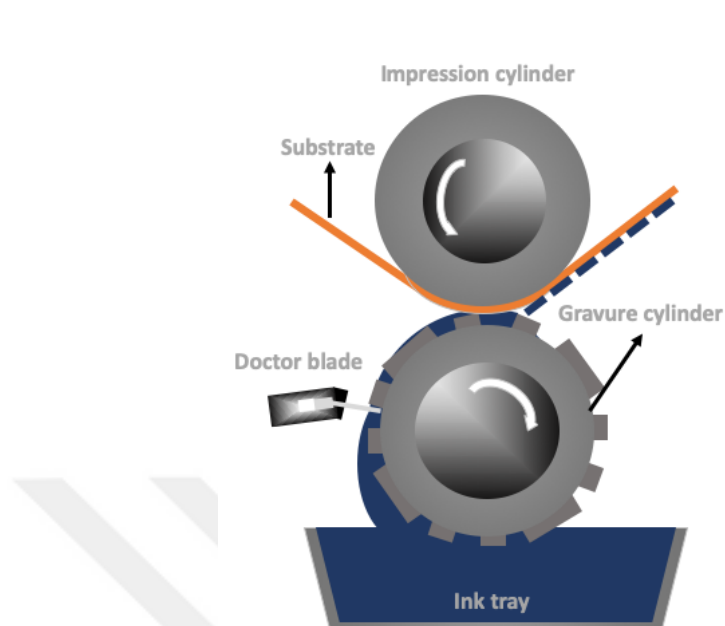


Figure 2.7 : Schematic of gravure printing process. Adapted from (Kipphan, 2001).

2.2.3 Flexographic printing

Flexographic printing is more desirable than gravure and offset for high resolution designs, and it has superior printing speeds for printed electronics. Photolithography is used to generate patterns, which are then applied to a cylinder as illustrated in Figure 2.8. These raised patterns are applied to the substrate that lies between print/plate and impression cylinders on contact with the ink-marked regions of the annilox cylinder. Due to the consistent thickness, this helps producing thin layers, resulting in greater pattern reliability and finer edges when printing with gravure. In case if any of the cells is blocked or damaged by continuous operations, flexography is an inconvenient method of producing continuous printing patterns. The pattern lines are also diverging from the desired resolutions because the flexible or polymer plate that transfers the ink deforms as a result of pressure applied to it (Khan et al., 2015).(Khan et al., 2015).

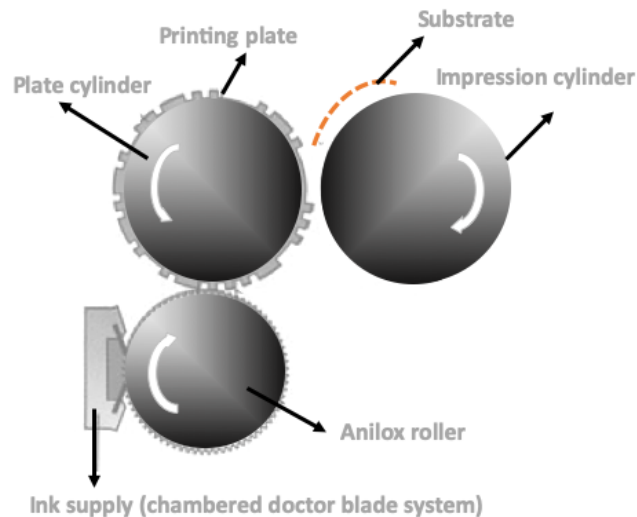


Figure 2.8 : Schematic diagram of flexographic printing unit. Adapted from (Kipphan, 2001).

2.2.4 Offset printing

Offset printing has two unique properties, as seen in Figure 2.9. Also, the design is not applied directly to the substrate but is first applied to an offset roller. In addition, the arrangement of hydrophobic and hydrophilic regions in the image roller is designed to respond to a hydrophobic ink. Along with a hydrophilic water-based ink, the hydrophobic ink is applied to the printing plate. The pattern from the underlying substrate is captured by these two repellers, which then transfer to the offset cylinder, where it completes its design. In addition to hydrophobic/hydrophilic ingredients, inks must also have very high viscosities ($> 100 \text{ Pa.s}$) (Weng et al., 2010).

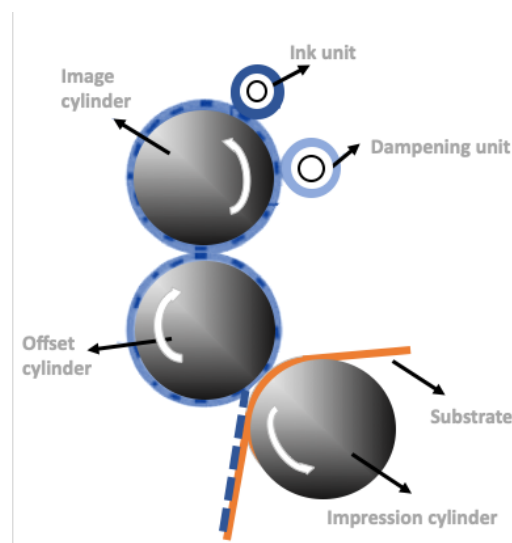


Figure 2.9 : Schematic diagram of offset printing unit. Adapted from (Kipphan, 2001).

2.2.5 Inkjet printing

Inkjet printing is a basic concept that refers to drops of colorant ink jetted into particular areas on a textile substrate to generate a printed pattern with a specific resolution (Matavž et al., 2017). The printed pattern and pattern geometry can be precisely controlled with inkjet printing (P. Chen et al., 2010). The characteristics of the textile are not altered by inkjet printing, which is completely automated and scalable for mass production (Khair et al., 2019).

Inkjet-printed conductive textiles are being used to manufacture wearable sensors, antennas, conductive paths, and energy devices (Ali, Khan, et al., 2019; Amendola et al., 2017; Delekta et al., 2017; Karim et al., 2017). Inkjet printing is a highly scalable fabrication technique that is improving and beginning to transform the area of flexible electronic sensors by offering lower manufacturing costs (Kai Yang et al., 2013). The inkjet printing has been emphasized by no masking, non-contact functionality, less material consumption, low temperature, and simple process (Krebs et al., 2013). Therefore, inkjet printing technique can enable the sensors to be low-cost and potentially disposable (Lo et al., 2020). The inkjet technology is being combined with conductive inks for producing complex electronic devices. This technique makes the process quick and easy to modify. The following section 2.3 discusses in detail the inkjet printing technology used during this study.

2.3 Inkjet Printing Technology

This section explains the inkjet printing technique utilized throughout the research, including its basics and principles, and the inkjet printability properties (viscosity, surface tension, contact angle, particle size, z dimensionless number, solidification, resolution, and surface roughness).

2.3.1 Fundamentals of inkjet printing

There has been a growing interest in the application of inkjet printing to electronic textiles in recent years. Advantages such as flexibility, simplicity, efficiency, creativity, and eco-friendliness lead to inkjet printing prestigious from other printing techniques.

Inkjet printing is a method of transferring ink to a wide surface area of textiles that is automated and controlled (Loffredo et al., 2009). It involves an ink-filled cartridge that

travels horizontally back and forth, making microdroplets of ink that are then fired out from a nozzle that is just one-tenth of a millimeter in diameter (Salim & Lim, 2017). Without the use of masking or stencils, it can print high-resolution functional patterns on textiles (Tortorich et al., 2018).

Inkjet printers are categorized into two classes of technologies, named, Continuous Inkjet (CIJ), and Drop-on-Demand (DOD). Types of inkjet printers are depicted in Figure 2.10.

2.3.1.1 Continuous ink-jet (CIJ) printing

A continuous jet of ink is generated at a high frequency (50-175 kHz) by a pressure pump. The solid ink stream is separated into small droplets using ultrasonic piezo-vibrations at the exit of the nozzle orifice. An electrode, which is close to the nozzle, charges the unnecessary droplets and transfers them to a recycling gutter using an electric field. The deflector (binary or multiple system) applies an electrostatic field capable of deflecting only charged droplets and provides precise and controlled patterning (Sette, 2014). The droplet sizes in a continuous system may be as tiny as 20 μm , although in most cases, 150 μm is common (Wallace, 2012).

This kind of system is referred to as a "continuous" system since droplets are created continuously and their trajectories are adjusted by the amount of charge provided (Wallace, 2012). Additionally, owing to the steady ink flow, continuous processing eliminates nozzle clogging, thus enabling the use of volatile solvents (Sette, 2014).

CIJ systems are used in a broad variety of product labeling applications, such as for food and medication packaging. Their throughput capacity is exceptionally high, and is ideal for applications where it is utilized continuously. CIJ systems have a certain percentage of unused ink droplets that must be recirculated or wasted in order to function. This limits the effectiveness of employing CIJ for micromanufacturing (Wallace, 2012).

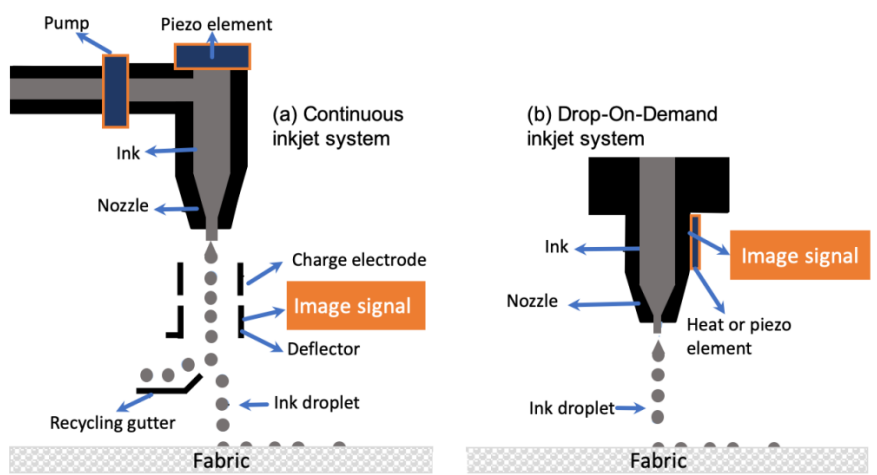


Figure 2.10 : (a) Continuous and (b) Drop-on-Demand inkjet systems. Redrawn from ref. (Khan et al., 2019).

2.3.1.2 Drop-on-Demand (DOD) inkjet printing

In the Drop-on-Demand Inkjet (DOD) inkjet system, drops are sprayed when they are required to print onto the surface. The inkjet nozzle head jets out droplets of extremely tiny size at the specified pulse applied by either thermal or piezoelectric actuators (Khan et al., 2015). In thermal DOD, when the temperature of a resistor in the ink chamber rises above a certain threshold, vapor from the neighboring liquid is produced, which results in the formation of a bubble. In piezoelectric DOD, the activity of a piezoelectric actuator generates a pressure pulse within the ink chamber, which is then used to eject a droplet from the chamber (Beedasy & Smith, 2020). Generally, drops are produced by the function of a pressure pulse generated by the dynamic deflection of the pulsed-voltage activated piezoelectric diaphragm (Hudd, 2011; Lau & Shrestha, 2017). Figure 2.11 depicts a single nozzle of a piezoelectric inkjet printhead. The resulting printable device includes a piezo actuator, an ink chamber, a cylindrical nozzle, and a nozzle plate. Pressure fluctuation, viscosity force in the ink, surface tension, and adhesion force between the ink and the nozzle's inner wall are among the major influencing forces on the nozzle (He et al., 2017). Accelerating voltage changes can be used to control the velocity and volume of ejected droplets on a piezo DOD printhead. By design, droplet size is primarily determined by the nozzle diameter, and if a user deserves smaller droplets or larger droplets, they can choose a nozzle that encompasses that need (Beedasy & Smith, 2020).

The piezo DOD printhead is the most common type of DOD printhead used in industrial and research applications. Printing machinery equipped with demand-mode

inkjet systems has mostly been utilized in desktop printers, and currently forms a large proportion of the low-end printer industry. In addition, demand mode inkjet systems do not require fluid recirculation, and this makes using them simpler than continuous mode technology (Wallace, 2012). For printing conductive ink, since less ink is wasted during printing, DOD is a preferred option (Lawes et al., 2015).

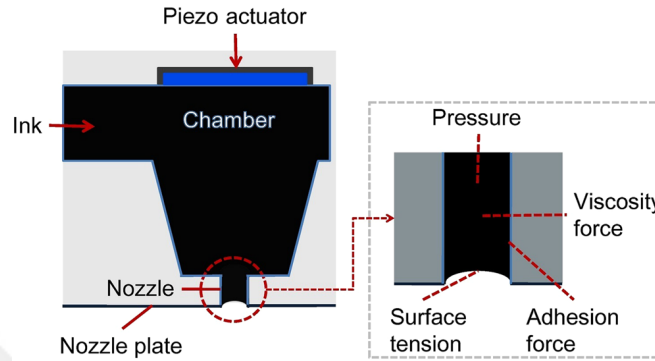


Figure 2.11 : Representation of the piezoelectric inkjet printhead and various forces that have a significant impact on the nozzle (He et al., 2017).

2.3.2 Inkjet printability issues

Like all graphics printing inks, conductive inkjet inks must meet some fundamental characteristics, including: good printability, adhesion to various surfaces, excellent resolution, low maintenance, and long shelf life (Kamyshny & Magdassi, 2014). Hence, the most critical features of an ink to consider while selecting it are its fluid characteristics, such as viscosity, density, and surface tension. Jetability, size and form of the deposited droplets, as well as wetting of the substrate, are affected by these parameters. As ink droplets contact the substrate, the ink interacts with the substrate, and this interaction is influenced by the ink's surface tension, viscosity, surface energy, and surface roughness (Karim et al., 2017).

In addition, to regulate the geometrical characteristics (Soltman & Subramanian, 2008; Stringer & Derby, 2010; J.-T. Wu et al., 2010) (for example; droplet diameter and line width) of the dried films, critical parameters such as viscosity (Tuladhar & Mackley, 2008), surface tension, evaporation rate (J. S. Park et al., 2010), drop volume of ink, and surface energy of the surface (Kettle et al., 2010; Li et al., 2008; Son & Kim, 2009; Street et al., 2006) must be precisely adjusted in inkjet printing. Table 2.5 and Figure 2.12 show a comparison of the characteristics of different printing techniques used in the printing of conductive materials.

Table 2.5 : Comparison of various properties of printing (Baeg et al., 2013; Cummins et al., 2012; Q. Huang & Zhu, 2019; Thomas et al., 2018).

Method	Viscosity (mPa s)	Surface tension (mN/m)	Throughput (m ² /s)
Screen	500-5000	35-50	2-3
Gravure	100-1000	25-45	3-60
Flexographic	50-500	10-30	3-30
Offset	100 - 10000	≈ 37	3-30
Inkjet	2 -20	25-50	0.01-0.5

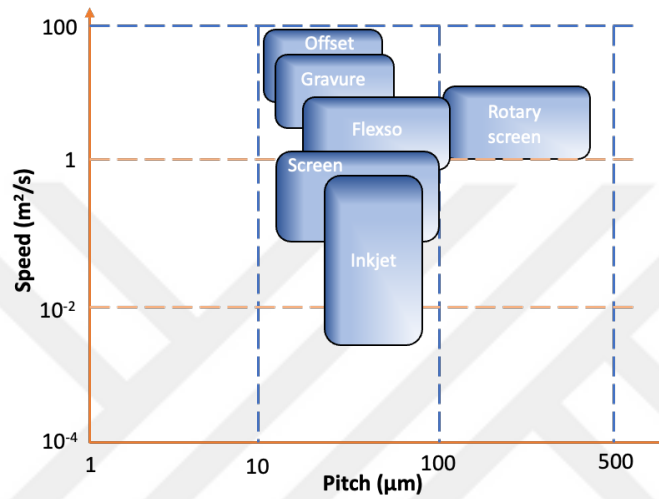


Figure 2.12 : Comparison of speed vs. fine pitch for different printing technologies. This was adapted from reference (Suganuma, 2014).

2.3.2.1 Viscosity

The viscosity must be sufficiently low to refill the ink reservoir in approximately 100ms and expel a drop from the nozzle by the transient pressure pulse. To avoid undesired dripping from the nozzle, the surface tension must be relatively high, but to make the ejected droplet able to break away from the nozzle, it must be sufficiently low. It's not mandatory that the properties of inkjet inks be restricted within the recommended range. They commonly have a viscosity range of 2-20 mPa·s and a surface tension of 25-50 mN/m (Cummins et al., 2012; Izdebska & Thomas, 2015).

One frequently known occurrence following solvent evaporation from a droplet is the creation of a circular ring pattern (coffee ring), which includes the majority of the solutes. This "coffee ring" is unacceptable because it has the potential to degrade the performance of inkjet-printed devices. When the solvent evaporates, the surface of the formed droplet is subjected to a much quicker evaporative loss, thus creating a "coffee ring" structure (Eshkalak et al., 2017). Pattern surface morphology is strongly affected

by coffee ring deposits on the substrate. That influences pattern resolution and device results. The viscosity of the ink is lower in inkjet printing, allowing for such an impact (Q. Huang & Zhu, 2019).

Most often, various co-solvents are inserted to alter the viscosity. In this research, adding ethylene glycol (EG) (16.1 mPa.s) helped to improve the viscosity of the solution to a better value.

2.3.2.2 Surface tension

Printing quality is highly affected by the interaction between the ink and the substrate, both during and after printing (Magdassi, 2009). In actuality, surface tension is a feature of the liquid's interface with another medium, rather than a property of the liquid alone. This interface refers to the ink-air interface in the case of drop formation (Reis & Derby, 2000).

Due to the fact that surface tension tends to pinch off the droplet tail and reduces the ink droplet towards a spherical shape, surface tension will have a considerable impact on the creation and development of the ink droplet (He et al., 2017). High surface tension is required to keep the nozzle from dripping, but low enough to enable proper jetting (Reis & Derby, 2000). In order to get a proper wetting of the substrate, the substrate's surface energy must be greater than the surface tension of the jetted fluid (Magdassi, 2009).

2.3.2.3 Surface energy

The situation of “wetting” can be described as the spread of a liquid throughout a substrate. In the performance of a print, the substrate plays a significant role. It is critical to distribute the functional ink throughout the substrate. A material with a low surface energy can produce randomly distributed clumps and agglomerates. Functional material placed onto a substrate with high surface energy typically ends up at the boundary of the drying print (Beedasy & Smith, 2020).

A liquid's surface energy can be easily obtained by evaluating the angle created by a droplet of the liquid with the substrate of interest, as illustrated in Figure 2.13. Each of the three interfaces, liquid-gas (LG), solid-gas (SG), and solid-liquid (SL), (Beedasy & Smith, 2020) affects the shape of the droplet based on the interfacial tensions γ_{xy} (Beedasy & Smith, 2020).

2.3.2.4 Contact angle

The wettability of a substrate surface is influenced by the chemical composition and micro geometric arrangement of the substrate. The physical parameter that defines the wettability of the substrate is the contact angle of a droplet. The contact angle of the substrates will be decreased if the wettability is increased. When the wettability is higher, the substrates' contact angle is lower. The adhesion of ink drops is also greatly affected by wettability, contact angle, and surface energy (Karim et al., 2017).

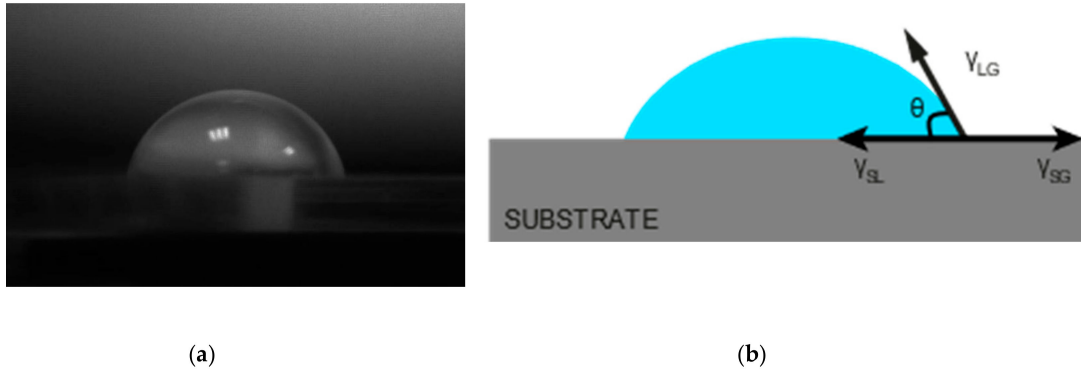


Figure 2.13 : (a) Image of a sessile drop in equilibrium on a polymer substrate. (b) Illustration of the forces occurring on a droplet on a substrate. The substrate is partially wetted because the contact angle, θ , is less than 90 degrees (Beedasy & Smith, 2020).

In order to calculate the equilibrium, contact angle, θ , the mentioned interfacial tensions in the static Young's equation must be analyzed.

$$\cos(\theta) = \frac{\gamma_{SG} - \gamma_{SL}}{\gamma_{LG}} \quad (2.5)$$

Once interfacial tensions are known, it is possible to identify the wetting level of the fluid. If the surface of the solid substrate is hydrophilic, the thermodynamic system will be in complete equilibrium, leading to complete wetting of the substrate (Figure 2.14a). In the situation where $\gamma_{SG} < \gamma_{SL} + \gamma_{SL}$, a droplet will spread across the surface, which will result in wetting to a limited degree (Figure 2.14c). On the other hand, if $\gamma_{SG} > \gamma_{SL} + \gamma_{SL}$, a droplet creates a spherical cap that lies on the surface, resulting in minimal wetting (Figure 2.14e), related to a hydrophobic surface (Beedasy & Smith, 2020).

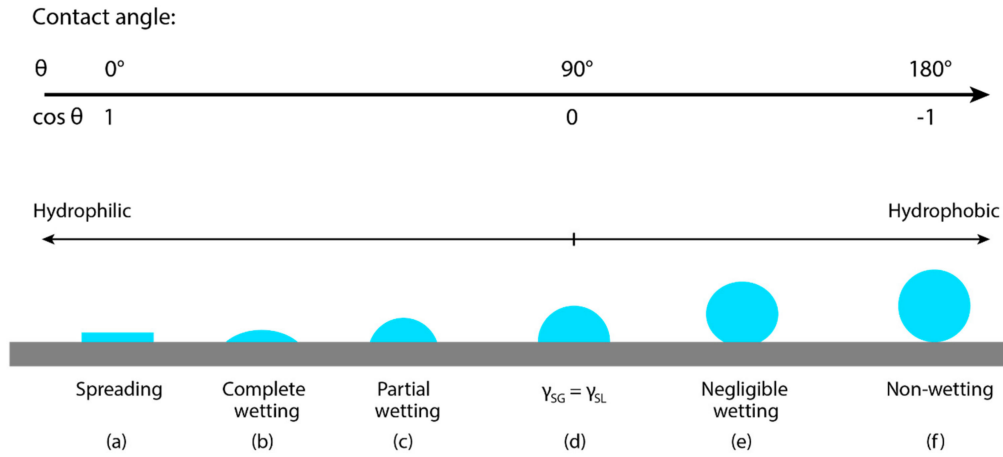


Figure 2.14 : Illustration of the potential wetting situations of a droplet as it deposits on a surface (Beedasy & Smith, 2020).

Materials that are hydrophobic have a contact angle of between $90 - 150^\circ$. As the contact angle is higher than 150 degrees, surfaces become super hydrophobic (Nosonovsky & Bhushan, 2009).

2.3.2.5 Particle size

Additional factors influencing print results include particle size and ink distribution. The optimum particle size varies for each print head, but is generally less than $1 \mu\text{m}$ and ideally less than 200 nm . Printability issues may arise when the particle size is in the order of $1 \mu\text{m}$ (Weng et al., 2010).

There are a variety of print heads with diverse nozzle diameters for sale. Smaller drop volume and smaller minimum possible dimension mean the smallest possible diameter (resolution). However, for certain inks, nozzle diameters of a few microns could cause clogging (Fromm, 1984).

2.3.2.6 Z dimensionless number

The dimensionless Reynolds (Re), Weber (We) and Ohnesorge (Oh) numbers provide an evaluation of the “jettability” of the material using fluid characteristics. The Reynolds number is defined as the ratio of inertial forces to viscous forces.

$$Re = \frac{\rho VL}{\eta} \quad (2.6)$$

Weber described it as the ratio of inertial forces to surface tension forces and calculated as following:

$$We = \frac{\rho V^2 L}{\sigma} \quad (2.7)$$

The Ohnesorge number is the ratio of viscous forces to surface tension and inertial forces that characterize droplet formation:

$$Oh = \frac{\sqrt{We}}{Re} = \frac{\eta}{\sqrt{\rho L \sigma}} \quad (2.8)$$

The density, viscosity, surface tension, nozzle diameter, and droplet velocity are represented by ρ , η , σ , L and V , respectively. To define the capability of inks for drop formation, Fromm was one of the first to employ dimensionless numbers. According to Fromm, stable droplets can only be formed if the reciprocal of the Ohnesorge number, Z , is bigger than 2. Reis and Derby modified this by demonstrating that a Z value of $1 < Z < 10$ is adequate for stable droplet production using numerical simulations.

2.3.2.7 Solidification

The solvent evaporates after the ink is spread on the substrate, and the ink solidifies during the subsequent annealing process. The degree of volatility, as well as the vapor pressure of the solvent, viscosity, and surfactants used in the ink composition, affect ink solidification (Karim et al., 2017).

A drying condition is important in this process. A lot of print methods, such as inkjet, use low concentration of functional materials in inks that contain a lot of solvent. Due to the high concentration of solvents, it is necessary to evaporate the solvent in order to obtain suitable solid tracks (Suganuma, 2014).

2.3.2.8 Surface roughness of substrates

The interlacing of warp and weft yarns, which causes ridges and holes in the structure, typically makes the structure of a woven fabric rough (Krykpayev et al., 2017). The adhesion factor between the substrate and the deposited layer is highly influenced by the substrate surface roughness (Wan et al., 2013). A thin coating on the surfaces is required to prevent rough impact (Karim et al., 2017). To maintain continuous

conductivity, consideration must be given to lowering the roughness of the substrates. For instance, to enable subsequent inkjet-printed conductive layers, a polyurethane-based interface paste was applied directly to the woven fabrics (Whittow et al., 2014).

2.4 CNT and PEDOT:PSS Based Conductive Ink Formulations Used for Printing

The primary focus of this section is a review of temperature-sensitive inks. Particular emphasis is placed on CNT and PEDOT:PSS ink formulations, which are target-sensitive materials that are used in this research in order to develop sensors. This section discusses the most critical properties of functional materials, as well as dispersibility, and the major processes involved, including ultrasonication, covalent and non-covalent functionalization, and their effect on the final product's quality.

Improvements in the field of printed electronics are attributable to functional, printable inks, whose success started with inkjet printing. Inks are classified according to the characteristics of materials with dimensions ranging from nanometers to micrometers. There are three kinds of dimensions. There are zero-dimensional materials (e.g., nanoparticles), one-dimensional materials (e.g., nanowires, nanotubes), and two-dimensional materials (e.g., graphene) (Tiwari et al., 2012).

Using functional materials in inkjet printing necessitates ink formulations that are compatible for deposition. In order to reduce the overall cost and reduce the amount of time and equipment necessary to produce printed conductive traces, several ink formulations were employed, including nanomaterial inks and conducting organic inks (Shen et al., 2014). The following materials were inkjet-printed onto various fabrics to create flexible wearable electronics: Ag nanoparticles, carbon nanotubes, graphene, and PEDOT:PSS (Kao et al., 2020; Karim et al., 2019; Stempien et al., 2016).

When utilizing conductive nanomaterials as the functional component of conductive inks, numerous challenges need to be overcome. One of the important challenges is to ensure that nanomaterials in the ink remain stable against aggregation and precipitation so that consistently reliable performance can be obtained. Therefore, an additive, such as a polymeric or surfactant, is needed to promote stability. However, the presence of the stabilizer may lead to significant problems such as lowering the conductivity (Kamyshny et al., 2011; Magdassi et al., 2010).

Numerous ink formulations exist for conductive printing. As a result of the high aspect ratio of CNTs, van der Waals forces lead them to adhere together, resulting in the formation of large bundles (Cummins et al., 2012; L. Hu et al., 2010). It is conceivable that such large bundles would create the risk of the printhead nozzles clogging. In printed electronics, conductivity level of the printed patterns is very important. Nevertheless, it has been claimed that when CNTs are aggregated, electric current flows solely via the bundle's outermost tubes, with the interior tubes contributing little to the current (L. Hu et al., 2010; Stahl et al., 2000). One of the main challenges when creating conductive inks is preparing dispersions of separated CNTs that are chemically and mechanically stable, and that do not demonstrate a change in chirality, which is critical for high electrical conductivity (Azoubel & Magdassi, 2010; Heller et al., 2005; Lu et al., 1996).

PEDOT:PSS is one of the most frequently utilized polymer-based inks. Because of its optical transparency, chemical stability, and structural elasticity, the optimal formulation of PEDOT:PSS ink has many advantages over other inks. In terms of electrical performance, the PEDOT:PSS pattern is comparable and reliable after an adequate number of layers have been printed; PEDOT:PSS printed pattern is currently being used in solar cells and energy storage devices. Biocompatible and biodegradable are the most essential features of PEDOT:PSS. It has been employed in lab-on-a-chip and organ-on-a-chip applications, as well as in biodegradable stretchable devices (Beedasy & Smith, 2020).

2.4.1 Functional materials

2.4.1.1 Carbon nanotube based inks

CNTs are cylindrical hollow nanostructures whose walls are made up of one-atom-thick sheets of carbon with the graphene structure (Kamysny & Magdassi, 2014). CNTs have electron-transport mechanisms consistent with π -conjugated structures, but their diameter determines whether they are metallic or semiconducting (Dresselhaus & Avouris, 2001; Schönenberger, 2006). Single-walled nanotubes (SWNT) are formed by rolling graphene and can be utilized as one-dimensional conductors (Gracia-Espino et al., 2010). Multiwalled nanotubes (MWNT) are made up of concentric tubes within tubes that can have metallic or semiconducting properties (Rice et al., 2007). Because of the average current density through the

characteristically different walls, these electrical hybrid tubes are supposed to behave like metallic nanowires. Furthermore, within the MWNT, wall-to-wall conduction is achievable, enabling charge transport to be dominated by the metallic MWNT tubes (Bachtold et al., 2000; Schoenenberger et al., 1999).

Because of their particular potential applications, carbon nanotubes (CNTs) have been widely researched (Zaporotskova et al., 2017). Nevertheless, their insolubility in both water and organic solvents limits their usage in practical applications. CNTs in organic (Ausman et al., 2000) and aqueous media (O'Connell et al., 2001) aggregate due to strong van der Waals interactions between CNT walls, and these interactions affect the efficiency of these materials (Girifalco et al., 2000). Pristine nanotubes come together to form bundles of hundreds of close-packed carbon nanotubes. CNTs are challenging to produce stable aqueous dispersions, and their insolubility has been a limiting factor in their potential implementation.

2.4.1.2 Conductive PEDOT:PSS polymer-based inks

These polymers have been widely used in sensing applications as their electrical or electrochemical properties can be sensitized to specific environmental or chemical factors by using an appropriate conducting polymer backbone or molecular dopant. Poly-(3,4-ethylene dioxythiophene) (PEDOT), Polypyrrole and Polyaniline are common conductive polymers that have been widely used as polymeric conductive ink for the inkjet printing process. Their electrical conductivity is typically lower than metallic inks (Cummins et al., 2012).

The easy hopping of electrons within and between polymer chains causes charge conduction in conductive polymers. In actuality, conductivity is the result of a complex combination of several parameters. The polymers have a conjugated backbone (Figure 2.15), which is created by a series of single and double bonds that alternate. A chemically strong, localized σ -bond exists in both single and double bonds, whereas a relatively less strongly localized π -bond exists in double bonds. The p-orbitals overlap each other in a series of π -bonds, making it easier to delocalize the electrons and to move freely amongst the atoms. The dopant is also crucial for providing these polymers' conductivity. The charge carriers (polarons and bipolarons) are introduced into the polymer during the doping process, making it conductive. It can occur in two forms, parallel to semiconductor technology. P-doping involves oxidizing the polymer

and giving it a positive charge, whereas n-doping involves reducing the polymer and giving it a negative charge (Balint et al., 2014).

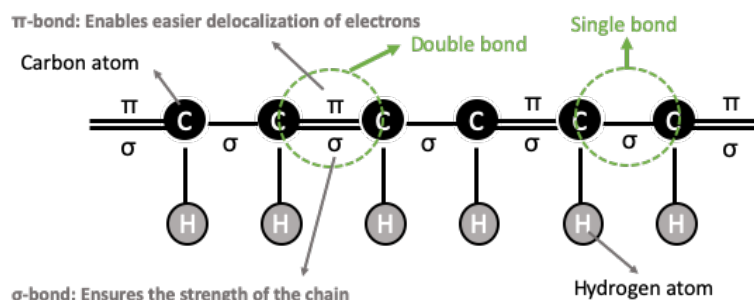


Figure 2.15 : A schematic of a conjugated backbone in simplified form. Adapted from (Balint et al., 2014).

PEDOT was thought to be one of the most durable conducting polymers. Because of its planar configuration and delocalized π electrons, PEDOT has high electrochemical stability in its oxidized state (Balint et al., 2014). PEDOT has higher conductivity and thermal stability than Polypyrrole. While PEDOT is a conductive polymer, its low solubility limits its use. PEDOT itself is very poor in processability since it is highly water-insoluble. Polystyrenesulfonate (PSS) as counterion is used to accomplish a stable dispersion in a water solution (Moulé et al., 2015). PEDOT:PSS is composed of conjugated poly(3,4-ethylenedioxythiophene) and PSS to counterbalance the charge of the charge carriers, as demonstrated in Figure 2.16a. The cationic PEDOT segments are dispersed in the aqueous medium with the help of PSS. The ratio of the two components has a significant impact on the conductivity of the resultant film produced from PEDOT:PSS aqueous solution. The use of polar solvents such as dimethyl sulfoxide, sorbitol, diethylene glycol, and N-methylpyrrolidone as "secondary dopants" increased the conductivity of PEDOT:PSS films by 2 to 3 orders of magnitude (J. Huang et al., 2005; J. Y. Kim et al., 2002; Ouyang et al., 2004, 2005). Primary dopants neutralize additional charges for conjugated polymers generated during the doping process by oxidation or reduction. As a consequence of the interaction between the dopants and the polymers, secondary dopants are mostly regarded as expanding polymer chains and therefore increasing their conductivity (Chiolerio et al., 2015; Kar, 2013). This procedure, according to experts, creates morphological alterations in the film during drying, which promotes the phase separation of PEDOT and PSS (Greczynski et al., 1999). This alteration results in a

better PEDOT conducting structure and, as a consequence, improved electrical conductivity.

PEDOT:PSS exhibits a core-shell structure in the aqueous medium (as well as the as-prepared film) (Figure 2.16b), with the core being PEDOT-rich and the shell being PSS-rich (Tseghai et al., 2020).

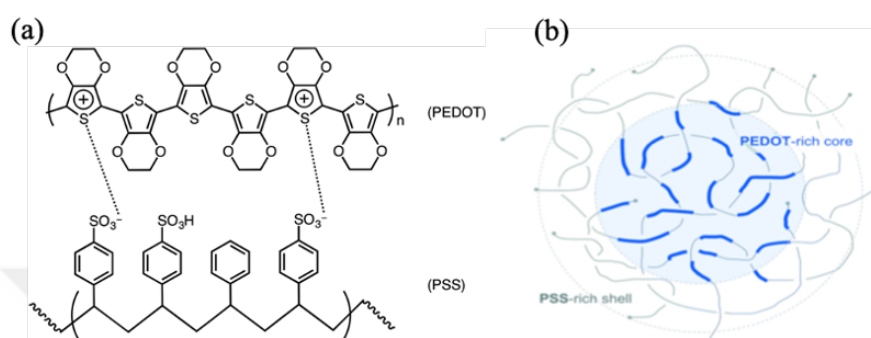


Figure 2.16 : (a) Chemical structure of PEDOT:PSS (Guo et al., 2016). (b) Core-shell structure of PEDOT:PSS (Tseghai et al., 2020).

Commercially available PEDOT: PSS can not be used directly as a conductive ink because of its low viscosity, solubility, and poor wettability, which would cause clogging of the print head and inconsistency of the ink droplet. The solution's viscosity and surface tension need to be adjusted by additives. PEDOT: PSS with additives prevents the formation of a coffee-stain effect and helps the improvement of electrical conductivity (L. Fan et al., 2014). Unlike metals, conducting polymers do not generate skin irritation or long-term toxicity when in direct touch. No cytotoxicity has been observed with PEDOT:PSS (Miriani et al., 2008).

2.4.1.3 Conductive CNT/PEDOT:PSS composite-based inks

The majority of conductive polymer/CNT composite sensor applications have centered on polyaniline, polypyrrole, polythiophene, and poly(3,4-ethylenedioxythiophene) (PEDOT) and the characterization of their synergistic electronic and sensing properties. With the conductive polymer, CNTs can produce binding sites. Covalent bonds can form when dispersed conductive polymer and carboxylated CNTs interact in a solvent, resulting in polymer-functionalized CNTs and CNT doped polymers (Baibarac et al., 2003; Baibarac & Gómez-Romero, 2006).

2.4.2 Dispersion of carbon nanotubes

The dispersion of CNT materials is critical for maintaining the nanotubes' electronic properties. While the CNTs are hydrophobic, mechanical and chemical methods for dispersing nanotubes are used for dispersing the CNTs. Some studies using carbon nanotube ink for inkjet printing are listed in Table 2.6. It contains information about the printer type, ink content, dispersant type, applied process, and electrical properties of the printed surface.

Table 2.6 : Comprehensive comparison of recently reported carbon nanotube inkjet printing and formulation details.

Ref. No.	Printer	Solvent	Dispersant / Concentration	CNT/ Concentration	Preparation	Sheet Resistance
(Kordás et al., 2006)	Canon	Water	Functionalized	MWCNT-COOH (0.26 mg/mL)	Sonication Stirring Centrifuge	40 kΩ/sq. (90 prints)
(Mustonen et al., 2008)	Dimatix	Water	Functionalized	SWCNT-COOH (0.1 mg/mL)	Sonication Centrifuge	Not reported
(Li Yang et al., 2009)	Dimatix	DMF	n/a	SWCNT (0.4 mg/mL)	Sonication	150 Ω/sq. (25 prints)
(Song et al., 2008)	MicroJet	DMF	n/a	SWCNT(0.02 mg/mL)	Centrifuge	333 Ω/sq. (8 prints)
(Matsuzaki et al., 2012)	MicroJet	DMF	n/a	SWCNT (0.001 µg/ml or 0.04 µg/mL)	Sonication Filtering	Not reported
(Okimoto et al., 2010)	MicroJet	DMF	n/a	SWCNT (0.001 µg/ml or 0.04 µg/mL)	Sonication Centrifuge Filtering Sonication	Not reported
(In Het Panhuis et al., 2007)	HP	Water	xanthan gum	SWCNT or MWCNT	Sonication	Not reported
(P. Chen et al., 2010)	Epson	Water	SDS (10 mg/ml)	SWCNT (0.2 mg/mL)	Sonication Centrifuge	78 Ω/sq. (200 prints)
(Kwon et al., 2013)	Epson	Water	3 different dispersants 150 mg/ml	MWCNT 0.15 mg/mL	Mixing Ball-milling Centrifuge	760 Ω/sq. (12 prints)
(Qin et al., 2016)	Meyer burger	Water	Nonionic surfactant	SWCNT-COOH (2 mg/ml)	Filtering Sonication	1 kΩ (200 print)
(da Costa et al., 2015)	HP	Water	SDS(7 mg/ml)	MWCNT 10mg/mL	Sonication Centrifuge	1 Ω/sq. (33 print)
(Michelis et al., 2015)	Dimatix	Dichlorobenzene	SDBS (3 mg/ml)	MWCNT 0.2mg/mL	Sonication Centrifuge	87 kΩ/sq. (30 print)

2.4.2.1 Ultrasonication

One method for dispersing CNTs is to use organic solvents without dispersing agents. Ultrasonication and high shear mixing are used in the mechanical alternative. These procedures take considerable time and are inefficient. They can generate CNT fragmentation and, as a result, a decrease in their aspect ratio, as well as poor dispersion stability (Lu et al., 1996). The fundamental problem with organic solvents that do not contain dispersing agents is the difficulty of achieving dispersions with high CNT concentrations, higher than 0.1 g/L (Kamyshtny & Magdassi, 2014).

