ANALYSIS ON THE MIXING PROCESS AND THE SUBSTRATE SURFACE TREATMENT OF THE STRETCHABLE CONDUCTIVE INK

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A report submitted

In fulfillment of the requirement for the degree of

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DECLARATION

I declare that this project entitled "Analysis on The Mixing Process and The Substrate Surface Treatment of The Stretchable Conductive Ink" is the result of my own work except as cited in the references.

Signature : ……………………………………………

Name : ……………………………………………

Date : ……………………………………………

APPROVAL

I hereby declare that I have read this project report and in my opinion this report is sufficient in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering (Structure and Materials)

Signature : ………………………………………

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Date : ………………………………………

DEDICATION

To my beloved

Noor Aziha Binti Bakar and Mohd Yusoff Bin Basir

ABSTRACT

This research demonstrates the optimized mixing parameters of stretchable conductive on the sheet resistivity performance and the effect of chemical surface treatment on the substrate of the stretchable conductive ink. The mixing parameter was divided into three ranges which are 400 rpm in 10 minutes, 1500 rpm in 30 minutes, and 2000 rpm in 60 minutes. A rectangular shape of TPU is used as the substrate in this experiment. The mixture of ink consists of GNPS as a filler, PEDOT:PSS as a functional binder, and a few types of solvents used such as DMSO, EG, and Triton X-100. Sheet resistivity performance measured for all samples shows that the best mixing parameter is 400 rpm in 10 minutes reached the lowest sheet resistivity compared to another sample. Two types of adhesion promoters were used in this experiment as chemical surface treatment such as Methyl Ethyl Ketone (MEK) and Anchor Spray Paint (ASP). The adhesion promoter was applied on the TPU substrate before printing the conductive ink. Then, the sheet resistivity performance was measured on the sample and plot the comparison with the sample without adhesion promoter. Thus, it can be concluded that the sample with adhesion promoter reached the lowest sheet resistivity performance.

ABSTRAK

Penyelidikan ini menunjukkan parameter pencampuran yang dioptimumkan bagi konduktif boleh renggang pada prestasi kerintangan kepingan dan kesan rawatan permukaan kimia pada substrat dakwat konduktif boleh renggang. Parameter pencampuran dibahagikan kepada tiga julat iaitu 400 rpm pada masa 10 minit, 1500 rpm pada masa 30 minit, dan 2000 rpm pada masa 60 minit. TPU yang berbentuk segi empat tepat digunakan sebagai substrat dalam eksperimen ini. Campuran dakwat terdiri daripada GNPS sebagai pengisi, PEDOT:PSS sebagai pengikat berfungsi, dan beberapa jenis pelarut yang digunakan seperti DMSO, EG, dan Triton X-100. Prestasi kerintangan kepingan yang diukur untuk semua sampel menunjukkan bahawa parameter pencampuran terbaik ialah 400 rpm pada masa 10 minit mencapai kerintangan kepingan terendah berbanding dengan sampel yang lain. Dua jenis promoter lekatan digunakan dalam eksperimen ini sebagai rawatan permukaan kimia seperti Methyl Ethyl Ketone (MEK) dan Anchor Spray Paint (ASP). Penganjur lekatan telah digunakan pada substrat TPU sebelum mencetak dakwat konduktif. Kemudian, prestasi kerintangan kepingan diukur pada sampel dan plotkan perbandingan dengan sampel tanpa promoter lekatan. Oleh itu, boleh disimpulkan bahawa sampel dengan promoter lekatan mencapai prestasi kerintangan kepingan terendah.

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TABLE OF CONTENT

LIST OF TABLES

LIST OF FIGURES

CHAPTER 1

INTRODUCTION

This chapter consists of the project background, problem statement, objectives, the scope of the project, and the general methodology of the Analysis on The Mixing Process and The Substrate Surface Treatment of The Stretchable Conductive Ink.

1.1 Background of Study

The research to complete this project was generally to study the process of a simple mixture of the ink to validate the optimized mixing parameter of stretchable conductive ink on the sheet resistivity performance and to investigate the effect of chemical surface treatment on the substrate of the stretchable conductive ink. The stretchable conductive ink is one of the effective material which allows the ink film to undergo better electrical conductivity after applied any forces into it such as stretching or folding. It has wide application possibilities in the branch of learning flexible electronic devices.

There are a few applications that can gain from this technology including neuro-prosthetics, flexible displays, soft robotics, cardio-stimulating implants, and other curvilinear systems such as an aircraft wing. A mixture of metallic particles with a solvent is described a conductive ink. The ink normally involves three different groups of materials. Metallic powders like Ag, Au, or Cu are labeled as the first group. It may include nano-materials to enhance sintering and low- temperature melting of the listed conductive powders.

A polymers binder is categorized in the second group which plays the greatest role in combining the metallic powders to the substrate during and after the sintering process. The third group generally includes non-volatile organic polymers and volatile solvents which bring about the rheological properties for screen printing. It will act as a transporter for the metallic powders and together with polymers binder during the sintering process and screening process. The binder performed an essential role in assist the ink to adhere to the substrate by contributing stretch-ability at the sintered state. The requirement of the stretchable conductive ink is it able to stretch after completing the sintering procedure and still keep up the ohmic connectivity between the metal flakes.

A flexible substrate such as polydimethylsiloxane (PDMS) and Thermoplastic Polyurethane (TPU), a listed metallic powders including Ag-PDMS and elastic components which are LED, transistor, resistor, capacitor, and integrated circuit mainly a part of a stretchable conductive circuit. Elastomers, TPU and PDMS are often used as substrates in numerous electronic devices and flexibly stand high deformations that bigger than 200% strain. This procedure shows the high electrical resistivity of the devices.

1.2 Problem Statement

There are several articles mentioned about the conductive filler that had been experimentally tested and used as a new engineering technology which is the most popular one is silver. However, based on the current literature review, they are considering the price range of the materials and some factors regarding mass production for widely used in electronic capsulation in the future. On the other hand, silver material is quite pricey and limited compared to graphene which saves costs with excellent environment stability and had the highest thermal conductivity among them, along with special properties suitable for range of applications. Graphene Nanoplatelets (GNPs) had been chosen in this project because of an effective reinforcement shown in polymer materials. GNPs were established to have uncomplicated process-ability with the added advantage of improving practical and biological properties of the composites. Plus, there also limited knowledge about the optimized results of the designed experiment such as the best mixing speed, and mixing time for the graphene materials and the effect of chemical surface treatment on the substrate of the stretchable conductive ink.

1.3 Objectives

The objectives of this project are listed below:

i. To optimize the mixing parameters of stretchable conductive ink on the sheet resistivity performance.

ii. To investigate the effect of chemical surface treatment on the substrate of the stretchable conductive ink.

1.4 Scope of the Project

The making of graphene is by “unzipping” the carbon nanotubes. Both of them have exceptional mechanical and electronic properties. Graphene is listed as one of the strongest materials that have superior properties and optical properties, good tensile strength, and excellent electrical conductivity. This project covered on an Analysis on The Mixing Process and The Substrate Surface Treatment of The Stretchable Conductive Ink. The optimized mixing parameters regarding on suitable mixing time and mixing speed will measured based on sheet resistivity performance of each samples. This experiment also considered the process of testing the chemical surface treatment by applying an adhesion promoter at the TPU substrate to measure the sheet resistivity performance and make a comparison with a samples without adhesion promoter. The adhesion promoter will only apply for the samples that have achieved the best mixing parameters which shows the lowest sheet resistivity. Two different adhesion promoter used as chemical surface treatment in this experiment including Methyl Ethyl Ketone (MEK) and Anchor Spray Paint. Adhesion promoter can rise up the adhesion by incorporating functional additives into the adhesive formulation that produce chemically bond to the substrate. It definitely will improve the initial joint strength and prevent delamination of the adhesive from the substrate.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Regarding this chapter, literature reviews conducted encompassed Thermoplastic Polyurethane (TPU) substrate, conductive ink, a solvents used, mechanical testing method, and adhesion promoter for chemical surface treatment. The main point is included substrates, ink, solvents used, and printing techniques, mechanical testing and adhesion promoter with detailed descriptions on each point.

2.2 Flexible Electronics

Technology in electronic circuits is majorly used a flexible electronics and has been around for several decades. Anything thin or long can become flexible. To fabricate a flexible device in an uncomplicated and efficient manner, manufacturing technologies should be readily accessible, inexpensive, and most importantly are ready to use. It is usually connected with pattern transfer, solution printing processes, additive manufacturing technologies, and roll-to-roll capabilities. Plus, the flexible circuit can be printed by using a spot of conductive ink.

Transfer and bonding of finalized circuits to a flexible substrate and fabrication of the circuit straight on the flexible substrate are the two basic approaches that have been employed to make flexible electronics. The whole transfer is fabricated by standard methods on a carrier substrate like a Si wafer on a glass plate in the transfer-and-bond approach. It advances over the original flexible wafer-based solar cell arrays. Their drawbacks are slight surface area coverage and expensive. In various applications, most of the surface will be covered with electronics fabricated straight on the substrate. There a few approaches to integrating disparate materials and oftentimes flexible substrates are not fit with the existing planar silicon microfabrication process.

To increase the power and weight ratio from the history of the flexible circuit, any kind of item thinned becomes flexible cause the silicon solar cell becomes thinner to approximately 100 µm. The thinned solar cells then were assembled on the plastic substrate to reach flexibility forty years ago. It can be classified into three different categories in terms of mechanical characteristics for flexibility which are (a) Rollable or bendable. (b) Elastically shaped and (c) permanently shaped. All the categories can be shown in Figure 2.1 below.

