EFFECTS OF SURFACTANTS AND THICKNESS OF CARBON NANOTUBE FILM ON CONDUCTIVE PROPERTY FOR TOUCH SCREEN

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ผลของสารลดแรงตึงผิวและความหนาของฟิล์มท่อนาโนคาร์บอนต่อสมบัติการนำไฟฟ้าสำหรับจอสัมผัส

น.ส.ฐิมาภรณ์ ชาญณรงค์

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ฐิมาภรณ์ ชาญณรงค์ : ผลของสารลดแรงตึงผิวและความหนาของฟิล์มท่อนาโนคาร์บอนต่อสมบัติการนำไฟฟ้าสำหรับจอสัมผัส . (EFFECTS OF SURFACTANTS AND THICKNESS OF CARBON NANOTUBE FILM ON CONDUCTIVE PROPERTY FOR TOUCH SCREEN) อ.ที่ปรึกษาหลัก : รศ. ดร.อรัญ หาญสืบสาย, อ.ที่ปรึกษาร่วม : ศ. ดร.ชิเกรุ ทากาฮาร่า

เทคโนโลยีฟิล์มโปร่งใสที่นำไฟฟ้าได้รับการพัฒนาอย่างกว้างขวางสำหรับอุตสาหกรรมอิเล็กทรอนิกส์ เช่นระบบสัมผัสและอื่น ๆ งานวิจัยนี้ได้ศึกษาท่อนาโนคาร์บอนแบบชั้นเดียวหรือผนังเดี่ยวที่กระจายตัวในสารละลายอะคริลิกและช่วยการกระจายตัวของท่อนาโนคาร์บอนโดยสารลดแรงตึงผิว การกระจายตัวของท่อนาโนคาร์บอนเป็นหนึ่งในปัจจัยสำคัญที่มีอิทธิพลอย่างมากต่อคุณสมบัติของฟิล์มโปร่งใส จึงทำการเปรียบเทียบผลของการใช้สารลดแรงตึงผิวไฮโดรคาร์บอนและซิลิโคน ซึ่งเป็นสารลดแรงตึงผิวประเภทโมเลกุลไม่มีประจุ เทคนิคการเคลือบแบบเส้นลวดถูกนำมาใช้ในการเคลือบสารท่อนาโนคาร์บอนบนพลาสติก PET วัตถุประสงค์ของงานวิจัยนี้เพื่อศึกษาผลของสารลดแรงตึงผิวและความหนาของฟิล์มท่อนาโนคาร์บอนต่อสมบัติการนำไฟฟ้า ความเข้มข้นของสารลดแรงตึงผิวที่ใช้ร้อยละ 0.1 - 2.0 โดยน้ำหนัก เคลือบบนแผ่นพลาสติกโดยใช้เส้นลวด K-bar เพื่อได้ความหนาแตกต่างกัน โดยใช้เทคนิคการเคลือบแบบชั้นเดียวและหลายชั้นเข้ามาช่วยในการเพิ่มความหนาของฟิล์มท่อนาโนคาร์บอนวิเคราะห์ความโปร่งใสของฟิล์มท่อนาโนคาร์บอนและความต้านทานไฟฟ้า ผลจากการทดลองพบว่าความเข้มข้นของสารลดแรงตึงผิวที่แตกต่างกัน เมื่อเคลือบที่ความหนาเดียวกัน พบว่า เมื่อเพิ่มความเข้มข้นของสารลดแรงตึงผิวทั้ง 2 ชนิด ทำให้ความโปร่งใสของฟิล์มนำไฟฟ้าเพิ่มขึ้นเล็กน้อย และค่าความต้านทานแผ่นทั้งสองลดลง แต่เมื่อเพิ่มของความหนาของการเคลือบสารท่อนาโนคาร์บอนพบว่า ค่าความโปร่งใสและค่าความต้านทานไฟฟ้ามีค่าลดลง เมื่อใช้สารลดแรงตึงผิวชนิดซิลิโคนและไฮโดรคาร์บอนที่ความเข้มข้นร้อยละ 2.0 โดยน้ำหนัก แผ่นที่ให้ค่าความต้านทานไฟฟ้าต่ำที่สุด มีค่า 8.91×10^{2} และ 2.63×10^{3} โอห์มต่อพื้นที่ สารลดแรงตึงผิวชนิดซิลิโคนและไฮโดรคาร์บอนตามลำดับ จากการเคลือบที่ความหนา 250 ไมครอน เห็นได้ว่าสารลดแรงตึงผิวชนิดซิลิโคนจะให้ผลการนำไฟฟ้าที่ดีกว่าสารลดแรงตึงผิวชนิดไฮโดรคาร์บอน

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Conductive transparent film technology has been developed extensively for electronic industry such as touch panel and so on. In this study, single-wall carbon nanotubes (SWCNTs) was chosen to disperse in acrylic resin and homogenized by surfactant. The dispersion of SWNCT is one of the key factors that strongly influences the properties of these products. Thus Hydrocarbon and Silicone surfactant types were compared. The wire-bar coating technique was used to produce a conductive film PET base. Our objectives were to study the effects of surfactants and thickness of SWCNT film on conductivity and transparency. Surfactant concentration was varied 0.1 – 2.0 % w/w. Film formation samples were done by using wire K-bar rods varied the thickness using single and multi-coating method. Transparency and sheet resistance were analyzed. Results showed that the varying surfactant concentration and coating thickness did slightly affect obviously the change of transmittance of conductive films and sheet resistance varied between 10^4 – 10^5 Ω/square for both surfactants. While sheet resistance declined relevant to the increase of SWCNT thickness and the amount of surfactants. Results showed Silicone surfactant gave better results than Hydrocarbon surfactant, the optimum sheet resistance was at 8.91×10^2 Ω/square with 87% transmittance (including PET film) and 2.63×10^3 Ω/square with 86% transmittance by using 2.0 %w/w of silicone and hydrocarbon surfactant respectively with the film thickness of 250 microns.

Field of Study:	Imaging Technology	Student's Signature ....
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		Co-advisor's Signature ....

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Thimaporn Channarong

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CHAPTER 1
Introduction

A touch screen is an input device and normally layered on the top of an electronic visual display of an information processing system. A user can give input or control the information processing system through simple or multi-touch gestures by touching the screen with a special stylus or one or more fingers. Touchscreens are common in devices such as smart phone, computer, personal digital assistants (PDAs) and so on. The touch screen technologies with different methods of sensing touch such as resistive and capacitive touch screen. Transparent conductive films electrodes have been widely used in electronic devices such as solar cells, displays, memories and batteries. The transparent conductive film is an important part of resistive touch screen. In particular, indium tin oxide (ITO) has attracted considerable attention as a transparent conductive electrode because of its excellent optical and electrical properties. ITO has some disadvantages in terms of production and cost. Moreover, ITO application in flexible devices is limited. To replace ITO in flexible devices, several types of conductive material such as graphene and carbon nanotubes (CNTs) [1]. The tubes, either single-walled carbon nanotubes (SWCNTs) or multiple-walled carbon nanotubes (MWCNTs). There is great interest in single-walled carbon nanotubes because of their excellent mechanical, thermal and electrical properties. This feature can be used in coating to get transparent and conductive property. Transparent and electrically conductive coatings on flexible films will be useful for electronic device for flat panel displays, touch screen panels, solar cell and light emitting diodes (LEDs). Methods are used in order to get transparent conductive film from carbon nanotube such as vacuum-filtration, spray coating, spin-coating and dip coating. However, short time, large area and continuous processes are important to producing SWCNT coating. The wire bar method, which wraps a stainless steel wire around a shaft, is useful equipment to spread coating and ink on flexible materials. Which can control the film thickness and simplicity [2]. Layer structure of the transparent conductive film may have a single or more layers. Improving the conductive properties of plastic materials will contribute to the development of transparent conductive film, packaging and other fields. Poly(ethylene terephthalate) (PET) film are widely used transparent thin film, solar cell, printed electronic and so on. Because their high transparency, flexibility, heat setting, gas and vapor barrier, excellent mechanical properties. A method for conductive transparent films was found which have properties with good performances, such as transmittance, low sheet resistance, flexibility and good adhesion on substrate. Single-walled carbon nanotubes (SWCNTs) have poor adhesion on PET film. To this problem, water base binder was chose as a binder to improve adhesion. Polyurethane (PU) was found to improve adhesion on substrate because it has high adhesion and flexible, widely used in adhesives and coating but it is low glass transition temperature (Tg) cause sticky surface problem. Acrylic binder was used to this research because it is thick, liquid and milky white when wet, but colorless and transparent when dry, has good adhesion on PET film and very good applicability, flow and leveling for wire- bar coating technique. The conductive transparent film must need low sheet resistance and high transmittance, when the transmittance of the conductive film is low, reduces the brightness of the display, a display for the display screen is dark become difficult to see. Carbon nanotubes were dispersed in water base resin or aqueous solution using a surfactant for increasing transmittance. The common surfactant were used carbon nanotube dispersion are non-ionic and anionic surfactants such as Triton X-100, Sodium dodecyl sulfate (SDS), Sodium dodecylbenzenesulfonate (SDBS), Sodium deoxycholate (DOC) and so on. Surfactants may act as dispersants, detergents, emulsifiers, wetting agents and foaming agents. The SWCNT dispersion in water base solution have been reported by using surfactant for homogenous solution. There are many research about dispersing CNTs by surfactant [3]. The silicone surfactants, their high lubricity and good spreading properties, the Si-O-Si chain has been confirmed that it has strong interaction with CNTs. Therefore, our research was used silicone surfactant for SWCNT dispersion.

The Wire-bar coating technique, which wraps a stainless steel wire around a shaft, is useful equipment to spread inks and coatings on flexible materials. The coating thickness can be controlled by the area in the groove between the coils of wire because the short coating time, ability to extend the coating area, and continuous processes are important means to improve the efficiency of producing SWCNT coatings.

Our objectives were to study the effects of surfactants and thickness of single-walled carbon nanotube (SWCNT) film on conductivity and transmittance using the single and multi-coating technique by wire-bar coater was used to produce a conductive film PET base and preparation SWCNT dispersion with hydrocarbon and silicone non-ionic surfactant types were varied concentration and thickness coating.

For this study, the conductive transparent films and material has been rapidly changing and continuous developing at all level: application such as flexible display, printed electronic, touch panel and solar cell. For technologies: the market of transparent conductive film increased and growth especially touch technologies in mobile, smart watch, tablets, notebook, AiOs, automotive display, and so on. And last, supplier or manufacturing are growth too.

CHAPTER 2

Literature reviews

2.1 Touchscreen or touch panel

Touch screen is a display device that allows the user to can control through simple touch by using their finger or pen/stylus. Touch screens are used on a variety of devices, such as computer and displays, smartphones, personal digital assistant (PDA) and tablets. The popularity of smart phone and personal digital assistant is driving the demand of touch screen and many functional electronic. The market for touch screen and electrical material has been rapidly changing and growing. Predicted that in the next 10 years, in 2029, the electrical industry (transparent conductive films) will grow up 50% to the present. History of the touch screen as shown in Figure 1 [4].

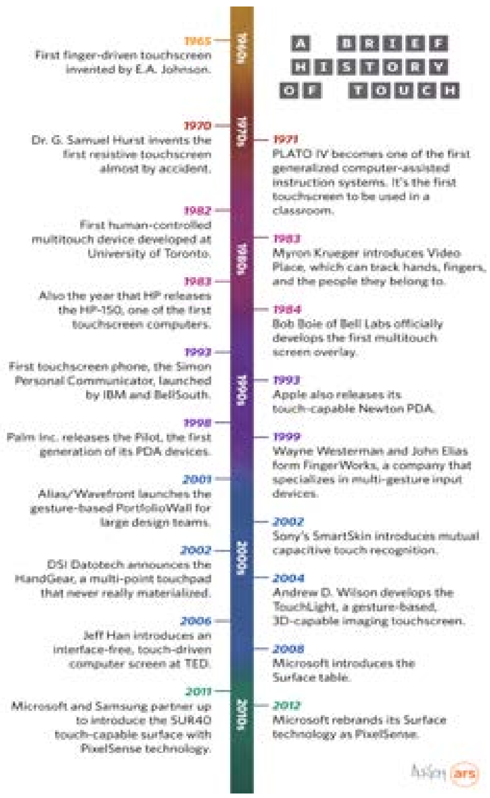


Figure 1 A brief history of touch

2.2 Two main types of touch screen technologies

2.2.1 Capacitive touch screen

A capacitive touch screen also consists of 2 spaced layer of glass, which are coated with conductor such as Indium Tin Oxide (ITO) and Carbon nanotubes (CNTs) Human body is electrical charge conductor. When a finger touches the glass of the capacitive surface. The system continuously monitors the movement of each tiny capacitor to find out the exact area where the finger had touched the screen as shown in Figure 2 and 3.

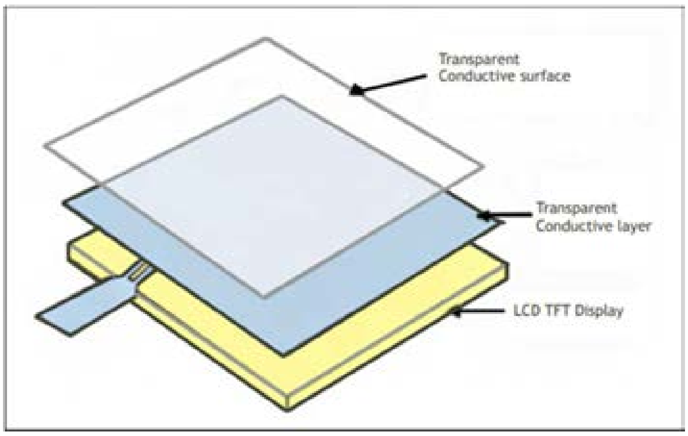


Figure 2 The main layer of capacitive touch screen

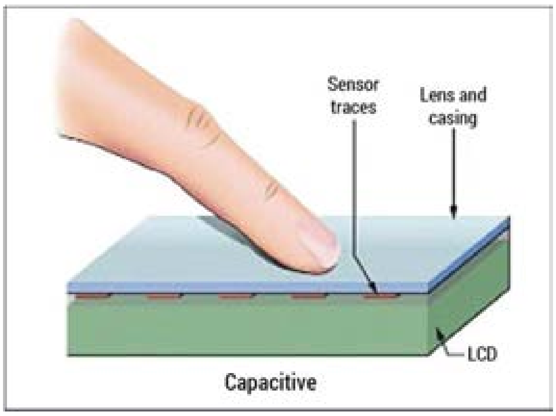


Figure 3. Structure of capacitive touch screen

2.2.2 Resistive touch screen

A resistive touch screen comprises of several layers, out of the flexible plastic and glass layers are to important electrically resistive layers. The top surface of resistive touch screen is a scratch resistant plastic with coating of conductive material such as carbon nanotube and ITO, printed underside. Both layers are separated with a thin gap in between. An electrical resistance is created between both layers, when a finger or pen presses down on the top, both the conductive transparent film meet and get an accurate measurement of the touch positive. The accuracy also relies on the evenness of the coating of conductive material on both the layers as shown in Figure 4 and 5 [5].