In general, the sonication method is used to disperse nanomaterials in an aqueous medium. The mechanical energy provided throughout the sonication process overcomes the van der Waals interactions in the CNT bundles and leads to the generation of CNT "exfoliation," whereas adsorption of the surfactant molecules occurs on the surface of the CNTs walls (Strano et al., 2015).

Ultrasonication process can be combined with a non-covalent approach. Short CNTs are dispersed by tip-sonication using SDS at a weight ratio of 1:2 CNTs: SDS, whereas a weight ratio of 1:7.5 CNTs: surfactant is required for exfoliation of long CNTs (Tkalya et al., 2012).

2.4.2.2 Non-covalent approach

One of the methods for dispersing CNTs is to use an aqueous medium with dispersants such as surfactants (anionic, cationic, and nonionic) or polymers. Non-covalent functionalization is desirable since the π -electron cloud of the CNT's graphene sheet is not affected, maintaining the CNT's characteristics. The tube surface can be modified by adsorption or wrapping of surfactants, polymers, or biomolecules to exfoliate the CNT bundles by van der Waals forces and π - π interactions. This method can also reduce the amount of time and expense needed to manufacture CNT reinforced nanocomposite materials as well as homogeneous CNT dispersed solutions for a variety of applications (Calvaresi et al., 2012). Various forms of amphiphilic compounds, surfactants such as sodium dodecyl sulfate (SDS) (Duan et al., 2011), sodium dodecyl benzenesulfonate (SDBS) (K. Yang et al., 2013), polymers (Nish et al., 2007; Sinani et al., 2005), and ionic liquids (Fatemi & Foroutan, 2015a) have all been utilized to disperse and stabilize CNTs in aqueous medium. The conditions of adsorption of a given surfactant on a CNT with a certain length, diameter, and chirality

that may have an impact on the interaction of the CNTs, define the arrangement and order of surfactant molecules around the CNTs (Ortiz-Acevedo et al., 2005). Surfactant adsorption is critical in the standard procedure for producing a high weight fraction and exclusively dispersing CNTs (M. F. Islam et al., 2003; Vaisman et al., 2006). The columbic force has no significant effect on the adsorption of an anionic surfactant on the surface of CNTs, according to experiments, but the hydrophobic interaction between the tail of the surfactant and the wall of CNTs has a major effect on stabilizing the structure of the CNT in an aqueous medium (Matarredona et al., 2003). According to recent molecular dynamics simulations, the molecular structure of surfactants has a significant impact on surfactant packing on nanotubes, decreasing the efficiency of nanotube–nanotube interaction. This is qualitatively consistent with experimental studies (Suttipong et al., 2011). Rastogi et al. (Rastogi et al., 2008) used UV–Vis spectroscopy and transmission electron microscopy to analyze the dispersion of multi-walled CNTs with four surfactants: Triton X-100, Tween 20, Tween 80, and SDS. They discovered that while Triton X-100 generates the maximum dispersions, SDS provides the minimum dispersions. The dispersing power of the four surfactants in terms of percentage extractability includes the following general trend, according to their findings: $\text{SDS} < \text{Tween 20} < \text{Tween 80} < \text{Triton X-100}$. Surfactant molecules can be available in two or three states in a solution that includes CNT and surfactant: in micelles, adsorbed onto the CNT surface, and as individual molecules, based on the temperature and surfactant concentration. In the case of Triton X-100, it has been demonstrated that most surfactant molecules do not occur in the state of micelles at surfactant loadings below 0.5 %, while at high concentrations, the micellar volume fraction approaches the solution value asymptotically (Fatemi & Foroutan, 2015b). A diagram illustrates several surfactants assemblies structures on a CNT (Figure 2.17).

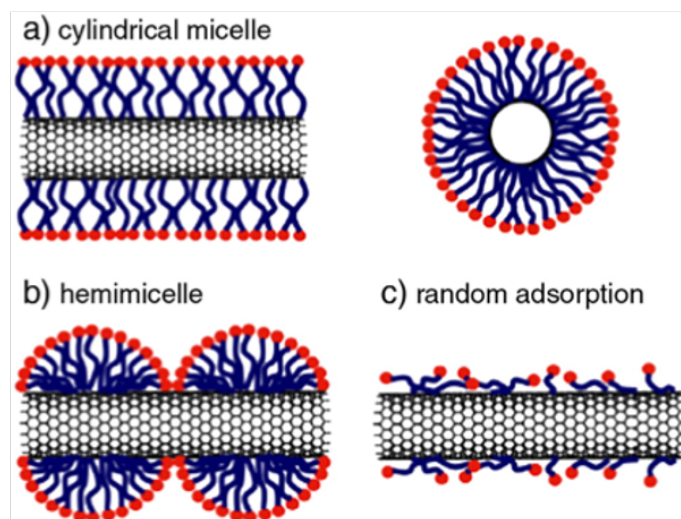


Figure 2.17 : Various surfactant assembly structures on a CNT are depicted schematically. (a) Cylindrical micelles, side and cross-section views, (b) hemimicelle, and (c) random adsorption (H. Wang, 2009).

Conductive polymeric based surfactants can also be used to decrease the contact resistivity between CNTs or graphene platelets. PEDOT/PSS is a conductive polymer that can be used to disperse CNTs in water. The minimum required PEDOT:PSS/CNT ratio is stated as 4:1 for stable dispersions and complete exfoliation. The percolation threshold is as low as 0.2 wt% of the loading of the CNTs. An ultimate conductivity of 500 S/m was achieved (Tkalya et al., 2012).

2.4.2.3 Covalent approach

Additionally, when needed, the CNTs can be chemically modified with functional groups, which enhance the probability of the CNTs' interaction with the dispersing medium (Kamyshny & Magdassi, 2014). The chemical modification of the surfaces of CNTs as covalent or non-covalent are general methods for dispersing CNTs in a solution (Grossiord et al., 2007; Zhao et al., 2004).

Chemical modification is commonly used to reduce CNT aggregation. To increase solubility in solvents, covalent methods include chemical modification (or chemical functionalization) of the CNT walls with different chemical moieties (Jiang et al., 2013). Carboxyl groups, for example, allow covalent coupling of molecules by forming amide or ester bonds. Long-chain aliphatic amines can be covalently or ionically attached to carboxyl groups to gain high solubility in organic solvents. The carboxyl groups decrease van der Waals interactions between the CNTs, enabling separation and chemical functionalization in aqueous or organic solutions (Fernando

et al., 2004; Trojanowicz, 2006; Ye & Sheu, 2006). This aggressive method can create defects in the CNT walls and alter the π -electron conjugation, leading to their unique properties deteriorating. The covalent and non-covalent approaches on CNT walls are depicted in Figure 2.18.

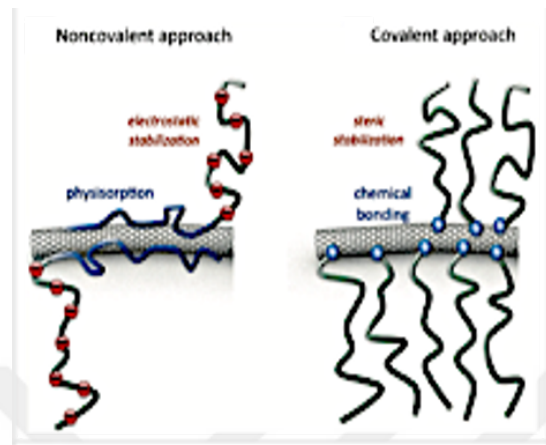


Figure 2.18 : Surface modification of CNTs (Richard et al., 2003).

2.5 Wearable E-Textile Based Solutions

Textile-based wearable devices can detect and respond to a wide range of conditions, including electrical, mechanical, chemical, magnetic, and thermal stimuli (Van Langenhove & Hertleer, 2004). Depending on the configuration and application, they perform a particular function in a variety of situations. Textile-based strain, humidity, pressure, and temperature sensors are examples of commonly occurring wearable sensing devices (Gonçalves et al., 2018). It's critical to manufacture sensors that are built on textiles with the use of conductive materials. This is accomplished by combining properties of textiles with expected sensor specifications.

Textile-based wearable electronics have the potential for medical applications, such as injury prevention, medical diagnosis, and therapy. As a result, high measurement precision is required and must take precedence over user experience design decisions (Bosowski et al., 2015; Leutheuser et al., 2017). The system criteria for a textile sensor differ depending on the application. The majority of textile-based temperature sensor solutions are focused on converting the operation techniques of traditional sensors to smart textile systems. At present, a broad range of methodologies for developing a textile-based temperature sensor have been studied. Fiber processing, coating, weaving, knitting, embroidery, and printing techniques have all gained considerable

interest. The reported temperature sensors are configured as thermocouples, thermistors, and resistive-type sensors depending on their working principle (Bosowski et al., 2015).

The temperature of a human body is an important indicator of a patient's health. The average human body temperature is between 37 and 37.5 degrees Celsius. When a person feels sick, whether it is a minor or serious illness, it can be stressful to be in a hospital. Thus, it is suggested that every physiological parameter be measured as quickly as possible (Nag & Mukhopadhyay, 2015). If the patient's condition isn't life-threatening, this assessment can be performed remotely, at the patient's comfort home. Thus, patients and industry will benefit from the application of healthcare devices, as it will minimize hospital bills, waiting times, and congestion problems (Mansor et al., 2013). A variety of wearable sensors have been developed for diverse applications with the progress of sensing technologies. For instance, for someone with a cardiovascular disease, real-time measurement of physiological signals during exercise and sports is useful with remote sensing (X. Wang et al., 2017). Another example is a temperature monitoring belt made of conductive textile wires rather than hard wires, which helps to improve the newborn infant's comfort (W. Chen et al., 2010). Moreover, continuous body temperature monitoring with wearable devices can be used in the field of physiology research and chronobiological study to mirror emotional alterations throughout the menstrual cycle for females, which can then be used to predict ovulation times (G. S. Kelly, 2007). These flexible wearable sensors could be a popular technology used in upcoming healthcare monitoring devices for remote medicine, according to the researchers (X. Wang et al., 2017).

Direct contact with the human body is desired for high-precision temperature measurements. Traditional rigid temperature detectors, on the other hand, are inadequate to obtain conformal contact with curved surfaces. This problem is vital to the success of wearable and flexible temperature sensors (Descent et al., 2018).

This section describes the general outlook for wearable technologies underlying textile-based sensors, their characteristics, and the factors affecting their fabrication.

2.5.1 Textile-based sensor

A sensor is a device that detects a particular target stimulus and transforms it into an electronic signal. As a result, sensors serve as a link between the physical world and

electrical devices. (G. M. N. Islam et al., 2020; Rida et al., 2010). Textile-based capacitive, resistive, and optical sensors are the most common wearable e-textiles that enable the textile to detect touch, strain, pressure, temperature, and humidity (Gonçalves et al., 2018). In accordance with the production process and material, a textile sensor can be defined on four levels of the textile structure, including fibrous, linear, planar, and spatial, which need to be accurately chosen based on the form of the textile (Gehrke et al., 2019). Functional fibers have sparked interest in the development of smart textiles with sensing, deformability, and biometric capabilities. Numerous techniques can be used to produce woven and knitted cotton fabric-based sensors (G. M. N. Islam et al., 2020). To expedite and provide a more practical means of making conductive textiles, using conductive inks to treat the polymers, fibers, yarns, fabrics, or ready-made garments at the fabrication stage is a promising option (Qu & Skorobogatiy, 2015). Coating, printing, pad-drying, in-situ polymerization, and dipping are some of the scalable fabrication techniques.

2.5.2 Attributes of the wearable textile-based sensors

Textiles can be produced in a variety of shapes and sizes, including required length, width, and thickness, enabling the development of "changeable" surface areas for "hosting" different numbers of devices for the specified function. Wearable textile devices are apparently the most valid human computer interfaces that people deserve, use, know and they can easily be adaptable (S. Park & Jayaraman, 2021). While integrating bulky silicon or inorganic electronics into textiles primarily seen at the early stages of research, placing bulky devices in textiles may lead to uncomfortable fitness or even an increase in the weight of textiles. Wearable sensor devices and systems utilizing fiber or textile-based materials have been employed to handle these difficulties (Heo et al., 2018). Furthermore, as compared to conventional printed circuit boards, textile-based devices are comparatively cost-effective to manufacture (S. Park & Jayaraman, 2021). The main attributes of textile-based sensors are shown in Figure 2.19.

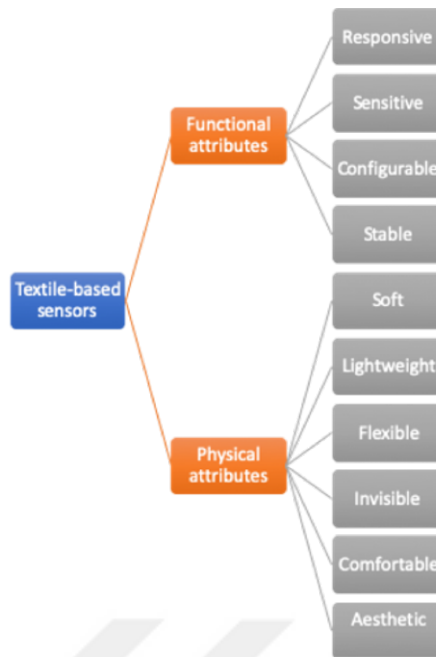


Figure 2.19 : Main attributes of textile-based sensors.

The responsiveness of the wearable is crucial, particularly when it is being used for actual data acquisition and monitoring. Textiles offer high flexibility due to the wide variety of fibers, yarns, fabrics, and production processes (such as weaving, knitting, and printing) that can be used to produce items with planned performance requirements for specific end-use applications. They are unique in their ability to combine strength and flexibility into one form. As a result, when bent, textiles adapt to the required shape while maintaining their strength. Moreover, their flexibility, and lightweight properties enable them to resist a variety of operational (stress/strain) and harsh environmental (biohazards and climatic) conditions (S. Park & Jayaraman, 2021).

A wearable device must be shape-conformable to fulfill the intended end use; in other words, it must react similarly to human skin. Aesthetic is also essential in deciding whether or not a device or technology is accepted and utilized. It is especially significant since other people can see the device. As a consequence, if the wearable device will be noticeable to others, it should be aesthetically pleasurable and, preferably, make a fashion statement when performing its function (S. Park & Jayaraman, 2021).

As a consequence, textiles are an excellent platform for integrating wearable devices to utilize situational awareness data while maintaining aesthetic and comfort properties, as well as many other textile-specific attributes (S. Park & Jayaraman, 2021).

2.5.3 Factors affecting the production of textile sensors

The main feature of textile-based wearable sensors is stretchability, which allows the sensor to stretch, flex, or bend without causing damage to the sensor during body movements (G. M. N. Islam et al., 2020). The improved sensor flexibility opens up additional possibilities for future applications, such as incorporation into various types of textiles.

Flexible electronic devices, unlike traditional rigid, 2D electronic devices, can conform to the shape of the human body. As a result, mechanically flexible substrates are necessary for the production of electronic components and circuits (Heremans et al., 2016). Owing to their thermal stability, chemical resistance, and excellent mechanical flexibility, flexible polymers, rubbers, and metal foils are commonly employed as substrates (X. Wang et al., 2017). Textile substrates enable sensors and other devices to be built into or embedded in a fabric-based network (Zeng et al., 2014). The thin, lightweight, soft, flexible, robust, deformable, breathable, and washable (Norstebo, 2003) structure of textiles enables them to be used as substrates for wearable temperature sensors. In general, owing to the extensibility properties and more porous structure of knit fabric, researchers prefer woven fabrics instead of knit fabrics, based on the type of sensor. Woven fabrics with a lower porosity are optimal for controlling ink throughout inkjet printing. Woven fabrics are also more dimensionally stable than knit fabrics. (Krykpayev et al., 2017).

The resistance of a flexible temperature sensor may be affected by human body-induced strain/ stress that would cause a decrease its reliability (C. Wang et al., 2019). One of the solutions to achieve this is the usage of highly deformable thermoplastic polymers, such as polyethylene terephthalate (PET), and polyurethane (PU), and silicone elastomers (PDMS, EcoFlex) (Kenry et al., 2016; Salvatore et al., 2017; Shih et al., 2010; J. Yang et al., 2015). Kim et al. presented a flexible organic temperature sensor based on polyvinyl alcohol that showed remarkable stability for 50 bending cycles (S. W. Kim et al., 2019). Dinh et al. tested a CNT-based thermal flow sensor with different folding angles. The folding experiments exhibited a maximum resistance change of about 0.2%, suggesting the high stability of a CNT-based sensor (Dinh et al., 2016). Hilal et al. demonstrated a graphene/nickel-based fiber-shaped temperature sensor. The developed sensor retained 75% of its sensitivity after the bending tests (Hilal & Han, 2020). Rajan et al. investigated the flexibility and

mechanical resilience of their developed temperature sensor based on graphene-coated polypropylene textile fibers. They observed no significant change in resistance with multiple bends (Rajan et al., 2020). In another study, PEDOT:PSS printed cotton and Kapton-based flexible sensors displayed high stability, and no significant changes after bending up to 300 times (Y. Zhang & Cui, 2019).

Humidity stability is another challenge for temperature sensors. Flexible and wearable sensors are inevitably subjected to ambient humidity. Therefore, developing a flexible temperature sensor that is insensitive to humidity would be of considerable significance. Kim et al. demonstrated a flexible temperature sensor based on ion channels. The developed temperature sensor's performance remained constant in the air and water (J.-S. Kim et al., 2018). Wang et al. developed a crosslinked PEDOT:PSS-based flexible temperature sensor which exhibits very good stabilization between 30% RH and 80% RH (Y.-F. Wang et al., 2020). Liu et al. presented a skin-attachable polyethyleneimine/ reduced graphene oxide-based temperature sensor resistant to humidity (Q. Liu et al., 2019).

2.6 Sensor Design Aspects

In this section, the sensor characteristics that are relevant to wearable temperature measurement system are summarized.

2.6.1 Sensitivity

The sensitivity of a sensor is one of the most important sensor characteristics. The ratio between the electrical variation of the output and the non-electrical variation of the input is sensitivity. It depicts the relationship between the electrical signal generated by the system and the non-electrical signal received by the system. There is a direct correlation between sensor sensitivity and the output signal change as the measured quantity varies. High sensitivity is required for sensors that sense extremely minute changes (De Marcellis & Ferri, 2011; Rida et al., 2010).

2.6.2 Linearity

The great majority of sensor-output signals occur in the form of electric signals. Transfer functions represent the relationship between sensor signals and physical

variations. The transfer function is a mathematical model that describes how the sensor signal influences the physical variable. A linear or nonlinear transfer function may describe the system's behavior in a continuous manner. Equation 2.9 that describes a linear relationship can be expressed as follows:

$$y = bx \quad (2.9)$$

where y denotes the electric signal. The physical stimulus is denoted by x . a is the y -axis intercept, which indicates the output signal for a zero input. The slope, given as b , is also referred to as the sensitivity (Webster & Eren, 2017).

Sensors that are ideal have the property of being linear. Such a sensor's output signal varies linearly with the property's value. A quality that is critical to long performance is overall sensitivity, described as the ratio between the output signal and the measured characteristic. (Rida et al., 2010).

2.6.3 Stability

Stability is defined as the capability of a temperature measurement system to perform consistent and predictable measurements over time, both in terms of bias (mean) and precision (standard deviation) (Krishnan, 2015).

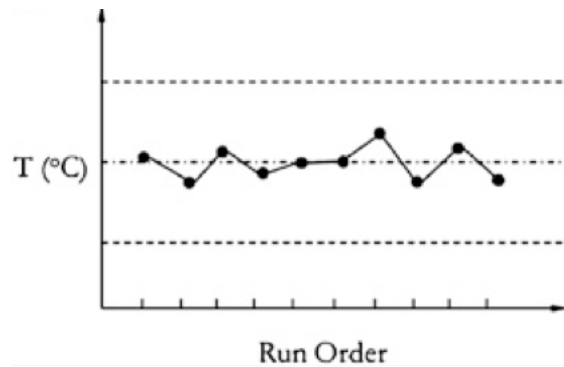


Figure 2.20 : Example chart of stability (Krishnan, 2015).

Due to the aging of sensor materials, the long-term stability may be attributed to a change in those properties (electrical, mechanical, chemical, or thermal). Drift is widely considered to have a one-way directional tendency. It generally occurs over an extended period of time, such as months and years. It is very vital for precision sensors to provide long-term stability (Figure 2.20). In particular, aging is only a problem for sensors with organic components. One good example of this is glass-coated metal-

oxide thermistors, which are even more stable over time as compared to epoxy-coated thermistors (Fraden, 1998).

2.6.4 Accuracy

The difference between the actual value and the average value of measurements is referred to as accuracy (Figure 2.21) (Krishnan, 2015). This is a reference to the measurement's uncertainty. An ideal sensor will adhere to the following guidelines: It is dependent on the thing being measured. It is not affected by any other property. It has no effect on the property being measured (Rida et al., 2010).

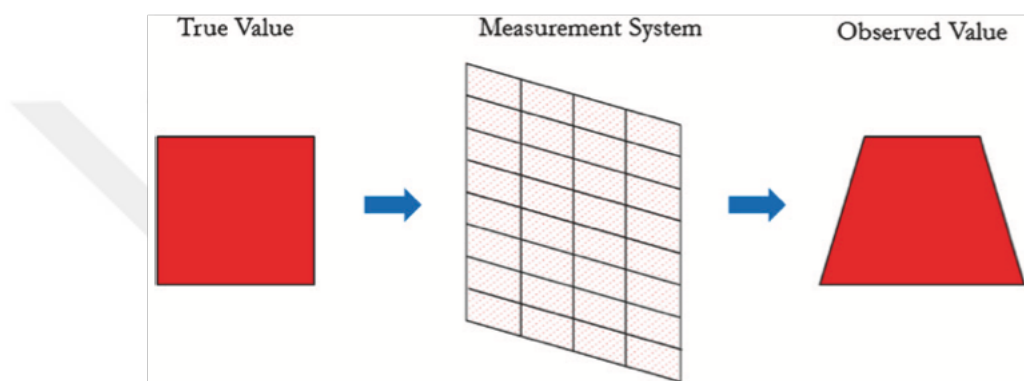


Figure 2.21 : A measurement system introduces a measurement error (Krishnan, 2015).

Precision (considered as the ability to replicate output signals with similar values) is closely related to accuracy. As a result, accurate measurement relies on the percentage error in relation to the ideal value (De Marcellis & Ferri, 2011).

2.6.5 Reliability

A sensor's reliability is defined as the capability of the sensor to meet performance requirements and operational constraints under stated conditions for a stated time period. It is defined in terms of probabilities, such as how likely it is that the device will not fail in a specified period of time or number of utilizations. Nevertheless, it is sometimes the situation that the sensor manufacturers take into account the requirement for reliability. It may be that, the unavailability of a generally established criterion for the term makes it so problematic to recognize (Fraden, 1998).

Designs of deformable structures (including buckling, noncoplanar, fractal, net-shaped, island-bridge, and serpentine designs) can prevent the influence of body motions on the performance of the temperature sensors while enhancing stretchability.

The priority is a high level of sensitivity. For instance, the use of a serpentine design increases sensitivity along the length direction of the sensor under a fixed strain due to the longer material path of the serpentine design in the direction of sensing with a minimum occupancy of the entire sensing region. The serpentine structure with lower length and width is preferred as more cycles and lower electrode width can enhance sensitivity (J. A. Fan et al., 2014; A. M. Hussain et al., 2015; Jason et al., 2016; D. Kim & Rogers, 2008; Someya et al., 2005; C. Wang et al., 2019; J. Wang et al., 2017; Y. Wu et al., 2017; Y. Zhang et al., 2016).

The washability is always a barrier in terms of implementation, which reduces textronic equipment's reliability. The wearable device can become unstable after several cycles of the washing process and, in some cases, start to fail (Tao et al., 2017). Rajan et al studied the washability of their textile-based sensor in a solution of laundry detergent and tap water at different temperatures by using a beaker and magnetic stirring bar. The developed sensor was found to be insensitive to washing (Rajan et al., 2020). Breathability is another significant issue for wearable electronics to maintain the comfort of the user. In research, it was revealed that the air permeability of the CNT printed fabric is similar to the primitive fabric that can fulfill the human skin's breathable requirement to long-term wrap comfortably around human skin with millions of pores (Cao et al., 2018; Zeng et al., 2014). The porosity design, low thickness and low modulus with extremely good substrate elongation make it possible for the wearer to be breathable when applied to human skin (Y. Y. Chen et al., 2015)

3. EXPERIMENTAL SET UP, SENSOR FABRICATION, CHARACTERIZATION AND TEST METHODS

This chapter introduces a comprehensive picture of the experimental techniques and equipment that were employed in this thesis. The presentation of the equipment used in the production of the ink formulations and temperature sensors developed in this thesis is provided in this section of the chapter. Finally, this chapter provides a description of the concepts and principles covered in this thesis, including techniques used for characterization and methodology of ink materials and sensors. Figure 3.1 represents the basic process flow of the process. At first, ink solutions formulated are transferred to ink-jet cartridges and then they are printed on top of the fabric structure in order to obtain sensor structure. Finally, the fabricated sensors via inkjet printing technique are covered with welding tapes to increase their durability against environmental factors.

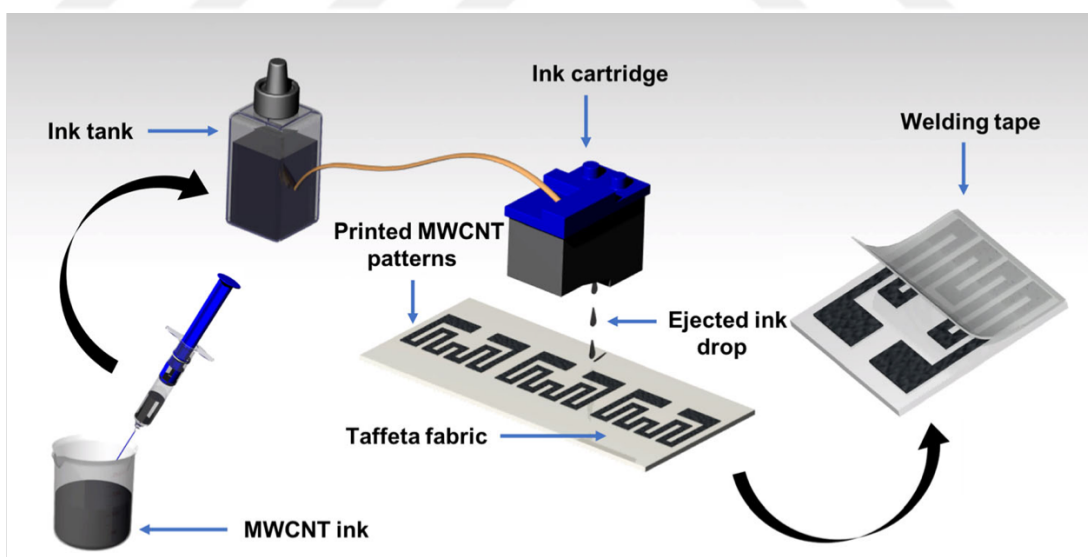


Figure 3.1 : The basic flowchart for the fabrication of textile-based printed temperature sensors.

3.1 Technique and Equipments for Ink Development

It is essential to utilize the proper materials when fabricating a flexible temperature sensor to meet the criteria for the electrical and mechanical properties that are required. Selection of suitable materials and formulations for ink solutions are essential, since suitable materials and a suitable fluid presence must be available for inkjet printing. In this thesis, inks containing CNT, PEDOT:PSS, and CNT/PEDOT:PSS composites were printed and then covered with a protective layer to create textile-based temperature sensors. Conductive inks developed in this thesis were fabricated using non-covalent functionalization, ultrasonication, centrifugation, and filtration techniques.

3.1.1 Non-covalent functionalization

The addition of a dispersant to a CNT-containing solution allows the solution to be dispersed via physical adsorption. After that, the solutions were sonicated with an ultrasonic sonicator.

3.1.2 Ultrasonication

The ultrasonication process is applied by soaking the ultrasonic horn directly into the nanomaterial solution or placing a beaker of nanomaterial solution in an ultrasonic bath. Cavitation breaking up nanofiller clusters in the liquid and exciting resonant vibrations of the clusters occurs all through ultrasonication. There are a variety of different factors to be considered to obtain optimum dispersion of the nanomaterial in the polymer. The first variable is the concentration of nanomaterial, followed by sonication bath, sonication tip, sonication times, surfactant type, and solvent type (F. Gao, 2012; Koo, 2019).

In this study, the sonication process was carried out with both an ultrasonic bath (60W, 40kHz) (Figure 3.2b) and a Hielscher UP400S sonicator (400W, 24kHz) (14 mm end cap diameter tip) (Figure 3.2a).

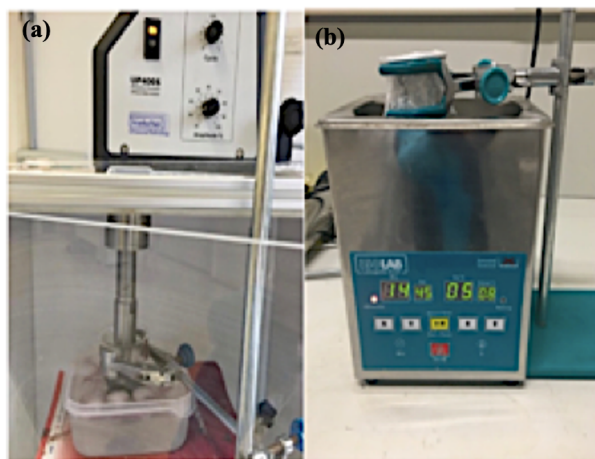


Figure 3.2 : (a) Ultrasonicator. (b) Ultrasonic bath.

3.1.3 Centrifugation

Centrifugation is a method in which centrifugal force is used to sediment mixtures (Nguyen et al., 2014). The pellet is defined as the precipitate while the solution remaining is known as the supernatant. The precipitate part is called the pellet, while the remaining solution is called the supernatant. (ACerS, 2010). When the density of the fluid equalizes with the density of the particles, the centrifuge is stopped. Performing further centrifugation does not yield results (Nguyen et al., 2014). Carbon nanotube supernatant left dark by a surfactant mixture was readily apparent with high dispersibility (ACerS, 2010).

Centrifugation to extract non-dispersed agglomerated tubes was performed using a 15 mL centrifugation tube and a centrifuge device (Elektromag M615) (Figure 3.3).



Figure 3.3 : Centrifuge device.

3.1.4 Filtration

After the centrifugation process, the supernatants were filtered using a syringe filter (Millex-FH 0.45 μm , $\varnothing$ 25 mm, PTFE) and transferred to a sample bottle to be stored for further analysis and applications.

A complete explanation of the materials and process parameters utilized in this research are given in Section 4.1

3.2 Sensor Fabrication Method and Equipments

This section discusses the techniques and equipments used in the fabrication and characterization of sensors. The steps including forming a conductive pattern, providing an interconnection, and applying a protective layer are briefly introduced.

3.2.1 Inkjet printing

The Canon Pixma G1400 and the Epson Stylus SX430W, piezo-based and low-cost inkjet printers with their properties exhibited in Table 3.1, were used to print temperature sensor's conductive patterns.

Table 3.1 : Printer properties of both the Canon Pixma and the Epson Stylus

Properties		
Printer name	Canon Pixma G1400	Epson Stylus SX430W
Print resolution	4800 x 1200 dpi	5760 x 1440 dpi
Minimum pitch	1/4800 inch	1/5760 inch
Max speed	Approx. 8.8 ipm	33 pages/min
Print technology	FINE Cartridges	DURABrite™ Ultra
Droplet size	2pL	3 pL
Simplicity	Difficulty in ink replacement	Easier ink replacement

3.2.2 Interconnection

In order to interconnect printed sensors, silver-plated polyamide conductive yarn and silver conductive paste were used in conjunction with each other.

3.2.3 Encapsulation

Welding tape was applied at 80°C for 20 seconds on fabric with 5.5 bar pressure using a textile heat-press machine (Megasistem) (Figure 3.4a) and a lamination machine (Mapilam 124) (Figure 3.4b).

A complete explanation of the fabrication processes is covered in more detail in Section 4.2.

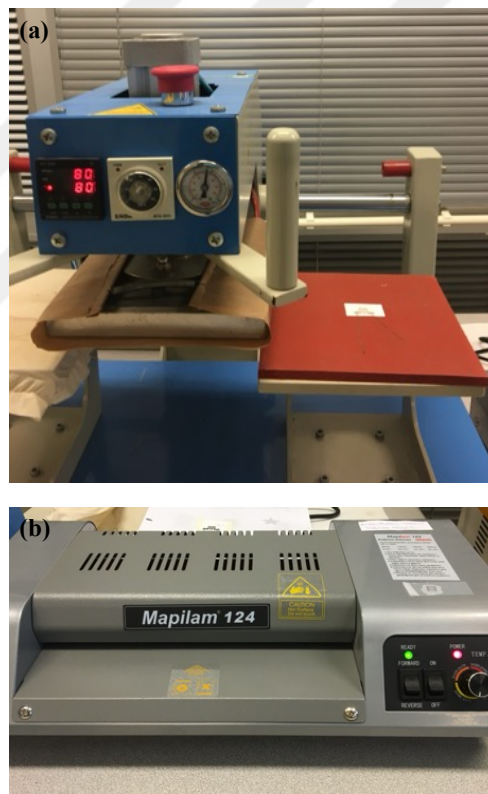


Figure 3.4 : (a) Heat-press machine, (b) Lamination machine.

3.3 Characterization and Test Methods for Ink Formulations, Printed Conductive Structures and Wearable Sensor

3.3.1 Characterization techniques for ink formulations

The properties of the prepared ink formulations have been analyzed in this subsection. The first objective was to discuss the dispersibility and stability of liquids that have

been formulated for ink application. Second, the print compatibility of the formulated dispersion was investigated.

3.3.1.1 UV-Vis spectroscopy

An Agilent 8453 spectrometer (Figure 3.5) was used to record UV-Vis absorption spectra which can range from 190 to 1100 nm.

Recognizing the characteristic properties of nanotube dispersion is difficult using traditional microscopy techniques, so quantitative analysis of dispersion is useful for understanding these properties more objectively and precisely in order to eliminate subjective evaluations. Dispersion of CNTs directly impacts suspension performance, so nano-ink production requires proper dispersion of CNTs (Njuguna et al., 2015).



Figure 3.5 : Agilent spectroscopy.

3.3.1.2 Viscometers

The viscosity of a fluid indicates the resistance to stress that the viscometer applies. There are many essential factors for fluid printability for inkjet ink formation, and viscosity is one of the crucial parameters. The Rheomat 100 rotational viscometer and the Fungilab alpha device were used in this research to measure the viscosity of the prepared CNT and PEDOT:PSS based inks. The sample volume is 5 mL for the Rheomat instrument and 15 mL for the Fungilab instrument (Figure 3.6).

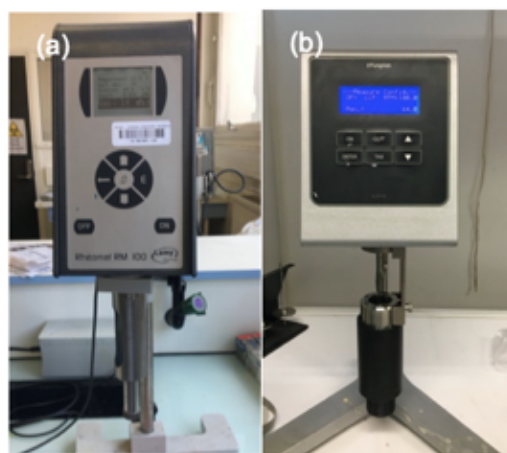


Figure 3.6 : (a) Rheomat and (b) Fungilab viscometers.

3.3.1.3 Optical tensiometer

For fluid systems, nanotube dispersions, optical profilometers were used to analyze physical properties like surface tension, contact angle, and surface energy. A Theta Lite optical tensiometer was used to measure the wetting properties of the prepared inks (Figure 3.7). The surface free energy of the substrate was obtained by measuring the contact angles using deionized water, diiodomethane and ethylene glycol via the Owens Wendt method.

It is possible to identify the equilibrium shape of a drop because external forces (like gravity) are in balance with the surface or interfacial tension of the drop by using shape techniques for the surface tension and contact angle testing. The syringe may hold a droplet that is attached to a capillary (pendant drop) or in contact with a horizontal solid surface (sessile drop) (Tropea et al., 2007).

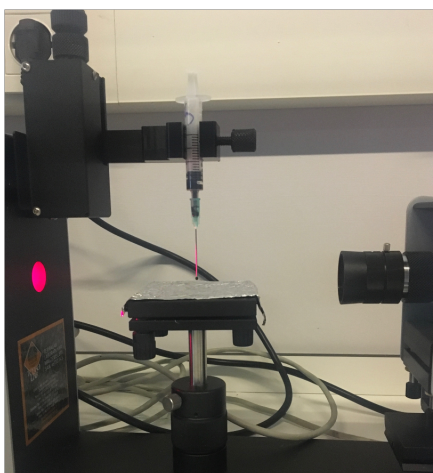


Figure 3.7 : Theta Lite optical tensiometer.

3.3.1.4 The Fourier transform infrared spectroscopy (FTIR)

FTIR has been widely utilized in the identification of the properties of a material (Figure 3.8). IR absorption by molecules gives absorption spectra. The wavenumber is a significant characteristic of a light wave. The wavenumber indicates the number of cycles a wave undergoes in a given unit of length. Wavenumbers are mostly defined as cm^{-1} units. Due to the fact that energy is proportional to wavenumber, higher wavenumber light contains more energy than lower wavenumber light. When displaying an infrared spectrum, the percent transmittance (%T) axis is commonly used to represent the percentage of light transmitted by a sample (Smith, 2011).

FTIR studies (Figure 3.8) have analyzed the functional groups present on the CNT and PEDOT:PSS surfaces in the range of 400 to 4000 cm^{-1} .

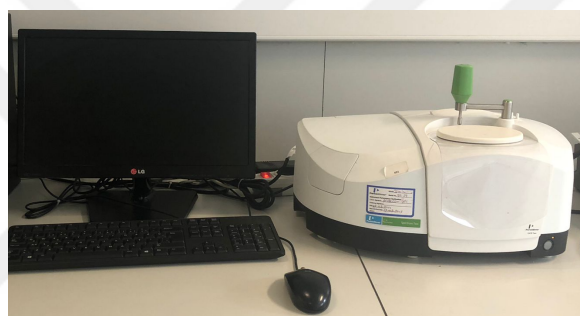


Figure 3.8 : Perkin Elmer Spectrum Two device.

