

**PRODUCTION OF SEGMENTED PIE BI-COMPONENT FIBERS FOR
ANTIBACTERIAL TEXTILES**



PIYANUT JINGJIT

**A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR THE DEGREE OF DOCTOR OF ENGINEERING
PROGRAM IN ENERGY AND MATERIALS**

FACULTY OF ENGINEERING

RAJAMANGALA UNIVERSITY OF TECHNOLOGY THANYABURI

ACADEMIC YEAR 2018

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| Program | D. Eng. Energy and Materials |
| Thesis Advisor | Mr. Natee Srisawat, Ph.D. |
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ABSTRACT

The increase of public concern regarding hygienic living environments has accelerated research on anti-microbial modifications for many commodity and technical textile products. This study attempted to produce a durable self-cleaning and antibacterial textile material with a relatively low production cost using inorganic photocatalytic nanoparticles embedded in the textile fibers. Segmented pie bi-component fibers have been attracting a lot of attention across the innumerable textile applications owing to their advanced multifunctional properties. The advantages of the split segmented pie bi-component fibers are that they can be spun and processed as larger fibers, then split into ultra-fine pie shape fibers at conventional melt spinning process rates.

The spinning of segmented pie bi-component fibers was conducted using a combination of polypropylene, polyethylene, and nylon 6 as base materials embedded with 1% TiO₂ in both components. This was conducted using a lab scale two-extruders system. The fibers were prepared by varying winding speeds from free-fall, 300 m/min., 500 m/min., and 700 m/min. The produced fibers were examined for their appearances and surface characteristics using an optical microscope, Scanning Electron Microscope (SEM), and Atomic Force Microscope (AFM). The Differential Scanning Calorimeter (DSC) was used for studying their thermal characteristics. The antibacterial effects of the fibers were assessed using a modified method based on the standard method, AATCC100: Assessment of Antibacterial Finishes on Textile Materials, by adding a visible light source above the test samples. The filament samples were tested with the two test organisms of *Staphylococcus aureus* and *Klebsiella pneumoniae*. The numbers of bacteria present were determined, and the percent reduction for the specimens with fibers embedded with TiO₂ and that with fibers without TiO₂ were calculated.

The extruded round fibers were split into irregular minuscule pie-shaped filaments, which significantly increased their photocatalytic surface areas. When the winding mechanism applied to the fibers, the average deniers were decreased by approximately 4.5k%. The calculation of the surface areas of the fibers indicates that the surface area of a 16-pie-shape-segments of a split filament are significantly higher than a round intact filament, by approximately 610.0%. The presence of Ti on the detected surface of the split fiber is much higher than that on the intact fiber. The atomic and weight percentages of the Ti on the split fiber surface were increased by 92% and 86% respectively. The percentage of the Gram-positive bacteria reduction (*Staphylococcus aureus*) and the Gram-negative bacteria reduction (*Klebsiella pneumoniae*) in the sample with TiO₂ were 80.00% and 42.94% respectively, compared to 0% in the sample without TiO₂.

Keywords: Segmented pie bi-component fiber, Antibacterial textile, Polypropylene, Polyethylene, Nylon 6, Titaniumdioxide

Dissertation Title การผลิตเส้นใยสององค์ประกอบแบบเช็กแมนไฟเพื่อสิ่งทอผ้าเนื้อ
Name – Surname Asst. Prof. Piyanut Jingjit
Program D. Eng. Energy and Materials
Thesis Advisor Mr. Natee Srisawat, Ph.D.
Academic Year 2018

ABSTRACT

การให้ความสนใจเกี่ยวกับสภาวะแวดล้อมที่ถูกสุขอนามัยที่เพิ่มสูงขึ้นกระตุ้นให้เกิดการวิจัยทางศาสตร์เพื่อการผ้าเนื้อของผลิตภัณฑ์สิ่งทอ การศึกษาวิจัยนี้มุ่งหวังที่จะผลิตวัสดุสิ่งทอที่มีสมบัติการต้านเชื้อที่มีความคงทน ในระดับต้นทุนการผลิตไม่สูงมากนัก โดยใช้การฝังอนุของ inorganic photocatalytic nanoparticles ในเส้นใยสิ่งทอ เส้นใยสององค์ประกอบแบบเช็กแมนไฟได้รับความสนใจในการนำไปใช้ในสิ่งทอเนื่องจากสมบัติที่ดีในหลายๆด้าน ข้อได้เปรียบของเส้นใยสององค์ประกอบแบบเช็กแมนไฟคือสามารถปั่นเส้นใยขนาดใหญ่ แล้วแตกออกเป็นเส้นใยขนาดเล็กที่มีรูปทรงไฟ โดยใช้วิธีการผลิตแบบการผลิตเส้นใยแบบปกติ ได้ทำการปั่นเส้นใยสององค์ประกอบแบบเช็กแมนไฟหลายชุด โดยใช้วัสดุพื้นฐานเป็น โพลีโพลีลีน โพลีเอทิลีน และไนลอน 6 และฝังด้วย TiO_2 ร้อยละ 1 ลงในทั้งสององค์ประกอบ โดยใช้ระบบฉีดเส้นใยสองด้าน ในการผลิตเส้นใยได้ใช้กลไกการม้วนเก็บโดยให้อัตราเร็วในการม้วนเก็บดังนี้ คือ ไม่ม้วนเก็บ 300 เมตร/นาที 500 เมตร/นาที และ 700 เมตร/นาที

เส้นใยที่ผลิตได้ถูกนำไปทดสอบลักษณะปรากฏและลักษณะผิวโดยใช้กล้องจุลทรรศน์แบบคอมพาวด์ เครื่อง Scanning Electron Microscope (SEM) และเครื่อง Atomic Force Microscope (AFM) การศึกษาสมบัติเชิงความร้อนของเส้นใยใช้เครื่อง Differential Scanning Calorimeter (DSC) ส่วนผลของการต้านเชื้อของเส้นใยใช้การประเมินด้วยวิธีการทดสอบมาตรฐาน AATCC100: Assessment of Antibacterial Finishes on Textile Materials โดยตัดแปลงวิธีการทดสอบให้ใช้แสงที่สามารถมองเห็นได้อยู่เหนือขึ้นทดสอบ โดยทดสอบกับสองเชื้อคือ *Staphylococcus aureus* และ *Klebsiella pneumoniae* จำนวนจำนวนเชื้อแบคทีเรียที่ปรากฏ การลดลงของเชื้อคิดเป็นร้อยละของชิ้นงานที่ TiO_2 และชิ้นงานที่ปราศจาก

เส้นใยที่ฉีดออกมาในลักษณะกลมได้แตกออกเป็นเส้นใยขนาดเล็กรูปทรงไฟ ซึ่งเป็นการเพิ่มพื้นที่ผิวที่เป็น photocatalytic อย่างมาก เมื่อเพิ่มกลไกในการม้วนเก็บ ค่าเฉลี่ยของ deniers ของเส้นใยลดลงร้อยละ 4.5k โดยประมาณ การคำนวณค่าพื้นที่ผิวของเส้นใยที่แตกออกเป็น 16 เส้นใยรูปทรงไฟ เพิ่มสูงขึ้นอย่างมาก เมื่อเทียบกับเส้นใยที่ยังเกาะกันเป็นวงกลม โดยเพิ่มขึ้นร้อยละ 610.0 จำนวนของไทเทเนียมที่ตรวจพบบนผิวของเส้นใยที่แตกเป็นเส้นเล็กมีปริมาณเพิ่มขึ้นจากเส้นใยที่เกาะกันเป็นวงโดยจำนวนอะตอมและน้ำหนักของไทเทเนียมบนเส้นใยที่แตกเป็นเส้นขนาดเล็กเพิ่มขึ้นร้อยละ 92 และร้อยละ 86 ตามลำดับ ร้อยละของการลดลงของเชื้อแกรมบวก (*Staphylococcus aureus*) และเชื้อแกรมลบ (*Klebsiella pneumoniae*) ในตัวอย่างที่มี TiO_2 คือ ร้อยละ 80.00 และ ร้อยละ 42.94 ตามลำดับ เมื่อเทียบกับตัวอย่างที่ไม่มี TiO_2 ซึ่งมีค่าการลดลงเป็นร้อยละ 0

Keywords: Segmented pie bi-component fibre, Antibacterial textile, Polypropylene, Polyethylene, Nylon 6, Titaniumdioxide

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Piyanut Jingjit



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List of Abbreviations

| | |
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| SEM | Scanning Electron Microscope |
| HEPA | High Efficiency Particulate Air media |
| AFM | Atomic Force Microscope |
| DSC | Differential Scanning Calorimetry |
| HAIs | Hospital Acquired Infections |
| AATCC | American Association of Textile Color and Chemistry |
| DBPs | Disinfection Byproducts |
| PP | Polypropylene |
| PE | Polyethylene |



CHAPTER 1

INTRODUCTION

The medical textiles marketplace is a large, complex and extremely diverse industrial sector of so-called the technical textiles. It is covered numerous product categories, segments, sub-segments, and sub-sub-segments. The medical textile goods beset an array of different sizes, shape, and configurations. The simplified terms of medical textiles segments are the two primary categories: outside body use and inside body use.

The outside-body medical textiles are the broadest and largest medical textile markets. The outside-body segments include wound care, compression, skin barrier and hygiene products. The outside-body medical textile items are the most familiar medical textiles for consumers. This segment has generated higher volumes, with comparatively lower cost and profit margins. The products may or may not come into contact with the human skin during use. The textile material technologies customarily are specific to the applications and implementations of the products. The applications and implementations of the medical textiles expect the requirement of certain level of testing and qualification prior to the usage acceptance.

1.1 Background and Statement of the Problems

1.1.1 Textile materials have been the most commonly used medias for medical purposes. Medical textiles are manufactured products used in hygiene, healthcare, personal care, as well as hospital and surgical end-uses. They are also used as health protection or filtration to capture undesired microbes, impurities, particles, soil or particulate compounds from fluids of many sources for many decades. The fabric and textile medium for medical implementation have grown in favouring to the whole of industrial spectrum. Fabric and textile filtration systems are also among the most typically used mechanical or physical operation for segregating and separating solid particles from liquids or gases by intervening the medium that only fluid or gas can pass through. Big and bulky particulates or solids in the fluid or gas are hold on the fabric or textile surface. The one of the key protecting and filtrating efficacies is depending on the pore size of the filter and the type and size of the particles.

Textiles and fabrics used in outside-body medical care are generally use for protect and filtrate undesired particles using mechanical and physical processes that liquid or gas containing solid particles pass through porous textile and fabric mediums. The textile and fabric protection and filtration capably detain surround environmental pollutants in gaseous and/or liquid streams. The textile and fabric protection and filtration can be used in air pollution control systems, by discarding particulates or soils from gaseous emissions; and in water purification by removing attached solids. A high efficiency particulate air (HEPA) filter is defined as a filter with an efficiency of 99.97% or greater for 0.3 μm diameter particles. A ULPA filters are defined as having an efficiency of 99.999% or greater against 0.1 – 0.2 μm diameter particles. HEPA and ULPA filters are traditionally formed using nonwoven fabric materials made of glass fibers with round cross sectional geometry. The current advanced melt spinning and melt-blown technologies have accelerated and broaden the uses of new materials in nonwoven fabric filters. The advanced fiber spinning technologies have enabled productions of sub-micron and even nano-scale fiber size.[2]

As diameter of fiber plays a significant role in the filtration efficiency, the means by which fiber size is achieved must be taken into serious consideration.

1) Glass fibers:

Advantages: extremely fine (~1.0 μ m in diameter)
superior packing efficiency
higher density filter

Disadvantage: compact packing depreciates filtration pressure

2) Synthetic fibers:

Made from traditional melt spinning techniques

Advantage: large in diameter
more open filter media
lower pressure drop
void spaces between fibers
higher capacity for particle capture

Disadvantage: void spaces
reduce chance of particle colliding and adhering to it

Modified- melt- blown, splittable and/ or dissolvable bi- component fiber and nano- scale electro- spinning fiber processing are the three fundamental technologies for making ultra- fine textile fibers.

The majority of the bi- component fibers are processed with melt spinning technology. A single strand of bi- component fibers consists of two polymer sections dividing into phases within itself. The potential bi- component fibers that are intended to split each components apart or dissolve out one component are segmented segmented pie, side by side, layer by layer, stripped, tipped as well as islands on the sea.

The explicit advantages of the splittable bi- component fibers are their high speed production as they are initially spun in larger fibers at the traditional melt spinning manufacturing rates, then purposely split or dissolved into minuscule non- circular cross sectional geometry strands. Some of the bi- component fiber technology have ability to produce nano- size fibers which is comparable to electro- spinning but at a much higher production rate. Besides, it also provide additional bulkiness for the fabric without increasing fabric weight. The bulkiness of the fabric is an indicator for a better permeability.

The primary goal of this research work is finding a selection for most applicable materials used for the splittable segmented pie bi- component fibers that meet the intention of the product end- uses. The desired properties of the fibers such as diameter and geometry, density, and special featured were subjected to be customized and evaluated throughout the research works.

1.2 Purpose of the study

1.2.1 To study materials used in segmented pie bi- component fiber process

1.2.2 To study diameter and geometry of segmented pie bi- component fiber with different materials

1.2.3 To produce and detect effects of diameter, geometry, and relevant parameters on the segmented pie bi- component fiber in laboratory scale plant

1.2.4 To produce splittable segmented pie bi- component fibers with special additives for antibacterial textiles

1.3 Scope of the study

1.3.1 To study common materials used in segmented pie bi-component fiber process such as Polypropylene, Polyethylene and Nylon

1.3.2 To produce and study diameter, surface, and geometry of segmented pie bi-component fiber with different materials by using optical compound microscope and scanning electron microscope

1.3.3 To produce splitable segmented pie bi-component fibers with special additives for antibacterial textiles using laboratory scale plant with different feeding speeds and flow rates

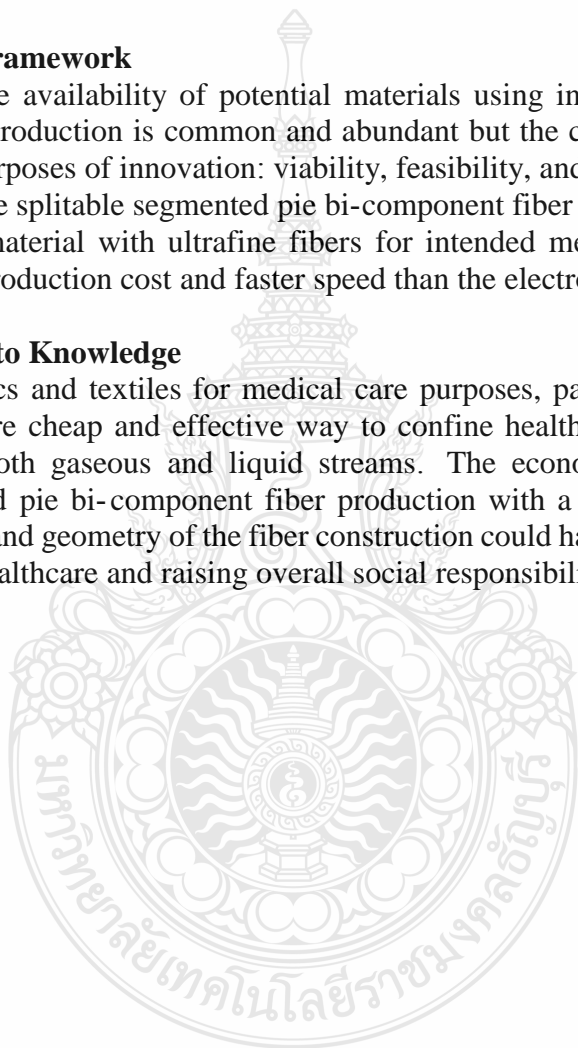
1.4 Conceptual Framework

1.4.1 The availability of potential materials using in the segmented pie bi-component fibers production is common and abundant but the correct match is to meet all the three key purposes of innovation: viability, feasibility, and desirability.

1.4.2 The splitable segmented pie bi-component fiber combination is likely to be an applicable material with ultrafine fibers for intended medical textile uses, in a significant lower production cost and faster speed than the electro-spinning materials.

1.5 Contribution to Knowledge

The fabrics and textiles for medical care purposes, particularly outside-body product segment are cheap and effective way to confine healthcare in highly polluted environment for both gaseous and liquid streams. The economically benefit of the splitable segmented pie bi-component fiber production with a correct combination of materials use, size and geometry of the fiber construction could have tremendous positive impact on social healthcare and raising overall social responsibility and quality of life.



CHAPTER 2

REVIEW OF THE LITERATURE

Increased public concern regarding hygienic living environments has driven recent developments in research to decontaminate air, water, and surfaces to combat the risk of pathogen spread. Even indoor air is a potential vehicle for many human pathogens. Airborne transmission of infectious agents can contaminate the environment and create secondary vehicles, leading to air–surface–air nexus points with viable transmission to prone hosts. Modern humans spend most of their time indoors, which has major implications for human health. Sources of airborne pathogens and surface contamination are ubiquitous indoors. [13] There are various groups of human pathogens known as potential airborne spread: vegetative bacteria (*staphylococci and legionellae*), fungi (*Aspergillus, Penicillium, and Cladosporium spp and Stachybotrys chartarum*), enteric viruses (norovirus and rotaviruses), respiratory viruses (influenza and coronaviruses), mycobacteria (tuberculous and nontuberculous), and bacterial spore formers (*Clostridium difficile* and *Bacillus anthracis*). [10]

Air quality is dynamic as effected by bounteous controllable and uncontrollable factors. Pathogens such as noroviruses and *Clostridium difficile*, are often detected in indoor air. The indoor airborne pathogens may grounded on surfaces, which become secondary carriers for the indoors infectious spreading. Therefore, the quality of indoor air is a marked public health burden which requires a profound understanding in order to develop and implemnet infection prevention and control measures. [10]

The method for studying airborne human pathogens were conducted by experimentally producing them, storing them and sampling them for residual infectious content over a period of predetermined time. The environmental factors that influence airborne microbial survival were also intensively studied as the aerosolised microbes must survive to infect a human host. Air temperature and relative humidity are key factors of the degree of airborne pathogen spreading in indoor environment. Currently, there have been upsurging and marketing of claimed air and surface decontamination technologies which no based on testing under relevant human health conditions. There is a gap in development of standardised testing method for inspect and indicate commercial decontaminating indoor air devices. [10]

Achieving antimicrobial for water disinfection and microbial control has also been a challenge as inappropriate disinfection by conventional chemical disinfectants would initiate harmful outgrowth as well as byproducts. The chemical disinfectants such as free chlorine, chloramines and ozone may form harmful disinfection byproducts (DBPs). The growing demand for decentralised or point-of-use water treatment and recycling systems stimulates the search for new technologies for water disinfection and microbial control. As outbreaks of water borne disease continue to arise more frequency. The antimicrobial mechanisms using nanomaterial as nanoparticles have been intensively discussed for their merits, limitations and applications to innovate nanomaterials for water treatment applications. [14]

The healthcare-associated infections (HAIs) such as *Clostridium difficile* (*C. difficile*) have been ubiquitous in healthcare setting such as hospital. The infections catagorise in HAIs are the infection which occurs more than 48 hours after admission to hospital; or within 10 days of discharge from hospital; or within 30 days for a surgical wound infection; or occurs within 72 hours of out-patient procedure. The key principles of

infection prevention and control are to reduce the scale of the problem by ascertaining potential sources of infection, routing of transmission, and determining role of host factor. The traditional role of textiles in infection prevention and control are bedding and patient and medical personnel dressings. The interest in advanced textile materials for medical care, infection prevention, and control toward nanotechnology, size and surface area related properties of materials has been upsurging among textile scientists and researchers. One area of the recent development of textiles for infection prevention and control are antimicrobial wound dressing and anti-adhesive wound dressing using antibacterial agents such as nanocrystalline silver and titania or silk fibroin nanofibres. [40]

Healthcare and medical textiles

Types of textiles and biomaterials for medical and healthcare applications are covered intired spectrum of natural and manufactured textile fibers from cotton to alginate, from polyester to polylactic acid. [40] Textile structures applicable for medical and healthcare products are catagorised depending on their specific application possibilities, such as vapour-permeable gauzes, tubular bandages, nonwoven fabric, double layered woven fabric, knitted medical stocking, tissue-engineerd vascular grafts, spacer fabrics, mesh grafts, hollow fibers, microfibers and other structural modifications. The suitability of textile materials for medical and healthcare application should have prominent properties for hygiene, protection, therapeutic, etc.

Studies on anti-microbial modifications for commodities are common; technologies for decontaminating human contact pathogens have been developed, including the Microgenix air purification system with chemical-coated filters and a UV source to reduce microbes in heating, ventilation, and air-conditioning (HVAC) systems. [10] Textiles are significant in everyday human life; people are in continuous contact with textiles. Therefore, textiles are potentially conducive for human health protection. The use of special textile materials for healthcare products has been widespread in human hygiene and medical practices.[23][4][5][15] Advanced healthcare textiles have developed significantly, with considerable research conducted globally on healthcare and medical textile materials and polymers. Textiles can be rendered antimicrobial in several ways, such as adding antimicrobial additives to the fiber during spinning or extrusion, combining with dyes or pigments, or adding coatings on the surfaces.[23][4][5]

The development of low-cost medical and healthcare textiles for efficient capture minuscule particles and microorganism protection and control has been many studied and commercially launch into consumer market everywhere. [6] The aim of this study is for a better understanding on how to produce textile material with segmented pie bi-component fibres of cheap polymeric materials such as polyethylene, polypropylene, and nylon added with TiO_2 to utilise its photocatalysis property. The segmented pie bi-component fibres offer alternative material choices for cheap textile product which can be engineered to meet various density, permeability, and protecting performance requirements. TiO_2 has been widely used for environmental products as it has excellent photocatalysis property such as water and air purification filter media.[17]

Titaniumdioxide (TiO_2)

Since the late 1960s, TiO_2 has been considered one of the most valuable photocatalytic materials, because it can be used for photoelectrochemical solar energy

conversion and the photocatalytic decomposition of organic compounds even under low-intensity illumination. [21][23] The material is stable, durable, inexpensive, and safe for health. [25][3][18] In addition, it can be used without additional catalysts. TiO₂ photocatalysts decompose microbial organisms and other organic compounds by generating an oxidizing mechanism under photonic excitation, where a photon generates an electron-hole pair on the surface that reacts with water or hydroxide ions to become a hydroxyl radical. [23] As mentioned earlier, TiO₂ photocatalysts can kill microbes; therefore, self-sterilizing materials have many potential uses for everyday products that make contact with human skin. [21][23][24]

A well-designed photocatalytic material can effectively break down organic compound under illuminated light make it useful for many potential textile applications such as environment purifications and basic healthcare products. The split bicomponent fibre embedded with photocatalytic material would enhance their advantageous properties such as self-cleaning, antifogging and stain-proofing, water purification and air cleaning, formaldehyde and acetaldehyde decomposition by multiple the effective surface area with split bicomponent fibres technique. Photocatalytic surfaces require a certain light intensity, but with the present of TiO₂ as its photocatalytic material the required energy of small amount of photons as in ordinary room light would be sufficiently induce the photocatalysis. The longer the material is illuminated with light at shorter wavelengths than ~400 nm (UV light), the higher photocatalytic effect on decomposing organic compound material. [21] With sufficient humidity and temperature, textiles are a potential substrate to grow micro-organisms and come to contact with human skin. The increasing concern in public hygiene and health safely environment has driven research and study in modification of anti-microbial textiles. The limitation of using some certain anti-microbial agents is mostly because of their possible harmful or toxic side-effects. The applications of inorganic particles have been studied as the alternatives. The inorganic structured materials with anti-microbial effects include TiO₂, metallic and non-metallic TiO₂ nano-composites, titania nanotubes (TNTs), silver nano-particles, silver-based nano-structured materials, gold nano-particles, zinc oxide nano-particles and nano-rods, copper nano-particles, carbon nanotubes (CNTs), nano-clay and its modified formation, gallium, liposomes loaded nano-particles, metallic and inorganic dendrimers nano-composite, nano-capsules and cyclodextrins containing nano-particles.

The photocatalytic material, Titanium dioxide (TiO₂) has become one of the most basic material in everyday life since it proved itself to be a superb photocatalytic material available in the most competitive price. It is also an extremely stable semiconducting material with sufficiently positive valence band edge to oxidize water to oxygen. [24]

The efficiency of photocatalytic mechanism is assessed by measuring photonic efficiency which is the rate of reaction of product formation divided by the incident photon flow. Time-resolved spectroscopic studies reveal that the most photogenerated e⁻/h⁺ pairs (approx. 90%) recombine rapidly after excitation. [12] The reaction dynamics of the photogenerated charge carriers in TiO₂ are generally obtained by means of time-resolved absorption spectroscopy, being a widely applied technique to study formation, relaxation, recombination, and transfer process of photogenerated charge carriers in photocatalytic materials. [21,26] The time-resolved absorption spectroscopy technique, the specimens will be excited by laser pulse with absorption of the formed intermediates being detected by time-resolved optical spectroscopy usually in the UV/vis/NIR region

with visible white light. [26] The measuring techniques have been improved and it is now possible to measure and evaluate on the femtosecond time scale, meaningful interpretation of observed kinetics or a chemical assignment of the detected absorption transients.

TiO₂ with stoichiometric surface, flat bands have been found from several photoemission experiments that the surface contains many of defects which can be understood as unpaired electrons that locate initially in an O 2p orbitals then transferred into the conduction band formed by Ti 3d orbitals. The electronic situation of these extra electrons in the vacancies act as donor-like states create an accumulation layer in the near-surface region resulting in a downward band bending. [23] The accumulation of electrons at surface determines surface chemistry of TiO₂. The processes occur at surface of semiconductors are driven to achieve an equilibrium between Fermi level potential and chemical potential of the adsorbates with position of Fermi level being equal to the work function of semiconductor. The calculation using density functional theory can define the influence of excess electrons on the adsorption behaviour of the adsorbates at the surface of TiO₂. The charge transfer from the reduced surface to the adsorbate occurs provided that the electronegativity of the adsorbate exceeds the surface electronegativity of TiO₂. The oxidation state of the surface and redox potential of the adsorbate, electrons can flow from or to the adsorbates, the negatively or positively charged species are formed. Therefore, it is possible to control the surface chemistry of TiO₂, when the adsorbates do not donate or accept electrons, no charge transfer will occur and the interaction strength will not depend on the oxidation state of the surface. [23,31]

Melt-spun bi-component fiber extrusion

Bi-component fibers are composed of two constituent polymers, unlike fibers of polymer blends, which can be purposely separated along fiber length which unique features that offer considerably advantages such enhancing desired properties. [43] Melt-spun bi-component fiber extrusion has been broadly employed for preparing functional and technical textile materials since the invention of this flexible and cost-effective technology in the 1990s.[6] Single filaments can be spun with two polymer components in designated morphologies, including side-by-side, island-on-the-sea, or segmented-pie shapes. The different designated types of bi-component fibers alter their characteristics, physical properties, shapes, and applicability for different potential uses.[6] The multiple inner sections can also be altered by removing one component by the proper selection of a solvent, or by separating the two components physically. Fibers can be spun from two polymers of the same or different generic types. The technology allows the production of ultra-fine fibers at the same rate as achieved in conventional fiber-spinning processes. The segmented-pie bi-component fiber morphology was selected for this experiment because the purpose was to produce fibers with very high surface areas. This was achieved by separating the injected round-shape bi-component fibers into pie-slice-shaped filaments.[6]

The melt-spun bicomponent fibres have been extensively used for functional and technical textile end-uses since the process was developed to be more flexible and cost effective in 1990s. The fibres can be spun from two polymers with same generic types and different types of polymers. One of the practical ways of forming ultra-fine fibres with same production speed as conventional fibre spinning process, is to use bicomponent fibre spinning technology. The bicomponent fibre spinning technology can

produce fibres as large fibres with multi-inner sections that can be further processed to separate the inner sections into much smaller individual fibres using well-designed sequential processes. The segmented pie bicomponent fibre is one of the common form of bicomponent fibre. The fibres are injected as round-shape fibres with 16 or 32 pie-shape segments section inside which later on can be separated apart. The fibres then become super fine pie-shape fibres which upsurges their surface area.[6]

Synthetic fibers are oriented materials and important to human's life today as their customised characteristics have made them capability manipulatable in vast applications. Melt spinning, dry spinning, and wet spinning are their productions regarding different polymers. The spinning method are depended on polymer material properties and their required performance. The fibers can be customised as solid, delta, hollow, or other cross-sections, and can be made from one, two, or more polymer materials. [6] Multicomponent fibers have been giving benefits to mechanical, physical, or chemical properties of two or more different polymer materials. Selection of materials is based on their production methods and intended applications. Spinning of bi-component fibers, the two selected materials will encounter each other at the spinneret head to form intended configurations such as segmented pie, core/shell, side by side, or islans in the sea. The extruded bi-component fibers exposition alter properties and characteristics such as self-crimped, solidification, based on each polymers' natures. Segmented pie bi-component fiber is among the most common configulations used in technical textile applications, providing desire properties from the both polymers. Besides, the segmented pie bi-component fiber with best fit polymers could be sepatated into many smaller fibers, multipling total active surface area of the fibers. [41]

The microscale fiber applications include filtration, composite materials, medicine delivery, tissue engineering, and biological textiles. The viscosity ratio of materials use in bi-component fiber productions is one of the most important aspects to be initail considered. Polypropylene and polyethylene are among the largest materials for segmented pie bi-component fiber productions. Some of the advanced bi-component fibers are applicable for using as artificial organs, such as muscules and vessels. The antibacterial bi-component fibers have been widely used in medical applications. The progress in spining of bi-component fibers and their treatment technologies will steady enhance their advanced applications. [30]

The melt spinning of bi-component fibers is one of the most common commercial processes. The melt spinning of segmented pie bo-component fibers, two extruders melt and pressurise the selected two polymers separately before join one another at the spinneret heads to make the segmented pie cross-sectioned bi-component filaments. The properties enhancing for bi-component fibers have been under intensive studies lately. The segmented pie bi-component fibers have significant advantages in increasing active surface area by segments separation. [6]

Splitable bi-component fiber can deliver softness, extensibility, and bulkyness to the material. Soft textile materials are gentle and affable to the skin and useful in some disposable materials. Decreassing of fiber diameters and/or increasing surface area of textile materials can significantly raise softness. The mechanical post-treatments are also benefit to the extensibility of the small scale diameter fibers. However, having small fiber diameter with high extensibility is difficult to attain, because when the diameter is reduced with higher spinning speed or drawing ratio, the extensibility will be decreased. The extensible nonwoven textiles that made from small-scale fibers using convention

thermoplastic polymers are in high demand. The invention of splittable fibers can provide small diameter and extensible fibers. The splittable fibers are cost-effective and easy process. The splitted bi-component fibers correspond to thermoplastic polymer components will have higher elongation rate than direct spun mono polymer fiber at equivalent mass. [42,43] The small diameter fiber can be produced at low spinning speed and provide better elongation property comparing to conventinal process with low mass through-put and consequently low cost fiber production. The configurations of the splittable bi-component fibers are side-by-side, segmented pie, hollow segmented pie, island-in-the-sea, segmented ribbon, tipped multilobal, and any combinaton of the configurations. Generally, segments of the two polymers within a single fiber will split from their adjacent segments and become smaller and different shape fibers. The segments can be split by chemical, mechanical, thermal, or other mechanisms. This splitability can also be obtained immediately upon their fiber formation through spinneret. Their rheology, thermal, solubility, surface energy, extensibility, and solidification will be differed from their initial extrusion stage. [41] The splittable bi-component fibers can deliver higher extensibility because they can be spun in relatively low molecular orientation and relatively large diameters, which can occur with relatively slow spinning speeds without high drawing forces. The fibers are usually subjected to be drawn to reduce the fiber diameter and increase fiber strength with softer feel for nonwoven textile materials, but the drawing mechanism increases molecular orientation of the fiber resulting decrease its elongation. The split fibers of bi-component fiber provide higher elongation comparing to mono-polymer fibers at the same diameter and mass through-put. Besides, the softness of the nonwoven fabric produced from split fibers will be improved as a result of the improved extensibility. [6,41]

The materials suitable for the bi-component fiber spinnings should have melting temperatures of thermoplastic materials (60°C to approx. 300°C). However, thermoplastic polymer materials that have melting temperature above 250°C, may use plasticiser or diluents to lower the melting temperature. The glass transition of the thermoplastic polymer materials should be less than 0°C. The molecular weight of the material should be sufficiently high in order to enable entanglement between molecules of the polymers but low enough to melt. The molecular weight that suitable for the thermoplastic melt spinning should be approximately 1,000k g/mol or below, and not less than 5k g/mol. They must be solidify rapidly under extensional flow. Polyolefins with low, high, linear low, or ultra low density polyethylene, polypropylene and their copolymers, polyamides and their copolymers such as nylon 6, nylon 11, nylon 12, nylon 46, nylon 66; polyesters and their copolymers such as polyethylene terephthalates; olefin carboxylic acid and their copolymer such as ethylene/acrylic acid copolymer, ethylene/maleic acid copolymer, ethylene/methacrylic acid copolymer, ethylene/vinyle acetate copolymers or combination among them; polyacrylates, polymethacrylates, and their copolymers such as poly(methyl methacrylates), are the examples of ideal polymers for melt spinning bi-component fibers. [41]

Some of the biodegradable thermoplastic polymers are also applicable for the melt spinning bi-component fibers. The biodegradable polymers are susceptible to assimilated by microorganisms such as molds, fungi, and bacteria, when the materials come into contact with microorganisms in conducive environment, the microorganisms will grow on them and become degraded using aerobic or anaerobic digestion procedures, or by exposing to environmental elements such as sunlight, rain, moisture, wind, temperature. The biodegradable thermoplastic polymers can be used individually or as a

combination or as a combination of biodegradable or non-biodegradable polymers. The biodegradable polyester containing aliphatic components, ester polycondensates containing aliphatic constituent and poly- (hydroxycarboxylic) acid and ester polycondensates such as diacids/ diol aliphatic polyester, polybutylene succinate, polybutylene succinate co-adipate, aliphatic/aromatic polyester, terpolymers made of butylenes diol, adipic acid, and terephthalic acid, are among the melt spinning applicable materials for bi-component fibers. The poly(hydroxycarboxylic) acids including lactic acid based homopolymers and their copolymers, polyhydroxybutyrate (PHB), or other polyhydroxyalkanoate homopolymer and their copolymers with higher chain length monomers such as C6-C12. [41,30]

The softness, textures and relevant properties of the fibers are depended on the selection of the polymers and amount of polymers used in the process. The thermoplastic polymers in the components can contain a single polymer specie or a blend of two or more of the non-starch thermoplastic polymers.