(a) (b)

(c)

Figure 2. 1: (a) Bendable Circuit (b) Silicon island on spherically shaped foil substrate (c) Concept of conformably shaped digital dashboard (Sarma, 2009)

The used electrical connections classify as one of the most advanced and important technologies because of their beneficial properties such as marginal weight, thinness factor, and flexibility. It will help in improving the device functionality by coming up with portability function, freedom form or flexible, resistance impact, reduce the product weight and thickness.

2.3 Conductive Ink

Conductive inks made of dispersed metallic, carbon, or conductive polymer nanoparticles have been prepared and placed on a low-cost substrate for radio frequency identification (RFID) capacitive coupled antennas. It is the most important component in the printing of metallic structures. A few conductive materials could be listed for this purpose such as conductive polymers, carbon, organic or metallic compounds, metal NPs and metal precursors. Most of the conductive inks are referred to as metal NPs. The reason that can be concluded from that is regarding their resistivity is near to that of bulk materials (2-3 times higher) as opposed to conductive materials whose resistivity is higher.

The metallization using NPs inks requires the following two steps in a row: printing of the inks and sintering to convert the inks into a conductive, solid metal track. For now, the major approach for printing metals is based on metal ink formulation, which can be printed and then sintered to acquire a metallic layer. Metal inks and pastes were grown for printing methods used in the graphics arts industry such as screen printing, flexography, inkjet printing, offset, and gravure.

To make sure a good conductivity, the ink needs to dry at room temperature or using cure techniques by cure the ink in the oven on a certain curing time to shift the solvents to strengthen the connections between metal particles. After the drying process is completed, the solvents will be removed by the evaporation process. Silver-based ink particles reach a conductivity of approximately 10^5 S/m compared to bulk copper is 59 x 10^7 S/m, unfortunately, this conductivity can be upgraded with an optimized annealing step. Organic inks based on graphene [HUA 11] and carbon nanotubes [DEN 09] are valid but the production is small with conductivities of approximately $10^2 – 10^3$ S/m. this guide to the considerable increase in losses by conduction in the conductive materials (Vena et al, 2016).

Conductive ink can be separated into two types which is particle and non-particle where also can be categorized into organic and non-organic because the solvent used a different ink (Khirotdin et al., 2016). Electrical properties that utilize a different kind of carbon because of a complex arrangement and interact between the morphology and size of the parts initiate properties, and the control techniques used to scatter them.

2.3.1 Carbon Ink

The type of ink highlighted in this project is carbon-based ink or graphene. Graphene is categorized as the mother of all graphitic forms of carbon. This is the basic structure of all carbon allotropes. Carbon atoms are uniformly arranged in a hexagonal lattice and tightly packed by sp2 hybrid bonds which is a one atom thick carbon layer (0.345 Nm thick). In 2014, Andre Geim and Kostya Novoselov isolated that this is the thinnest and world's first 2D material (Iqbal et al., 2020). The structure of graphene owns numerous fascinating properties such as good mechanical strength with Young's modulus of 1 Tpa and ultimate tensile strength of 130 Gpa, high stiffness, and extremely high thermal conductivity up to 5000 Wm-1K-1 plus elevated specific surface area.

Normally, graphene avoids to use in unalloyed form because of its insufficient yield rather than graphene derivatives such as graphene oxide (GO), reduced graphene oxide (rGO), and functionalized reduced graphene oxide (frGO) are being used universally as they exhibit similar properties of graphene. Consequently, the requirement of graphene materials over the past few years has been increased to cover the demand in both industry and academia for commercial applications and research.

2.4 Dimethyl Sulfoxide (DMSO)

Dimethyl Sulfoxide (DMSO) classified as organosulfur compound with the formula $(CH_3)_2SO$. It is a colourless liquid that important polar aprotic solvent that dissolves bothpolar and non-polar compounds in a wide range of organic solvent as well as water. It was first Russian scientist Alexander Zaytsev who reported his findings in 1867. DMSO is undustrially produced from dimethyl sulfide, a by-product of the Kraft process, by oxidation with oxygenor nitrogen dioxide. The kraft process known as kraft pulping or sulfate process is a method for conversion of wood into wood pulp, which consists of all pure cellulose fibers, the main component of paper. DMSO evaporates slowly at normal atmospheric pressure because of its high boiling point, 189 ºC (372 °F) while the high freezing point of DMSO, 18.5 °C (65.3 °F). Samples mixed with DMSO cannot be easily recuperated compared to other solvents, as it is very hard to erase all traces of DMSO by conventional rotary evaporation. DMSO also can be used as *in vitro* and *in vivo* drug testing, biology, medicine, alternative medicine, veterinary medicine and taste. DMSO used as the primary solvent that exhibits good conductivity and low baseline resistance at room temperature (Seekaew et al., 2014).

2.5 Triton X-100

It is a nonionic surfactants that has a hydrophilic polyethylene oxide chain and comes with aromatic hydrocarbon lilophilic or hydrophobic group. Triton X-100 was registered trademark of Rohm & Haas Co. Undiluted Triton X-100 is a clear viscous fluid which have less viscous than undiluted glycerol which produced a viscosity about 270 centipoise at 25 ºC which comes down to about 80 centipoise at 50 °C. It is soluble in water at 25 ºC, toluene, xylene, trichloroethylene, ethylene glycol, ethyl ether, ethyl alcohol, isopropyl alcohol and ethylene dichloride while insoluble in kerosene, mineral spirits and naphtha unless a coupling agent like oleic acid is used.

In common, Triton X-100 used in detergent in laboratories and widely used to lyse cells to extract protein organelles, or to pemeabilize the membranes of living cells. J. -B. Raoof eta al. [82] studied the effect of TX-100 film on the carbon (carbon nanotube) paste electrodes for detecting formaldehyde. They found that, TX-100 increases the electrocatalytic activities of the carbon surfaces by increasing its active surface area for the formaldehyde redox reactions. B. E. K. Swamy et al. [35, 77, 79, 83] modified the carbon (graphite) paste electrodes by TX-100 for sensor applications. They 135 found that, TX-100 increases the voltammetric signals of the carbon electrode sensors by increasing graphite active surface area. S. Ghasemi and F. Ahmadi [84] has proved the effect of TX-100 on the charge transfer properties of graphene. They found that charge transfer properties of graphene were improved by TX-100 (Jayaprakash & Flores-Moreno, 2017).

2.6 Ethylene Glycol (EG)

It is an organic compound with the formula of $(CH2OH)_2$ is mainly used as a raw material in the manufacture of polyester fibers and for antifreeze formulation. Ethylene Glycol is a colorless, an odorless, toxic, sweet-tasting and viscous fluid. Ethylene Glycol is produced via intermediate ethylene oxide form ethylene (ethane). Ethylene glycol will produced according to the chemical equation after ethylene oxide is react with water. Acidic occur when ethylene glycol reach the highest yield or neutral pH with a large excess of water.

Ethylene Glycol is an important precursor to polyester fibers and resin in the plastic industry. A popular and promising strategy has been the functionalization with poly (ethylene glycol) (PEG-GO) through covalent bonding or non-covalent interactions, which imparts remarkably high aqueous stability as well as enhanced biocompatibility (Ghosh & Chatterjee, 2020). EG and triton x-100 were added to improve of the viscosity and surface tension as well as to prevent of rapid drying and clogging in the printer head (Seekaew et al., 2014).

2.7 Poly(3,4-Ethylenedioxythiophene) Polystyrene Sulfonate (PEDOT:PSS)

PEDOT:PSS is a polymer mixture of two ionomers. Polymer known as substance or material that consisting very large molecules, or macromolecules, composed of various repeating subunits. Both synthetic and natural polymers play essential roles in everyday life due to their spectrum of properties. PEDOT is a conjugated polymer and carries positive charges and is based on polythiophene. Polythiopenes or PTs are polymerized thiopenes, a sulfur heterocycle. PEDOT:PSS can be served by mixing an aqueous solution of PSS with EDOT monomer and to the resulting mixture, a solution of sodium persulfate and ferric sulfate.

It has the largest efficiency among conductive organic thermoelectric materials and can be used in flexible and biodegradable thermoelectric generators. The conductivity of PEDOT:PSS can be improved by a post-treatment with various compound such as dimethyl sulfoxidde (DMSO), ethylene glycol (EG), salts, cosolvents, acids, alcohols, phenol and amphilic fluoro-compounds. A water-based inks of PEDOT:PSS generally used in slot die coating, flexography, rotogravure and inkjet printing. It has a lower electrical mobility than silicon which can incorporated into flexible electronics.

Poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS) is a conjugated polymer (a mixture of two ionomers) widely used as the active material in flexible and printed electronics because of its good electrical conductivity, high transparency, low redox potential and good processability. However, its limited structural and chemical properties remain major obstacles that inhibit its use in various practical applications (Seekaew et al., 2014).

2.8 Substrate

There are various substrate that useful as each of the substrate owns their surface resistivity. The substrate that commonly used for a research purposes are Polyethylene Terephthalate (PET), Polypropylene (PP), Polyvinyl Chloride (PVC), Thermoplastic Polyurethane (TPU), and some other more. Polyethylene Terephthalate (PET) and Thermoplastic Polyurethane (TPU) are chosen to be used in this project. Polyethylene Terephthalate (PET) is an excellent thermoplastic polymer resin of the polyester family.

It can be semi-rigid to rigid form depending on its thickness and very lightweight. Low cost, good thermal stability, surface inertness, good spin ability and excellent moisture resistance of the PET substrate has attracted interest in a wide arrays of fields. PET considered as flexible display films materials in several display technologies such as organic light emitting displays and resistive touch-screens. Certain applications such as electronic touch drawing pads, where a ball-pen is beneficially in order to make contact with the screen, must show high mechanical.