3.3.2 Morphological analysis of conductive printed structures

3.3.2.1 Scanning electron microscopy (SEM)

Nanotechnology techniques like electron microscopy always require verification, since any new structures of nanomaterials should always be revealed by an electron microscope study (Kharissova & Kharisov, 2017). Electron microscopy (EM) is a technology that evaluates quite fine objects by utilizing highly energetic electron beams rather than electromagnetic radiation.

In this research, we utilized SEM to characterize morphology and to analyze developed composite structures using both Philips-FEI XL30 ESEM-FEG and Invenso IEM SEM (Figure 3.9). The gold coated specimen is first placed into the device's vacuum chamber. It utilizes a beam of electrons that is focused on the specimen to be analyzed. When the electrodes connect with the specimen, secondary electrons and backscattered electrons are emitted and magnified images are generated (Goldstein et al., 2017).

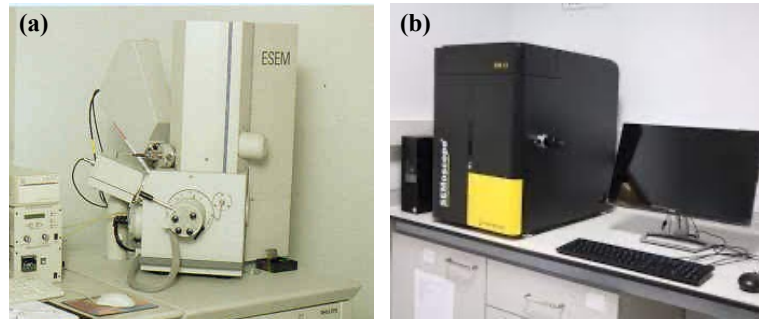


Figure 3.9 : (a) Philips-FEI XL30 ESEM-FEG, (b) Inovenso IEM SEM.

3.3.2.2 Atomic force microscope (AFM)

A test procedure to investigate the topography of a sample surface is called AFM. When given the RMS value, an RMS surface roughness value is frequently used (Lehmann et al., 2014).

AFM images were acquired using the AFM (Ambios-Quesant USPM) running in tapping mode (Figure 3.10). In Atomic Force Microscopy (AFM), a cantilever holds a probe which scans the surface of the sample, much like a scanning electron microscope. When the probe's tip taps on the sample surface, the cantilever is deflected by the forces between the tip and sample. The data collected throughout the probe's interaction with the surface is transferred to a computer, which generates an image of the surface.



Figure 3.10 : Ambios-Quesant AFM device.

3.3.2.3 Optical profilometer

A profilometer is a type of instrument that is used to define the topography or profile of the surface under test. Optical probes determine the form of a test object by detecting the optimum focus region on the sample surface (Pandey, 2018). The profilometer was

used to analyze the properties of the conductive ink printed substrate. However, in contrast to the AFM, the profilometer functions in a manner very similar to it, but with scan lengths that are substantially larger.

An optical profilometer (Zygo) (Figure 3.11) was used to measure the printed surface roughness of the conductive ink printed patterns on the textile substrate. Three dimensional-data for surface characterization can be obtained using an optical profilometer (W. L. Kelly et al., 1977).

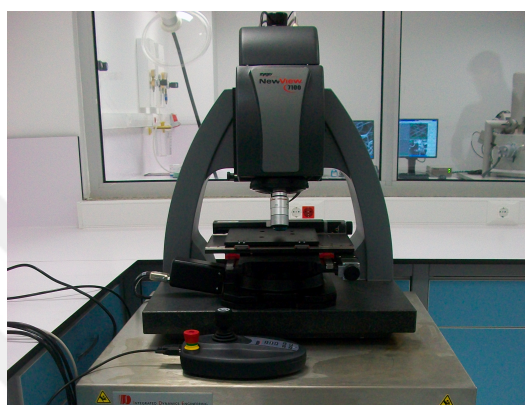


Figure 3.11 : Zygo New view 7100 optical profilometer.

3.3.2.4 Optical microscope

By looking directly at the particles, this technique is able to identify their shape and differentiate agglomeration from single particles, which is useful in certain situations (Nguyen et al., 2014). Motic B1 Advanced Series optical microscope (Figure 3.12) was used to capture images of CNT coated areas of the samples.

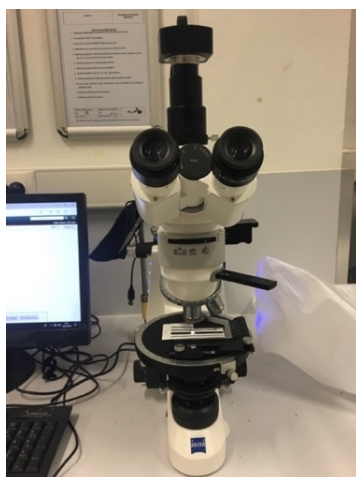


Figure 3.12 : Motic B1 Advanced Series optical microscope.

3.3.2.5 Spectrophotometer

The CIE LAB color space system was employed in this study. A spectrophotometer (Datacolor 650, USA) was used to analyze the deposition of the CNT coating according to the number of print-passes and the color properties of the samples, which were presented in the form of CIELab color space values (Schanda, 2007) (Figure 3.13). Applying the CIELAB color classification system, we are able to determine a given color using three distinct measurements ($L^*a^*b^*$) which are coordinated in a 3D color space. The L symbolizes lightness, and the a and b for color positions on a red-green and yellow-blue axis, respectively (Sharma, 2018). The spectrophotometer was used to perform colorimetric experimentation by averaging four measurements on each specimen.



Figure 3.13 : Spectrophotometer (Datacolor 650, USA).

3.3.3 Assessment of wearable sensor performance

While there are many different types of temperature sensor devices available, printed sensors have become prominent for their flexibility and comfort, making them particularly well suited for continuous measurement situations in healthcare applications. The properties of the sensor printed patterns have been analyzed in this subsection in terms of conductivity, temperature measurement ability, flexibility, stability and reliability.

3.3.3.1 4-probe multimeter system

The electrical resistance of the 10-pass printed sensors was measured using a 4-probe multimeter system (Keithley 2700 multimeter). The electrical resistance tests were conducted with samples placed between two insulated crocodile clips. The data was transferred to the computer through an RS232 to a universal serial bus (USB) converter

cable. All output data was displayed by using the Keithley 2700 Data Acquisition System, which interfaces with the PC using Excelinx software.

3.3.3.2 Hot plate with a special type thermocouple

The resistive response of the 10-pass printed temperature sensors was evaluated at the temperature range of room temperature to 50 °C on a hotplate (DAIHAN 20D), under a hot water containing beaker and under the fingertip with a special Omega TT-K-30-SLE(ROHS) type thermocouple with a diameter of 0.6 mm × 1.0 mm (Figure 3.14). Here the abbreviation “SLE” stands for -particular limits of error-. Thermocouples use the multimeter called the Keithley 2700 to obtain data, which is sent to the computer via the data acquisition/switch systems and the Keithley 7700 20 channel differential multiplexer module. The finger tapping test configuration was illustrated using a Fluke thermal camera.

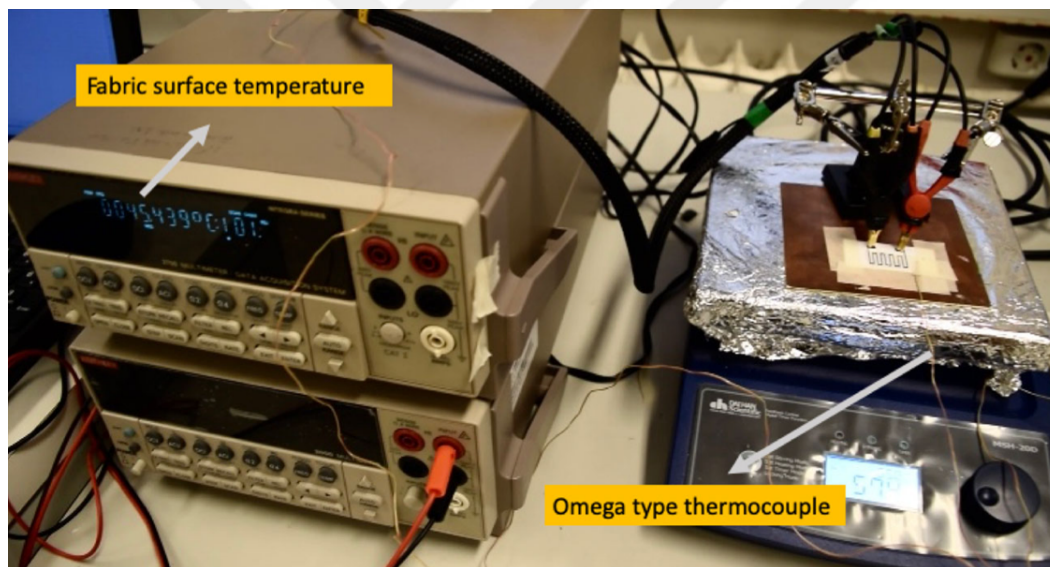


Figure 3.14 : Measurement system of a temperature sensor.

3.3.3.3 Custom-made bending cycle machine

The repetitive bending cycle tests were performed using a custom-made device. Adhesive fabric sensors were placed on the gloves to perform wrist bending tests (Figure 3.15). Resistance measurements were carried out at a flat position using a Fluke hand multimeter for gloved finger tests.

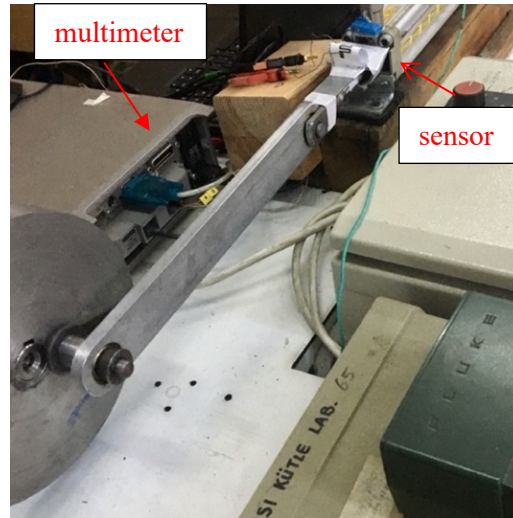


Figure 3.15 : Custom-made bending machine.

3.3.3.4 Light box

To test the effects of light exposure, we used the Gretag Macbeth Spectralight device. To test the sensor's performance stability, the temperature sensor is placed in a light box for up to eight hours. The amount of UV (Lamp: 30 Watt, 4100K; Two (2) F30T8 BLB & One (1) F6T5 BLB) (*SpectraLight III Operation Manual*, n.d.) found in natural daylight simulates the effects on sensor performance.

3.3.3.5 Crockmeter

A crockmeter (*AATCC 116 Colorfastness to Crocking*, n.d.)) is used to simulate the action of a human finger and forearm touching on a sensor surface (Buras Jr & Harris Jr, 1983). A cotton rubbing fabric was used to make contact with each specimen. The rubbing tests were conducted in dry conditions using a Crockmeter (Figure 3.16), which was crocked back and forth 20 times by making 10 full turns. After the rubbing test, the resistance and performance of the textile-based temperature sensor were evaluated using a hotplate in combination with the Keithley multimeter system.



Figure 3.16 : SDL Atlas Crockmeter.

3.3.3.6 Humidity cabinet

Multiple printed temperature sensors were placed in a hood and were subjected to varying relative humidity (RH) up to 85% RH, where the temperature was changed between 25 °C and 50 °C.

3.3.3.7 Domestic washing

Textile-based sensors should be capable of resisting washing in standard washing machines, as they will eventually be laundered at customers' homes using readily available machines. As a result, the emphasis is placed on commercially easy-accessible washing machines. For washing, we used an ECE standard/reference detergent type B without optical brightener. Each washing cycle consumed a total of 20 g of detergent and 2.5 kg ballasts. The "wool" cycles with a final tumbling speed of 600 rpm were chosen for washing tests even though woolen washing is gentle (30°C, 40 min) (uz Zaman et al., 2019). For wash durability testing, textile-based sensors were placed in a nonwoven bag. A domestic washing machine (BEKO 9102E) was used.

3.3.3.8 Perspiration

Acidic and basic perspiration tests were applied according to TS EN ISO 105 E04 (*ISO - ISO 105-E04:2013 - Textiles — Tests for Colour Fastness — Part E04: Colour Fastness to Perspiration*, n.d.) to determine the effects of perspiration on printed sensors. Although this test is typically developed to determine colorfastness to perspiration, it was utilized in this study to evaluate a difference in electrical resistivity after exposure to perspiration.

4. DEVELOPMENT OF WEARABLE TEMPERATURE SENSOR ARCHITECTURE & SENSOR PERFORMANCE ANALYSIS AND RESULTS

4.1 Development of Temperature Sensitive Ink Formulations

While flexibility is critical for an electronic system, it is extremely important to choose materials with the appropriate electrical and mechanical properties when fabricating a flexible electronic system. Additionally, viscosity is a factor to consider when printing on textiles with an inkjet printer. Moreover, when considering the wetting of the ink on the substrate, both the substrate's surface energy and the surface tension of the ink influence wetting property. This section first discusses three distinct types of dispersions that are developed under this research to be used as inks for creating temperature sensor. CNT is the first ink to be compatible with inkjet printers. This prepared ink is designed to be a practical commercial office-type inkjet printer.

The ink formulation section describes the accomplishment of two distinct goals for ink formulation: the development of a quality evaluation guideline for CNT inks and the formulation of PEDOT: PSS and CNT/PEDOT composite inks in our laboratory. The concepts and influential qualities of functional materials are examined in this chapter, as well as the essential ingredients during the printing process, which can significantly impact the fabrication of the printed sensors. The main problem with ink-containing dispersed nanomaterials is aggregation and, therefore, sedimentation. The aggregation-prevention mechanism needs a method to solve the attraction. This section also covers the related problems and solutions associated with the development of ink formulations.

4.1.1 Materials

In this research, MWCNT powder was purchased from Molchem Company and the short-length and carboxylic functionalized (-COOH) MWCNTs (Figure 4.1a) and silver adhesive paste (Figure 4.1b) were supplied by Nanografi Nano Technology. The properties of the CNTs used in this thesis project are shown in Table 4.1. Dodecyl

sulfate sodium salt (SDS) (Product No. 822050), from Sigma-Aldrich Chemical Company, and Triton X-100 from Merck Millipore (No: 108603) were used as surfactants. Ethylene glycol (EG) was from EMPLURA®. The poly(3,4-ethylenedioxythiophene): polystyrene sulfonate (PEDOT:PSS) (3-4 wt% in water) was purchased from Sigma-Aldrich. Silver adhesive paste had an average grain size of 5-10 μm . Silver plated polyamide yarn (Yarn count: 235f $\times$ 34 $\times$ 2, and Linear electrical resistance: <85 Ω/m) was purchased from Shieldex. The polyamide-based taffeta fabric from Huzhou Hengxin Label Manufacture Co., Ltd. was used as the substrate. Polyamide-based adhesive taffeta fabric (Thickness: 0.105-0.115 mm and average weight: $62 \pm 5 \text{ g/m}^2$) was purchased from Huzhou Hengxin Label Manufacture Co., Ltd. The fabric has a polyamide resin coating. An elastic, washable and translucent polyurethane welding tape was used as the encapsulation layer (Bemis ST604). Pressure and temperature are required to activate the tape. The softening temperature of the tape is 105 $^\circ\text{C}$. It is possible to provide hydrophobicity of the entire surface and to prevent air contact with this tape. The tape is also elastic and washable.

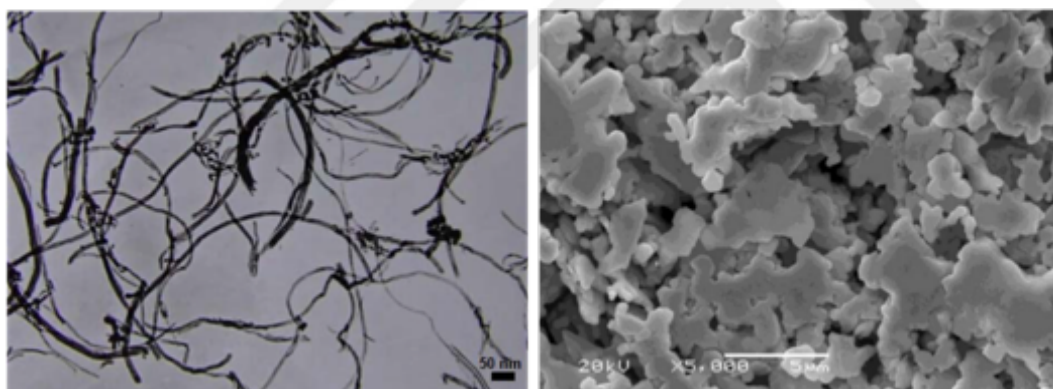


Figure 4.1 : SEM images of (a) purchased short-length CNT (2nd version), and (b) silver adhesive paste were taken from Nanografi Nano Technology.

Table 4.1 : Properties of two different CNTs used in this thesis.

MWCNT	Outer diameter (nm)	Inner Diameter (nm)	Length (μm)	COOH content (%)	Manufacturing process	Purchased company
CNT-1 st version	8-18	5-10	10-20	1.0	CVD	Molchem
CNT- 2 nd version	8-18	5-10	0.5 – 2.5	2.2	CVD	Nanografi

4.1.2 CNT ink formulations

4.1.2.1 CNT ink-Type 1

Multiple inks were prepared to achieve an optimum ratio between the SDS and the carbon nanotube concentration (Table 4.2). To produce the temperature-sensitive ink, the CNT material was prepared by dispersing it in 50 mL of distilled water with the help of a surfactant. After the addition of CNT and SDS to the distilled water, the mixture was sonicated using an ultrasonic probe sonicator (400W, 24kHz and using a 14 mm end cap diameter tip) with an amplitude of 70%, until obtaining a homogeneous mixture. The sonication time was 20 min. Figure 4.2 shows the carbon nanotubes before and after the sonication process.

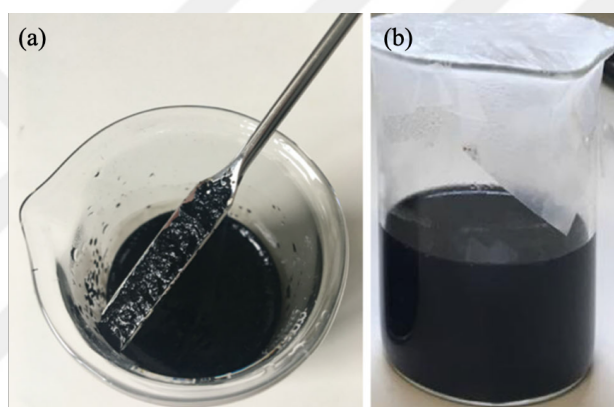


Figure 4.2 : Carbon nanotubes (a) without sonication and (b) with sonication.

Table 4.2 : Inkjet ink compositions for CNT.

Ink	CNT (g)	SDS (g)	Distilled water (mL)
A0	0.1	-	50
A1	0.1	0.1	50
A2	0.1	0.3	50
A3	0.1	0.5	50
A4	0.1	1	50
A5	0.2	0.5	50

4.1.2.2 CNT ink-Type 2

A process of the sensor fabrication system is shown in Figure 4.3. Homogenous CNT dispersions can not be obtained in the non-surfactant environment. Therefore, a surfactant, Triton X-100, was used to help disperse CNTs via a non-covalent approach.

The dispersions were prepared by mixing different concentrations of MWCNTs with a fixed Triton X-100 concentration (0.5 wt%). The concentration of Triton X-100 was determined based on previous studies (Azoubel & Magdassi, 2010; Fatemi & Foroutan, 2015b). The optimal CNT concentrations were established to obtain inks for the inkjet printing process. For this purpose, various CNT concentrations were prepared (Table 4.3). Carboxylic functionalized and Triton X-100 were first mixed with 50 mL of distilled water in an ultrasonic bath (60W, 40kHz) for 20 min. The mixture was then sonicated in a bath of ice water for 20 min. The sonication process was carried out with a sonicator (400W, 24kHz) and using a 14 mm end cap diameter tip). The CNT dispersions were then centrifuged at 5000 rpm for 10 min (3 times). The upper supernatant solutions were collected carefully and filtered using a syringe filter (0.45 μ m pore size) to obtain well dispersed CNT solutions.

Table 4.3 : Sample codes of CNT-based various inks at different concentrations.

Ink	CNT (g)	Triton X-100 (g)	Distilled water (mL)
B0	0.05	-	50
B1	0.05	0.25	50
B2	0.10	0.25	50
B3	0.15	0.25	50
B4	0.20	0.25	50
B5	0.25	0.25	50
B6	0.20	0.25	50 + 12.5 mL EG

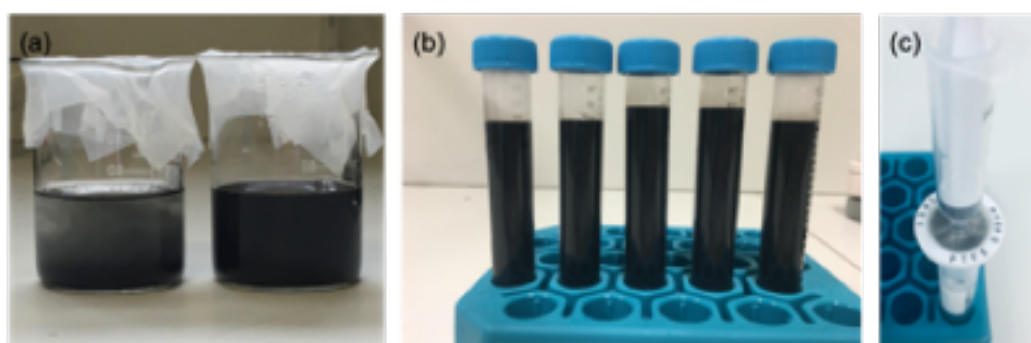


Figure 4.3 : (a) CNT ink before and after sonication. (b) Inks after centrifugation. (c) Filtration process with syringe filter.

4.1.3 PEDOT:PSS and CNT/PEDOT:PSS composite ink formulations

PEDOT: PSS (3-4 wt% in water) was diluted with distilled water (polymer solution to water ratio of 1:1 by volume) to make a polymer stock solution. The composite ink was prepared by mixing 8 ml of B6 solution with 2 ml of polymer stock solution for 1 hour using a magnetic stirrer and for 30 minutes using an ultrasonic bath. Similarly, PEDOT:PSS ink was prepared for comparison by adding 2 ml of polymer stock solution to 8 ml of distilled water-EG mixture (4:1 by volume) containing 0.5 wt % Triton X-100.

4.1.4 Characterization methods

The morphology of the suspension was characterized by optical microscopy. The absorbance values of CNT-based inks were measured by UV/Vis spectroscopy. It is aimed at examining the degree of dispersibility and stability of the ink by using absorbance data taken from UV/Vis spectroscopy. The viscometer was used to measure the dynamic viscosities of conductive inks. All measurements were carried out at 23 ± 0.5 °C. Data in MPa.s (= cPoises) was obtained. The surface free energy of the substrate was obtained by measuring the contact angles using deionized water, diiodomethane and ethylene glycol via the Owens Wendt method. The surface tensions of the prepared inks were measured with the pendant drop method and the contact angle measurements were done with the sessile drop method. Surface tension, surface free energy and contact angle measurements were done with the use of an optical tensiometer equipped with a video-camera system and computer software. The FTIR spectrophotometer was used to detect the chemical structure and different functional groups present in the inkjet inks that had been formulated.

4.1.5 Results and discussion

In order to formulate inks that are ideal for the inkjet printing process, the physical and chemical properties of the fluid must be well handled in order to ensure that drops are continuously generated at a specified electric pulse. The viscosity of inks should be comparable to water when printing with a commercial printhead such as those made by HP, Canon, or Epson (Yoshioka & Jabbour, 2005).

The viscosity and surface tension of the inkjet ink should be in the range of 2–20 mPa.s and 25–50 mN/m, respectively (Cummins et al., 2012; Izdebska & Thomas, 2015). It is necessary that the ink viscosity be low enough to allow for the refilling of the ink

channel in approximately 100 microseconds, but high enough to prevent the creation of satellites (small droplet segments that are separated from the main stream of ink) all through the printing process (Blazdell & Evans, 2000). When a droplet comes into contact with a hydrophilic substrate, it spreads out across the surface to replace a larger surface area. A high surface energy, on the other hand, results in a small impact and insufficient wetting of the substrate surface. Surfactants, which increase the wetting of the ink by lowering the surface tension of the ink, are required if one wishes to achieve complete area coverage (layer formation) (Yoshioka & Jabbour, 2005).

It is essential to disperse the required CNTs suspended in a suitable liquid in order to achieve the desired result. In actuality, commercial inkjet printer inks currently contain color pigments with sizes ranging from 100 to 400 nm, instead of colorants that have been dissolved to produce an ink solution. To prevent sedimentation and aggregation of particles, the dispersion of the particles must be remarkably efficient (Yoshioka & Jabbour, 2005).

4.1.5.1. CNT ink-Type 1

The morphology and aggregate state of the suspensions on a micrometer scale were examined by sensitively sucking up the drops of suspension with a burette and dipping them in the fabric. Optical microscopic images were taken at varying SDS levels and CNT concentrations. The dispersion of CNTs in suspension drastically alters with the increase in SDS concentration. The dispersibility of suspensions was low when the SDS concentration was too low; when the SDS concentration gradually increased, dispersibility improved. The dispersibility degree can be seen in Figure 4.4. However, the increase in SDS level decreases the electrical conductivity. The agglomeration of nanotubes starts as the concentration of CNT increases beyond the value of A5. As shown in Table 4.4, A4 has the lowest electrical conductivity. The homogeneous single-phase suspension and the highest conductivity were obtained from the A5 composition, which has the highest concentration of CNT and was, therefore, selected as the inkjet printing process in this study. The ink viscosity and surface tension were measured as 2.64 ± 0.10 mPa.s and 34.10 ± 1.00 mN/m respectively, which are in line with the inkjet inks. The printer that we used in experiments has a nozzle diameter of 9 μ m, and the density of ink is 1.01 g/cm³.

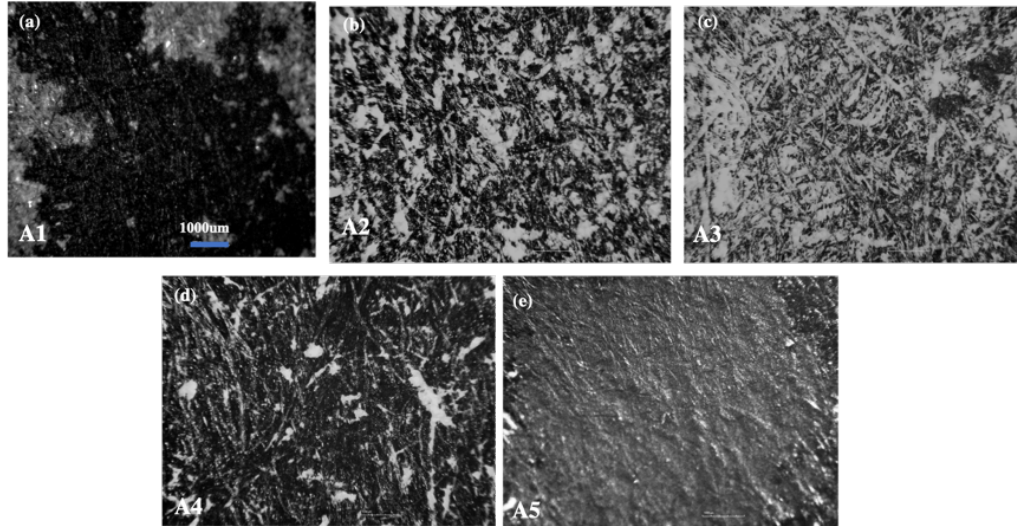


Figure 4.4 : Optical microscope images taken for different CNT – SDS concentrations at the same scale.

Table 4.4 : The prepared ink viscosities and electrical resistance values of the CNT ink-coated paper

Ink	Electrical resistance (k Ω /cm)
A1	-
A2	0.40 ± 0.02
A3	0.50 ± 0.02
A4	1.31 ± 0.08
A5	0.30 ± 0.02

The surface free energy of taffeta fabric was measured at 46.45 ± 0.30 mN/m. This value indicates that the surface free energy of the substrate is higher than the surface tension of the prepared ink. Therefore, when evaluated in terms of printability, the prepared ink was compatible with the substrate (Turkani et al., 2019).

A dimensionless number analysis was necessary to determine the eligibility of the prepared ink for inkjet printing. Therefore, we used a characteristic dimensionless number (Z) to discuss the physical properties of the prepared ink. Jang et al. defined the inks as printable in the range of $4 \leq Z \leq 14$ by considering single droplet generation, the minimum standoff distance, positional accuracy, and maximum allowable jetting frequency (Jang et al., 2009).

Table 4.5 : Calculation of Z dimensionless number for the CNT based prepared ink for CNT ink-Type 1.

Ink	Surface tension, σ (mN m ⁻¹)	Viscosity, η (mPa.s)	Density, ρ (g/cm ³)	Nozzle diameter, L (μ m)	Z
CNT(A5)	34.1 $\pm$ 1.0	2.64 $\pm$ 0.1	1.01	9	$\sim$ 7

The ink properties and associated Z dimension number were presented in Table 4.5. The calculated Z number for the CNT ink, as shown, undoubtedly proves to be acceptable for the ejection of the ink. Nonetheless, nozzle clogging problems occurred after one month of printing. It demonstrates that the sonication process is not enough to make the ink suitable for the printer. Therefore, we agreed to use carboxylated and shorter CNTs (D'Souza & Kadish, 2012), use Triton X-100 surfactant, and apply additional processes, including centrifugation and filtration. Therefore, we continued on CNT ink-Type 2

4.1.5.2. CNT ink-Type 2

A UV/vis analysis reveals individual carbon nanotubes to be active in the UV range (200 to 1200 nm) with a specific wavelength peak of $\sim$ 288 nm reported in this work. The absorbance of CNT dispersions increases until it reaches a maximum, and then decreases gradually from ultraviolet to near-infrared wavelengths, which is partially related to scattering (Isfahani et al., 2016). The obtained results by Yu et al. are parallel to the UV-Visible absorption spectra of CNTs (Yu et al., 2007). One example of the UV-vis spectra of CNT is shown in Figure 4.5.

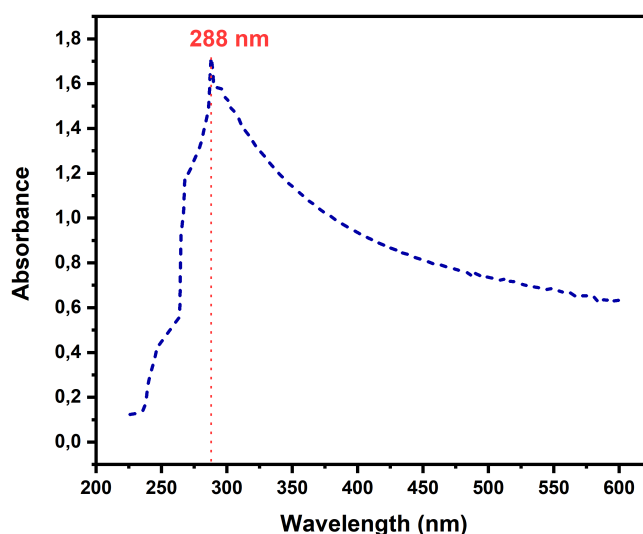
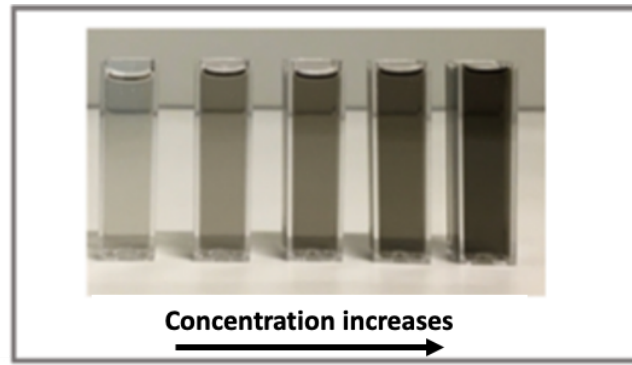
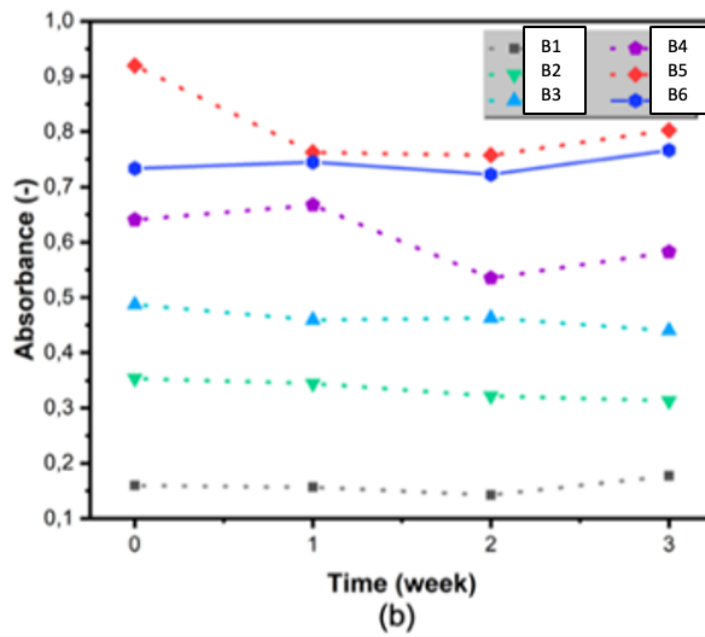


Figure 4.5 : UV-vis spectra of B6.



(a)



(b)

Figure 4.6 : (a) Photographic image of diluted CNT dispersions. (b) UV- visible spectral data as a function of the elapsed time.

The CNTs could not be dispersed without the help of Triton X-100. The B0 ink was not suitable for spectroscopy analysis. Therefore, only the CNT-Triton X-100 suspensions were diluted by a factor of 1000 for UV/Vis spectroscopy analysis. Photographic images of diluted CNT dispersions are shown in Figure 4.6a. The stability of the nanofluid is evaluated on the basis of CNT concentration by measuring the absorbance. The degree of aggregation of MWCNTs in the ink was characterized at a wavelength of 500 nm. This is because, at the given absorbance value measurement wavelength, CNT-based inks were virtually unaffected by ambient conditions (Rastogi et al., 2008). Figure 4.6b shows the absorption of the diluted supernatant suspensions with Triton X-100 versus time. The surfactant to CNT ratio is a critical factor to ensure homogeneous and stable CNT suspensions. The data shows that the absorbance value increases with the concentration of CNTs. However, high

amounts of CNT in suspensions reveal an instable characteristic over time as a result of the insufficient surfactant to CNT ratio. CNTs can not be properly covered and separated with smaller amounts of surfactant and thus precipitation occurs (Alafogianni et al., 2016). The ink with the upper level concentration of CNT (about 4 mg/mL) and stable for one week is B4. The ink B4 is hence a good candidate for further investigation. Accordingly, ethylene glycol (EG) was added (20%) to B4 dispersion (ink named B6) to enhance stability, viscosity, and surface tension as well as to prevent clogging issues.

The compatibility of the ink is important for the inkjet printing process. The measurements of contact angle were carried out at 5 seconds after the deposition of the ink drop on the fabric. The contact angle of samples exhibited an increasing trend with an increase in CNT concentration, while the surface tension properties of inks are slightly affected by CNT concentration (Table 4.6). When the viscosity of the samples was evaluated from B1 to B5, it was observed that increasing the CNT concentration exhibited no significant effect on the viscosity. The viscous behavior of EG enhances the viscosity in addition to B4. Moreover, PEDOT:PSS particles increase the viscosity of B6 by enhancing depletion interactions between the nanotubes (Maillaud et al., 2015).

Table 4.6 : Printing properties of CNT ink-Type 2 inks

Material	Viscosity (mPa.s)	Surface tension (mN/m)	Contact angle ($^{\circ}$) at 5s
B1	1.49 ± 0.01	26.03 ± 0.4	6.86
B2	1.44 ± 0.02	30.94 ± 0.3	9.87
B3	1.45 ± 0.02	30.12 ± 0.4	11.50
B4	1.52 ± 0.01	31.53 ± 0.6	12.17
B5	1.49 ± 0.02	30.19 ± 1.2	14.19
B6	2.44 ± 0.02	29.88 ± 0.4	15.51
PEDOT:PSS ink	6.43 ± 0.02	27.08 ± 1.5	27.16
B6/PEDOT:PSS ink	3.25 ± 0.01	29.33 ± 0.4	11.38

It is also required to examine whether all of the prepared inks and fabric substrate meet inkjet printing requirements. Therefore, the surface free energy of the fabric was also investigated. The surface free energy of the polyamide based taffeta fabric was

measured as 46.45 ± 0.30 mN/m, which is higher than the surface tensions of the prepared inks. Thus, surface free energy is considered convenient for inkjet printing (Turkani et al., 2019). To obtain quantitative information, we needed to use Z dimensionless number as the CNT ink- Type 1. The definition used by Jang et al. referred to inks that could be printed (Table 4.7) (Jang et al., 2009). In the continuation of the study, B6 was defined as the CNT ink.

Table 4.7 : Calculation of Z dimensionless number for the prepared inks for CNT ink- Type 2.

Ink	Surface tension, σ (mN m ⁻¹)	Viscosity, η (mPa.s)	Density, ρ (g/cm ³)	Nozzle diameter, L (μ m)	Z
CNT (B6)	29.88 ± 0.4	2.44 ± 0.02	1.030	20	~ 10
PEDOT:PSS	27.08 ± 1.5	6.43 ± 0.02	1.005	20	~ 4
CNT(B6)/ PEDOT:PSS	29.33 ± 0.4	3.25 ± 0.01	1.025	20	~ 7

As shown in Figure 4.7, the carboxylated CNT, PEDOT:PSS, and their composite inks have different FTIR spectra, with several peaks ranging from 500 to 4000 cm⁻¹ appearing. The 889 cm⁻¹ peak is assigned to the C-S bond in the thiophene ring's vibration. The peak at 1062 cm⁻¹ and the area around 986 cm⁻¹ correspond to -SO₃ stretching vibrations (Mannayil et al., 2018).

The O-H stretching vibration of hydroxyl groups can be found in the broad peak at 3700 cm⁻¹, and the carboxylic (C=O) group attached to MWCNTs is observed in the peak at 1733 cm⁻¹ (Figure 4.7). The presence of these two peaks indicates acid functionalization. The peaks at 2922 cm⁻¹ and 2855 cm⁻¹ were attributed to the CH stretching vibration of methylene produced at acid-oxidized MWCNT surface defect sites (Singh et al., 2015).

The nozzles of the printhead frequently clog up during the jetting process. There are several possible explanations for this, including the presence of particles (or aggregation) within the ink or potentially the rapid evaporation of ink on the nozzle plate, where it tends to dry up non-volatile materials within the ink and clog the printhead (Kang, 1991).