The term 'split fiber' is used for the obtained fibers upon separation or splitting from the bi-component fibers. Splitting or separation mechanism can be any techniques such as chemical removal of one component polymer or dissolving of an acid to facilitate the separation of the component, as well as mechanical separation, or a combinations of them. The component mechanical separation can be accomplished by applying force such as drawing, hydroentangling, stretching. The bi-component fibers with incompatible polymer materials, its components may split or separate apart upon spinning or normal handling of the fibers. The dissolve is also one a technique for splitting the components in bi-component fibers by plasticizer, or solvent or reactive medium (liquid or gas). [41,43]

References

Photocatalytic materials for antibacterial: 03, 08, 09, 11, 12, 13, 14, 15, 18, 20, 21, 23, 24, 26, 28, 30, 31, 32

Antibacterial textile applications: 04, 16, 22, 33, 34, 35, 36, 37, 38

Textile testing: 05, 07, 08, 10, 24, 25, 29, 30

Bicomponent fiber spinning: 01, 05, 06, 10, 17, 27, 30, 33, 37, 41, 42, 43, 44

CHAPTER 3 RESEARCH METHODOLOGY

To fully understand how bi-component fibers are processed using commonly found fiber polymers and to study splitting mechanism of the bi-component fibers to produce ultra-fine detached fibers, the series of laboratories works have been sequentially conducted in Rajamangala University of Technology Thanyaburi at the Faculty of Engineering using the Synthetic Fiber Processing Laboratory and related facilities at the Department of Textile Engineering.

Some testing, such as SEM (Scanning Electron Microscope), AFM (Atomic Force Microscopy), and assessment of antibacterial activity were conducted using commercial certified technical laboratories.

The different characteristics of the produced fibers using different production settings assisted the selection of the most applicable of the materials for the intended splittable bi-component fibers. Additionally, examining the fibers produced in the laboratory gave a comprehensive view on their physical and biological properties for the medical textile uses.

This research aims to select the most suitable materials for producing prototypes of textile fibers for special uses in medical or healthcare with the bi-component fiber technology. The research did not include encompassing views of medical text and relevant works of medical policy such as studies on healthcare statistics and the law reform affect the actions of healthcare professional.

3.1 Research Planning

The research and laboratories works were carried out roughly as the timeline shows in table 3.1.

Table 3.1 Research planning of the production of segmented pie bi-component fibers for antibacterial textile

| Task | 2016 | | | | 2017 | | | | 2018 | | | |
|---|-------|-----|-----|------|------|-----|-----|------|------|-----|-----|--|
| | month | | | | | | | | | | | |
| | 1-3 | 4-6 | 7-9 | 1-12 | 1-3 | 4-6 | 7-9 | 1-12 | 1-3 | 4-6 | 7-9 | |
| 1. Research planning | ←→ | | | | | | | | | | | |
| 2. Literature review | ←→ | | | | | | | | | | | |
| 3. Proposal examination | ←→ | | | | | | | | | | | |
| 4. Polymer selection and material testing | | | ←→ | | | | | | | | | |
| 5. Fiber spinning and testing | | | | ←→ | | | | | | | | |
| 6. Progress examination | | | | ←→ | | | | | | | | |
| 7. Study fiber detaching mechanism | | | | | ←→ | | | | | | | |
| 8. Performance testing | | | | | | ←→ | | | | | | |
| 9. Results and discussion | | | | ←→ | | | | | | | | |
| 10. Final report | | | | | | | | ←→ | | | | |

3.1.1 Literature review

The literature review in this thesis was selected and organised around the intended ideas of producing splittable segmented pie bi-component fiber for medical or healthcare uses. The research theme and issues were connected and sourced together to present different solutions that initially missing from the field. The review also presented the relevant materials and portray the core concept of the research ideas according to appropriate theories with a reveal of the trend and current applications in the field.

The review used information that was as current as possible. However, the research concepts and ideas were built on long existing technologies, the initial literature unavoidably needed because the importance of how the technology has evolved through the years.

The review contained the introduction and background information which give a quick idea of the topic of the review and central theme, the discussion of sources, and the conclusion and recommendations.

The reviews of literature were the thematic review type which organised around core topic and issue of the preparation and characterisation application of segmented pie bi-component fibers for antibacterial textile. The reviews emphasized the current development of the materials, technology, and implementation of antibacterial additives. The selections of the most important points in each source were highlight in the review which is related directly to the review's focal points.

3.1.2 Polymer selection

The polymers used for the intended experimental bi-component fibers vary depending on the spinning ability of the polymers, machine setting, compatible between the two polymers, and their end-uses. The early fiber materials for medical uses were mostly made from natural fibre polymers, which are relatively inexpensive but have several limitations. Therefore, fibers made from synthetic polymers, which can withstand higher tension and better chemical and biological resistant, are more widely used today. The advancement in melt spinning and melt-blown technologies have broaden the list of materials in medical textile applications, even fibres with cross-section as sub-micron and nano-scale size range.

There are several polymers potentially use for the segmented pie bi-component fibers, depending on the desired performance. The purposes of polymer selection for this research are to find the matching polymers that meet the machine capability and be able to withstand fiber spinning process conditions such as spinning and winding speed, extruder and spinning heating temperatures, the geometry and shape of the fibers, and the ability to detach the fiber segments within the fiber, at the same time the selected polymers must have the desire properties such as ability to detach segments apart into minuscule strands and desire properties for medical or health care uses.

Bi-component fibres are the fibres that being extruded using two polymer materials through the same spinneret head at the same time. A single extruded filament contains both polymers in separate areas. The polymers given below are the primary selected polymers for this research, because they are common and low cost.

- 1) Polypropylene
- 2) Polyethylene
- 3) Nylon 6

3.1.3 Material and fiber testing

Testing was a crucial aspect of the research to detect the performance and properties of the selected materials. The extruded fibers must be properly evaluated, compared, and assessed using recognized testing procedures and standards. The testing gives the results to direct the next route for the research experiments. It also detected the faults of the machine settings and gave exact investigation for the better spinning conditions. The testing results enhanced and efficient output of the experiments.

The tests for this research were for examining the physical, mechanical, chemical, and biological properties of the fibers. Some of the tests were done while fibers were being extruded, to identify problems or faults in the machinery setting conditions and to double-check that materials used were appropriate.

Some of the tests are physical test for the appearance of the fiber which were done on individual filaments and strands of detached segmented fibers. The mechanical tests were those in which fibers were subjected to pressure and stress, including tensile strength, the force applied to break a fiber to ensure that the fiber were strong enough to maintain integrity when use. The biological test was conducted as to evaluate, compare, and assess for outside-body textile for medical and health care applications.

The test results were properly attained for accuracy and precision of the experimental product interpretations. The examination and testing of the fibres will be either before and/or after various process treatments.

The test procedures and standard methods for this research were for the structure and performance of the materials such as:-

- 1) Thermal analysis
- 2) Visual and microscopic analysis
- 3) Three-dimensional shape (topography) of surface at high resolution
- 4) Surface topography and composition of sample with back-scattered electron imaging
- 5) Quantitative X-ray analysis and X-ray mapping of specimens
- 6) Tensile strength
- 7) Antimicrobial analysis

The tests were the essential part of this research as they provided the examined and assessed of the properties of the subjected materials. They also helped to select the suitability of the raw materials and provided information in creating textiles for the intended specific uses.

3.1.4 Fiber Spinning

Bi-component Extruder System.

The fiber spinning experiments in this research were carried out using A Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS- 100 by The Hills Inc.

The machine was designed to produce any cross-section of bi-component fibers and used for bi-component micro-denier applications. The take-up speeds was up to 1,500 meters per minute, and equipped with central process control system. The schematic of the bi-component extruder system is given in Fig. 3.1 and the images of The Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS- 100 are given in Fig. 3.2.

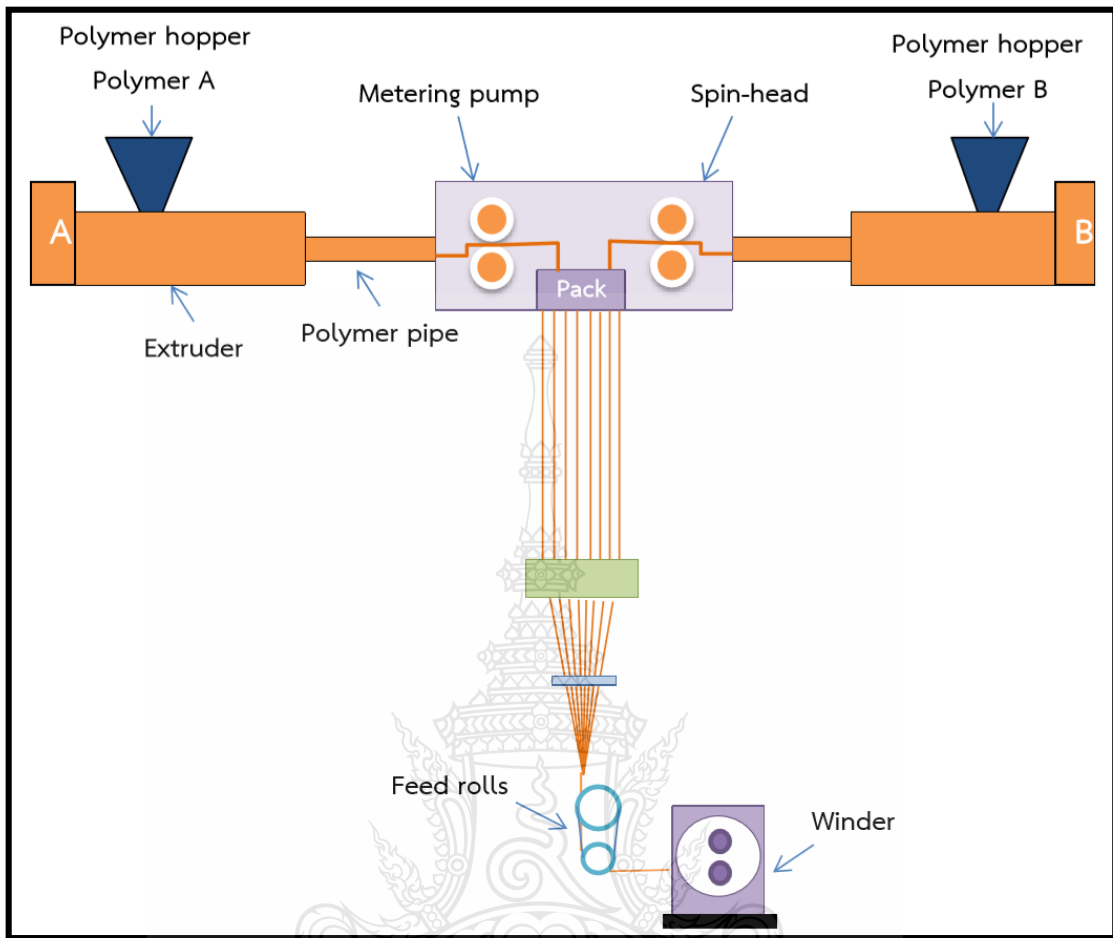


Figure 3.1 Schematic of Bi-component Fiber Extruder

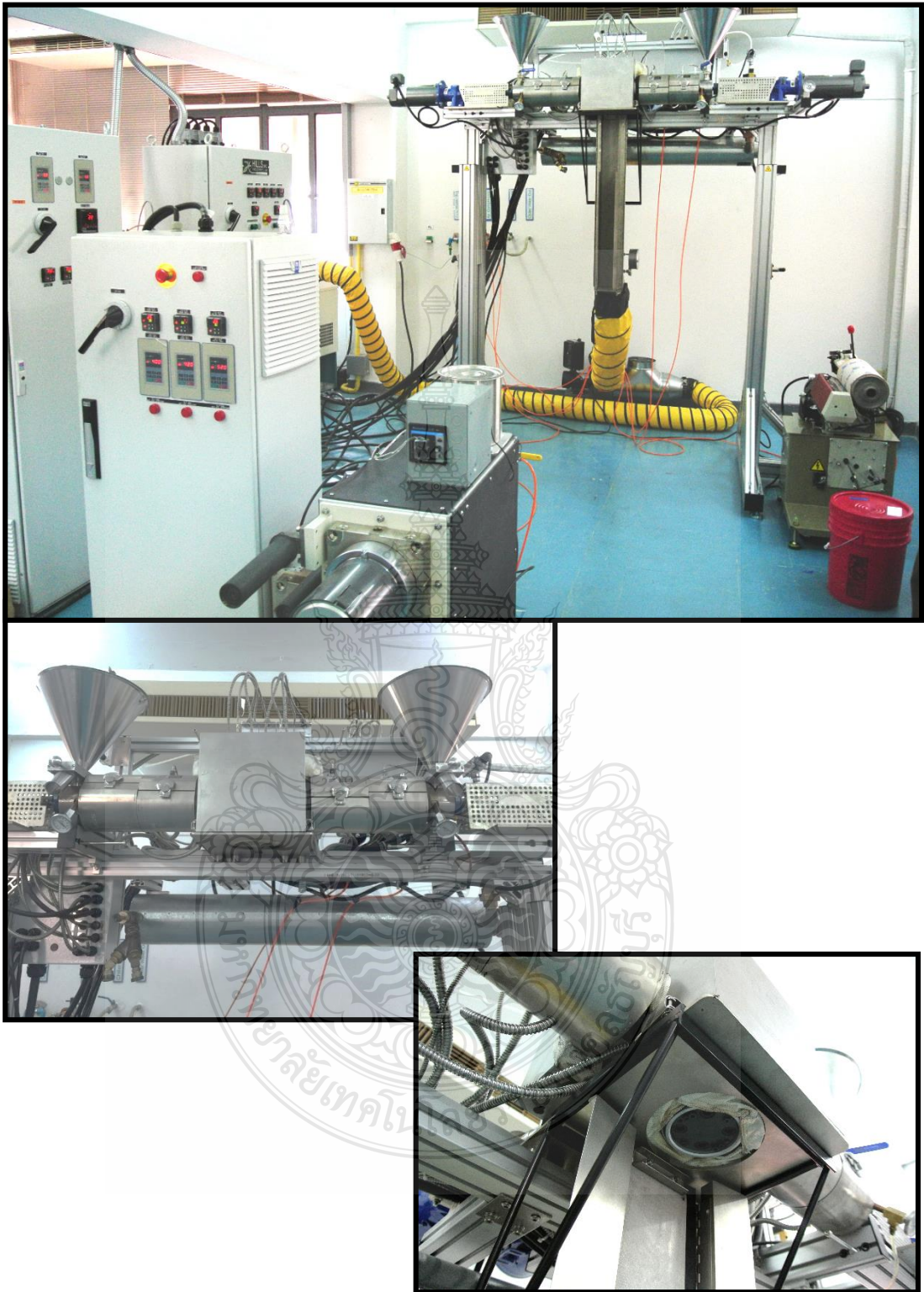


Figure 3.2 The image of The Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100

This machine is designed for laboratory use to make small amounts of bi-component fiber from small amounts of polymer. It requires as little as 20 grams of polymer to make 100 meters of fiber. Its simplified design also makes it easy to operate and maintain. The system consists of 2 single screw extruders connected to a spinneret block. Metering pumps are optional to reduce the minimum polymer requirements and maintenance. The extruder speeds are set to deliver the desired flow rates. Mechanical torque limiters are provided to prevent over pressure. That the simplified design means the tolerances of fiber properties (denier & bico ratio) are limited to +/- 10%. Meter pumps can be added as an option to get production fiber tolerances of +/- 0.5%. The machine is very versatile. With optional parts, the machine is convertible to run monofilaments, high temperature polymers, melt-blown and spun-bond webs in almost any cross section desired.

The machine specifications with complete unit ready to run for this research:

- 1) Extruder unit, with drive mechanisms
(Variable speed D.C. electric motor driven)
 - (1) Extruder Barrel Pressure rating of 7,500 psi (500 bar)
 - (2) Screw size 5/8" dia – 24:1 l/d (16mm)
 - (3) Can deliver polymer pressure of 2500 psi (166 bar)
 - (4) Temperature capability of up to 310°C with temperature control of $< \pm 3.5^{\circ}\text{C}$
 - (5) Electrically heated, 5 zones: 2 / extruder; 1 spinneret
 - (6) Pack block - 17-4 ph stainless steel
 - (7) Torque limitation on extruder screw for barrel over pressure Protection
 - (8) Throughput range: 0.05 to 1 kg / hour for each extruder
 - (9) Control of extruder screw speed $\pm 5\%$ via KBIC dc motor controller (pressure controls optional)
 - (10) Dial gauge type pack pressure indicators
 - (11) Quantity of unused polymer in each system less than 10 cc
2. Spin Pack
 - (1) Spinneret: 19 round holes, 0.5mm dia, shaped holes available
 - (2) Screen sandwich type filters
 - (3) Distribution plates for homo, side-by-side, sheath/core, 16 segment pie and islands-in-the-sea
3. Fiber Winder, lab style
 - (1) 50 – 1000 meter / minute, 75 mm id x 150 mm cardboard bobbins
4. Utilities: 240 vac – 1 phase, 90 amp
 - (1) 240 vac 3 phase 25 amp (melt-blown option)
 - (2) 35 cfm 90 psi compressor air (intermittent) for string up aspirator
 - (3) 40 cfm 80 psi compressor air for melt-blown pack
 - (4) 35 cfm 90 psi compressed air for spun-bond
 - (5) 5 cfh nitrogen for hopper purge
 - (6) 1 gpm water @ 30oC for extruder cooling

3.1.5 Fiber Tests

3.1.5.1 Tests for structure of the materials [39]

The structure evaluation of textile materials in this research were based on sources of information such as:

Thermal analysis

Visual and microscopic analysis

Three- dimensional shape (topography) of surface at high resolution

Surface topography and composition of sample with back-scattered electron imaging

Quantitative X-ray analysis and X-ray mapping of specimens

Tensile strength

Antimicrobial analysis

The techniques used in the research were:

1) Optical microscopy

The optical microscopy used in this research were compound light microscope with magnified lens. The images were captured and generate digital micrographs by digital camera mounted to the microscope and equipped with observation computer screen. The magnification of the microscope is the powers of eyepiece and the objective lens and the maximum of the magnification of this research was 1,000x.

The use of the microscope in this research was for visual analysis at high pixel magnified images of the experimented materials and fibers, as well as the precise measurements.

2) Electron microscopy and electron diffraction

The scanning electron microscopy (SEM) is a method for examining surface detail of the fibers. SEM gave the three-dimensional shape (topography) of the fiber surface at high resolution. The principle is to traverse a spot of electrons across the fiber surface to form an image on a screen scanned synchronously with the spot. In the usual mode of operation, the image are as ordinary enlarged picture of the fiber surface. There are also several special modes of use that provide added information of the materials. The range of the magnification of the image using in fiber science is medium to high magnification which is beyond the limit of the optical microscope. The scanning electron microscope has considerable advantage of much clearer depth of focus.

Atomic force microscopy (AFM) was also used for examining the experimented fibers. The fiber specimens were examined using a probe with a minute tip mounted on a cantilever arms. As the probe move across a fiber surface, the tip of the probe rested on the surface of the fiber, the tip rose and fell and the deflection of the cantilever was a measure of force. This was viewed as scanning over an area on the specimen and conversion of the response to a grey scale gave images of the fiber surface topography.[39]

3) Thermal analysis

The experimented fibers were measured the variation of specific heats of the polymers with temperature for transition effects in the fiber structures. The measured values were spread over ranges of temperature, therefore, the heat did not appeared as latent heat of fusion at single temperatures bur rather contributed to heat

changes over whole melting ranges. By the integration of the areas under the peaks of each specimens, the values of the latent heats of fusion were informed.[39]

3.1.5.2 Tests for performance [40]

Fibers and polymeric materials that could acquire bio-functions as well as acceptable mechanical properties and able to carry medication agents are the potential materials for medical and healthcare textile products. The polymeric materials that suitable for outside-body healthcare must have properties that enable them to be applied as hygiene, protection, therapeutic, etc.

The important properties of textiles for outside-body healthcare and medical uses are stable and spatial structure, purity and non-toxicity, sterility, effective barrier against microorganisms, dirt, liquid and other foreign bodies.[40]

1) Tensile strength

A mechanical properties of a single fiber in responding to applied force and deformation is one of the most crucial properties. It contributes both to behaviour of the fiber in processing and to the performance of the intended end-uses.

2) Antimicrobial analysis

The measurement of reducing bacterial count or preventing microbial growth is the key index for antimicrobial properties of materials. The textiles for outside-body healthcare or medical uses commonly follow a standard test method such as AATCC100. This research followed the AATCC100 test method to measure the reduction in bacteria after 24 hours incubation under static conditions. The test organisms used in the test were *Staphylococcus aureus* which specified for Gram positive and *Klebsiella pneumoniae* specified for Gram negative.

Spinning of splittable segmented pie bi-component fibres were developed to form minuscule fibres using a two extruders system. Areas of the study include the examinations of their physical configurations such as size, shape, and surface using optical microscope, Scanning Electron Microscope, and Atomic Force Microscope. The assessments of the filaments' thermal characteristics, the amount of energy their absorbed and released when heated and cooled and their transformation temperatures with determination of crystalline to amorphous transition temperatures using Differential Scanning Calorimeter were performed.

The selection criteria for material used in the experiment was that they must be compatible to fibre injection in melt spinning process, considerable low cost, common and available in the market, applicable for technical and functional textile end uses and capable to thoroughly accommodate the photocatalytic material. The materials use in the bicomponent fibre forming that intend to separate them into smaller fibres is a crucial factor, because the cohesion of the two selected materials will influence the attempt in splitting them apart. Polyolefin (polypropylene and polyethylene) and Nylon 6 are among the typical fibres use in clothing, technical and functional textiles nowadays. There are vast variety of types and grades available in commercial market, therefore, they were selected as the base material for the experiment. For the photocatalytic material, Titanium dioxide (TiO_2) has proved itself to be a superb photocatalytic material available in competitive price in everywhere, it has become one of the most basic material in everyday life. It is also an extremely stable semiconducting material with sufficiently positive valence band edge to oxidize water to oxygen.[21] The experiment is to examine the achievability of the fibre extrusion and splitting mechanism using the selected base materials and the photocatalytic material. The outcomes of this

report are their characteristic, properties, and possibility to use them in the applications of medical and healthcare textiles.

The further intention of the experiment was that a well-designed photocatalytic material can effectively break down organic compound under illuminated light make it useful for many potential textile applications such as environment purifications and basic healthcare products. The attempt to use split bicomponent fibre embedded with photocatalytic material is for enhancing their advantageous properties such as self-cleaning, antifogging and stain-proofing, water purification and air cleaning, formaldehyde and acetaldehyde decomposition by multiple the effective surface area with split bicomponent fibres technique. Photocatalytic surfaces require a certain light intensity, but with the present of TiO₂ as its photocatalytic material the required energy of small amount of photons as in ordinary room light would be sufficiently induce the photocatalysis. The longer the material is illuminated with light at shorter wavelengths than ~400 nm (UV light), the higher photocatalytic effect on decomposing organic compound material.[21]

3.2 Segmented pie bi-component fiber spinning: Experiment I

The experiment I (PP/PE free fall)

The experiment I was for an initial tentative step for producing segmented pie bi-component fibers using the available materials with the bi-component extruder system in the laboratory.

Objectives of the experiment I (PP/PE free fall)

- 1) To produce segmented pie bi-component fibers
- 2) To study the applicability of the two selected materials for producing segmented pie bi-component fiber
- 3) To study the system condition for producing segmented pie bi-component fiber using the two selected materials
- 4) To study the implications of the setting condition on the produced fibers.
- 5) To study the bi-component fiber without applying winding speed (free fall)
- 6) To study shapes of the produced segmented pie bi-component fibers using light compound microscope

Instruments:

- 1) Bi-component Extruder System

The fiber spinning experiments in this research were carried out using A Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS- 100 by The Hills Inc. which consists of 2 single screw extruders connected to a spinneret block. (Fig. 3.2)

Materials:

There were several potential polymers for the experiment I for the segmented pie bi- component fiber spinning available in the laboratory. For the first attempt, Polypropylene and polyethylene were selected to be spun. The purposes of raw material selection for this tentative step were to find the compatible polymer resins that meet the machine system capability. The resin must be able to withstand the setting spinning process conditions such as spinning speed, winding speed, extruding temperatures, and spinning heating temperatures. Then the geometry and shape of the fibers were to be examined. Besides, the detaching mechanism of each polymer segments were to be analysed.

1) Polypropylene: Moplen HP561R (introductory product)
 Polypropylene (PP) used in the experiment was MoPlen HP561R produced by HMC Polymers Company Limited which is a member of the Basell family of polypropylene, polyethylene and advanced polyolefin companies.

Features

- (1) Polypropylene Homo-polymer Resin for Spunbond Fiber
- (2) High melt flow with very narrow MWD resin for high speed spin continuity of low denier fiber
- (3) Very uniform melt flow rate and low gel
- (4) Excellent processability and wide processing operating window
- (5) Spinnability improved with enchanted fabric properties
- (6) Very good gas fading resistance

Typical Applications:

- (1) Spunbonded nonwoven fabric using for hygiene products, medical, agriculture, geotextile, etc.

The resin meets FDA requirement in the Code of Federal Regulations in 21 CFR 177.1520 for all food contact. All ingredients in the resin meets the chemical registration requirements of TSCA (U.S) and DSL (Canada)

Table 3.2 shows the resin properties of Moplen HP561R used in the experiment.

Table 3.2 Resin properties of polypropylene: Moplen HP561R

| Resin properties (a) | Moplen HP561R | ASTM method (b) |
|---|---------------|-----------------|
| Melt flow rate, dg/min | 25 | D 1238 |
| Density, g/cm ³ | 0.90 | D 792B |
| Tensile strength at yield, MPa | 32 | D638 |
| Elongation at yield, % | 11 | D 638 |
| Flexural modulus, MPa | 1240 | D 790A |
| Notched izod impact strength at 23°C, J/m | 24 | D256A |
| Deflection temperature, at 455 kPa, °C | 94 | D 648 |

(a) Values shown are averages and not to be considered as specifications.

(b) ASTM test methods are the latest under Society's current procedures. All molded specimens are prepared by injection.

2) Polyethylene: EL-Lene H5818J

Polyethylene (PE) used in the experiment was EL-Lene H5818J produced by SCG Plastics Co., Ltd. which is a company under the family companies of SCG Siam Cement Group Chemical. It is a general purpose high density polyethylene resin suitable for using in injection molding process.

Features

- (1) High density polyethylene for injection molding
- (2) Good flowability grade
- (3) Excellent processibility
- (4) Good impact resistance
- (5) Food contact applicable (complies with US FDA 21 CFR177.1520)

The recommended processing conditions for the resin is at melting temperature: 200-250°C, mold temperature: 20-60°C, injection speed: fast, screw speed: 40-70 rpm, pressure: injection 30-70%, packing and holding: 30-50%, back: 10%, and of Max: pressure. Table 3.3 shows the resin properties of EL-Lene H5818J used in the experiment.

Table 3.3 Resin properties of polyethylene: EL-Lene H5818J

| Resin properties (a) | EL-Lene H5818J | Test Method |
|---|----------------|---|
| Melt flow rate, g/10min | 18 | ASTM D 1238 @190°C, 2.16 kg. |
| Density, g/cm ³ | 0.962 | ASTM D 1505 |
| Tensile strength at yield, Kg/cm ² | 280 | ASTM D 638 @ Crosshead speed 50 mm/min |
| Tensile strength at break, Kg/cm ² | 160 | ASTM D 638 @ Crosshead speed 50 mm/min |
| Elongation at break, % | 150 | ASTM D638 @ Crosshead speed 50 mm/min |
| Flexural modulus, Kg/cm ² | 13000 | ASTM D 790 |
| Notched izod impact strength at 23°C, kg.com/cm | 2.5 | ASTM D 256 |
| Hardness, shore D | 66 | ASTM D 2240 |
| ESCR, hrs, F ₅₀ | 2 | ASTM D 1693 @50°C Condition B, Compression molded, 25% Igepal |
| Melting point, °C | 131 | ASTM D 2117 |
| Vicat softening point, °C | 122 | ASTM D 1525 |
| Heat deflection temperature (HDT), °C | 75 | ASTM D 648 @4.6 kg/cm ² |
| Brittleness temperature, °C | -60 | ASTM D 746 |
| % Shrinkage (2mmt) MD, % | 2.02 | TPE method |
| TD, % | 2.46 | TPE method |

Note: conversion factor for changing unit from kg/cm² to MPa is divided by 10.20

Procedures:

- 1) Material ratio of the two components:
Polypropylene (Moplen HP561R):Polyethylene (EL-Lene H5818J)
1:1

The resins were used as received.

- 2) Extrusion spinneret plates

The spinneret plates for segmented pie bi-component fiber extrusion were assembled following the sequences specified in the assembly chart for the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100, Fig. 3.3. The plates were preheated in the oven at 250 °C for not less than 3 hours before assemble into the spinneret head. The plates were assembled consecutively into the spinneret head immediately after preheated, as show in Fig. 3.4.

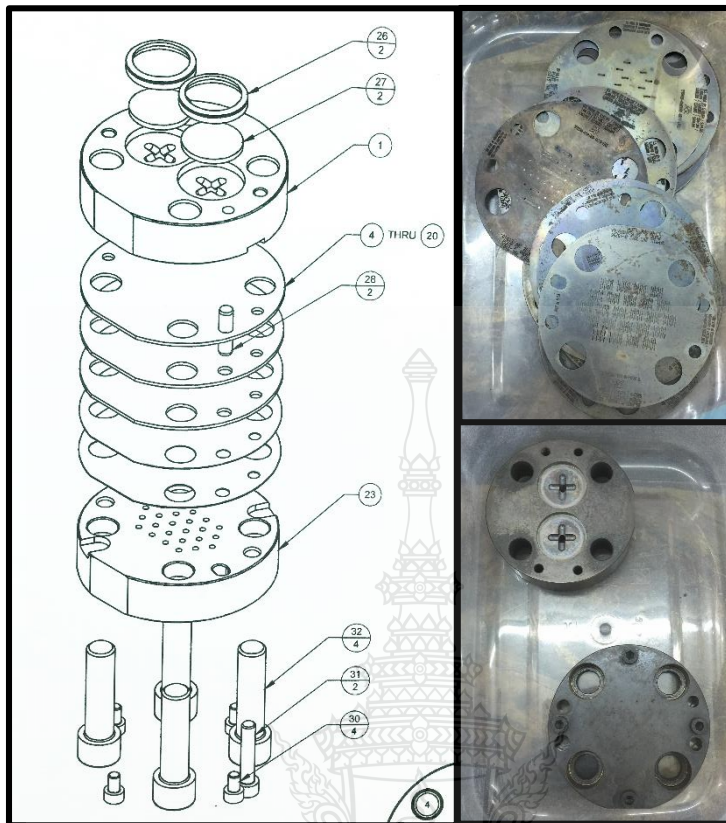


Figure 3.3 The spinneret plates of The Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 in the experiments

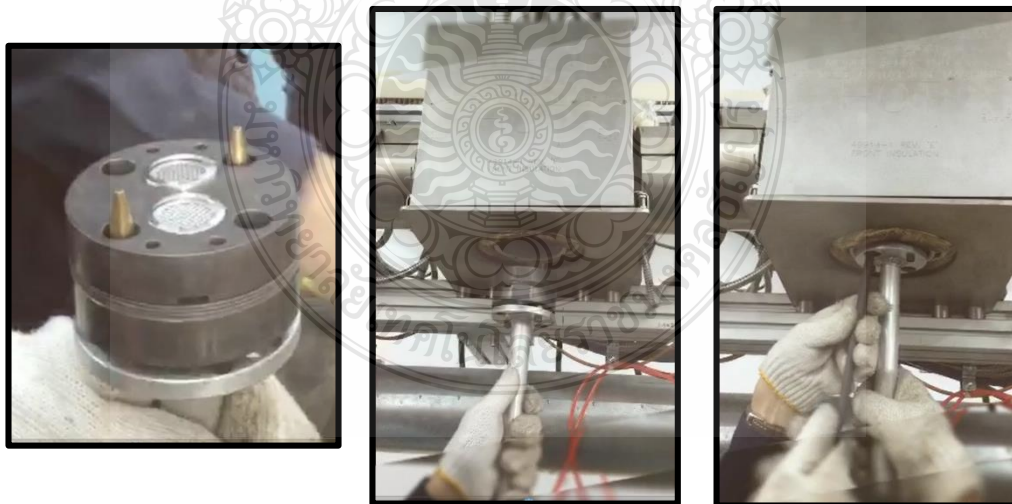


Figure 3.4 The spinneret plates assembly for the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 in the experiments

3) Bi-component Extruder control system

The tentative system conditions were set according to the recommended conditions for the two materials compatible with the machine capability. The system was equipped with the central process control, therefore, the setting mechanism were all at the control cabinet as shown in Fig. 3.5.

The conditions were appointed at the central controlling panel on the controlling cabinet. The conditions were set according to the specification of the machine and the recommendation of the experimental materials (MoPlen HP561R and EL-Lene H5818J). The machine automatically conditioned itself according to the setting, which took approximately 30 min. for the machine to reach the appointed conditions and become stable at approximately one hour after the setting. The system were ready to operate when it reach the stable stage.

4) Polymer hoppers

There were two polymer hoppers in the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100, each hopper for each component material. According to Fig. 3.1, the hopper A (hopper left) for one material and hopper B (hopper right) for another material, both hoppers were cleaned before use.

The polymer chips were not to be poured into the hoppers before the machine reached the stable stage. As soon as the system reached at the stationary condition, the hopper A was filled with the polypropylene (PP) chips and the hopper B was filled with the polyethylene (PE) chips (Fig. 3.6), both chips were used as received.

5) Segmented pie bi-component fiber extrusion

The polymer chips from the both hoppers were melted and became homogeneous liquidised polymers, then flew through polymer pipes into the metering pumps inside the spin-head before pass through the pack of the assembled spinneret plates. (Fig. 3.1) Eventually the melted polymers were extruded as bi-component strands of fibers and fell through surrounding ambiance to cool down. The extruded fibers were segmented pie bi-component fibers.

The experiment I was to be a trial of the polymer chips and the system, so the winding mechanism was not applied which means that the extruded fibers was to be free fall without any tension.

The first few minutes of the extrusion was pre-run period. The extruded fibers from the pre-run period were discarded. The fibers to be subjected to analyse were the latter part which come out after the flow became stable and consistence.



Figure 3.5 The condition control panel of The Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 in the experiments



Figure 3.6 The polymer hopper of The Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100

Analysis:

- 1) Material applicability: observing the production process throughout the experiment.
- 2) Suitability of the system condition: observing stability of the control panel, the flow of the materials, the continuation of the extruded filaments, etc.
- 3) Implications of the system condition: observing overall filament extrusion.
- 4) Shapes and geometry of the filament: observing the extruded filaments under light compound microscope. The light compound microscope used for the experiment was the Carl ZEISS Primo Star (Fig. 3.7), which equipped with digital camera to capture the images and a computer screen for display real time images.
- 5) Implication of the experiment I (PP/PE free fall): analysing the information received from this experiment for the following experiments.