DSC investigated the PET substrate under dry nitrogen at a heating and cooling rate of 5 ^{o}C per minute from 20 to 250^{o}C. PET known as an aliphatic polyester that obtained from polycondensation reaction of the monomers obtained either by esterification reaction between terephthalic acid and ethylene glycol or trans-esterification reaction between ethylene glycol and dimethyl terephthalate. It is hygroscopic which absorbs water from surroundings and must be dried before the resin can be processed in a molding machine.

Thermoplastic Polyurethane (TPU) film is a dynamic substrate that can be altered for a diversity of performance applications. TPU known for their extremely high wear resistance, excellent tear strength, good compression set, high elasticity, high transparency, low temperature performance and excellent process. It is part of a huge group of elastomers called thermoplastic elastomers or TPEs. The TPEs are a birth of polymers that can be frequently stretched without permanently deforming the shape of the part. When exposed to the saturated hydrocarbons, the TPU will expand resulting lead to an interim reduction in a TPU's tear resistance.

2.9 Printing Method

The process of printing have developed for centuries, mainly with the growing need for information in bigger volumes and at a smaller price. There are few methods of ink printing available nowadays, includes screen printing, offset, flexography, inkjet, laser and gravure. Besides, there are various process of printing the conductive ink onto the substrate, screen printing is chosen as it compliments with the ink suitability for this research.

3.0 Screen Printing

Screen printing is known as a stencil process. The ink is transferred to the substrate through a stencil covering a fine fabric mesh of threads (Blayo & Pineaux, 2005). There are a few things that need to consider to achieve good conductivity which is ink resistance. The resistance of the ink that will be used needs to consider when designing a circuit. This is because all the conductive ink has different resistances, which means a design for a certain type of ink will probably not work for another type of ink. Plus, the conductivity and resistance are depending on the width and the length of the traces.

Secondly, the conductive ink trace thickness will also give an impact on the resistance and the conductivity of the traces. This condition will vary depending on the substrate that is printed. As an example, four layers of conductive ink are more conductive compared to the two layers. The thicker trace can compensate for the loss of conductivity in length. Lastly, the type of substrate and the surface will have an impact on the conductivity of the inks. A test needs to be conducted on each of the substrates as the results are not transferable.

It has been introduced since 1980s offered huge scale of manufacturing in reasonable price electrode system. The inks will be squeeze through the screen mask and stencil for patterned onto substrate. Usually, the ink will cured at high temperature or let it dry in a room temperature for a certain time. Screen printing can be classify in two methods which is flat bed screen printing by setting the stencil on the substrate. The second method shows layered ink on the stencil and pull it along the stencil surface by using a squeegee. The ink remain on the substrate while the stencil are removed.

3.1 Mixing Process

Different mixing results can be achieved by change the speed of rotation and revolution and the ratio between them. Two standard quality modes are used to reach optimal mixing results which are mixing mode and de-foaming mode or de-gassing mode. This mixing method adds no air to the materials. It has a propensity to expel the entrapped air. Mixing quality and speed are optimized by using an acceptable container.

This experiment used a THINKY mixer to stir the mixture. The mixer holds a small container with an optional adapter. Various sizes of jars, syringes, and cartridges can be used. The adapters can be made as requested. The freeze gel act as a cooling adapter that will help to keep the material cools throughout the mixing process. The heat insulation adapter allows proceeding heater materials up to 130 ^{o}C. The THINKY mixers are effective for effortless, homogeneous mixing of all types of engineered compounds. The accuracy can be simply confirmed when inspecting under a microscope. There are a few benefits of using THINKY mixers which suitable for high viscosity with different densities. No materials could harm unlike processes involving mixing blades, rollers, or propellers.

3.2 Curing Process

The conductive ink can be made to dry very speedily by using a speedy evaporating solvent system. The snag to this is that if it dries rapidly in the oven, it will also dry in on the screen more quickly, causing clogging. Quality ink systems will supply an optimal balance between long screen time and short drying time. One of the considerable problems area when screening conductive inks is that they are not dried totally after screening. An easy way to inspect the completeness of drying inks is to run a circuit through the drier and measured the point-to-point resistance on one of the longer ink traces. Run the circuit for a second time through the drier again and measured the resistance. If the drop is lower than 10%, then the ink can be observed as dried after the first pass.

Three factors donate to how fast they will dry; temperature, the quantity of airflow, and air humidity. To enhance drying efficiency it is necessary to profile the oven by running a small thermocouple connected to a spool of wire and a handheld temperature readout. The thermocouple is placed on a circuit and measured the temperature at systematic intervals as the circuit runs through the drier. There will be an amazing difference between the actual temperature result and the oven readouts, especially on a hot air oven with no IR heat sources. After an oven is profiled, the temperature setting can be increased until reaches an optimal actual temperature on the substrate that does not damage the substrate.

3.3 Mechanical Testing

Hardness tests evaluate a material's resistance to indentation. The shape and size of the indenter vary between the most common methods, but the common principle is to apply a known force to an intender of a given shape onto the surface of materials. The size of the indentation that is left is then connected to a measurement of hardness. The testing needs a flat, often ground and polished surface which may be a section etched so that the different parts of the sample can be identified and the hardness indents located in the correct region. The most frequent hardness testing methods are Vickers, Rockwell, and Brinell hardness testing, valid for general testing of bulk materials and quality control during processing. It is common to use the Vickers hardness test in the UK and Europe.

This is the oldest equipment and has a large hardness scale, making it suitable for most metals and welds. It used a 136° pyramidal diamond indenter that structures a square indent. The load is tried for a time of 10-15. The two axes of the diamond-shaped indentation measured in millimeters are mid-point and the hardness is decided. Vickers hardness testing has fulfilled standards such as ASTM E384 or ISO 6507 which has four parts mentioning the test itself, calibration, and hardness tablets (The, 2015).

The most common method used for measuring sheet resistance is the four-probe method. This method involves using four equally-spaced, co-linear probes known as a four-point probe to make electrical touch with the material. Most four-point probes economically use sharp needles as probes. These can scratch or pierce fine materials often used in thin-film electronic devices. The movement of the electrons designates passed electricity through a material. There are normally many electrons flowing through the material at the same time and for ease of explanation and only one electron is examined in this model and also solid material consist of immobile atoms.

The solid material reveals to external force which is electrical power, the electron tends to proceed from the opposite ways of their electrical charge. The collision of free electrons with atoms will occur in the green circles. This collision will retarded the movement of the electrons. If the atoms cause the slightest amount of scattering of electrons, then the material has low resistivity. High resistivity materials rely on the temperature and the applied magnetic field. Figure 2.2 shows a simple microscopic model of electricity flowing. (Ghorbani & Taherian, 2019).

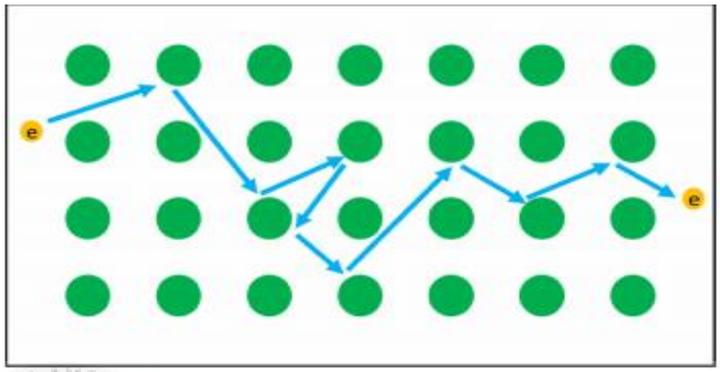


Figure 2. 2: Simple microscopic model of electricity flowing

3.4 Adhesion Promoter

Adhesion promoter or known as a coupling agents are chemical that act at the interface between an organic polymer and an inorganic surface to create adhesion between the two materials. In its optimal sense will act effectively at the organic or inorganic interfacial region to chemically and physically wed these different materials into a powerful cohesive bond structure. The use of adhesion promoters can provide a “glue” or compatibility bridge to give a much greater level of adhesion instead of alter physical and chemical forces at the interface. Additionally, it can transmit resistance to environmental and other destructive forces such as heat and moisture that always act on the bonded place to break adhesive strength. Silicon-based chemical that will function as an adhesion promoter, or a coupling agent, has a general structure of four substituents attached to a single silicon atom. The most common structure has three inorganic-reactive alkoxy groups, methoxy or ethoxy, and one organic group (Verkholantsev, 1999).

CHAPTER 3

METHODOLOGY

3.1 Introduction

The list of method and procedure of sample preparation, the apparatus and materials used, the test implementation, and data collection for the research were discussed in this chapter. The methodology described the whole process of the final year project from the start until the end of the project. The processes involved some research from journals to guide the whole journey of the project. Next, the process continues with spot the materials used for the testing and study the plan of actions of the experiments. Then, the apparatus was prepared and set up for conducting the test. Based on the experiments, the data collected will be analyzed and prepare the complete final report to present to the panel.

This study focused on the mixing parameters to finalize what is the optimum mixing time and mixing speed to achieve the best conductivity and resistivity of the ink. The mixing time is set in three different range which are 400 rpm, 1500 rpm and 2000 rpm as the maximum range with 10 minutes, 30 minutes and 60 minutes respectively based on the articles researched. The samples will be cured in the oven for 15 minutes on 60°C fixed for all samples. The conductive ink is printed on the stretchable Thermoplastic Polyurethane (TPU) as a substrate.

The filler type used in this project is Graphene Nanoplatelets (GNPs) mixed with a functional binder which is poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) and a few types of solvents including Dimethyl Sulfoxide (DMSO), Ethylene Glycol (EG) and

Triton X-100.The sheet resistivity of the samples is measured using Four-Point Probe Measurement and analyzed which samples have the lowest sheet resistivity to be label as an optimized result for mixing parameters.