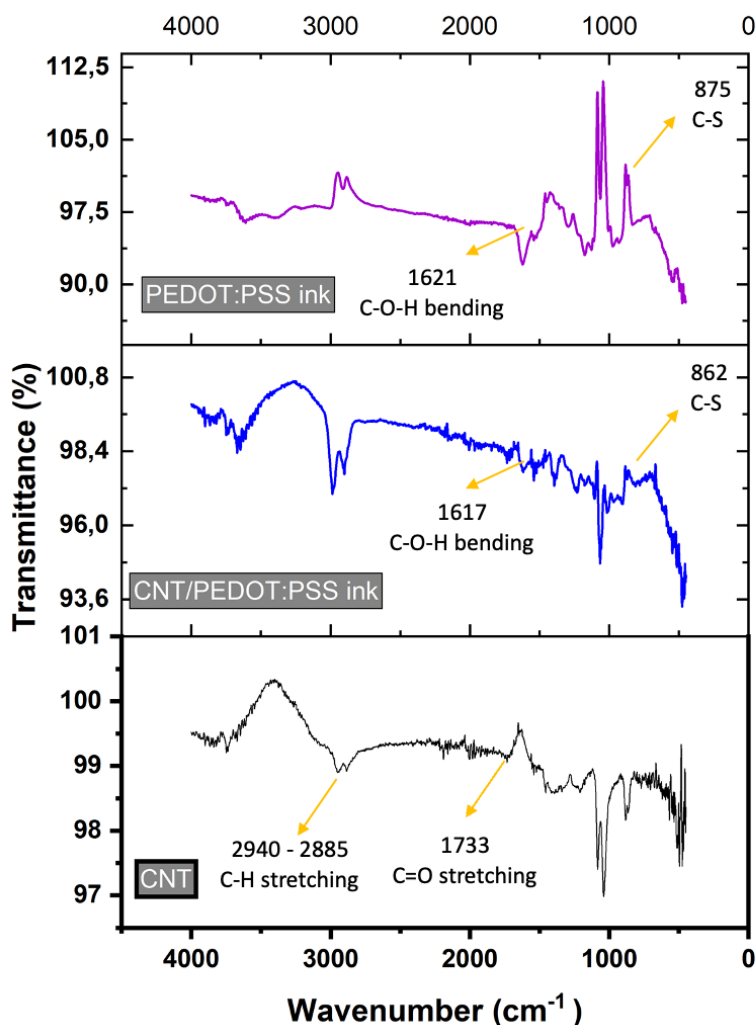


Figure 4.7 : FTIR spectra of CNT, CNT/PEDOT:PSS and PEDOT:PSS inks.

The vapor pressure of organic solvents is critical in the development of inkjet fluids because it determines how rapidly the solvent evaporates. The solvent evaporates because the nozzle orifice is open to the atmosphere whenever the printer is not printing. The viscosity at the nozzle orifice tends to increase as a consequence. The physical properties of the droplet (such as velocity, volume, and angularity) are significantly changed, making the droplet features very complex. High vapor pressure solvents exhibit this situation more noticeably. The carbon nanotube materials may precipitate in the jet during the vaporization of low boiling solvents at the nozzle orifice (Hoth et al., 2013). For this reason, in order to preserve moisture during ink formulation, a humectant was added, (Kang, 1991) ethylene glycol, which is a water-soluble, high boiling point liquid (Yoshioka & Jabbour, 2005). The evaporation properties of solvents, which were used in this study is given in Table 4.8.

Table 4.8 : Evaporation properties.

Name	Structure	Vapor pressure (hPa) (20 °C)	Boiling point	Ref.
Water	$\text{H}-\text{O}-\text{H}$	23	100	(Gamache, 2017)
Ethylene glycol	$\begin{array}{c} \text{H}_2 \\ \\ \text{HO}-\text{C}-\text{C}-\text{OH} \\ \\ \text{H}_2 \end{array}$	0.053	197.6	(<i>Ethylene Glycol</i> <i>EMPLURA®</i> <i>107-21-1</i> , n.d.)

The carbon nanotubes were deposited from the nozzles, but it will be demonstrated that these nanotubes are difficult to deposit due to their length (generally less than 1 μm and ideally less than 200 nm (Weng et al., 2010). It is only reasonable to conclude that the nanotubes underwent fluid positioning while being jetted from the actuation system and passing through the nozzle throughout the inkjet process.

4.1.6 Conclusion

All the above findings demonstrate that the inkjet device performance is strongly related to the chemical composition of the solvent, to its stability, and to the influence of the functional materials. Because of this, formulation research should be conducted for high ink storage period and performance, and the ink should be suited to the printing device. A vital stage in the development of suitable ink formulations for functional materials is to maintain the morphology and printing reliability in order to achieve flexible electronic devices.

Since the quality of the printing method is extremely dependent on the formulation of the ink, it is absolutely necessary to recognize that performance is very much related to optimization. Proper ink formulations with appropriate physical and chemical properties were successfully developed for printed sensors, which typically affects homogeneous printing surface characteristics with low defect levels over desired places according to sensor design. Long storage time, high stability in concentration, surface tension, and viscosity are all characteristics of inks that the manufacturer should aim for. In addition, the substrate showed a stable and reliable surface, as well as good homogeneity and printability, during the printing process.

Carbon nanotube jetting has primarily been successful in achieving low viscosity and well dispersed particle ink while maintaining a high nanotube concentration, which has allowed for the creation of conductive printed materials to be produced.

4.2 Development of Wearable Temperature Sensor via Inkjet Printing

Printing using an inkjet device is perhaps the most prominent method of creating flexible sensors. One of the potential advantages of printing is that it enables you to perform your operations at room temperature, which reduces the production costs (Krebs, 2009).

This section discusses the inkjet printing method, in which a textile-based temperature sensor was developed. The description of fundamental design information is based on general information gathered from a review of the literature. The production of carbon nanotubes and PEDOT:PSS containing printed patterns is the main topic of this section. A strategy for encapsulating inkjet-printed structures that does not require a chemical process has been also demonstrated. This is accomplished by coating the printed conductive area with a special welding tape and applying pressure and heat to encapsulate the printed area. The ink deposition via the inkjet printing technique is examined in detail, and its morphology with its features are also explained. The schematic illustration of the processes is given in Figure 4.8.

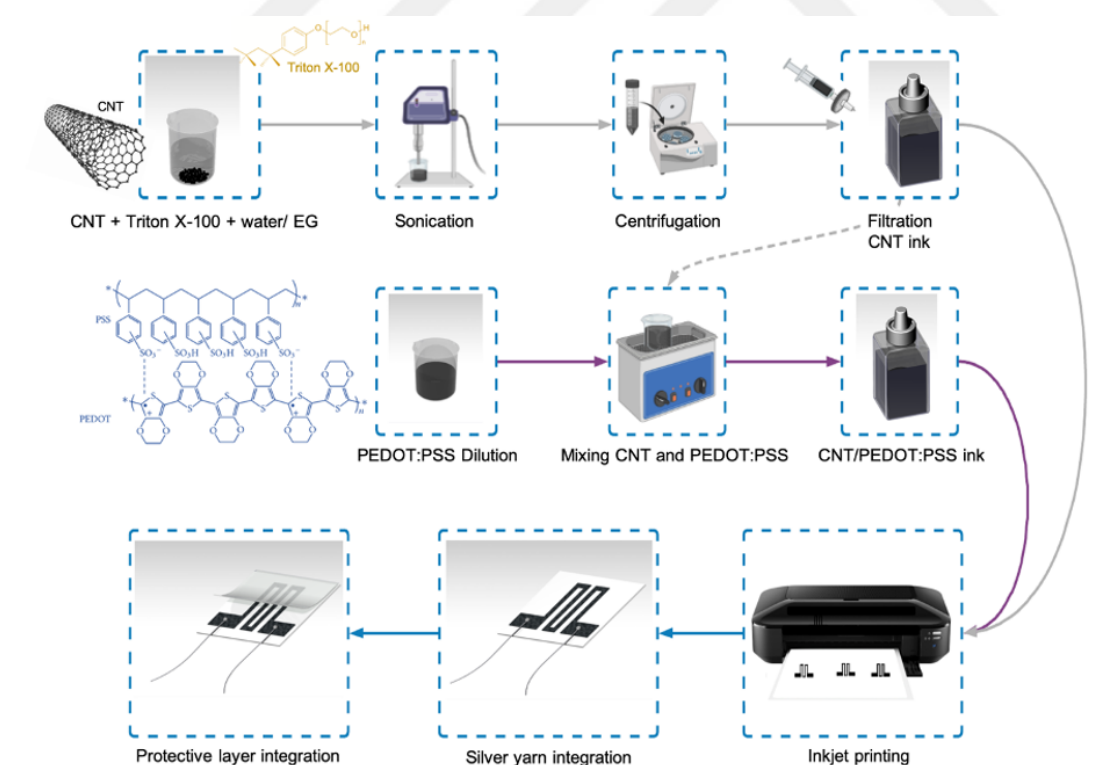


Figure 4.8 : Schematic view of inkjet printed sensor fabrication.

4.2.1 Inkjet process

A wide variety of materials in the form of colloidal or chemical solutions can be printed on a variety of substrate materials using the inkjet printing method (Khan et al., 2015; Moonen et al., 2012). Conductive lines can be formed on a variety of substrate materials using the inkjet printing method. It is analyzed that an inkjet ink is usable by conducting dimensionless studies that are based on its physical properties, as well as the printing system (Jang et al., 2009; Kosmala et al., 2011).

Commercial inkjet printers (for example, Dimatix) are very expensive. Therefore, two different types of office-type printers were used in this research. Due to experiencing clogging issues in the CNT ink-Type 1 after studying nearly two months, we decided to use the Epson Stylus SX430W, which allows us to easily switch out empty cartridges for newly formulated ones.

The formulated inks were syringe-filled into the ink tank of the printhead cartridge (piezo-based Canon Pixma G1400 printer with an optical resolution of 4800 x 1200 dots per inch (DPI)) after the inks were optimized for viscosity with respect to preferred jetting properties. The ink tank had a capacity of 135 mL for black ink and 70 mL for each of the three other colors.

The temperature sensors were printed using an Epson Stylus SX430W, a piezo-based, low-cost inkjet printer with an optical resolution of 5760 x 1440 dots per inch and a piezoelectric print head. The cartridge has a 15 mL capacity. It is possible to purchase empty cartridges, which aid in the speeding up of the printing process by allowing users to fill them in a simple manner. The viscosity of the original ink used in office printers is typically 2 mPa.s. (Beisteiner & Zagar, 2016), according to the manufacturer. Prior to beginning the printing process, the ink was allowed to settle in the cartridge for a few minutes to ensure that it was stable.

4.2.2 Design of temperature sensors

The temperature sensor containing CNT and PEDOT:PSS as the sensing materials, was patterned into different serpentine shapes, as shown in Figures 4.9a and 4.9b, to improve its performance. Because serpentine retains its mechanical stability even when subjected to mechanical deformation (Yao et al., 2020). The sensor pattern was created using Inkscape® software.

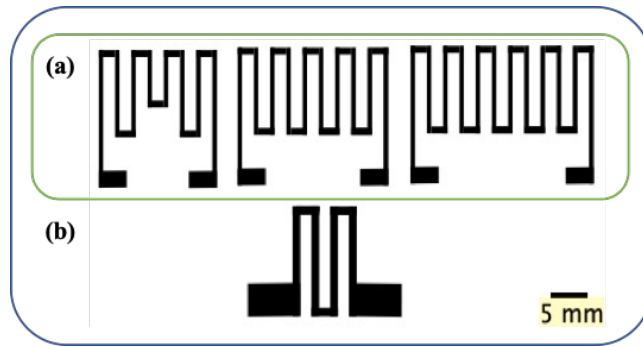


Figure 4.9 : (a) Layout of sensor designs of CNT Ink-Type 1, and (b) CNT Ink-Type 2 based sensors.

4.2.3 Encapsulation

It is critical to insulate conductive threads in order to prevent short circuits and maintain the overall consistency of the textile materials. According to the criteria of the target purpose, various methodologies indeed might an be used to overcome this issue (Castano & Flatau, 2014).

While the stretchability, conductivity, and stability of a printed pattern are all significant factors in practical applications, there are several other factors to consider as well, such as the encapsulation of printed areas for insulation and protection, and the welding of printed sensor structures with other electrical interconnections, e.g with conductive yarns.

The silver-plated polyamide conductive yarn was properly attached to the printed interconnect electrodes after printing. The conductive yarn and the electrode surfaces were connected with the help of a silver paste adhesive which cured at 80 °C for about one hour. After that, the printed sensor was encapsulated with a polyurethane tape to have a hydrophobic surface and prevent air contact. The softening temperature of the tape is 105 °C. Pressure and temperature are required to activate the tape. The tape was applied at 80 °C for 20 seconds on fabric with 5.5 bar pressure using a textile heat-press machine (Megasisitem). No linear electrical resistance change was observed after lamination with this tape.

4.2.4 Fabricated wearable temperature sensors

A wearable temperature sensor commonly includes a textile-based substrate, a sensing element, an electrode, and a protective layer. The Substrate performs the essential function of helping wearable sensors achieve their level of flexibility, as well as determining the endurance and comfort of users. Temperature changes are converted

into electrical outputs by the temperature sensitive material. The interconnector is formed of conductive material and serves as a transport system for signals. The transparent welding tape was laminated to protect the sensor. As seen in the Figure 4.10, the protective layer and a sensing layer were cured together with temperature and pressure to form the sensor structure.

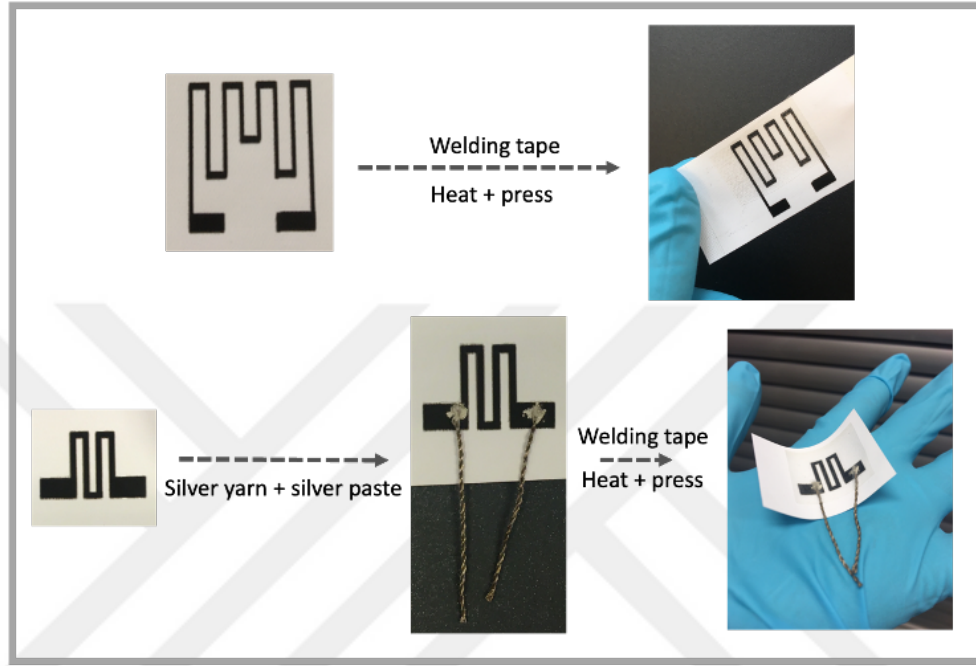


Figure 4.10 : Fabrication of sensors.

4.2.5 Characterization methods

The morphological properties of the printed conductive surfaces were examined using optical microscopy (Motic B1 advanced series), scanning electron microscope (SEM) (Inovenso IEM 11 and Philips-FEI XL30 ESEM-FEG), and an atomic force microscope (AFM) (Ambios-"Universal Quesant's Scanning Probe Microscope (USPM)). A optical profilometer with the model " Zygo 7100 New View" was utilized to analyze the surface profilometry results of the printed and base fabrics and also the printed surface thickness. A spectrophotometer (Datacolor 650, Datacolor, USA) was utilized to examine the CNT coating based on the number of print passes, as well as the color properties of the specimens, which were expressed in the form of CIELab color space values. The electrical resistance of printed sensors was determined using a four-probe multimeter system (Keithley 2700 multimeter).

4.2.6 Results and discussion for printed conductive patterns

4.2.6.1 Morphological and structural analysis

CNT ink-Type 1

For inkjet-printed e-textiles, the main difficulty is ensuring that conductive patterns are produced continuously and effectively on rough and porous textiles. In woven fabrics, the various fibres are arranged in a grid that cause planar anisotropy of the overall quality (Salvado et al., 2012). Even more, the structure of the fiber itself varies as a result of the interaction of water molecules with the external environment, which results in a changeable network of conductive lines that is particularly challenging to generate by employing low viscous inkjet ink solutions. As a result, we chose taffeta fabric that was coated in order to achieve the desired effect (Chauraya et al., 2013).

The sensor structure, which can be seen in Figure 4.11a, is composed of a CNT-printed sensing layer, and a taffeta fabric as a substrate. Figure 4.11b and Figure 4.11d demonstrate the sensor structure in more detail. When examined under an optical microscope (Figure 4.11b), it was clear that the CNT-based ink had only been deposited in the areas that had been defined during the printing process. The raw fabric (Figure 4.11c) and the printed fabric (five-print deposition) (Figure 4.11d) were both shown in SEM images for comparison. It was discovered that the printed sensor had a homogeneous deposition of CNT ink on the taffeta fabric.

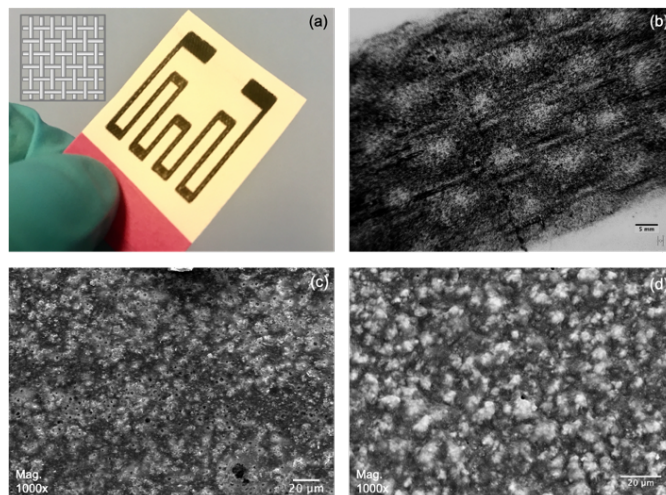


Figure 4.11 : (a) A digital photograph of the inkjet-printed sensor on taffeta fabric (Inset: Woven fabric structure), (b) an optical microscope image of the sensor surface (Lighter points on the CNT ink-1 printed fabric area are because of the fabric structure), (c) a SEM image of the raw fabric and (d) the CNT ink-1 printed sensor surface.

CNT ink-Type 2

From 1 to 10 prints, as illustrated in Figure 4.12, a gradual darkening of the printed films occurred as the number of printing passes increased. When the number of printing passes increased from 1 to 10, there was a significant improvement in the consistency of printed patterns. At 10-pass printing, very regular and continuous printed surfaces were obtained.

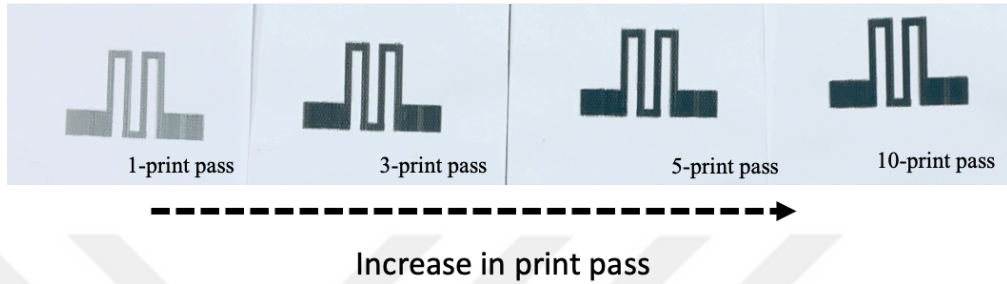


Figure 4.12 : A digital image of the printed sensors after several printing passes.

Using the resulting colorimetric values, the color disparities between the printed samples and bare fabric as standard are determined. The values for brightness (L) and total color difference (E) in the color space defined by the Commission Internationale de l'Eclairage (CIE) are as follows (Schanda, 2007):

DL= difference in lightness/darkness value (plus= lighter, minus = darker)

DE = total colour difference value

As illustrated in Table 4.9, the similar tendency of increasing darkness is observed in DL and DE values in general, especially after the initial printing. This is also a confirmation that as the number of prints increases, the print color darkness increases correspondingly.

Table 4.9 : Delta L and Delta E values of CNT printed samples

Sample	CIE DL	CIE DE
1-pass	-58,24	60,25
2-pass	-67,00	68,39
4-pass	-67,70	68,92
6-pass	-67,64	68,82
8-pass	-67,13	68,30
10-pass	-68,74	69,92

To obtain a quick perspective of the surface morphology, the image shown in Figure 4.13 uses a magnification of 1000x, which provides a good view for comparison of both the bare fabric and the CNT ink-Type 2 printed textile substrates. The morphology of the CNT deposited ink was also investigated in relation to the number of printed layers at higher magnifications (Figure 4.14).

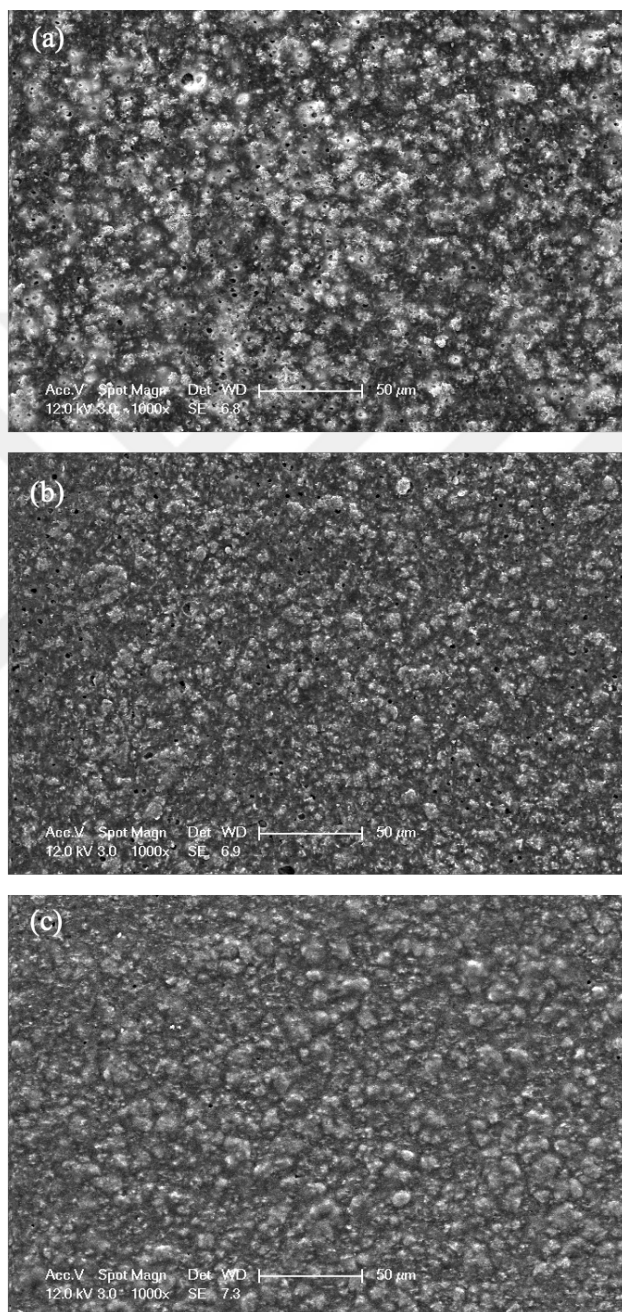


Figure 4.13 : SEM image of (a) the bare fabric; SEM image of the nanostructure of a CNT ink-Type 2 fabric surface printed using (b) 1 pass; (c) 10 passes.

When viewed at higher magnification, images demonstrate that carbon nanotube networks are clearly visible in the SEM images (Figures 4.13c and 4.13f), which show the formation of tangled, randomly oriented, and random networks of carbon nanotubes. Figure 4.14a, 4.14b, and 4.14c exhibit 1-pass printed surfaces. As illustrated in Figures 4.14d, 4.14e, and 4.14f, the 10-pass printed surfaces formed a dense network that filled almost all of the voids on the surface. The integration of PEDOT:PSS polymer on the CNT walls was confirmed by the images in Figure 4.15.

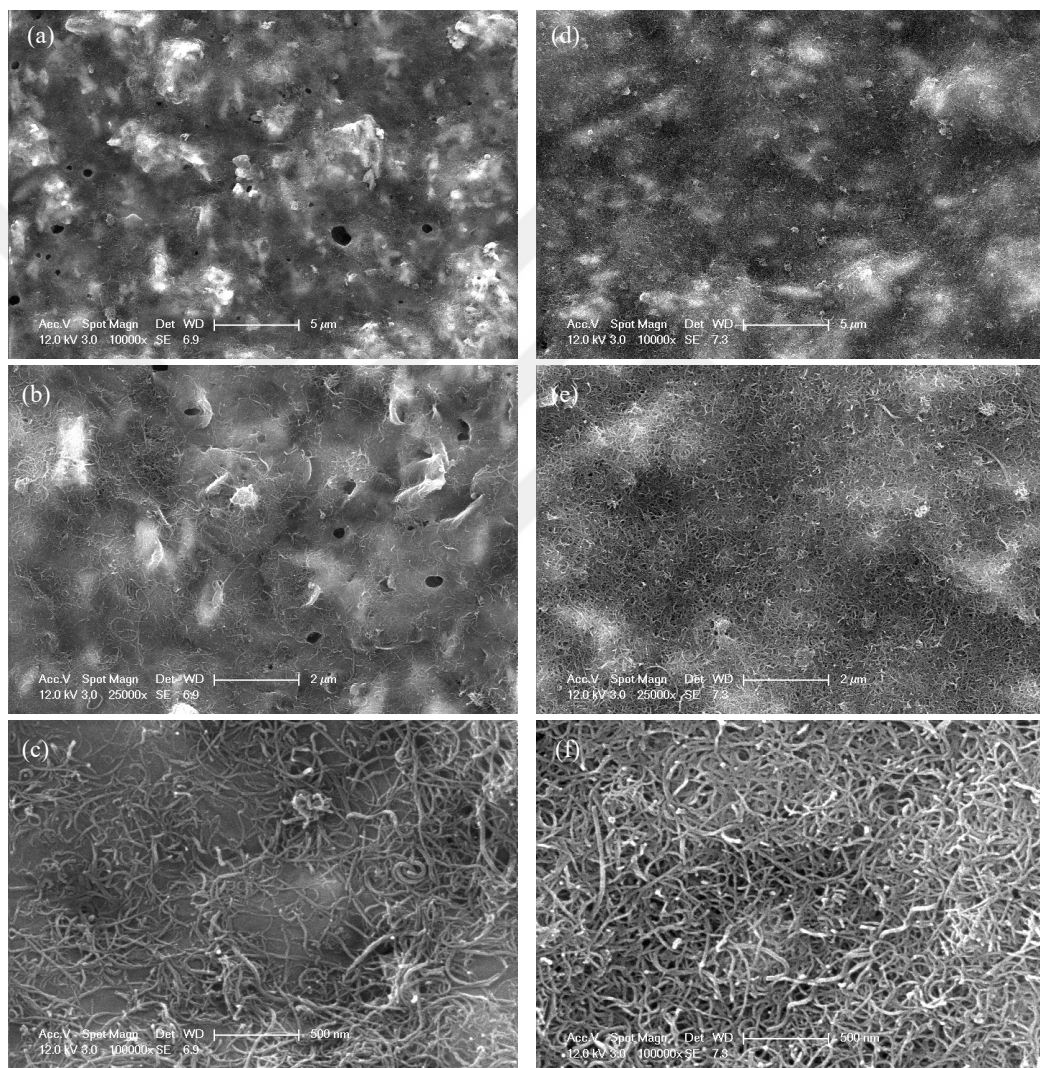


Figure 4.14 : SEM images showing the surface properties of the inkjet printed surfaces: (a-c) 1-print pass CNT (d-f) 10-print passes CNT at different magnifications.

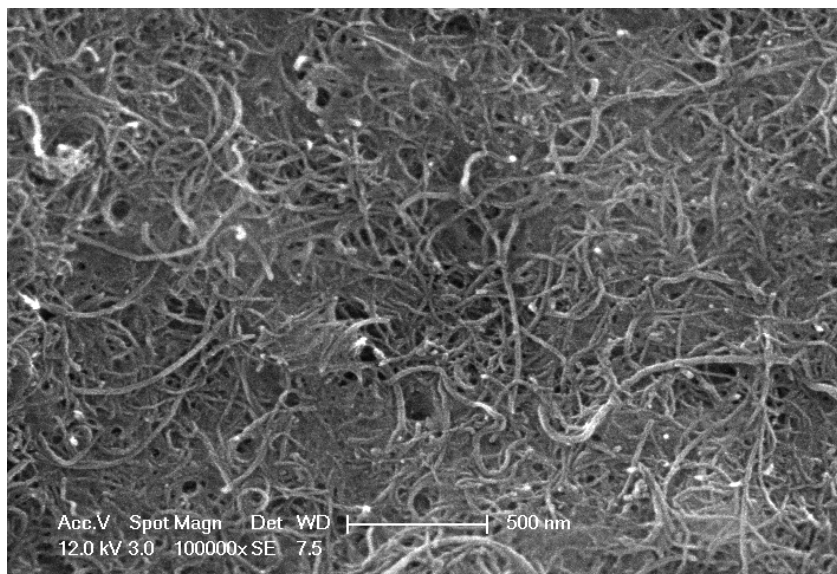


Figure 4.15 : SEM image of CNT/PEDOT:PSS printed fabric for 10-pass printing.

The textile substrate, on the other hand, has a porous structure. While some ink percolates into the textile substrate, the thickness of the printed patterns on the fabric can not be measured. As a result, the thicknesses of the printed layers can not be measured using either a scanning electron microscope image of cross sections or an optic profilometer-derived profiles. Figure 4.16 depicts a SEM cross section image of a 10-pass carbon nanotube printed fabric. CNTs are also embedded within the yarns, as illustrated in the Figure 4.16.

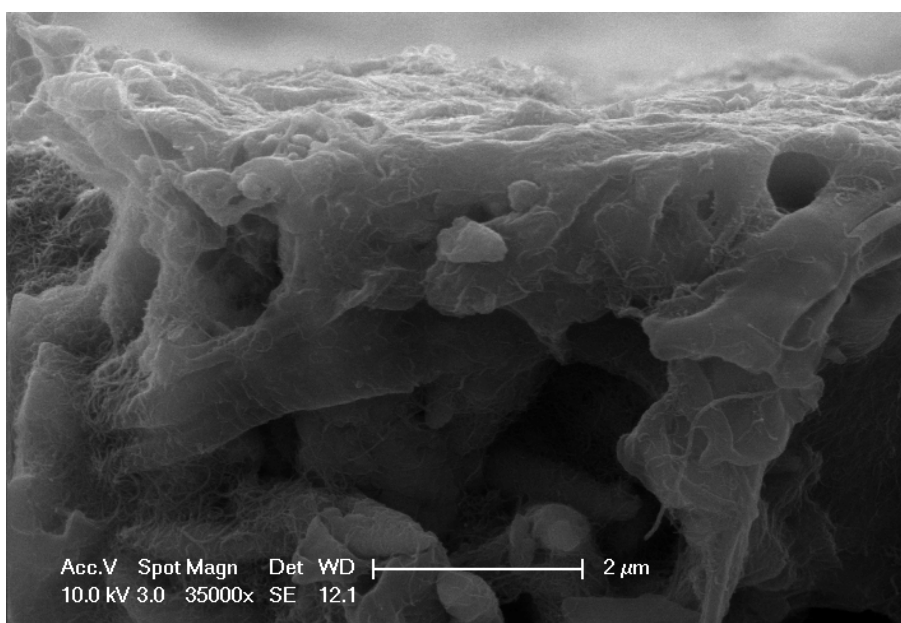


Figure 4.16 : Cross section image of 10-pass CNT printed fabric.

The optic profilometer with an X range of 180 μm , Y range of 140 μm , and a Z range of 20 μm was used to analyze the bare fabric and printed substrates, while AFM scanning probe microscopy with an X-Y range of 5 μm and a Z range of 900 nm was used to analyze the bare fabric and printed substrates. The sections in blue on the images in Figure 4.17 display the base and the lowest points, and the red regions indicate the highest points. Owing to the texture of the fabric, the distribution of ink is not consistent. Table 4.10 and Figure 4.19 exhibit the root mean square height (Sq) and arithmetic mean height (Sa) roughness values of the bare fabric and CNT printed fabrics obtained by the optic profilometer.

In Figure 4.18, the inkjet printed CNT and CNT/PEDOT:PSS ink depositions have a roughness of nanometer size, which shows that the individual carbon nanotubes are laterally implemented on the textile substrate. AFM images of the CNT printed fabric surface and the CNT/PEDOT:PSS printed fabric surface are shown in Figure 4.18. The Sq and Sa roughness values of the unprinted fabric, CNT and CNT/PEDOT:PSS printed surfaces that were obtained by AFM are shown in both Table 4.10 and in Figure 4.20. Data shows that the CNT/PEDOT:PSS printed fabric has a smoother appearance. The surface roughness showed a steady increase upon increase in CNT concentration among the samples without PEDOT:PSS. AFM has a smaller stylus tip radius than an optical profilometer, which implies a greater level of accuracy (Bhushan, 2013).

Table 4.10 : Roughness of bare fabric and CNT printed substrates

Sample description	AFM		Profilometer	
	Sa (nm)	Sq (nm)	Sa (μm)	Sq (μm)
Bare fabric	67.36	87.34	0.654	0.834
CNT - 1 print	67.58	88.85	0.679	0.878
CNT - 5 prints	92.32	106.6	0.812	0.944
CNT - 10 prints	130.07	166.9	1.192	1.412

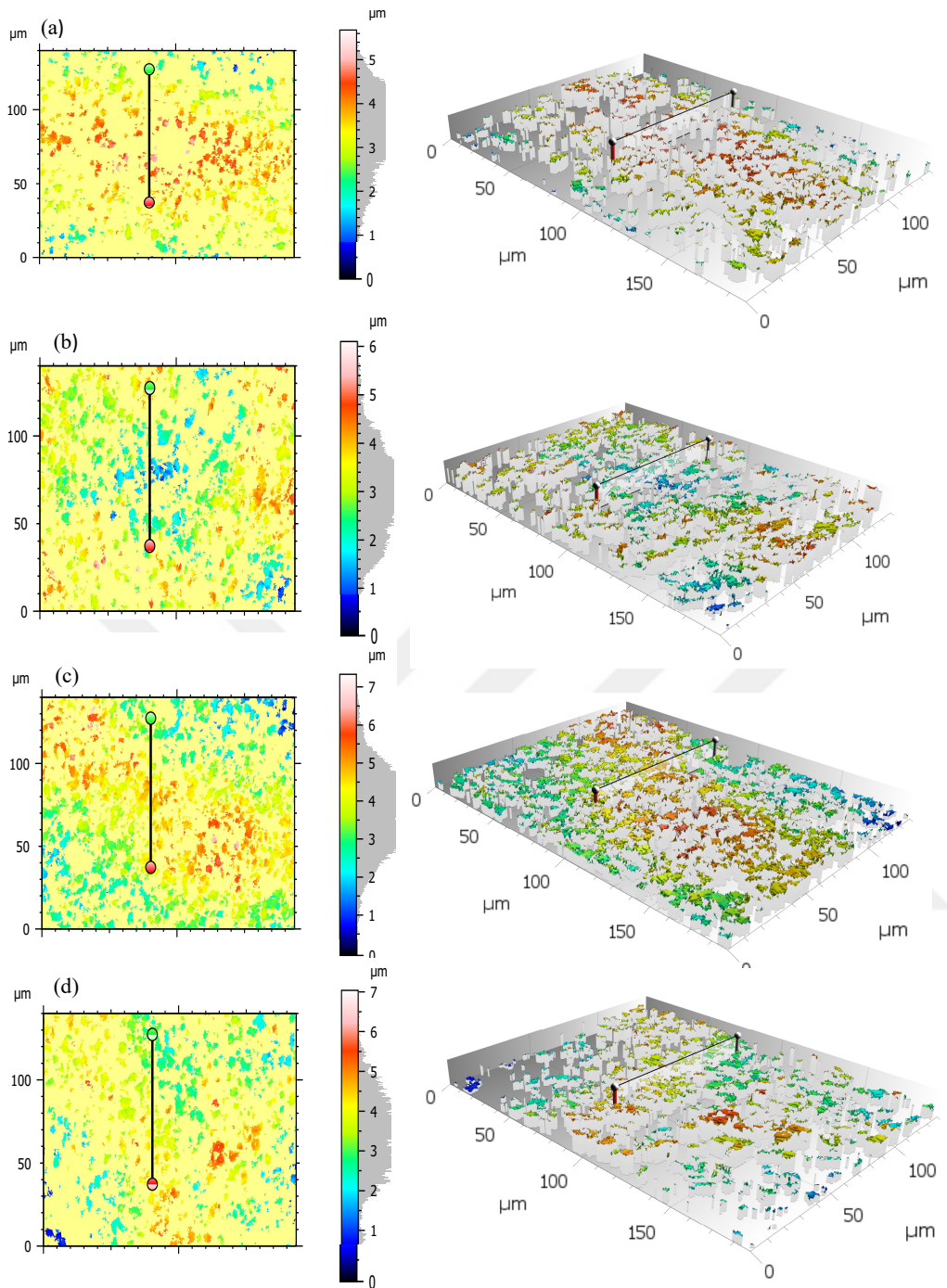


Figure 4.17 : Optic profilometer images (left column) and 3D profiles (right column) of (a) bare fabric, (b) 1-print pass CNT, (c) 5-print passes CNT, (d) 10-print passes CNT.

By using AFM and an optic profilometer, we were able to receive additional data about the topography of the printed substrates. Roughness plots of printed patterns on textile substrate using an optic profilometer and using an AFM are shown in Figure 4.17 and Figure 4.18, respectively.