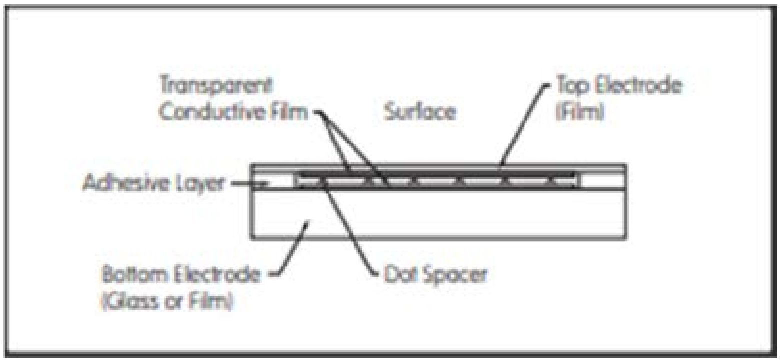


Figure 4. Cross section view for resistive touch screen

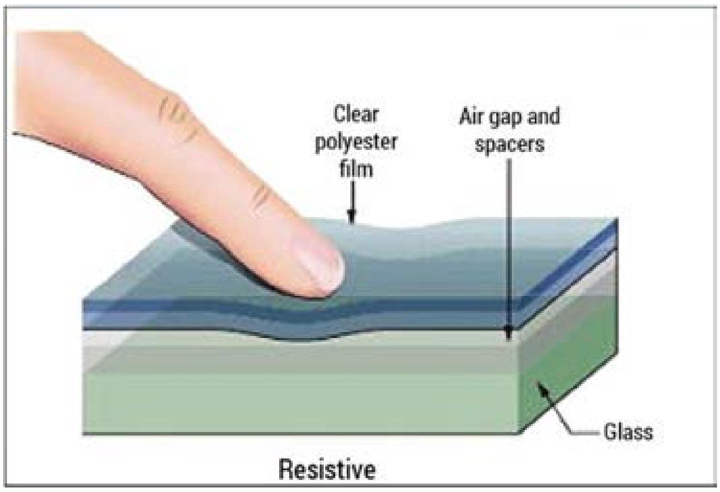


Figure 5. Structure of capacitive touch screen

In touch screen applications in each industry, can choose to use as appropriate for that product by comparing the advantages and disadvantages of the capacitive and resistive touch screen as shown in Table 1 and 2.

Table 1. Comparison of capacitive touch screens

Advantages	Disadvantages
Brighter and sharper display because capacitive touch screen has glass layer	Expensive to produce
High touch sensitivity	Cannot use without gloves or stylus (finger only)
Support multi-touch	Glass is more prone to breaking

Table 2 Comparison of resistive touch screens

Advantages	Disadvantages
Low production cost	Low sensitivity, you have to press down hardness
High resistance to dust and water	Does not support multi-touch
Can be used with a glove or stylus	Poor contrast because of having additional reflections from extra layer of material placed over the screen
Less resistant to breakage	More susceptible to scratching

2.3 Transparent conductive films (TCFs)

Transparent conductive films (TCFs) are thin films of optically transparent and electrically conductive material. They are an important component in a number of electronic devices including flexible display such as touch screens and touch panel, LEDs and printed electronics applications such as transparent electrodes, RFID tags, thin-film transistors, light-emitting devices, and solar cells. Two main factors in the production of conductive films are transparency and electrical resistance. Which % transmittance is greater than 80% and the sheet resistance is between 120 – 1500 Ω/square [6]. In particular, indium tin oxide (ITO) has attracted considerable attention as a transparent conductive electrode for touch panel because of its excellent optical and electrical properties [3]. The transparent conductive film layer is laminated surface by transparent resin sheet bonded laminated via an adhesive. The pressure-sensitive adhesive, it has transparency such as acrylic adhesive, silicone adhesive and rubber based adhesive. ITO has some disadvantages for touch panel of finding alternatives to expensive and brittle ITO-based devices. ITO films have a better electrical conductivity than CNT films. The transmittance is also important for touch screen and is typically between 85-90 % (the higher than better) [1].

Furthermore the comparison ITO and CNT material in term of simple conductive coating using spray-coating. The resulted presented that ITO have a better electrical conductivity than CNT but CNT is good transmittance. SWCNTs are more suitable for transparent conductive coating than MWCNTs [7]. Moreover, ITO application in flexible devices is limited. While carbon nanotubes (CNTs) is widely used for flexible display. Preparation of carbon nanotube thin films for transparent conductive film is composed of three steps: first step, the carbon nanotubes growth process, putting the CNTs in solution, and finally, creation of the CNT thin film. Carbon nanotubes (CNTs) are cylindrical molecules that consist of rolled-up sheets of graphene.

2.4 Carbon nanotubes (CNTs)

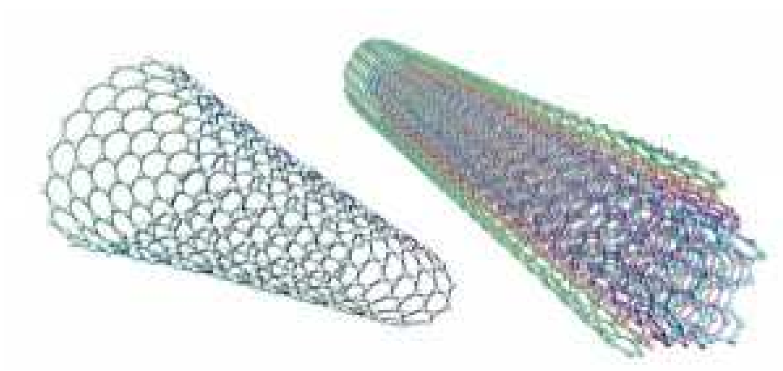
CNTs can be single-walled carbon nanotubes (SWCNT) if made from one layer of carbon atoms, or multi-walled carbon nanotubes (MWCNT) when consisting of several layers of graphene sheets. The structure of CNT in Figure 6. The rolling-up direction of the graphene layers determines the electrical properties of the nanotubes [8]. Single-walled carbon nanotubes properties and applications which take full advantage of CNTs aspect ratio, mechanical strength, electrical and thermal conductivity. The properties of SWCNT are below:

Mechanical: Individual SWCNTs are significantly stronger than steel. Calculated values for tensile strength of SWCNT are ~ 100 times greater than steel at 1/16th the weight. The highest measured value is approximately half of the predicted theoretical strength, the difference possibly being due to defects in the structure.

Electrical: Individual SWCNTs have current carrying capacities of 10^9 amp.cm^{-2}, higher than those of copper or gold, and semiconducting species exhibit higher electron mobility than silicon.

Optical: SWCNTs have a distinct optical absorption and fluorescence response, with each chirality demonstrating its own characteristic absorption and fluorescence spectrum.

Thermal: Room temperature thermal conductivity of a single nanotube may be comparable to that of diamond or in-plane graphite, which is generally thought to display the highest measured thermal conductivity of any known material at moderate temperatures.



(a) SWCNT (b) MWCNT

Figure 6 Structure of carbon nanotube: (a) SWCNT and (b) MWCNT

Single wall carbon nanotube can be described as a graphene sheet rolled into a cylindrical shape so that the structure is one-dimensional with axial symmetry. Nano tube structures are represented by the following parameters [9].

1. Arm chair type

   The symmetrical classification of an armchair nanotube is an achiral nanotube. Achiral means the nanotube has a structure that is a mirror image of the original one. An armchair nanotube has a chiral vector where n = m, therefore vector = (n, n). The chiral angle is equal to 30°.

2. Zigzag type

   The symmetrical classification of a zigzag nanotube is an achiral nanotube, the same as an armchair nanotube. A zigzag nanotube has a chiral vector where m = 0, therefore vector = (n, 0). The chiral angle is equal to 0°.

3. Chiral type

   The symmetrical classification of a chiral nanotube is a chiral nanotube. The structure of a chiral nanotube. A chiral nanotube has general n and m values, therefore vector = (n, m). The chiral angle is between 0° and 30°, therefore 0° < chiral angle < 30°.

Four main methods are currently available for the production of CNTs: arc discharge, laser ablation, chemical vapor deposition (CVD), and high-pressure carbon monoxide disproportionation (HiPCO). The CVD growth method is popular, as it yields high quantity and has a degree of control over diameter, length and morphology. Various methods have been used in the manufacture of SWCNTs. These include laser ablation, carbon arc and CVD processes, either involving a gaseous catalyst as in the HIPCO process or using a supported catalyst as in the CoMoCAT process. The laser ablation process is used primarily for research materials. The carbon arc process produces long tubes with diameters in the range 1.4 to 2.0 nm, but carbon arc material has a large amount of impurities and for most applications will require extensive purification. The CVD processes offer the best approach to the manufacture of larger SWCNTs quantities, with perhaps the most scalable being the CoMoCAT process which uses a fluidized bed reactor similar to those used in petroleum refining, albeit, currently on a much smaller scale. The supported catalyst approach also offers the unique ability to provide a substantial degree of chirality control during synthesis.

Optical absorption can be used to evaluate the distribution of (n,m) species in a given sample. The spectrum in Figure 7 corresponds to a sample produced by the standard CoMoCAT method, which exhibits a high concentration of the specific (6,5) nanotube type and a narrow range of diameters and chiral angles (around 0.76 nm and 27 degrees, respectively) and the concentration of semiconducting nanotubes is higher than 90% [10].

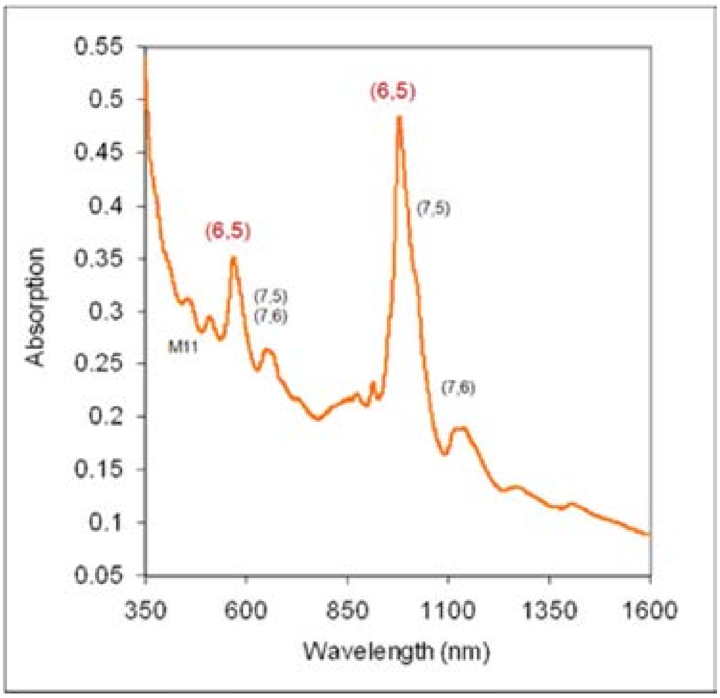


Figure 7. Optical Absorption Spectra showing the predominance of the (6,5) nanotube when the CoMoCAT method

SWCNTs have stimulated a great of activity in both the research and industry because it can be easily twisted and is more flexible, characterization, evaluation is easy and have better conductivity than MWCNTs but have poor adhesion to the PET substrate. Second step, preparation of SWCNT dispersion.

2.5 Binders

Binders are liquid or dough-like substances that harden by a chemical or physical process and bind fibers. Binders are loosely classified as organic and inorganic. These can be either metallic or ceramic as well as polymeric depending on the nature of the main material. The binder, or resin, is usually what gives the coating its name, such as polyurethane or acrylic. Though there are many types of resins, there are two setting methods for the synthetic resins used in paints: thermoset and thermoplastic resin [11].

2.5.1 Thermoplastic resins

Resins which are thermoplastic remain plastic after setting. They do not

cure irreversibly, but soften with heat. Film formation occurs through evaporation of the solvent or water. Thermoplastic resins for paint provide excellent protection against corrosive materials and environments

2.5.2 Thermosetting resins

These resins cure irreversibly when exposed to the right conditions.

These can be extreme heat, chemical reaction or irradiation in the case of UV-cured thermoset resins. Uncured, thermoset resins are in a viscous, liquid state. During the curing process, the molecules that make up the resin form crosslinks, combining into long, tightly-bound polymers which set permanently. Because of these crosslinks, thermoset resins provide excellent adhesion as well as resistance to heat, chemicals, and water.

A resin is a solid or highly viscous substance of plant or synthetic origin. The term “resin” is applied to the component of a liquid that dries to a hard film or finish. Five types of binders with unique properties

Epoxy (thermoset)

Epoxy resins, like polyurethanes, are thermosetting polymers that form

a tough film through the process of crosslinking. There are a variety of formulations of epoxy resin including bisphenol A epoxy, bisphenol F epoxy (which has lower viscosity than bisphenol A epoxy) and aliphatic epoxy. Each of these resin formulations result in a slightly different epoxy resin coating, with different strengths. Epoxy resins are generally two component, though single component formulations are available, and they are water or solvent based.

Polyester and vinyl ester resins (thermoset)

A polyester resin is formed by the polymerization of an alcohol and an

acid. A vinyl ester resin is a subclass of polyester resins. There are both thermosetting resins, and the resin is usually dissolved in a monomer such as styrene in order to reduce their viscosity and make them workable. Polyester and vinyl ester provide superior chemical and temperature resistance to most coatings. They are also abrasion resistant, quick curing, and provide long term corrosion protection.

Acrylic resins (thermoset & thermoplastic)

Acrylic resins account for nearly 30% of the coating resin market.

Acrylic resin is derived from the polymerization of acrylate and methacrylate monomers (such as methyl methacrylate or MMA) and can be thermoset or thermoplastic. Acrylic resins are known for their good color and gloss retention, as well as their superior weathering and UV resistance.

Polyurethane resins (thermoset)

Polyurethanes are reaction polymers created by reacting an isocyanate

with a polyol. The types of isocyanate and polyol. Polyurethane coatings come in one or two component varieties and can be either water or solvent based. Polyurethanes have excellent chemical and solvent resistance, corrosion resistance, weather resistance, UV stability, abrasion resistance, gloss durability, hardness, and flexibility.

Alkyd resins (thermoset & thermoplastic)

An alkyd is the result of the polymerization reaction between an alcohol (like glycerol) and an acid, modified by the addition of oils. They are also known as oil-modified polyesters. They are also known as oil-modified polyesters. The oil content of the formulation varies; a short oil alkyd has the lowest percentage of fatty acid by weight (less than 40%), then medium oil (40-60%), then finally long oil (60-70%). Long oil alkyds are the slowest drying and most flexible, where short oil alkyds are fast drying and form the hardest films. The desirable properties of alkyds include good adhesion, hardness, flexibility, corrosion resistance, and gloss retention.

Binders (or resins) are the film-forming element of coating or adhesive. It provide adhesion to the substrate, pigments and extenders together, and determines properties such as durability, flexibility, hardness, gloss, chemical and heat resistance. Water based binder and resin (emulsion) for coating, typically water based such as styrene, full acrylic, styrene acrylic, vinyl resin, aromatic and aliphatic urethanes. When one choose a resin or binder it affects, price and evaluate the following: wetting/flow/leveling, flexibility, adhesion/cohesion, stability with the colorant, abrasion wet and dry, method of the coating and so on. The water-based polyurethane (WPU) as the binder to improve the adhesion between the carbon nanotube and the PET for antistatic application. WPU is a linear thermoplastic polymer and has many advantage such as non-toxicity and has been used in adhesive, coating materials and so on. The antistatic films obtained has special characteristic, such as high transparency, low sheet resistance and excellent resistance to water and heat, like as touch screen application [12]. In this research, acrylic copolymer is binder to improve the adhesion between SWCNT and substrate for touch screen application. Acrylic copolymer is a yellowish translucent liquid but colorless and transparent when dry, that is a self-cross-linking acrylic co-polymer emulsion. It is for flexo and gravure printing and offers excellent chemical resistance and good adhesion properties on a variety packaging films. It does not yellow when exposed to sunlight, even after many years. The important factor for choosing binder is the flow and viscosity of polymer. Because it will greatly affect the coating by the flow characteristics and viscosity of the polymer as shown in Figure 8.