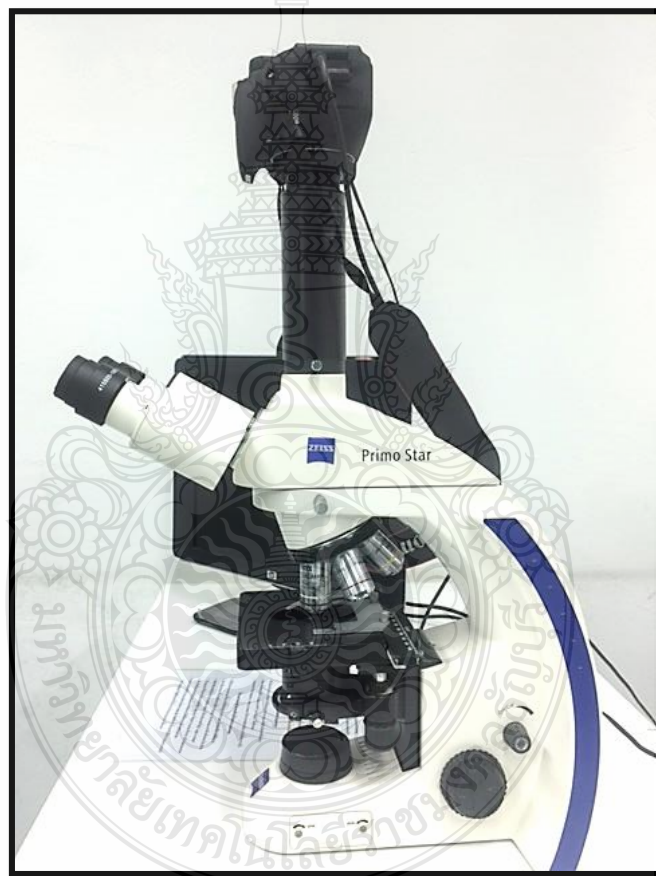


Figure 3.7 The light compound microscope: Carl ZEISS Primo Star

3.3 Segmented pie bi-component fiber spinning: Experiment II (PP/PE 300m/min)

The experiment II (PP/PE 300 m/min)

The experiment II was for an initial step for producing segmented pie bi-component fibers using polypropylene and Polyethylene at 300 m/min winding speed.

The experiment II was designed to follow the implication of the experiment I. The experiment II used the same materials as the experiment I: polypropylene (MoPlen HP561R) and polyethylene (EL-Lene H5818J) with materials ratio at 1:1, using the bi-

component extruder system in the laboratory, but altered the process by applying the winding mechanism at the end of the filament extrusion. The applied winding speed for the initial tentative step was 300 metre/min, which considerably slow.

Objectives of the experiment II (PP/PE 300 m/min)

- 1) To produce segmented pie bi-component filament with winding speed at 300 m/min.
- 2) To study the implications of the winding speed at 300 m/min in producing the bi-component filaments.
- 3) To study the bi-component fiber with winding mechanism at 300 m/min.
- 4) To study shapes of the produced segmented pie bi-component fibers using light compound microscope

Instruments:

- 1) Bi-component Extruder System

The fiber spinning experiments in this research were carried out using A Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 by The Hills Inc. which consists of 2 single screw extruders connected to a spinneret block. (figure. 3.2)

Materials:

The materials used in the experiment II were polypropylene (MoPlen HP561R) and polyethylene (EL-Lene H5818J) with materials ratio at 1:1. The information and specifications of the two materials were the same as in the experiment I.

Procedures:

- 1) Material ratio of the two components:
Polypropylene (Moplen HP561R):Polyethylene (EL-Lene H5818J)
1:1

The resins were used as received.

- 2) Extrusion spinneret plates

The spinneret plates for segmented pie bi-component fiber extrusion were assembled following the sequences specified in the assembly chart for the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100, Fig. 3.3. The plates were preheated in the oven at 250 °C for not less than 3 hours before assemble into the spinneret head. The plates were assembled consecutively into the spinneret head immediately after preheated, as show in Fig. 3.4.

- 3) Bi-component Extruder control system

The system conditions were set in same manner as the experiment I.

- 4) Polymer hoppers

The two hoppers of the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 were filled in same manner as the experiment I.

- 5) Segmented pie bi-component fiber extrusion

The polymer chips from the both hoppers were melted and became homogeneous liquidised polymers, then flew through polymer pipes into the metering pumps inside the spin-head before pass through the pack of the assembled spinneret plates. Eventually the melted polymers were extruded as bi-component strands of fibers and fell through surrounding ambiance to cool down.

The experiment II differed from the experiment I at the latter part of the process where the extruded bi-component filaments were subjected to winding tension at 300 metre/min. into round paper bobbin, as show in Fig. 3.8.



Figure 3.8 The winding unit for reeling filaments into bobbins

Analysis:

- 1) Material applicability: observing the production process throughout the experiment.
- 2) Suitability of the system condition: observing stability of the control panel, the flow of the materials, the continuation of the extruded filaments, etc.
- 3) Implications of the system condition: observing overall filament extrusion.
- 4) Shapes and geometry of the filament: observing the extruded filaments under light compound microscope (Fig. 3.4).
- 5) Implication of the experiment II (PP/ PE 300 m/ min): analysing the information received from this experiment for the following experiments.

3.4 Segmented pie bi-component fiber spinning: Experiment III (PP/PE 500m/min)

The experiment III (PP/PE 500 m/min)

The experiment III was for a further step for producing segmented pie bi-component fibers using polypropylene and Polyethylene at 500 m/min winding speed.

The experiment III was designed to follow the implication of the experiment II. The experiment III used the same materials as the experiment I and II: polypropylene (MoPlen HP561R) and polyethylene (EL-Lene H5818J) with materials ratio at 1:1, using the bi-component extruder system in the laboratory, but altered the process by applying the winding mechanism at the end of the filament extrusion. The applied winding speed for the initial tentative step was 500 metre/min, which considerably medium speed.

Objectives of the experiment III (PP/PE 500 m/min)

- 1) To produce segmented pie bi-component filament with winding speed at 500 m/min.
- 2) To study the implications of the winding speed at 500 m/ min in producing the bi-component filaments.
- 3) To study the bi-component fiber with winding mechanism at 500 m/min.

4) To study shapes of the produced segmented pie bi-component fibers using light compound microscope

Instruments:

1) Bi-component Extruder System

The fiber spinning experiments in this research were carried out using A Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS- 100 by The Hills Inc. which consists of 2 single screw extruders connected to a spinneret block. (figure. 3.2)

Materials:

The materials used in the experiment III were polypropylene (MoPlen HP561R) and polyethylene (EL-Lene H5818J) with materials ratio at 1:1. The information and specifications of the two materials were the same as in the experiment I and II.

Procedures:

1) Material ratio of the two components: Polypropylene (Moplen HP561R): Polyethylene (EL-Lene H5818J)

1:1

The resins were used as received.

2) Extrusion spinneret plates

The spinneret plates for segmented pie bi-component fiber extrusion were assembled following the sequences specified in the assembly chart for the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100, Fig. 3.3. The plates were preheated in the oven at 250 °C for not less than 3 hours before assemble into the spinneret head. The plates were assembled consecutively into the spinneret head immediately after preheated, as show in Fig. 3.4.

3) Bi-component Extruder control system

The system conditions were set in same manner as the experiment I and II.

4) Polymer hoppers

The two hoppers of the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 were filled in same manner as the experiment I and II.

5) Segmented pie bi-component fiber extrusion

The polymer chips from the both hoppers were melted and became homogeneous liquidised polymers, then flew through polymer pipes into the metering pumps inside the spin-head before pass through the pack of the assembled spinneret plates. Eventually the melted polymers were extruded as bi-component strands of fibers and fell through surrounding ambiance to cool down.

The experiment III differed from the experiment I and II at the latter part of the process where the extruded bi-component filaments were subjected to winding tension at 500 metre/min. into round paper bobbin, as show in Fig. 3.8.

Analysis:

1) Material applicability: observing the production process throughout the experiment.

2) Suitability of the system condition: observing stability of the control panel, the flow of the materials, the continuation of the extruded filaments, etc.

3) Implications of the system condition: observing overall filament extrusion.

4) Shapes and geometry of the filament: observing the extruded filaments under light compound microscope (Fig. 3.4).

5) Implication of the experiment III (PP/PE 500 m/min): analysing the information received from this experiment for the following experiments.

3.5 Segmented pie bi-component fiber spinning: Experiment IV (PP/PE 700m/min)

The experiment IV (PP/PE 700 m/min)

The experiment IV was for a further step for producing segmented pie bi-component fibers using polypropylene and Polyethylene at 700 m/min winding speed.

The experiment IV was designed to follow the implication of the experiment III. The experiment IV used the same materials as the experiment I, II, and III: polypropylene (MoPlen HP561R) and polyethylene (EL-Lene H5818J) with materials ratio at 1:1, using the bi-component extruder system in the laboratory, but altered the process by applying the winding mechanism at the end of the filament extrusion. The applied winding speed for the initial tentative step was 700 metre/ min, which considerably medium speed.

Objectives of the experiment IV (PP/PE 700 m/min)

- 1) To produce segmented pie bi-component filament with winding speed at 700 m/min.
- 2) To study the implications of the winding speed at 700 m/min in producing the bi-component filaments.
- 3) To study the bi-component fiber with winding mechanism at 700 m/min.
- 4) To study shapes of the produced segmented pie bi-component fibers using light compound microscope

Instruments:

- 1) Bi-component Extruder System

The fiber spinning experiments in this research were carried out using A Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS- 100 by The Hills Inc. which consists of 2 single screw extruders connected to a spinneret block. (figure. 3.2)

Materials:

The materials used in the experiment IV were polypropylene (MoPlen HP561R) and polyethylene (EL-Lene H5818J) with materials ratio at 1:1. The information and specifications of the two materials were the same as in the experiment I, II, and III.

Procedures:

- 1) Material ratio of the two components:
Polypropylene (Moplen HP561R):Polyethylene (EL-Lene H5818J)
1:1
The resins were used as received.

- 2) Extrusion spinneret plates

The spinneret plates for segmented pie bi-component fiber extrusion were assembled following the sequences specified in the assembly chart for the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100, Fig. 3.3. The plates were preheated in the oven at 250 °C for not less than 3 hours before assemble into the spinneret head. The plates were assembled consecutively into the spinneret head immediately after preheated, as show in Fig. 3.4.

- 3) Bi-component Extruder control system

The system conditions were set in same manner as the experiment I, II, and III.

4) Polymer hoppers

The two hoppers of the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 were filled in same manner as the experiment I, II, and III.

5) Segmented pie bi-component fiber extrusion

The polymer chips from the both hoppers were melted and became homogeneous liquidised polymers, then flew through polymer pipes into the metering pumps inside the spin-head before pass through the pack of the assembled spinneret plates. Eventually the melted polymers were extruded as bi-component strands of fibers and fell through surrounding ambiance to cool down.

The experiment IV differed from the experiment I, II, and III at the latter part of the process where the extruded bi-component filaments were subjected to winding tension at 700 metre/min. into round paper bobbin, as show in Fig. 3.8.

Analysis:

1) Material applicability: observing the production process throughout the experiment.

2) Suitability of the system condition: observing stability of the control panel, the flow of the materials, the continuation of the extruded filaments, etc.

3) Implications of the system condition: observing overall filament extrusion.

4) Shapes and geometry of the filament: observing the extruded filaments under light compound microscope (Fig. 3.4).

5) Implication of the experiment IV (PP/PE 700 m/min): analysing the information received from this experiment for the following experiments.

3.6 Segmented pie bi-component fiber spinning: Experiment V (PP/PE 1% TiO₂ Free fall)

The experiment V (PP/PE 1% TiO₂ Free fall)

The experiment V was for a further step for producing segmented pie bi-component fibers using polypropylene and Polyethylene with 1.0% TiO₂ without winding mechanism at the end of the fiber spinning.

The experiment V was designed to follow the implication of previous experiments. The filaments from the earlier experiments using polypropylene and polyethylene were considerable agreeable as the extrusion processes were steady and manageable. Besides, the extruded filaments from the first four experiments were smooth, unbroken, uncrease, and no significant fiber breakage and fatigue.

The experiment V was designed to add special feature to the fiber, particularly for healthcare or medical textile approach. The first attempt for the special feature supplementation was the ability to anti-bacteria using common additive. The intention of supplementing additive into the extruded filaments was for anti- bacteria ability of outside-body healthcare textiles.

There are several materials could be potential additives to textile materials for health and medical care. Metals, such as silver, copper, and titanium, are among the metal elements that has high effective in biocidal activity. However, the use of such metals in textiles has faced several tough challenges, for example, silver is one of the most expensive metals while copper is considerably cheap but high corrosive. The development of metal use in health and medical care has been in target for funding and supports in the field of material sciences in recent years, because it could be easily applied

by coating, chelating, and impregnating into materials or on their surfaces. The metal/polymer composites has taken a certain percentage usages of hospital acquired infection (HAIs) materials and gradually growing its share in the market.[9]

Therefore, cheap and effective antimicrobial polymers have been in high demand as to deal with the HAIs. The metal that is common, cheap, and harmless to human is probably the hidden treasure for the health and medical care products.

Titanium dioxide (TiO_2) is one of the most important metal oxides which universally subjected to investigate and research nowadays. Titanium dioxide can be apply to variety of technical fields. Titanium dioxide is used in many material applications such as heterogeneous catalysis, photocatalyst, in solar cells, optical coating, in ceramics, white pigment, in paints and cosmetic products, corrosion-protective coating, etc. One of the pursued applied research on titania is its capability in degrading organic molecule. It is a semiconductor which is created upon irradiation with sunlight that can lead to absorb and decomposition of organic molecule. [28]

The experiment V used the same base materials as the experiment I, II, and III: polypropylene (MoPlen HP561R) and polyethylene (EL-Lene H5818J) with materials ratio at 1:1. The additive used in the experiment was 1.0% of TiO_2 . The experiment carried out using the bi-component extruder system in the laboratory,

Objectives of the experiment V (PP/PE 1% TiO_2 free fall)

- 1) To produce segmented pie bi-component fibers using polypropylene and polyethylene as base polymer material and supplement with 1% TiO_2
- 2) To study the applicability of the two selected materials with the supplement of 1% TiO_2 for segmented pie bi-component fiber extrusion
- 3) To study the system condition for producing segmented pie bi-component fiber using the selected base material and the additive
- 4) To study the implications of the setting condition on the produced fibers.
- 5) To study the bi-component fiber without applying winding speed (free fall)
- 6) To study shapes of the produced segmented pie bi-component fibers using light compound microscope

Instruments:

1) Bi-component Extruder System

The fiber spinning experiments in this research were carried out using A Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS- 100 by The Hills Inc. which consists of 2 single screw extruders connected to a spinneret block. (figure. 3.2)

Materials:

The base materials used in the experiment IV were polypropylene (MoPlen HP561R) and polyethylene (EL-Lene H5818J), same as in the experiental I - IV. The information and specifications of the two materials were as in the experiment I - IV.

The additive material for the intention of enhancing the antimicrobial in this experiment was Titanium dioxide. The 20% of Titanium dioxide Polypropylene master batch and 20% Titanium dioxide Polyethylene master batch were used as functional supplement in the components.

The TiO_2 used in the experiment was the photocatalytic standard commercial type of P25, comprising mixed rutile and anatase phases. It has the average primary particle size of 21 ± 5 nm, specific surface area of 50 ± 10 m^2/g , less than 0.3 wt% Al_2O_3 , and less than 0.2 wt.% SiO_2 .

Procedures:

1) The preparation of the materials:

Preparation of 200 gram of Polypropylene with 1.0% of TiO₂

(1) MoPlen HP561R 190 gram

(2) Titanium dioxide Polypropylene master batch 10 gram

(3) combine and blend the polypropylene chips and Titanium dioxide

Polypropylene master batch chips

Preparation of 200 gram of Polyethylene with 1.0% of TiO₂

(1) EL-Lene H5818J 190 gram

(2) Titanium dioxide Polyethylene master batch 10 gram

(3) combine and blend the polyethylene chips and Titanium dioxide

Polyethylene master batch chips

Material ratio:

Polypropylene, 1.0% TiO₂: Polyethylene, 1.0% TiO₂

1:1

The resins were used as received.

2) Extrusion spinneret plates

The spinneret plates for segmented pie bi-component fiber extrusion were assembled following the sequences specified in the assembly chart for the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100, Fig. 3.3. The plates were preheated in the oven at 250 °C for not less than 3 hours before assemble into the spinneret head. The plates were assembled consecutively into the spinneret head immediately after preheated, as show in Fig. 3.4.

3) Bi-component Extruder control system

The system conditions were set in same manner as the experiment I - IV.

4) Polymer hoppers

The two hoppers of the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 were filled in same manner as the experiment I, II, and III.

The polymer chips were not to be poured into the hoppers before the machine reached the stable stage. As soon as the system reached at the stationary condition, the hopper A was filled with the mixture of 1.0% TiO₂ polypropylene chips and the hopper B was filled with the mixture of 1.0% TiO₂ polyethylene (PE) chips (Fig. 3.6).

5) Segmented pie bi-component fiber extrusion

The polymer chips from the both hoppers were melted and became homogeneous liquidised polymers, then flew through polymer pipes into the metering pumps inside the spin-head before pass through the pack of the assembled spinneret plates. Eventually the melted polymers were extruded as bi-component strands of fibers and free fell through surrounding ambience to cool down.

Analysis:

1) Material applicability: observing the production process throughout the experiment.

2) Suitability of the system condition: observing stability of the control panel, the flow of the materials, the continuation of the extruded filaments, etc.\

3) Implications of the system condition: observing overall filament extrusion.

4) Shapes and geometry of the filament: observing the extruded filaments under light compound microscope (Fig. 3.4).

5) Implication of the experiment V (PP/PE 1% TiO₂ free fall): analysing the information received from this experiment for the following experiments.

3.7 Segmented pie bi-component fiber spinning: Experiment VI (PP/PE 1% TiO₂ 300 m/min)

The experiment VI (PP/PE 1% TiO₂ 300 m/min)

The experiment VI was for a further step for producing segmented pie bi-component fibers using polypropylene and Polyethylene with 1.0% TiO₂ at 300 m/min winding speed).

The experiment VI was designed to follow the implication of previous experiments. The filaments from the earlier experiments were considerable agreeable as the extrusion processes were steady and manageable. Besides, the extruded filaments from the first four experiments were smooth, unbroken, uncrease, and no significant fiber breakage and fatigue.

The experiment VI was designed to add special feature of antimicrobial using Titanium dioxide supplement as mentioned in the experiment V.

The experiment VI used the same base materials as the experiment V: polypropylene (MoPlen HP561R), polyethylene (EL-Lene H5818J), and same additives: 20% of Titanium dioxide Polypropylene master batch and 20% Titanium dioxide Polyethylene master batch. The component material ratio for this experiment was 1:1. The experiment carried out using the bi-component extruder system in the laboratory.

Objectives of the experiment VI (PP/PE 1% TiO₂ 300 m/min)

1) To produce segmented pie bi-component fibers using polypropylene and polyethylene as base polymer material and supplement with 1% TiO₂ at 300 m/min applied winding speed

2) To study the implications of the winding speed at 300 m/min in producing the bi-component filaments.

3) To study the bi-component fiber with winding mechanism at 300 m/min.

4) To study shapes of the produced segmented pie bi-component fibers using light compound microscope

Instruments:

1) Bi-component Extruder System

The fiber spinning experiments in this research were carried out using A Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS- 100 by The Hills Inc. which consists of 2 single screw extruders connected to a spinneret block. (figure. 3.2)

Materials:

The base materials used in the experiment V were polypropylene (MoPlen HP561R) and polyethylene (EL-Lene H5818J), same as in the experiental I - V. The information and specifications of the two materials were as in the experiment I - V.

The additive material for the intention of enhancing the antimicrobial in this experiment was Titanium dioxide. The 20% of Titanium dioxide Polypropylene master batch and 20% Titanium dioxide Polyethylene master batch were used as functional supplement in the components.

The TiO₂ used in the experiment was the same as in earlier experiment.

Procedures:

1) The preparation of the materials:

Preparation of 250 gram of Polypropylene with 1.0% of TiO₂

- | | |
|---|------------|
| (1) MoPlen HP561R | 237.5 gram |
| (2) Titanium dioxide Polypropylene master batch | 12.5 gram |
| (3) combine and blend the polypropylene chips and Titanium dioxide Polypropylene master batch chips | |
| Preparation of 250 gram of Polyethylene with 1.0% of TiO ₂ | |
| (1) EL-Lene H5818J | 237.5 gram |
| (2) Titanium dioxide Polyethylene master batch | 12.5 gram |
| (3) combine and blend the polyethylene chips and Titanium dioxide Polyethylene master batch chips | |
| Material ratio: | |
| Polypropylene, 1.0% TiO ₂ : Polyethylene, 1.0% TiO ₂ | |
| 1:1 | |
| The resins were used as received. | |

2) Extrusion spinneret plates

The spinneret plates for segmented pie bi-component fiber extrusion were assembled following the sequences specified in the assembly chart for the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100, Fig. 3.3. The plates were preheated in the oven at 250 °C for not less than 3 hours before assemble into the spinneret head. The plates were assembled consecutively into the spinneret head immediately after preheated, as show in Fig. 3.4.

3) Bi-component Extruder control system

The system conditions were set in same manner as the experiment I - V.

4) Polymer hoppers

The two hoppers of the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 were filled in same manner as the experiment I - V.

The polymer chips were not to be poured into the hoppers before the machine reached the stable stage. As soon as the system reached at the stationary condition, the hopper A was filled with the mixture of 1.0% TiO₂ polypropylene chips and the hopper B was filled with the mixture of 1.0% TiO₂ polyethylene (PE) chips in the same manner as in experiment V. (Fig. 3.6)

5) Segmented pie bi-component fiber extrusion

The polymer chips from the both hoppers were melted and became homogeneous liquidised polymers, then flew through polymer pipes into the metering pumps inside the spin-head before pass through the pack of the assembled spinneret plates. Eventually the melted polymers were extruded as bi-component strands of fibers and fell through surrounding ambiance to cool down.

The experiment VI differed from the experiment V at the latter part of the process where the extruded bi-component filaments were subjected to winding tension at 300 metre/min. into round paper bobbin, as show in Fig. 3.8.

Analysis:

- 1) Material applicability: observing the production process throughout the experiment.
- 2) Suitability of the system condition: observing stability of the control panel, the flow of the materials, the continuation of the extruded filaments, etc.
- 3) Implications of the system condition: observing overall filament extrusion.
- 4) Shapes and geometry of the filament: observing the extruded filaments under light compound microscope (Fig. 3.4).

5) Implication of the experiment VI (PP/PE 1% TiO₂ 300 m/min): analysing the information received from this experiment for the following experiments.

3.8 Segmented pie bi-component fiber spinning: Experiment VII (PP/PE 1% TiO₂ 500 m/min)

The experiment VII (PP/PE 1% TiO₂ 500 m/min)

The experiment VII was for a further step for producing segmented pie bi-component fibers using polypropylene and Polyethylene with 1.0% TiO₂ at 500 m/min winding speed).

The experiment VII was designed to follow the implication of previous experiments. The filaments from the earlier experiments were considerable agreeable as the extrusion processes were steady and manageable. Besides, the extruded filaments from the first four experiments were smooth, unbroken, uncrease, and no significant fiber breakage and fatigue.

The experiment VII was designed to add special feature of antimicrobial using Titanium dioxide supplement as mentioned in the experiment V - VI.

The experiment VII used the same base materials as the experiment V - VI: polypropylene (MoPlen HP561R), polyethylene (EL-Lene H5818J), and same additives: 20% of Titanium dioxide Polypropylene master batch and 20% Titanium dioxide Polyethylene master batch. The component material ratio for this experiment was 1:1. The experiment carried out using the bi-component extruder system in the laboratory,

Objectives of the experiment VII (PP/PE 1% TiO₂ 500 m/min)

1) To produce segmented pie bi-component fibers using polypropylene and polyethylene as base polymer material and supplement with 1% TiO₂ at 500 m/min applied winding speed

2) To study the implications of the winding speed at 500 m/min in producing the bi-component filaments.

3) To study the bi-component fiber with winding mechanism at 500 m/min.

4) To study shapes of the produced segmented pie bi-component fibers using light compound microscope

Instruments:

1) Bi-component Extruder System

The fiber spinning experiments in this research were carried out using A Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 by The Hills Inc. which consists of 2 single screw extruders connected to a spinneret block. (figure. 3.2)

Materials:

The base materials used in the experiment VII were polypropylene (MoPlen HP561R) and polyethylene (EL-Lene H5818J), same as in the experiment I - VI. The information and specifications of the two materials were as in the experiment I - VI.

The additive material for the intention of enhancing the antimicrobial in this experiment was Titanium dioxide. The 20% of Titanium dioxide Polypropylene master batch and 20% Titanium dioxide Polyethylene master batch were used as functional supplement in the components.

The TiO₂ used in the experiment was the same as in earlier experiment.

Procedures:

1) The preparation of the materials:

Preparation of 250 gram of Polypropylene with 1.0% of TiO₂

- | | |
|--|------------|
| (1) MoPlen HP561R | 237.5 gram |
| (2) Titanium dioxide Polypropylene master batch | 12.5 gram |
| (3) combine and blend the polypropylene chips and Titanium dioxide | |
- Polypropylene master batch chips
- Preparation of 250 gram of Polyethylene with 1.0% of TiO₂
- | | |
|---|------------|
| (1) EL-Lene H5818J | 237.5 gram |
| (2) Titanium dioxide Polyethylene master batch | 12.5 gram |
| (3) combine and blend the polyethylene chips and Titanium dioxide | |
- Polyethylene master batch chips
- Material ratio:
- Polypropylene, 1.0% TiO₂: Polyethylene, 1.0% TiO₂
- 1:1
- The resins were used as received.

2) Extrusion spinneret plates

The spinneret plates for segmented pie bi-component fiber extrusion were assembled following the sequences specified in the assembly chart for the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100, Fig. 3.3. The plates were preheated in the oven at 250 °C for not less than 3 hours before assemble into the spinneret head. The plates were assembled consecutively into the spinneret head immediately after preheated, as show in Fig. 3.4.

3) Bi-component Extruder control system

The system conditions were set in same manner as the experiment I - VI.

4) Polymer hoppers

The two hoppers of the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 were filled in same manner as the experiment I - VI.

The polymer chips were not to be poured into the hoppers before the machine reached the stable stage. As soon as the system reached at the stationary condition, the hopper A was filled with the mixture of 1.0% TiO₂ polypropylene chips and the hopper B was filled with the mixture of 1.0% TiO₂ polyethylene (PE) chips in the same manner as in experiment V - VI. (Fig. 3.6)

5) Segmented pie bi-component fiber extrusion

The polymer chips from the both hoppers were melted and became homogeneous liquidised polymers, then flew through polymer pipes into the metering pumps inside the spin-head before pass through the pack of the assembled spinneret plates. Eventually the melted polymers were extruded as bi-component strands of fibers and fell through surrounding ambiance to cool down.

The experiment VII differed from the experiment VI at the latter part of the process where the extruded bi-component filaments were subjected to winding tension at 500 metre/min. into round paper bobbin, as show in Fig. 3.8.

Analysis:

- 1) Material applicability: observing the production process throughout the experiment.
- 2) Suitability of the system condition: observing stability of the control panel, the flow of the materials, the continuation of the extruded filaments, etc.
- 3) Implications of the system condition: observing overall filament extrusion.
- 4) Shapes and geometry of the filament: observing the extruded filaments under light compound microscope (Fig. 3.4).

5) Implication of the experiment VII (PP/PE 1% TiO₂ 500 m/min): analysing the information received from this experiment for the following experiments.

3.9 Segmented pie bi-component fiber spinning: Experiment VIII (PP/PE 1% TiO₂ 700 m/min)

The experiment VIII (PP/PE 1% TiO₂ 700 m/min)

The experiment VIII was for a further step for producing segmented pie bi-component fibers using polypropylene and Polyethylene with 1.0% TiO₂ at 700 m/min winding speed).

The experiment VIII was designed to follow the implication of previous experiments. The filaments from the earlier experiments were considerable agreeable as the extrusion processes were steady and manageable. Besides, the extruded filaments from the first four experiments were smooth, unbroken, uncrease, and no significant fiber breakage and fatigue.

The experiment VIII was designed to add special feature of antimicrobial using Titanium dioxide supplement as mentioned in the experiment V - VII.

The experiment VIII used the same base materials as the experiment V - VII: polypropylene (MoPlen HP561R), polyethylene (EL-Lene H5818J), and same additives: 20% of Titanium dioxide Polypropylene master batch and 20% Titanium dioxide Polyethylene master batch. The component material ratio for this experiment was 1:1. The experiment carried out using the bi-component extruder system in the laboratory,

Objectives of the experiment VIII (PP/PE 1% TiO₂ 700 m/min)

1) To produce segmented pie bi-component fibers using polypropylene and polyethylene as base polymer material and supplement with 1% TiO₂ at 700 m/min applied winding speed

2) To study the implications of the winding speed at 700 m/min in producing the bi-component filaments.

3) To study the bi-component fiber with winding mechanism at 700 m/min.

4) To study shapes of the produced segmented pie bi-component fibers using light compound microscope

Instruments:

1) Bi-component Extruder System

The fiber spinning experiments in this research were carried out using A Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 by The Hills Inc. which consists of 2 single screw extruders connected to a spinneret block. (figure. 3.2)

Materials:

The base materials used in the experiment VIII were polypropylene (MoPlen HP561R) and polyethylene (EL-Lene H5818J), same as in the experiental I - VII. The information and specifications of the two materials were as in the experiment I - VII.

The additive material for the intention of enhancing the antimicrobial in this experiment was Titanium dioxide. The 20% of Titanium dioxide Polypropylene master batch and 20% Titanium dioxide Polyethylene master batch were used as functional supplement in the components.

The TiO₂ used in the experiment was the same as in earlier experiment.

Procedures:

1) The preparation of the materials:

Preparation of 250 gram of Polypropylene with 1.0% of TiO₂

- | | |
|---|------------|
| (1) MoPlen HP561R | 237.5 gram |
| (2) Titanium dioxide Polypropylene master batch | 12.5 gram |
| (3) combine and blend the polypropylene chips and Titanium dioxide Polypropylene master batch chips | |
| Preparation of 250 gram of Polyethylene with 1.0% of TiO ₂ | |
| (1) EL-Lene H5818J | 237.5 gram |
| (2) Titanium dioxide Polyethylene master batch | 12.5 gram |
| (3) combine and blend the polyethylene chips and Titanium dioxide Polyethylene master batch chips | |

Material ratio:

Polypropylene, 1.0% TiO₂: Polyethylene, 1.0% TiO₂

1:1

The resins were used as received.

2) Extrusion spinneret plates

The spinneret plates for segmented pie bi-component fiber extrusion were assembled following the sequences specified in the assembly chart for the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100, Fig. 3.3. The plates were preheated in the oven at 250 °C for not less than 3 hours before assemble into the spinneret head. The plates were assembled consecutively into the spinneret head immediately after preheated, as show in Fig. 3.4.

3) Bi-component Extruder control system

The system conditions were set in same manner as the experiment I - VII.

4) Polymer hoppers

The two hoppers of the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 were filled in same manner as the experiment I - VII.

The polymer chips were not to be poured into the hoppers before the machine reached the stable stage. As soon as the system reached at the stationary condition, the hopper A was filled with the mixture of 1.0% TiO₂ polypropylene chips and the hopper B was filled with the mixture of 1.0% TiO₂ polyethylene (PE) chips in the same manner as in experiment V - VII. (Fig. 3.6)

5) Segmented pie bi-component fiber extrusion

The polymer chips from the both hoppers were melted and became homogeneous liquidised polymers, then flew through polymer pipes into the metering pumps inside the spin-head before pass through the pack of the assembled spinneret plates. Eventually the melted polymers were extruded as bi-component strands of fibers and fell through surrounding ambiance to cool down.

The experiment VIII differed from the experiment VII at the latter part of the process where the extruded bi-component filaments were subjected to winding tension at 700 metre/min. into round paper bobbin, as show in Fig. 3.8.

Analysis:

- 1) Material applicability: observing the production process throughout the experiment.
- 2) Suitability of the system condition: observing stability of the control panel, the flow of the materials, the continuation of the extruded filaments, etc.
- 3) Implications of the system condition: observing overall filament extrusion.
- 4) Shapes and geometry of the filament: observing the extruded filaments under light compound microscope (Fig. 3.4).

5) Implication of the experiment VIII (PP/PE 1% TiO₂ 700 m/min): analysing the information received from this experiment for the following experiments.

3.10 Segmented pie bi-component fiber spinning: Experiment IX (PE/Nylon 6 free fall)

The experiment IX (PE/Nylon6 free fall)

The experiment IX was for an initial tentative step for producing segmented pie bi-component fibers using polyethylene and Nylon 6.

Objectives of the experiment I (PE/Nylon 6 free fall)

- 1) To produce segmented pie bi-component fibers
- 2) To study the applicability of the two selected materials for producing segmented pie bi-component fiber
- 3) To study the system condition for producing segmented pie bi-component fiber using the two selected materials
- 4) To study the implications of the setting condition on the produced fibers.
- 5) To study the bi-component fiber without applying winding speed (free fall)
- 6) To study shapes of the produced segmented pie bi-component fibers using light compound microscope

Instruments:

- 1) Bi-component Extruder System

The fiber spinning experiments in this research were carried out using A Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 by The Hills Inc. which consists of 2 single screw extruders connected to a spinneret block. (Fig. 3.2)

Materials:

The early experiments for this research used common materials widely available such polypropylene and polyethylene. The purposes of raw material selection for this experiment was also to find the compatible polymer resins that meet the machine system capability. The resin must be able to withstand the setting spinning process conditions such as spinning speed, winding speed, extruding temperatures, and spinning heating temperatures. Then the geometry and shape of the fibers were to be examined. Besides, the detaching mechanism of each polymer segments were to be analysed.

The early attempts for the research using polypropylene and polyethylene in a variation of additives and winding speeds showed that the two materials were compatible to produce segmented pie bi-component filaments by the Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS- 100 by The Hills Inc. The filaments produced by the earlier experiments were considerable agreeable as the extrusion processes were steady and manageable. The filaments were smooth, unbroken, uncrease, and no significant fiber breakage and fatigue.

The focal point of the experiments were to produce segmented pie bi-component fiber with special feature as antimicrobial textiles by enhancing the ability to impair the microorganism when the segments in the filaments split or detach after being reeled into bobbins. The point were not as succeed as expected as the filaments were not easily split or detached into finer filaments with higher active surface area. Therefore, the pursuit of improving of result the research goal was to select new materials which expected to detach or split into finer filaments easily after apply winding mechanism.

The base materials used in the experiment IX were polyethylene (EL-Lene H5818J) which is the same as in the experiment I - VIII. The information and

specifications of EL-Lene H5818J was as in the experiment I. The base material for the other component of the filaments was polyamide (Nylon 6). Nylon 6 was selected as the second component to the experiment of segmented pie bi-component fiber because it is one of the simplest raw material for forming fibers for textile products. It has high strength, abrasive performance, and good thermal properties. The Nylon 6 used in this research was of semi-dull textile grade with the consistent viscosity of R. V. 2.6 and the melting temperature of 220 – 240 °C. The material resin were produced by Thai Toray Synthetic Co., Ltd.

Procedures:

1) Material ratio of the two components:

Polyethylene (EL-Lene H5818J): Nylon 6

1:1

The polyethylene resins were used as received.

The nylon 6 resins were dried by incubating in hot air oven at temperature 80°C for approximately 6 – 8 hours before transferred immediately after took out from the incubator into a desiccator to cool the resins down to room temperature before use. This was because Nylon 6 as received had considerably medium moisture regain which could cause hydrolytic reaction to the material. The hydrolytic reaction in Nylon 6 occurs when Nylon is melting at high level of moisture content (>500 ppm). The hydrolytic reaction hinders the extrusion flow of the material and debilitates the quality of the extruded filaments. The incubation reduced the moisture content in the material so the hydrolytic reaction during the melting process were insignificantly affected the extruded filaments.