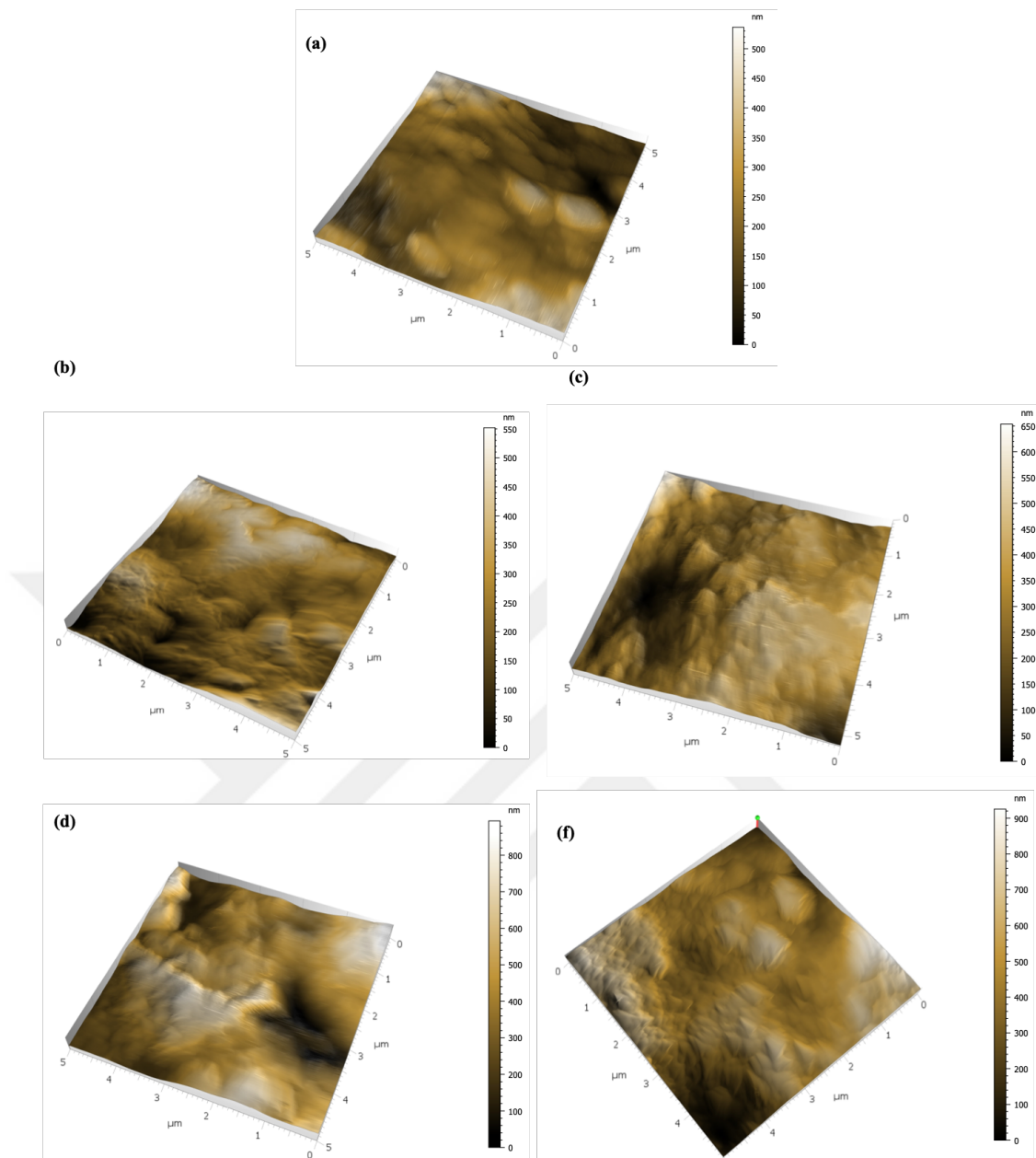


Figure 4.18 : AFM 3D profiles showing the surface roughness of the bare fabric and inkjet printed surfaces as assessed by an atomic force microscope: (a) Bare fabric, (b) 1-print pass CNT, (c) 5-print passes CNT, (d) 10-print passes CNT, and (f) 10-print passes CNT/PEDOT:PSS.

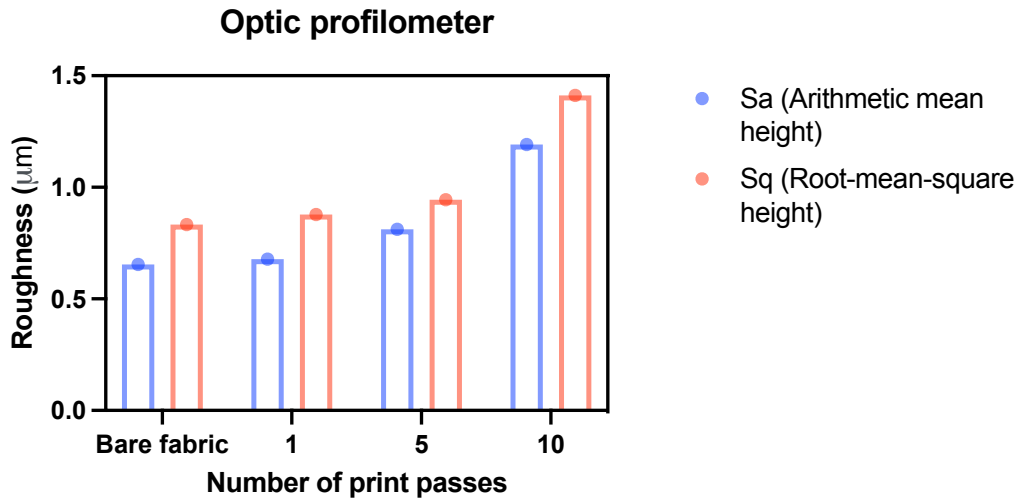


Figure 4.19 : Graph exhibiting the variation in Sa and Sq roughness values obtained by optic profilometer vs the number of print passes.

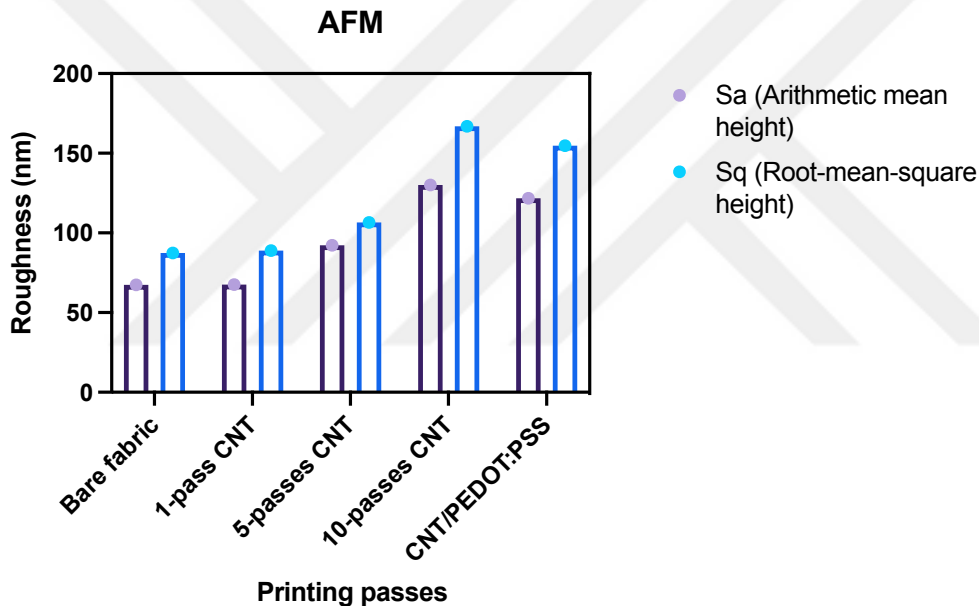


Figure 4.20 : Graph exhibiting the variation in Sa and Sq roughness values obtained by AFM vs the number of print passes.

4.2.6.2 Electrical properties of printed conductive patterns

CNT ink-Type 1

The initial stage involved varying the number of print passes to determine the electrical resistance of the printed lines. The line resistance of the CNT network depends on sequential prints over the same pattern. As expected, the resistance decreases as the number of prints increases. Figure 4.21a presents the line resistance of the CNT-printed fabric according to the number of printings per specified sensor area and also shows the resistance as a function of the line length (width = 2 mm) for multiple printed

lines. The resistance scales linearly with the length up to $L = 50$ mm. The increase in length causes resistance to increase as predicted. Figure 4.21b shows that the CNT-printed sensor can be used to light up a blue LED with a 6 V power source.

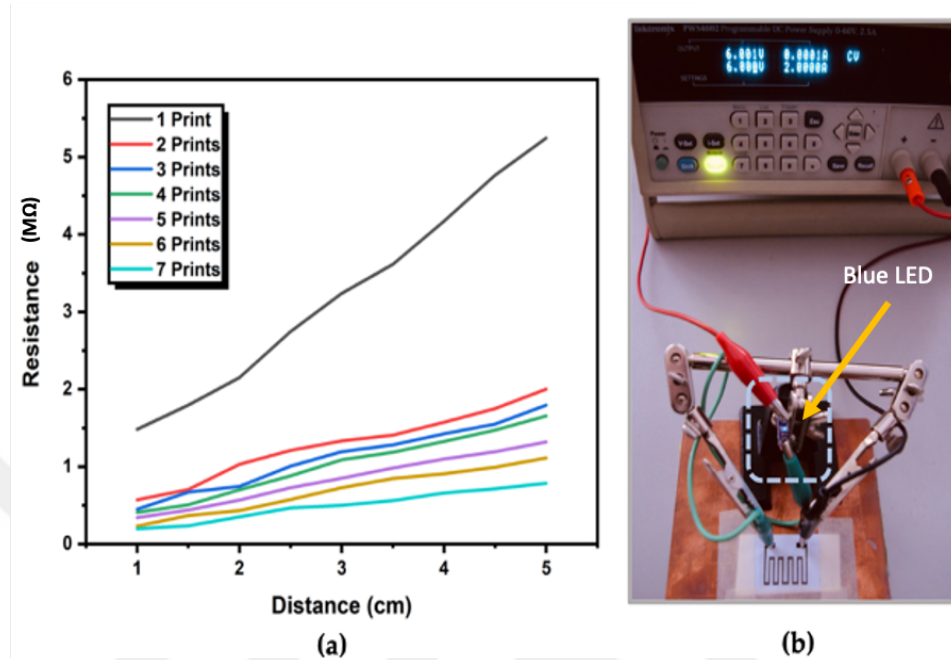


Figure 4.21 : (a) The electrical resistance of multiple printed lines plotted as a function of the interelectrode distance (line length), (b) Blue LED lighting of the CNT-printed sensor.

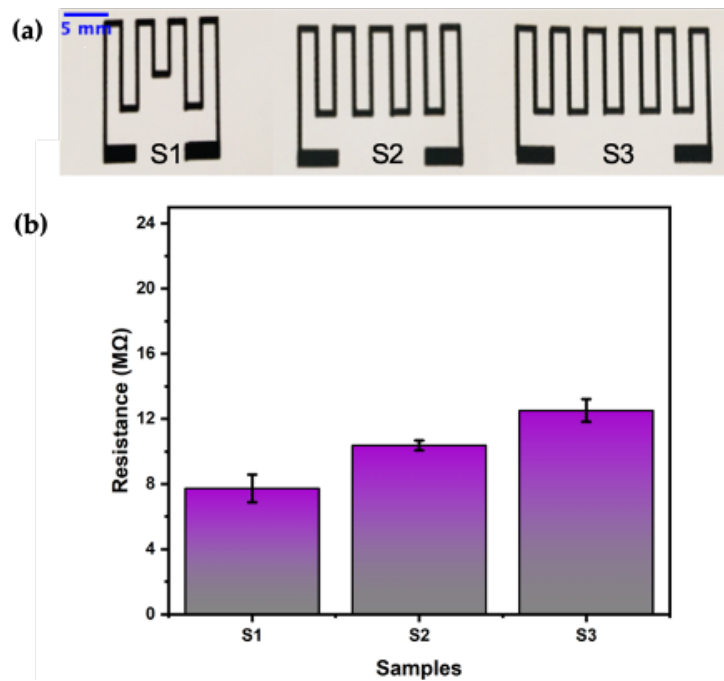


Figure 4.22 : (a) Digital images, (b) electrical resistance values of printed sensors.

Figure 4.22a exhibits the printed sensors having a different number of serpentine arms. As a result, the measured resistances are $7.73 \pm 0.85 \text{ M}\Omega$, $10.37 \pm 0.30 \text{ M}\Omega$, and $12.51 \pm 0.70 \text{ M}\Omega$ for S1, S2, and S3, respectively (Figure 4.22b). As the number of arms of the serpentine design increases (the distance between the electrodes increases), the conductivity decreases.

CNT ink-Type 2

Figure 4.23a shows the resistance of the CNT-printed sensors as a function of the number of print passes. The nylon taffeta fabrics become conductive after 2-pass printing and their conductivity increases with more print passes. The electrical resistance of the CNT printed fabric is $6.38 \pm 0.9 \text{ M}\Omega$ after the 2-pass printing and decreases dramatically after 2 passes. The increase in electrical conductivity is due to the formation of a denser nanotube network with an increasing number of prints. The Inset plotted in Figure 4.23a is the semi-log-scale plot for the same data.

After the printing process, the drying and fixation of ink on the substrate are important factors for obtaining high quality printing (Nir et al., 2010). In Figure 4.23b, the measured resistance of an inkjet printed carbon nanotube-based sensor structure is plotted against drying time. A comparison of the resistance values against drying time at 100°C in Figure 4.23b shows that drying samples at a longer time helped to lower the resistance. Printed patterns were totally dried after 7-8 minutes, achieving higher conductivity. Resistance did not change significantly thereafter. The electrical resistance of a pure CNT printed sensor is measured as $304.9 \pm 12 \text{ k}\Omega$ for 10-pass printing.

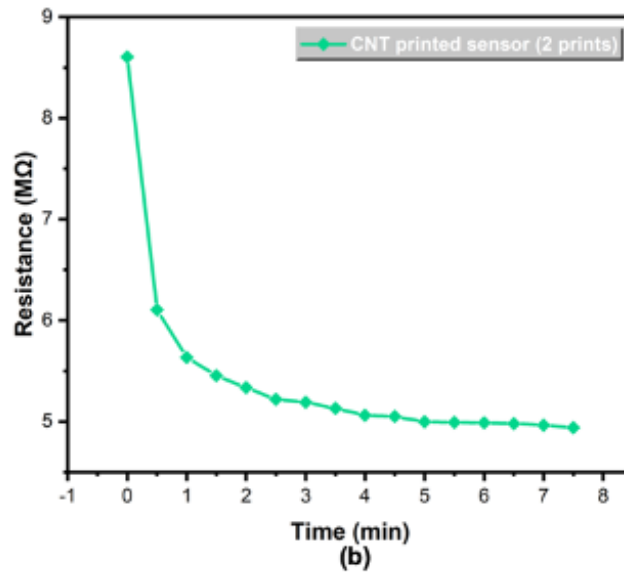
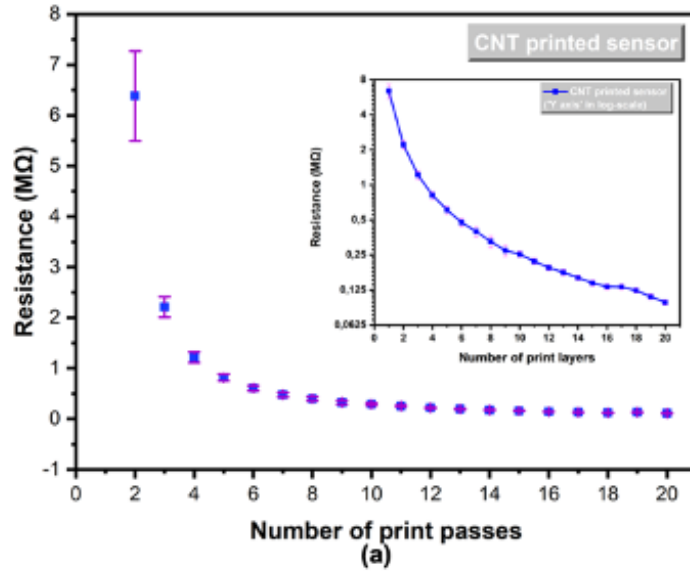


Figure 4.23 : Electrical resistance of CNT printed sensors as a function of (a) number of prints (Inset plotted in semilog-scale) and (b) as a function of drying time.

An SEM, an AFM, and an optic profilometer were utilized to examine the structures of the printed textile substrates. The CNTs have established a randomly oriented network that covers the majority of the printed surface area. Although the SEM cross-section imaging could not be used to determine the thickness of the printed pattern. We can clearly observe that nanotubes are distributed evenly throughout the substrate.

4.2.7 Results and discussion for wearable temperature sensor's electrical and mechanical performance

In this section, we used our formulated temperature-sensitive materials to manufacture printed temperature sensors. The performance of the produced temperature sensors

was studied by subjecting the printed sensor to varying temperatures ranging from room temperature to 50 °C on a hotplate (DAIHAN 20D) under a beaker filled with hot water, and under the fingertip using a special Omega thermocouple with dimensions of 0.6 mm x 1.0 mm. The effect of designed serpentine patterns on the resistance and TCR value was investigated. The configuration of the finger tapping test was demonstrated using a Fluke thermal camera. The data was sent to the computer via an RS232 to USB converter adapter. The Keithley 2700 Data Acquisition System, which integrates with the PC using Excelinx software, was used to monitor all output data. Moreover, wrist bending tests were carried out with the help of fabric sensors in the form of stickers. Resistance measurements were carried out in the flat position with a hand multimeter (Fluke). The repetitive bending cycle tests were carried out with the aid of a specially designed instrument.

4.2.7.1 Temperature measurement performance of CNT ink-Type 1 based wearable temperature sensors

The performance of sensors was evaluated using a hot plate with a thermocouple for calibration. Figure 4.24 shows the DC current (I) vs. voltage (V) curves (1 V–15 V) of the sensor when the temperature increases. The I–V curves of the sensor 1 showed linear behavior with prominent ohmic characteristics and an apparent reduction in electrical resistance with increasing temperature. The current of the sensor increased from 0.014 mA at 35 °C to 0.015 mA at 50 °C at 10 V.

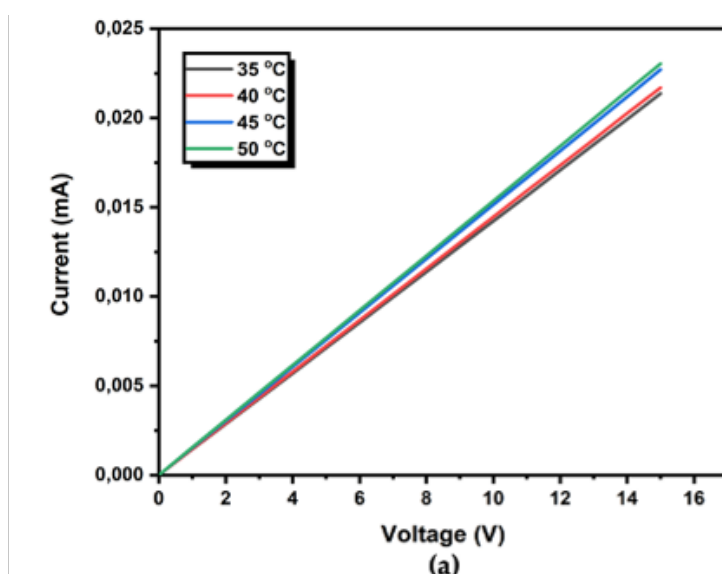
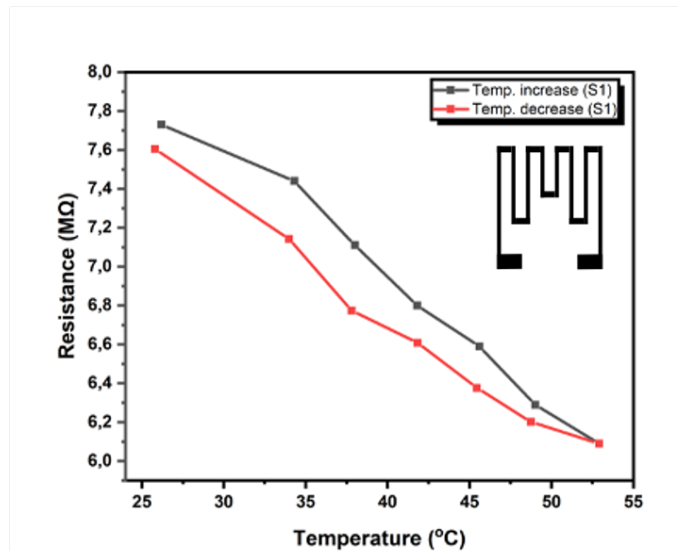


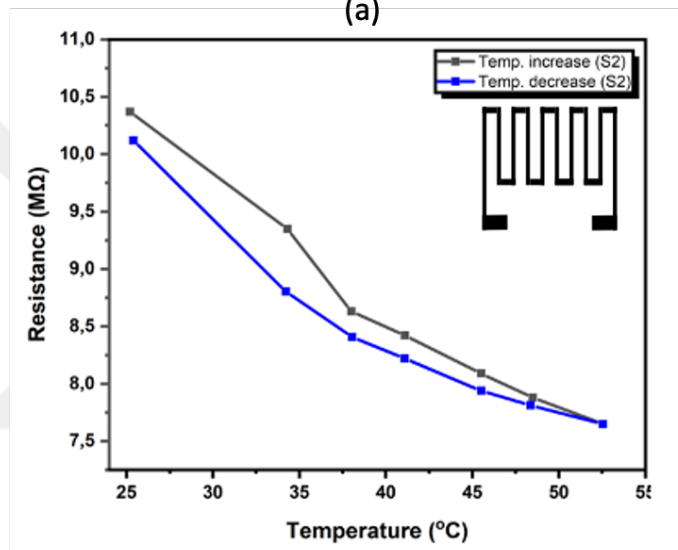
Figure 4.24 : I-V curves of the printed temperature sensor (S1).

Figure 4.25a, 4.25b, and 4.25c show the response of the sensors to the temperature varying from room temperature to 50 °C, which was applied by placing and removing sensors from the surface of the hot plate. The resistance of the produced temperature sensor decreases when the temperature increases, indicating the negative temperature coefficient (NTC) regime. The reason for the increase in conductivity of CNT-based temperature sensors under the influence of temperature is the increase in charge carrier concentration, as in semiconductors (Neamen, 2012). The closeness of the heating-cooling cycle values indicates the stability of the sensor produced. In Figure 4.26a, a non-dimensional resistance parameter is introduced as $\Delta R/R$ where $\Delta R = R(T) - R(T_0)$. It demonstrates that the responsivity of the sensor varies as the temperature increases from room temperature to 50 °C. Three differently patterned printed sensors show almost the same trend of responsivity.

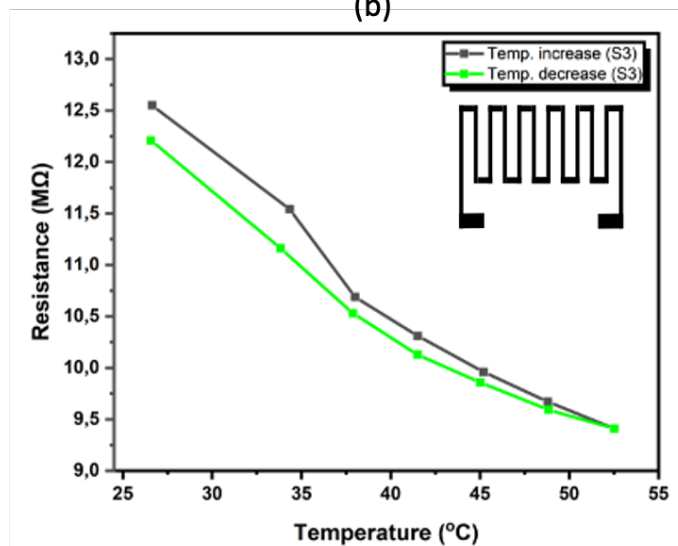
The temperature coefficient of resistance (TCR, α) is a crucial parameter for analyzing temperature sensitivity for resistance-type temperature sensing elements. The sensitivity of the sensors along the length direction increases under constant strain by creating a long material path, taking the minimum space in the sensing area with a serpentine structure (Y. Wu et al., 2017). The structure of the serpentine design of the sensors affects the sensitivity of the temperature sensor. An increase in the number of serpentine arms causes an increase in sensitivity (J. Kim et al., 2001).



(a)



(b)



(c)

Figure 4.25 : Electrical resistance changes according to the heating-cooling cycle of (a) S1, (b) S2, and (c) S3.

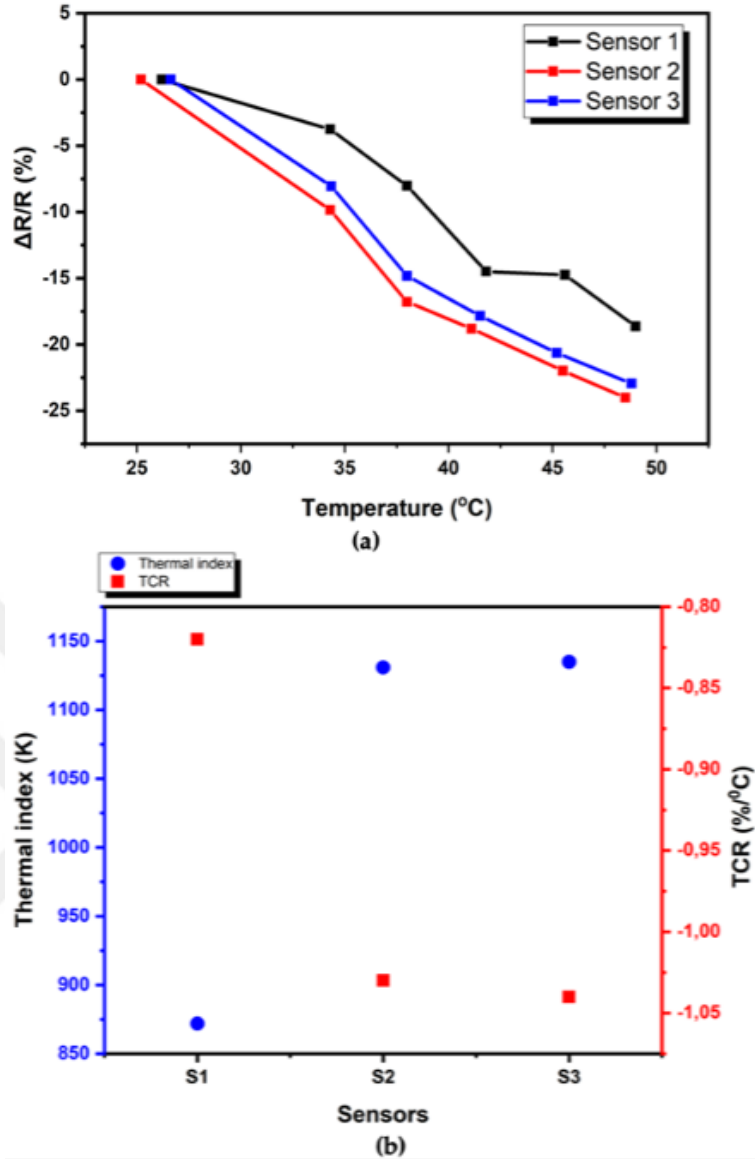


Figure 4.26 : (a) The variation in electrical resistance with temperature for CNT-printed temperature sensors. Sensors were placed on a hotplate; (b) TCR and thermal index values of printed sensors.

The TCR values between room temperature and 50 $^{\circ}\text{C}$ of S1, S2, and S3 were -0.82%/°C, -1.03%/°C, and -1.04%/°C, respectively (Figure 4.26b). The negative value of TCR is because of the dominant barrier tunneling conduction mechanism over pure metallic conduction across percolating paths (Zhu et al., 2018). The high sensitivity is due to continuous CNT lines with lower defects and adequate thickness that can cause a decrease in the electron scattering effect (Yan et al., 2015). The calculated TCRs are higher than that of standard commercial platinum temperature sensors (0.392%/K) (Sibinski et al., 2010). Even though the TCR of our sensors is lower than that of some related CNT-based temperature sensors produced with different methods, the

sensitivity obtained herein is closer and higher than some others listed in Chapter 2 Table 2.3. In addition, our sensor is one step closer to wearable sensor applications, with a lower-cost approach than those used in some other studies, both in terms of devices and materials.

The thermal index (thermal constant, β), which is also a sensitivity parameter, commonly lies between 1000 K and 10,000 K for temperature sensors using ceramic and semiconductor materials (Sahoo et al., 2014). The high β value indicates high-temperature stability (Shahil & Balandin, 2012). The calculated β values of S1, S2, and S3 temperature sensors are ~ 872 K, 1131 K, and 1135 K, respectively. Our sensor's thermal indexes are lower than conventional ceramic/semiconductor-based sensors, but comparable with previously reported flexible temperature sensors (Shahil & Balandin, 2012; Yan et al., 2015).

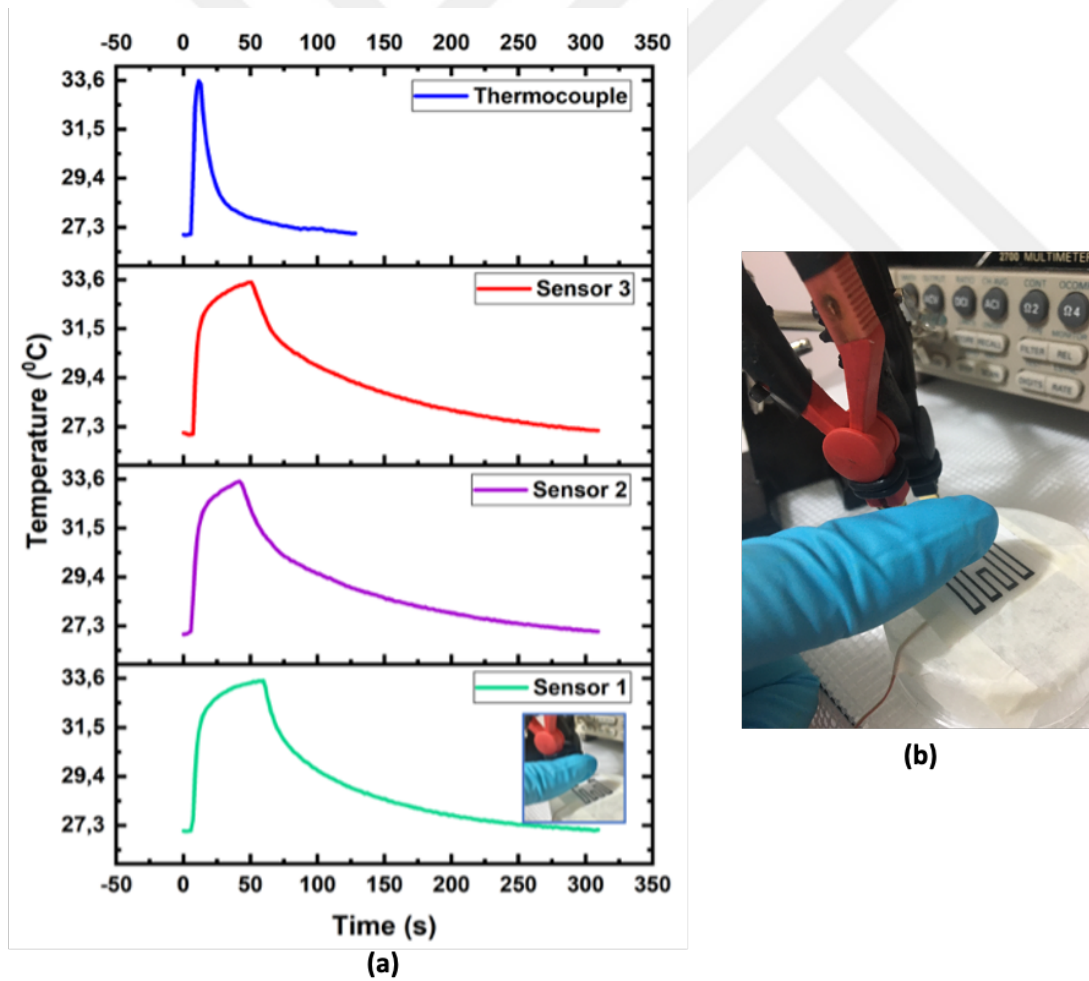


Figure 4.27 : (a) Responses of printed temperature sensors on taffeta fabric and a K-type thermocouple during the fingertip touch and leave process, (b) finger approaches the sensor.

Additionally, to compare the temperature response performance of the printed temperature sensor (S1), two different testing methods were also used. A finger temperature experiment (Dinh et al., 2016) was applied to test the reactions of printed temperature sensors to temperature variations. A gloved finger was placed over the top surface of the printed sensors once equilibrated to a temperature of $\sim 33^{\circ}\text{C}$ and then located at 27°C . The response time of the printed temperature sensors is shown in Figure 4.27. The test results revealed that the printed temperature sensors and the reference k-type thermocouple could respond to temperature changes in 1–1.5 s. The transfer of heat from the fingertip to the sensor gradually occurs. As shown in the graph, all sensors had an identical equilibrium time scale, but due to the larger heat capacitance of the fabric substrate, the time needed to achieve equilibrium was longer than the thermocouple. Furthermore, the large capacitance of the fabric also caused a longer recovery time of the sensor than the thermocouple. The response of the printed temperature sensor to the temperature of the beaker, which contained hot water (45°C), was exhibited in Figure 4.28. Once the beaker was placed on the sensor surface, the temperature of the CNT-printed sensor increased, resulting in a reduction in electrical resistance. The electrical resistance became stable at the equilibrium state of temperature $\sim 31^{\circ}\text{C}$.

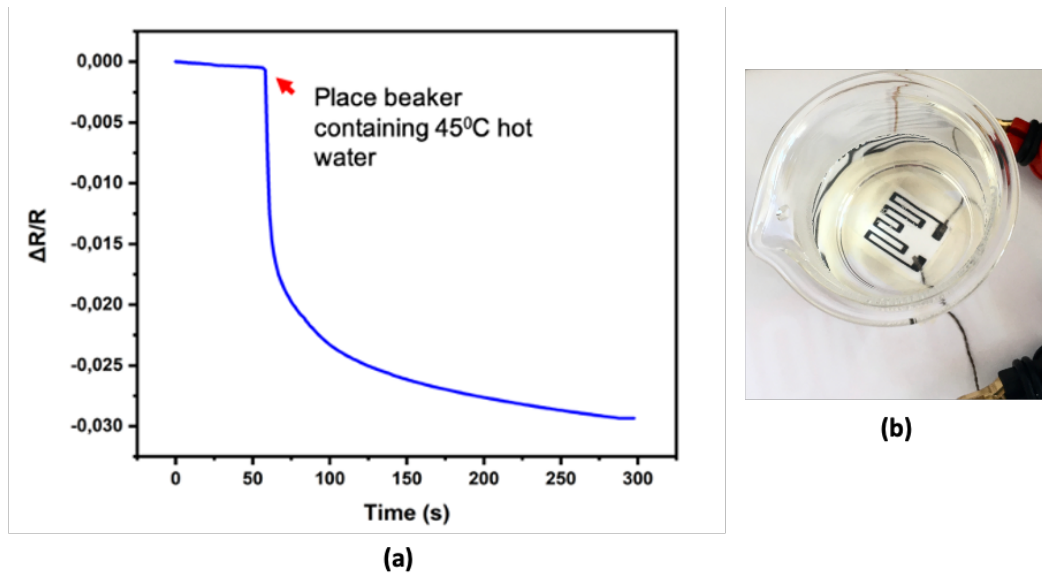


Figure 4.28 : (a) The response of the printed temperature sensor to the temperature under beaker containing hot water, (b) sensor under beaker.

4.2.7.2 Mechanical stability of CNT ink-Type 1 based wearable temperature sensors against bending and folding

The mechanical stability of the printed temperature sensor, S1, was characterized. The resistance change as a function of wrist joint motion was given in Figure 4.29. This graph demonstrates the resistance changes of the flattened sensor after repetitive wrist bending motions. The maximum resistance variation value was 9.3% between the initial state and the state after 30 cycles. In another test, the sensor was bent at different angles, which resulted in changes in the resistance (Figure 4.30). The conductivity decreases with increasing folding angles. The maximum resistance variation value was found to be 7.3%. Moreover, multiple folding tests showed a maximum change in resistance of nearly 6.2% (Figure 4.31), only a 1% TCR increase was observed at the folded condition compared to the flat condition (Figure 4.32). Both bending and folding have a significant influence on the resistance of the temperature sensor. Therefore, for wearable sensor applications, it may be a more convenient method to apply the printed temperature sensor by sticking it onto the garment, rather than stitching on the temperature sensing areas where folding and bending can be minimized.

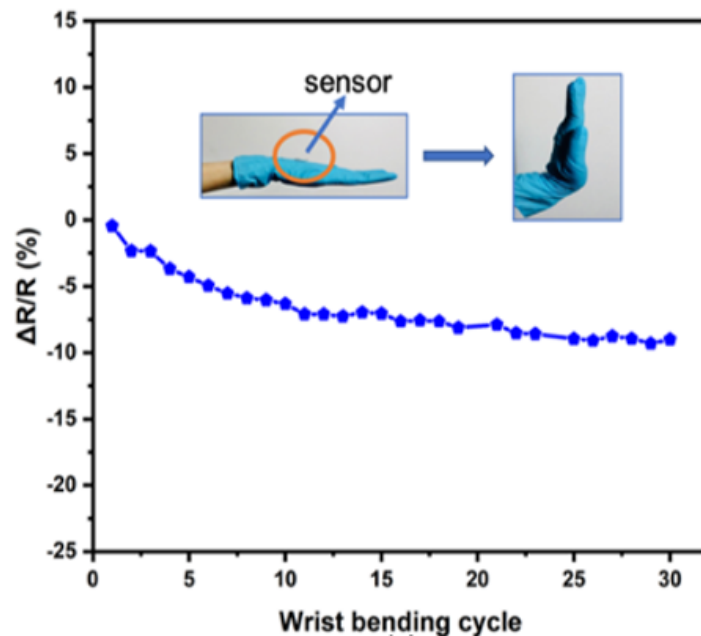


Figure 4.29 : The electrical resistance measurement of S1 during the wrist joint motion. The response to the wrist bending to 90° was measured. (Inset: photographs of the wrist joint motion).

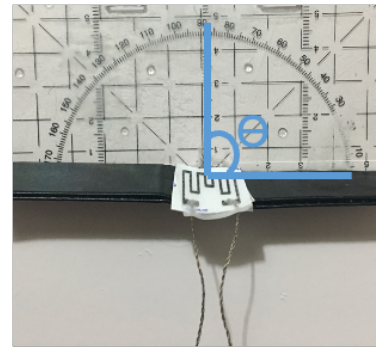
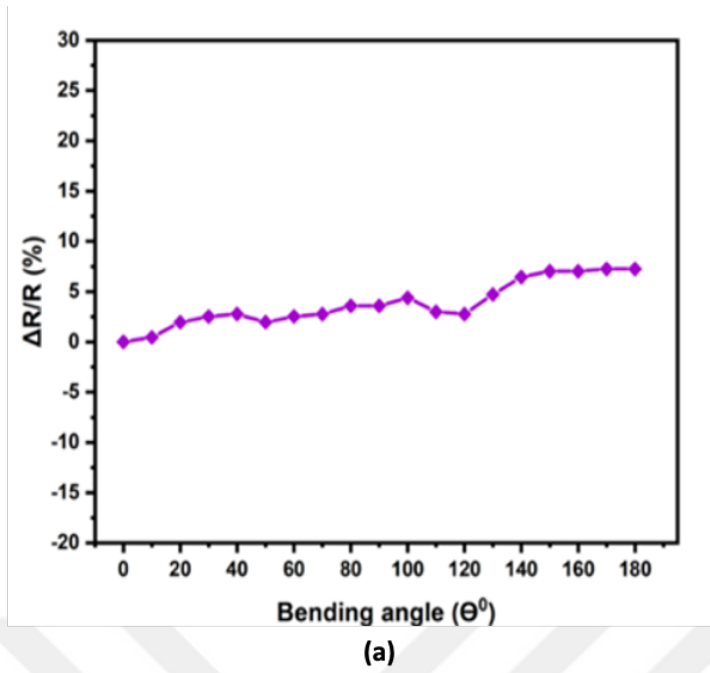


Figure 4.30 : (a) Sensor resistance variation of the sensor folded under various curvature angles, (b) test set-up.