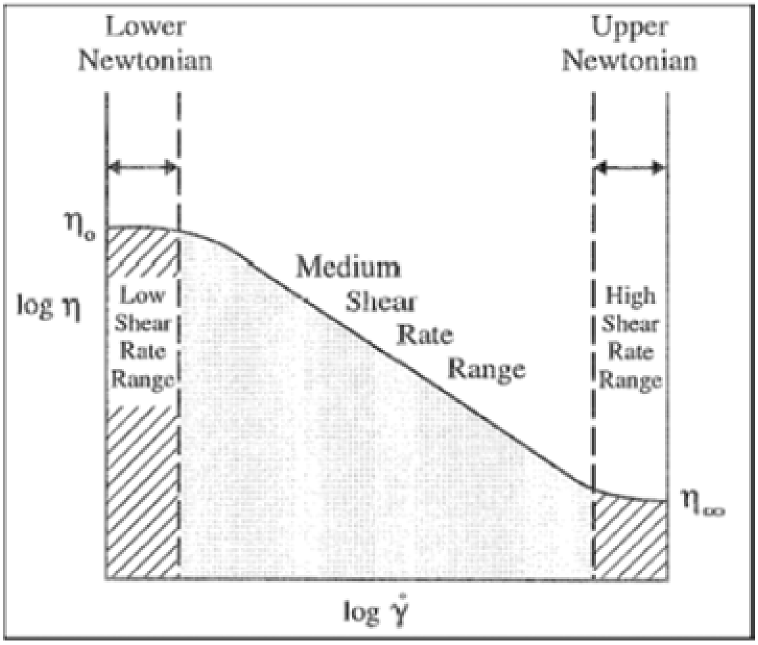


Figure 8. The relationship between shear viscosity and shear rate

2.6 Surfactants

SWCNT dispersions were prepared in aqueous solution, carbon nanotubes are not well distributed. Therefore the surfactant is used to help dispersing. Single-walled carbon nanotubes (SWCNTs) have been suspended in aqueous media using various anionic, cationic, nonionic surfactants and polymers.

Surfactants are materials that lower the surface tension (or interfacial tension) between two liquids or between a liquid and a solid. In the general sense, any material that affects the interfacial surface tension, can be considered a surfactant, but in the practical sense, surfactants may act as wetting agents, emulsifiers, foaming and anti-foaming agents, and dispersants. Surfactant must have a chemical structure with two different functional groups with different affinity within the same molecule. Usually the molecules of the substances called surfactants have both an alkyl chain with 8–22 carbons. This chain is called a hydrophobic group, which does not show affinity to water (they are called hydrophobic groups since surfactants are often used in water systems. Surfactants are classified into ionic surfactants and nonionic surfactants. Ionic surfactants are sub-classified into anionic surfactants where the hydrophilic group dissociates into anions in aqueous solutions, cationic surfactants that dissociate into cations, and amphoteric surfactants that dissociate into anions and cations often depending on the pH [13]. Surfactant of anionic, cationic, and nonionic surfactants and polymers have been tested for their ability to suspend individual single-walled nanotubes. A nonionic surfactant or polymer's ability to suspend nanotubes appears to be due mostly to the size of the hydrophilic group, with higher molecular weights suspending more nanotube material because of enhanced steric stabilization with longer polymeric groups [14]. The nonionic surfactant are not in the ionic state in the solution, thereby having high stability and being less susceptible to the effect of strong electrolyte inorganic salts as well as acid and alkalis; it has excellent compatibility with other types of surfactants and have excellent solubility (which vary depending on different structures) in both water and organic solvents. Moreover, it also has good compatibility with anionic, cationic surfactants or amphoteric surfactants and can be used in formulation. The shape of the molecules depends on the balance in size between hydrophilic head and hydrophobic tail. A measure of this is the HLB. The HLB value is an indicator of solubility of surfactant. Surfactant selected on the basis of hydrophilic-lipophilic balance (HLB) number can be used. Hydrophilic-lipophilic balance (HLB) value of surfactant shown in table 3.

Table 3 Hydrophilic-lipophilic balance (HLB) value of surfactant

Hydrophilic-lipophilic balance (HLB)	**Use**
1-4	Anti-foams
4-6	Water-in-oil emulsifiers
7-9	Wetting and spreading agent
8-12	Oil-in-water emulsifiers
13-15	Detergent
15-18	Solubilizing agent

The surfactant to dispersants are surface-active substances added to a suspension, usually a colloid, to improve the separation of particles and to prevent settling or clumping. Dispersants consist normally of one or more surfactants in a solution. Most surfactants' "tails" are fairly similar, consisting of a hydrocarbon chain, which can be branched, linear, or aromatic. Siloxanesurfactants have siloxane chains. Recent advances in surfactant technology has seen the development of mixed chains or/and complex structures. Many research about dispersing CNTs by surfactant, silicone surfactant applied in aqueous solution. However, the Si-O-Si chain has strong interaction with carbon nanotubes[15] .The silicone surfactant molecule consists of hydrophobic Si-O-Si chain and hydrophilic part of polyethylene oxide chain (EO) with a combination molecular structure. The Si-O-Si chain is flexible due to long Si-C bond and it can easily wrap onto surface of carbon nanotube via hydrophobic and other intermolecular interaction. The silicone surfactant could wrap onto the surface of CNTs leading to steric stabilization so that it could well disperse CNTs, and Van der Waals attraction was the dominating force of the silicone surfactant adsorbing onto CNT [16].

2.7 Substrates

Substrates (or materials) were used for conductivity of carbon nanotube such as membrane, glass and plastic. Plastic was chose the flexible display device, there are so many different types of plastic material [17] . The following are the seven of the most popular and commonly used plastics:

1. Acrylic or Polymethyl Methacrylate (PMMA), also known as acrylic or acrylic glass, is a transparent and rigid thermoplastic material widely used as a shatterproof replacement for glass. PMMA has many technical advantages over other transparent polymer. Limitations of PMMA: poor impact resistance, limited heat resistance (80°C), Limited chemical resistance and Poor wear and abrasion resistance
2. Polycarbonate (PC) is a high-performance tough, amorphous and transparent thermoplastic polymer. It is used as an engineering plastics and many properties: good electrical properties, high impact strength, transmittance and heat resistance. There are also certain limitations associated with polycarbonate plastics: Low fatigue endurance, proper drying before processing is needed and Yellows after long exposure to UV.
3. Polyethylene (PE) is used in applications ranging for films, tubes, plastic parts, laminates, etc. in several markets (packaging, automotive, electrical, etc.). Disadvantages of PE: susceptible to stress cracking, lower stiffness than polypropylene, high mold shrinkage and poor UV- and low heat resistance.
4. Polypropylene (PP) is a tough and rigid, crystalline thermoplastic produced from propene (or propylene) monomer. PP is widely used in various applications due to its good chemical resistance and weldability. Some common uses of polypropylene include: packaging applications, automotive applications, medical applications and industrial applications. Disadvantages

of PP: poor resistance to UV, impact and scratches, Low upper service temperature, 90-120°C and poor paint adhesion.

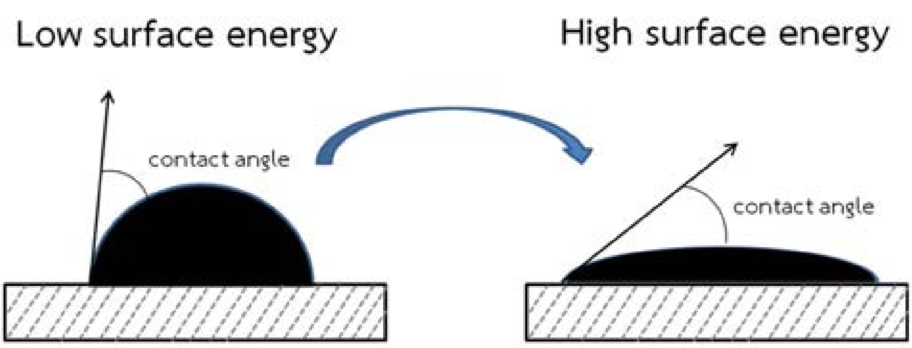
5. Polyethylene Terephthalate (PET) is one of the most recycled. It is highly flexible, colorless and semi-crystalline resin in its natural state. It shows good dimensional stability, resistance to impact, moisture, alcohols and solvents. PET is successfully being used in many applications in the automotive industry and electrical and electronic industry. It exhibits excellent electrical insulating properties. PET has broad range of use temperature, from -60 to 130°C and it is a good gas (oxygen, carbon dioxide), and moisture barrier properties. PET is suitable for transparent applications, when quenching during processing.
6. Polyvinyl Chloride (PVC) is an economical and versatile thermoplastic polymer widely used in building and construction industry to produce door and window profiles, pipes (drinking and wastewater), wire and cable insulation, medical devices etc. Limitations of Polyvinyl Chloride: poor heat stability and properties can change with time, due to plasticizer migration
7. Acrylonitrile-Butadiene-Styrene (ABS) is an impact-resistant engineering thermoplastic & amorphous polymer. ABS is made up of three monomers: acrylonitrile, butadiene and styrene. Limitations of ABS: poor weathering resistance, scratches easily and can suffer from stress cracking in the presence of some grease.

Polyethylene Terephthalate (PET) is among those plastics which are an important part of your everyday life. It is an important commercial polymer having application ranging from packaging, fabrics, films, molded parts for automotive and electronics. PET is a general-purpose thermoplastic polymer which belongs to the polyester family of polymers and highly flexible, colorless and semi-crystalline resin in its natural state. Depending upon how it is processed, it can be semi-rigid to rigid. It shows mechanical, thermal, chemical resistance as well as dimensional stability dimensional stability, resistance to impact, moisture, alcohols and solvents [18].

Coating of carbon nanotubes has adhesion study using UV-ozone treatment using dip-coating on polyethylene naphthalate (PEN) and polyethylene terephthalate (PET) substrates. The two types of polymer films have relatively high optical transmittance at 400-700 nm wavelengths which render them suitable as substrate for optical display and plastic electronic application and comparison of different kind of SWCNT performance and ITO. There found that between PEN and PET substrate, PEN gives better optical transmittance and conductivity with CNT coating than PET does which will be most likely due to the thickness difference. The report also observed better CNTs adhesion with PEN. The properties of PEN film are similar to PET but PEN film offers improved performance over PET in the areas of dimensional stability, stiffness, UV weathering resistance, low oligomer content, tensile strength, hydrolysis resistance and chemical resistance. The carbon nanotube coated filmed exhibit good mechanical flexibility that exceeds ITO coated film [19].

2.8 Surface energy

Increasing of surface energy (surface tension) is important the adhesion property on PET sheets and can be done in many way such as primer with polymer, plasma and corona treatment. If you put this round drop on a flat material which has a weak surface energy, the internal attracted forces of the drop will be preponderant and the drop will have a minimum surface contact with the material (e.g., plastic film). In this way, the corona treatment is good choice for increase the surface energy. Corona treatment is a surface modification technique that uses a low temperature corona discharge plasma to impart changes in the properties of a surface. Plastic is a man-made synthetic material, which contains long homogeneous molecular chains that form a strong and uniform product. The chains of molecules are normally joined end to end forming even longer chains, leaving only a few open chain ends, thus providing only a small amount of bonding points at the surface. The small amount of bonding points cause the low adhesion and wettability, which is a problem in converting processes. A high frequency charge would provide both a more efficient end controllable method of increasing the adhesion and wettability of a plastic surface. During corona discharge treatment, electrons are accelerated into the surface of the plastic causing the long chains to rupture, producing a multiplicity of open ends and free valences are formed. The dyne level of a material is called its surface energy. If the liquid has a dyne level lower than the material's surface energy, then the liquid will spread out over its entire surface in a uniform wet layer. Testing materials to verify treatment levels have been around for decades. For some research using a contact angle measuring device was the best most accurate method for checking materials. Figure 9 represents a surface that supports adhesion and the droplet of liquid is wetting out to the surface is an untreated surface and the droplet is clinging to itself rather than the solid surface. Surface energy is normally measured in energy units called dynes/cm. Surface energy test were chose surface energy levels by Corona-check dyne test pens. There are quick and convenient to use to determine treatment level (dyne/cm), this unique pen is easy to read [20].



Large contact angle, bad adhesion | Small contact angle, good adhesion

Poor wettability and printability | Good wettability and printability

Figure 9. A liquid drop showing the quantities before and after corona treatment

The effectiveness of the Corona treatment depends on the specific material being used. Different materials have different characteristics and different amounts of slip and additives, which will determine the effect of the Corona treatment. There are no limits with regard to the materials that can be corona treated. However, the required intensity of the treatment (watt/min/m^2) may vary significantly. The treatment level can be calculated by using the following formula:

Power (watt) = T x S x W x M

P = Total Power (Watt) required

T = Number of sides to Treat (single/double sided)

S = Line Speed (in meters per minute)

W = Film Width (in meters)

M = Material factor (required Watt per m^2 per minute)

The exact value is best determined by testing a sample of the actual film that is used for a specific application. Surface energy of base material and surface energy for adhesion on materials are shown in Table 4 and 5 respectively.

Table 4. Surface energy of base material

Substrates	Surface energy (dyne/cm)
Polyethylene (PE)	32
polypropylene (PP)	30
Polycarbonate (PC)	34
Polyethylene Terephthalate (PET)	38
Polyvinyl Chloride (PVC)	39
Acrylonitrile-Butadiene-Styrene (ABS)	34

Table 5 Surface energy for adhesion on materials (substrates)

Materials	Surface energy (dyne/cm)
UV ink	48-56
Water based	50-56
Coatings	46-52
UV glue	44-50
Water based glue	48-56

2.9 Coating process

Preparation of SWCNT-coated film by vacuum-filtering, spray-coating, spin-coating, Langmuir–Blodgett deposition, wire-bar coating and dip-coating have been reported. Comparison of coating process shown Table 6. However, the short coating time, ability to extend the coating area, and continuous processes are important means to improve the efficiency of producing SWCNT coatings. Wire-bar coating, which wraps a stainless steel wire around a shaft, is useful equipment to spread inks and coatings on flexible materials. The coating thickness can be controlled by the area in the groove between the coils of wire (wire-bar thickness). Thick wire can coat coating materials thickly and narrow wire can coat coating materials thinly. Wired K bars are produced by winding precision drawn stainless steel wire onto a stainless steel rod resulting in a pattern of identically shaped grooves. K Control Coater is automatic machine (detailed on a separate leaflet) has variable speed and a constant pressure exerted between the bar and substrate. The coating thickness applied is controlled by the area of the groove between the coils of wire. As the material levels a smooth uniform thickness is produced. Impression beds hold the substrate material firmly in place to ensure accurate draw down. Wire bar of stainless steel, these rods apply accurately coatings of paint, varnish, ink, adhesive etc. on any substrate provided that this is perfectly flat. Ideal for multi-layer coatings and parallel testing. Structure of wire bar coater shown in Figure 10. Available for wet film thickness of 4-120 microns. Transparent and conductive SWCNT thin films were prepared using wire-bar coating. This method improves the productive efficiency of the SWCNT coating process [21].