The experiment is continued by applying two different adhesion promoter on the substrate surface to investigate the chemical surface treatment on the stretchable conductive ink. The samples will be cured in the oven for 15 minutes on 60°C fixed for both adhesion promoter layered. After a while, the samples will be tested under Four-Point Probe Measurement to compare the sheet resistivity performance with non-layered samples. Both adhesion promoters used as chemical surface treatment in this experiment are Methyl Ethyl Ketone (MEK) and Anchor Spray Paint.

3.2 Gantt Chart

A gantt chart is used in project management as one of the most popular useful ways of listing activities, tasks or events displayed against time. On the left side of the chart is a list of the activities and along the top shows a suitable time scale. Table 3.1 and 3.2 shows the gantt chart of PSM 1 and 2.

Table 3.1: Gantt Chart for PSM 1

Bil.	Works/tasks \ Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1.	Registration title selection for Projek Sarjana Muda (PSM 1)								MID SEMESTER BREAK						
2.	Briefing with supervisor for PSM 1														
3.	Literature review: explore and review journals and articles														
4.	Literature review: research gaps for PSM 1														
5.	Methodology: process framework for PSM 1														
6.	Submission progress report for PSM 1														
7.	Discussion with the supervisor about design the process														
8.	Result and analysis														
9.	Submission final report														
10.	Make slides preparations for PSM 1														
11.	Seminar PSM 1														

Table 3. 2: Gantt Chart for PSM 2

Bil.	Works/tasks \ Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1.	Literature review								MID SEMESTER BREAK							
2.	Design of experiment															
3.	Fabricate the sample															
4.	Testing : Sheet Resistivity															
5.	Data analysis															
6.	Report writing															
7.	Report submission															
8.	PSM presentation															

3.3 Project Methodology

The overall process of the project flowchart shown in Figure 3.1 below. The details description for each method and process are elaborated in the subsequent section.

3.3.1 Literature Review

One of the dominant process to complete the project are literature review. Fundamentally, it is an overview of the formerly published works on a specific topic that helps us to refer on. The project planning is important to make sure that the process are well-prepared and able to attain the objectives at the end of the study. This study focused on information regarding flexible electronics, carbon-based conductive ink, TPU, a functional binder, list of solvents, the optimum mixing time, mixing speed, suitable curing time and curing temperature, applying adhesion promoter on the substrate and comparing the sheet resistivity performance of the ink. Based on the input gained, the final data is concluded. Journals, articles, and authorized websites are the main sources for completion of literature review.

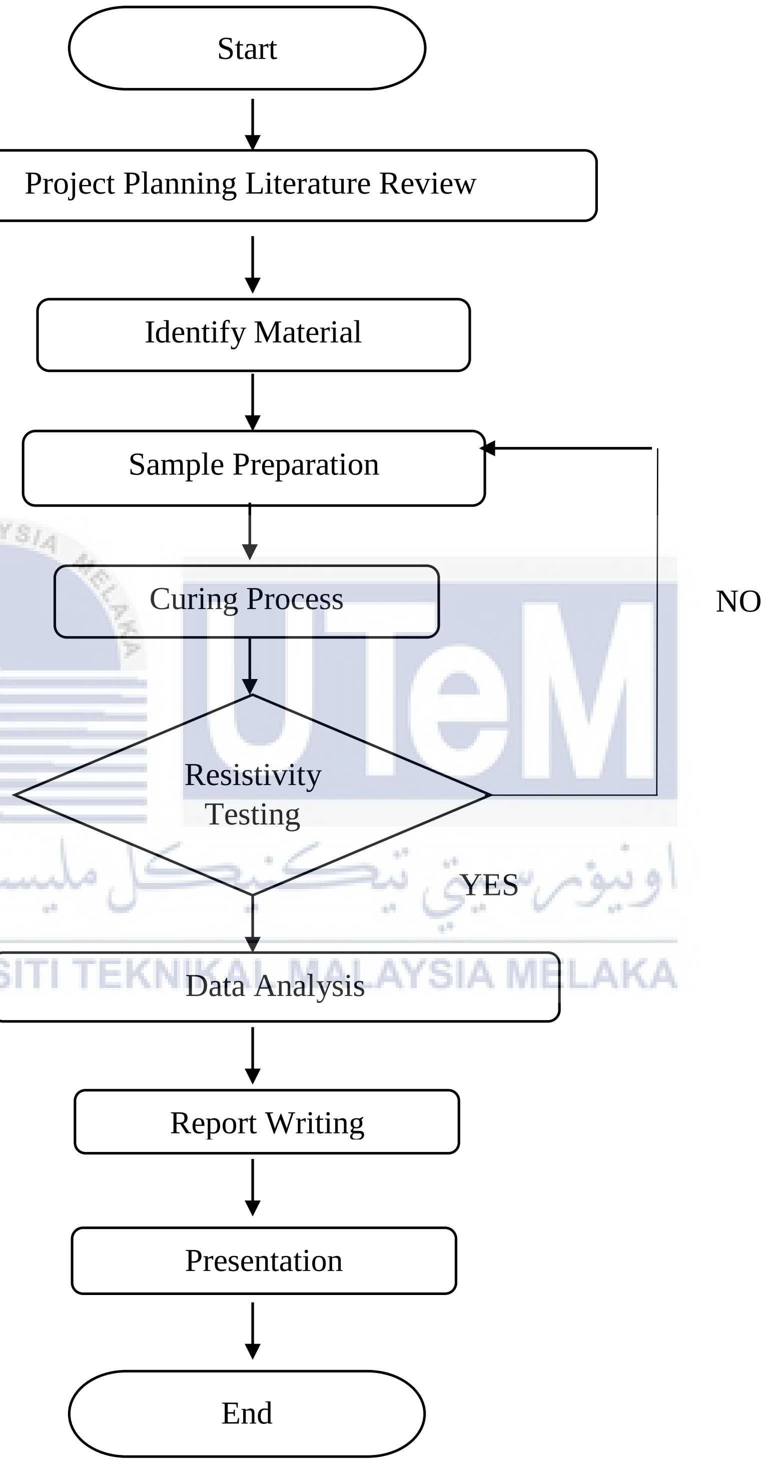


Figure 3.1: Overall Project Flowchart

3.3.2 Material Identification

The material used in this experiment is Graphene Nanoplatelets (GNPs). It is a platelet-liked graphite nanocrystal with multi-graphene layers. These materials and other printed electronics technologies have been used to create a wide arrays of sensors. GNPs has a great possible in upgrade simultaneously mechanical, tribological, electrical, thermal and biological properties. Graphene has been known as the thinnest, most flexible and strongest material that conducts heat and electricity well. Moreover, GNPs can be expected to show better reinforcement in polymer composites due to their ultrahigh detail ratio. It allows greater surface contact area with polymer derive in the enhancement of the composite thermal conductivity.

There is a functional binder and a few solvents used and mixed together with graphene. The binder used are Poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) known as polymer mixture of two ionomers which of them is made up of sodium polystyrene sulfonate which is a sulfonated polystyrene. It can be used in flexible and biodegradable thermoplastic generators thus shows PEDOT:PSS has the highest efficiency among conductive organic thermoplastic materials. Its biggest application is as transparent, conductive polymer with high ductility. A high viscous paste of PEDOT:PSS is suitable in screen printing method.

Moreover, one of the solvents mentioned is Triton X-100 which is a nonionic surfactant that has a hydrophilic polyethylene oxide chain. Surfactants is a compound that lower the surface tension between two liquids, between gases and liquid or between a liquid and a solid. Surfactants may act as wetting agents, foaming agents, detergents, emulsifier or dispersants. A clear viscous fluid is visible for undiluted Triton-X.

Next, Ethylene Glycol (EG) is a colorless, an odorless, toxic, sweet-tasting and viscous fluid. Ethylene Glycol (EG) is produced via intermediate ethylene oxide form ethylene (ethane).

Ethylene glycol will produced according to the chemical equation after ethylene oxide is react with water. Acidic occur when ethylene glycol reach the highest yield or neutral pH with a large excess of water. Ethylene Glycol (EG) is an important precursor to polyester fibers and resin in the plastic industry. EG was added to improve the viscosity and surface tension as well as to prevent of rapid drying and clogging in the printer head.

Dimethyl Sulfoxide (DMSO) classified as organosulfur compound with the formula $(CH_3)_2SO$. It is a colourless liquid that important polar aprotic solvent that dissolves bothpolar and non-polar compounds in a wide range of organic solvent as well as water. It was first Russian scientist Alexander Zaytsev who reported his findings in 1867. DMSO is undustrially produced from dimethyl sulfide, a by-product of the Kraft process, by oxidation with oxygenor nitrogen dioxide. DMSO used as the primary solvent that exhibits good conductivity and low baseline resistance at room temperature.

The substrate used in this project is TPU which is classified in a category of plastic created when a polyaddition reaction occurs between diisocyanate and one or more diols. It can be used as a soft engineering plastic or as a renewal for hard rubber. TPU comes with lots of typical properties such as high elongation and tensile strength, excellent abrasion resistance, low temperature performance, excellent mechanical properties, high transparency and also good oil and grease resistance. Technically, they are thermoplastic elastomers that contain of linear segmented block copolymers composed of hard and soft segments. The properties of graphene are shown in Table 3.3 below:

Table 3. 3: Properties of Graphene.