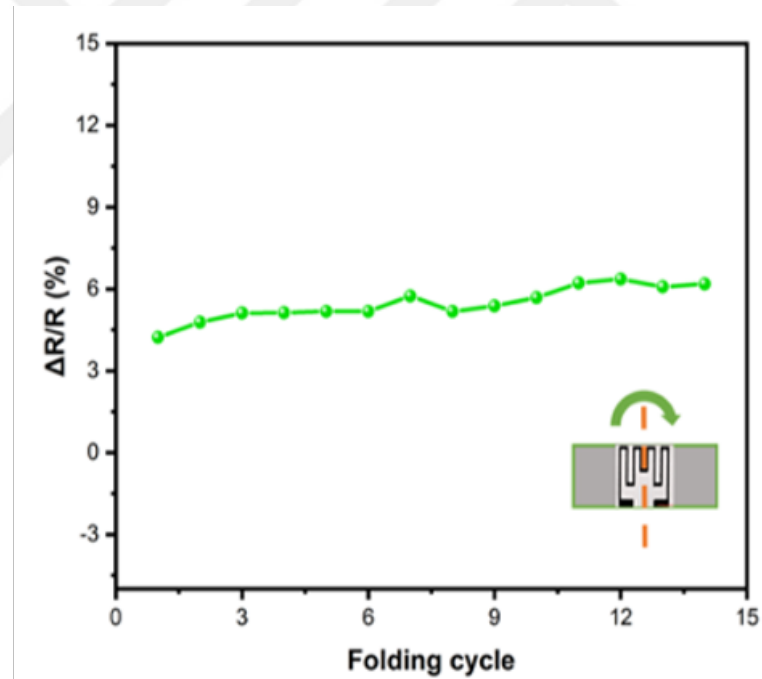


Figure 4.31 : Cyclic folding tests of the printed temperature sensor.

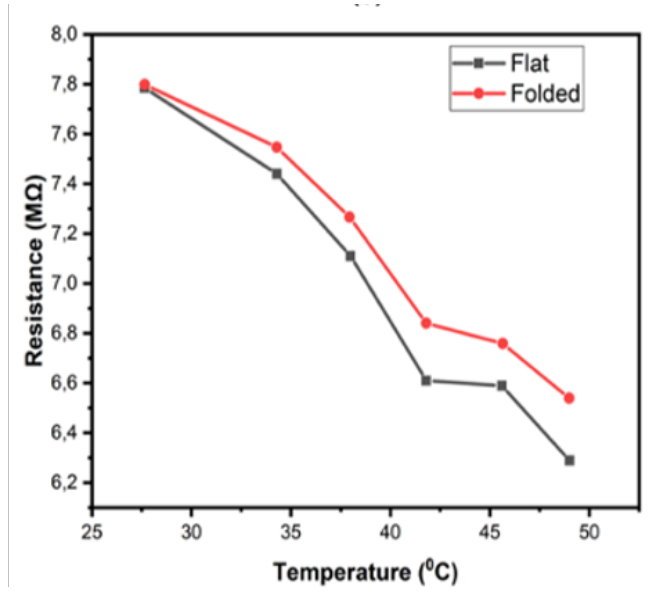


Figure 4.32 : Comparison of sensor performance between flat and folded positions.

4.2.7.3 Linearity of CNT ink-Type 1 based wearable temperature sensors

The dependence of fitted data and the accuracy of the temperature sensor measurement were evaluated using the Steinhart Equation 4.1.

$$\frac{1}{T} = A + B \ln R + C (\ln R)^3 \quad (4.1)$$

where T is the temperature in Kelvin, and R is the resistance at temperature T. A, B, and C are Steinhart coefficients used to calculate temperature associated resistance. Coefficients are calculated using Equation 4.2 and the results are listed in Table 4.11.

$$\begin{pmatrix} 1 & \ln(R_1) & \ln^3(R_1) \\ 1 & \ln(R_2) & \ln^3(R_2) \\ 1 & \ln(R_3) & \ln^3(R_3) \end{pmatrix} \begin{pmatrix} A \\ B \\ C \end{pmatrix} = \begin{pmatrix} \frac{1}{T_1} \\ \frac{1}{T_2} \\ \frac{1}{T_3} \end{pmatrix} \quad (4.2)$$

Table 4.11 : Steinhart coefficients of printed sensors calculated at temperatures of 307.15 K, 315.15 K and 322.15 K.

	A	B	C
Sensor 1	2.68×10^{-2}	2.42×10^{-3}	-2.10×10^{-6}
Sensor 2	4.38×10^{-2}	4.10×10^{-2}	-5.30×10^{-5}
Sensor 3	-4.56×10^{-2}	4.22×10^{-2}	-5.27×10^{-5}

The calculated Steinhart coefficients of the printed sensors are given in Table 4.11. The accuracy of the S1, S2, and S3 printed sensors was investigated (Figure 4.33). The accuracies of the sensors were calculated as $\pm 0.7^{\circ}\text{C}$, $\pm 0.5^{\circ}\text{C}$, and $\pm 0.2^{\circ}\text{C}$ for S1, S2, and S3, respectively. These values obtained are comparable data to thermistors on the market with an accuracy between ± 0.001 - 0.1°C (Feteira, 2009). In another study in the literature, the accuracy of the sensor produced using CNT gravure printing was found to be $\pm 0.5^{\circ}\text{C}$. The sensors we produced performed better in terms of accuracy (Turkani et al., 2018).

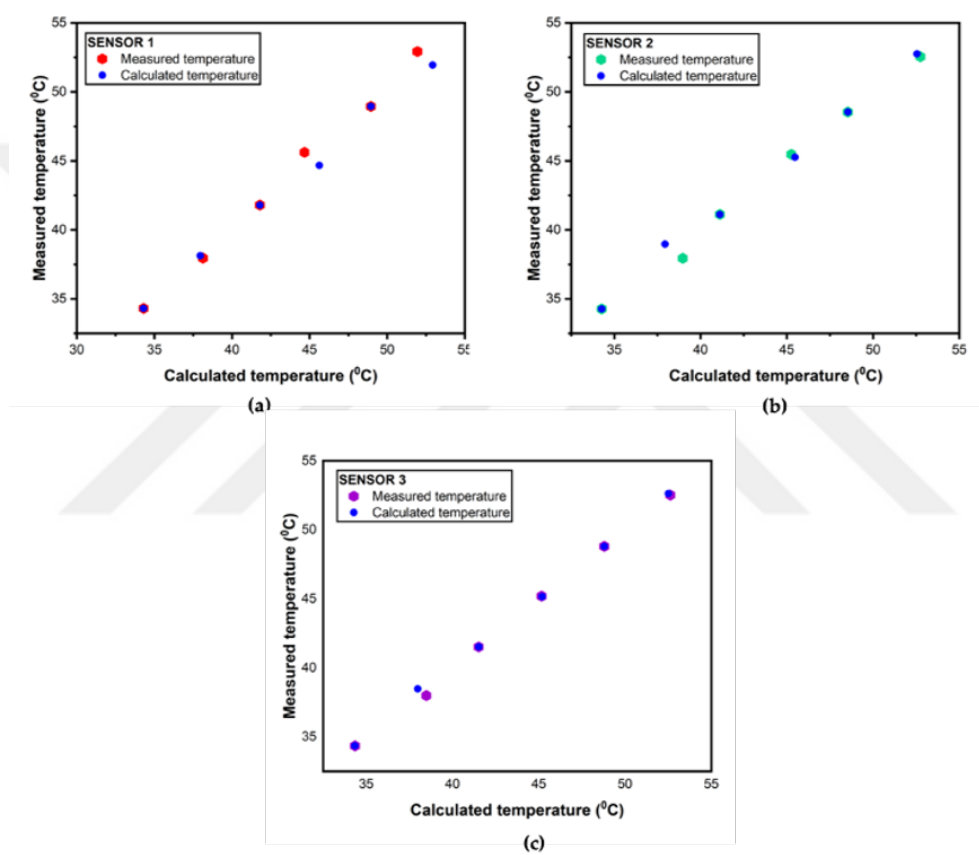


Figure 4.33 : Accuracy graphs of CNT printed temperature sensors.

4.2.7.4 Temperature measurement performance of CNT ink-Type 2 based wearable temperature sensors

A 10-pass printed sensor's temperature-sensing capabilities were assessed by varying the resistance using a hot plate from room temperature to 50°C . Temperature-resistance change graphs of the temperature sensor printed on nylon taffeta fabric are shown in Figures 4.34. The resistance-temperature curve is nearly linear. The average sensitivity is $-0.15\%/^{\circ}\text{C}$, with a standard deviation of 0.02. The TCR value is better than some CNT-based temperature sensors previously found in the literature (Di

Bartolomeo et al., 2009; Dinh et al., 2016; Giuliani et al., 2014). As shown in Graph 4.34, the sensor proceeds in approximately the same temperature-resistance data at one temperature cycle, indicating that the sensor is performing in an almost identical way. The thermal index was 152.7 K with a 23.3 standard deviation. The temperature drop was slower due to the thermal conductivity level of the substrate. The thermal conductivity of taffeta fabric is 16.12 mW/mK. The reaction of the printed temperature sensor to the temperature of the beaker containing hot water (50 °C) is shown in Figure 4.35. Following the positioning of the beaker on the surface of the sensor, the temperature of the CNT printed sensor increased, leading to a decrease in resistance as estimated. With increasing temperature, sensors' electrical resistance reduces significantly, indicating a negative temperature coefficient characteristic.

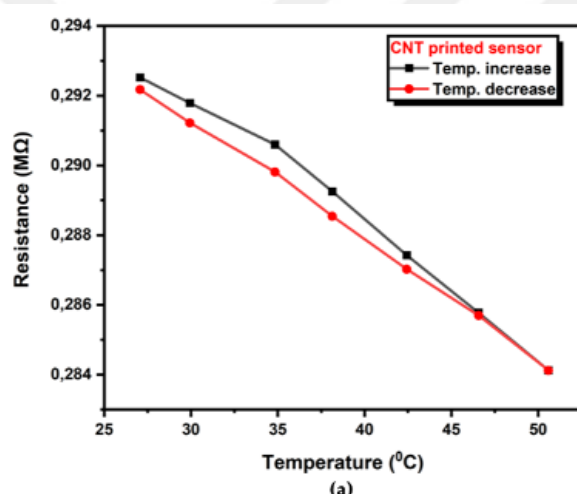


Figure 4.34 : The heating-cooling cycle of the CNT printed sensor pattern.

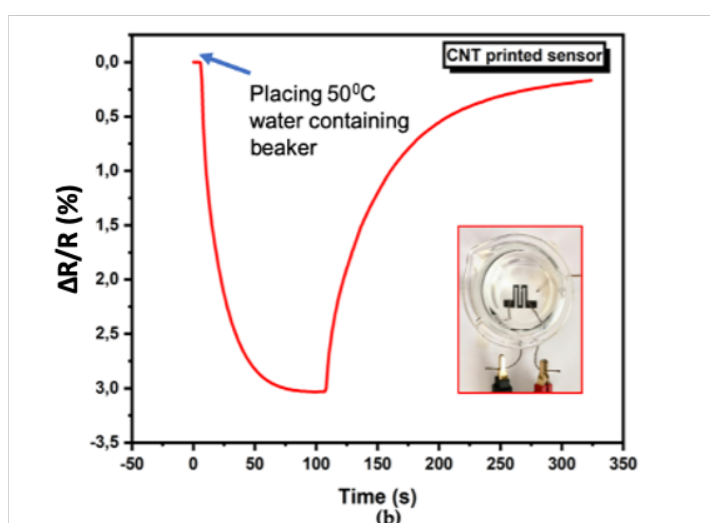


Figure 4.35 : The response of the CNT printed temperature sensor to the temperature under the beaker containing hot water.

4.2.7.5 Mechanical Stability of CNT ink-Type 2 based wearable temperature sensors against bending and folding

For a wearable temperature sensor, a structure that can sustain long-term bending or stretching is required while maintaining its conductivity. For this reason, sensors should be tested for multiple bending or folding cycles (M. Gao et al., 2017). The resistance changes that occurred when the printed sensor was subjected to bending are shown in Figure 4.36-4.38.

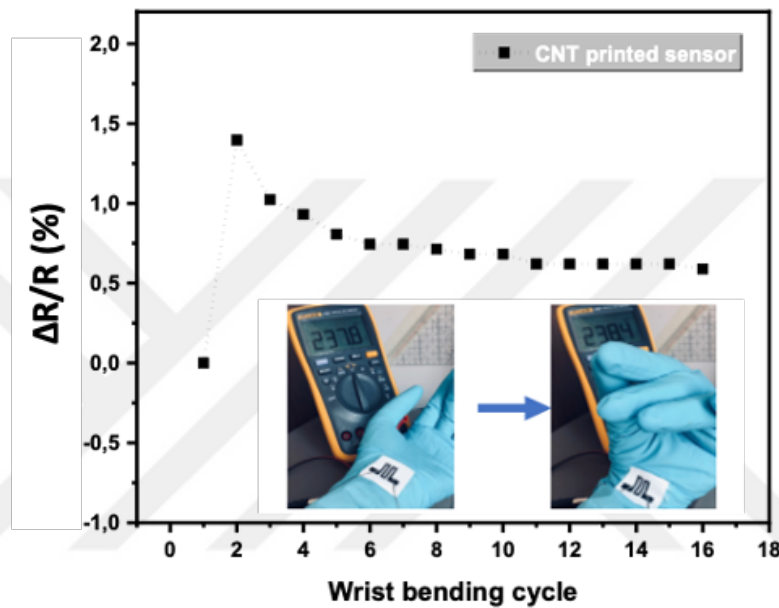


Figure 4.36 : Electrical resistance changes of the sensor during wrist joint movement. The response to wrist twist from 0 ° to about 90 ° was measured.

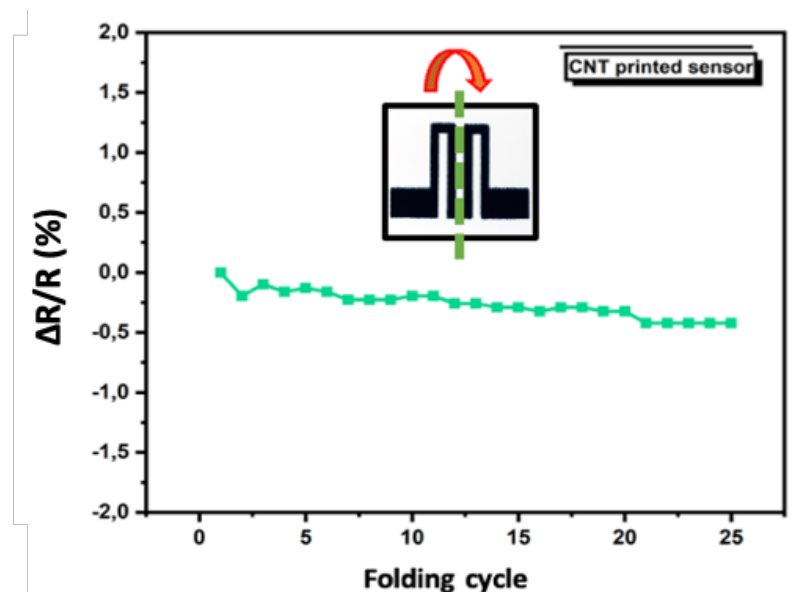


Figure 4.37 : Resistance changes under the folding tests.

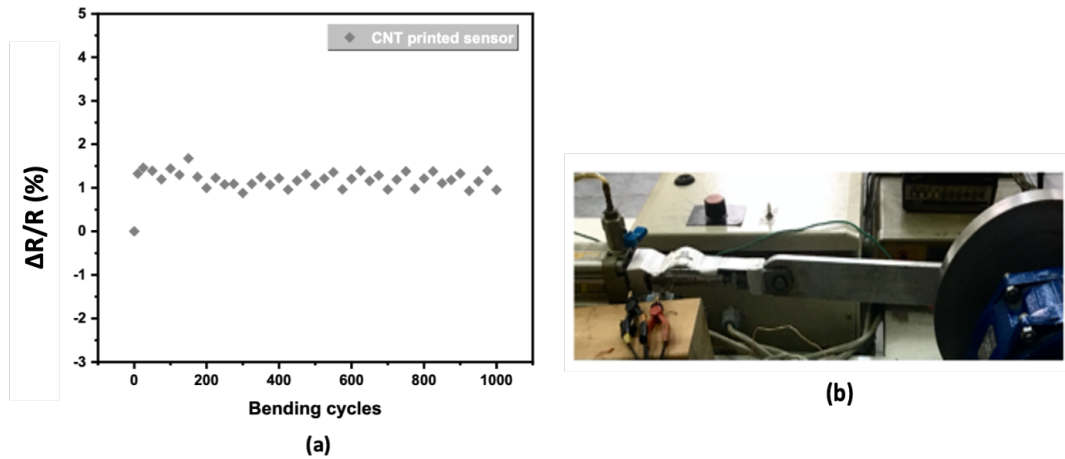


Figure 4.38 : The resistance variations of the sensors repetitive bending tests up to 1000 cycles.

The mechanical stability of the CNT printed temperature sensor was characterized. Resistance change as a function of wrist joint motion is given in Figure 4.36. This graphic shows the resistance changes of the flattened sensor after repeated wrist bending movements. The maximum resistance change value was 0.6% between the baseline and the state after 15 cycles. In addition, multiple folding tests showed the highest resistance change of approximately 0.4% (Figure 4.37). Figure 4.38 demonstrates repetitive bending tests up to 1000 cycles with a bending radius of 6 mm. Tests were performed with a duration of 3 seconds for each bending cycle. The variation of electrical resistance was 0.9 % for CNT printed sensor.

4.2.7.6 Linearity of CNT ink-Type 2 based wearable temperature sensors

The Steinhart equations 4.1 and 4.2 were used to determine the dependence of fitted data and the accuracy of the CNT ink printed temperature sensor measurement. Table 4.12 contains the determined Steinhart coefficients for the printed sensors. The graph indicates how well the computed data for the CNT printed sensor matches the experimental data. The coefficient of determination (R^2) for Figure 4.39 is almost 0.97.

Table 4.12 : Steinhart coefficients of CNT printed sensors calculated at temperatures of 300 K, 308 K and 316 K

	A	B	C
CNT printed sensor	7.84	-0.94	2.0×10^{-3}

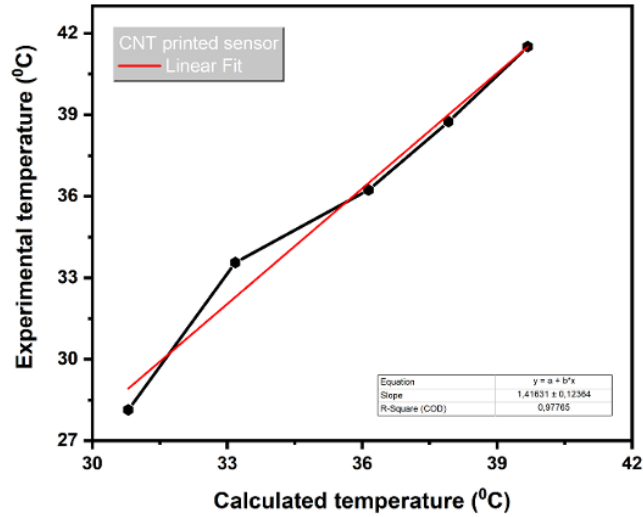


Figure 4.39 : Dependence of experimental data of CNT printed sensor with calculated data from the Steinhart–Hart formula.

4.2.7.7 Temperature measurement performance of PEDOT:PSS ink based wearable temperature sensors

For pure PEDOT:PSS printed sensor, no conductivity is observed until 10-print passes. The obtained resistance of the pure PEDOT:PSS printed sensor was only 3.67 ± 0.48 M Ω for 10-pass printing. The low conductivity of PEDOT:PSS printed sensor was due to the nature of its structure. The conductive PEDOT-rich core region is surrounded by an insulating PSS-rich shell region in the pure PEDOT:PSS printed sensor. Therefore, it is difficult to transport charge carriers on the PEDOT chain through the printed line (Chou et al., 2015).

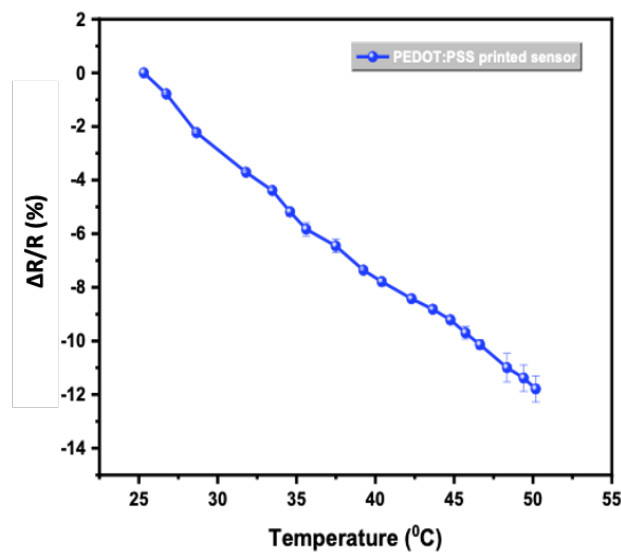


Figure 4.40 : Resistance change as a function of temperature for PEDOT:PSS printed temperature sensors.

The resistance of a 10-pass PEDOT:PSS ink printed sensor was varied from ambient temperature to 50°C to determine its temperature sensing performance. Figure 4.40 illustrates the temperature-resistance change graphs for the PEDOT:PSS ink-based temperature sensor printed on nylon taffeta fabric. TCR value is -0.41%/°C with a standard deviation of 0.03. The electrical resistance of the sensors decreased as the temperature increased, indicating NTC behavior.

Another temperature performance test of the PEDOT:PSS printed sensor is demonstrated in Figure 4.41 by placing a sensor under a beaker containing 50 °C water. As illustrated, sensors first display a high resistance on the Y-axis. Following the beaker's placement on the sensor, an increase in resistance was measured. After cooling the printed sensors to room temperature, the resistance values reach to their initial levels.

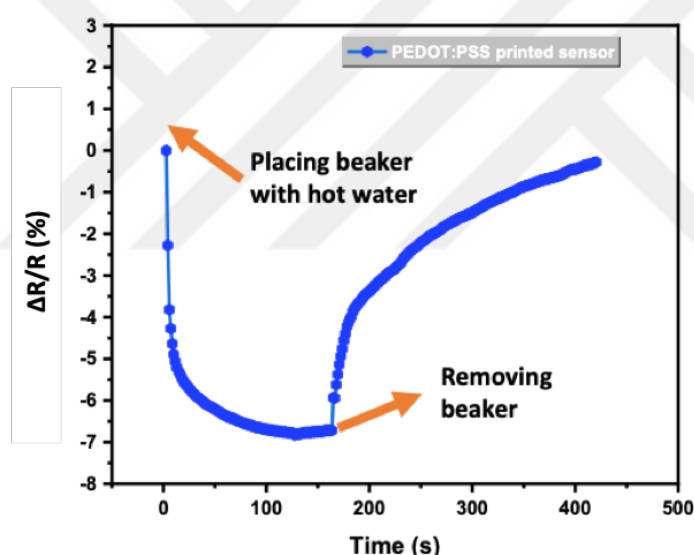


Figure 4.41 : The response of the PEDOT:PSS printed temperature sensor to the temperature under the beaker containing hot water.

4.2.7.8 Mechanical stability of PEDOT:PSS ink based wearable temperature sensors against bending and folding

Figure 4.42-4.44 illustrates the resistance changes that observed when the printed sensor was bent. We investigated the mechanical stability of a PEDOT:PSS-printed temperature sensor.

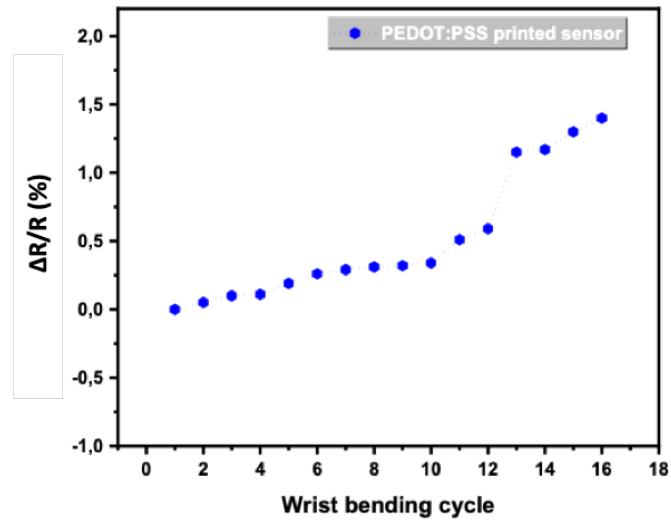


Figure 4.42 : Electrical resistance changes of the sensor during wrist joint movement. The response to wrist twist from 0 ° to about 90 ° was measured.

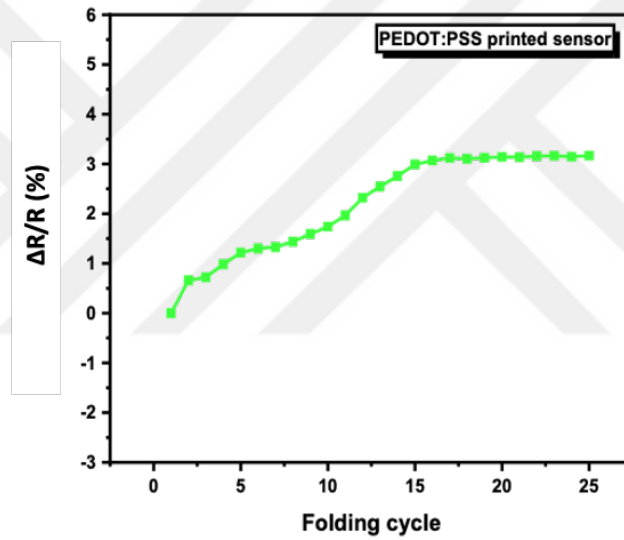


Figure 4.43 : Resistance changes under the folding tests.

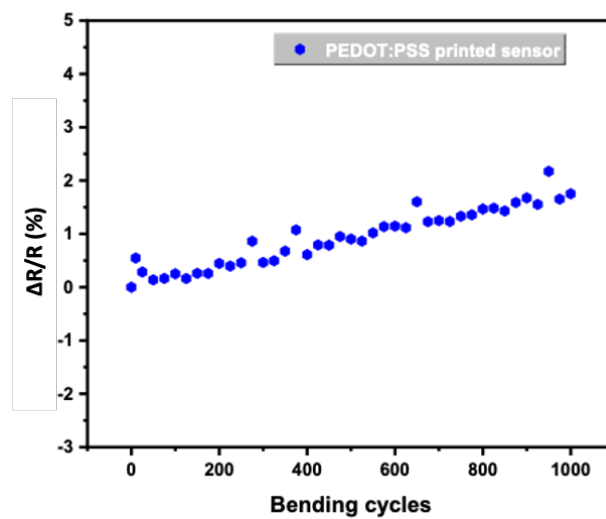


Figure 4.44 : The resistance variations of the sensors repetitive bending tests up to 1000 cycles.

Figure 4.42 illustrates the resistance change as a function of wrist joint motion. The resistance changes in the flattened sensor as a result of repeated wrist bending movements are depicted in this graph. After 15 cycles, the greatest resistance change was 1.4 % between the baseline and the state. Additionally, several folding experiments revealed that the greatest change in resistance is about 3.17 % (Figure 4.43). Lastly, Figure 4.44 illustrates repetitive bending tests with a bending radius of 6 mm up to 1000 cycles. Each bending cycle lasted three seconds. Electrical resistance fluctuation was 1.75 % for the PEDOT:PSS ink printed sensor.

4.2.7.9 Linearity of PEDOT:PSS ink based wearable temperature sensors

The dependence of fitted data and the accuracy of the PEDOT:PSS ink printed temperature sensor measurement were evaluated using the Steinhart equations 4.1. and 4.2. The calculated Steinhart coefficients of the printed sensors are given in Table 4.13. The graph demonstrates that the calculated data for the PEDOT:PSS printed sensor is very close to the experimental data. For Figure 4.45, the coefficient of determination (R^2) is nearly 0.97.

Table 4.13 : Steinhart coefficients of PEDOT:PSS printed sensors calculated at temperatures of 300 K, 308 K and 316 K

	A	B	C
PEDOT:PSS printed sensor	-1.38	0.14	-1.92×10^{-4}

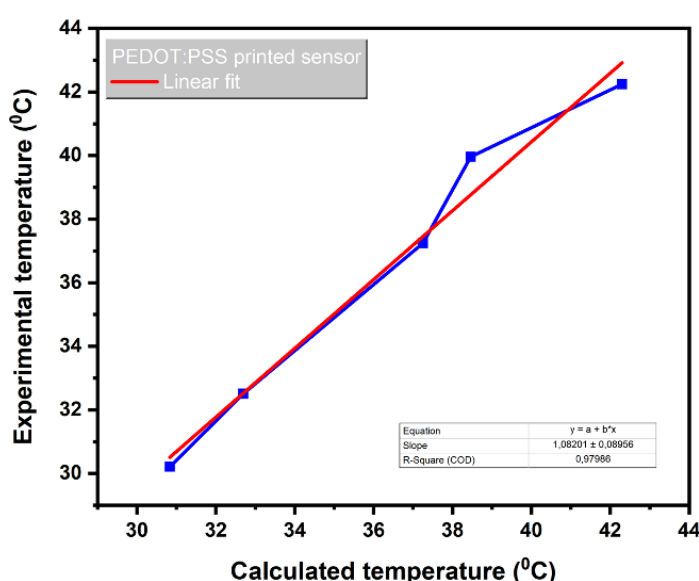


Figure 4.45 : Dependence of experimental data of PEDOT:PSS printed sensor with calculated data from the Steinhart–Hart formula.

4.2.7.10 Temperature measurement performance of CNT ink-Type 2 /PEDOT:PSS composite ink based wearable temperature sensors

Determining the temperature sensing performance of a CNT/ PEDOT:PSS ink-printed sensor was tested from room temperature to 50°C. The temperature-resistance graph for the CNT/PEDOT:PSS ink printed sensor is illustrated in Figure 4.46. Temperature sensor made of CNT/PEDOT:PSS ink and printed on nylon taffeta fabric. The temperature coefficient of resistance is $-0.31\ \%/^{\circ}\text{C}$ with a standard variation of 0.03. As the temperature increased, the electrical resistance of the sensors decreased, confirming NTC behavior.

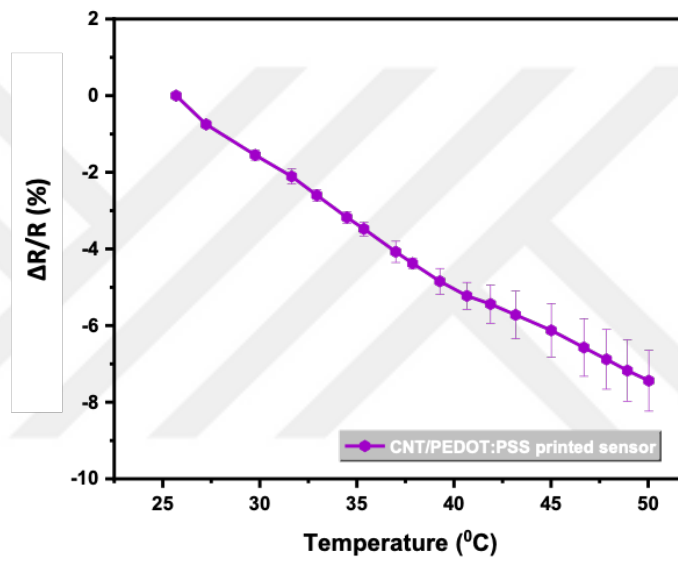


Figure 4.46 : Resistance change as a function of temperature for CNT ink-type 2/PEDOT:PSS printed temperature sensor

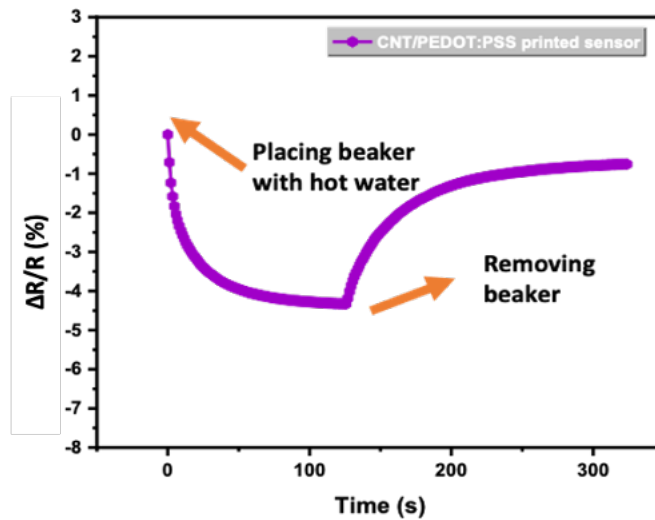


Figure 4.47 : The response of the CNT ink-type 2/PEDOT:PSS printed temperature sensor to the temperature under the beaker containing hot water.

Figure 4.47 exhibits another temperature performance test of the CNT/PEDOT:PSS printed sensor by placing it under a beaker of 50 °C water. As seen, there is a resistance change on the Y-axis. A resistance decrease was observed when the beaker was placed on the sensor. Once the printed sensors are cooled to room temperature, their resistance values return to their initial values.

4.2.7.11 Mechanical stability of CNT ink-Type 2/PEDOT:PSS composite ink based wearable temperature sensors against bending and folding

The resistance changes that were detected when the printed sensor was bent are depicted in Figures 4.48-4.50, respectively. The mechanical stability of a CNT ink-type 2/PEDOT:PSS-printed temperature sensor was examined. The resistance change as a function of wrist joint motion is depicted in Figure 4.48. This graph illustrates the resistance changes in the flattened sensor as a result of repeated wrist bending movements. After 15 cycles, the biggest difference in resistance between baseline and state was 0.3 %. Additionally, multiple folding studies demonstrated that the biggest change in resistance occurs at a rate of approximately 1.15 %. (Figure 4.49). Finally, Figure 4.50 demonstrates repetitive bending tests up to 1000 cycles with a bending radius of 6 mm. Each cycle of bending lasted three seconds. The electrical resistance fluctuation was 0.3 % for the sensor printed with CNT/PEDOT:PSS ink.

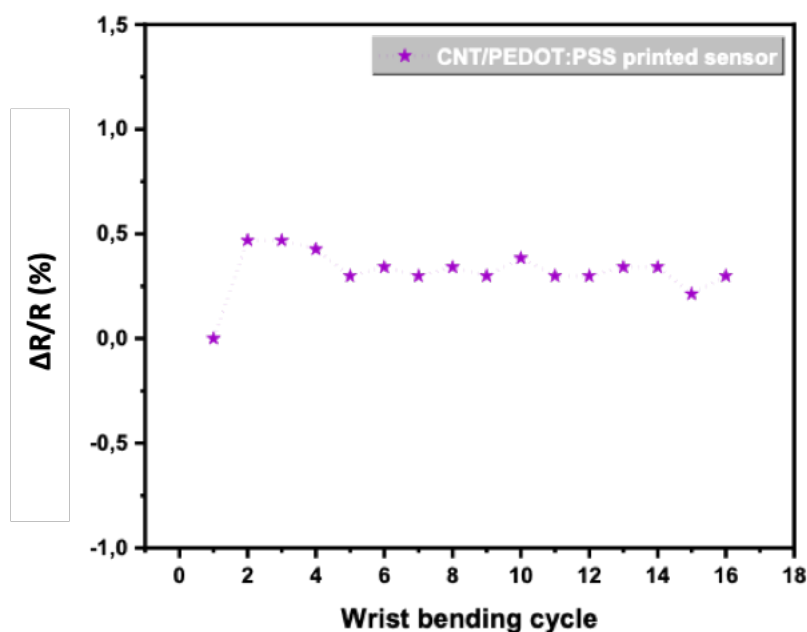


Figure 4.48 : Electrical resistance changes of the sensor during wrist joint movement. The response to wrist twist from 0 ° to about 90 ° was measured.

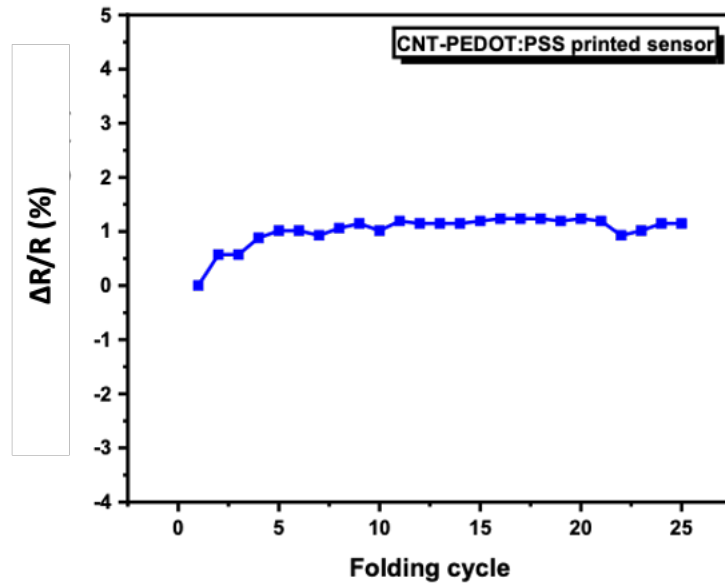


Figure 4.49 : Resistance changes under the folding tests.

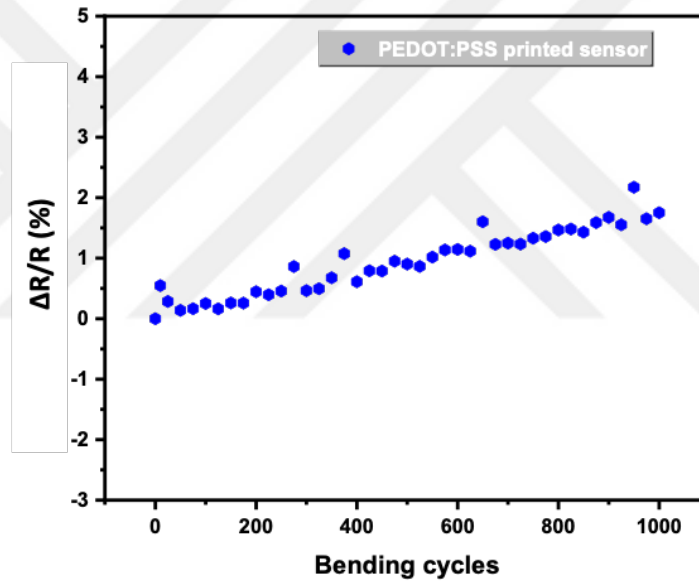


Figure 4.50 : The resistance variations of the sensors repetitive bending tests up to 1000 cycles.

4.2.7.12 Linearity of CNT ink-Type 2/PEDOT:PSS composite ink based wearable temperature sensors

The Steinhart Equations 4.1 and 4.2 were used to compare the dependence of fitted data and the accuracy of the CNT ink-type 2/PEDOT:PSS ink printed temperature sensor measurement. Steinhart coefficients calculated in Table 4.14 are given in the calculation above. The graph shows that the CNT ink-type 2/PEDOT:PSS printed sensor's predicted data better approximates the experimental data. R^2 is about 0.99 for Figure 4.51.