Table 6 Comparison of coating process

Method	Coating time	Extend coating	Continuous process
vacuum-filtering	Long	Impossible	Impossible
spray-coating	Long	Possible	Possible
spin-coating	Short	Impossible	Impossible
Langmuir–Blodgett deposition	Long	Impossible	Impossible
dip-coating	Short	Possible	Impossible
wire-bar coating	Short	Possible	Possible

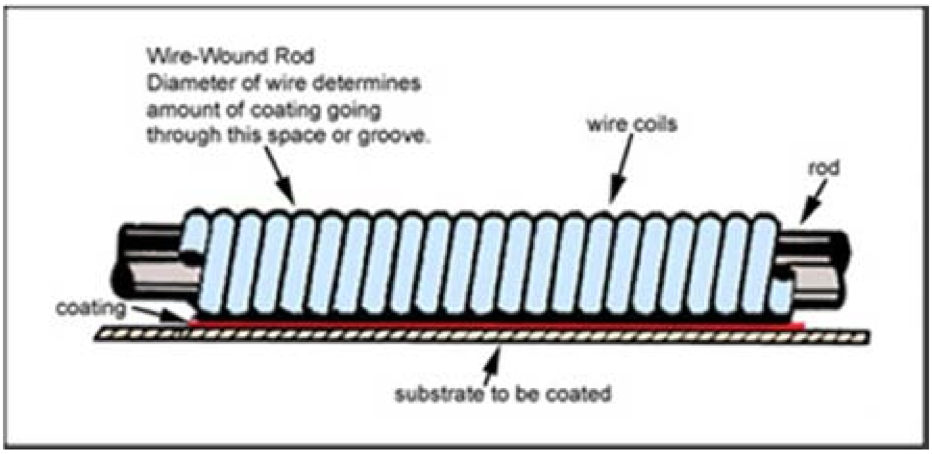


Figure 10 Structure of wire-bar coater

2.10 Evaluation

2.10.1 Adhesion test (tape test)

Preparation of carbon nanotube thin films for transparent conductive film is composed of three steps: finally step, the films of SWCNT tested strongly adhesion to the PET surface using a laboratory Scotch-tape test. Tape tests have been criticized when used for substrates other than metal, such as plastics [22]. Test Method A—An X-cut is made in the film to the substrate, pressure-sensitive tape is applied over the cut and then removed, and adhesion is assessed qualitatively on the 0 to 5 scale. Rate the adhesion in accordance with the following scale illustrated in Table 7 and Figure 11:

Table 7. Rate of the adhesion by tape test method

Rate the adhesion	Description
5	The edges of the cuts are completely smooth; none of the squares of the lattice is detached.
4	Small flakes of the coating are detached at intersections; less than 5 %of the area is affected.
3	Small flakes of the coating are detached along edges and at intersections of cuts. The area affected is 5 to 15 % of the lattice.
2	The coating has flaked along the edges and on parts of the squares. The area affected is 15 to 35 % of the lattice.
1	The coating has flaked along the edges of cuts in large ribbons and whole squares have detached. The area affected is 35 to 65 % of the lattice.

0	Flaking and detachment worse than Grade 1.

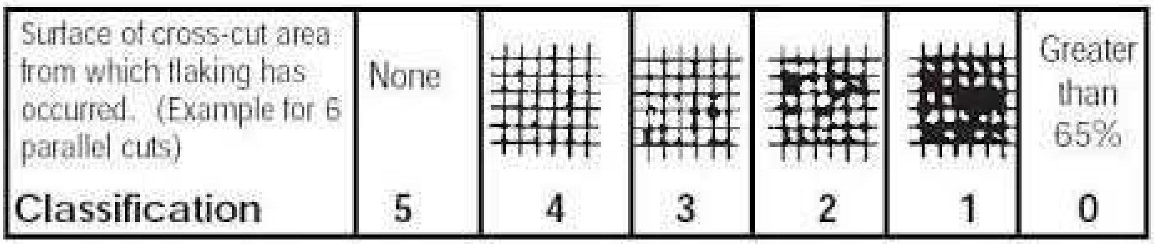


Figure 11. Classification of the adhesion by tape test

The properties of carbon nanotubes vary with the individual SWCNT chirality and SWCNT conductive transparent film. Since at this time all SWCNTs are produced as a mixture of chiralities, the properties of the material will depend on the proportions of chiralities present and coated SWCNT dispersion on PET film to conductive transparent film. Many analytical techniques have been deployed to determine the structure of SWCNT materials. These range from observational techniques such as SEM, AFM, sheet resistance, transmittance and Raman spectrum.

2.10.2 Raman spectrophotometer

Analysis by Raman spectroscopy has been widely used for determining both the detailed combination of chiralities present in the SWCNTs material and for assessing purity. There are three areas of the Raman spectrum of primary interest for SWCNTs. Raman spectra in Figure 12 show the radial breath mode (RBM) from approximately 120 to 300 cm^{-1} is unique to SWCNTs and can be used to determine tube diameter from the equation. There are two additional bands seen in the Raman spectrum of SWCNTs: the D band at ~ 1350 cm^{-1} is indicative of disordered carbon, multiwall tubes and microcrystalline graphite, and the G band at 1500 to 1600 cm^{-1} is a result of the tangential stretching mode from graphitic-like materials. The ratio of the height of the G band to that of the D band has been widely used as a measure of the purity of SWCNTs. It is probably best to say that a high G/D ratio is a necessary condition for high purity SWCNTs [23].

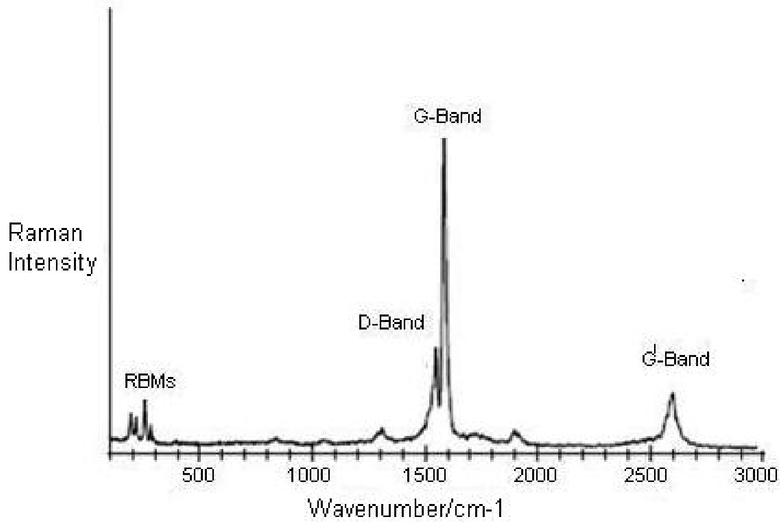


Figure 12. Raman spectrum of single-walled carbon nanotubes (SWCNTs)

2.10.3 Scanning electron microscopic (SEM)

The scanning electron microscope (SEM), which uses the electrons that are reflected or knocked off the near-surface region of a sample to create an image. SEM can be measured thickness of the film thickness and thickness surface morphology shown in Figure 13. SEM help researchers optimize their material characterization processes and save valuable time. The SEM images show a dense and homogeneous network of SWCNT. Figure 14 show SEM image of single-walled carbon nanotube.

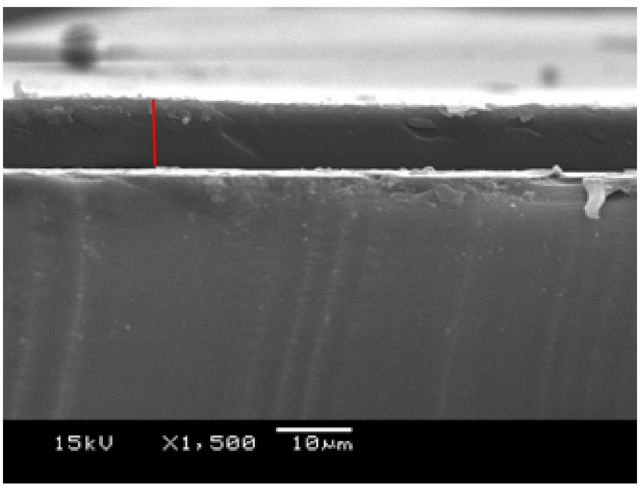


Figure 13. Cross section-SEM image of thickness of SWCNT film

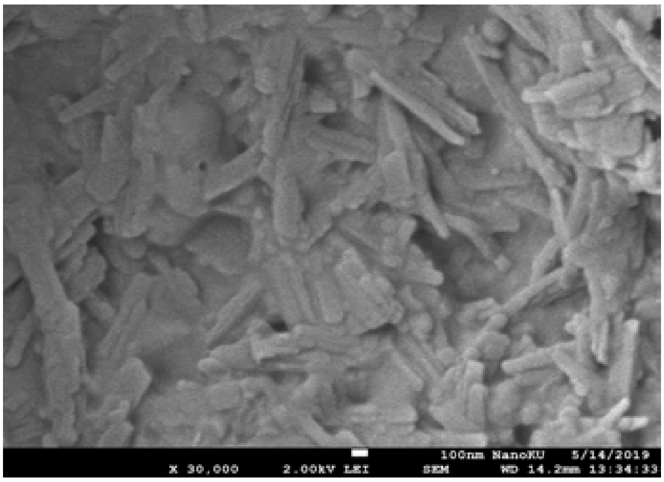


Figure 14. SEM image of single-walled carbon nanotube.

2.10.4 % Transmittance

Transmittance is the ratio of the light passing through to the light incident on the specimens and the reflectance the ratio of the light reflected to the light incident. The amount of monochromatic light absorbed by a sample is determined by comparing the intensities of the incident light (Io) and transmitted light (I). Figure 15 show transmittance processing, the ratio of the intensity of the transmitted light (I) to the intensity if the incident light (Io) is called transmittance (T) [24].

The intensity of the transmitted light (I) is never greater than the intensity of the incident light (Io), transmittance (T) is always less than 1, calculated from T = I / Io

In practice, one usually multiplies T by 100 to obtain the percent transmittance (%T), which ranges from 0 to 100%. %T = T * 100

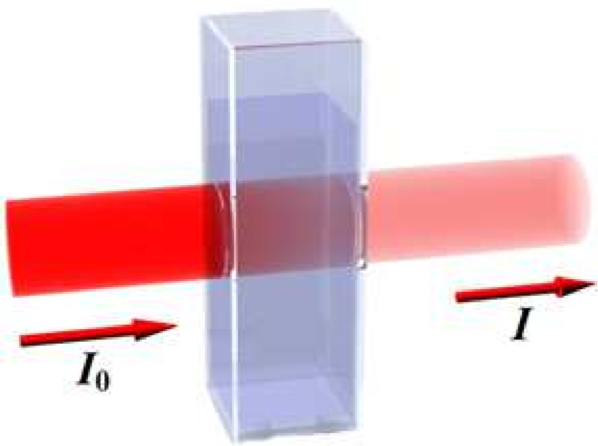


Figure 15 Schematic of transmittance

Optical absorption in transmittance measurement show characteristic of individual type of single-walled carbon nanotube. For example, the (6,5) SWCNT absorb at 566 and 976 nm. The (7,6) SWCNT absorb at 645 and 1024 nm [10].

2.10.5 Sheet resistance

Electrical resistivity is a basic material property that quantifies a material's opposition to current flow; it is the reciprocal of conductivity. The resistivity of a material depends upon several factors, including the material doping, processing, and environmental factors such as temperature and humidity. The resistivity of the material can affect the characteristics of a device of which it's made, such as the series resistance, threshold voltage, capacitance, and other parameters. The four-point collinear probe technique involves bringing four equally spaced probes in contact with a material of unknown resistance. Figure 16 show four-point probe resistivity test circuit, sheet resistance is a measure of resistance of thin films that are nominally uniform in thickness. It is commonly used to characterize materials made by semiconductor doping, metal deposition, resistive paste printing, and glass coating, and the resistors that are screen printed onto the substrates of thick-film hybrid microcircuits. Its measure of difficulty to pass an electric current through that conductor. Sheet resistance measurements are very common to characterize the uniformity of conductive or semi-conductive coatings and materials.

The sheet resistance measure of difficulty to pass an electric current through that conductor. One of the most common ways of measuring the resistivity of some thin, flat materials, such as semiconductors or conductive coatings, uses a four-point collinear probe. The four-point probe technique involves bringing four equally spaced probes in contact with a material of unknown resistance [25].

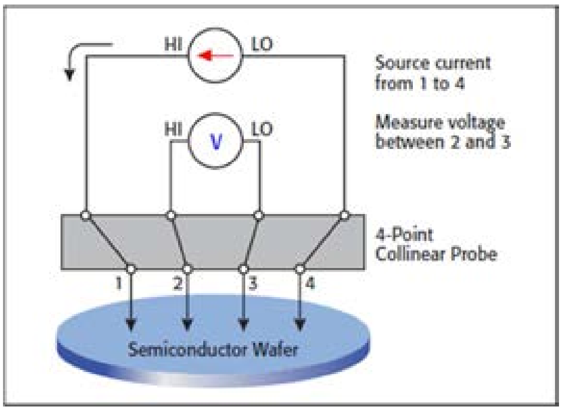


Figure 16 Four-point probe resistivity test circuit.

For thin films and coatings, the sheet resistance, or surface resistivity, is determined instead, which does not take the thickness into account. The sheet resistance (**σ**) is calculated as follows:

$$\sigma = \frac{\pi}{ln2}\frac{v}{I}k = 4.532\frac{v}{I}k$$

where:

σ = the sheet resistance (Ω/square)

V = the voltage measured between probes 2 and 3 (voltage)

I = the magnitude of the source current (amps)

k = a correction factor based on the ratio of the probe to wafer diameter and on the ratio of wafer thickness to probe separation.

CHAPTER 3
Experimental

3.1 Materials

3.1.1 Single-walled carbon nanotubes (SWCNTs)

The (6,5) chirality, ≥95% carbon basis (≥95% as carbon nanotubes), 0.78 nm average diameter with median length of 1 µm of powder (freeze-dried) form. SWCNTs were prepared by CoMoCAT catalytic chemical vapor deposition (CVD) method and purchased from Sigma-Aldrich Co., Ltd.

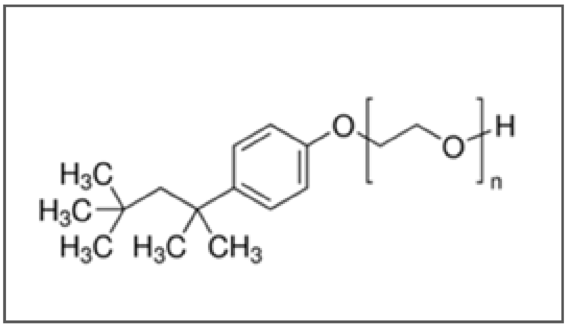
3.1.2 Non-ionic surfactants

3.1.2.1 Triton X-100 is hydrocarbon surfactant (HLB value 13.5). It was purchased from Sigma-Aldrich Co., Ltd

3.1.2.2 Dow corning 193C fluid is silicone surfactant (HLB value 12). It was purchased from Dow corning Co., Ltd.

The Structure of Triton x-100 and Dow corning 193C fluid as shown in Figure 17.

a.



b.