GRAPHENE	
Properties	**Description**
Surface Area (meter^2/gram)	1520
Solubility (Water)	Insoluble
Solubility (Other)	Dispersed in organic solvent
Thermal Conductivity (Watt/meter*Kelvin)	$4.84x10^3$
Drying Temperature	At room temperature/ Oven
Conductivity	High Electrical Conductivity

The properties of TPU are as shown in Table 3.4 below:

Table 3. 4: Thermoplastic Polyurethane (TPU))

THERMOPLASTIC POLYURETHANE (TPU)	
Properties	**Description**
Abrasion Resistance	Excellent
Mechanical Properties	Excellent
Low Temperature Flexibility	Good
Heat Aging	Good
Hydrolysis Resistance	Poor
Adhesion Strength	Good
Chemical Resistance	Excellent

The properties of Dimethyl Sulfoxide (DMSO) are as shown in Table 3.5 below:

Table 3. 5: Properties of Dimethyl Sulfoxide (DMSO)

DIMETHYL SULFOXIDE (DMSO)	
Properties	**Description**
Appearance	Colourless Liquid
Density	1.1004 g·cm^{-3}
Melting Point	19 °C (66 °F; 292 K)
Boiling Point	189 °C (372 °F; 462 K)
Viscosity	1.996 cP at 20 °C
Solubility in Water	Miscible

The properties of Poly(3,4-ethylenedioxythiophene) Polystyrene Sulfonate (PEDOT:PSS) are as shown in Table 3.6 below:

Table 3. 6: Poly(3,4-ethylenedioxythiophene) Polystyrene Sulfonate (PEDOT:PSS)

POLY(3,4-ETHYLENEDIOXYTHIOPHENE) POLYSTYRENE SULFONATE (PEDOT:PSS	
Properties	**Description**
Appearance	Dark Blue
Form	Liquid
Solid Content	3.0 – 4.0 %
Storage Temperature	2 - 8 °C
Conductivity	> 200 S/cm

The properties of Triton X-100 are as shown in Table 3.7 below:

Table 3. 7: Triton X-100

TRITON X-100	
Properties	**Description**
Appearance	Viscous Colourless Liquid
Density	1.07 g/cm^3
Melting Point	6 °C (43 °F; 279 K)
Boiling Point	270 °C (518 °F; 543 K)
Solubility in Water	Soluble

The properties of Ethylene Glycol (EG) are as shown in Table 3.8 below:

Table 3. 8: Ethylene Glycol (EG)

ETHYLENE GLYCOL (EG)	
Properties	**Description**
Appearance	Celar, Colourless Liquid
Density	1.1132 g/cm^3
Melting Point	−12.9 °C (8.8 °F; 260.2 K)
Boiling Point	197.3 °C (387.1 °F; 470.4 K)
Solubility in Water	Soluble
Viscosity	1.61×10^{-2} Pa·s[4]

3.3.3 Sample Preparation to Optimized a Mixing Parameters

The experiment was started by dropping a binder and solvents in the small container was placed on the digital weight balance. The previous research article referred to each weight percentage of the dropped binder and solvents (Seekaew et al., 2014). The binder mentioned is Poly(3,4-ethylenedioxythiophene) Polystyrene Sulfonate (PEDOT:PSS) while the solvents are Ethylene Glycol (EG), Triton X-100, and Dimethyl Sulfoxide (DMSO). The formulation was mixed for the first time using THINKY Mixers with a different range of mixing parameters.

There is three range of mixing parameter used: 400 rpm for 10 minutes, 1500 rpm for 30 minutes, and 2000 rpm for 60 minutes based on articles. Then, the formulation of graphene was added together with the mixture before continuing to mix in the THINKY Mixers for the second time using the same mixing parameter as the first one. The filler size of graphene used is 15 µm. The Mixers are effective for effortless, homogeneous mixing of all types of engineered compounds. The table 3.9 below shows the complete formulations of graphene, PEDOT:PSS, and solvents. The figure 3.2 below shows the THINKY Mixers and the mixture of conductive ink.

Table 3. 9: Graphene – PEDOT:PSS formulation

Sample	SCI Mass, g	Graphene (wt%)	Graphene Mass, g	PEDOT:PSS Solution (wt%)	PEDOT:PSS Solution Mass, g	Substrates
1	4	7.5	0.3	92.5	3.7	TPU

Table 3. 10: PEDOT:PSS solution and solvents.

PEDOT:PSS Solution Mass, g	PEDOT:PSS (wt%)	PEDOT:PSS Mass, g	DMSO (wt%)	DMSO Mass, g	EG (wt%)	EG mass, g	Triton X-100 (wt%)	Triton-X-100 mass, g
3.7	89.82	3.323	5.98	0.2213	3.99	0.1467	0.199	0.00736

(a) (b)

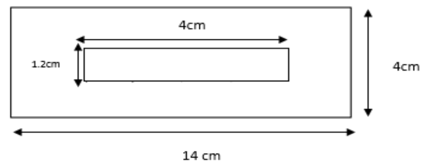
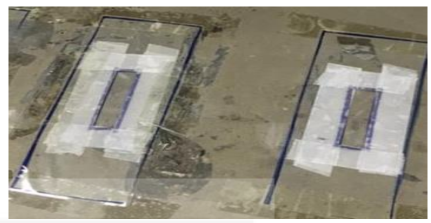
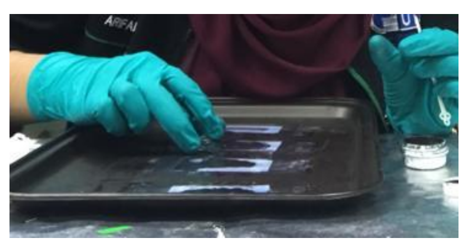
Figure 3. 2: a) A THINKY Mixer, b) A mixture of conductive ink

3.3.4 Sample Design and Ink Printing Preparation

The complete mixture of the ink was printed on the simple designed TPU substrate. The TPU substrate was cut into a rectangular shape with an area of 14 cm × 4 cm and a small rectangular shape inside with an area of 4 cm × 1.2 cm. Scotch tape is used to achieve smooth printing on the substrate and maintain the thickness while printing the ink. To print the ink smoothly, the TPU substrate was laid on the tray neatly.

Each of the substrates was labeled with the mixing parameters value. A glass slide was used to align the conductive ink on the substrate. It is a thin, 1 mm thick, flat, rectangular piece of glass that resulted in the thickness of printed ink maintained. Normally, slides are made of common glass and their edges are often in small sharp fragments or polished and make the printing step easier compared to other tools. The sample preparation procedure applied in this project was described in the Table 3.11 below:-

Table 3. 11: Sample preparation procedure

STEP	PROCEDURE	OVERVIEW
1	Cut the TPU substrate into a rectangular shape with a correct measurement	
2	Applied a double layer of Scotch Tape around the small rectangular shape	
3	Laid the substrate on the tray neatly with a complete labeled	
4	Pour the conductive ink on the TPU substrate using a plastic spoon stirrer	
5	Align the conductive ink using a glass slide	

3.3.4 Curing Process

Curing is a method during which a chemical reaction such as polymerization or physical action such as evaporation takes place, concluding in a harder, tougher, or become stable linkage for adhesive bond or substance. Some of the processes desired maintenance of a certain temperature or humidity phase, others require a certain pressure. The cure monitoring process allows a significant insight into the chemical process and elaborate process actions towards achieving specific quantity indices of the cured substance. Good curing can help reduce the appearance of curing.

This process will involve curing a mixture of the conductive filler with PEDOT:PSS and the other solvents. The curing process in this experiment took place at 60^{o} C for 15 minutes fixed for all samples. After the printed sample is fully cured, it was then let to be fully dried at room temperature. The samples are ready to be analyzed. The figure 3.3 below shows the example of a heating and drying oven used to cure the samples and the condition of conductive ink after fully cured.

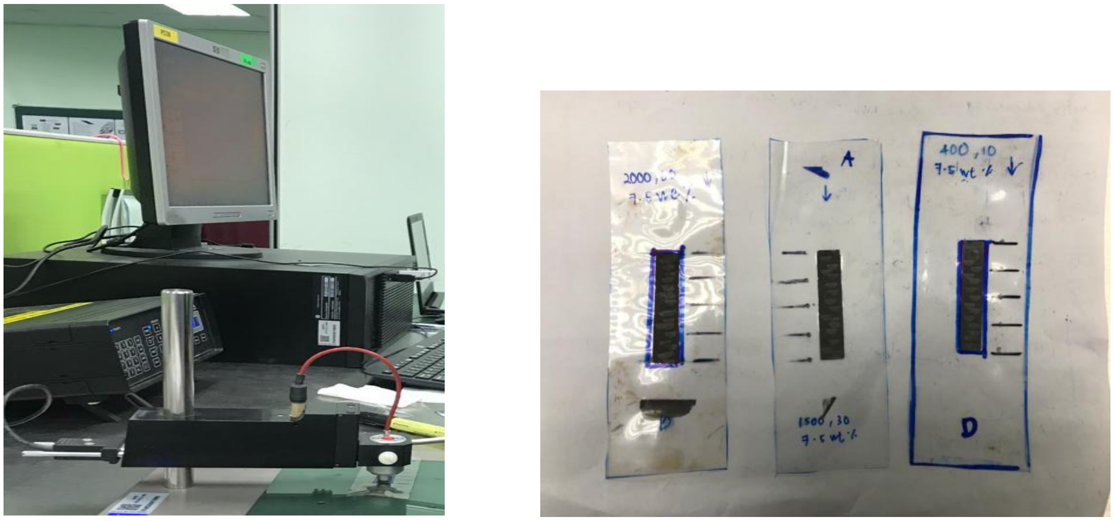
(a) (b)

Figure 3. 3: a) Example of heating and drying oven, (b) The conductive ink after cured

3.3.5 Sheet Resistivity Testing

To measure the resistivity of the sample, the four-point probe measurement is used in this experiment. The four-point probe is to determine the resistivity of a material at different temperatures. It is called four-point probe because the four points touch the sample's surface. This method is working when the sample is in the form of a thin wafer. The four-point probes are arranged linearly in a straight line at an equal distance of 1 mm from each other a high impedance source is used to generate the current through the outer two probes. A digital voltmeter measures the voltage across the inner two probes to measure the resistivity.