2) Extrusion spinneret plates

The spinneret plates for segmented pie bi-component fiber extrusion were assembled following the sequences specified in the assembly chart for the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100, Fig. 3.3. The plates were preheated in the oven at 250 °C for not less than 3 hours before assemble into the spinneret head. The plates were assembled consecutively into the spinneret head immediately after preheated, as show in Fig. 3.4.

3) Bi-component Extruder control system

The system conditions were set in same manner as the experiment I - VIII.

The tentative system conditions were set according to the recommended conditions for the two materials compatible with the machine capability. The system was equipped with the central process control, therefore, the setting mechanism were all at the control cabinet.

The conditions were appointed at the central controlling panel on the controlling cabinet. The conditions were set according to the specification of the machine and the recommendation of the experimental materials. The machine automatically conditioned itself according to the setting, which took approximately 30 min. for the machine to reach the appointed conditions and become stable at approximately one hour after the setting. The system were ready to operate when it reach the stable stage.

4) Polymer hoppers

There were two polymer hoppers in the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100, each hopper for each component material. According to Fig. 3.1, the hopper A (hopper left) for one material and hopper B (hopper right) for another material, both hoppers were cleaned before use.

The polymer chips were not to be poured into the hoppers before the machine reached the stable stage. As soon as the system reached at the stationary condition, the hopper A was filled with the nylon 6 chips and the hopper B was filled with the polyethylene chips (Fig. 3.6). The polyethylene chips were used as received while nylon 6 chips were used after being oven-cured for at least 6 hours to reduce moisture content in the material until under 500 ppm.

5) Segmented pie bi-component fiber extrusion

The polymer chips from the both hoppers were melted and became homogeneous liquidised polymers, then flew through polymer pipes into the metering pumps inside the spin-head before pass through the pack of the assembled spinneret plates. (Fig. 3.1) Eventually the melted polymers were extruded as bi-component strands of fibers and fell through surrounding ambiance to cool down. The extruded fibers were segmented pie bi-component fibers.

The experiment IX was to be a trial of the polymer chips and the system, so the winding mechanism was not applied which means that the extruded fibers was to be free fall without any tension.

The first few minutes of the extrusion was pre-run period. The extruded fibers from the pre-run period were discarded. The fibers to be subjected to analyse were the latter part which come out after the flow became stable and consistence.

Analysis:

1) Material applicability: observing the production process throughout the experiment.

2) Suitability of the system condition: observing stability of the control panel, the flow of the materials, the continuation of the extruded filaments, etc.

3) Implications of the system condition: observing overall filament extrusion.

4) Shapes and geometry of the filament: observing the extruded filaments under light compound microscope. The light compound microscope used for the experiment was the Carl ZEISS Primo Star (Fig. 3.7), which equipped with digital camera to capture the images and a computer screen for display real time images.

5) Implication of the experiment IX (PE/Nylon 6 free fall): analysing the information received from this experiment for the following experiments.

3.11 Segmented pie bi-component fiber spinning: Experiment X (PE/ Nylon 300m/min)

The experiment X (PE/Nylon6 300m/min)

The experiment X was for a further step for producing segmented pie bi-component fibers using polyethylene and Nylon 6.

The experiment X was designed to follow the implication of the experiment IX. The experiment X used the same materials as the experiment IX: polyethylene (EL-Lene H5818J): nylon 6 with materials ratio at 1:1, using the bi-component extruder system in the laboratory, but altered the process by applying the winding mechanism at the end of the filament extrusion. The applied winding speed for the tentative step was 300 metre/min, which considerably slow.

Objectives of the experiment X (PE/Nylon6 300 m/min)

1) To produce segmented pie bi-component filament with winding speed at 300 m/min.

2) To study the implications of the winding speed at 300 m/min in producing the bi-component filaments.

3) To study the bi-component fiber with winding mechanism at 300 m/min.

4) To study shapes of the produced segmented pie bi-component fibers using light compound microscope

Instruments:

1) Bi-component Extruder System

The fiber spinning experiments in this research were carried out using A Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 by The Hills Inc. which consists of 2 single screw extruders connected to a spinneret block. (figure. 3.2)

Materials:

The materials used in the experiment X were polyethylene (EL-Lene H5818J) and nylon 6 with materials ratio at 1:1. The information and specifications of the two materials were specified in the experiment IX.

Procedures:

1) Material ratio of the two components:

Polyethylene (EL-Lene H5818J):Nylon 6

1:1

The polyethylene resins were used as received.

The nylon 6 resins were dried by incubating in hot air oven at temperature 80°C for approximately 6 – 8 hours before transferred immediately after took out from the incubator into a desiccator to cool the resins down to room temperature before use, the same manner as in the experiment IX.

2) Extrusion spinneret plates

The spinneret plates for segmented pie bi-component fiber extrusion were assembled following the sequences specified in the assembly chart for the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100, Fig. 3.3. The plates were preheated in the oven at 250 °C for not less than 3 hours before assemble into the spinneret head. The plates were assembled consecutively into the spinneret head immediately after preheated, as show in Fig. 3.4.

3) Bi-component Extruder control system

The system conditions were set in same manner as the experiment IX.

4) Polymer hoppers

The two hoppers of the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 were filled in same manner as the experiment X.

5) Segmented pie bi-component fiber extrusion

The polymer chips from the both hoppers were melted and became homogeneous liquidised polymers, then flew through polymer pipes into the metering pumps inside the spin-head before pass through the pack of the assembled spinneret plates. Eventually the melted polymers were extruded as bi-component strands of fibers and fell through surrounding ambiance to cool down.

The experiment X differed from the experiment IX at the latter part of the process where the extruded bi-component filaments were subjected to winding tension at 300 metre/min. into round paper bobbin.

Analysis:

1) Material applicability: observing the production process throughout the experiment.

- 2) Suitability of the system condition: observing stability of the control panel, the flow of the materials, the continuation of the extruded filaments, etc.
- 3) Implications of the system condition: observing overall filament extrusion.
- 4) Shapes and geometry of the filament: observing the extruded filaments under light compound microscope (Fig. 3.4).
- 5) Implication of the experiment X (PE/Nylon 6 300 m/min): analysing the information received from this experiment for the following experiments.

3.12 Segmented pie bi-component fiber spinning: Experiment XI (PE/ Nylon6 500m/min)

The experiment X (PE/Nylon6 300m/min)

The experiment X was for a further step for producing segmented pie bi-component fibers using polyethylene and Nylon 6.

The experiment XI was designed to follow the implication of the experiment X. The experiment XI used the same materials as the experiment IX and X: polyethylene (EL-Lene H5818J) and Nylon 6 with materials ratio at 1:1, using the bi-component extruder system in the laboratory, but altered the process by applying the winding mechanism at the end of the filament extrusion. The applied winding speed for the initial tentative step was 500 metre/min, which considerably medium speed.

Objectives of the experiment XI (PE/Nylon6 500 m/min)

- 1) To produce segmented pie bi-component filament with winding speed at 500 m/min.
- 2) To study the implications of the winding speed at 500 m/min in producing the bi-component filaments.
- 3) To study the bi-component fiber with winding mechanism at 500 m/min.
- 4) To study shapes of the produced segmented pie bi-component fibers using light compound microscope

Instruments:

- 1) Bi-component Extruder System

The fiber spinning experiments in this research were carried out using A Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS- 100 by The Hills Inc. which consists of 2 single screw extruders connected to a spinneret block. (figure. 3.2)

Materials:

The materials used in the experiment XI were polyethylene (EL-Lene H5818J) and Nylon 6 with materials ratio at 1:1. The information and specifications of the two materials were the same as in the experiment IX and X.

Procedures:

- 1) Material ratio of the two components:
Polyethylene (EL-Lene H5818J):Nylon 6
1:1

The polyethylene resins were used as received.

The nylon 6 resins were dried by incubating in hot air oven at temperature 80°C for approximately 6 – 8 hours before transferred immediately after took out from the incubator into a desiccator to cool the resins down to room temperature before use, the same manner as in the experiment IX and X.

- 2) Extrusion spinneret plates

The spinneret plates for segmented pie bi-component fiber extrusion were assembled following the sequences specified in the assembly chart for the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100, Fig. 3.3. The plates were preheated in the oven at 250 °C for not less than 3 hours before assemble into the spinneret head. The plates were assembled consecutively into the spinneret head immediately after preheated, as show in Fig. 3.4.

3) Bi-component Extruder control system

The system conditions were set in same manner as the experiment IX and X.

4) Polymer hoppers

The two hoppers of the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 were filled in same manner as the experiment IX and X.

5) Segmented pie bi-component fiber extrusion

The polymer chips from the both hoppers were melted and became homogeneous liquidised polymers, then flew through polymer pipes into the metering pumps inside the spin-head before pass through the pack of the assembled spinneret plates. Eventually the melted polymers were extruded as bi-component strands of fibers and fell through surrounding ambiance to cool down.

The experiment XI differed from the experiment IX and X at the latter part of the process where the extruded bi-component filaments were subjected to winding tension at 500 metre/min. into round paper bobbin.

Analysis:

1) Material applicability: observing the production process throughout the experiment.

2) Suitability of the system condition: observing stability of the control panel, the flow of the materials, the continuation of the extruded filaments, etc.

3) Implications of the system condition: observing overall filament extrusion.

4) Shapes and geometry of the filament: observing the extruded filaments under light compound microscope (Fig. 3.4).

5) Implication of the experiment XI (PE/Nylon 6 500 m/min): analysing the information received from this experiment for the following experiments.

3.13 Segmented pie bi-component fiber spinning: Experiment XII (PE/ Nylon6 700m/min)

The experiment XII (PE/Nylon6 700m/min)

The experiment XII was for a further step for producing segmented pie bi-component fibers using polyethylene and Nylon 6.

The experiment XII was designed to follow the implication of the experiment IX - XI. The experiment XII used the same materials as the experiment IX, X, and XI: polyethylene (EL-Lene H5818J) and Nylon 6 with materials ratio at 1: 1, using the bi-component extruder system in the laboratory, but altered the process by applying the winding mechanism at the end of the filament extrusion. The applied winding speed for the initial tentative step was 700 metre/min, which considerably medium speed.

Objectives of the experiment XII (PE/Nylon6 700 m/min)

1) To produce segmented pie bi-component filament with winding speed at 700 m/min.

2) To study the implications of the winding speed at 700 m/min in producing the bi-component filaments.

- 3) To study the bi-component fiber with winding mechanism at 700 m/min.
- 4) To study shapes of the produced segmented pie bi-component fibers using light compound microscope

Instruments:

1) Bi-component Extruder System

The fiber spinning experiments in this research were carried out using A Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 by The Hills Inc. which consists of 2 single screw extruders connected to a spinneret block. (figure. 3.2)

Materials:

The materials used in the experiment XII were polyethylene (EL-Lene H5818J) and Nylon 6 with materials ratio at 1:1. The information and specifications of the two materials were the same as in the previous experiments.

1) Material ratio of the two components:

Polyethylene (EL-Lene H5818J): Nylon 6
1:1

The polyethylene resins were used as received.

The nylon 6 resins were dried by incubating in hot air oven at temperature 80°C for approximately 6 – 8 hours before transferred immediately after took out from the incubator into a desiccator to cool the resins down to room temperature before use, the same manner as in the experiment IX - XII.

2) Extrusion spinneret plates

The spinneret plates for segmented pie bi-component fiber extrusion were assembled following the sequences specified in the assembly chart for the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100, Fig. 3.3. The plates were preheated in the oven at 250 °C for not less than 3 hours before assemble into the spinneret head. The plates were assembled consecutively into the spinneret head immediately after preheated.

3) Bi-component Extruder control system

The system conditions were set in same manner as the experiment IX - XI.

4) Polymer hoppers

The two hoppers of the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 were filled in same manner as the experiment IX - XI.

5) Segmented pie bi-component fiber extrusion

The polymer chips from the both hoppers were melted and became homogeneous liquidised polymers, then flew through polymer pipes into the metering pumps inside the spin-head before pass through the pack of the assembled spinneret plates. Eventually the melted polymers were extruded as bi-component strands of fibers and fell through surrounding ambience to cool down.

The experiment XII differed from the experiment IX - XI at the latter part of the process where the extruded bi-component filaments were subjected to winding tension at 700 metre/min. into round paper bobbin.

Analysis:

1) Material applicability: observing the production process throughout the experiment.

2) Suitability of the system condition: observing stability of the control panel, the flow of the materials, the continuation of the extruded filaments, etc.

- 3) Implications of the system condition: observing overall filament extrusion.
- 4) Shapes and geometry of the filament: observing the extruded filaments under light compound microscope.
- 5) Implication of the experiment XII (PE/Nylon 6 700 m/min): analysing the information received from this experiment for the following experiments.

3.14 Segmented pie bi-component fiber spinning: Experiment XIII (PE/Nylon 6 1% TiO₂ Free fall)

The experiment XIII (PE/Nylon6 1% TiO₂ Free fall)

The experiment XIII was for a further step for producing segmented pie bi-component fibers using polyethylene and Nylon 6.

The experiment XIII was designed to follow the implication of previous experiments. The filaments from the earlier experiments using polyethylene and nylon 6 were considerable agreeable as the extrusion processes were steady and manageable. Besides, the extruded filaments from the first four experiments were smooth, unbroken, uncrease, and no significant fiber breakage and fatigue.

The experiment XIII was also designed to add special feature to the fiber, particularly for healthcare or medical textile approach by using antibacterial supplement.

The experiment XIII used the same base materials as the experiment IX - XII: polyethylene (EL-Lene H5818J) and nylon 6 with materials ratio at 1: 1. The additive used in the experiment was 1.0% of TiO₂. The experiment carried out using the bi-component extruder system in the laboratory,

Objectives of the experiment XIII (PE/Nylon 6 1% TiO₂ free fall)

- 1) To produce segmented pie bi-component fibers using polyethylene and nylon6 as base polymer material and supplement with 1% TiO₂
- 2) To study the applicability of the two selected materials with the supplement of 1% TiO₂ for segmented pie bi-component fiber extrusion
- 3) To study the system condition for producing segmented pie bi-component fiber using the selected base material and the additive
- 4) To study the implications of the setting condition on the produced fibers.
- 5) To study the bi-component fiber without applying winding speed (free fall)
- 6) To study shapes of the produced segmented pie bi-component fibers using light compound microscope

Instruments:

- 1) Bi-component Extruder System

The fiber spinning experiments in this research were carried out using A Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS- 100 by The Hills Inc. which consists of 2 single screw extruders connected to a spinneret block.

Materials:

The base materials used in the experiment XIII polyethylene (EL- Lene H5818J) and Nylon 6, same as in the earlier experiments. The information and specifications of the two base materials were as in the experiment IX - XII.

The additive material for the intention of enhancing the antimicrobial in this experiment was Titanium dioxide. The 10% of Titanium dioxide Nylon 6 master batch and 20% Titanium dioxide Polyethylene master batch were used as functional supplement in the components.

The TiO₂ used in the experiment was the photocatalytic standard commercial type of P25, comprising mixed rutile and anatase phases. It has the average primary particle size of 21 ± 5 nm, specific surface area of 50 ± 10 m²/g, less than 0.3 wt% Al₂O₃, and less than 0.2 wt.%SiO₂.

Procedures:

1) The preparation of the materials:

Preparation of 200 gram of Polyethylene with 1.0% of TiO₂

(1) EL-Lene H5818J 190 gram

(2) Titanium dioxide Polyethylene master batch 10 gram

(3) combine and blend the polyethylene chips and Titanium dioxide

Polyethylene master batch chips

Preparation of 200 gram of Nylon 6 with 1.0% of TiO₂

(1) Nylon 6 180 gram

(2) Titanium dioxide Polyethylene master batch 20 gram

(3) combine and blend the Nylon 6 chips and Titanium dioxide Nylon 6

master batch chips

Material ratio:

Polyethylene, 1.0% TiO₂:Nylon 6, 1.0% TiO₂

1:1

The polyethylene resins were used as received.

The nylon 6 resins were dried by incubating in hot air oven at temperature 80°C for approximately 6 – 8 hours before transferred immediately after took out from the incubator into a desiccator to cool the resins down to room temperature before use, the same manner as in the experiment XII.

2) Extrusion spinneret plates

The spinneret plates for segmented pie bi-component fiber extrusion were assembled following the sequences specified in the assembly chart for the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100. The plates were preheated in the oven at 250 °C for not less than 3 hours before assemble into the spinneret head. The plates were assembled consecutively into the spinneret head immediately after preheated.

3) Bi-component Extruder control system

The system conditions were set in same manner as the experiment IX.

4) Polymer hoppers

The two hoppers of the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 were filled in same manner as the experiment IX - XII.

The polymer chips were not to be poured into the hoppers before the machine reached the stable stage. As soon as the system reached at the stationary condition, the hopper A was filled with the mixture of 1.0% TiO₂ polyethylene chips and the hopper B was filled with the mixture of 1.0% TiO₂ Nylon 6 chips.

5) Segmented pie bi-component fiber extrusion

The polymer chips from the both hoppers were melted and became homogeneous liquidised polymers, then flew through polymer pipes into the metering pumps inside the spin-head before pass through the pack of the assembled spinneret plates. Eventually the melted polymers were extruded as bi-component strands of fibers and free fell through surrounding ambiance to cool down.

Analysis:

- 1) Material applicability: observing the production process throughout the experiment.
- 2) Suitability of the system condition: observing stability of the control panel, the flow of the materials, the continuation of the extruded filaments, etc.
- 3) Implications of the system condition: observing overall filament extrusion.
- 4) Shapes and geometry of the filament: observing the extruded filaments under light compound microscope.
- 5) Implication of the experiment XIII (PE/Nylon 6 1% TiO₂ free fall): analysing the information received from this experiment for the following experiments.

3.15 Segmented pie bi-component fiber spinning: Experiment XIV (PE/Nylon6 1% TiO₂ 300 m/min)

The experiment XIV (PE/Nylon6 1% TiO₂ 300m/min)

The experiment XIV was for a further step for producing segmented pie bi-component fibers using polyethylene and Nylon 6.

The experiment XIV was designed to follow the implication of previous experiments. The filaments from the earlier experiments were considerable agreeable as the extrusion processes were steady and manageable. Besides, the extruded filaments from the first four experiments were smooth, unbroken, uncrease, and no significant fiber breakage and fatigue.

The experiment XIV was also designed to add special feature of antimicrobial using Titanium dioxide supplement as mentioned in the experiment XIII.

The experiment XIV used the same base materials as the experiment IX - XIII: polyethylene (EL-Lene H5818J) and Nylon 6, and same additives: 20% of Titanium dioxide Polyethylene master batch and 10% Titanium dioxide Nylon master batch. The component material ratio for this experiment was 1:1. The experiment carried out using the bi-component extruder system in the laboratory,

Objectives of the experiment XIV (PE/Nylon 6 1% TiO₂ 300 m/min)

- 1) To produce segmented pie bi-component fibers using polypropylene and polyethylene as base polymer material and supplement with 1% TiO₂ at 300 m/min applied winding speed
- 2) To study the implications of the winding speed at 300 m/min in producing the bi-component filaments.
- 3) To study the bi-component fiber with winding mechanism at 300 m/min.
- 4) To study shapes of the produced segmented pie bi-component fibers using light compound microscope

Instruments:

- 1) Bi-component Extruder System

The fiber spinning experiments in this research were carried out using A Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS- 100 by The Hills Inc. which consists of 2 single screw extruders connected to a spinneret block.

Materials:

The base materials used in the experiment XIV were polyethylene (EL-Lene H5818J) and Nylon 6, same as in the experiment IX - XIII. The information and specifications of the two materials were as in the experiment IX – XIII.

The additive material for the intention of enhancing the antimicrobial in this experiment was Titanium dioxide. The 20% of Titanium dioxide Polyethylene master batch and 10% Titanium dioxide Nylon 6 master batch were used as functional supplement in the components.

Procedures:

1. The preparation of the materials:

Preparation of 250 gram of Polyethylene with 1.0% of TiO₂

(1) EL-Lene H5818J 237.5 gram

(2) Titanium dioxide Polyethylene master batch 12.5 gram

(3) combine and blend the polyethylene chips and Titanium dioxide Polyethylene master batch chips

Preparation of 250 gram of Nylon 6 with 1.0% of TiO₂

(1) Nylon 6 225 gram

(2) Titanium dioxide Polyethylene master batch 25 gram

(3) combine and blend the Nylon 6 chips and Titanium dioxide Nylon 6 master batch chips

Material ratio:

Polyethylene, 1.0% TiO₂: Nylon 6, 1.0% TiO₂

1:1

The polyethylene resins were used as received.

The nylon 6 resins were dried by incubating in hot air oven at temperature 80°C for approximately 6 – 8 hours before transferred immediately after took out from the incubator into a desiccator to cool the resins down to room temperature before use, the same manner as in the experiment XIII.

2) Extrusion spinneret plates

The spinneret plates for segmented pie bi-component fiber extrusion were assembled following the sequences specified in the assembly chart for the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100. The plates were preheated in the oven at 250 °C for not less than 3 hours before assemble into the spinneret head. The plates were assembled consecutively into the spinneret head immediately after preheated.

3) Bi-component Extruder control system

The system conditions were set in same manner as the experiment IX – XIII.

4) Polymer hoppers

The two hoppers of the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 were filled in same manner as the earlier experiments.

The polymer chips were not to be poured into the hoppers before the machine reached the stable stage. As soon as the system reached at the stationary condition, the hopper A was filled with the mixture of 1.0% TiO₂ polyethylene chips and the hopper B was filled with the mixture of 1.0% TiO₂ Nylon 6 chips in the same manner as in the earlier experiments.

5) Segmented pie bi-component fiber extrusion

The polymer chips from the both hoppers were melted and became homogeneous liquidised polymers, then flew through polymer pipes into the metering pumps inside the spin-head before pass through the pack of the assembled spinneret plates. Eventually the melted polymers were extruded as bi-component strands of fibers and fell through surrounding ambiance to cool down.

The experiment XIV differed from the experiment XIII at the latter part of the process where the extruded bi-component filaments were subjected to winding tension at 300 metre/min. into round paper bobbin.

Analysis:

- 1) Material applicability: observing the production process throughout the experiment.
- 2) Suitability of the system condition: observing stability of the control panel, the flow of the materials, the continuation of the extruded filaments, etc.
- 3) Implications of the system condition: observing overall filament extrusion.
- 4) Shapes and geometry of the filament: observing the extruded filaments under light compound microscope.
- 5) Implication of the experiment XIV (PE/Nylon6 1% TiO₂ 300 m/min): analysing the information received from this experiment for the following experiments.

3.16 Segmented pie bi-component fiber spinning: Experiment XV (PE/Nylon6 1% TiO₂ 500 m/min)

The experiment XV (PE/Nylon6 1% TiO₂ 500m/min)

The experiment XV was for a further step for producing segmented pie bi-component fibers using polyethylene and Nylon 6.

The experiment XV was designed to follow the implication of previous experiments. The filaments from the earlier experiments were considerable agreeable as the extrusion processes were steady and manageable. Besides, the extruded filaments from the first four experiments were smooth, unbroken, uncrease, and no significant fiber breakage and fatigue.

The experiment XV was aslo designed to add special feature of antimicrobial using Titanium dioxide supplement as mentioned in the earlier experiments.

The experiment XV used the same base materials as the experiment IX - XIV: polyethylene (EL-Lene H5818J) and Nylon 6, and same additives: 20% of Titanium dioxide Polyethylene master batch and 10% Titanium dioxide Nylon master batch. The component material ratio for this experiment was 1: 1. The experiment carried out using the bi-component extruder system in the laboratory.

Objectives of the experiment XV (PE/Nylon 6 1% TiO₂ 500 m/min)

- 1) To produce segmented pie bi-component fibers using polypropylene and polyethylene as base polymer material and supplement with 1% TiO₂ at 500 m/min applied winding speed
- 2) To study the implications of the winding speed at 500 m/min in producing the bi-component filaments.
- 3) To study the bi-component fiber with winding mechanism at 500 m/min.
- 4) To study shapes of the produced segmented pie bi-component fibers using light compound microscope

Instruments:

- 1) Bi-component Extruder System

The fiber spinning experiments in this research were carried out using A Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS- 100 by The Hills Inc. which consists of 2 single screw extruders connected to a spinneret block.

Materials:

The base materials used in the experiment XV were polyethylene (EL-Lene H5818J) and Nylon 6, same as in the experiment IX - XIV. The information and specifications of the two materials were as in the experiment IX – XIV.

The additive material for the intention of enhancing the antimicrobial in this experiment was Titanium dioxide. The 20% of Titanium dioxide Polyethylene master batch and 10% Titanium dioxide Nylon 6 master batch were used as functional supplement in the components.

Procedures:

1. The preparation of the materials:

Preparation of 250 gram of Polyethylene with 1.0% of TiO₂

(1) EL-Lene H5818J 237.5 gram

(2) Titanium dioxide Polyethylene master batch 12.5 gram

(3) combine and blend the polyethylene chips and Titanium dioxide Polyethylene master batch chips

Preparation of 250 gram of Nylon 6 with 1.0% of TiO₂

(1) Nylon 6 225 gram

(2) Titanium dioxide Polyethylene master batch 25 gram

(3) combine and blend the Nylon 6 chips and Titanium dioxide Nylon 6 master batch chips

Material ratio:

Polyethylene, 1.0% TiO₂: Nylon 6, 1.0% TiO₂

1:1

The polyethylene resins were used as received.

The nylon 6 resins were dried by incubating in hot air oven at temperature 80°C for approximately 6 – 8 hours before transferred immediately after took out from the incubator into a desiccator to cool the resins down to room temperature before use, the same manner as in the experiment XIV.

2. Extrusion spinneret plates

The spinneret plates for segmented pie bi-component fiber extrusion were assembled following the sequences specified in the assembly chart for the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100. The plates were preheated in the oven at 250 °C for not less than 3 hours before assemble into the spinneret head. The plates were assembled consecutively into the spinneret head immediately after preheated.

3. Bi-component Extruder control system

The system conditions were set in same manner as the experiment IX – XIV.

4. Polymer hoppers

The two hoppers of the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 were filled in same manner as the earlier experiments.

The polymer chips were not to be poured into the hoppers before the machine reached the stable stage. As soon as the system reached at the stationary condition, the hopper A was filled with the mixture of 1.0% TiO₂ polyethylene chips and the hopper B was filled with the mixture of 1.0% TiO₂ Nylon 6 chips in the same manner as in the earlier experiments.

5) Segmented pie bi-component fiber extrusion

The polymer chips from the both hoppers were melted and became homogeneous liquidised polymers, then flew through polymer pipes into the metering

pumps inside the spin-head before pass through the pack of the assembled spinneret plates. Eventually the melted polymers were extruded as bi-component strands of fibers and fell through surrounding ambiance to cool down.

The experiment XV differed from the experiment XV at the latter part of the process where the extruded bi-component filaments were subjected to winding tension at 500 metre/min. into round paper bobbin.

Analysis:

- 1) Material applicability: observing the production process throughout the experiment.
- 2) Suitability of the system condition: observing stability of the control panel, the flow of the materials, the continuation of the extruded filaments, etc.
- 3) Implications of the system condition: observing overall filament extrusion.
- 4) Shapes and geometry of the filament: observing the extruded filaments under light compound microscope.
- 5) Implication of the experiment XV (PE/Nylon6 1% TiO₂ 500 m/min): analysing the information received from this experiment for the following experiments.

3.17 Segmented pie bi-component fiber spinning: Experiment XVI (PE/Nylon6 1% TiO₂ 700 m/min)

The experiment XVI (PE/Nylon6 1% TiO₂ 700m/min)

The experiment XVI was for a further step for producing segmented pie bi-component fibers using polyethylene and Nylon 6.

The experiment XVI was designed to follow the implication of previous experiments. The filaments from the earlier experiments were considerable agreeable as the extrusion processes were steady and manageable. Besides, the extruded filaments from the first four experiments were smooth, unbroken, uncrease, and no significant fiber breakage and fatigue.

The experiment XVI was also designed to add special feature of antimicrobial using Titanium dioxide supplement as mentioned in the earlier experiments.

The experiment XVI used the same base materials as the experiment IX - XV: polyethylene (EL-Lene H5818J) and Nylon 6, and same additives: 20% of Titanium dioxide Polyethylene master batch and 10% Titanium dioxide Nylon master batch. The component material ratio for this experiment was 1: 1. The experiment carried out using the bi-component extruder system in the laboratory.

Objectives of the experiment XVI (PE/Nylon 6 1% TiO₂ 700 m/min)

- 1) To produce segmented pie bi-component fibers using polypropylene and polyethylene as base polymer material and supplement with 1% TiO₂ at 700 m/min applied winding speed
- 2) To study the implications of the winding speed at 700 m/min in producing the bi-component filaments.
- 3) To study the bi-component fiber with winding mechanism at 700 m/min.
- 5) To study shapes of the produced segmented pie bi-component fibers using light compound microscope

Instruments:

- 1) Bi-component Extruder System

The fiber spinning experiments in this research were carried out using A Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 by The Hills Inc. which consists of 2 single screw extruders connected to a spinneret block.

Materials:

The base materials used in the experiment XVI were polyethylene (EL-Lene H5818J) and Nylon 6, same as in the experiment IX - XV. The information and specifications of the two materials were as in the experiment IX – XV.

The additive material for the intention of enhancing the antimicrobial in this experiment was Titanium dioxide. The 20% of Titanium dioxide Polyethylene master batch and 10% Titanium dioxide Nylon 6 master batch were used as functional supplement in the components.

Procedures:

1) The preparation of the materials:

Preparation of 250 gram of Polyethylene with 1.0% of TiO₂

(1) EL-Lene H5818J 237.5 gram

(2) Titanium dioxide Polyethylene master batch 12.5 gram

(3) combine and blend the polyethylene chips and Titanium dioxide Polyethylene master batch chips

Preparation of 250 gram of Nylon 6 with 1.0% of TiO₂

(1) Nylon 6 225 gram

(2) Titanium dioxide Polyethylene master batch 25 gram

(3) combine and blend the Nylon 6 chips and Titanium dioxide Nylon 6 master batch chips

Material ratio:

Polyethylene, 1.0% TiO₂: Nylon 6, 1.0% TiO₂

1:1

The polyethylene resins were used as received.

The nylon 6 resins were dried by incubating in hot air oven at temperature 80°C for approximately 6 – 8 hours before transferred immediately after took out from the incubator into a desiccator to cool the resins down to room temperature before use, the same manner as in the experiment XV.

2. Extrusion spinneret plates

The spinneret plates for segmented pie bi-component fiber extrusion were assembled following the sequences specified in the assembly chart for the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100. The plates were preheated in the oven at 250 °C for not less than 3 hours before assemble into the spinneret head. The plates were assembled consecutively into the spinneret head immediately after preheated.

3. Bi-component Extruder control system

The system conditions were set in same manner as the experiment IX – XV.

4. Polymer hoppers

The two hoppers of the Hills' Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 were filled in same manner as the earlier experiments.

The polymer chips were not to be poured into the hoppers before the machine reached the stable stage. As soon as the system reached at the stationary condition, the hopper A was filled with the mixture of 1.0% TiO₂ polyethylene chips and the hopper B

was filled with the mixture of 1.0% TiO₂ Nylon 6 chips in the same manner as in the earlier experiments.

5) Segmented pie bi-component fiber extrusion

The polymer chips from the both hoppers were melted and became homogeneous liquidised polymers, then flew through polymer pipes into the metering pumps inside the spin-head before pass through the pack of the assembled spinneret plates. Eventually the melted polymers were extruded as bi-component strands of fibers and fell through surrounding ambiance to cool down.

The experiment XVI differed from the experiment XV at the latter part of the process where the extruded bi-component filaments were subjected to winding tension at 700 metre/min. into round paper bobbin.

Analysis:

1) Material applicability: observing the production process throughout the experiment.

2) Suitability of the system condition: observing stability of the control panel, the flow of the materials, the continuation of the extruded filaments, etc.

3) Implications of the system condition: observing overall filament extrusion.

4) Shapes and geometry of the filament: observing the extruded filaments under light compound microscope.

5) Implication of the experiment XVI (PE/Nylon6 1% TiO₂ 700 m/min): analysing the information received from this experiment for the following experiments.

3.18 The analysis of the experiments

In the medical textile industry the focus herein lies on the potential applications of fine fibers for its superiority of active surface area. The segmented pie bi-component fiber processing is a high productivity where polymers melts are pushed through the specific spinneret. This fiber exists out of pie shape segments arranged in a segmented component that is detached afterwards. The miniscule size fibers were obtained but required specific polymer material make this technique relatively difficult.

It is possible to obtain different fiber properties by combining different polymers and percentage ratios with a good combinations additives and proper process conditions, the desire result properties of the textiles for medical use can be tailored.

A perfect match of all the ingredient in these trial experiments were not easily reached. The fibers as results from each experiments in this research were tested for their performance and properties, and subjected to be analyzed according to their implications. The structure and performance tests also gave the results to direct the next route for the following experiments. It also detected the faults of the machine settings and gave exact investigation for the better spinning conditions. The test results gave deeper understanding of factors underlined the behaviour and properties of the filament variations.

However, not every extruded filaments were subjected to every tests. Some of the filaments were examined and tested for their physical, mechanical, chemical, and biological properties, considering upon indications of each filament.

The test procedures and standard methods for this research were for the structure and performance of the materials such as:-

1) Thermal analysis

2) Visual and microscopic analysis

- 3) Three-dimensional shape (topography) of surface at high resolution
- 4) Surface topography and composition of sample with back-scattered electron imaging
- 5) Quantitative X-ray analysis and X-ray mapping of specimens
- 6) Tensile strength
- 7) Antimicrobial analysis

The filaments from the experiments were subjected to some specific tests, which could not be conducted at the university facilities, such as SEM (Scanning Electron Microscope), AFM (Atomic Force Microscopy), and AATCC test method 100 (assessment of antibacterial activity), were sent to government service agencies or commercial certified technical laboratories. The different characteristics of each produced fibers from different material variations and production settings were initially visual examined, then accounted for the further experiment and investigation.



CHAPTER 4 RESEARCH RESULT AND DISCUSSION

A series of laboratory and experimental works have been sequentially conducted in Rajamangala University of Technology Thanyaburi at the Faculty of Engineering using the Synthetic Fiber Processing Laboratory and related facilities at the Department of Textile Engineering, as well as, laboratories under government offices and private sectors.

This research goal was to produce splittable segmented pie bi-component fibers suitable for textile material for healthcare or medical applications.

4.1 Fiber Spinning

Bi-component Extruder System.

The fiber spinning experiments in this research were carried out using A Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 by The Hills Inc. The series of fiber production experiments were performed by varying the materials and metal supplement, and machine condition and setting. Each experiment gave unique results which were subjected to be examined and tested for their characteristics, structure, and performance.

4.1.1 Segmented pie bi-component fiber spinning: Experiment I

The experiment I (PP/PE free fall)

The experiment I was the initial tentative step for producing segmented pie bi-component fibers using the available materials.

The results of the experiment I (PP/PE free fall)

1) The production of segmented pie bi-component fibers

A batch of segmented pie bi-component fibers was produced successfully using the Lab Scale Bi-component Extruder Fed Spinning Machine Model LBS-100 by The Hills Inc.

2) The applicability of the two selected materials for producing segmented pie bi-component fiber

The selected materials were considered applicable as the process flew smoothly without any production difficulty, as soon as the system was stable the filaments were extruded persistently.