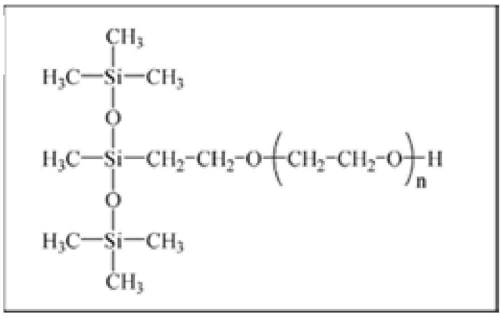


Figure 17. Structure of surfactants (a) Triton x-100 and (b) Dow corning 193C fluid

3.1.3 Neocryl A-1127 is anionic acrylic resin was purchased from DSM coating resin.(Viscosity 25-175 cps. At 25°C, pH = 7.3-7.9, Solid content 43-45% w/w)

3.1.4 Poly (ethylene terephthalate) (PET) sheets were purchased Virtiflex Co., Ltd.(91% transparency, 125 μm thickness)

3.1.5 Deionized water

3.2. Equipments

3.2.1 The 2450 is Keithley's sourcemeter source measure unit (SMU) instrument, the sheet resistance was measured using a four-point probe.

3.2.2 Transmittance was measured using a spectrophotometer (HunterLab). Ultraviolet- spectrophotometers can also be used to measure light transmittance over a wavelength range of 400 to 700 nm when samples are films.

3.2.3 Scanning electron microscopic (SEM) (JEOL, JSM-6610LV).

3.2.4 Field-emission scanning electron microscope (FE-SEM)

(Accelerating voltage: 2.0 kV; JEOL, JSM-7600F).

3.2.5 Raman spectrophotometer (Horiba Xplora plus one from Horiba Instrument Co., Ltd.) Raman spectra of SWCNTs measure using excitation wavelengths of 785 nm.

3.2.6 Wire K bar coater is automatic machine.Wire K bar coater were used NO. 3, 5, 7 and 8: wet film thickness 24, 50, 80 and 100 micron respectively.

3.2.7 Brookfield viscometer

3.2.8 Transparent tape No.600 (3M)

3.3. Preparation of PET films

The PET sheets were increased surface energy by corona treatment and checked surface tension by corona-check dyne test pen at 50 dyne. After that the PET films was soaked in methanol in 30 min to remove the dispersant reagent, dried in an oven at 80°C for 5 min. Preparation of PET films as shown in Figure 18.

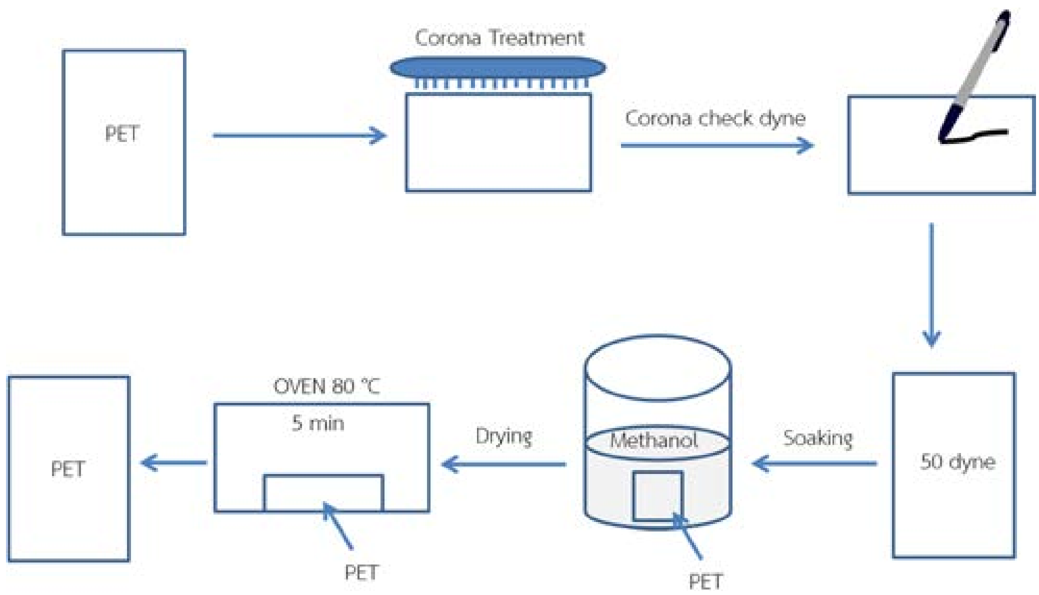


Figure 18 Scheme of the preparation of PET films

3.4 Preparation of Acrylic solution

Preparation of acrylic solution were mixed Neocryl A1127 and deionized water (1:1 w/w) in a beaker. Acrylic solution was mixed magnetic stirrer at 500 rpm for 30 min. Then measure the viscosity by Brookfield viscometer at 25°C.

3.5 Preparation of SWCNT dispersion

SWCNT dispersion were mixed acrylic solution with Triton X-100 (Dow corning 193C fluid) and concentration of surfactant varied 0.1, 0.2, 0.5, 1.0 and 2.0 %w/w were placed in 10 g acrylic solution into each vials bottle was mixed magnetic stirrer at 500 rpm for 30 min. After that prepared 0.01 g. of SWCNT were placed in to each vials bottles. The suspension were mixed magnetic stirrer at 1500 rpm for 1 hour, and then sonicated in a baht sonicator for 30 min. Formulation of SWCNT dispersion with Triton X-100 and Dow corning 193C fluid as shown in Table 8 and 9 respectively.

Table 8. Formulation of SWCNT dispersion with Triton X-100

Concentration of Triton x-100 (% w/w)	Acrylic solution (g)	SWCNT (g)	Triton X-100 (g)	DI water (g)
0.1	10.00	0.010	0.011	0.979
0.2	10.00	0.010	0.022	0.968
0.5	10.00	0.010	0.055	0.935
1.0	10.00	0.010	0.110	0.880
2.0	10.00	0.010	0.220	0.770

Table 9. Formulation of SWCNT dispersion with Dow corning 193C fluid

Concentration of Dow corning 193C fluid (% w/w)	Acrylic Solution (g)	SWCNT (g)	DC193C fluid (g)	DI water (g)
0.1	10.00	0.010	0.011	0.979
0.2	10.00	0.010	0.022	0.968
0.5	10.00	0.010	0.055	0.935
1.0	10.00	0.010	0.110	0.880
2.0	10.00	0.010	0.220	0.770

3.6 Coating method

3.6.1 Experiment 1. Varying concentration of surfactants

Preparation of SWCNT from item 3.5, the SWCNT dispersion (4 ml) was drawn in a uniform line over the fixed treated PET film from top to bottom and control all coating in the same place by using wire K bar coater No.5 corresponding to a wet film thickness 50 micron. The SWCNT films were dried at 80°C for 5 min. To study the effect of concentration of surfactants, analyze the results by choosing the best of concentration of SWCNT dispersion of each surfactant that have the lowest sheet resistance and the highest transmittance to be used to study the effect of thickness of conductive transparent film in experiment 2

3.6.2 Experiment 2. Varying thickness of SWCNT dispersion

Chosen the best of concentration of each surfactant of SWCNT dispersion from experiment 1 for experiment 2. Then vary the thickness of the SWCNT films by using the wire-bar coating wet film thickness at 24, 50, 80 and 100 μm size by using wire k bar coater No.3, 5, 7 and 8 respectively, were chosen for single coating, and 100+50, 100+100 and 100+100+50 μm sizes for multiple coating technique from wire K bar coater No. 5 and 8. To study the effect of thickness on electrical conductivity of SWCNT films for touch screen. Preparation of SWCNT dispersion, coating method and characterization of SWCNT films as shown in Figure 19.

3.7 SWCNT films characterization

3.7.1 Measure the viscosity of the acrylic solution with brookfield viscometer for the coating ability with the wire-bar coating technique at room temperature.

3.7.2 The films of SWCNT tested strongly adhesion to the PET surface using tape test by transparent tape No.600.

3.7.3 Sheet resistance was measured using a 4-point probe.

Cut four point of the SWCNT film into a small square with dimension of 3x3 cm per sheet, five sheets of SWCNT film, then measured the sheet resistance on the small SWCNT film and calculate the average of sheet resistance of SWCNT films.

3.7.4 Transmittance was measured using a spectrophotometer.

% Transmittance measure of the amount of light that passes through a transparent material. Ultraviolet- spectrophotometers can also be used to measure light transmittance over a wavelength range of 400 to 700 nm when samples are films. Single-walled carbon nanotube (6,5) chirality absorb at 560 nm.

3.7.5 Scanning electron microscopic (SEM) observations were carried out using field-emission scanning electron microscope (FE-SEM). SEM images were used to analyze the surface morphology of SWCNT films and dry film thickness.

3.7.6 Raman spectroscopy, this is an important tool to effectively measure the characteristics of single-walled carbon nanotubes and conductive property.

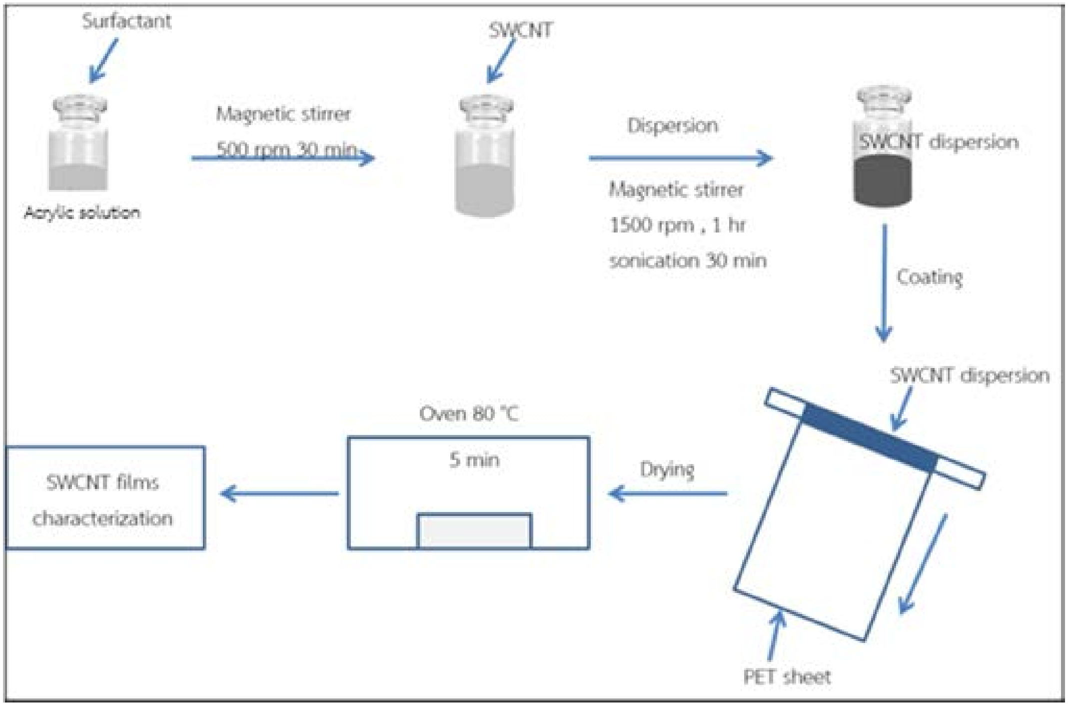


Figure 19. Preparation of SWCNT dispersion, coating method and characterization of SWCNT films.

CHAPTER 4
Result and Discussions

4.1 Experiment 1

Preparation acrylic solution, which were mixed acrylic resin and deionized water (1:1 w/w) and measured viscosity using brookfield viscometer at room temperature, found that when increasing the share rate, the viscosity decreases. Acrylic solution can using wire bar coating technique shown in Figure 20.

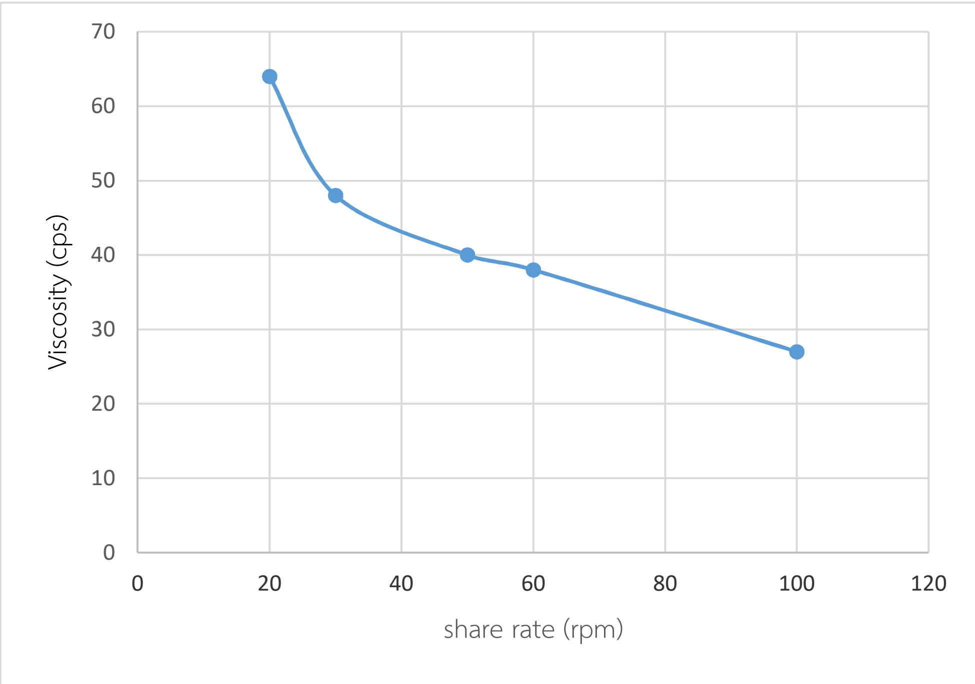


Figure 20. The relationship between viscosity and shear rate

Increasing the surface energy of PET film by corona treatment. The surface energy test were chose surface energy levels by corona-check dyne test pen at 50 dyne/cm because surface energy for adhesion on substrates at 50-56 for water base coating The corona treated film and the surface energy values 50 dyne/cm, by corona-check dyne test pen is permanent. The area where the surface energy is lower than 50 dyne/cm found the ink of the corona-check dyne test pen cannot cover on the film (poor wettability) shown in Figure 21.

Figure 21. Comparison between treated and untreated corona treatment on PET film by corona-check dyne test pen at 50 dyne/cm

Preparation of acrylic solution and surfactants using 0.1, 0.2, 0.5, 1.0 and 2.0 % w/w of Triton X-100 and Dow corning 193C fluid were used respectively without SWCNTs powder, then coated on PET film at 50 micron by wire-bar coater. To study the effect of surfactants on electrical conductivity. The sheet resistance of PET film is 1.782×10^8 Ω/square and acrylic solution mixed surfactants without SWCNTs in Table 10.

Table 10 Sheet resistance of SWCNT films, coating thickness 50 micron

Concentration of surfactant (% w/w)	Sheet resistance (Ω/square)	
	Triton X-100	Dow corning 193C fluid
0.1	9.891×10^6	9.590×10^6
0.2	9.725×10^6	9.470×10^6
0.5	9.426×10^6	9.390×10^6
1.0	9.205×10^6	9.115×10^6
2.0	9.033×10^6	8.969×10^6

Preparation of SWCNT dispersion without SWCNT and varied the concentration of both surfactants coated on PET sheet, the reduction of the sheet resistance compared with uncoated PET film, but when increasing the concentration of Triton X-100 and Dow corning, There are found that the electrical resistance was slightly reduced, so the acrylate solution and the both surfactants can reduced the sheet resistance.