The sample is put carefully in the sample space provided and mounted on the four probes on the surface of the sample without damaging it. The samples were divided into four sections which each section will produce four data of sheet resistivity in the conclusion of sixteen data per sample. All the data recorded through the voltmeter will automatically store and transferred to the Jandel Multipurpose Four Point Probe System. Up to 99 data can be stored in one time. Download the data and export it to the chosen file. It is a combination of the Multiheight Probe Stand with the RM3000 Test Unit, with the added feature of a removable X-Y microsposition table. A wide variety of samples is allows to be measured from glass slides with TCOs or metal layers, to wafers and even ingots up to 250 mm deep. Figure 3.4 below shows the four-point probe measurement and the measured samples.



(a) (b)

Figure 3. 4: a) Four-Point Probe Measurement, b) Measured samples

3.3.6 Sample Preparation for Chemical Surface Treatment

Two different types of adhesion promoters are used in this experiment as chemical surface treatment for the substrates. An adhesion promoter act at the interface between an organic polymer and an inorganic substrate to enhance adhesion between two materials. It helps to increase the adhesive strength between the conductive ink and the TPU substrate. Two types of adhesion promoters are Methyl Ethyl Ketone (MEK) and Anchor Spray Paint.

The TPU substrate was prepared with the same measurement and size. The conductive ink formulation was also prepared same as the previous experiment. However, the mixing time and mixing speed used to mix the ink are referred from the optimized mixing parameter. In this case, the optimized mixing parameter goes to the sample that reached the lowest sheet resistivity performance. Laid down the substrate on the tray before applying the adhesion promoter. MEK was poured on the TPU substrate and the plastic spoon stirrer was used to scatter the chemical uniformly. Labeled the substrate with correct information to avoid misunderstanding between other samples.

Set up the oven at 60 °C for 15 minutes to cure the applied MEK on the substrate. The curing process helps the chemical to dry faster. Poured the conductive ink on the substrate using a plastic spoon stirrer and aligned with a glass slide uniformly. The sample was cured again for the second time using the same curing temperature and curing time. Next, the experiment continued by applying two layer of Anchor Spray Paint on the new TPU substrate. The substrate was let to dry at the room temperature for 15 minutes before print the conductive ink. The samples were then cured in the oven for 60 °C in 15 minutes.

3.3.7 Sample Testing for Chemical Surface Treatment

Testing that had been done for the samples was using a four-point probe measurement to measure the sheet resistivity performance after applying the adhesion promoter on the surface. The samples were divided into four sections which each section will produce four data of sheet resistivity in the conclusion of sixteen data per sample. All the data recorded through the voltmeter will automatically store and transferred to the Jandel Multipurpose Four Point Probe System. The sample is put carefully in the sample space provided and mounted on the four probes on the surface of the sample without damaging it. Download the data and export it to the chosen file. The collected data was plotted in the graph to make a comparison of the sheet resistivity performance between the sample with adhesion promoter and without adhesion promoter. Figure 3.5 below shows the schematic picture to show the distance measured on the sample.

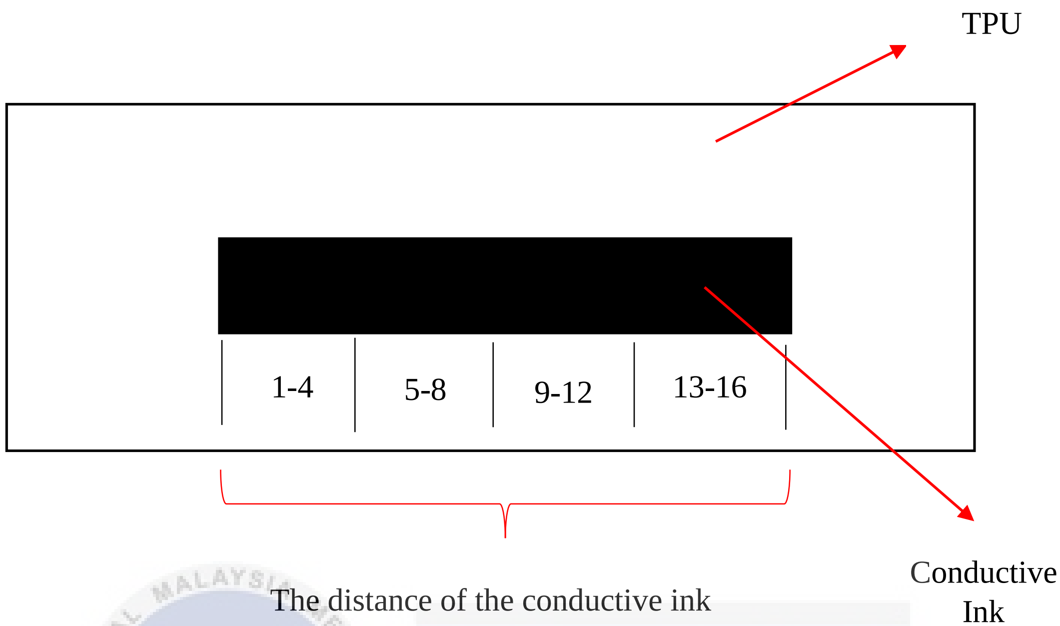


Figure 3. 5: The distance marked on the conductive ink

CHAPTER 4

RESULT AND DISCUSSION

4.1 Overview

After completion of preparing different samples, this chapter will elaborate on the data collected and value of sheet resistivity performance measured from the four-point probe measurement. Table below shows the data tabulation of sheet resistivity for conductive ink on different mixing parameters value which are 400 rpm in 10 minutes, 1500 rpm in 30 minutes, and 2000 rpm in 60 minutes. The plotted graphs below mentioned on the sheet resistivity against the distance for different mixing parameters. There are also mentioned on the adhesion promoter data involving Methyl Ethyl Ketone (MEK) and Anchor Spray Paint (ASP) with a comparison graph between the sheet resistivity performance on adhesion promoter and non-adhesion promoter.

4.2 Sample Data

All of the resistance data obtained after the curing process were recorded and by using the resistivity data, resistance-distance curve was formed.

4.2.1 Data Comparison of Different Mixing Process Parameter

Table 4. 1: Sheet Resistivity on 2000 rpm, 60 minutes

Distance of Conductive Ink	Sheet Resistivity, Ω/sq	Average Sheet Resistivity, Ω/sq	Standard Deviation
1 - 4	3.56	2.99	0.6291
	2.10		
	3.08		
	3.24		
5 – 8	3.70	4.68	1.1093
	4.06		
	6.21		
	4.76		
9 - 12	6.04	4.69	1.2606
	5.47		
	3.44		
	3.81		
13 – 16	3.27	3.68	0.2794
	3.73		
	3.82		
	3.89		

Table 4. 2: Sheet Resistivity on 1500 rpm, 30 minutes

Distance of Conductive Ink	Sheet Resistivity, Ω/sq	Average Sheet Resistivity, Ω/sq	Standard Deviation
1 - 4	19.15	10.95	5.6264
	9.87		
	8.17		
	6.61		
5 – 8	7.39	5.5375	1.3407
	5.63		
	4.39		
	4.74		
9 - 12	7.28	6.6375	0.6346
	7.08		
	6.02		
	6.17		
13 – 16	3.61	4.4275	0.8936
	3.75		
	4.9		
	5.45		

Table 4. 3: Sheet Resistivity on 400 rpm, 10 minutes

Distance of Conductive Ink	Sheet Resistivity, Ω/sq	Average Sheet Resistivity, Ω/sq	Standard Deviation
1 - 4	6.28	4.80	1.3037
	5.03		
	4.81		
	3.11		
5 – 8	3.34	3.21	0.0962
	3.21		
	3.11		
	3.18		
9 - 12	3.27	3.35	0.0845
	3.37		
	3.46		
	3.3		
13 – 16	4.09	3.61	0.3916
	3.33		
	3.26		
	3.78		