3) The system condition for production

The condition set for the experiment I appears in appendix A.

4) The implications of the setting condition on the produced fibers

The controlled condition for the experiment I implied that the condition was suitable to produce segmented pie bi-component fibers using the two materials – polypropylene and polyethylene.

5) The result of the fiber without applying winding speed (free fall)

The filaments were extruded out from the spinneret in liquidized form before fell freely through the laboratory ambient air then become solid filaments. The solid filaments were transparent colourless and considerably bulky in size. The fiber segments in a single filament were still intact and visually homogeneous solid features.

6) To study shapes of the produced segmented pie bi-component fibers using light compound microscope

The filaments from the experiment I were randomly examined under light compound microscope.

4.1.2 Segmented pie bi-component fiber spinning: Experiment II (PP/PE 300m/min)

The experiment II (PP/PE 300 m/min)

The experiment II was conducted after the experiment I. The result of the experiment I indicated that the materials used and the machine conditions were applicable to produce segmented pie bi-component fiber.

The experiment II used the same materials as in the experiment I: Polypropylene and polyethylene with material ratio at 1:1. The experiment I was designed to have the extruded filament drop freely from the spinneret through ambient air to cool down but the experiment II was designed to apply a winding mechanism to the filament as soon as they were extruded and become solid filaments at 300 metre/min.

The results of the experiment II (PP/PE 300 m/min)

1) The production of segmented pie bi-component filaments with winding speed at 300 m/min.

A batch of segmented pie bi-component fibers was produced successfully using the selected materials, machine conditions, and 300 m/min. winding speed at the end of the process.

2) The implications of the winding speed at 300 m/min in producing the bi-component filaments.

The winding mechanism was set at 300 metre/min and applied to the filament as soon as they were extruded out from the spinneret and become solid without any technical difficulty.

3) The bi-component fiber with winding mechanism at 300 m/min.

The filaments were extruded out from the spinneret in liquidized form before fell through the laboratory ambient air then quickly become solid filaments. And as soon as the filaments become solid the winding mechanism was applied at speed 300 metre/min. The solid filaments were semi-transparent colourless. The fiber segments in a single filament were still intact and visually homogeneous solid features.

4) The shapes of the produced segmented pie bi-component fibers using light compound microscope

The filaments from the experiment II were randomly examined under light compound microscope.

4.1.3 Segmented pie bi-component fiber spinning: Experiment III (PP/PE 500m/min)

The experiment III (PP/PE 500 m/min)

The experiment III was for a further step for producing segmented pie bi-component fibers using polypropylene and Polyethylene at 500 m/min winding speed.

The experiment III was designed to follow the implication of the experiment II. The experiment III used the same materials as the experiment I and II: polypropylene (MoPlen HP561R) and polyethylene (EL-Lene H5818J) with materials ratio at 1:1, the applied winding speed for the initial tentative step was 500 metre/min.

The results of the experiment III (PP/PE 500 m/min)

1) The material applicability

The materials used in the experiment III were the same as in the earlier experiments to produce segmented pie bi-component. The materials were applicable and withstand the winding speed at 500 m/min. at the end of the process.

2) The system condition

The condition set for the experiment III appears in appendix A.

3) The implications of the system condition

A batch of segmented pie bi-component fibers was extruded through spinneret using the control condition with no complication. The winding mechanism applied at the end of the process also run smoothly. The filaments were wound into paper bobbins continuously and evenly.

4) Shapes and geometry of the filament

The filaments from the experiment III were randomly examined under light compound microscope.

5) Implication of the experiment III (PP/PE 500 m/min)

The filaments were successfully extruded and evenly wound into bobbins with winding speed at 500 metre/min. The solid filaments were semi-transparent colourless. The fiber segments in a single filament were still intact and visually homogeneous solid features.

4.1.4 Segmented pie bi-component fiber spinning: Experiment IV (PP/PE 700m/min)

The experiment IV (PP/PE 700 m/min)

The experiment IV produced a batch of segmented pie bi-component fibers using polypropylene and Polyethylene at 700 m/min winding speed.

The experiment IV used the same materials as the earlier experiments: polypropylene (MoPlen HP561R) and polyethylene (EL-Lene H5818J) with materials ratio at 1:1, with winding speed at 700 metre/min.

The results of the experiment IV (PP/PE 700 m/min)

1) The material applicability

The materials used in the experiment IV were applicable and withstand the winding speed at 700 m/min.

2) The system condition

The condition set for the experiment IV appears in appendix A.

3) The implications of the system condition

The segmented pie bi-component fibers were produced with winding speed at 700 m/min. without any difficulty. The filaments were wound into bobbins quickly and evenly throughout the process.

4) Shapes and geometry of the filament

The filaments from the experiment IV were randomly examined under light compound microscope.

5) The implication of the experiment IV (PP/PE 700 m/min)

The experiment IV was conducted in similar manner as the experiment II and III, but using the faster winding speed. The filaments were wound into bobbins with quicker than the previous experiments.

4.1.5 Segmented pie bi-component fiber spinning: Experiment V (PP/PE TiO₂ Free fall)

The experiment V (PP/PE 1% TiO₂ Free fall)

The experiment V was designed to follow the implication of previous experiments for producing segmented pie bi-component fibers using polypropylene and Polyethylene with 1.0% TiO₂ without winding mechanism at the end of the fiber spinning.

The filaments from the earlier experiments using polypropylene and polyethylene without applying winding mechanism were successfully spun. The

production of segmented pie bi-component fibers using polypropylene and polyethylene with applied winding mechanism at the final step of the process also did not face any difficulty for all the applied winding speeds: 300 m/min, 500 m/min, and 700 m/min. Therefore, the system conditions for the previous experiments were chosen for this experiment because the fibers would certainly be reproducible in the same manner.

The first experiment for this series continued to use the same base materials to produce segmented pie bi-component fibers – polypropylene (MoPlen HP561R) and polyethylene (EL-Lene H5818J). The additives selected for enhancing antimicrobial activity was titanium dioxide (TiO_2). The percentage of TiO_2 in the experiment was 1.0%.

The result of the experiment V

1) The material applicability

The materials and additive used in the experiment V were applicable. The base materials and the master batch of TiO_2 were blended well and the fibers were homogeneously extruded.

2) The system condition

The condition set for the experiment V

3) The implications of the system condition

The experiment V was conducted in same system condition as the earlier experiments but mixing the photocatalytic supplement agent from polypropylene and polyethylene master batches into the polymer chips before pour each material into the signified hoppers.

4) The shapes and geometry of the filaments

The filaments from the experiment V were randomly examined under light compound microscope.

5) The implication of the experiment V (PP/PE 1% TiO_2 free fall)

The system condition were set in the similar manner as in the earlier experiments and the production run smoothly as expected. The extruded filaments flew down continuously without disruption. The extruded filaments were colourless and transparent. The size of the filaments were bulky because there was no winding mechanism applied.

4.1.6 Segmented pie bi-component fiber spinning: Experiment VI (PP/PE TiO_2 300 m/min)

The experiment VI (PP/PE 1% TiO_2 300 m/min)

The experiment VI was for producing segmented pie bi-component fibers using polypropylene and Polyethylene with 1.0% TiO_2 at 300 m/min winding speed.

The experiment VI was designed after the experiment V which conducted successfully and produced the segmented pie bi-component fibers with polypropylene and polyethylene as base materials with 1.0% of TiO_2 additive. Therefore, this experiment was processed with the same materials and additives but altered the system by applying the winding mechanism at 300 m/min. just immediately after the filaments were cooled by ambient air.

The result of the experiment VI:

1) The material applicability

The materials and the additive were the same as the experiment V, therefore, the materials were applicable as expected.

2) The system condition

The condition set for the experiment VI appears in appendix A.

3) The implications of the system condition

The experiment VI was conducted in similar system condition as the previous experiment but alter with winding mechanism at 300 m/min. The segmented pie bi-component filaments were produced smoothly under this committed system condition.

4) The shapes and geometry of the filament

The produced filaments from the experiment VI were initially examined under light compound microscope.

5) The implication of the experiment VI (PP/PE 1% TiO₂ 300 m/min)

The experiment were run smoothly as expected with the similar condition and materials as earlier experiments. The segments in the filaments were still intact and visually consistency.

4.1.7 Segmented pie bi-component fiber spinning: Experiment VII (PP/PE TiO₂ 500 m/min)

The experiment VII (PP/PE 1% TiO₂ 500 m/min)

The experiment VII was for producing segmented pie bi-component fibers using polypropylene and Polyethylene with 1.0% TiO₂ at 500 m/min winding speed.

The experiment VII was considered to be the further experiment from the experiment VI by accelerated up the winding speed from 300 m/min to 500 m/min, as the implications of all the previous experiments had tendency to reproducible with higher winding speed.

The results of the experiment VII:

1) The material applicability

The materials and the additive were applicable.

2) The system condition

The condition set for the experiment VII appears in appendix A.

3) The implications of the system condition

The experiment VII was conducted in similar manner of the experiments VI altering only the winding speed from 300 m/min. to 500 m/min. The extruded filaments were smooth and constantly wound into bobbins.

4) The shapes and geometry of the filament

The extruded filaments were examined under light compound microscope.

5) The implication of the experiment VII (PP/PE 1% TiO₂ 500 m/min)

The accelerated winding speed from 300 m/min to 500 m/min were not affected the experiment as the filament were wound into bobbins faster without decelerated. The solid filaments were wound into bobbins smoothly.

4.1.8 Segmented pie bi-component fiber spinning: Experiment VIII (PP/PE TiO₂ 700 m/min)

The experiment VIII (PP/PE 1% TiO₂ 700 m/min)

The experiment VIII was intended to speed up the production of segmented pie bi-component fibers using polypropylene and Polyethylene with 1.0% TiO₂ by 200 m/min from the previous experiment.

The result of the experiment VIII:

1) The material applicability

The base materials and additive used in this experiment were the same as the last experiment which is all applicable.

2) The system condition

The condition set for the experiment VIII appears in appendix A.

3) The implications of the system condition

The experiment VIII was another untrobling production of segmented pie bi-component fibers using polypropylene and polyethylene with 1.0% TiO₂. The filaments were visually indifferent from the filaments from the previous experiment. The faster winding speed made it quicker to be wound into bobbins.

4) The shapes and geometry of the filament

The extruded filaments were examined under light compound microscope.

5) The implication of the experiment VIII (PP/PE 1% TiO₂ 700 m/min)

The accelerated applied winding speed at 700 m/min had unnoticeable effect on the production only that it made the filaments wound into bobbins faster than the earlier experiments.

4.1.9 Segmented pie bi-component fiber spinning: Experiment IX (PE/Nylon 6 Free fall)

The experiment IX (PE/Nylon6 free fall)

The experiment IX was for an initial tentative step for producing segmented pie bi-component fibers using polyethylene and Nylon 6.

The series of the earlier experiments used polypropylene and polyethylene as the base materials to produce segmented pie bi-component fibers. The experiments were successfully done without process hiccup.

However, the extruded filaments from all of the previous experiments were not split lengthwise or the segments within the filaments were firmly attached to one another, which against the intended hypothesis to have the filament detached into smaller filaments with greater surface area. This experiment changed the base materials from polypropylene and polyethylene to polyethylene and Nylon 6, for better segments detachable anticipation. This experiment was the first in its series of experiments using same base materials without photocatalytic agent with varying the filament winding speed.

The result of the experiment IX:

1) The material applicability

The applicability of the selected polyethylene and Nylon 6 were met the experiment expectations. The process using the two base materials were manageable.

2) The system condition

The condition set for the experiment IX appears in appendix B.

3) The implications of the system condition

The experiment IX was the first experiment in its series of experiments using polyethylene and Nylon 6 without winding mechanism. The system condition were set according to the recommendation of the two materials. The production run smoothly without any difficulty.

4) The shapes and geometry of the filament

The filaments from this experiment were examined under light compound microscope.

5) The implication of the experiment IX (PE/Nylon 6 free fall)

The system condition were changed as one of the base materials was different from all of the earlier experiments. The system conditions were adjusted

according to the recommendation of both materials. The production of the experiment were conducted in the similar manner to the experiments using polypropylene and polyethylene. The outcome of this experiments had no significant differences from other related experiments. The segmented pi bi- component filaments were extruded uncomplicated. The winding mechanism was not applied so the filaments flew freely to the container below.

4.1.10 Segmented pie bi-component fiber spinning: Experiment X (PE/Nylon 6 300m/min)

The experiment X (PE/Nylon6 300m/min)

The experiment X was to produce segmented pie bi-component fibers using polyethylene and Nylon 6.

This experiment was designed to produce a new series of segmented pie bi-component fibers without photocatalytic function. The base materials were selected the same as the previous experiment but the winding mechanism were applied at the end of the process to wind the filaments into bobbins. The winding speed for this experiment was 300 m/min.

The result of the experiment:

1) The material applicability

The materials were not different from the experiment IX, therefore, there was no difference of the material applicability.

2) The system condition

The condition set for the experiment X appears in appendix B

3) The implications of the system condition

The experiment X was the second experiment in its series of experiments using polyethylene and Nylon 6. The system condition were set in the same manner of the experiment IX but applied 300 m/min. winding speed to roll the filaments into bobbins. The production run smoothly without any difficulty.

4) The shapes and geometry of the filament

The filaments from this experiment were examined under light compound microscope.

5) The implication of the experiment X (PE/Nylon 6 300 m/min)

The winding speed at 300 m/min applied at the end of the process of producing segmented pie bi-component fibers using polyethylene and nylon 6 as base materials were feasible. The fibers were wound into the bobbins continuously and the filament were unbroken.

4.1.11 Segmented pie bi-component fiber spinning: Experiment XI (PE/Nylon 6 500m/min)

The experiment XI (PE/Nylon6 500m/min)

The experiment XI was for a further step for producing segmented pie bi-component fibers using polyethylene and Nylon 6.

The experiment XI was designed to follow the implication of the experiment X but speed up the winding speed to 500 m/min.

The results of the experiment XI:

1) The material applicability

The materials were not different from the experiment IX and X, which was all applicable.

2) The system condition

The condition set for the experiment XI appears in appendix B.

3) The implications of the system condition

The experiment XI was the third experiment in its series of experiments using polyethylene and Nylon 6. The system condition were set in the same manner of the experiment IX and X but applied 500 m/min. winding speed at the final step of the process. The production run smoothly without any difficulty.

4) The shapes and geometry of the filament

The filaments from the experiment XI were examined under light compound microscope.

5) The implication of the experiment XI (PE/Nylon 6 500 m/min)

The winding speed at 500 m/min applied at the end of the process of producing segmented pie bi-component fibers using polyethylene and nylon 6 were considered feasible because the filaments were wounded into bobbins continuously and unbroken.

4.1.12 Segmented pie bi-component fiber spinning: Experiment XII (PE/Nylon 6 700m/min)

The experiment XII (PE/Nylon6 700m/min)

The experiment XII was for a further step for producing segmented pie bi-component fibers using polyethylene and Nylon 6.

The experiment XII was designed to follow the implication of the experiment IX - XI. The experiment XII used the same materials as the experiment IX, X, and XI: polyethylene (EL-Lene H5818J) and Nylon 6 with materials ratio at 1:1. The applied winding speed for this experiment was 700 metre/min, the fastest speed in its series of the experiments.

The results of the experiment XII:

1) The material applicability

The material used in this experiment were the same as the earlier experiment of its series – polyethylene and nylon 6 without functional additive, so the materials applicability still showed the same result.

2) The system condition

The condition set for the experiment XII appears in appendix B.

3) The implications of the system condition

The experiment XII was the last experiment in its series of experiments using polyethylene and Nylon 6. The system condition were set in the same manner of the experiment IX and X but applied 700 m/min. winding speed which was the fastest applied speed this series of the experiments. The production faced some difficulty in producing segmented pie bi-component fibers. The extruded filaments tended to break soon after wounded into bobbins. There were several attempts to extrude the filament and wind up them into bobbins but most of the attempts faced the similar problem of breaking filaments. Therefore, the winding speed at 700 m/min. was not feasible for the two base materials.

4) The shapes and geometry of the filament

The filaments from the experiment XII were examined under light compound microscope.

5) The implication of the experiment XII (PE/Nylon 6 700 m/min)

The winding speed at 700 m/min applied at the end of the process of producing segmented pie bi-component fibers using polyethylene and nylon 6 were

considered unlikely to be feasible because the filaments were too weak to withstand the winding speed at 700 m/min. as they tended to break soon after the winding mechanism was applied.

4.1.13 Segmented pie bi-component fiber spinning: Experiment XIII (PE/Nylon 6 1% TiO₂ Free fall)

The experiment XIII (PE/Nylon6 1% TiO₂ Free fall)

The experiment XIII was for a further step for producing segmented pie bi-component fibers using polyethylene and Nylon 6 with 1% TiO₂ as functional additive.

The series of the earlier experiments used polypropylene/polyethylene and polyethylene/ Nylon 6 as the base materials and to produce segmented pie bi-component fibers. The most of the experiments were successfully done. The only experiment with difficulty was the polyethylene and Nylon 6 with 700 m/min. applied winding speed.

The segments in the extruded filaments from the polypropylene and polyethylene tended to cling tight to one another, while the filaments from polyethylene and nylon 6 were split or detach lengthwise easier. Therefore, in this series of experiments began with the similar selection of the material – polyethylene and nylon 6, the alteration was the varying winding speeds. The experiment XIII was the first in its series which the production was not applied with the winding mechanism.

The result of the experiment XIII:

1) The material applicability

The applicability of the selected polyethylene and Nylon 6 and the TiO₂ additive were met the experiment expectations. The process using the two base materials were manageable.

2) The system condition

The condition set for the experiment XIII appears in appendix B.

3) The implications of the system condition

The experiment XIII was the first experiment in its series of experiments using polyethylene and Nylon 6 with 1.0% TiO₂ without winding mechanism. The system condition were set according to the recommendation of the two materials and the master batch of TiO₂. The production run smoothly without any difficulty.

4) The shapes and geometry of the filament

The filaments from this experiment were examined under light compound microscope.

5) The implication of the experiment XIII (PE/Nylon 6 1.0% TiO₂ free fall)

The system condition were similar to the earlier experiments where polypropylene and nylon 6 were selected as base materials. The production of the experiment were conducted in the similar manner to the experiments using polyethylene and nylon 6 without functional additive. The filaments from this experiment had no significant differences from the experiment using the same base materials. The winding mechanism was not applied so the filaments flew freely to the container below.

4.1.14 Segmented pie bi-component fiber spinning: Experiment XIV (PE/Nylon 6 1% TiO₂ 300 m/min.)

The experiment XIV (PE/Nylon6 1% TiO₂ 300m/min)

The experiment XIV was for a further step for producing segmented pie bi-component fibers using polyethylene and Nylon 6 with functional additive and applied winding speed.

The experiment XIII was run successfully so the experiment XIV was designed to follow the implication of the experiment XIII but adding the winding mechanism at 300 m/min. in the end of the process.

The results of the experiment XIV:

1) The material applicability

The selected polyethylene, nylon 6, and functional additive were applicable.

2) The system condition

The condition set for the experiment XIV appears in appendix B.

3) The implications of the system condition

The experiment XIV was the second experiment in its series of experiments using polyethylene and Nylon 6 with 1.0% TiO₂. The system condition were altered by applying winding mechanism at 300 m/min. The filament extruding were not facing any difficulty throughout the process.

4) The shapes and geometry of the filament

The filaments from this experiment were examined under light compound microscope.

5) The implication of the experiment XIV (PE/Nylon6 1% TiO₂ 300 m/min)

The system condition were similar to the earlier experiments XIII which used the same base materials and additive. The process were progress in similar manner to the experiment XIV but after by adding the 300 m/min winding speed to roll the filaments onto bobbins. The production run smoothly and the filaments were consistence, smooth and unbroken.

4.1.15 Segmented pie bi-component fiber spinning: Experiment XV (PE/Nylon 6 1.0% TiO₂ 500 m/min.)

The experiment XV (PE/Nylon6 1% TiO₂ 500m/min)

The experiment XV was the third experiment for producing segmented pie bi-component fibers using polyethylene and Nylon 6 with 1.0% TiO₂ additive.

The experiment XV was designed to follow the implication of previous experiments. The filaments from the experiment XIV were considerable agreeable as the extrusion processes were steady and manageable. The attempt of this experiment was to speed up the production by 200 m/min. by changing the winding speed from 300 m/min. to 500 m/min.

The results of the experiment XV:

1) The material applicability

The materials used in this experiment were the same materials used in the experiment XIV which all applicable.

2) The system condition

The condition set for the experiment XV appears in appendix B.

3) The implications of the system condition

The experiment XV was the third experiment of its series of experiments using polyethylene and Nylon 6 with 1.0% TiO₂. The system condition were altered by speeding up the applied winding mechanism to 500 m/min. The filament

extruding were facing a slightly difficulty as the filaments broke a while after applied winding mechanism. An enough amount of the filaments were still managed to be wounded onto bobbins.

4) The shapes and geometry of the filament

The filaments from this experiment were examined under light compound microscope.

5) The implication of the experiment XV (PE/Nylon6 1% TiO₂ 500 m/min)

The system condition were used the same condition as the experiment XIV but alter the winding speed to 500 m/min. instead of 300 m/min. The production was experiencing small set back as the filaments broke during the winding process. A few further experimental attempts were conducted to wound more filaments onto the prepared bobbins enough for examining the characteristics and properties of the filaments.

4.1.16 Segmented pie bi-component fiber spinning: Experiment XVI (PE/Nylon 6 1.0% TiO₂ 700 m/min.)

The experiment XVI (PE/Nylon6 1.0% TiO₂ 700m/min)

The experiment XVI was the further attempt to produce segmented pie bi-component fibers using polyethylene and Nylon 6 with 1.0% TiO₂ as functional additive.

This experiment was designed to try to produce segmented pie bi-component fibers using polyethylene and Nylon 6 with 1.0% TiO₂ with high winding speed than the previous experiments.

The results of the experiment XVI:

1) The material applicability

The materials used in this experiment were the same materials used in the experiment XV which all applicable.

2) The system condition

The condition set for the experiment XVI appears in appendix B.

3) The implications of the system condition

The experiment XVI was the final experiment of its series of experiments using polyethylene and Nylon 6 with 1.0% TiO₂. The system condition were altered by accelerated the winding speed to 700 m/min. The filament extruding were facing complication as the filaments broke soon after being wounded onto the filament bobbins. A small amount of the filaments were managed to be wounded onto bobbins.

4) The shapes and geometry of the filament

The filaments from this experiment were examined under light compound microscope.

5) The implication of the experiment XVI (PE/Nylon6 1% TiO₂ 700 m/min)

The system condition were the same condition as the experiment XIV but alter the winding speed to 700 m/min. instead of 500 m/min. The production was experiencing set back as the filaments broke during the winding process. A few further experimental attempts were conducted to wound more filaments onto the prepared bobbins enough for examining the characteristics and properties of the filaments.

4.2 Synopsis and discussion of the production of the experiment I to XVI

A series of 16 laboratories and experimental works have been sequentially conducted to produce splittable segmented pie bi-component fibers suitable for textile material for healthcare or medical applications. The experiments were carried out sequentially following the results of the previous experiments. The results from the earlier experiments showed the possibility of producing and spinning of splittable segmented pie bi-component fibres using selected base polymers. The spinning process were emphasized on producing minuscule fibres using a two extruders system. The nanoparticles of titanium dioxide (TiO_2) were embedded in the bi-component fibers during the extrusion stage by mixing additive master-batches of the base polymers with the pure base polymers. The intention of adding titanium dioxide particles in the extrusion stage was that the particles would be dispersed thoroughly inside and on the surface of the both components of the extruded filaments. The particles of titanium dioxide were secured and disseminated throughout fiber polymer [27] by production adding them into polymer during fiber extrusion stage. The production of the fibers embedded with 0.1% TiO_2 in some of the 16 experiments did not show significant production differences with the ones without TiO_2 . Therefore, the particles of the TiO_2 did not burden the productions.

The experiments designed to apply winding mechanism to the extruded filaments at the end of the process. The winding mechanism was applied at vary speeds in order to detect the maximum stretch that the experimented filaments could withstand. The applied winding mechanism would probably affect the two base polymers in different degree which would make them split from one another easier since the different polymers have different degree of resilience and flexibility toward mechanical force. The materials use in the bicomponent fibre forming that intend to separate them into smaller fibres was a crucial indicator for the success of the research, because the cohesion of the two selected materials will influence the endeavour in separate them lengthwise. The base polymers used in the experiments were applicable to the fiber extrusion in melt spinning process, cheap, common and easy to find in the market. They were applicable for technical and functional textile end uses. The additional feature of the base polymers used in the experiments was to have a capability to hold and accommodate the photocatalytic materials. Polyolefin (polypropylene and polyethylene) and Nylon 6 are among the typical fibres use in clothing, technical and functional textiles nowadays. There are many different types and grades of the polymers available in commercial market nowadays. [33] Therefore, it would be a wise choice to choose them as the base materials for the experiments. The base polymers selected for the experiments were commercial grade polymers: polypropylene (MoPlen HP561R), polyethylene (EL-Lene H5818J), and nylon 6 (semi dull grade) that could be find easily with competitive prices in the material market. The two base polymers used for each experiment were commercial grade and the material ratio was 1:1. The system condition was set according to the recommendation of the two materials and capability of the machine. The titanium dioxide used in the experiment was P25 which is also a commercial photocatalytic agent, comprising mixed rutile and anatase phases.

The batches of segmented pie bi-component fibers using the selected base materials were produced successfully. The production of most of the experiments were smooth, stable and the materials were applicable. The filaments were extruded out from the spinneret in liquidized form before fell freely through the laboratory ambient air then become solid filaments. The experiment without winding mechanism were extruded and

fell freely without any tension after the filaments were emerge from the spinneret. The other experiments attempted to apply tension to the extruded filaments using winding mechanism soon after the filament emerged from the spinneret. The aims of the winding mechanism at 300 m/min., 500 m/min., and 700 m/min. were to reduce the size of the extruded filaments, accelerate the productivity, and separate the filament lengthwise. The filaments from the experiments with winding mechanism were wounded onto paper bobbins continuously and evenly. The synopsis of the selected base materials and the TiO₂ additives in the experiments were applicable for most of the experimental productions except for the spinning of polyethylene and nylon 6 at 700 m/min. winding speed. Therefore, the filament of the base materials that applicable for the spinning processes would withstand the winding mechanism at as high speed as 700 m/min. The experiments were successfully conducted, the system condition setting and base polymers used in the experiments were applicable and the filaments from the fourteen experiments indicated that producing the fibers with high winding speed up to 700 m/min. was achievable because the extruded filaments were smooth, unbroken and withstand the tension applied as the winding mechanism.

The experiment of polyethylene and nylon 6 were conducted for the approach of finding a better selection of the base materials to be spun as segmented pie bi-component fibers that easily split lengthwise than the fibers from the experiment of polypropylene and polyethylene. The reason of the selection was that the former pair of the base materials – polypropylene and polyethylene - were both olefin class of polymers which tend to have similar properties, that cohesion between the two polymers were rather high [6,39,17] so when the liquidized polymers emerged from the spinneret in the form of segmented pie bi-component filaments, the two polymer phases reacted similarly toward the ambient air around them. The new selection was to find base materials with different class of polymers. The hypothesis for the selection was that polyethylene and nylon 6 reacted to the fiber spinning condition and process differently, particularly when they come into contact with the ambient air after they immerge from the spinneret head. Their different degree crystallinity and thermal properties [33,34,37] would effects the contraction and shrinking of their polymer phases when encountered the surrounding air would cause their segments to be divided lengthwise. The attempts to separate the segments within the filaments in lengthwise axis in the new base materials indicated more promising affect. The additional experiment were the spinning nylon filaments with and without TiO₂. The experiments were successfully spun at high speeds. However, the attempts to spin the fine-denier polyethylene filaments with and without TiO₂ were unsuccessful at high winding speeds because the branch chains on the polyethylene counteracted the fiber orientation. [33,34,35] The spinning of the bi-component filaments of nylon segmented with polyethylene facilitated the formation of fine-denier segmented solid filaments from the melted polyethylene because of the support provided by the adjoining nylon filaments.

4.3 Fiber Analysis

In this research the focus herein lies on the potential medical textile applications. The production of segmented pie bi- component fibers was selected because the hypothesis of the research was to enhance active surface area of the textile materials by producing extremely fine fibers from initial segmented pie bi- component fibers.

Therefore, the examinations and tests were mostly on the appearance, shape and geometry of the extruded filaments and the antimicrobial activity of the materials.

4.3.1 Thermal Analysis

The Differential Scanning Calorimetry (DSC)

The assessment of the polymer percent crystallinity were performed using differential scanning calorimetry.

The extruded filament from experiment I to XVI were selected to be subjected to the thermal analysis test. The base materials in the first attempt of the experiment were polypropylene and polyethylene. The results of the thermal analysis of filaments using the two base materials were presented in the table 4.1. The table shows the onset temperature, melting temperature, ΔH_m , and percentage of crystallinity of the filament using the two material with the free fall condition. The results from the DSC thermal analysis of the two selected base materials with free fall condition also shows in fig. 4.1

Table 4.1 The thermal analysis of the selected polypropylene and polyethylene filaments using free fall condition.

| Sample | Winding Speeds (m/min) | Onset Temperature (°C) | | Melting Temperature (°C) | | ΔH_m (J/g) | | Crystallinity (%) | |
|--------|------------------------|------------------------|-------|--------------------------|-------|--------------------|-------|-------------------|-------|
| | | PP | PE | PP | PE | PP | PE | PP | PE |
| PP | - | 159.7 | - | 168.5 | - | 76.28 | - | 36.85 | - |
| PE | - | - | 124.9 | - | 130.9 | - | 150.0 | - | 51.02 |

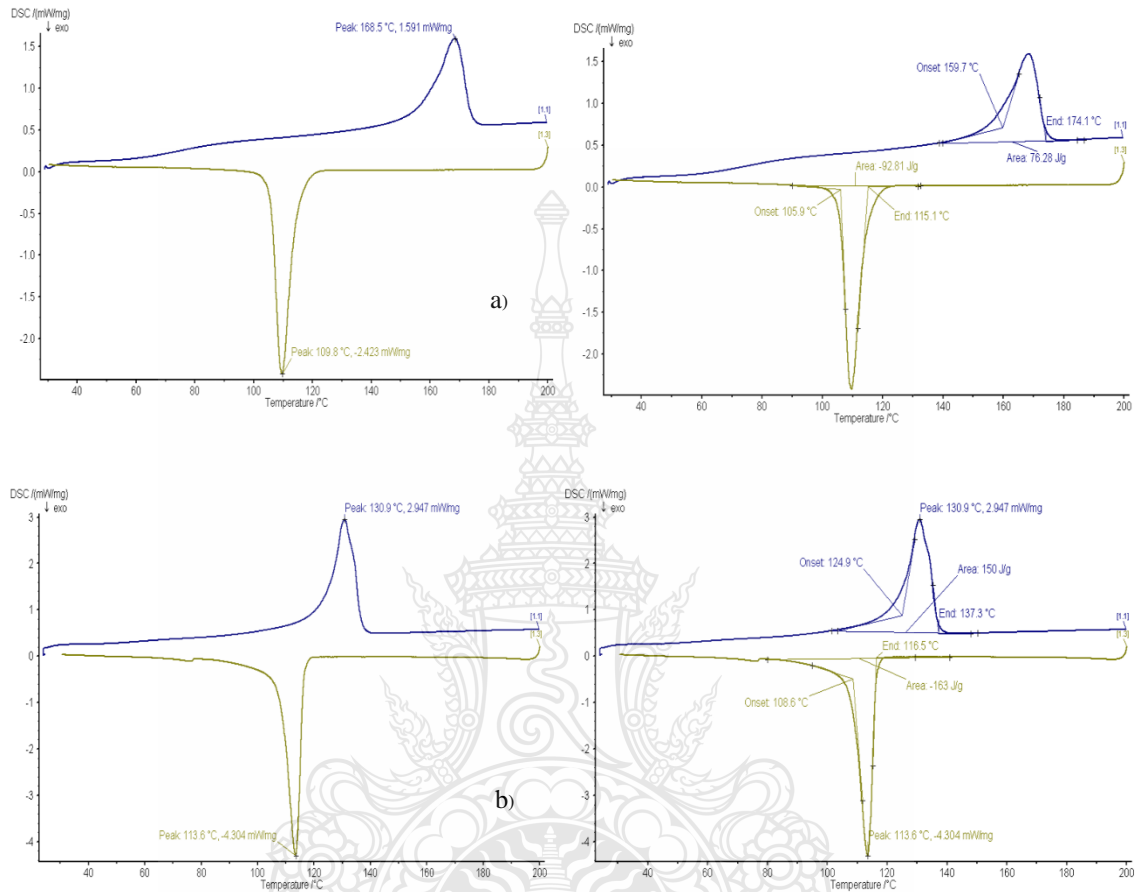


Figure 4.1 The thermal analysis of a) polypropylene and b) polyethylene filaments using free fall condition

The results of the thermal analysis of the segmented pie bi-component filaments using the selected polypropylene and polyethylene as base materials with alterations of winding speeds from 300 m/min to 700 m/min. shows in table 4.2, presents the onset temperature, melting temperature, ΔH_m , and percentage of crystallinity. The results from the DSC thermal analysis of the two selected base materials with winding speeds at 300 m/min, 500 m/min, and 700 m/min. also shows in fig. 4.2

Table 4.2 The thermal analysis of the segmented pie bi-component polypropylene and polyethylene filaments using with winding speeds at 300 m/min, 500 m/min, and 700 m/min.

| Sample | Winding Speeds (m/min) | Onset Temperature (°C) | | Melting Temperature (°C) | | ΔH_m (J/g) | | Crystallinity (%) | |
|--------|------------------------|------------------------|-------|--------------------------|-------|--------------------|-------|-------------------|-------|
| | | PP | PE | PP | PE | PP | PE | PP | PE |
| PP/PE | 300 | 156.6 | 123.0 | 168.3 | 129.4 | 47.92 | 54.28 | 23.15 | 18.46 |
| PP/PE | 500 | 157.2 | 123.0 | 167.9 | 130.5 | 60.74 | 38.22 | 29.34 | 13.00 |
| PP/PE | 700 | 170.0 | 123.2 | 169.2 | 129.2 | 35.41 | 32.02 | 17.11 | 10.89 |

The results of the thermal analysis of the segmented pie bi-component filaments using the selected polypropylene and polyethylene as base materials and 1.0% TiO₂ as functional additive with alterations of winding speeds from 300 m/min to 700 m/min. shows in table 4.3, presents the onset temperature, melting temperature, ΔH_m , and percentage of crystallinity. The results from the DSC thermal analysis of the two selected base materials with 1.0% TiO₂ functional additive at altering winding speeds at 300 m/min, 500 m/min, and 700 m/min. also shows in fig. 4.3

Table 4.3 The thermal analysis of the segmented pie bi-component polypropylene and polyethylene filaments with 1.0% TiO₂ as functional additive using with winding speeds at 300 m/min, 500 m/min, and 700 m/min.