Preparation of SWCNT dispersion using 0.1, 0.2, 0.5, 1.0 and 2.0 % w/w of Triton X-100 and Dow corning 193C fluid were used respectively. After that coated of SWCNT dispersion on PET films was carried out using wire bar wet film thickness 50 micron. Images of the SWCNT dispersions in Figure 22 and 23. Found that, the distribution of SWCNTs in acrylic solution are clearly at 0.5, 1.0 and 2.0 %w/w concentration of both surfactants.

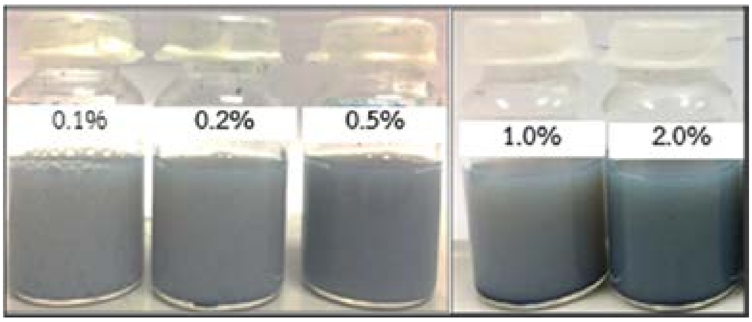


Figure 22. The SWCNT dispersions with Triton X-100 at 0.1, 0.2, 0.5, 1.0 and 2.0 % w/w

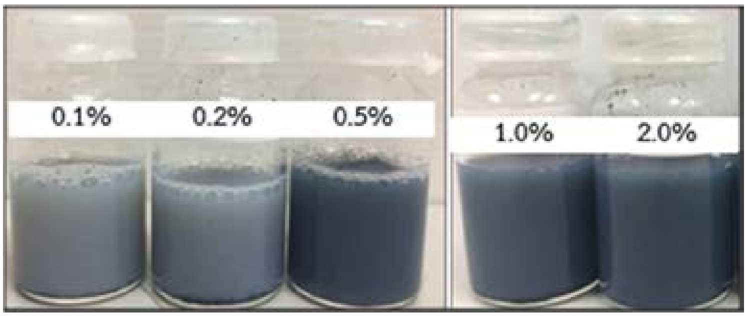


Figure 23. The SWCNT dispersions with Dow corning 193C fluid at 0.1, 0.2, 0.5, 1.0 and 2.0 % w/w

The films of SWCNT tested strongly adhesion to the PET surface using tape test by transparent tape No.600 according to standard found the SWCNT films are excellent adhesion on treated PET film, qualitatively on the 5 scale of rate of the adhesion by tape test. Therefore, the surface energy value of 50 dyne/cm is suitable for PET film and SWCNT dispersion from acrylic resin.

The sheet resistance of SWCNT films on PET films was measured with four-point probe at room temperature and transmittance was measured with spectrophotometer wavelength at 560 nm. The optical absorption spectra of the characteristic band of (6,5) SWCNTs at 566 nm. The sheet resistance and transmittance of SWCNT films are shown in Table 11. When creating a relationship between sheet resistance and varied concentration of surfactants of SWCNT films are shown in Figure 24.

To consider sheet resistance and transmittance of coated films, that there was relationship between the increase of surfactant concentration and sheet resistant value. The concentration 0.1% to 2% of surfactant, the sheet resistant was decreased 63% and 57% for Triton X-100 and Dow corning 193C fluid surfactants respectively. Conversely, the transmittance of coated films showed similar manner, but the values slightly increased.

Table 11 Sheet resistance (R) and transmittance (T) of SWCNT films, coating thickness 50 micron

Concentration of surfactant (% w/w)	Triton X-100		Dow corning 193C fluid	
	R (Ω/sq)	T (%)	R (Ω/sq)	T (%)
0.1	1.82×10^5	86.5	1.11×10^5	88.2
0.2	1.25×10^5	87.3	9.14×10^4	88.3
0.5	9.74×10^4	87.9	8.57×10^4	88.8
1.0	8.12×10^4	88.0	7.87×10^4	89.6
2.0	6.69×10^4	88.2	4.70×10^4	89.8

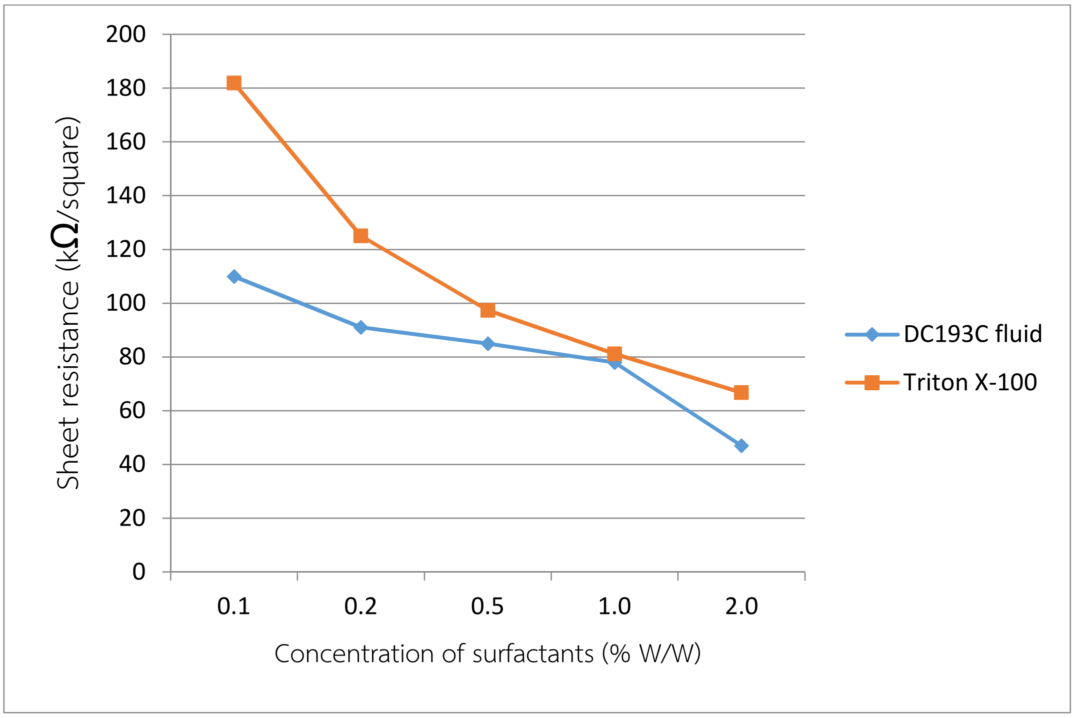


Figure 24. Sheet resistance of SWCNT dispersion with varied concentration of surfactants, coating thickness 50 micron.

In addition, increased the surfactant to 2.5, 3.0, 3.5, 4.0, 4.5 and 5.0 % w/w of Triton X-100 and Dow corning 193C fluid were used respectively, and coated on the

PET films was carried out using wire bar thickness 50 micron and dried at 80°C for 5 min. that found that SWCNT dispersion of 3.5 to 5.0 % w/w of both surfactants, the SWCNT dispersion have low surface tension, characteristic like as oil when increasing the concentration of surfactants, The SWCNT film have sticky surface after dried, Therefore cannot use the concentration higher than 3 % w/w of both surfactants. Images of the SWCNT dispersions in Figure 25 and 26.

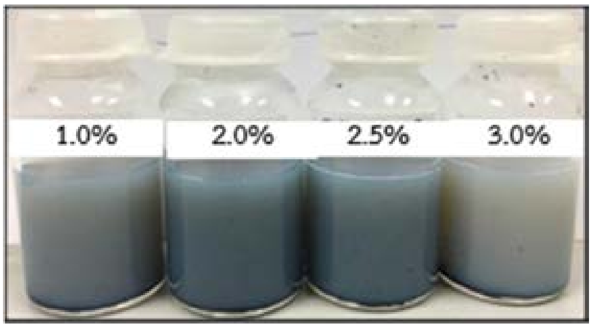


Figure 25. The SWCNT dispersions with Triton X-100 at 1.0, 2.0, 2.5 and 3.0 % w/w

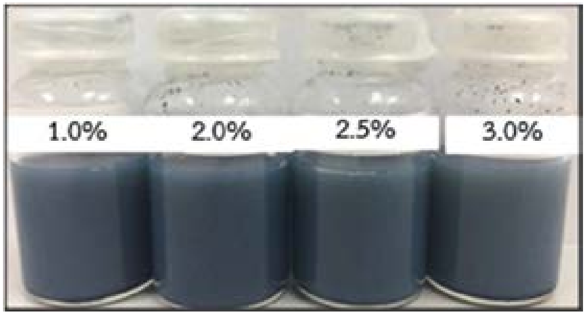


Figure 26 The SWCNT dispersions with Dow corning 193C fluid at 1.0, 2.0, 2.5 and 3.0 % w/w

The sheet resistance and transmittance of SWCNT films are shown in Table 12 and creating a relationship between sheet resistance and varied concentration of surfactants of SWCNT films are shown in Figure 27. the varying surfactant concentration did slightly affect obviously, the change of transmittance and sheet resistance of conductive films. For the experiment 2, chose the proper surfactant concentration at 2.0 % w/w.

Table 12 Sheet resistance (R) and transmittance (T) of SWCNT films, coating thickness 50 micron

Concentration of surfactant (% w/w)	Triton X-100		Dow corning 193C fluid	
	R (Ω/sq)	T (%)	R (Ω/sq)	T (%)
0.1	1.82×10^5	86.5	1.11×10^5	88.2
0.2	1.25×10^5	87.3	9.14×10^4	88.3
0.5	9.74×10^4	87.9	8.57×10^4	88.8
1.0	8.12×10^4	88.0	7.87×10^4	89.6
2.0	6.69×10^4	88.2	4.70×10^4	89.8
2.5	6.25×10^4	88.75	4.56×10^4	89.75
3.0	6.24×10^4	88.92	4.96×10^4	89.92

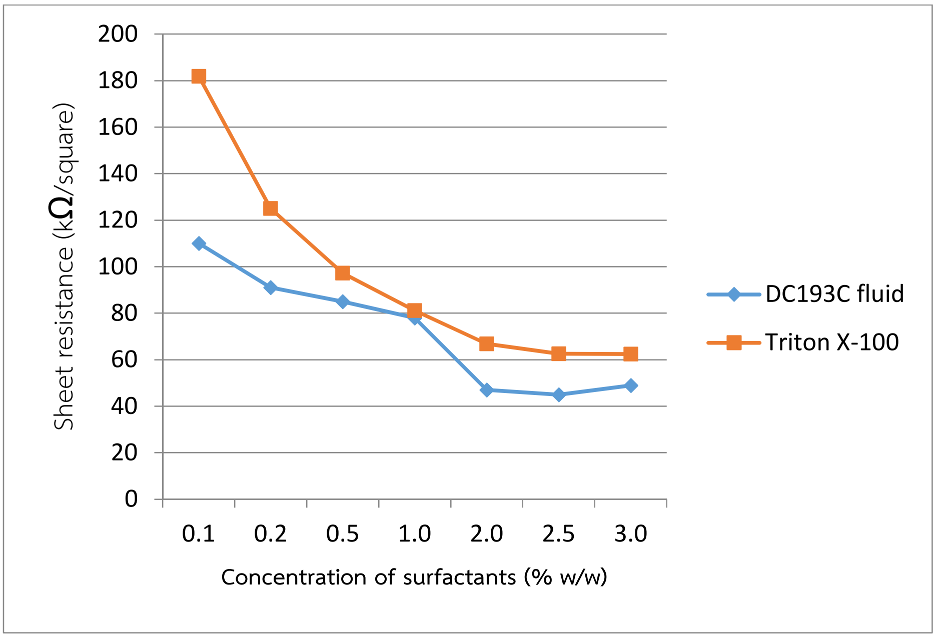


Figure 27. Sheet resistance of SWCNT dispersion with varied concentration of surfactants, coating thickness 50 micron.

The FE-SEM images of SWCNT coated on PET films by varying the concentration of Triton X-100 and Dow corning 193C fluid respectively shown in Figure 26 and 27. The FE-SEM images show a dense and homogeneous network of SWCNTs. Which is clearly at 1.0 and 2.0% concentration of both surfactant. From FE-SEM images can see explicitly the distributed SWCNT at surfactant concentration 1.0 and 2.0 % w/w. Which corresponds to Figure 28 and 29. The distribution of SWCNT was considered. It was found that higher surfactant concentration gave better SWCNT distribution, particularly at the surface of coated film. This implied that there was less flocculation of SWCNT in the coated films. SWCNT dispersion with Dow corning 193C fluid resulted in better result of SWCNT distribution than SWCNT dispersion with Triton X-100.

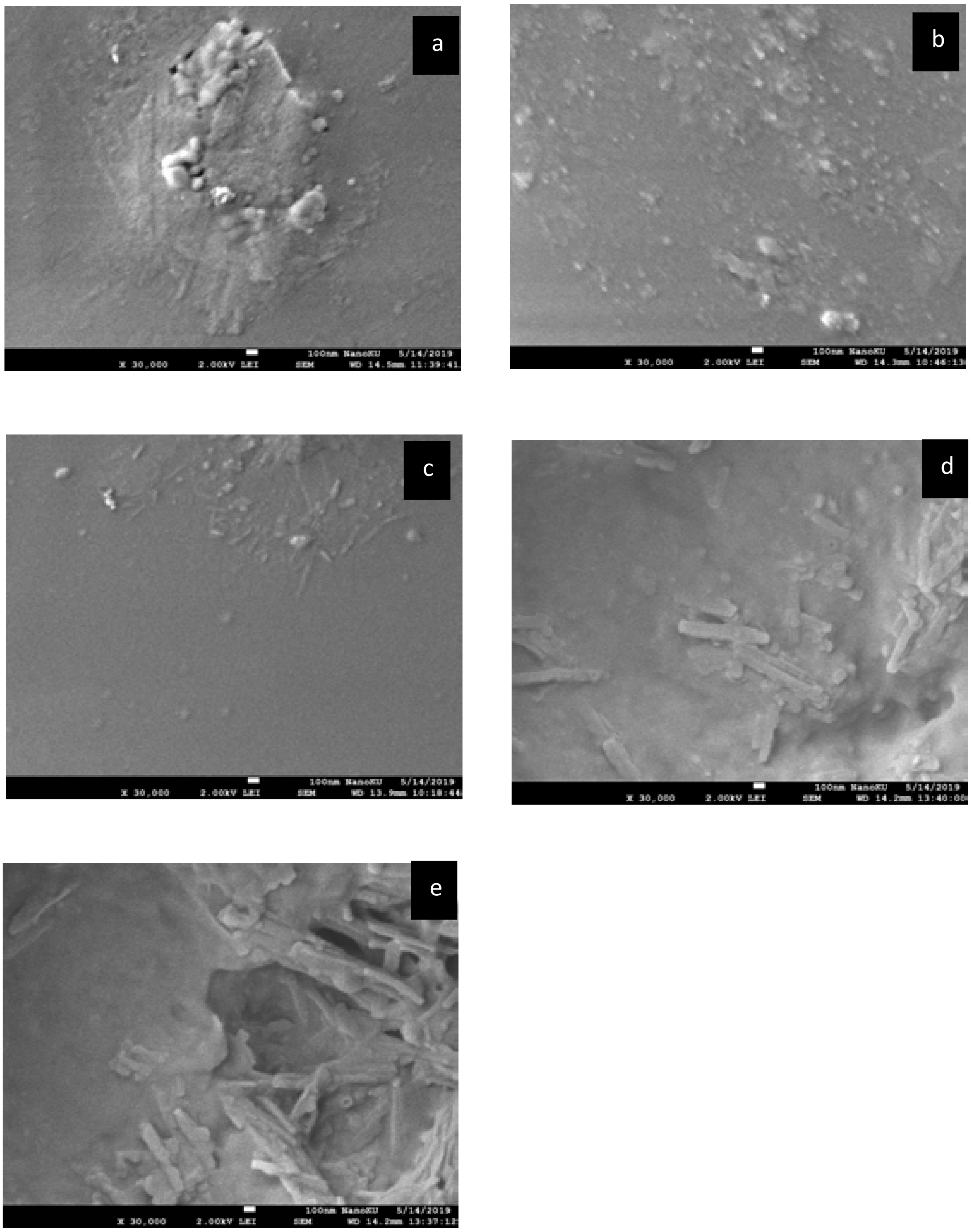

a
b
c
d
e

Figure 28 FE-SEM images of SWCNTs coated onto PET film: (a) – (e): dispersion of SWCNTs with Triton X-100; concentration are (a) 0.1% w/w (b) 0.2% w/w (c) 0.5% w/w (d) 1.0% w/w (e) 2.0% w/w, coating thickness 50 micron