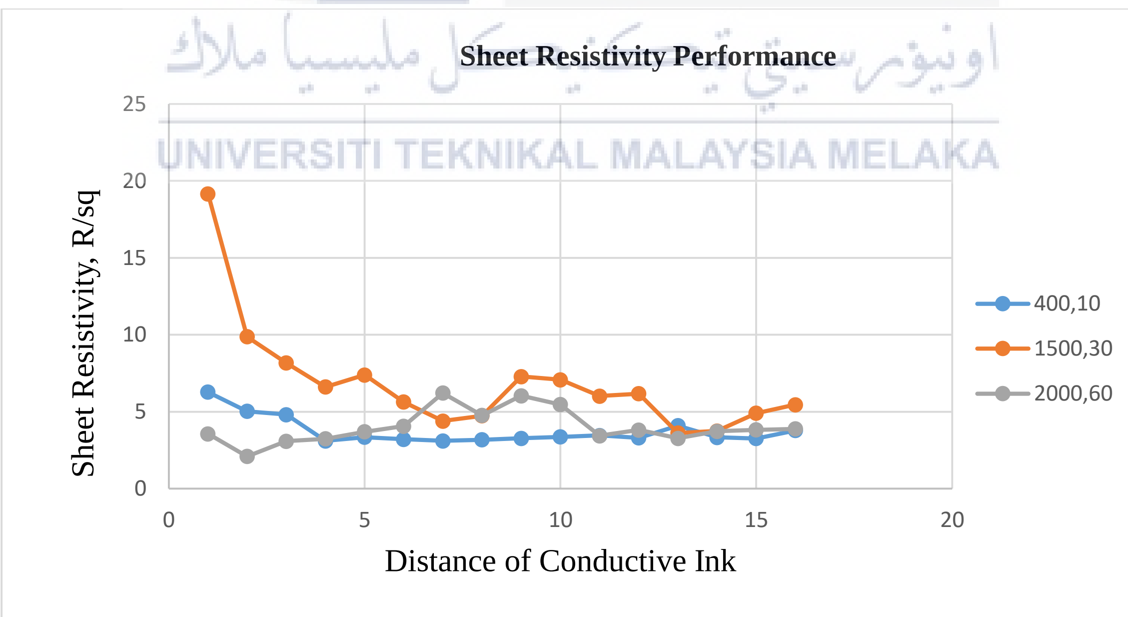


Figure 4. 1: Sheet Resistivity Performance of 2000,60, 1500, 30 and 400, 10

Table 4.1, 4.2, and 4.3 shows the distance range, average sheet resistivity, and the standard deviation data for each sample. Figure 4.4 shows the graph pattern on the data comparison between the mixing parameters. Each of the lines marked the highest point at the range distance of 1 to 4. Average value shows 10.95 Ω/sq for the 1500,30 sample, 4.8075 Ω/sq for 400,10 sample and 2.99 Ω/sq for 2000,60 sample. As for the standard deviation on the distance of 1 to 4 can be concluded that the lowest value mentioned to the 2000,60 sample with 0.6291 and 1.3037 for 400, 10 samples. The highest standard deviation is 5.6264 represented on the 1500,30 sample.

A high standard deviation proved that the data is widely spread which is less reliable and a low standard deviation shows that the data are clustered closely around the more reliable mean. This shows the highest resistivity and lowers electrical performance at the distance range from 1 to 4 on the 1500,30 sample. Meanwhile, 2000,60 sample marked the lowest sheet resistivity performance for the distance of 1 to 4 with higher electrical performance

Based on the graph line showed on Figure 4.1, the lowest point was marked at the distance range from 13 to 16. The 400,10 sample shows the lowest average sheet resistivity with 3.615 Ω/sq and a standard deviation of 0.3916. 4.4275 Ω/sq with a standard deviation of 0.8936 for the 1500, 30 samples. The average value for 2000, 60 sample is 3.68 Ω/sq and 0.2794 for the standard deviation value. This shows that 400,10 and 2000,60 samples have lower sheet resistivity performance compared to 1500,30 samples.

A conclusion can be made from the graph pattern which a sample of 400, 10 has the lowest sheet resistivity with higher electrical performance compared to the other samples. However, the graph lines between 400,10 and 2000, 60 are not significantly different but to consider the factor of time taken, 400, 10 samples can be highlighted as the optimized mixing parameter. The shorter the time taken the more the product can be produced at one time.

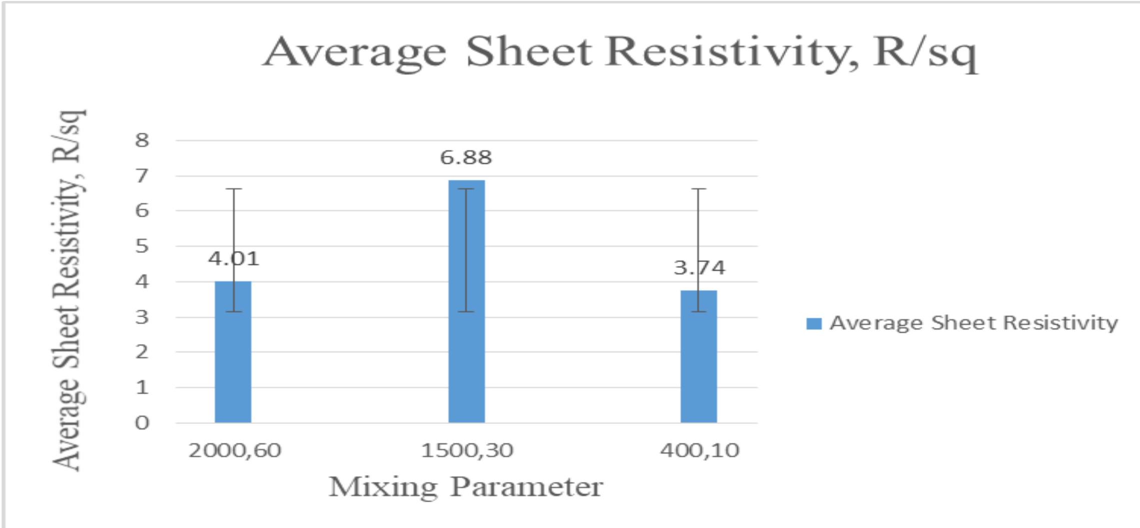


Figure 4. 2: A bar chart for Average Sheet Resistivity, Ω/sq against Mixing Parameters with Standard Deviation.

Figure 4.2 and mentioned on the bar chart for the Average Sheet Resistivity, Ω/sq against Mixing Parameters with Standard Deviation. Both chart shows that 400,10 marked the lowest value with 3.74 for average sheet resistivity, Ω/sq and 0.469 for the standard deviation. The highest ranked mentioned on the 1500,30 mixing parameter with the average sheet resistivity, ,Ω/sq of 6.88 and 2.1238 of standard deviation.

4.2.2 Data Comparison of Adhesion Promoter with Non-adhesion Promoter

Table 4. 4: Sheet Resistivity on Methyl Ethyl Ketone (MEK)

Distance of Conductive Ink	Sheet Resistivity, Ω/sq	Average Sheet Resistivity, Ω/sq	Standard Deviation
1 - 4	3.49	2.912	0.8167
	3.73		
	2.33		
	2.1		
5 – 8	2.72	2.520	0.6309
	3.27		
	2.31		
	1.78		
9 - 12	2.93	2.195	0.4972
	1.89		
	1.89		
	2.07		
13 – 16	2.29	2.352	0.3653
	1.97		
	2.85		
	2.3		

Table 4. 5: Sheet Resistivity on Anchor Spray Paint (ASP)

Distance of Conductive Ink	Sheet Resistivity, Ω/sq	Average Sheet Resistivity, Ω/sq	Standard Deviation
1 - 4	2.27	2.350	0.2851
	2.12		
	2.29		
	2.72		
5 – 8	2.43	2.132	0.2275
	2.18		
	2.01		
	1.91		
9 - 12	2.59	2.315	0.3944
	2.69		
	2.13		
	1.85		
13 – 16	1.69	1.882	0.2156
	1.85		
	1.8		
	2.19		

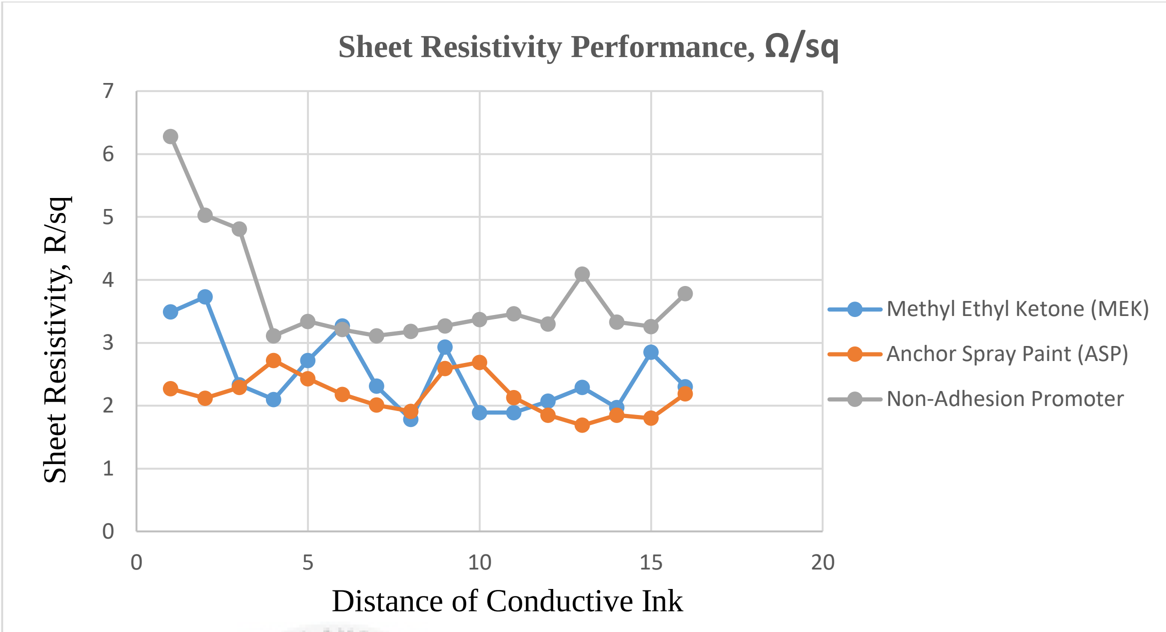


Figure 4. 3: Sheet Resistivity Performance of Adhesion Promoter and Non-Adhesion Promoter

Table 4.4 nd 4.5 mentioned the distance of conductive ink, average sheet resistivity and standard deviation on the adhesion promoter data involving Methyl Ethyl Ketone (MEK) and Anchor Spray Paint (ASP). Figure 4.3 shows the comparison of graph pattern between the sheet resistivity performance on adhesion promoter and non-adhesion promoter. The non-adhesion promoter refer to the sample that classified as the best mixing parameter which is 400, 10 sample.