| Sample | Winding Speeds (m/min) | Onset Temperature (°C) | | Melting Temperature (°C) | | ΔH_m (J/g) | | Crystallinity (%) | |
|-----------------------------|------------------------|------------------------|-------|--------------------------|-------|--------------------|-------|-------------------|-------|
| | | PP | PE | PP | PE | PP | PE | PP | PE |
| PP/PE 1%TiO ₂ | 300 | 156.1 | 122.8 | 164.8 | 128.3 | 35.11 | 35.64 | 16.96 | 12.12 |
| PP/PE 1%TiO ₂ | 500 | 164.5 | 123.0 | 167.6 | 128.5 | 32.53 | 62.6 | 15.71 | 21.29 |
| PP/PE 1%TiO ₂ | 700 | 155.8 | 122.9 | 163.4 | 128.4 | 33.67 | 68.09 | 16.27 | 23.16 |

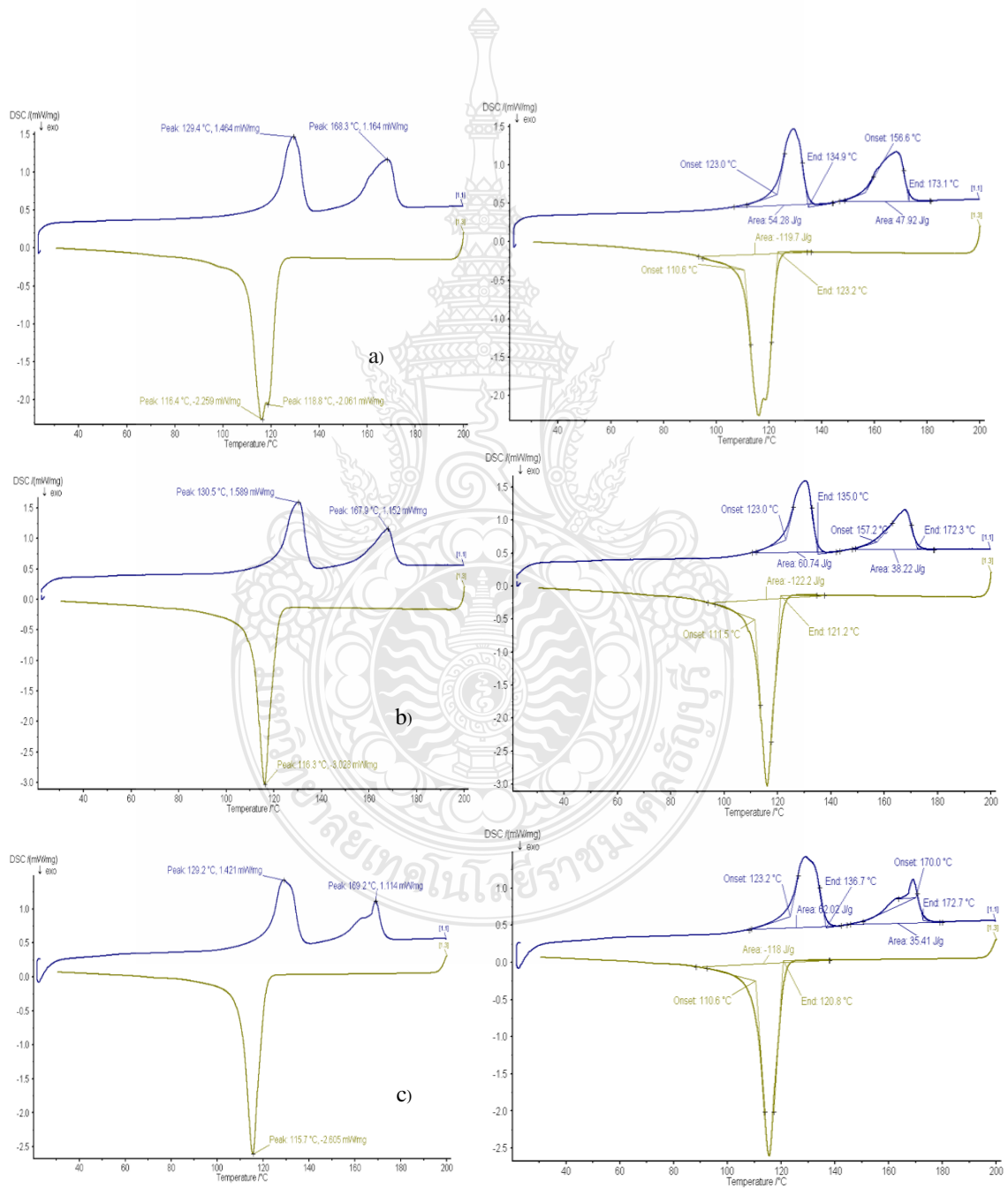


Figure 4.2 The thermal analysis of a) filament of PP/PE at 300 m/min. winding speed, b) filament of PP/PE at 500 m/min. winding speed, and c) filament of PP/PE at 700 m/min. winding speed

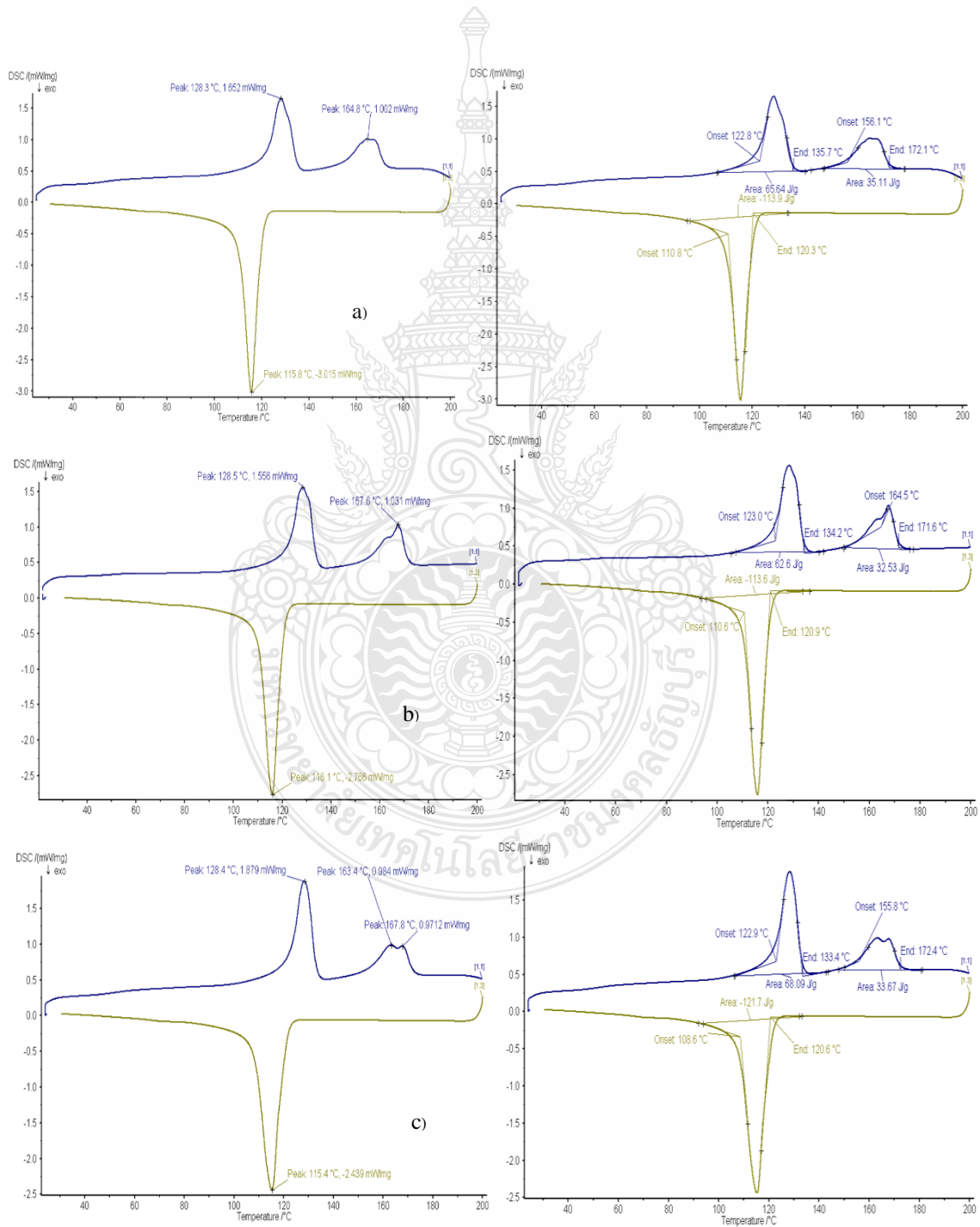


Figure 4.3 The thermal analysis of a) filament of PP/PE with 1.0% TiO₂ at 300 m/min. winding speed, b) filament of PP/PE with 1.0% TiO₂ at 500 m/min. winding speed, and c) filament of PP/PE with 1.0% TiO₂ at 700 m/min. winding speed

The thermal analysis of all of the filament using polypropylene and polyethylene as their base materials with their variety were showed in tables and figures earlier. The fig. 4.4 shows the comparison of the thermal analysis of eight variable filaments

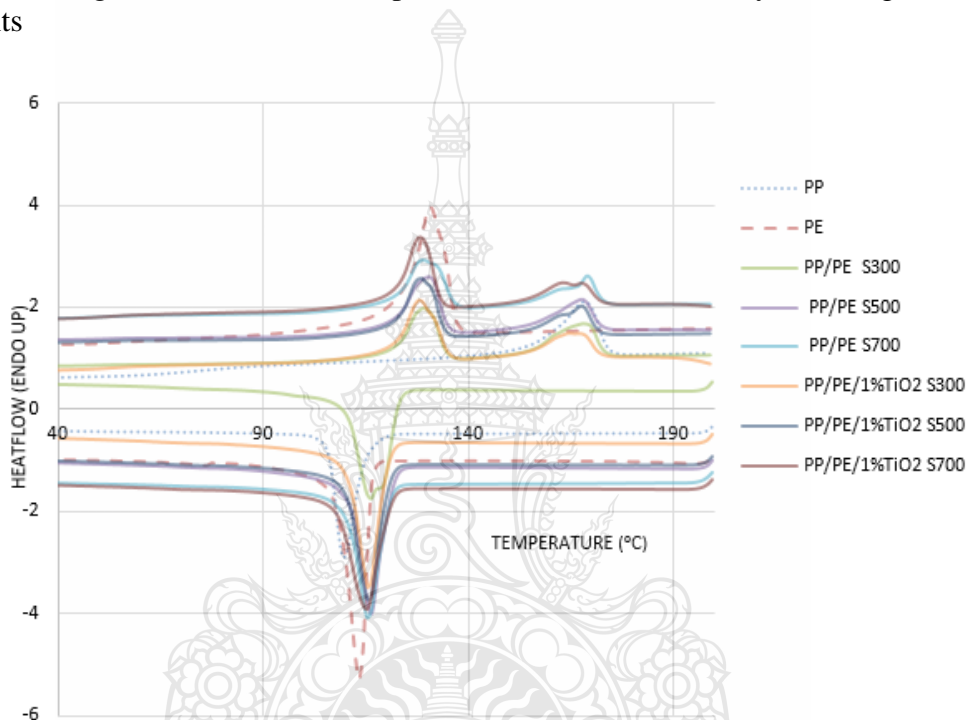


Figure 4.4 The thermal analysis of the eight variable filaments from the experiments

The results of the thermal analysis of the segmented pie bi-component filaments using the selected polyethylene and nylon 6 as base materials and 1.0% TiO₂ as functional additive with alterations of winding speeds from 300 m/min to 700 m/min. shows in table 4.4, presents the onset temperature, melting temperature, ΔH_m , and percentage of crystallinity. The results from the DSC thermal analysis of the two selected base materials with 1.0% TiO₂ functional additive at altering winding speeds at 300 m/min, 500 m/min, and 700 m/min. also shows in fig. 4.5

Table 4.4 The thermal analysis of the segmented pie bi-component polyethylene and nylon 6 filaments with 1.0% TiO₂ as functional additive using with winding speeds at 300 m/min, 500 m/min, and 700 m/min.

| Sample | Winding Speeds | Onset Temperature | Melting Temperature (°C) | ΔH_m (J/g) | Crystallinity (%) |
|--------|----------------|-------------------|--------------------------|--------------------|-------------------|
|--------|----------------|-------------------|--------------------------|--------------------|-------------------|

| | (m/min) | (°C) | | | | | | | |
|----------------------------------|----------|-------|---------|-------|---------|-------|---------|-------|---------|
| | | PE | Nylo n6 | PE | Nylo n6 | PE | Nylo n6 | PE | Nylon 6 |
| PE/Nylon 6 1%TiO ₂ | 300 | 122.5 | 211.1 | 128.1 | 217.2 | 59.85 | 24.1 | 39.9 | 10.4 |
| PE/Nylon 6 1%TiO ₂ | 500 | 112.5 | 203.2 | 128.7 | 213.7 | 60.52 | 21.85 | 40.35 | 9.10 |
| PE/Nylon 6 1%TiO ₂ | 700 | 119.5 | 195.7 | 127.7 | 215.8 | 56.85 | 31.53 | 37.9 | 13.13 |

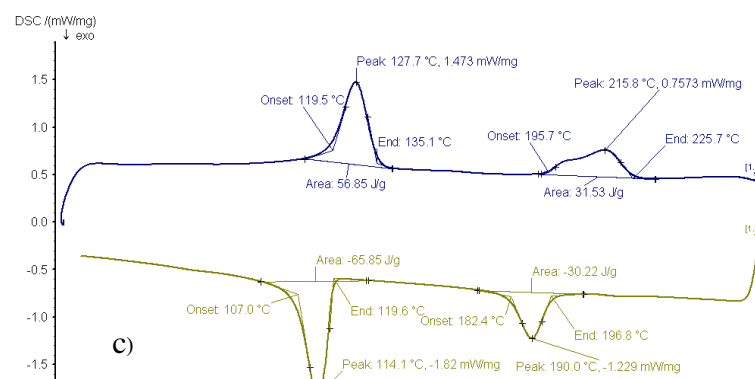
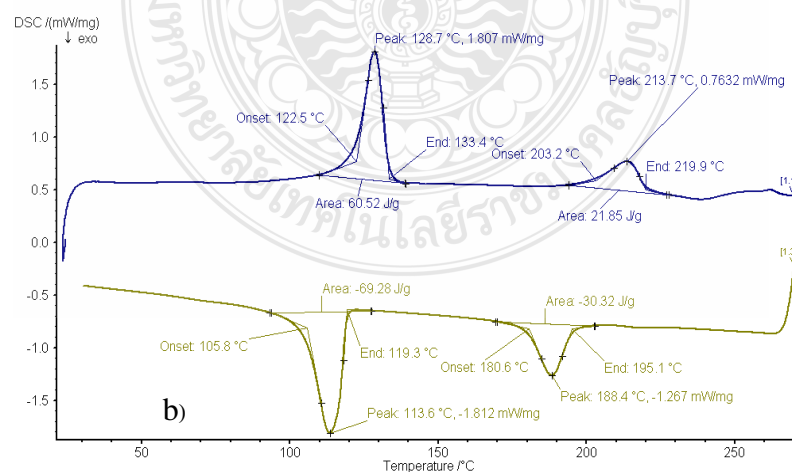
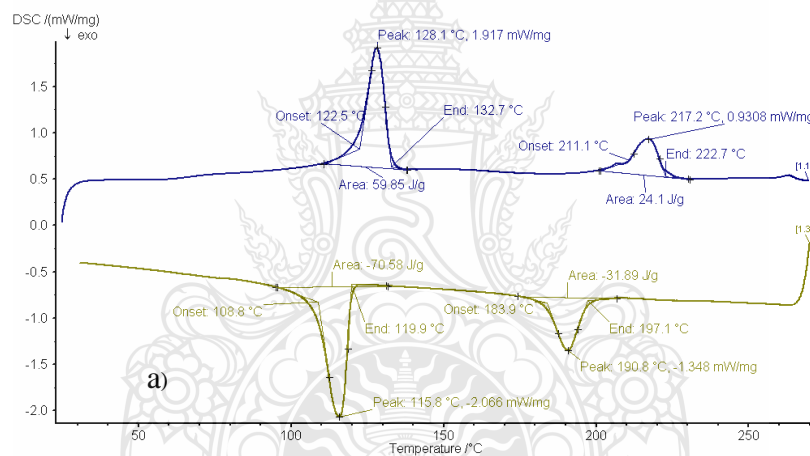


Figure 4.5 The thermal analysis of a) filament of PE/Nylon 6 with 1.0% TiO₂ at 300 m/min. winding speed, b) filament of PE/Nylon 6 with 1.0% TiO₂ at 500 m/min. winding speed, and c) filament of PE/Nylon 6 with 1.0% TiO₂ at 700 m/min. winding speed

4.3.2 Microscopic examination

The microscopic examination of the extruded filaments from the experiments were carried out using light compound microscope with 10x, 40x, and 100x magnifications.

The extruded filament from experiment I to XVI were selected to be subjected to the microscopic examination. The specimens to be examined was random pick from each experiment. The results show in this report would be only the filaments that gave clear microscopic images, because some of the filaments were too dull for the light to transmit throughout the filaments, and some of images show unclear boundary.

The specimen from the experiment I (PP/PE free fall) - segmented pie bi-component fibers using polypropylene and polyethylene, polymer ratio 1:1, no applied winding speed - were examined under light compound microscope. The fig. 4.6 shows the image of shape and geometry of the specimen from the experiment I.

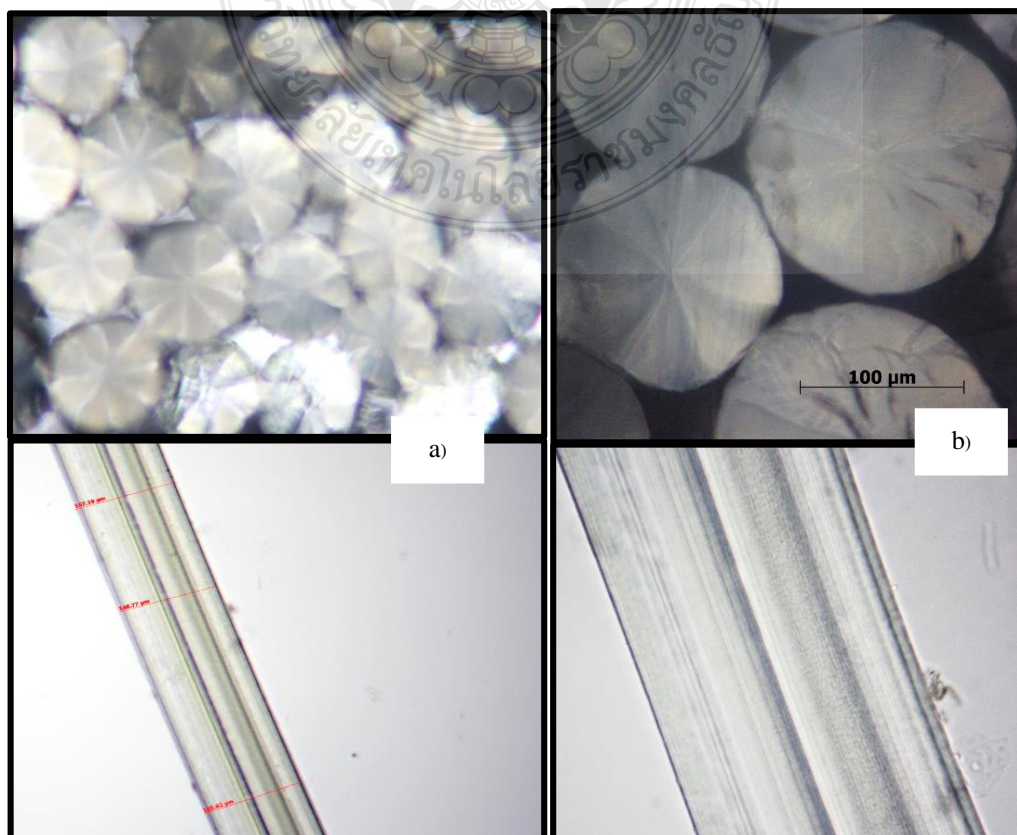


Figure 4.6 The microscopic images of the fibers from the experiment I (PP/PE free fall)
a) cross-section and longitudinal view of PP/PE free fall 10x
b) cross-section and longitudinal view of PP/PE free fall 40x

The specimen from the experiment II (PP/PE 300 m/min) - segmented pie bi-component fibers using polypropylene and polyethylene, polymer ratio 1:1, with 300 m/min winding speed - were examined under light compound microscope. The fig. 4.7 shows the image of shape and geometry of the specimen from the experiment II.

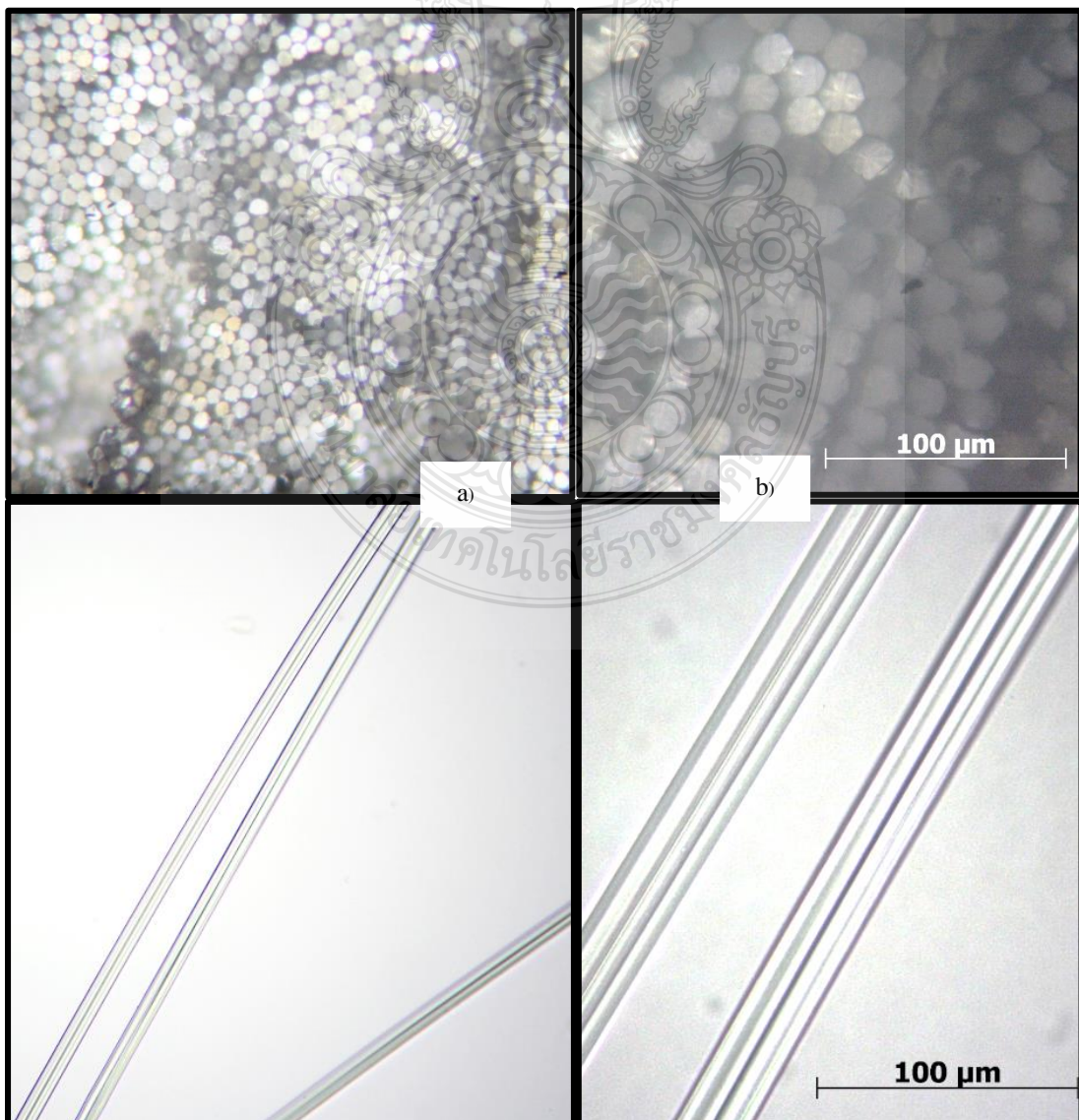


Figure 4.7 The microscopic images of the fibers from the experiment II (PP/PE 300 m/min)

- a) cross-section and longitudinal image of pp/pe 300 m/min with 10x magnification
- b) cross-section and longitudinal image of pp/pe 300 m/min with 40x magnification

The specimen from the experiment III (PP/PE 500 m/min) - segmented pie bi-component fibers using polypropylene and polyethylene, polymer ratio 1: 1, with 500 m/min winding speed - were examined under light compound microscope. The fig. 4.8 shows the image of shape and geometry of the specimen from the experiment III.

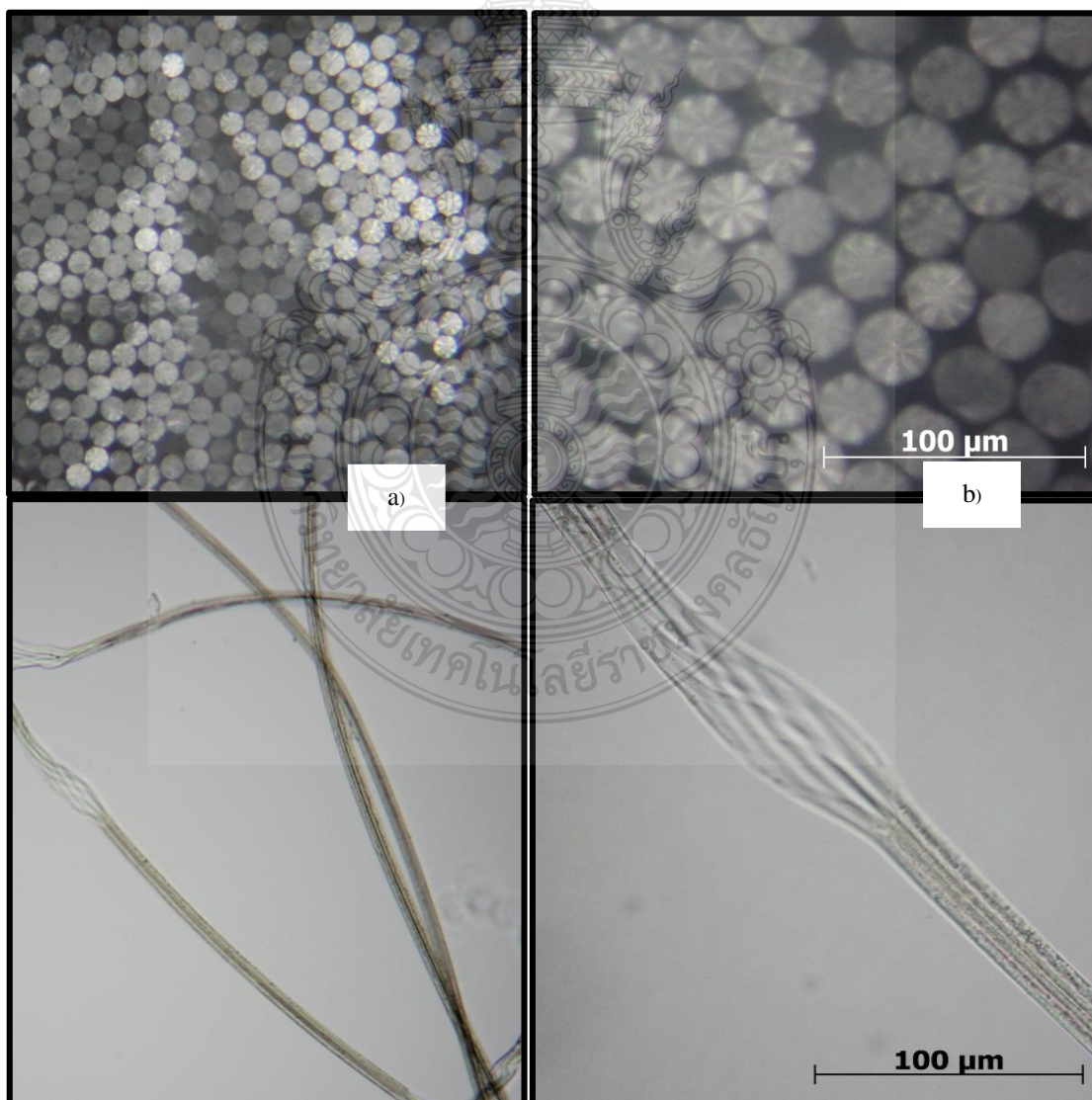


Figure 4.8 The microscopic images of the fibers from the experiment III (PP/PE 500m/min)

a) cross-section and longitudinal view of PP/PE 500 m/min 10x

b) cross-section and longitudinal view of PP/PE 500 m/min 40x

The specimen from the experiment IV (PP/PE 700 m/min) - segmented pie bi-component fibers using polypropylene and polyethylene, polymer ratio 1:1, with 700 m/min winding speed - were examined under light compound microscope. The fig. 4.9 shows the image of shape and geometry of the specimen from the experiment IV.

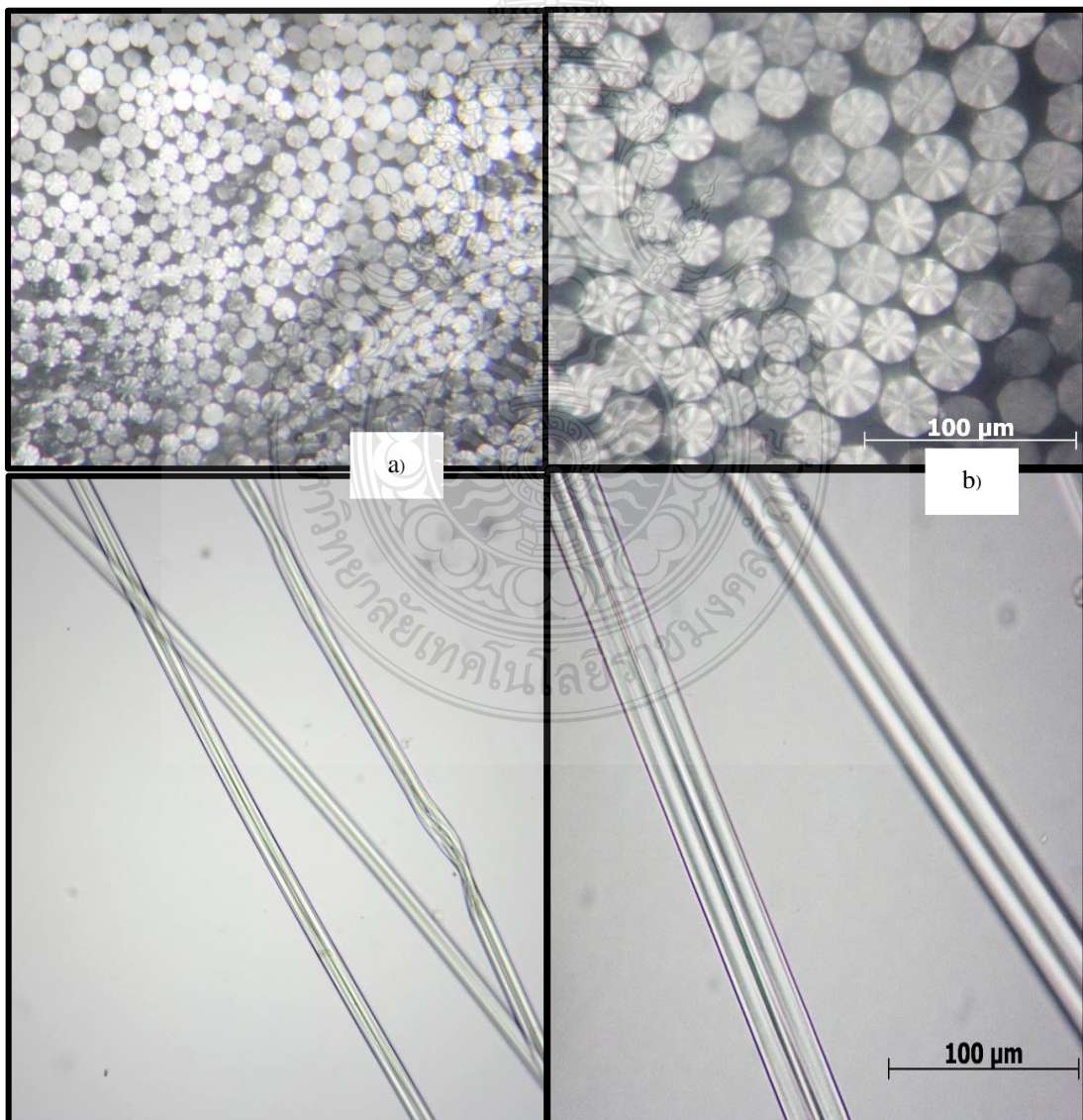


Figure 4.9 the microscopic images of the fibers from the experiment IV (PP/PE 700m/min)
 a) Cross-section and longitudinal view of PP/PE 700 m/min 10x
 b) Cross-section and longitudinal view of PP/PE 700 m/min 40x

The specimen from the experiment V (PP/PE 1% TiO₂ free fall) - segmented pie bi-component fibers using polypropylene and polyethylene, polymer ratio 1:1, without applied winding speed - were examined under light compound microscope. The fig. 4.10 shows the image of shape and geometry of the specimen from the experiment V.

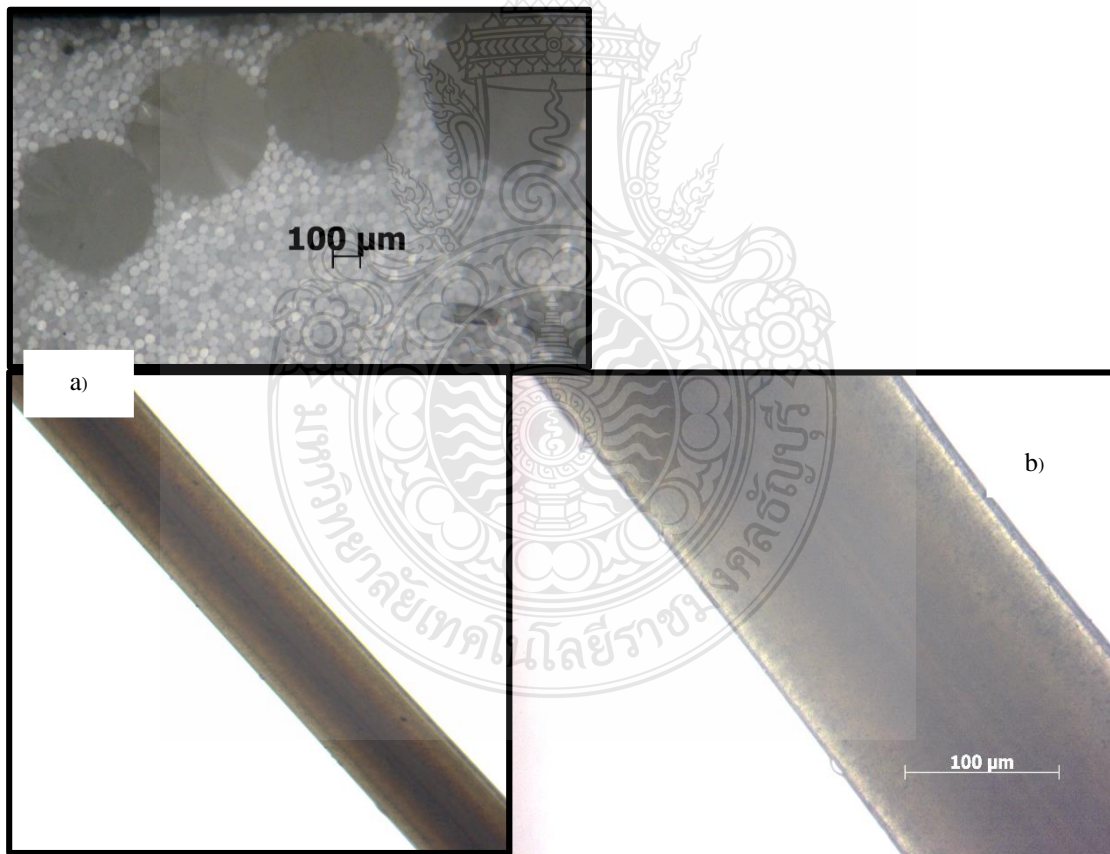


Figure 4.10 The microscopic images of the fibers from the experiment V (PP/PE 1% TiO₂ Free fall)
 a) cross-section and longitudinal view of PP/PE 1%TiO₂ Freefall, 10x
 b) longitudinal view of PP/PE 1%TiO₂ Freefall, 40x

The specimen from the experiment VI (PP/PE 1%TiO₂ 300 m/min.) - segmented pie bi-component fibers using polypropylene and polyethylene, polymer ratio 1:1, with 300 m/min applied winding speed - were examined under light compound microscope. The fig. 4.11 shows the image of shape and geometry of the specimen from the experiment VI.