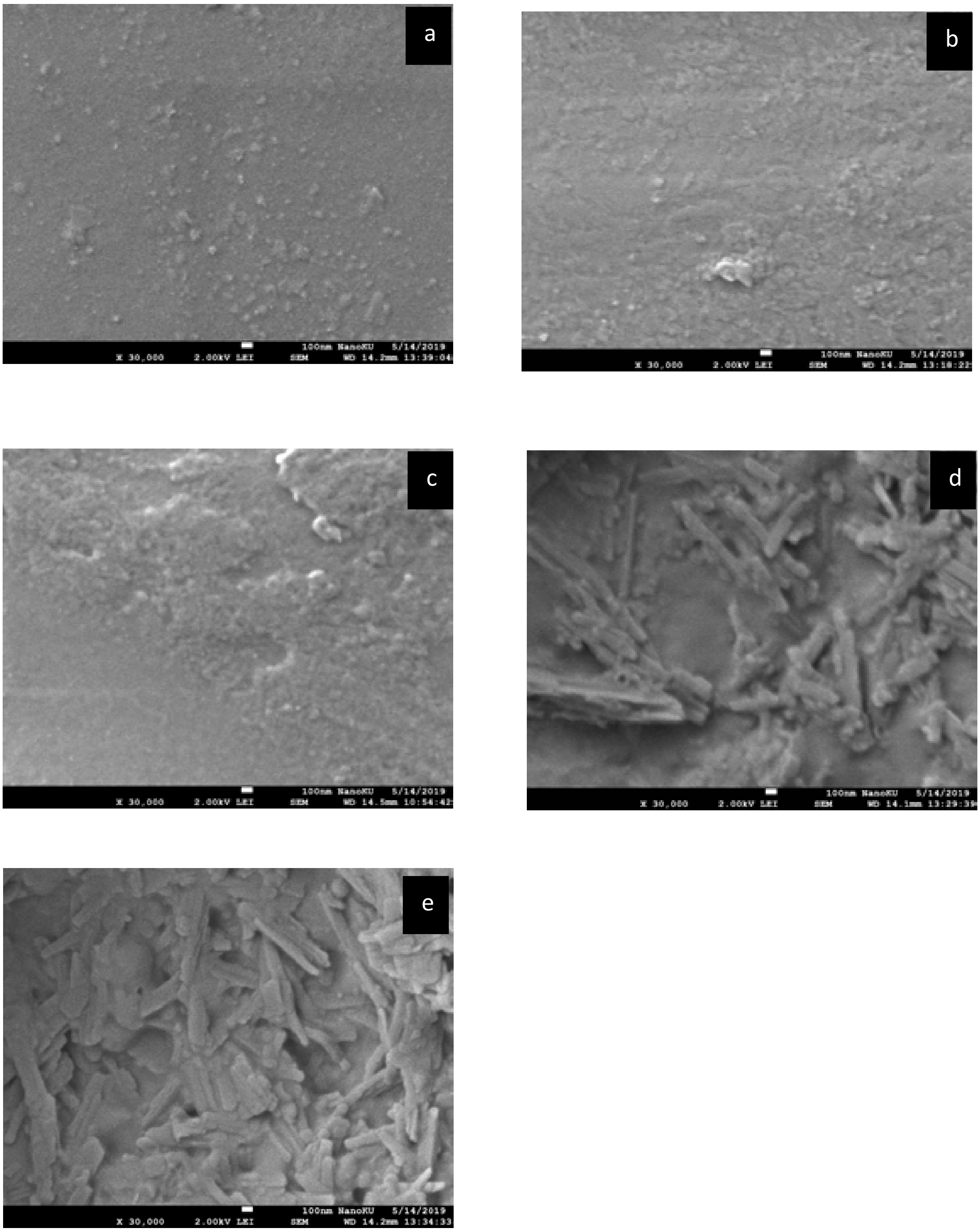
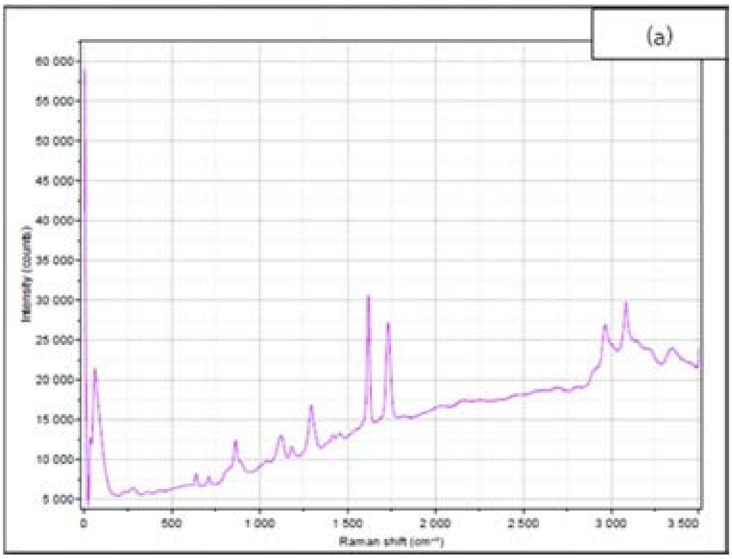


Figure 29. FE-SEM images of SWCNTs coated onto PET film: (a) – (e): dispersion of SWCNTs with Dow corning 193C fluid; concentration are (a) 0.1% w/w (b) 0.2% w/w (c) 0.5% w/w (d) 1.0% w/w (e) 2.0% w/w, coating thickness 50 micron

The FE-SEM images can see explicitly the distributed SWCNT at surfactant concentration 1.0 and 2.0 % w/w, when analyzed Raman spectra of SWCNT films with (a) 1.0% w/w and (b) 2.0% w/w of Triton X-100 in Figure 30 and (a) 1.0% w/w and (b) 2.0% w/w of Dow corning 193c fluid in Figure 31 were measured using excitation wavelengths of 785 nm. The characteristic peaks, the radial breathing mode (RBM), disordered carbon mode (D band), and the tangential Raman mode (G band) are depicted.



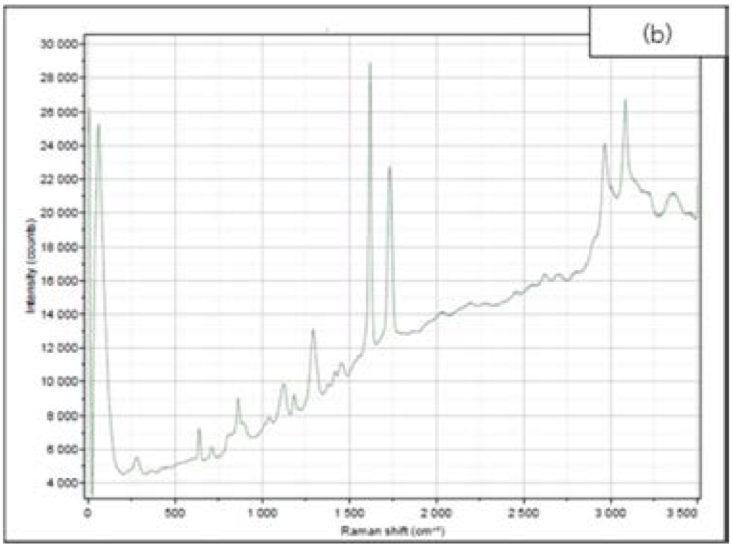
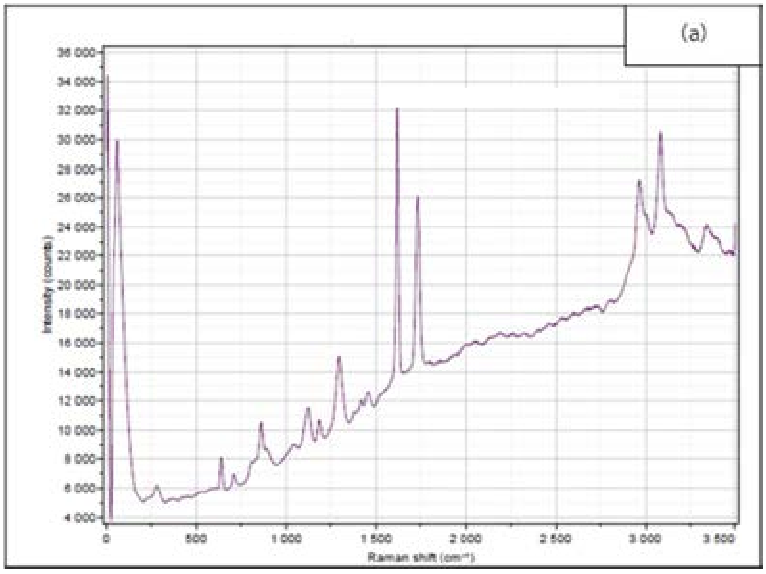


Figure 30. Raman spectra of SWCNT film (a) 1.0 % w/w and (b) 2.0 % w/w concentration of Triton X-100, coating thickness 50 micron.



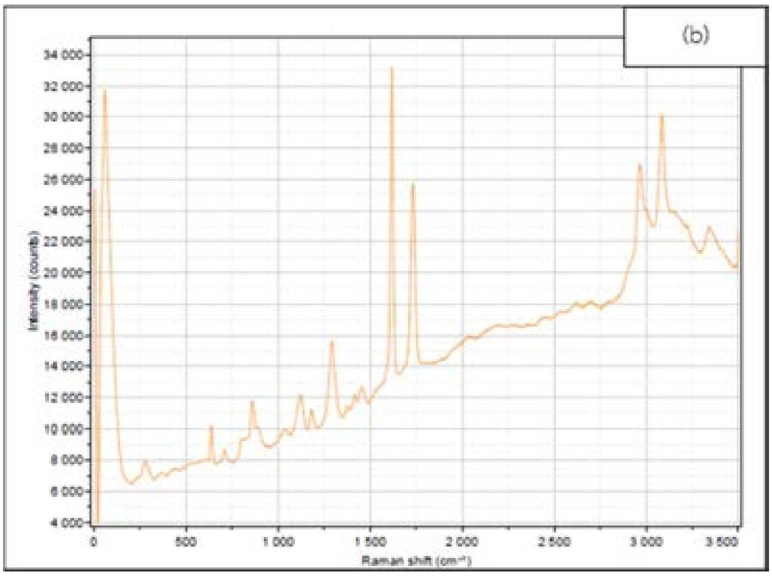


Figure 31 Raman spectra of (a) 0.1 % w/w and (b) 2.0 % w/w concentration of Dow corning 193C fluid, coating thickness 50 micron.

The Raman spectra of SWCNT films, The G band at 1500 to 1600 cm^{-1} is a result of the tangential stretching mode from graphitic-like materials as affects to electrical conductivity. When increased concentration of surfactants, the intensity of the tangential Raman mode (G band) peak increased. At 1.0% w/w of Triton X-100 and Dow corning 193C fluid, the intensity of G-band are 29 and 32 respectively, and 2.0% w/w of Triton X-100 and Dow corning 193C fluid, the intensity of G-band are 30 and 33 respectively. SWCNT dispersion with Dow corning 193C-fluid have better electrical conductivity than SWCNT dispersion with Triton x-100 as the same concentration.

4.2 Experiment 2

Choose the proper concentration at 2.0 % w/w of both surfactants, and then preparation of the SWCNT dispersion coated on PET films by varied thickness of wire-bar coater for wet film thickness at 24, 50, 80 and 100 micron by using wire k bar coater No.3, 5, 7 and 8 respectively, were chosen for single coating, and 100+50, 100+100 and 100+100+50 micron for multiple coating technique from wire K bar coater No. 5 and 8.

In addition, the SWCNT dispersion of 2.5 and 3.0% w/w of Triton X-100 and Dow corning 193C fluid were used respectively, and coated on the PET films by using multi-coating technique, that found that the SWCNT films cannot coated because increasing the concentration of surfactant, it reduce the surface energy. When using multi-coating technique found the SWCNT dispersion cannot cover (non-wettability) on the SWCNT film shows in Figure 32.

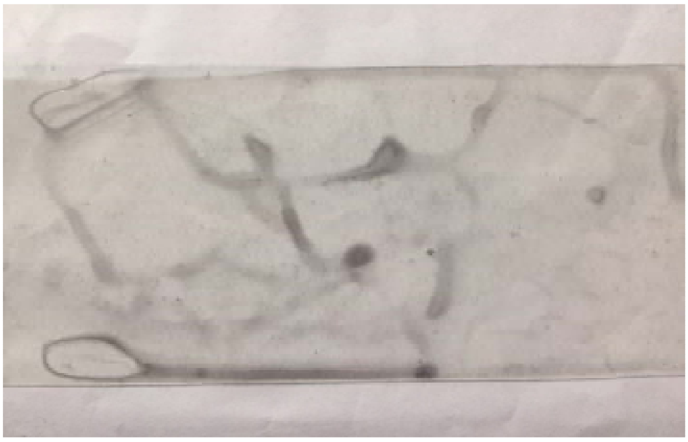


Figure 32 Non-wettability surface of high concentration of surfactant of surfactant of SWCNT dispersion using multi-coating technique

To consider the effect of coated film thickness by using single-coating and multi-coating technique to increase the SWCNT film thickness. Table 13 and Figure 33 show the averaged data of sheet resistance and transmittance of coated films with varying thickness by using different wire-bar sizes.