Table 4.14 : Steinhart coefficients of CNT ink-type 2/PEDOT:PSS printed sensors calculated at temperatures of 300 K, 308 K and 316 K

	A	B	C
CNT/PEDOT:PSS printed sensor	1.44	-0.17	3.78×10^{-4}

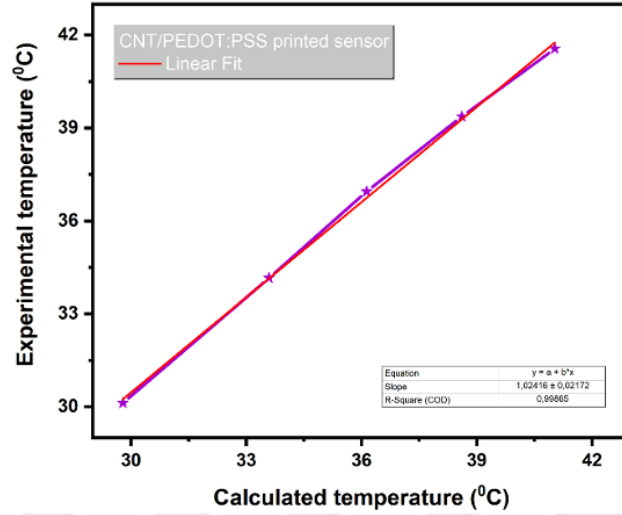


Figure 4.51 : Dependence of experimental data of CNT ink-type 2/PEDOT:PSS printed sensor with calculated data from the Steinhart–Hart formula.

4.2.7.13 Comparison of all type of fabricated sensors with regard to temperature measurement performance

The CNT network in PEDOT:PSS polymer matrix increases the circulation of electric charge due to “ π - π interaction” between the thiophene ring and carbon nanotubes, hence resulting in an increase in conductivity (Z. Chen et al., 2014; Denneulin et al., 2009). Hence, the electrical resistance of the pure PEDOT:PSS printed sensor decreased to $242.6 \pm 21 \text{ k}\Omega$ drastically after the contribution of CNT into the ink.

As illustrated in Figure 4.52 and Figure 4.53, the temperature-sensing properties of 10-pass printed sensors were characterized by measuring the variance of resistance at a regulated temperature on a hot plate from room temperature to 50 °C. Figure 4.52 exhibits the normalized resistance change where each sensor resistance is normalized to its initial resistance value at room temperature. The average TCR value is $-0.41\%/^{\circ}\text{C}$ with a standard deviation of 0.03 for pure PEDOT:PSS printed sensor. For the pure CNT and CNT/PEDOT:PSS printed sensors, TCR values were $-0.15 \pm 0.02 \text{ } \%/^{\circ}\text{C}$, and $-0.31 \pm 0.03 \text{ } \%/^{\circ}\text{C}$, respectively. The higher sensitivity of the composite ink printed sensor than the pure CNT printed sensor is most probably attributed to the electron hopping at the CNT and PEDOT: PSS interface (Honda et al., 2014b; Soni et al., 2020).

The TCR value of composite ink printed sensor is better than some CNT-based temperature sensors previously reported in the literature (Bali et al., 2016; Turkani et al., 2018; Vuorinen et al., 2016)

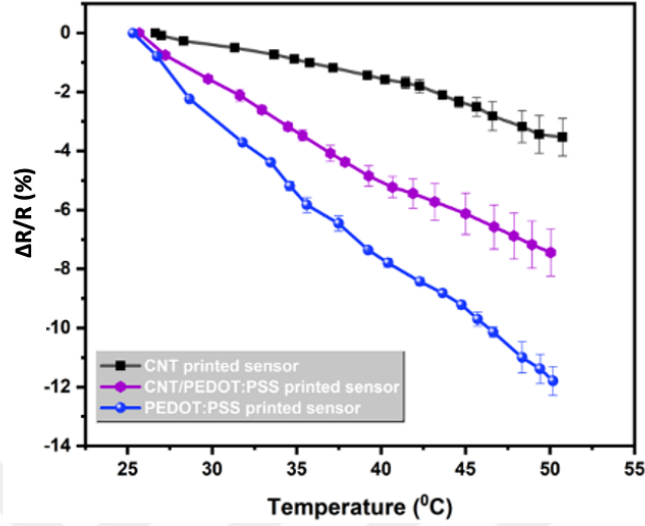


Figure 4.52 : Resistance changes as a function of temperature for printed temperature sensors.

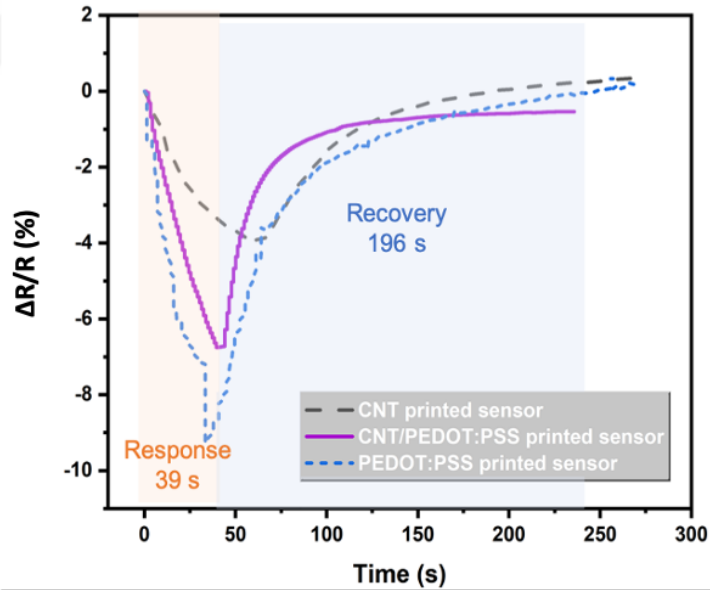


Figure 4.53 : The real-time response curves of printed sensors.

The recovery time was also evaluated by comparing sensors as shown in Figure 4.53. The fabricated CNT/PEDOT:PSS sensor exhibited a response of ~39 s and a recovery of ~196 s from room temperature to 50°C. The performance of composite ink printed sensors is better than the pure CNT and PEDOT:PSS printed sensors. The longer recovery time of sensors is possibly due to the large heat capacitance of the fabric substrate.

Figure 4.54 shows a temperature sensing test by placing a sensor under a beaker containing 50 °C water. It can be seen in the figure; initially, sensors exhibit high resistance on the Y-axis. After the placement of the beaker on the sensor, an increase in resistance change was observed. Printed sensors were removed to room temperature and the resistance values returned to the initial values.

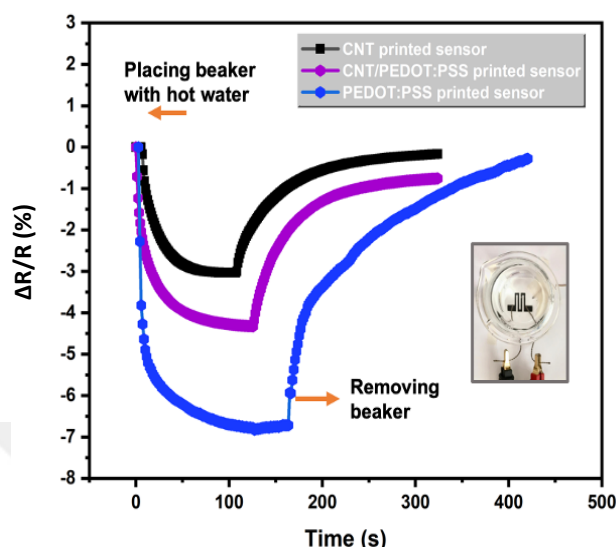


Figure 4.54 : The response of the CNT, PEDOT:PSS and CNT/PEDOT:PSS printed sensors to the temperature under a beaker containing hot water.

Repeatability

The thermal camera image of the experimental configuration is shown in Figure 4.55. Figure 4.56 (a-c) shows the repetitive temperature sensing test of the sensors with a gloved human finger in the ambient room environment between 25 °C and the finger temperature on the sensor (an equilibrium temperature of ~ 32).

The resistance of the sensors decreases with tapping the sensor with the finger tip. After repeated taps, the resistance of the CNT/PEDOT:PSS printed sensor recovers to the initial values, revealing better repeatability and consistency than the pure CNT and PEDOT:PSS printed sensors. The response time to the tapping was about 10 s, and the recovery time to its initial resistance value upon withdrawing the finger was nearly 105 s for the CNT/PEDOT:PSS printed sensor.

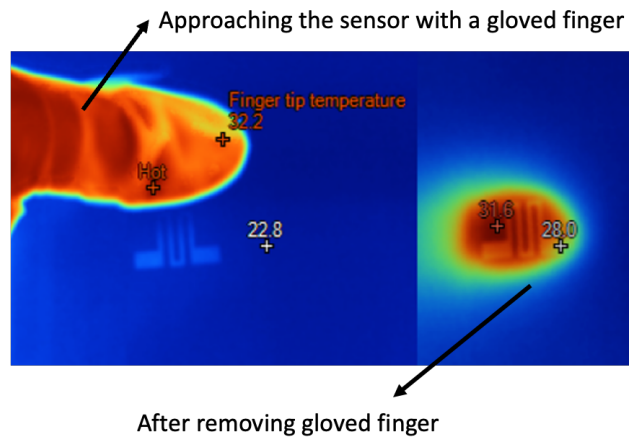


Figure 4.55 : Thermal camera image of experimental configuration.

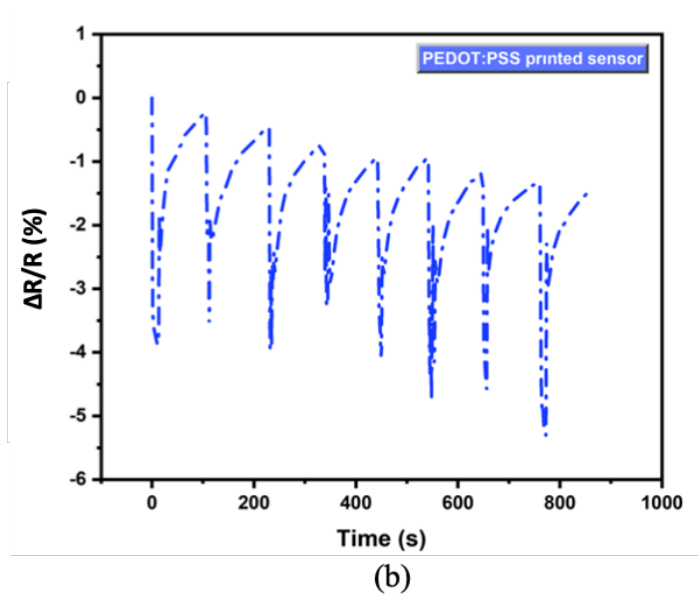
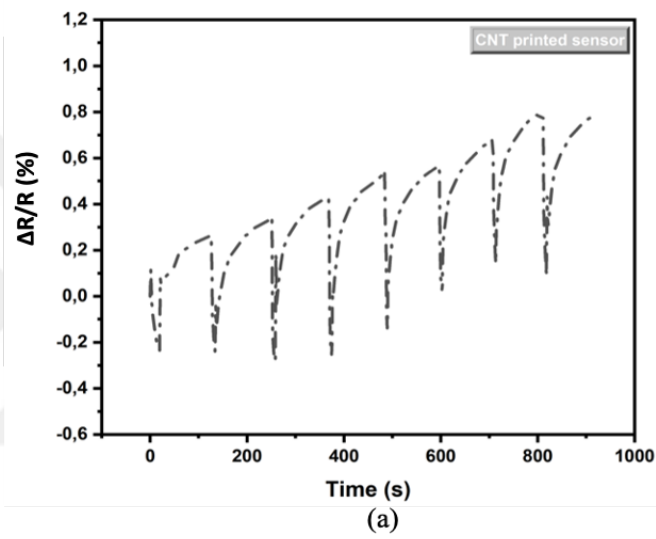


Figure 4.56 : Thermal response graphs of the (a) CNT, (b) PEDOT:PSS, and (c) CNT/PEDOT:PSS printed sensors when the finger touches and withdraws from the sensor.

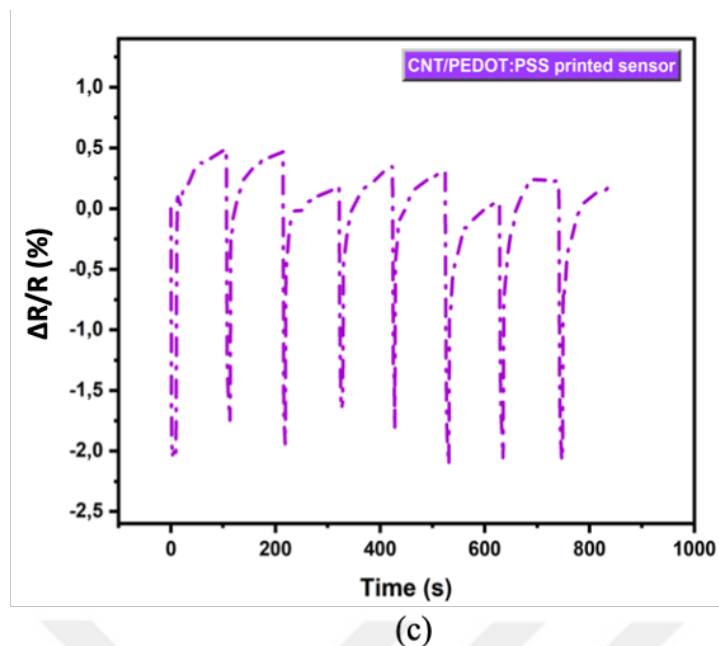


Figure 4.56 (continued) : Thermal response graphs of the (a) CNT, (b) PEDOT:PSS, and (c) CNT/PEDOT:PSS printed sensors when the finger touches and withdraws from the sensor.

The effect of humidity

The change in electrical resistance of printed sensors as a function of temperature at different relative humidity is also shown in Figure 4.57. The electrical conduction mechanism of the CNTs is dominated by the holes. When the humidity is increased, some of the water molecules are adsorbed onto the sensor. As electrons from water molecules pass to MWCNTs, the holes in MWCNTs are reduced and, therefore, conductivity decreases (X. Zhang et al., 2020). PEDOT:PSS is a water-soluble polymer and it is highly affected by environmental humidity. PSS shells can swell due to the absorption of water molecules, resulting in greater distances between adjacent PEDOT enriched cores, thereby increasing film resistance. On the other hand, the decrease in resistance will be generated by moisture desorption. This would cause the measurement of temperature to be unreliable (Y.-F. Wang et al., 2020). When humidity is increased to 85% RH, the TCR of PEDOT:PSS printed sensor increases to $-1.18\% \text{ }^{\circ}\text{C}^{-1}$ between $25 \text{ }^{\circ}\text{C}$ and $50 \text{ }^{\circ}\text{C}$ (Figure 4.58). The humidity effect on CNT/PEDOT:PSS printed sensors is low compared to the pristine PEDOT:PSS and pure CNT printed sensors when the relative humidity level is increased from 65% to 85%. Therefore, it may be more adequate to use CNT and CNT/PEDOT:PSS based printed sensors for insensitive humidity applications.

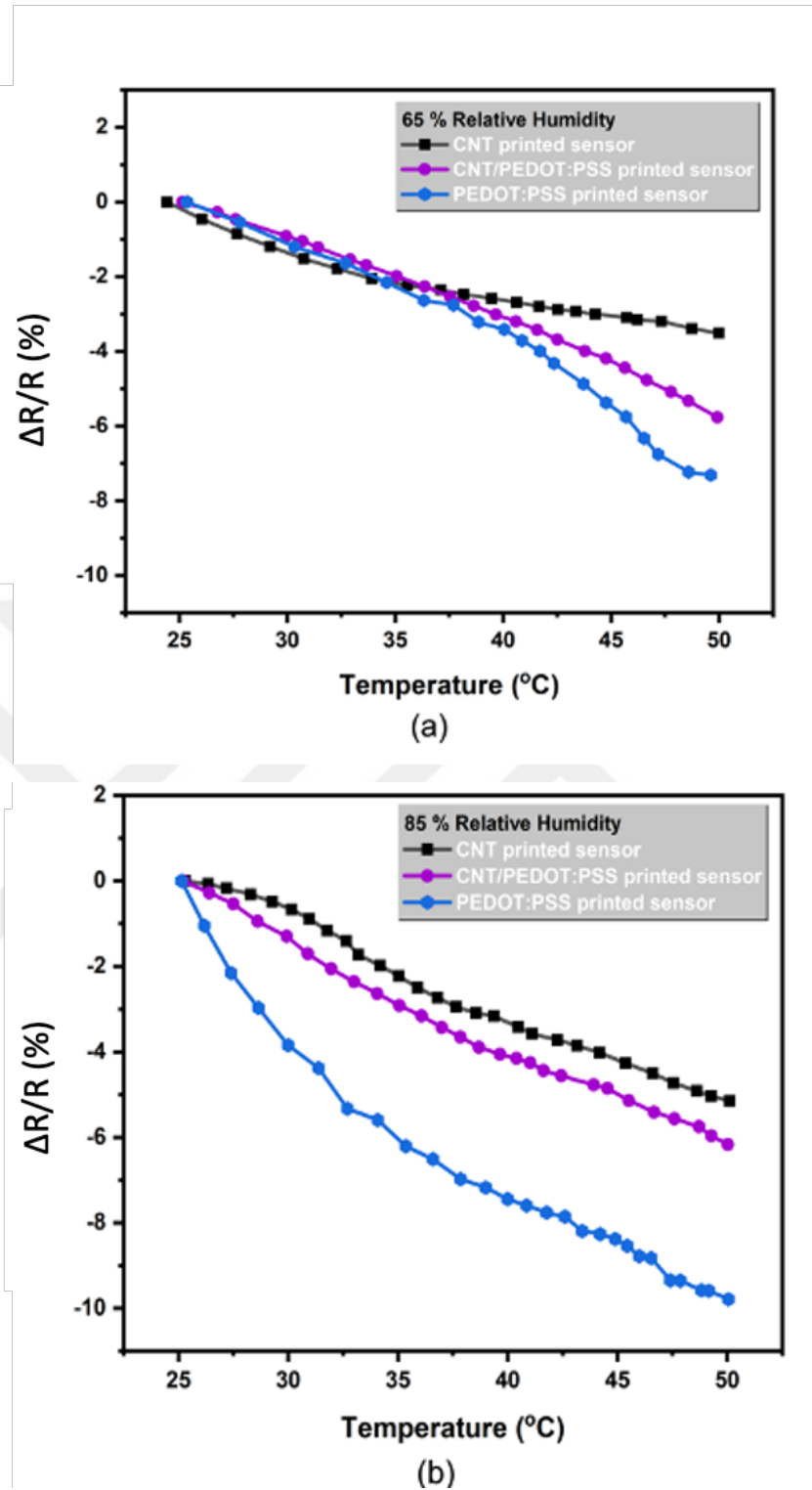


Figure 4.57 : Normalized resistance change as a function of temperature for CNT, PEDOT:PSS and CNT/PEDOT:PSS ink printed sensors (a) at 65 % RH, and (b) at 85 % RH.

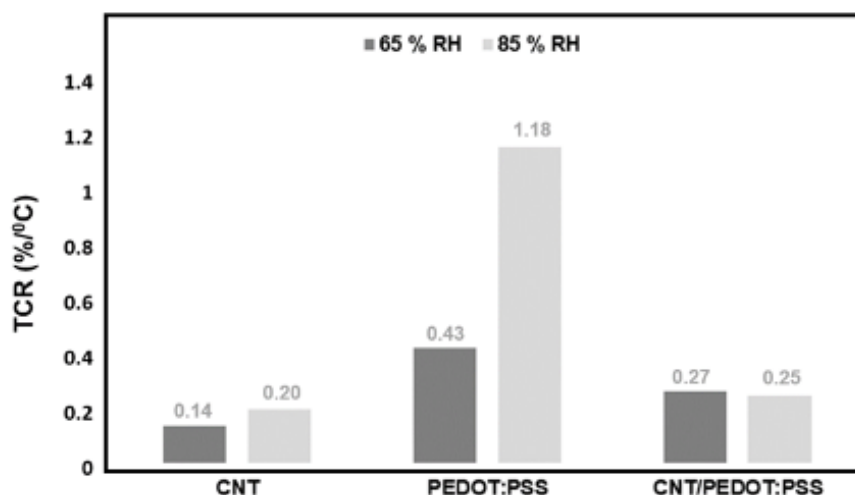


Figure 4.58 : TCR values of printed sensors at 65 % RH, and at 85 % RH.

4.2.7.14 Comparison of all type of fabricated sensors with regard to mechanical performance against bending and folding

The resistance changes that occurred when the CNT, PEDOT:PSS and CNT/PEDOT:PSS printed sensors were subjected to wrist joint bending are shown in Figure 4.59. This graphic exhibits the resistance changes of the flattened sensor after repeated wrist bending movements. The maximum resistance change values between the baseline and the state after 15 cycles were found to be 0.6 %, 0.3 %, and 1.4 % for CNT, CNT/PEDOT:PSS, and PEDOT:PSS printed sensors, respectively. Figure 4.60 exhibits repetitive bending tests up to 1000 cycles with a bending radius of 6 mm. Tests were performed with a duration of 3 seconds for each bending cycle. The variation of electrical resistance was 0.9 %, 0.3 %, and 1.75 % for pure CNT, CNT/PEDOT:PSS, and pure PEDOT:PSS printed sensors, respectively. The higher stability in resistance after repetitive bending cycles was observed for the CNT/PEDOT:PSS printed sensor. The pure PEDOT: PSS printed sensor behaved unstable in repetitive bending tests. Although the electrical conductivity of the pure PEDOT:PSS printed sensor is not totally lost after bending cycles, the ink on the substrate is presumed to deform the surface coating after bending tests. The enhanced stability of the CNT/PEDOT:PSS printed sensor on bending is due to the wrapping of PEDOT:PSS around the CNT junctions that prevents the CNT from separation. Furthermore, this also reveals the better adhesion of the ink to the fabric surface (Mannayil et al., 2018).

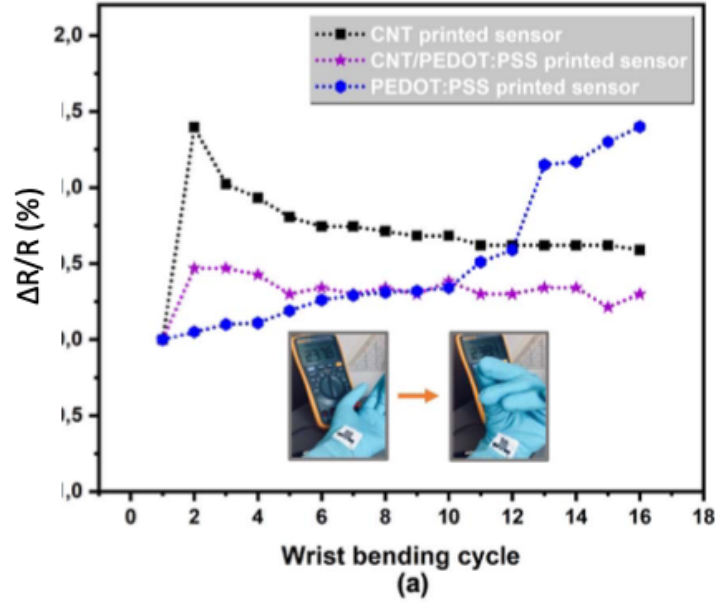


Figure 4.59 : The electrical resistance changes of sensors during the wrist joint motion (Inset: photograph of the wrist joint motion).

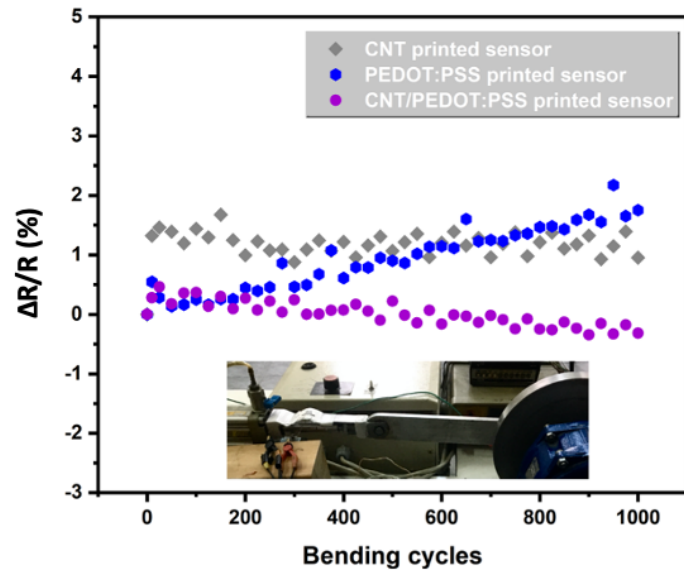


Figure 4.60 : The resistance variations of the sensors repetitive bending tests up to 1000 cycles (Inset: test set-up).

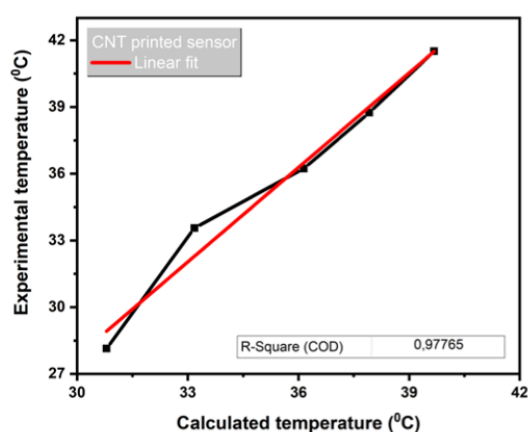
4.2.7.15 Comparison of all type of fabricated sensors with regard to linearization

In Figures 4.61a, 4.61b and 4.61-c experimentally measured temperature versus Steinhart–Hart equation based calculated temperature comparisons are plotted respectively for (a) pure CNT, (b) pure PEDOT:PSS and (c) CNT/PEDOT:PSS printed sensors. A, B, and C are Steinhart coefficients (Table 4.15) used to calculate temperature associated resistance. It can be seen from the graphs that the calculated

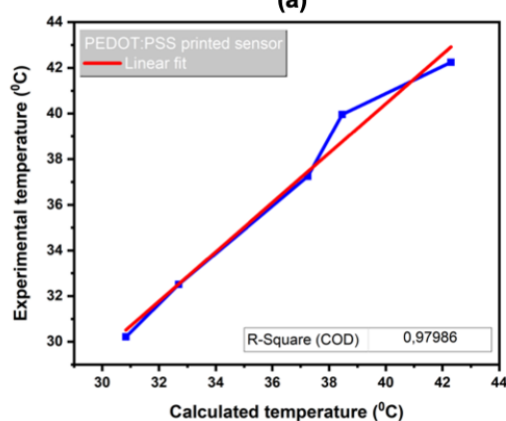
data of the CNT/PEDOT:PSS printed sensor almost matches the experimental data with R^2 of 0.99. The coefficient of determination value (R^2) for all type of sensors are higher than 0.97 as shown in Figures 4.60(a-c). The combined total error due to electric resistance measurement, temperature measurement and curve-fitting is within a very good experimental limit based on the comparison here.

Table 4.15 : Steinhart coefficients of printed sensors calculated at temperatures of 300 K, 308 K and 316 K

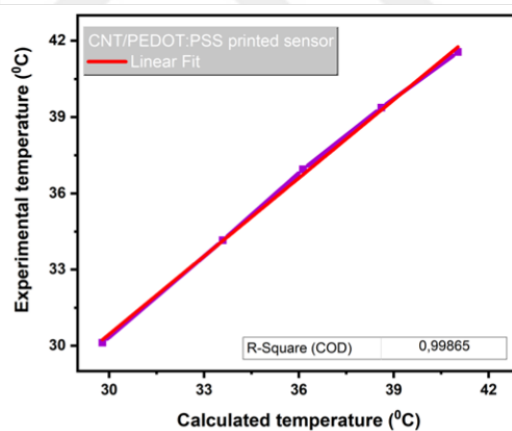
	A	B	C
CNT printed sensor	7.84	-0.94	2.0×10^{-3}
PEDOT:PSS printed sensor	-1.38	0.14	-1.92×10^{-4}
CNT/PEDOT:PSS printed sensor	1.44	-0.17	3.78×10^{-4}



(a)



(b)



(c)

Figure 4.61 : Dependence of experimental data of (a) pure CNT, (b) CNT/PEDOT:PSS, and (c) pure PEDOT:PSS printed sensors with calculated data from the Steinhart–Hart formula.

4.2.7.16 Conclusion

Hereby, we propose three kinds of resistive temperature sensors with sensitive materials, including CNT (ink-Type 2 and ink-Type 2), PEDOT:PSS ink and CNT/PEDOT:PSS composite ink. The prepared CNT ink-Type 1 gives promise for printed sensor applications. However, clogging problems have occurred in the inkjet printing machines used over the last two months. Therefore, additional processes, including centrifugation filtration for ink formation, were applied, and also small length CNT was used to avoid inkjet nozzles from clogging. Therefore, the progress in CNT ink was further continued by development of CNT ink-Type 2 formulation.

To determine the most appropriate material for obtaining reliable, sensitive, and flexible sensors, sensors manufactured by using inks based on CNT ink-Type 2, PEDOT:PSS, and CNT ink-Type 2/PEDOT:PSS were compared. The CNT/PEDOT:PSS printed sensor showed the lowest electrical resistance of $242.6 \pm 21 \text{ k}\Omega$ than pure CNT ink-Type 2 and pure PEDOT:PSS printed sensors. The most robust performance was obtained with the CNT ink-Type 2/PEDOT:PSS printed temperature sensor. The sensor possessed a temperature sensitivity of $-0.31 \pm 0.03 \text{ }^\circ\text{C}^{-1}$ from room temperature to 50°C . The TCR values of CNT ink-Type 2/PEDOT:PSS printed sensors were found to be more humidity stable than CNT ink-Type 2 and PEDOT:PSS printed sensors. The ability to bend is also critical for real-time applications of temperature sensors. CNT ink-Type 2/PEDOT:PSS printed temperature sensor exhibits good mechanical stability. It demonstrates a resistance change of 0.3 % after repetitive bending up to 1000 cycles. In addition, the effect of bending can be minimized by placing these wearable sensors on the torso area of the garment.

4.2.8 Results and discussion for wearable temperature sensor's wear and usability performance

Wearable sensors have the potential to be regularly exposed to bending, humidity, abrasion, washing, light, and also perspiration light. Because of this, it is crucial to improve the lifespan of wearable sensors. However, there is no way to prevent exposure. This, along with the excellent fastness exhibited in their sensor performance, was conducive to their use in the textile industry, as well as being incorporated into

the creation of wearable sensors for industrial manufacturing. Combined with a lack of standardized and consistent production techniques, there is additionally a deficiency of regular and comparable tests that may be used during the development and implementation processes for wearable e-textile sensors. In particular, this creates difficulties for industrialization in this area. However, in light of previous studies, I utilized some characterization methodologies with some modifications to investigate the reliability of sensors. Therefore, sensor performance was studied to identify potential applications before the integration of printed sensors.

We utilized the Gretag Macbeth Spectralight instrument to examine the effects of light exposure. To determine the temperature sensor's performance durability, it is placed in a light box for up to eight hours.

Crockmeter equipment was used for the rubbing tests, which were carried out in dry conditions. The device was crocked back and forth 20 times by turning it ten times completely.

Multiple printed temperature sensors were inserted in a humidity cabinet and exposed to different relative humidity (RH) values while the temperature varied between 25 and 50°C.

The washing process was carried out in the laboratory using a domestic washing machine (BEKO 9102E). In the test, we used ECE type B detergent. A total of 20 grams of detergent and 2.5kg ballasts were used for each washing cycle. Wool washing (also known as "gentle") is done at a wash temperature of 30°C and a wash time of 40 minutes. Express washing, however, includes a short process (at a wash temperature of 30°C, for 16 minutes) (uz Zaman et al., 2019).

Acidic and basic perspiration experiments were performed to determine if there was a difference in electrical resistivity following perspiration exposure. Besides that, water was syringed onto the encapsulated sensor and the resistance was measured to determine if there was a change in resistance.

Although, CNT/PEDOT:PSS composite ink exhibited relatively better performance than pure CNT and PEDOT:PSS printed sensors, PEDOT:PSS based inks create manufacturing problems such as nozzle clogging for inkjet printing due to required storage temperature 2-8°C. Therefore, considering manufacturing issues in terms of easy processing and having low-cost, we decided to continue with pure CNT ink Type-2 in order to test wear and usability performance of the fabricated sensors, which has also exhibited comparable performance. The developed CNT ink printed sensor is

potentially useful for applications in continuous measurement of human body temperature.

4.2.8.1 Humidity effect

Figure 4.61 illustrates the change in electrical resistance of printed sensors as a function of temperature and relative humidity. While the TCR of the CNT printed sensor was -0.15%/C at 65 % relative humidity, it was -0.21 %/C at 85 % relative humidity.

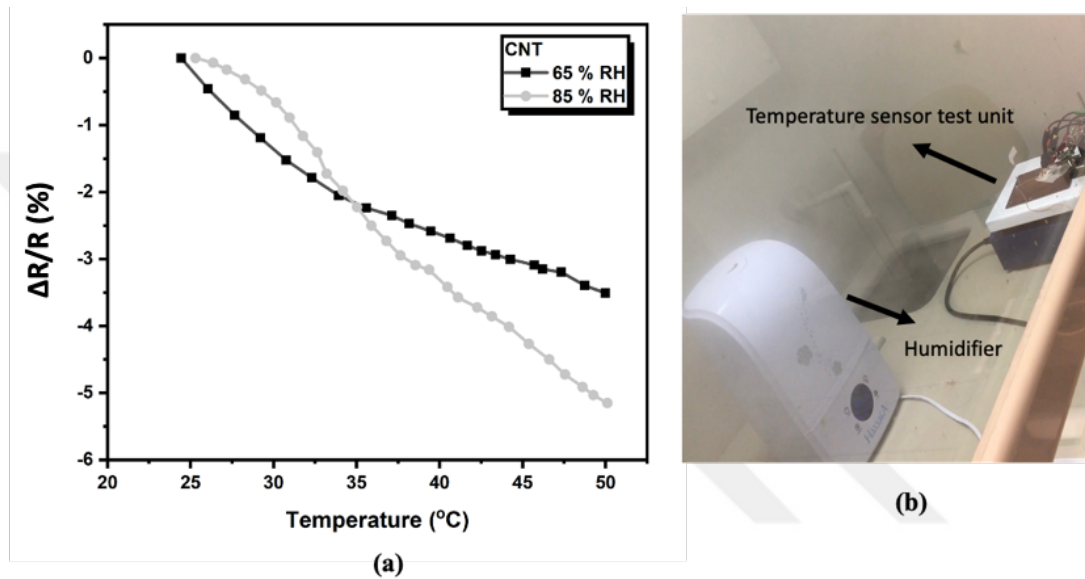


Figure 4.62 : (a) Resistance change as a function of temperature for CNT printed sensors at 65 % RH, and at 85 % RH. (b) Humidity test cabin.

Hydrophilic and hydrophobic materials have various impacts on the quantity of humidity on different materials (Fukuda et al., 2019). CNTs' electrical characteristics are related to charge transfer and doping. The density of the charge carriers in the CNT can vary due to interactions with molecules that have negatively charged molecules or molecules that have positive charges. A change in electrical resistance due to humidity can be an outcome of these interactions (Shooshtari et al., 2020).

The electrical conduction mechanism of the CNTs is dominated by the holes. When the humidity is increased, some of the water molecules are adsorbed onto the sensor from the backside of the fabric. As electrons from water molecules pass to MWCNTs, the holes in MWCNTs are reduced and, therefore, conductivity decreases (X. Zhang et al., 2020).

In addition, the functionalization of MWCNTs with acid generated defects that resulted in an increased number of nucleation sites, which in turn lead to good moisture

sensor sensitivity (Jung et al., 2014). Since the compensating doping effect was weakened, more water molecules may be adsorbed on the sensor during the acid treatment process (C. Wang et al., 2019; J. Zhang et al., 2019).

4.2.8.2 Resistance to rubbing

Subsequently, rubbing also effects negatively flexible textile-based sensors's surfaces worn on the body. The crocking test is utilized to identify the degree of electrical resistance change that occurs in inkjet printed sensors as a result of the rubbing. After 20 times of crocking, the resistance of the printed sensors had changed by nearly 3.5 % from their initial value. In accordance with Figure 4.62, after the rubbing test, there were some resistance irregularities that increased in correlation with the temperature. Even through, the TCR value is nearly similar to each other. The TCR of the sensor after crocking test is found as $-0.19 \pm 0.03 \text{ \%}/\text{C}$.

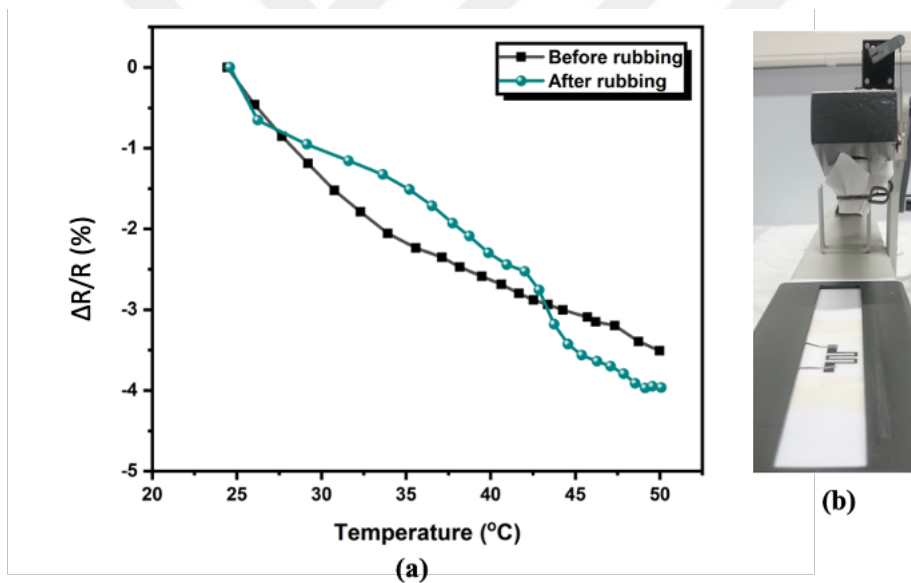


Figure 4.63 : (a) Resistance changes as a function of temperature for CNT printed sensors before and after rubbing test. (b) Crocking test unit.

4.2.8.3 Washing performance

The findings given in Figure 4.63 reveal that laundering damages the printed area. While the printed sensors surface remained conductive, they were unacceptably high in resistance. The variation in measured resistance and TCR values seen in Figure 4.63 is most likely attributable in part to the improper contact methods utilized with printed patterns. Due to the conductive thread integration, water leaking within the encapsulated sensor may cause the connections to not accurately represent the measured electrical resistance. The physical adhesion might be formed between both

the $-NH$ and $C=O$ groups of the polyurethane matrix and the $-COOH$ group of MWNTs due to hydrogen bonding (Barick & Tripathy, 2011). The decrease in conductivity may be attributed to partial separation of polyurethane welding tape together with CNT in small quantities from the fabric surface during the washing process.