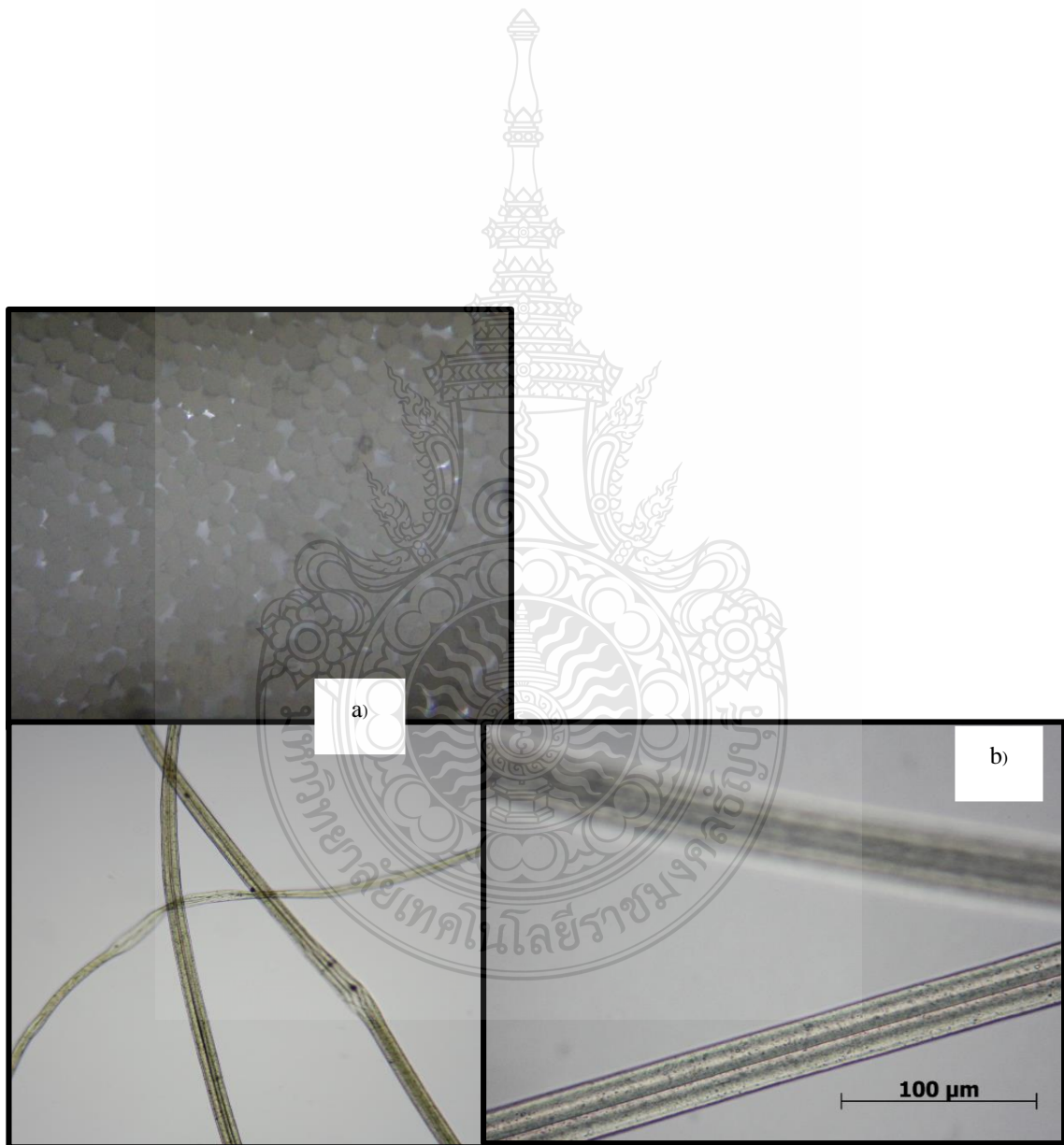


Figure 4.11 The microscopic images of the fibers from the experiment VI (PP/PE 1% TiO₂ 300 m/min)
a) cross-section and longitudinal view of PP/PE 1%TiO₂ 300 m/min, 10x

b) longitudinal view of PP/PE 1%TiO₂ 300 m/min, 40x

The specimen from the experiment VII (PP/PE 1%TiO₂ 500 m/min) - segmented pie bi-component fibers using polypropylene and polyethylene, polymer ratio 1:1, with 500 m/min applied winding speed - were examined under light compound microscope. The fig. 4.12 shows the image of shape and geometry of the specimen from the experiment VII.

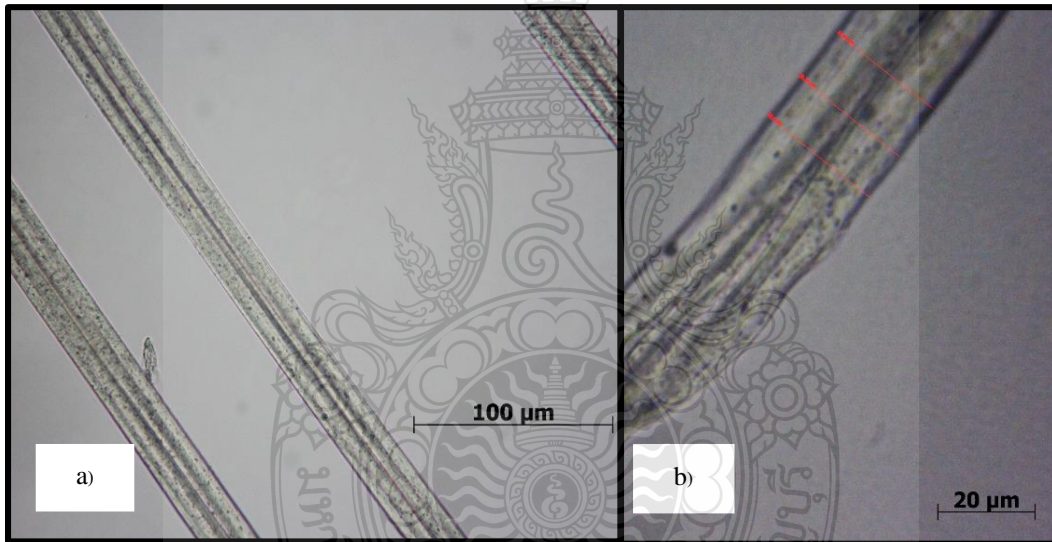
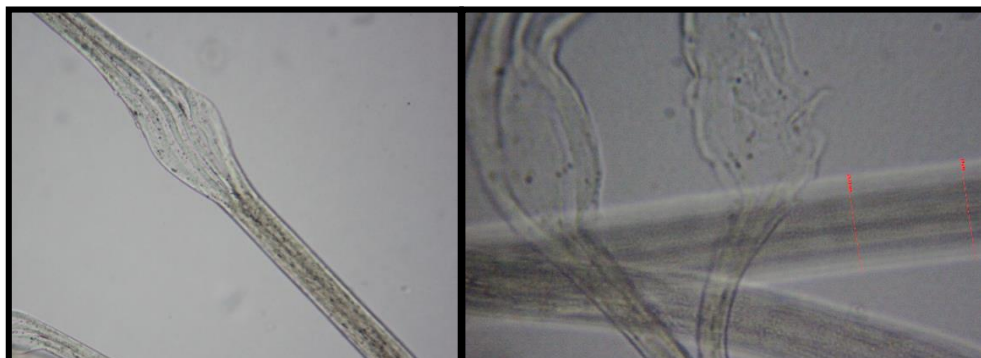


Figure 4.12 The microscopic images of the fibers from the experiment VII (PP/PE 1% TiO₂ 500 m/min)

a) longitudinal view of PP/PE 1%TiO₂ 500 m/min, 40x

b) longitudinal view of PP/PE 1%TiO₂ 500 m/min, 100x

The specimen from the experiment VIII (PP/PE 1% TiO₂ 700 m/min) - segmented pie bi-component fibers using polypropylene and polyethylene, polymer ratio 1:1, with 700 m/min applied winding speed - were examined under light compound microscope. The fig. 4.13 shows the image of shape and geometry of the specimen from the experiment VIII.



a) b)

Figure 4.13 The microscopic images of the fibers from the experiment VIII (PP/PE 1% TiO₂ 700 m/min)
a) longitudinal view of PP/PE 1%TiO₂ 700 m/min, 40x
b) longitudinal view of PP/PE 1%TiO₂ 700 m/min, 100x

The specimen from the experiment XI (PE/ Nylon6 500 m/ min) - segmented pie bi-component fibers using polyethylene and nylon 6, polymer ratio 1: 1, with 500 m/ min applied winding speed - were examined under light compound microscope. The fig. 4.14 shows the image of shape and geometry of the specimen from the experiment XI.

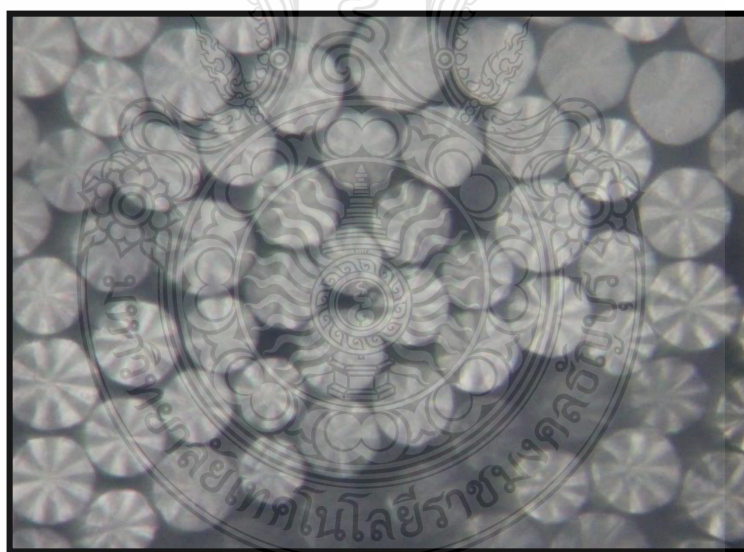


Figure 4.14 Micrograph of the cross-section of the polyethylene/nylon 6 bi-component fibers (100x)

The specimen from the experiment XV (PE/Nylon6 1%TiO₂ 500 m/min) - segmented pie bi-component fibers using polyethylene and nylon 6, polymer ratio 1: 1, with 500 m/ min applied winding speed - were examined under light compound microscope. The fig. 4.15 shows the image of shape and geometry of the specimen from the experiment XV.

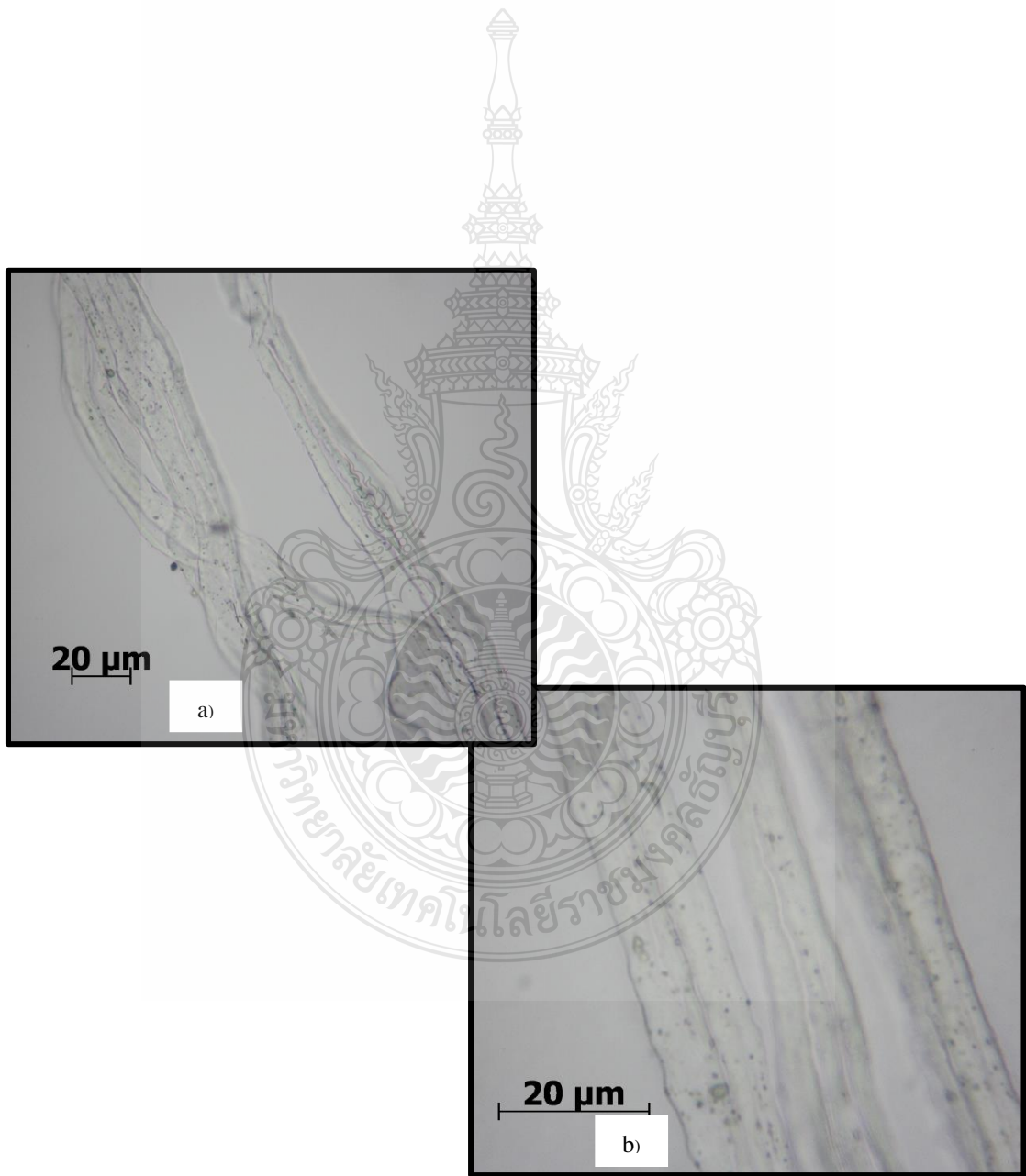


Figure 4.15 The microscopic images of the fibers from the experiment XV (PE/Nylon 6 1% TiO₂ 500 m/min)

- a) longitudinal view of PE/Nylon 6 1%TiO₂ 500 m/min, 40x
- b) longitudinal view of PE/Nylon 6 1%TiO₂ 500 m/min, 100x

4.3.3 Atomic Force Microscopy (AFM)

The surface of the selected filaments were examined using Atomic Force Microscopic technique. The images from this technique showed the surface of small section of the fiber specimen and immersing particles on its surface.

The specimen from the experiment (PP/PE 300 m/min) - segmented pie bi-component fibers using polypropylene and polyethylene, polymer ratio 1: 1, with 300 m/min winding speed, and the specimen from the experiment (PP/PE 1% TiO₂ 300 m/min) – segmented pie bi-component fibers using polypropylene and polyethylene, polymer ratio 1: 1, with 300 m/min winding speed - were examined under Atomic Force Microscope. The fig. 4.16 shows the AFM images of shape and geometry of the specimen from the two experiments.

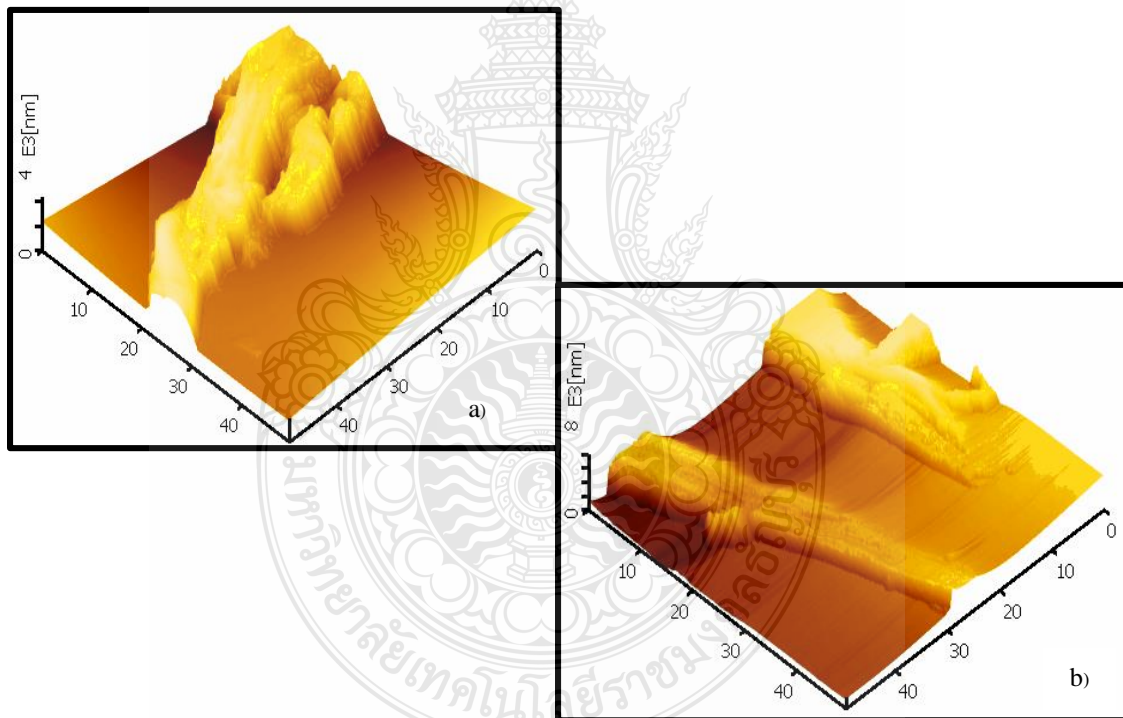


Figure 4.16 The images of the fiber surface using AFM
 a) longitudinal view of PP/PE 300 m/min
 b) longitudinal view of PP/PE 1%TiO₂ 300 m/min

4.3.4 Scanning Electron Microscopy (SEM)

The fiber surface morphologies were inspected using Scanning Electron Microscope (SEM) to deliver ultrahigh resolution at 1.0k magnifications. The Scanning Electron Microscope used in these experiments was JIOL JSM-7610F FE-SEM with EDS detector. The specimen from the experiment II (PP/PE 300 m/min) and the experiment

VI (PP/PE 1% TiO₂ 300 m/min) were examined under the Scanning Electron Microscope. The fig. 4.17 shows the SEM images of shape and geometry of the specimen from the two experiments.

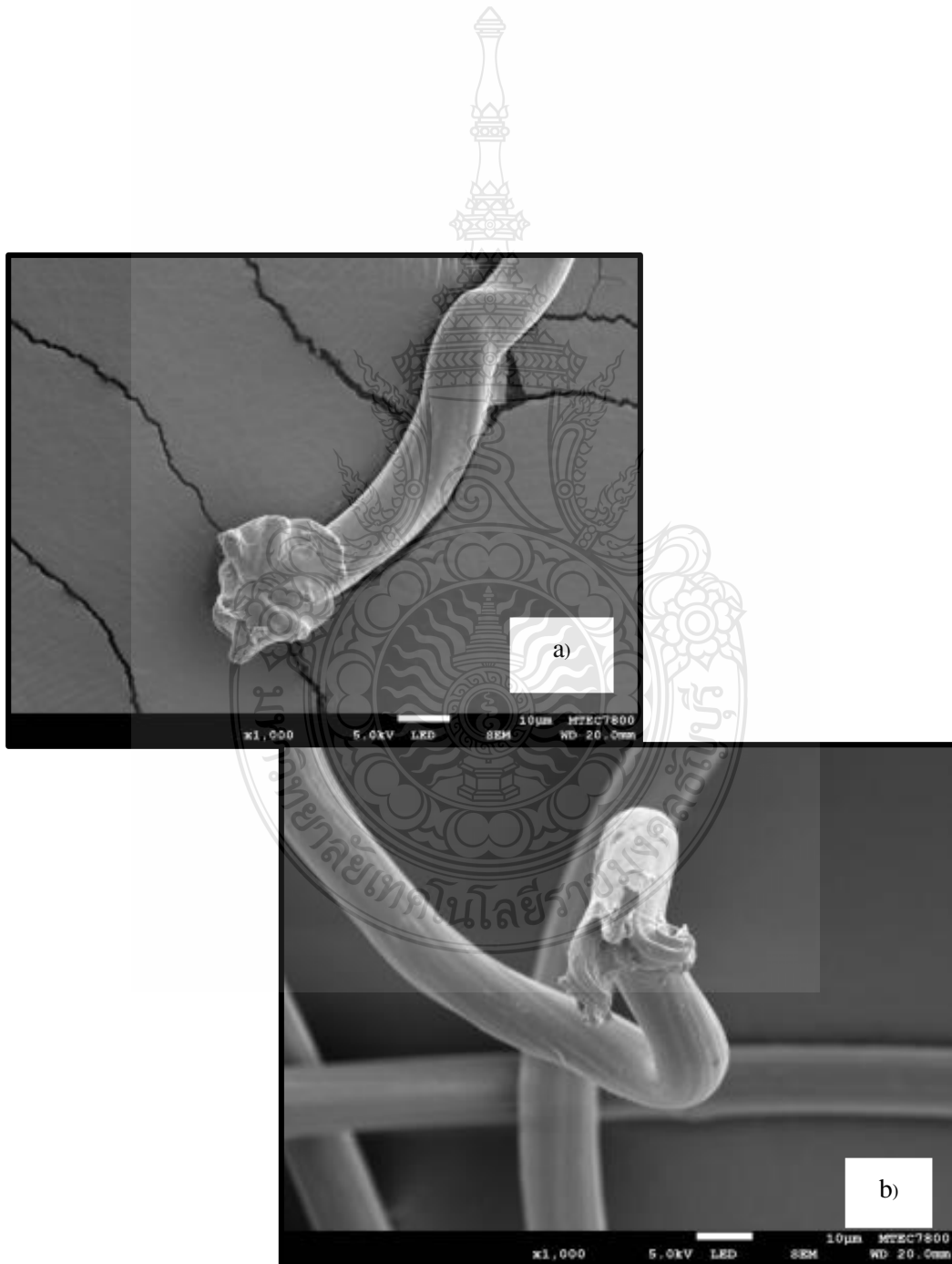
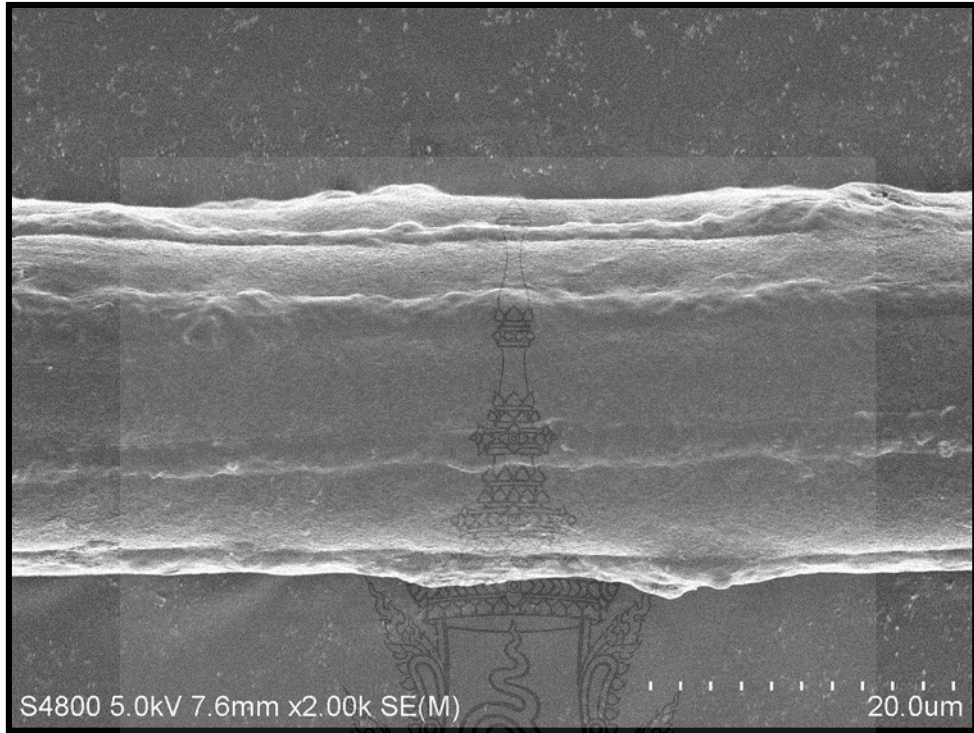


Figure 4.17 The images of the fiber ends using SEM

a) PP/PE 300 m/min



b)
PP/PE

1%TiO₂ 300 m/min



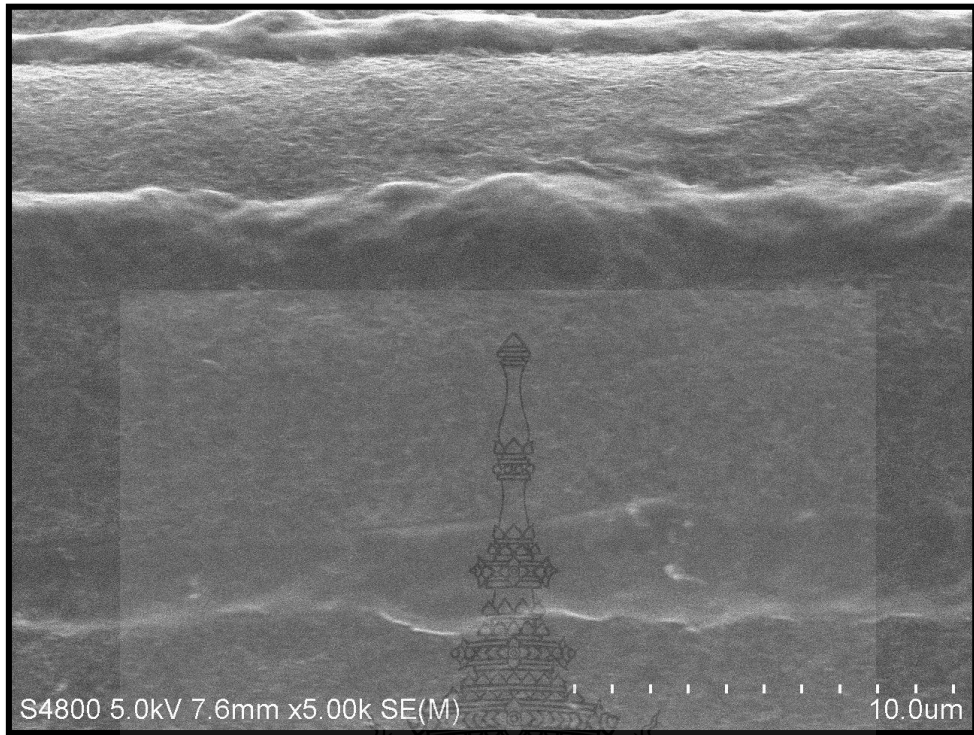


Figure 4.18 SEM image of the segmented pie bicomponent filaments: polyethylene/nylon 6 with 1%TiO₂ winding speed at 500 m/min.

The specimens from the experiment XV (PE/Nylon 6 1% TiO₂ 500 m/min) were examined under the Scanning Electron Microscope. The fig. 4.19 shows the SEM images of shape and geometry of the specimen from the experiments and the fig. 4.20 shows the EDX spectrums of the intact segmented pie bi-component fiber and the partly split segmented pie bi-component fiber.

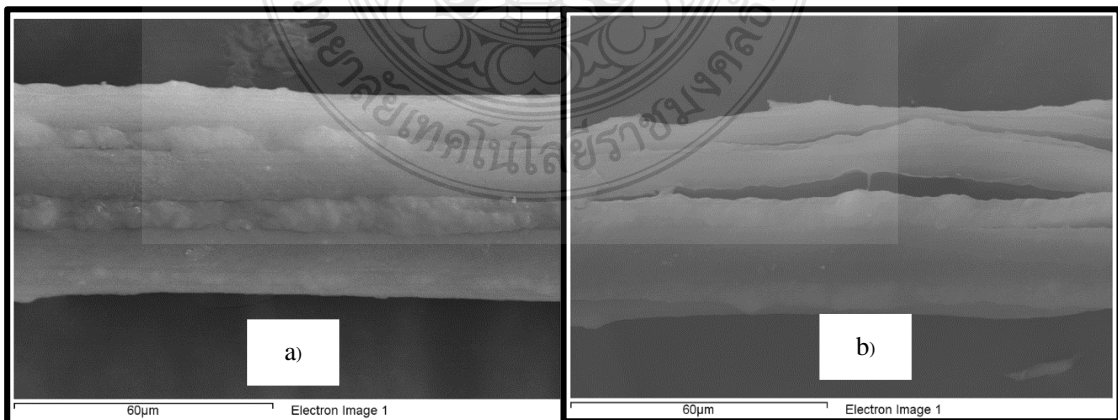


Figure 4.19 The SEM images of the fibers from experiment a) intact fiber b) partly split fiber

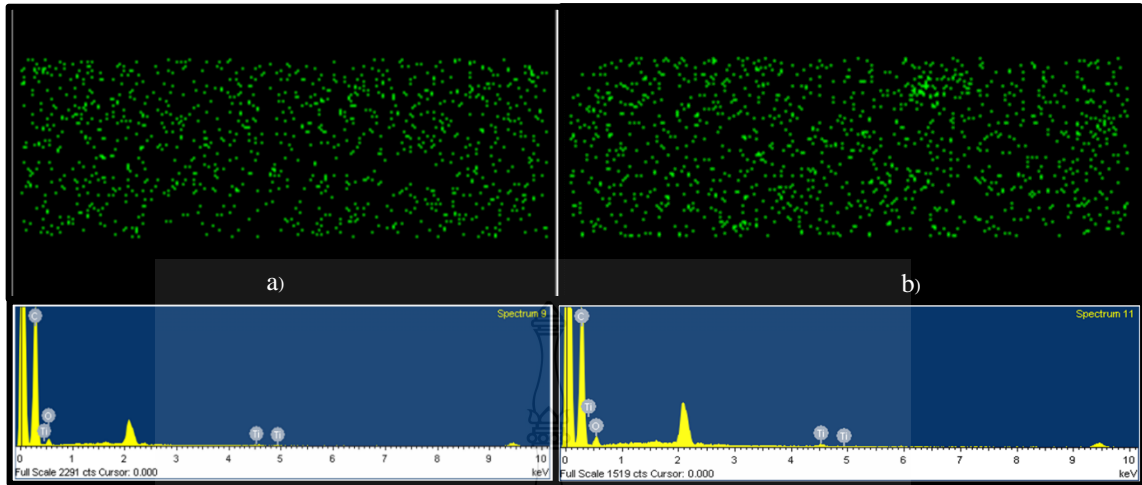


Figure 4.20 The EDX spectrums a) the intact segmented pie bi-component fiber b) the partly split segmented pie bi-component fiber

The specimens from the experiment XV (PE/Nylon 6 1% TiO₂ 500 m/min) were examined under the Scanning Electron Microscope equipped with EDX detector, and the results of the element composition of the intact segmented pie bi-component fiber from the experiment was indicated in table 4.5.

Table 4.5 SEM/EDX: The element composition information of the intact segmented pie bi-component fiber.

| Element | App. Conc. | Intensity Corn. | Weight% | Weight% Sigma | Atomic% |
|---------|------------|--------------------|---------|------------------|---------|
| C K | 5.57 | 1.8246 | 84.31 | 1.22 | 88.20 |
| O K | 0.23 | 0.4240 | 14.69 | 1.21 | 11.54 |
| Ti K | 0.03 | 0.7679 | 1.00 | 0.24 | 0.26 |
| Totals | | | 100.00 | | |

The specimens from the experiment XV (PE/Nylon 6 1% TiO₂ 500 m/min) were examined under the Scanning Electron Microscope equipped with EDX detector, and the results of the element composition of the split segmented pie bi-component fiber from the experiment was indicated in table 4.6.

Table 4.6 SEM/EDX: The element composition information of the split segmented pie bi-component fiber.

| Element | App. Conc. | Intensity Corn. | Weight% | Weight% Sigma | Atomic% |
|---------|------------|--------------------|---------|------------------|---------|
| C K | 3.78 | 1.7175 | 79.42 | 1.47 | 84.55 |
| O K | 0.23 | 0.4502 | 18.72 | 1.48 | 14.96 |

| | | | | | |
|--------|------|--------|--------|------|------|
| Ti K | 0.04 | 0.7713 | 1.86 | 0.33 | 0.50 |
| Totals | | | 100.00 | | |

4.3.5 Cross-section of the fiber

The filaments from a selected experiments were inspected using light compound microscope to examine and measure the cross-section of the randomly selected filaments.

The specimen from the experiment I to experiment IV (PP/PE free fall, and at 300 m/min, 500 m/min, and 700 m/min) were inspected and measure the lengths for their cross-section. The table 4.7 and fig. 4.21 shows the cross-section lengths of the PP/PE filaments from the experiment I to IV.