Table 13 Sheet resistance (R) and transmittance (T) of 2.0% w/w of surfactant of SWCNT films

Wet film thickness (micron)	Triton X-100		Dow corning 193C fluid	
	R (Ω/sq)	T (%)	R (Ω/sq)	T (%)
24	9.74 x 105	88.6	8.42 x 105	89.6
50	6.69 x 104	88.2	4.70 x 104	89.8
80	3.58 x 104	87.2	2.45 x 104	88.7
100	1.09 x 104	87.1	9.17 x 103	88.6
150	9.89 x 103	86.8	3.51 x 103	88.2
200	5.86 x 103	86.3	9.38 x 102	88.1
250	2.63 x 103	86.1	8.91 x 102	87.7

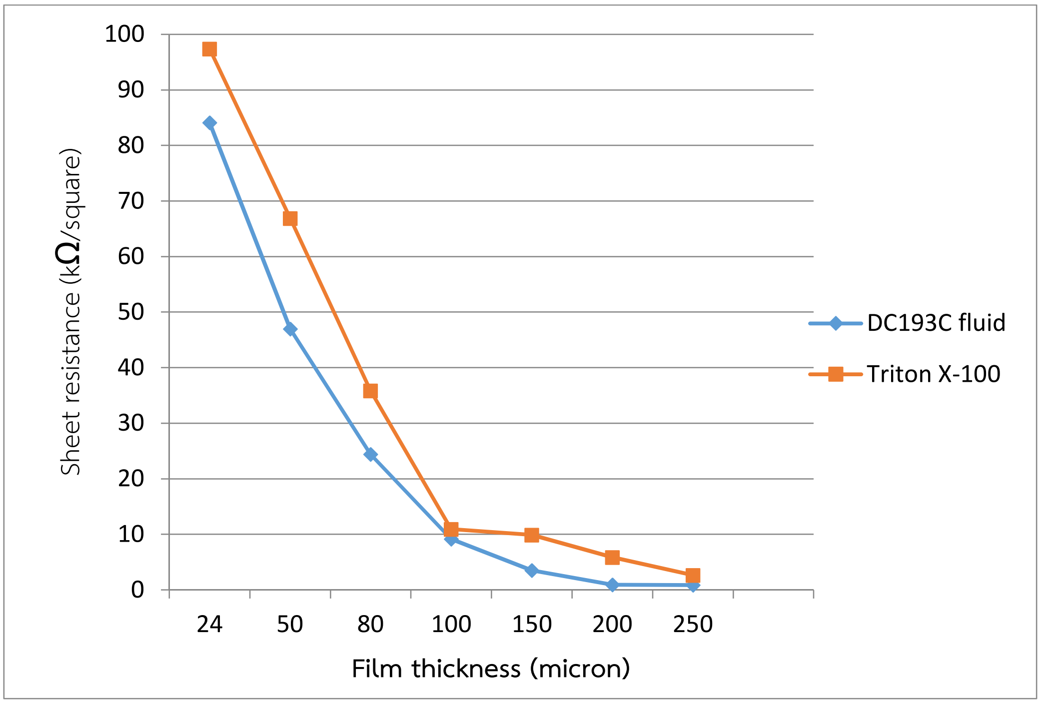


Figure 33. Sheet resistance of 2.0% w/w of surfactant of varied SWCNT film thickness

It was found that there was a trend of sheet resistance decreasing when the thickness of coated films were increased. Interestingly, the transmittance values showed similar results. Multiple coating played an important role in increasing the thickness of coated films. We could achieve the optimum sheet resistance values 891 Ω/square and 2630 Ω/square for Dow corning 193C fluid and Triton X-100 respectively by using multiple coating at 250 µm wet-film thickness. While % transmittance of coated films was slightly decreased not over 2.0%. Note that the display devices available in the market today have a target of % Transmittance values not lower 85% and sheet resistive value between 120-1500 Ω/square for resistive touch screen.

The FE-SEM images of SWCNT coated film prepared from SWCNT dispersion with Triton x-100 and Dow corning 193c fluid are show homogeneous distribution of

SWCNT dispersion of 2.0% w/w concentration of surfactant at 250 micron thickness shown in Figure 34. Which is smooth surface at 250 micron due to the high thickness, therefore resulting in the distribution of carbon nanotubes not as clear as coating thickness at 50 micron as the FE-SEM images in Figure 26 and 27.

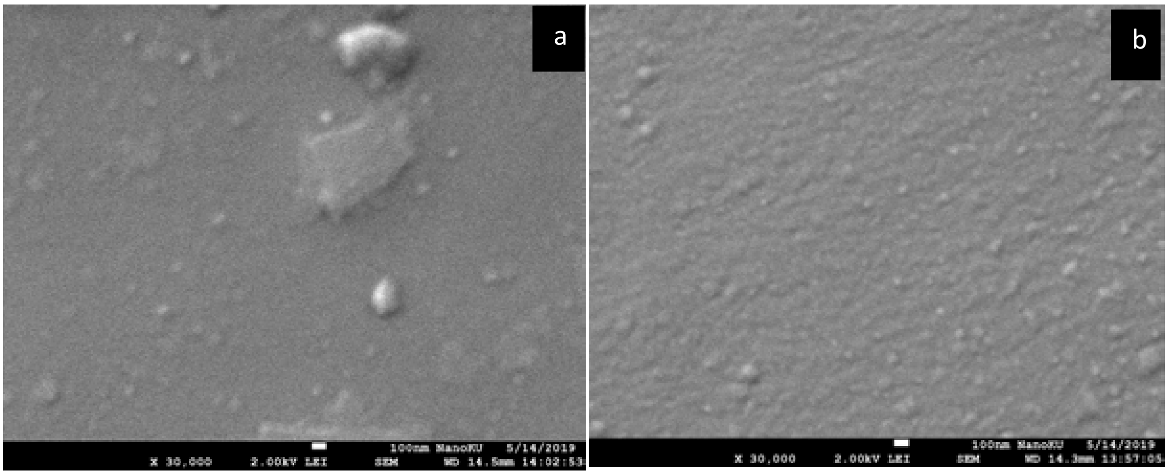


Figure 34. FE-SEM images of SWCNT dispersion with 2.0% Triton X-100 (a), 2.0% DC193C fluid (b), thickness is 250 μm

Raman spectra of SWCNT films with 2.0% w/w of triton X-100 and Dow corning 193c fluid and coated thickness at 250 micron were measured using excitation wavelengths of 785 nm. The characteristic peaks, the radial breathing mode (RBM), disordered carbon mode (D band), and the tangential Raman mode (G band) are depicted in Figure 35. The intensity of the tangential Raman mode (G band) peak of the SWCNT film of Triton X-100 and Dow corning 193C fluid are 29 and 32 respectively SWCNT dispersion with Dow corning 193C-fluid have better electrical conductivity than SWCNT dispersion with Triton x-100 as the same thickness and concentration.

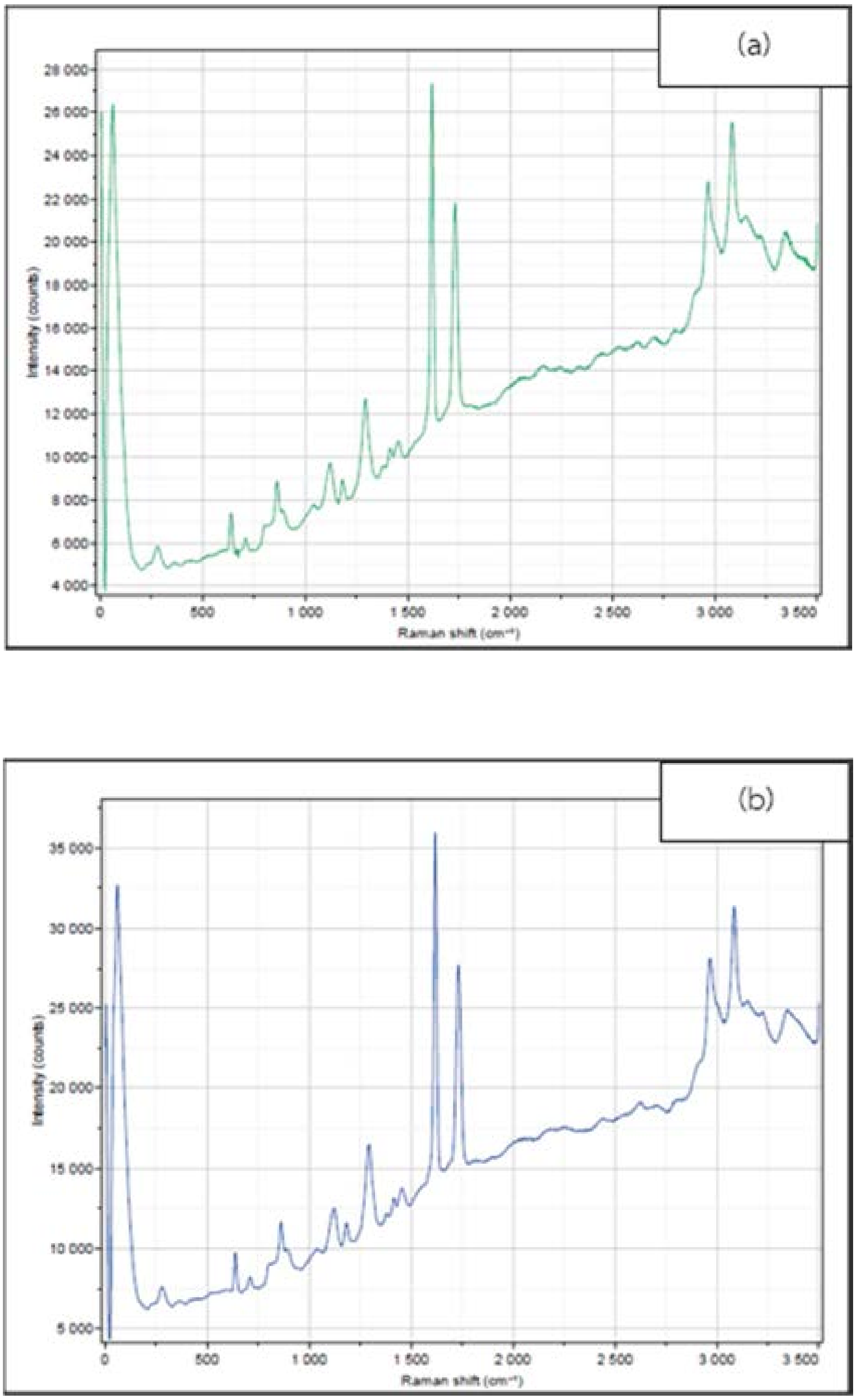


Figure 35. Raman spectra of 2.0% w/w concentration of SWCNT dispersion with (a) Triton X-100 and (b) Dow corning 193C fluid at 250 nm thickness.

In addition, the coated of SWCNT films thickness more than 250 micron using multi-coating method by wire k bar coater. The coated film thickness at 300 micron, the optimum sheet resistance was at 745 Ω/square with 84% transmittance (including PET film) and 2470 Ω/square with 83% transmittance by using surfactant of Dow corning 193c fluid and triton X-100 respectively. The transmittance are decreased lower 85 % transmittance of conductive transparent film. When increased SWCNT powder in acrylic solution from 10 g. to 20 g. and then coated on PET film at 250 micron. We found the optimum sheet resistance values 450 Ω/square and 890 Ω/square, the transmittance are decreased at 82% and 83% of Dow corning 193C fluid and Triton X-100 surfactants respectively. The SWCNT film can be measured dry film thickness with SEM shown in Table 14.

Table 14. Comparison between wet and dry film thickness of SWCNT film

Wet film thickness (micron)	Dry film thickness (micron)
24	2.5
50	6.0
80	9.5
100	11.0
150	16.0
200	22.0
250	26.0
300	32.0

CHAPTER 5

Conclusion

The increase of surfactant concentration affected the reduction of sheet resistance, while % Transmittance was increased. Surfactant concentration was varied 0.1 – 2.0 % w/w of the SWCNT dispersion coated on the PET film by wire bar coater wet thickness at 50 micron, the optimum sheet resistance was at 4.70 x 10^4 Ω/square with 89.8 % transmittance (including PET film) and 6.69 x 10^4 Ω/square with 88.2 % transmittance by using 2% w/w of Dow corning 193c fluid and triton X-100 respectively. Increasing surfactants has limitation due to the sheet resistance and % transmittance are slightly change. Increasing the concentration of the surfactant up to 3.0 % did slightly affect obviously, the sheet resistance was at 4.96 x 10^4 Ω/square with 89.9 % transmittance (including PET film) and 6.24 x 10^4 Ω/square with 88.9 % transmittance by using Dow corning 193c fluid and triton X-100 respectively. DC 193C fluid surfactant can the dispersion of SWCNT better than Triton X-100.

The increase of coated SWCNT film thickness affected the reduction of both sheet resistance and % transmittance, the optimum sheet resistance was at 890 Ω/square with 87% transmittance and 2630 Ω/square with 86% transmittance by using 2.0% w/w of Dow corning 193C fluid and Triton X-100 respectively with the film thickness of 250 microns. Sheet resistance depend on the thickness of SWCNT film. Transmittance release to the thickness of SWCNT film. The optimum thickness is 250 micron because % transmittance for conductive transparent film for resistive touch screen is typically between 85-90 % and the sheet resistance is value between 120-1500 Ω/square. The coated film thickness at 300 micron, the optimum sheet resistance was at 745 Ω/square with 84% transmittance and 2470 Ω/square with 83% transmittance by using 2% w/w of Dow corning 193c fluid and Triton X-100 respectively. The transmittance are decreased lower 85 % transmittance of conductive transparent film. On some devices or application for resistive touch screens that are medium quality for higher competition both in terms of cost and quality. The transmittance value higher 80% is a good choice.

The multi-coat technique can increase the thickness of SWCNT film and increasing surfactants has limitation due to the reduction of surface energy because this coating is a multi-layer coating overlapping when the first layer is coated with a high concentration of SWCNT dispersion, the resulting in lower surface energy values. Therefore, when coating on the next layer cannot be coated.

The SWCNT film thickness is tunable depending not only on wire bar thickness but also on the SWCNTs and surfactant concentration in dispersion. This depends on the concentration of surfactants and the thickness of SWCNT coated films. The wire-bar coater technique improve the productive efficiency of SWCNT coating process. In addition, the use of acrylic solutions to prepare carbon nanotubes can be prepared. Because carbon nanotubes can be dispersed in aqueous solutions and acrylic solutions have high transparency and good adhesion properties on PET film.

Silicone surfactant, Dow corning was employed to disperse single-walled carbon nanotube in acrylic solutions, which showed advantages in dispersing ability for preparation of SWCNT dispersion. DC 193C fluid surfactant can the dispersion of SWCNT better than Triton X-100 and SWCNT dispersion with DC 193C fluid have a better electrical conductivity than SWCNT dispersion with Triton X-100. Which the cost of Dow corning is about 50% cheaper than Triton X-100 can be used as an alternative to research. For nonionic surfactant, the silicone surfactant gave better results than hydrocarbon surfactant did, because the Si-O-Si chain of the silicone surfactant has strong interaction with carbon nanotubes. The Si-O-Si chain is flexible due to long Si-C bond and it can easily wrap onto surface of carbon nanotube via hydrophobic and other intermolecular interaction. The silicone surfactant could wrap onto the surface of SWCNTs leading to steric stabilization so that it could well disperse SWCNTs, and Van der Waals attraction was the dominating force of the silicone surfactant adsorbing onto SWCNTs.

From the experiment, it was found that conductive transparent film could be prepared for the touch screen from single-walled carbon nanotubes and nonionic surfactants in acrylic solution by using a single and multi-coating by wire bar coater increased the film thickness

For this research, using a surfactant-free surfactant, which can help the dispersion of carbon nanotubes for dispersing in acrylic solution. There are many types of surfactant such as anionic, and cationic surfactant, that can be used in the distribution of carbon nanotubes in acrylic solution and the multi-coat method has limitation for increasing the thickness of SWCNT from high concentration of surfactant, can use other coating for increasing the thickness of SWCNT film such as dip coating and spray coating.

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