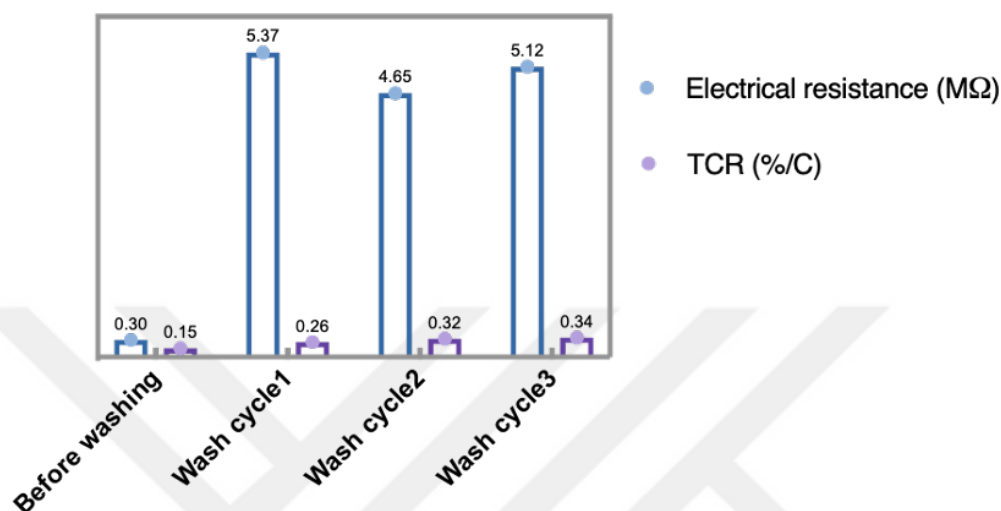


Figure 4.64 : Change in resistance and TCR values with washing processes.

4.2.8.4 Durability to ultraviolet radiation

Besides humidity effect, rubbing resistance, and washing performance, light fastness is another factor that should be taken into consideration when developing a textile-based wearable device. Figure 4.64 demonstrates that the electrical resistance and TCR values of the CNT printed sensors exposed to UV light after 8 hours of measurement are nearly the same as the relative resistance change of the CNT printed sensor prior to the experiment, indicating that the sensor has good light durability.

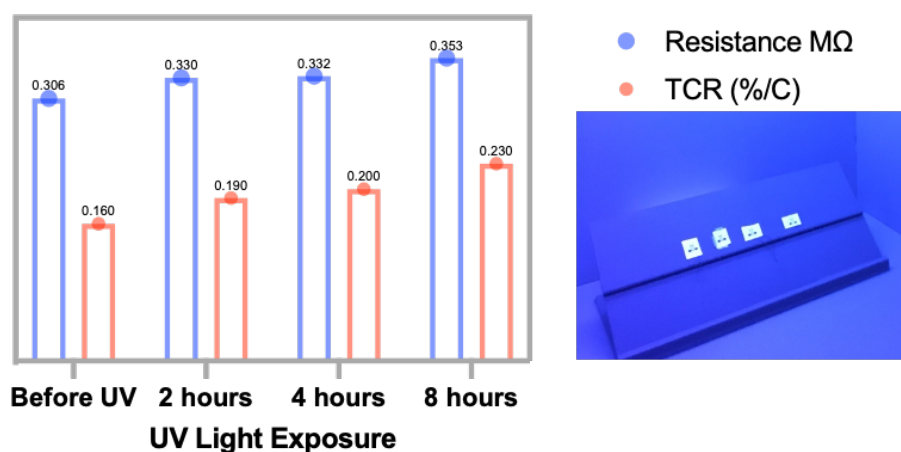


Figure 4.65 : Light durability of CNT printed sensor.

4.2.8.5 Durability to human sweat

There was a considerable change in the electrical resistance of the CNT printed fabric in relation to the two solution types used in the experiment, including acid perspiration solution, and basic perspiration solution. As can be seen from Figure 4.65, resistance change is very similar for both untreated CNT printed fabric and treated CNT printed sensor in basic solution. However, there is a huge increase in electrical resistance before and after perspiration tests. Electrical resistance increases from 300 to 480 k Ω for after acidic treatment, and to 750 k Ω for after basic treatment. Therefore, it is much more suitable for our sensors to be placed in body regions which are less affected by human body sweat (Table 4.16).

Table 4.16 : The density of sweat glands in various body regions (Wilke et al., 2007).

Body parts	Sweat gland density (in cm ⁻²)
Palm	644
Forearm	134
Abdomen	127
Upper arm	90
Armpit	68
Thigh	57
Face	59
Chest	20

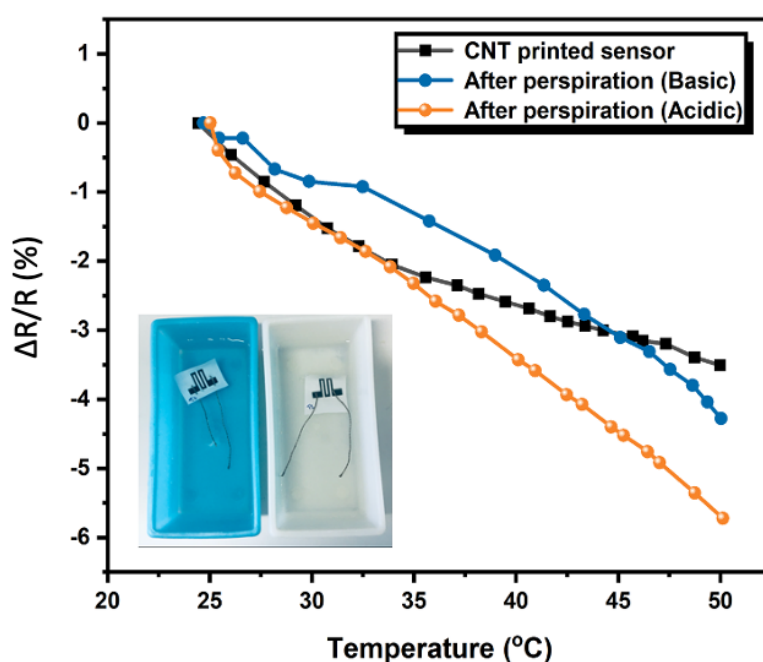


Figure 4.66 : Perspiration effects on temperature sensor performance.

4.2.8.6 Conclusion

Hereby, it is reported that the textile-based temperature sensors have been fabricated easily, with low cost a fast-prototyping process in which they are created via an ink-jet printing. Carbon nanotube and PEDOT:PSS based inks were directly implemented onto woven fabric via an inkjet printing technique to produce a flexible temperature sensor. This process is simple, efficient, inexpensive, and scalable, and, more importantly, preserves the textile's sensory and quality properties, including durability and comfort.

In addition, the effect of mechanical and perspiration can be minimized by placing these wearable sensors on the torso area of the garment. This sensor was intended to have simple and affordable fabrication capabilities. Because of this, it may be attached to a garment and utilized as a disposable sensor. In this direction, we presented that nanomaterial ink-printed textiles can be fabricated via low-cost inkjet printing technique in order to be used as wearable sensors .

4.2.9 Results and discussion for wearable sensor real-time measurement

Currently, the most attractive feature of the wearable sensors is to provide remote monitoring of patients' body temperature in regions where medical care facilities are not readily accessible. As a result, precision of measurement is critical for such applications. To demonstrate our developed sensor's accuracy, we measured the temperature on a mannequin and gloved hand with the developed inkjet printed sensor.

4.2.9.1 Measurement on a mannequin & results

After attaching the sensor to the torso of a mannequin, an experimental study was performed to check its reliability in real-life situations. Once it was stuck on the cloth, the resistance value was accurately recorded (Figure 4.66). The value of the electrical resistance reading of the CNT ink printed sensor on the mannequin is 25.59 °C, while the value of the temperature obtained by the Omega TT-K-30-SLE(ROHS) type thermocouple is 25.54 °C, and the Fluke 17+ thermocouple is 25.90 °C. It should be noted that the Fluke 17+ thermocouple has an accuracy of $2\% \pm 2\text{ }^{\circ}\text{C}$ between 0 and 50°C. But, the Omega TT-K-30-SLE(ROHS) type has an accuracy of 1 °C. When compared with the Omega TT-K-30-SLE(ROHS) type thermocouple, the developed wearable sensor shows approximately the same value. The resulted relative difference

of the accuracy of the developed sensor with respect to Omega TT-K-30-SLE(ROHS) type thermocouple is found to be 0.05 °C.

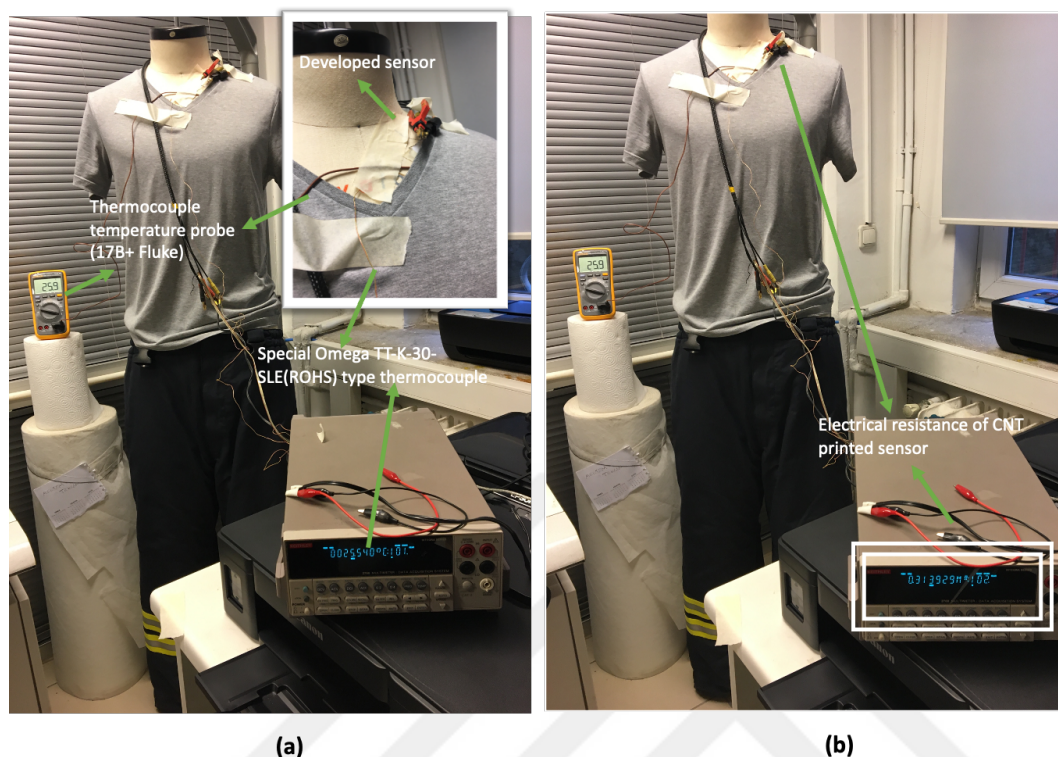


Figure 4.67 : (a) Photograph of temperature values of two different thermocouples on mannequin skin (Inset: magnified photograph showing placement of sensors). (b) Exhibition of electrical resistance value of the our developed temperature sensor on mannequin skin.

4.2.9.2 Measurement on a human body: gloved hand & results

The developed wearable sensor attached to the gloved hand of a volunteer creates a stable body temperature that can be measured (Figure 4.67). The value from the electrical resistance reading of the CNT ink printed sensor on the gloved hand is 31.71 °C, while the value obtained by the Omega TT-K-30-SLE(ROHS) type thermocouple is 31.72 °C, and the Fluke 17+ thermocouple is 32.2 °C. When compared to the Omega TT-K-30-SLE(ROHS) type thermocouple, the developed wearable sensor displays a value that is nearly identical. The resulted relative difference of the accuracy of the developed sensor with respect to Omega TT-K-30-SLE(ROHS) type thermocouple is found to be 0.01 °C while monitoring measurements on a human body.

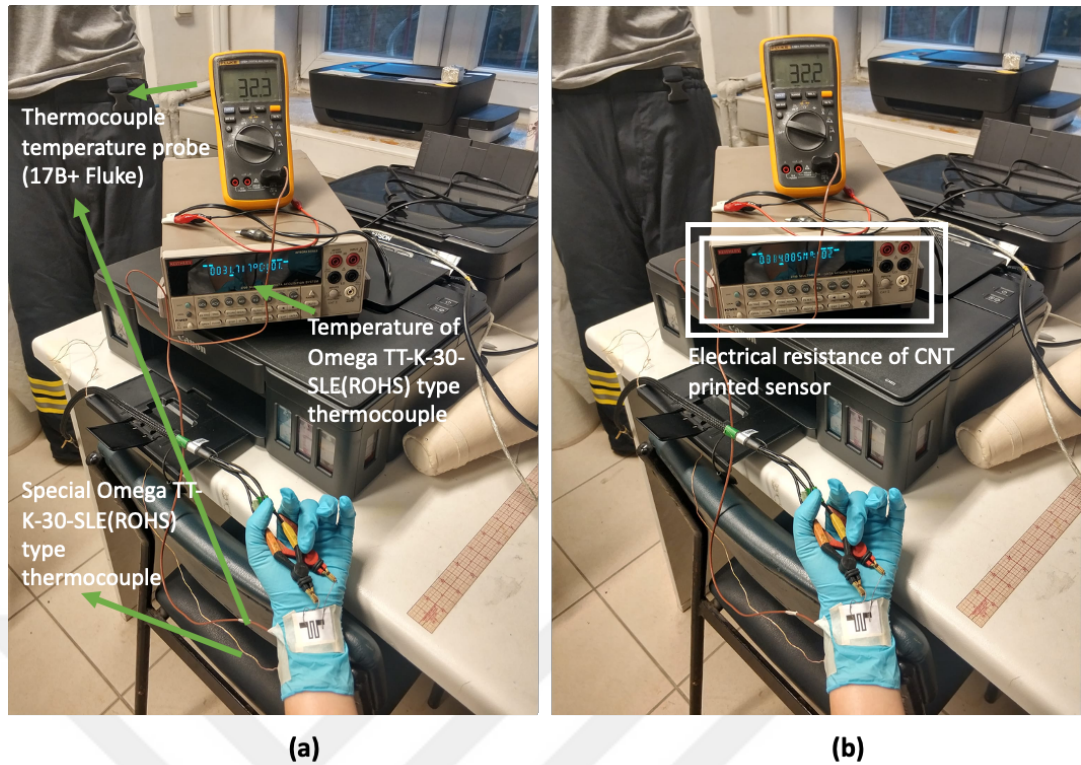


Figure 4.68 : (a) Photograph of temperature values of two different thermocouples on a gloved hand. (b) Exhibition of the electrical resistance value of our developed temperature sensor on a gloved hand.

4.2.9.3 Conclusion

Consequently, our developed temperature sensor was examined as a proof-of-concept for the real-time monitoring of a mannequin's skin and human body on a gloved hand temperatures. The skin's resistance was monitored while wearing the CNT ink printed sensor. Temperature monitoring with excellent accuracy was achieved by developing an inkjet printed wearable sensor that performed a real-time test. The robust development of this proposal will certainly encourage the widespread acceptance of smart wearable systems.

However, the biocompatibility and toxicity research on CNTs are still ongoing, and the results have not yet been derived (Yamamoto et al., 2017). Therefore, in order to work with CNT ink printed sensors attached to the directly human skin, some precautions should be taken before it is directly worn such as sensors's surface should be covered with a film that prevents the direct contact with human skin as we did in our research work.



5. MODELING OF WEARABLE SENSOR IN VARIOUS TEMPERATURE AND HUMIDITY CONDITIONS BY ARTIFICIAL NEURAL NETWORKS

In this chapter, the behavior of the sensor printed on the textile surface with formulated carbon nanotube based ink for wearable sensor applications against temperature and humidity was modeled using artificial neural networks. While humidity and temperature are defined as network input variables, the linear electrical resistance value is defined as a network output variable.

The purpose of this study is to provide an artificial neural network (ANN)-based model for predicting the final electrical resistance of a wearable sensor in a temperature and humidity environment. An ANN model for providing prediction of electrical resistance of the wearable sensor against temperature and different humidity conditions is also novel. By comparing synthetically collected electrical resistance data with experimentally observed results, the functionality of the ANN method in estimating sensor characteristics has been studied. The presented model could be a solution for researchers interested in flexible sensor applications by eliminating the time-consuming pre-product fabrication process, particularly for wearable sensors.

5.1 Development of Artificial Neural Network Model of Wearable sensor

Artificial neural networks are well-known for their functionalities, including decreasing the error rate to as small as possible during the training process using the forward feed back propagation algorithm, not requiring complex mathematical information while operating with analog and digital data. The ANN architecture has an advantage in functioning with uncertain and complex data and it has the capability of predicting the data and learning properly. It has the potential to be more efficient than traditional tools. Artificial neural network (ANN) is a useful technique that is widely used in modeling nonlinear functions (Bahiraei et al., 2019). It's noticeable that there's not too much research on implementing ANN into wearable electronics.

Artificial neural networks, a type of artificial intelligence tool, are capable of learning nonlinear complex functions, complex analytical correlations, and nonlinear relations with very low error rates (Bahiraei et al., 2019).

The Neural Network Toolbox of the MATLAB 9.5 (R2018b) mathematical software was used to predict the electrical resistance of the sensor on e-textile structures under various applied temperature and humidity values in this research.

A two-layer feed-forward network is used in the developed model. The amount of data to be used in ANN training is a significant step that has a serious influence on the ANN's prediction results (Çolak, 2021). The signal flow in feed-forward networks is entirely in a feed-forward direction, from input to output units (Abraham, 2005). Figure 5.1 depicts the developed network model's simple configuration design. Temperature (T) and humidity (H) are the input variables, and the output is electrical resistance. A total of 167 experimental data were used to train the ANN model developed in this study. 117 data is used for the training phase, whereas 25 and 25 data are used for validation and testing, respectively. Table 5.1 shows a limited experimental data set used for this study.

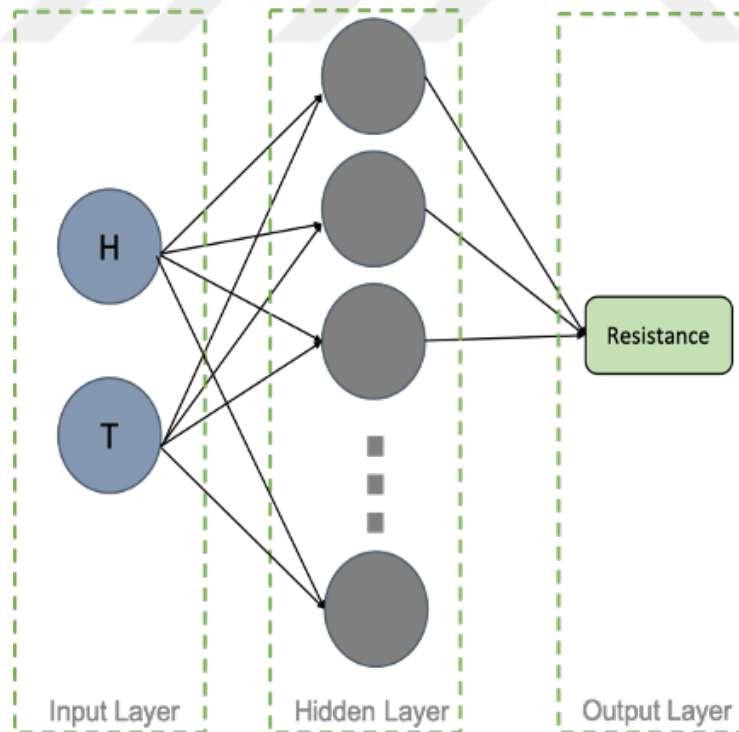


Figure 5.1 : The proposed artificial neural network model's structure.

Table 5.1 : A small experimental set

Data no	Humidity (%)	Temperature (°C)	Resistance (kΩ)
1	50	22.34	312.03
15	50	42.47	307.06
42	60	20.20	313.09
56	60	41.65	305.84
111	70	35.70	310.60
...	...	...	...
167	80	50.12	309.04

The network's capability to separate data is influenced by the number of hidden neurons. The network may be inadequate to learn the relationships between the data if there are too few hidden neurons, and the error will not decrease below an appropriate number (Abraham, 2005). For determining the number of neurons to use in neural network models, no explicit technique has been generated (Güzel & Çolak, 2021). As a result, several different ANN models with different neuron numbers have been created. In this part of the study, Levenberg Marquardt (LM) and Bayesian Regularization (BR) were used as the learning algorithms. The logarithmic sigmoid in hidden layers and fitnet in output neurons are used as activation functions. The logarithmic sigmoid (Log-Sig) and linear (fitnet) functions were given in Equations (5.1) and (5.2), respectively (Yadav et al., 2015).

$$\text{Logsig}(x) = \frac{1}{(1 + e^{-x})} \quad (5.1)$$

$$\text{linear}(x) = x \quad (5.2)$$

Understanding the consistency of the obtained outputs with the target values requires analyzing the ANN model's prediction performance. The output of the established multilayer perceptron (MLP) model was evaluated using the MSE (Mean Square Error) and coefficient of determination (R) parameters. The R is a standard parameter for evaluating a model's accuracy based on its prediction, whereas the mean-square error (MSE) is used to indicate the precision of a model. To evaluate overall model results, it is beneficial to use a combination of parameters (Ahmad et al., 2014; Güzel & Çolak, 2021). The below described parameters are used to assess ANN model's prediction success in this study.

The mean-squared error performance function is expressed as follows:

$$\text{MSE} = \frac{1}{n} \sum_{k=1}^n (y(k) - t(k))^2 \quad (5.3)$$

The coefficient of determination criterion is defined as follows:

$$R = \sqrt{1 - \frac{\sum_{k=1}^n (y(k) - t(k))^2}{\sum_{k=1}^n (y(k))^2}} \quad (5.4)$$

where n is the number of observations, $y(k)$ is the experimental data, and $t(k)$ is the predicted data.

5.2 Results and Discussion

In the present study, the modeling of the CNT printed textile-based sensor was carried out using a two-layer feed-forward network with temperature (room temperature -50°C) and relative humidity (50 – 80 %) as the input parameters. Various approaches to the number of neurons from 1 to 25 and two transfer functions were trained and evaluated by comparing some fit statistics with MSE and R to determine the best predictive configuration of ANN models. Depending on these parameters, ANN models offered better predictive results than nonlinear regression methods, with lower MSE values and higher R . The highest predictive ANN model based on network architecture with the activation functions of log-sig function between input layer and hidden layer and fitnet function between hidden layer and output layer with 15 number of neurons resulted in MSE of 5.6232×10^{-3} and R of 0.9999 with the use of the BR algorithm (Figure 5.2 and 5.3). Table 5.2 shows the performance of algorithms in more detail.

Table 5.2 : Mean Square Error (MSE) values of the two developed artificial neural network models.

Learning Algorithm	Number of Neurons	Training Data Set		Testing Data Set	
		MSE	R	MSE	R
Levenberg Marquardt	1	10.231	0.8065	8.7973	0.7081
	3	1.0497	0.9801	0.8191	0.9879
	5	0.0971	0.9982	0.3331	0.9946
	7	0.2307	0.9955	0.2869	0.9941
	9	0.0702	0.9986	0.0399	0.9995
	11	0.0269	0.9994	0.0836	0.9992
	13	0.0239	0.9995	0.3969	0.9933
	15	0.0870	0.9986	0.0903	0.9996
	17	0.0221	0.9996	0.1317	0.9986
	19	0.0638	0.9987	0.0195	0.9990
	21	0.0245	0.9995	0.1936	0.9976
	23	0.1765	0.9965	0.6437	0.9945
	25	0.0795	0.9986	0.3664	0.9879
Bayesian Regularization	1	1.6257	0.9677	1.6492	0.9687
	3	0.1403	0.9972	0.1669	0.9973
	5	0.0775	0.9985	0.1050	0.9977
	7	0.0557	0.9989	0.3861	0.9929
	9	0.0482	0.9990	0.7610	0.9979
	11	0.0765	0.9985	0.0280	0.9994
	13	0.00853	0.9998	0.0182	0.9995
	15	0.00562	0.9999	0.0432	0.9991
	17	0.0766	0.9985	0.0459	0.9994
	19	0.00581	0.9999	0.5953	0.9950
	21	0.0695	0.9987	0.0483	0.9981
	23	0.0479	0.9990	0.2821	0.9955
	25	0.0623	0.9988	0.0527	0.9991

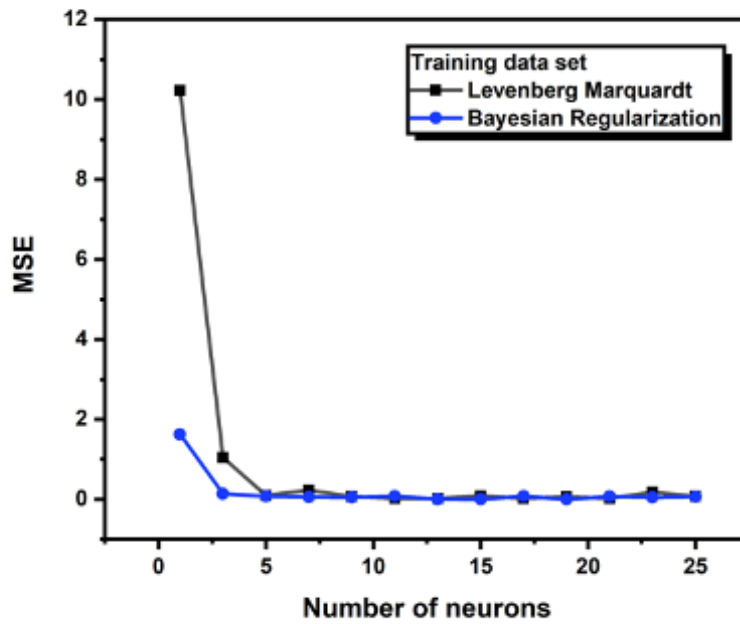


Figure 5.2 : The performance of the algorithms was compared as a function of the number of hidden neurons used in the training samples.

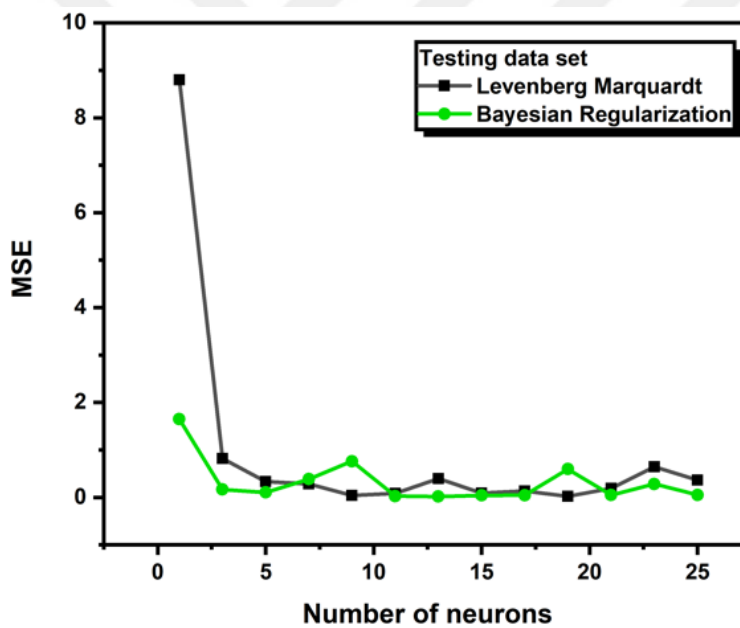


Figure 5.3 : The performance of the algorithms was compared as a function of the number of hidden neurons used in the testing samples.

In Figure 5.4, the developed ANN's training performance is demonstrated. The ANN performance is stopped at a point where the overgeneralization occurred (around 580 epochs). At this epoch, the ANN's training was completed, and the lowest MSE value was noted.

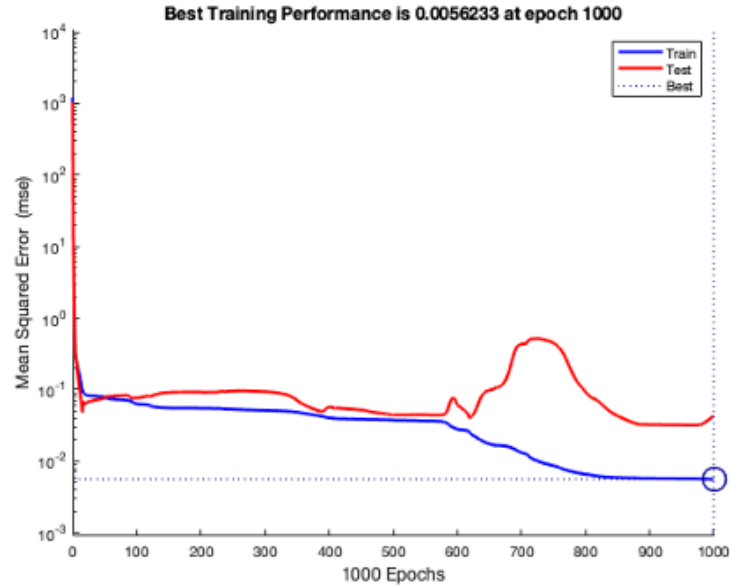


Figure 5.4 : ANN training performance depending on epoch.

Figure 5.5 illustrates the points obtained from all the data used in the training process of the two-layer feed-forward network with 117 data. It is clear that all the data points are fitted on the line. The R value was found to be 0.99989. The developed ANN model can accurately predict the electrical resistance values of the temperature sensor based on temperature and humidity changes.

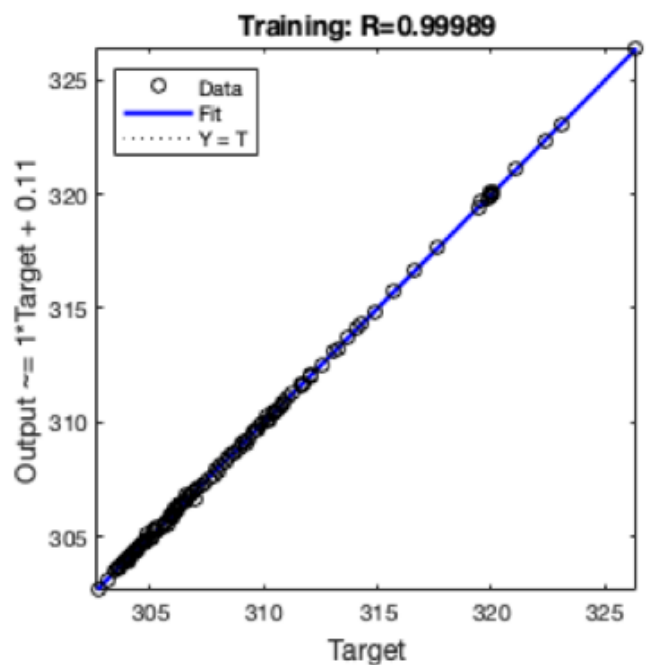


Figure 5.5 : Training phase: correlation between outputs and targets.

Figure 5.6 depicts the data collected during the ANN model's test phase. The data points from the test phase are clearly apparent in the graphic, and they are positioned

on the green line. For the test phase, the R value was calculated as 0.99913. These findings indicate that the ANN model's testing phase was concluded with a quite low error rate.

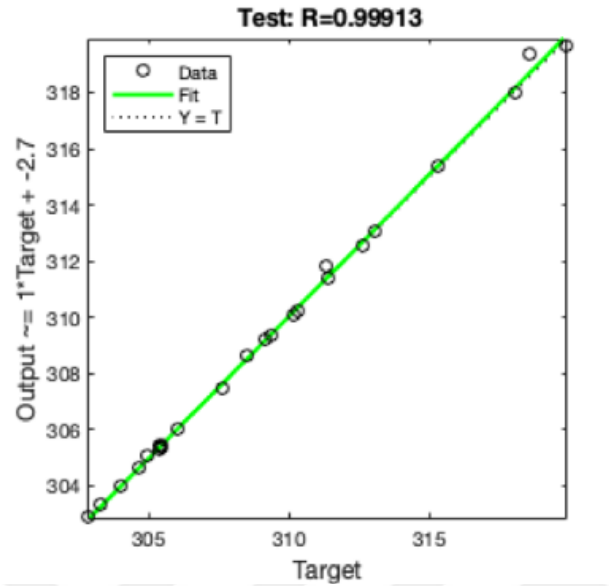


Figure 5.6 : Testing phase: correlation between outputs and targets.

Figure 5.7 exhibits the points obtained from all of the data used in the developed network model. The R value was 0.99978. When all of the data is analyzed, it is obvious that the developed ANN model can accurately predict the electrical resistance values of the sensor according to temperature and humidity changes.

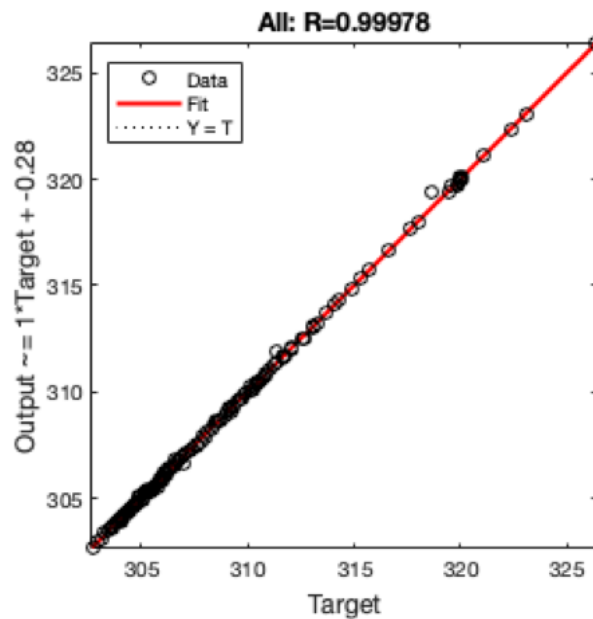


Figure 5.7 : Overall data of the ANN: correlation between outputs (predicted) and targets (experimental).

When analyzing the results in Figure 5.8, it's completely observable that the ANN data points and the experimental data points are in good accordance. This exact fit of ANN outputs with experimental data confirms that the developed model can predict the electrical resistance values of the textile-based sensor at four different humidity conditions from 50 to 80 % relative humidity and temperature conditions from room temperature to 50°C with a very low error rate.

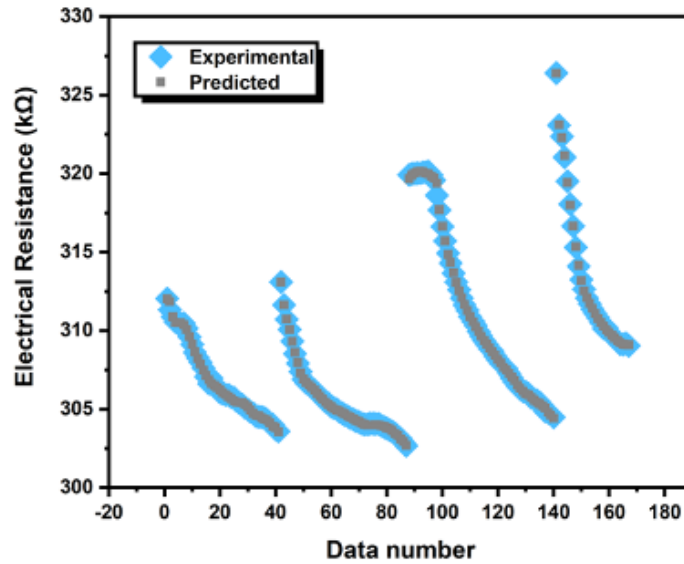


Figure 5.8 : Experimental data and predicted outputs according to data numbers.

5.3 Conclusion

The aim of this study is to accurately predict the change in electrical resistance of textile-based sensors containing carbon nanotubes against changing temperature and humidity using artificial neural networks.

A two-layer feedforward network with 15 neurons in the hidden layer and one output layer was selected as the most appropriate structure. The Bayesian Regularization algorithm was found to be the most suitable learning algorithm with a sigmoid function in the hidden layer. It is obvious to conclude that the developed ANN model has the capability of predicting the electrical resistance value of the sensor with sufficient accuracy ($R^2 > 0.99$) and low MSE error (< 0.01) against various temperature and humidity changes.



6. GENERAL CONCLUSIONS AND RECOMMENDATIONS

Wearable and flexible electronics will definitely be prominent alternatives in the development of future electronic device technology, and they are going to have unique and inspiring uses.

In this thesis, first, a critical evaluation of textile-based wearable temperature sensors, including various design aspects along with literature articles related to the design, was mentioned. In this respect, A detailed description of textile-based sensors, along with their specifications and requirements, was provided. Then, temperature-sensitive inks for temperature sensors were introduced. In detail, with examples of carbon nanotube and PEDOT:PSS ink, the formulation guidelines for conductive inkjet inks were provided. These concepts, including those of materials and inks, were explained in detail. After that, various printing technologies such as screen, gravure, and flexographic printing for sensor fabrications were introduced. Then, the details about the inkjet printing process paying particular attention to the concepts and principles necessary for a thorough understanding of the technique, as well as the printing factors that enable inkjet printability were presented.

Temperature-sensitive inkjet inks were successfully developed with two distinct ink formulation goals at our research laboratory: a thorough evaluation guideline for CNT inks was developed, and PEDOT: PSS and CNT/PEDOT composite inks were successfully created. All of the inks manufactured have exhibited good temperature sensor performance. However, the CNT/PEDOT:PSS composite ink printed temperature sensor yielded the most reliable results. This sensor was extremely sensitive; it could measure temperature changes as small as $0.3\%/^{\circ}\text{C}$ between room temperature and 50°C .

Moreover, the developed temperature sensor was examined as a proof-of-concept for the real-time monitoring of temperature one mannequin's skin and human body specifically to monitor temperature on a human gloved hand. The skin's resistance was monitored while wearing the CNT ink printed sensor.

The resulted relative difference of the accuracy of the developed sensor with respect to Omega TT-K-30-SLE(ROHS) type thermocouple is found to be 0.01 °C while monitoring measurements on a human body, which proves that temperature monitoring with excellent accuracy was achieved in a real-time environment with the wearable sensor developed under this thesis. Simple and affordable fabrication capabilities can also make our wearable sensor be used as a disposable sensor. The sensors developed with high accuracy have the potential to be used in healthcare applications particularly for continuous monitoring of human body temperature without disturbing wearer's comfort. The robust development of this proposal will certainly encourage the widespread acceptance of smart wearable systems.

Finally, an artificial intelligence based algorithm for modeling our newly developed textile-based printed sensor's performance in different temperature and humidity conditions has been presented. The developed CNT Type-2 ink-printed sensors were modeled using artificial neural networks in a diverse range of temperature and humidity conditions. It is concluded that the developed ANN model has the capability of predicting the electrical resistance value of the sensor with sufficient accuracy ($R^2 > 0.99$) and low MSE error (< 0.01) against various temperature and humidity changes. The presented model can assist researchers interested in flexible sensor applications particularly for wearable sensors by eliminating the time-consuming pre-product fabrication process as well as avoiding the need to perform unnecessary trials.

Despite these enhancements, wearable textile-based technologies face additional challenges in terms of manufacturing highly reliable products. Therefore, more research is required to better understand interconnection issues and their effects on encapsulation, and to examine more suitable encapsulation materials and methods for protecting the printed conductive ink areas on a substrate so that the sensor's performance doesn't weaken over time owing to mechanical stresses that may be encountered when the sensor is worn and laundered.

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