Based on the data measured, the average sheet resistivity and standard deviation on the sample with Anchor Spray Paint (ASP) is the lowest compared to Methyl Ethyl Ketone (MEK) and Non-Adhesion Promoter sample with the value of 2.35 Ω/sq and 0.2851 respectively. Methyl Ethyl Ketone (MEK) reached 2.912 Ω/sq and 0.8167 for standard deviation while the non-adhesion promoter is the highest value with 4.80 Ω/sq and 1.3037 of standard deviation. It can be proved from the plotted graph on the range distance from 1 to 4 where the non-adhesion promoter has higher sheet resistivity performance. Hence, the sample shows that it has low electrical performance compared to the sample that applied adhesion promoter.

The lowest average sheet resistivity is 1.882 Ω/sq with a standard deviation of 0.2156 for the Anchor Spray Paint (ASP) in distance range from 13 to 16. The decreasing value shows that it has higher electrical performance. Meanwhile, the non-adhesion promoter still remarked as the highest average sheet resistivity with a value 3.61 Ω/sq and 0.3916 standard deviation. From the plotted graph, a conclusion can be made that applying an adhesion promoter on the substrate before print the conductive ink resulted the lowest sheet resistivity performance compared to the non-adhesion promoter substrate.

This might happen because the ink adhered well to the substrate and provide a consistent path for electrons flow that increase electrical performance. As an example, most of the spray paint have Volatile Organic Compound (VOCs) that include variety of chemicals. Each brand can differ but some of the common spray paint ingredients include acetone, xylene and carbon black that also contributed to conductivity. Each brand can differ but some of the common spray paint ingredients include silane coupling agent that produce the interaction with the organic polymer can be very complex. Silane bonds to the TPU substrate through chemical reactivity of the silane and polymer. The layered silane coupling agents can be effective and will give the best combination of properties in the final bonded materials.

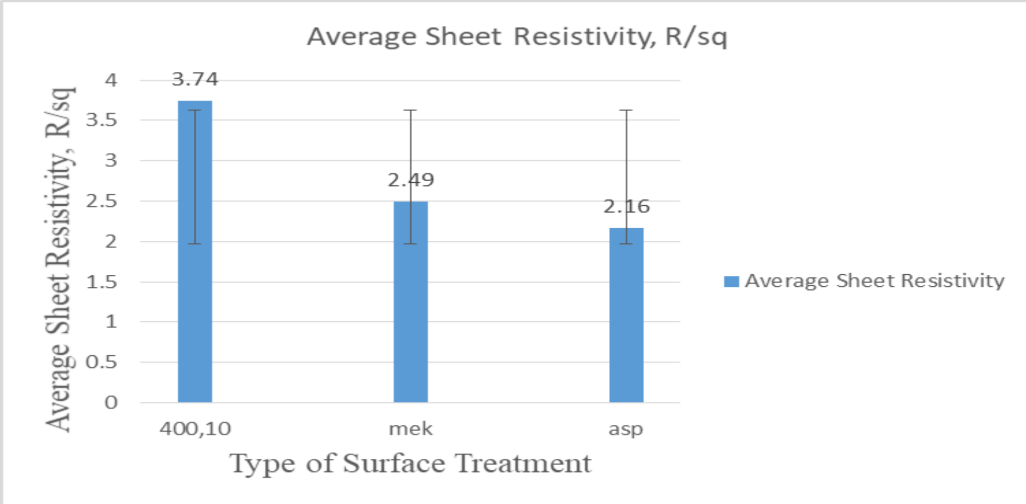


Figure 4. 4: A Bar Chart of Average Sheet Resistivity, Ω/sq against Types of Surface Treatment with Standard Deviation.

Figure 4.4 above shows on the bar chart for the Average Sheet Resistivity, Ω/sq against the Types of Surface Treatment with Standard Deviation. Both chart shows that ASP marked the lowest value with 2.16 for average sheet resistivity, Ω/sq and 0.281 for the standard deviation. The highest ranked mentioned on the 400,10 mixing parameter for the average sheet resistivity, Ω/sq of 3.74 and MEK concluded as the highest standard deviation among others with 0.578.

4.2.3 Surface of the Sample under Digital Microscope

Figure 4. 5: Without adhesion promoter

Figure 4.5 shows the image of conductive ink without adhesion promoter applied under a digital microscope. The picture shows on some uneven surface on certain area due to thin layered of printed ink.

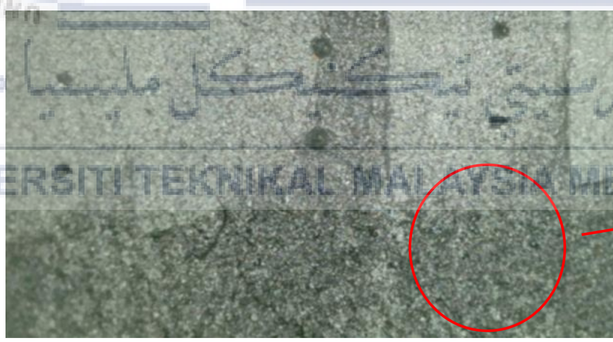


Figure 4. 6: Methyl Ethyl Ketone (MEK)

Figure 4.6 shows the image of conductive ink with adhesion promoter, Methyl Ethyl Ketone (MEK) applied under a digital microscope. The picture shows on some uneven surface on certain area due to thin layered of printed ink.

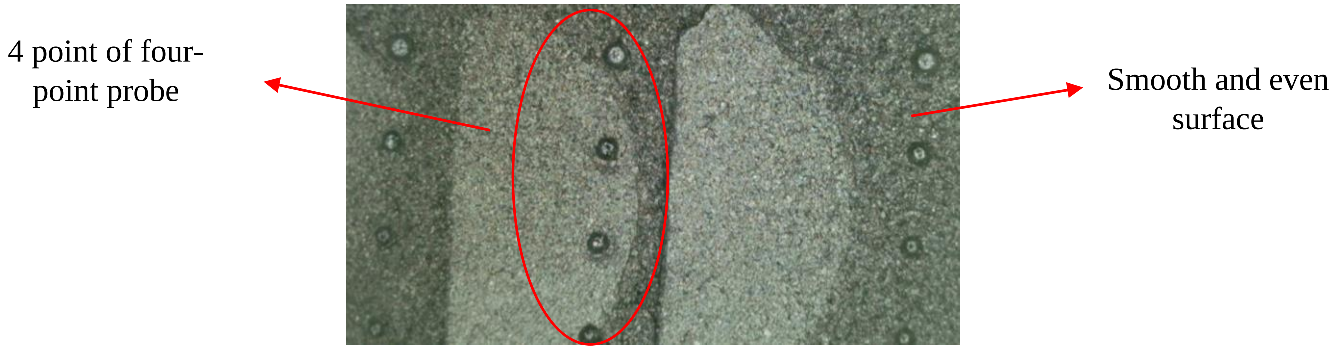


Figure 4. 7: Anchor Spray Paint (ASP)

Figure 4.7 shows the image of conductive ink with adhesion promoter, Anchor Spray Paint (ASP) applied under a digital microscope. The picture shows the ink surface was in a good condition, smooth and even.

Based on the image captured,. the conductive ink sticks to the substrate neatly without any cracks occurring unless for the uneven surface due to thin layered while printing the ink. There is no significant difference on the substrate surface or conductive ink condition after applied an adhesion promoter or without applied adhesion promoter. As for the ink printed on the TPU substrate treated with ASP is much smoother and even. It contributed to consistent path for electron to flow resulted in low resitivity with higher electrical performance. This might happen because the formulation used for conductive ink is acceptable and the mixing parameters are fit together with the formulation, generating an ideal ink paste. Moreover, the steps and tools used to print the ink are important to ensure that the conductive is spread on the TPU substrate uniformly while printing.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The main objectives of this research are to optimize the mixing parameter of stretchable conductive ink on the sheet resistivity performance and to investigate the effect of chemical surface treatment on the substrate of the stretchable conductive ink. A total of five samples were printed by using the manual-printing method for this research. The filler used is 7.5 wt% of graphene fix for all samples. PEDOT:PSS acts as a binder and a few solvents are also added in the formulation such as DMSO, EG, and Triton X-100. There were three different mixing parameters as manipulated variables which are 400 rpm for 10 minutes, 1500 rpm for 30 minutes, and 2000 rpm for 60 minutes. The substrate used was Thermoplastic Polyurethane (TPU) which is commonly known with many properties including elasticity, transparency, and resistance to oil, grease, and abrasion.

Next, the sheet resistivity performance was recorded for all the samples using a four-point probe measurement. The data collected shows that samples with mixing parameters of 400 rpm in 10 minutes reached the lowest sheet resistivity performance compared to the other samples. Hence, it can conclude that the optimized mixing parameter goes to 400 rpm in 10 minutes. Two types of adhesion promoters were used in this experiment as the chemical surface treatment such as Methyl

Ethyl Ketone (MEK) and Anchor Spray Paint (ASP) to test the sheet resistivity performance on the conductive ink.

The data measured were compared with the samples without adhesion promoter on the sheet resistivity performance. The sample of 400 rpm in 10 minutes only was chosen to plot the comparison. The result shows that the sample with an adhesion promoter have better sheet resistivity performance. As for the extra information on the sheet resistivity performance between ASP and MEK, the lowest sheet resistivity represented more on ASP.

5.2 Recommendation

For the future work and improvement of study, to improve the sheet resistivity performance result, the step should be done properly, good printing technique, suitable tools to print the ink on the substrate, and have complete equipment. This has to take serious action to reduce any human error on sheet resistivity performance for all samples. The curing time should follow the types of substrate used whether it can withstand high temperature or above its range. Plus, detailed experimental observation data must acquire to alert the conductive ink characteristics. To minimize the testing error during conducting the test, follow the steps and safety precautions in a correct order.

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