Table 4.7 The cross-section lengths of the fibers from the experiment I to experiment IV (PP/PE free fall, and at 300 m/min, 500 m/min, and 700 m/min)

| filament | Cross-section length (micron) | | | |
|----------|-------------------------------|-----------|-----------|-----------|
| | freefall | 300 m/min | 500 m/min | 700 m/min |
| 1 | 155.23 | 24.99 | 27.3 | 33.53 |
| 2 | 156.49 | 24.61 | 28.32 | 28.71 |
| 3 | 173.62 | 24.88 | 28.42 | 32.05 |
| 4 | 168.57 | 27.14 | 28.51 | 31.19 |
| 5 | 173.14 | 20.38 | 26.36 | 26.58 |
| 6 | 157.99 | 22.08 | 30.21 | 27.3 |
| 7 | 165.34 | 21.2 | 29.06 | 27.34 |
| 8 | 181.4 | 20.61 | 28.92 | 27.24 |
| 9 | 150.86 | 28.5 | 27.04 | 29.39 |
| 10 | 152.47 | 29.17 | 30.26 | 32.61 |
| average | 163.511 | 24.356 | 28.44 | 29.594 |

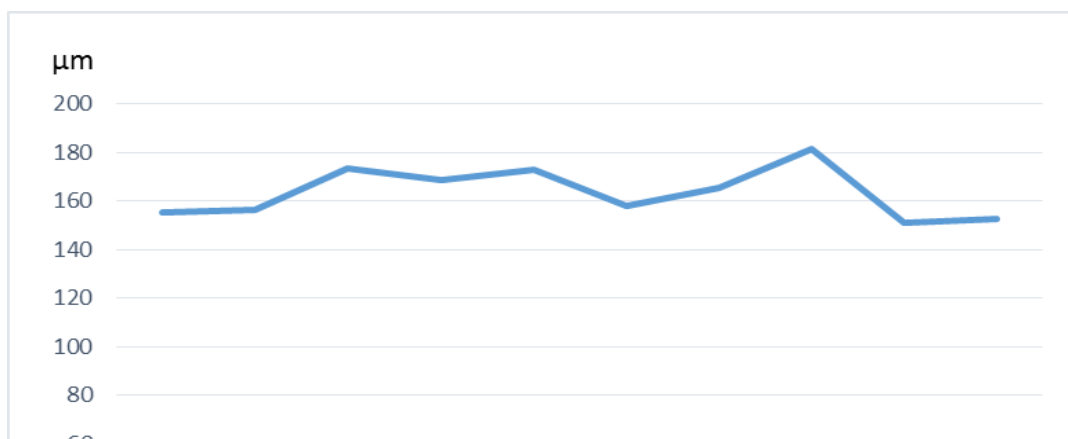


Figure 4.21 The cross-section lengths of the fibers from the experiment I to experiment IV (PP/PE free fall, and at 300 m/min, 500 m/min, and 700 m/min)

The average denier size of the round intact filaments from the experiments were calculated by Eq. (1)

$$d = 11.9\sqrt{\text{denier}/\rho} \quad (4.1)$$

Where: d = diameter of the cross-section (micron)
 ρ = density of the polymer (g/cm^3)

Note:
 ρ of Nylon 6 = 1.14 gram/cm^3
 ρ of Polypropylene = 0.95 gram/cm^3
 ρ of Polyethylene = 0.97 gram/cm^3

The average surface areas of the intact filaments from the experiments were calculated by Eq. (2)

$$\text{Surface area} = 2\pi rL \quad (4.2)$$

The average surface areas of the split filaments from the experiments were calculated by Eq. (3) and (4)

$$\text{Surface area for single strand of split filament} = (2\pi r/16 + 2r)L \quad (4.3)$$

$$\text{Surface area for 16 segmented strands of split filament} = 16*(2\pi r/16 + 2r)L \quad (4.4)$$

Where: r = radius of the cross-section (micron)
 L = length of the filament

The specimen from the experiment I to experiment IV (PP/PE free fall, and at 300 m/min, 500 m/min, and 700 m/min) were calculated for their average size (denier) and average surface areas (intact filament and split filament) and show in the table 4.8 and 4.9 respectively.

Table 4.8 The denier of the fibers from the experiment I to experiment IV (PP/PE free fall, and at 300 m/min, 500 m/min, and 700 m/min)

| PP/PE Filament | freefall | 300 m/min | 500 m/min | 700 m/min |
|---------------------------|----------|-----------|-----------|-----------|
| Average diameter (micron) | 163.511 | 24.356 | 28.44 | 29.594 |
| Radius (micron) | 81.25 | 12.178 | 14.22 | 14.797 |
| Denier of round PP | 179.35 | 3.98 | 5.43 | 5.88 |
| Denier of round PE | 183.13 | 4.06 | 5.54 | 5.99 |

Table 4.9 The average surface areas of the fibers from the experiment I to experiment IV (PP/PE free fall, and at 300 m/min, 500 m/min, and 700 m/min)

| PP/PE Filament | freefall | 300 m/min | 500 m/min | 700 m/min |
|--|-----------|-----------|-----------|-----------|
| Average diameter (micron) | 163.511 | 24.356 | 28.44 | 29.594 |
| Radius (micron) | 81.25 | 12.178 | 14.22 | 14.797 |
| Surface area of intact filament (sq. micron) | 510.25*L | 76.47*L | 89.30*L | 92.93*L |
| Surface area of single strand of split filament (sq. micron) | 194.39*L | 29.136*L | 34.02*L | 34.40*L |
| Surface area of split filament (sq. micron) | 3110.24*L | 466.176*L | 544.32*L | 566.43*L |

Table 4.10 The cross-section lengths of the fibers from the experiment X to experiment XII (PE/Nylon 6 at 300 m/min, 500 m/min, and 700 m/min)

Cross-section length (micron)

| Sample no. | PE/Nylon S300 | PE/Nylon S500 | PE/Nylon S700 |
|------------|------------------|------------------|------------------|
| 1 | 15.05 | 15.35 | 14.4 |
| 2 | 14.66 | 13.03 | 17.89 |
| 3 | 15.44 | 13.34 | 16.92 |
| 4 | 14.45 | 12.58 | 17.81 |
| 5 | 15.11 | 14.13 | 16.2 |
| 6 | 13.92 | 14.51 | 19.28 |
| 7 | 12.93 | 15.88 | 18.98 |
| 8 | 15.2 | 13.59 | 19.46 |
| 9 | 12.92 | 12.75 | 18 |
| 10 | 14.8 | 12.83 | 18.65 |
| 11 | 13.69 | 14.98 | 18.86 |
| 12 | 14.74 | 16.16 | 19.06 |
| 13 | 17.72 | 14.67 | 19.14 |
| 14 | 15.06 | 14.22 | 17.51 |
| 15 | 17.67 | 13.19 | 18.57 |
| average | 14.89 | 14.08 | 18.05 |

4.3.6 Mechanical Property of the fiber

The intact filaments from a selected experiments were inspected and measured for their strength and breaking elongation. The results of the inspection and measuring are shown in table 4.11 and fig. 4.22 and fig. 4.23.

The specimen from the experiment I to experiment IV (PE/Nylon 6 at 300 m/ min, 500 m/ min, and 700 m/ min) comparing with the experiments that used Polypropylene and Nylon 6 at the same applied winding speeds.

Table 4.11 The Strength and breaking elongation of the segmented pie bi-component fiber using polyethylene/nylon 6 and polypropylene/nylon 6

| | PE/Nylon S300 | PE/Nylon S500 | PE/Nylon S700 | PP/Nylon S300 | PP/Nylon S500 | PP/Nylon S700 |
|--------------------|------------------|------------------|------------------|------------------|------------------|------------------|
| Maximum load cN | 3.04 | 1.74 | 1.6 | 6.05 | 3.44 | 1.79 |
| Elongation % | 141.26 | 90.33 | 109.95 | 89.89 | 69.53 | 97.90 |

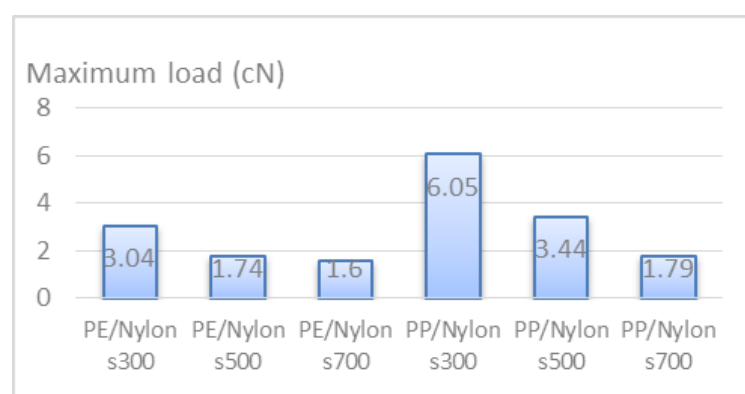


Figure 4.22 The maximum load (cN) of the selected bi-component fibers

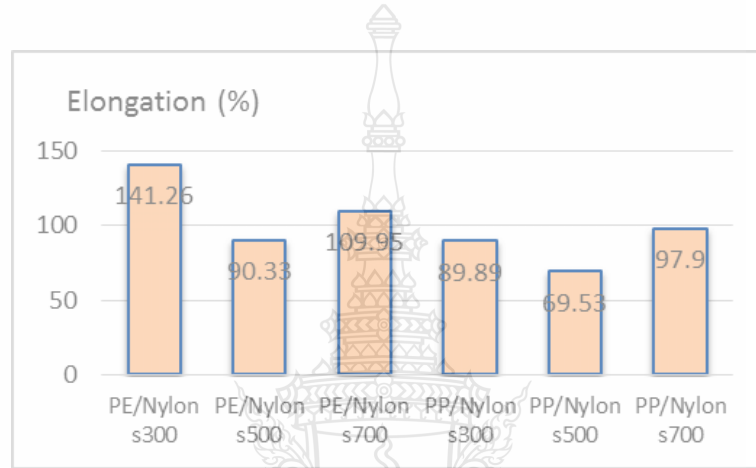


Figure 4.23 The elongation (%) of the selected bi-component fibers

4.3.7 Antibacterial assessment

The filament samples were sent to ISO/IEC17025 accredited laboratory for antibacterial tests. The specimens for some selected filaments were subjected to be assesses their antibacterial activity using the AATCC standard test method 100-2012: Assessment of Antibacterial Finishes on Textile Materials, with designated visible cold light source during the 24 hours incubation. The test bacterias were the gram positive organism, *Staphylococcus aureus*, ATCC No. 6538 and the gram negative organism, *Klebsiella pneumoniae*, ATCC No. 4352. The quantitative procedure evaluated and compared the degree of antibacterial activity after incubation at 35°C for a 24 hours exposure to the test bacterias on the test specimens, then after the incubation, the bacterial challenges were eluted from the swatches and enumerated and the percent reductions were calculated. The tests against each tested microorganism were performed in triplicate. The test specimens were sterilized before testing by using the autoclave at 121°C, 15 psi, 15 minutes. The Light-emitting diode (LED) in the visible light band gap with 330 lumens/sq M was used in 24 hour incubation of the specimens. The distance from the LED to the specimen was set at 25 cm.¶

The report bacterial counts as the number of bacteria per sample and the calculation of percent reduction of bacteria by the formula (1):

$$100 (C - A)/C = R \quad (4.5)$$

Where:

R = % reduction

A = the number of bacteria (CFU/sample) recovered from the inoculated treated test specimen swatches in the jar after '24 hours' contact time.

C = the number of bacteria (CFU/sample) recovered from the inoculated untreated control swatches in the jar at '0 hour' contact time

The results of the inspection and measuring of the specimens from the experiment XV Pare shown in table 4.12 to 4.15 and fig. 4.24 and fig. 4.25.

Table 4.12 Reduction of the number of bacteria recovered from the incubated treated test specimen swatches in the jar at 0 h and 24 h contact time with the bi-component fiber containing 1% TiO₂

| Tested organisms | 0 hour | 24 hours | % reduction |
|--|-----------------------|-----------------------|-------------|
| <i>Staphylococcus aureus</i> ATCC6538 | 2.0 x 10 ⁵ | 4.0 x 10 ⁴ | 80.00 |
| <i>Klebsiella pneumoniae</i> ATCC4352 | 1.7 x 10 ⁵ | 9.7 x 10 ⁴ | 42.94 |

Table 4.13 Reduction of the number of bacteria recovered from the incubated treated test specimen swatches in the jar at 0 h and 24 h contact time with the bi-component fiber without TiO₂

| Tested organisms | 0 hour | 24 hours | % reduction |
|--|-----------------------|------------------------|-------------|
| <i>Staphylococcus aureus</i> ATCC6538 | 2.0 x 10 ⁵ | 1.6 x 10 ⁶ | 0 |
| <i>Klebsiella pneumoniae</i> ATCC4352 | 1.7 x 10 ⁵ | >3.0 x 10 ⁶ | 0 |

Table 4.14 Reduction of the number of bacteria recovered from the incubated treated test specimen swatches in the jar at 0 h and 24 h contact time with the nylon 6 fiber containing 1% TiO₂

| Tested organisms | 0 hour | 24 hours | % reduction |
|--|------------------------|------------------------|-------------|
| <i>Staphylococcus aureus</i> ATCC6538 | 1.38 x 10 ⁵ | 8.00 x 10 ² | 99.42 |
| <i>Klebsiella pneumoniae</i> ATCC4352 | 1.41 x 10 ⁵ | 5.08 x 10 ⁵ | 0 |

Table 4.15 Reduction of the number of bacteria recovered from the incubated treated test specimen swatches in the jar at 0 h and 24 h contact time with the nylon 6 fiber without TiO₂

| Tested organisms | 0 hour | 24 hours | % reduction |
|--|--------------------|--------------------|-------------|
| <i>Staphylococcus aureus</i> ATCC6538 | 1.38×10^5 | 2.21×10^4 | 83.99 |
| <i>Klebsiella pneumoniae</i> ATCC4352 | 1.41×10^5 | 5.15×10^5 | 0 |

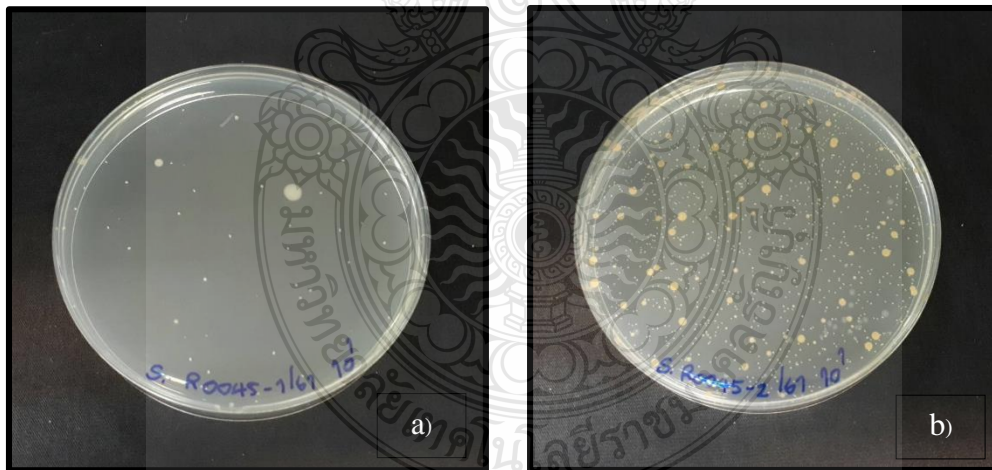


Figure 4.24 The antibacterial efficiency tests of a) the experimental specimen with 1%TiO₂ and b) the experimental specimen without TiO₂, against *Staphylococcus aureus* ATCC6538

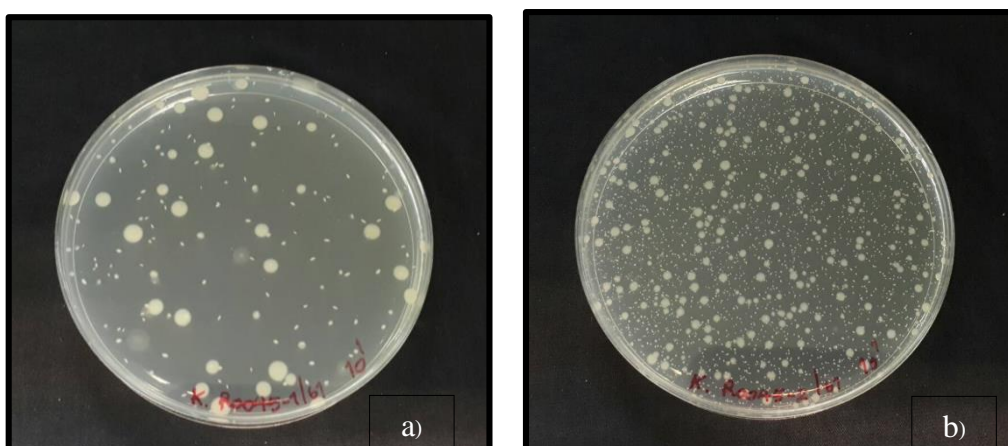


Figure 4.25 The antibacterial efficiency tests of a) the experimental specimen with 1%TiO₂ and b) the experimental specimen without TiO₂, against *Klebsiella pneumoniae* ATCC4352

4.4 Synopsis and discussion of the fiber analysis

The main hypothesis for this research was that the total surface areas of the fibers would significantly increase after the separation mechanism applied. The TiO₂ particles embedded on the inside surfaces of the intact filaments became surface particles, thus promoting a higher degree of photocatalytic. [3,4] Therefore, the experiment analysis were more focus on the degree fiber separation, their surface area, the position of the TiO₂ particles within the fibers, and the degree of antibacterial. [5] A light-emitting diode (LED) in the visible light region was used as the photocatalytic illumination source because it converts electric energy directly into light and employs cold light. [8] The LED was selected to evaluate the antibacterial activity of the filament embedded with 1.0% TiO₂ without additional harm to the bacteria by the heat produced by conventional light sources.[8][11] Besides, the experiments were intended to evaluate the antibacterial activity of the textile for indoor use, where there is limited UV light; and the LEDs are now common in household lamps.[13][14] The nanoparticles of TiO₂ were embedded in the bi-component fibers during the extrusion stage as the particles would disperse thoroughly inside and on the surface of both component segments of the fibers. When the intact bi-component fibers were separated into smaller pie-slice-shaped fibers by mechanical force thus the total surface areas of the split fibers were significantly increased; and the examinations clearly indicated that the particles embedded on the inside surfaces of the intact filaments became surface particles. The photocatalytic material was intended to break down organic compounds under common visible illuminated light for potential indoor and household uses. Photocatalytic surfaces require a certain light intensity, but with the presence of TiO₂, the required energy to induce photocatalytic is small.[11] The selected photocatalytic materials would enhance properties such as self-sterilization, air and water purification, and stain-repelling. To prove that the bi-component fibers with 1.0% embedded TiO₂ from the experiments were subjected to antibacterial tests against the Gram-positive *Staphylococcus aureus* and Gram-negative *Klebsiella pneumoniae* bacteria, using the American Association of Textile Chemists and Colorists (AATCC) test method 100-2012: Assessment of Antibacterial Finishes on Textile Materials. The antibacterial test results of the bi-component fibers were compared with those for single-polymer filaments with and without TiO₂ nanoparticles. The tests were modified by using a designated visible light for 24 h of incubation.[3,7, 9,19,17]

The cross-section microscopic images of the filaments from light compound microscope (fig. 4.6) show distinct boundary of the segments of different polymers within filaments. The filaments were intact as the images show that all the segments attach themselves to the group. The longitudinal images of the filaments also show the distinct line along the lengthwise of the filaments which indicates the boundary of different polymers. The filaments were colourless and transparent that the light of the microscope passed throughout them. The average diameter of the filaments from the experimental I is 163.511 micron (table 4.7), which is considerably big. The images of the segmented pie bi-component fibers in fig. 4.7 show the filaments in 10x magnification and 40 x magnification. The images indicate that the fiber segments in a single filament were still intact with segment boundary dim lines across the cross-section of the filaments and distinct line along the lengthwise of the fibers. The average cross-section size of the filaments was reduced significantly from the experiment without the applied winding mechanism indicates that the winding mechanism at 300 m/min. had an impact on the size of the extruded filament as expected. The average size of the cross-section of the fiber was reduce to 24.356 micron which is approximately 85% reduction of the filaments without winding mechanism. However, the images do not show any indicator of the effect of the winding mechanism on separating segments in the filaments. The images taken from the light compound microscope show in fig 4.8 and fig. 4.9. The filament cross-section images reveal the definite borderlines of each segment within one filament, with different degree of light transmission of the two base polymers. The longitudinal images of the filaments from the two experiments show clear and fine lines along the lengthwise axis of the filaments. The images of the longitudinal axis also show some section of the filaments that polymer segments detached or deformed lengthwise. The average cross-section diameter of the filaments from the two experiments were not significant different, 28.44 micron and 29.594 micron respectively, which means that the faster winding speeds than 300 m/min. had less effect on the size of the filaments. Table 4.7 and fig. 4.21 show the average size of the filaments measured in cross-section axis, table 4.8 shows the average denier of the fibers, and table 4.9 shows the average surface areas of the fibers. The fibers where the winding speeds were applied show insignificant differences between each winding speed, but the size of the filaments were decrease significantly when the winding speed applied compared to the filament with free fall mechanism. The average denier of the filaments from experiment II (PP/PE 300 m/min) was decrease to 3.98 denier from 179.35 denier of the filaments compared to the filaments from the experiment I (PP/PE free fall), which decreased approximately 4.5k%. The cross-section microscopic images of the filaments without applied winding mechanism from the light compound microscope in fig. 4.10 show cloudy round fiber that the light could hardly pass through the inner part of them, so the boundary line of each segment were obscured. The fibers appear to be bulky similar to the fibers from the similar experiment but free from particles of TiO₂, as they both were not subjected to the tension of the winding mechanism at the end of the process. The boundary lines of the segments that appeared in the filaments from the experimental I were not visible in the filaments from the similar conditioned experiment but with particles of 1% TiO₂, denoted that the particle of the photocatalytic agent were dispersed throughout the filaments. The filaments were still intact as the images show that all the filaments were round shape.

The images of the segmented pie bi-component fibers from the experiment VI (PP/PE 1%TiO₂ at 300 m/min.) show in fig. 4.11, the experiment VII (PP/PE 1% TiO₂ at

500 m/min.) show in fig. 4.12 and the experiment VIII (PP/PE 1% TiO₂ at 700 m/min.) show in fig. 4.13. The cross-section images were dusky ill-defined shape as the light was faintly pass through the filaments. However, the longitudinal image clearly shows the segment boundary lines along the lengthwise of the filaments and the particles of the photocatalytic agent appear all over the filaments. The images also show that some small parts of the filaments were deformed or the segments within the filaments seemed slightly split. The images of the filaments from the three experiments show the similar prospects such clear boundary lines along the lengthwise of the filaments, the photocatalytic particle densely distribute all over the filaments. [17] Some small parts of the filaments were deformed and split lengthwise, which clearly appears in fig. 4.13. indicate the positive sign of filament splitting.

The optical microscopic images of the extruded fibers of the experiment XV (PE/Nylon6 1%TiO₂ 500 m/min.) were taken with an optical microscope at 40× and 100× magnifications. The sample preparations were follow the plate method in reference [29]. However, the cross-sectional axial images of the fibers with nanoparticles of 1.0% TiO₂ were poorly defined because of the low light transmittance through polymer materials clouded with light-absorbing particles. Future attempts in obtaining undimmed cross-section images of such materials should probably use different sample preparation techniques, such as cutting the specimens into thinner slices. However, the cross-sectional axial images of the bi-component fibers with the same materials but without the added TiO₂ particles, using the same specimen preparation techniques, showed clear boundaries between the two materials within the filaments, as shown in fig. 4.14.

The longitudinal axial images of the fibers are shown in figure 4.15. Figures 4.15 (a) and (b) apparently indicate that the bi-component fiber separation mechanism was successfully executed, as the intact fiber strands are split into several smaller individual fiber strands. [6] The efficacy of the separation mechanism applied to the intact filaments is confirmed. The micrographs also show that the particles of TiO₂ are distributed homogeneously within the filaments; some particles are scattered near the surfaces, while others are adhered to the surfaces. The split fiber strands were uneven in length; therefore, the cross-sections of the split fibers were expected to be irregular.

The calculation of the surface areas of the filaments was showed in table 4.8 which indicates the surface areas of all the filaments from the experiment I to IV. The surface areas of the intact filaments compare to the surface areas of the split filaments indicate that the surface area of a 16-pie-shape-segments of a split filament have significant higher surface area than a round intact filament. The surface area of 16-pie-shape-segments of a split filament from experiment II was $466.176 * L$ where the surface area of a round intact filament was $76.47 * L$, which means that the surface area of the split filaments was increased approximately 609.62%. Therefore, the calculation of surface areas of filaments as 16-pie-shape-segments of a split filament shows remarkably increase comparing to a round intact filament.

The assessment of the polymer percent crystallinity were performed using differential scanning calorimetry. Some specific extruded filaments from experiment I to XVI were subjected to the thermal analysis test. The base materials in the first attempt of the experiment were polypropylene and polyethylene. The results of the thermal analysis of filaments using the first two base materials (PP, PE) were presented in the table 4.1. The table shows the onset temperature, melting temperature, ΔH_m , and percentage of crystallinity of the filament of the two materials. The results from the DSC thermal

analysis of the two selected base materials also shows in fig. 4.1. The results of the onset temperatures, melting temperatures, ΔH_m , and percentage of crystallinity from the DSC analysis of polyethylene and nylon 6 with the three winding speed varieties are display in table 4.4. The DSC curves of the three variations of the bi-component filaments display in fig. 4.5.

The segmented pie bi-component filaments of polypropylene and polyethylene without applied winding mechanism had melting temperature at 168.5°C and 130.9°C respectively, while the average melting temperatures of the filaments with 300 m/min, 500 m/min, and 700 m/min. were 168.47°C for polypropylene and 129.7°C for polyethylene, as show in the table 4.1 and fig. 4.1. The winding mechanism at the different speeds did not have significant impact on the melting temperature of the both materials. The similar results, in the table 4.3 and fig. 4.3, also point that the different winding speeds had little effect on the melting temperature of the both materials. However, the melting temperature of the filament with the additive (TiO₂) were slightly lower in both sides of the materials. The percentages of the crystallinity of the polypropylene and polyethylene bi-component fibers without winding mechanism were 36.85 and 51.02 respectively, while the percentages of the crystallinity of the two sides of the filaments were inconclusive as the percentages of the polypropylene side at 300 m/min, 500 m/min, and 700 m/min. were 23.15, 29.34 and 17.11, and the percentages of the polyethylene side were 18.46, 13.00, and 10.89 respectively. The results obviously show fluctuation and did not correspond with the speeds of the winding mechanism. However, the percentages of crystallinity of the filaments with TiO₂ additive reveal different directions for the polyethylene which increased when the speed increase. The crystallization temperatures of the filaments of polypropylene and polyethylene without applied winding mechanism were 109.8°C and 113.6°C respectively, while the crystallization temperatures of the filaments of the same materials with winding mechanism at 300 m/min., 500 m/min., and 700 m/min., in fig. 4.2, were almost at the same peak which is between 115.7°C to 118.8°C. The filaments with TiO₂ additive shows the single peak for all the three applied winding speeds between 115.4°C to 116.1°C as show in fig. 4.3.

On the other hand, the thermal analysis of the filaments using nylon 6 and polyethylene as base polymers adding with 1% TiO₂, in the table 4.4 and fig. 4.5, shows different indications. The melting temperatures of the nylon 6 were fluctuated between 213.7°C to 217.2°C (approximately 3.5°C differences) in different winding speeds, while the melting temperatures of the polyethylene were less fluctuated (between 127.7°C to 128.7°C, approximately 1.0°C differences). The percentage of the crystallinity of the both base materials were fluctuated approximately 2.45% for polyethylene sides and 4.3% for the nylon 6 sides. The most significant indicator for the thermal analysis was the crystallinity temperatures of the filaments using polyethylene and nylon 6. The crystallinity temperatures of the two base materials adding with 1% TiO₂ show two peaks in all the three winding speed varieties. The differences of the temperatures between the two peaks were between 74.8°C to 75.9°C, indicating that the nylon 6 segments were solidified before the polyethylene segments.

Comparing the crystallinity temperatures of the filaments using polypropylene/polyethylene and nylon 6/polyethylene as base materials, the graphs of thermal analysis show one peaks of crystallinity temperatures for PP/PE, and two peaks with great distances between them in the latter. The crystallinity temperatures indicated

that the differences between peaks underlined the splitability of the segmented pie bi-component fibers. The filaments using nylon6/polyethylene clearly split easier than the polypropylene/polyethylene filaments.

The Scanning Electron Microscope (SEM) delivered images of the surface morphologies of the filaments at ultrahigh resolution at 2.0k magnifications. The images in fig. 4.18 a) shows the surface morphology of polyethylene/nylon 6 with 1% TiO₂ speed 500 m/min and fig. 4.18 b) shows the surface morphology of polypropylene/nylon 6 with 1% TiO₂ speed 500 m/min. The images display distinct segment boundaries of the two polymers. The SEM images of the polyethylene/nylon 6 with 1.0% TiO₂ in figure 4.19 a) as an intact fiber and figure 4.19 b) as a partly split fiber show that a single fiber of the experimental segmented- pie bi- component fibers with polyethylene in eight small segments and polyamide in another eight segments is successfully created, reflecting both production feasibility and the efficacy of the splitting mechanism. The SEM image in figure 4.19 a) shows the distinct segment boundaries visible on a single extruded segmented- pie bi- component fiber before separation, with differences in surface appearance distinguishing the segments. The surfaces of the segments of each base polymer show noticeably different characteristics, as one polymer base is smoother and thicker than the other. The segmented fiber after separation, in figure 4.19 b), shows that each segmented component is smaller in size than the intact fiber; the split fiber strands are also irregular, affirming the observations from optical microscopy.

SEM with EDX was used to identify and qualify the elemental composition information of the intact and split fibers. EDX was used to define areas on the specimen surfaces, as shown in figure 4.20 a) and 4.20 b) of the intact and split fibers, respectively. The figures show peaks of the X-rays given off as electrons returning to the K electron shell; one of the detected peaks in figure 4.20 a) and b) corresponds to Ti. The presence of Ti on the detected surface of the split fiber is much higher than that on the intact fiber. The atomic percentage of the Ti on the split fiber surface was 0.50, while that on the intact fiber was 0.26, indicating a 92% increase. The weight percentage of the Ti on the split fiber surface was 1.86 while that on the intact fiber surface was 1.00, an increase of 86%. The qualitative and quantitative elemental composition information of the EDX is shown in tables 4.5 and 4.6 for the intact and split segmented- pie bi- component fibers, respectively.

The segmented- pie bi- component fibers of polyethylene and nylon 6 with and without 1.0% TiO₂ were tested using the AATCC Test Method 100-2012: Assessment of Antibacterial Finishes on Textile Materials, with a designated visible cold light source during 24 h incubation. [7,13,15,24] The modified antibacterial test method was based on the earlier research on the antibacterial performance of photocatalyst thin film fabricated by defection effect in visible light and visible-light-responsive nano-TiO₂ with mixed crystal lattice and its photocatalytic activity, both indicated that the visible light could enhanced the photocatalytic activity of the material using TiO₂ as photocatalytic agent. [8,11] The single polymer nylon filaments with and without TiO₂ were tested likewise for the comparison of the photocatalytic surface areas for intact fibers and split fibers.

The antibacterial activity against *Staphylococcus aureus* of the segmented- pie bi- component fiber with 1.0% TiO₂ is presented in figure 4.24 a); that of the fiber without TiO₂ is presented in figure 4.24 b). The figures show significant reductions of the tested bacteria in the sample containing TiO₂. The percentage of reduction of *Staphylococcus*

aureus recovered from the incubated treated test specimen swatches in the jar at 0 h and after 24 h contact time of the experimented bi-component fiber with the presence of 1.0% TiO₂ is shown in table 4.12. The percentage of the Gram-positive bacteria reduction (*Staphylococcus aureus*) in the sample with TiO₂ was 80.00% compared to 0% in the sample without TiO₂. The antibacterial activity against *Klebsiella pneumoniae* of the fiber with 1.0% TiO₂ is presented in figure 4.25 a) and that of the fiber without TiO₂ is presented in figure 4.25 b). The figures also show noticeable reduction of the tested bacteria in the sample with the presence of TiO₂. The percentage of reduction of the number of *Klebsiella pneumoniae* recovered from the incubated treated test specimen swatches in the jar at 0 h and after 24 h contact time of the experimental bi-component fiber with the presence of 1.0% TiO₂ is shown in table 4.14. The percentage of the Gram-negative bacteria reduction (*Klebsiella pneumoniae*) in the sample with TiO₂ was 42.94% compared to 0% in the sample without TiO₂.

The antibacterial assessments for the nylon filaments with and without TiO₂ showed different results. The percentage of reduction of the number of *Staphylococcus aureus* recovered from the incubated treated test specimen swatches in the jar at 0 h and after 24 h contact time of the nylon fiber with 1.0% TiO₂ is shown in table 4.14. The percentage of reduction of the number of *Klebsiella pneumoniae* recovered from the incubated treated test specimen swatches in the jar at 0 h and after 24 h contact time of the nylon fiber without TiO₂ is shown in table 4.15. The percentage of the Gram-positive bacteria reduction (*Staphylococcus aureus*) in the nylon sample with TiO₂ was 99.42% compared to 83.99% in the sample without TiO₂. The percentage of the Gram-negative bacteria reduction (*Klebsiella pneumoniae*) in the nylon samples was 0% for both samples. The higher reduction of the Gram-positive bacterial in nylon with and without added TiO₂ arises from the presence of small amounts of TiO₂ microparticles in the semi-dull grade nylon polymer chips used in the fiber spinning. However, the comparison test results of the bi-component fibers against the nylon fibers are inconclusive, as the numbers of bacteria recovered from the inoculated untreated control swatches in the jar at 0 hour are distinctly different, and the antibacterial assessment tests of the bi-component fibers used 0.7 grams of fiber, while that of nylon used 1.5 grams of fiber. The amount of fibers used in the antibacterial tests were not specified in the standard test method, which instead stated only to use the amount of specimen that absorbed 1.0 ± 0.1 mL of inoculum and left no free liquid in the jar. Therefore, the doubled mass of nylon fiber used in the tests relative to that of the bi-component fiber could be the reason underlying the higher degree of reduction of the Gram-positive bacteria (*Staphylococcus aureus*).

CHAPTER 5 CONCLUSION

A series of laboratory and experimental works have been sequentially conducted to produce splittable segmented pie bi-component fibers that is suitable for textile material for healthcare or medical applications. The experiments were carried out sequentially following the results of the previous experiments. The results from the earlier experiments showed the possibility of producing and spinning of splittable segmented pie bi-component fibers using selected base polymers. The spinning process were emphasized on producing minuscule fibers using a two extruders system. The experiments were also designed to investigate the morphologies and characteristics of the fibers and the particles fastened inside and on the surface of the injected filaments before and after the applied bi-component fiber separation mechanism. The separation mechanism was to be applied to the bi-component filaments for specific purpose of increasing active surface area of the lengthwise-split filaments. The variations of segmented pie bi-component fibers using polyolefin (PP, PE) and Nylon 6 with and without 1% TiO₂ master-batch as base materials were produced and their characteristics were profoundly studied and examine.

The emphasis of the research was to produce small-scale diameter fiber and embedded nanoparticles of titanium dioxide (TiO₂) within the two components of the fibers. The spinning of the filaments were successfully conducted using different combinations of base materials. The selected materials were applicable to the process and process conditions were manageable. Adding titanium dioxide particles in the extrusion stage was to have their particles dispersed thoroughly inside and on the surface of the both components of the extruded filaments. The images from optical microscope and SEM which clearly showed the distinct segments of the two polymers. The fibers without TiO₂ appeared to have higher transparency than the fibers with TiO₂ which clearly showed small dark particles scattering around the fibers. This means that adding the TiO₂ particle using master batches of the both sides of the base materials at the fiber extrusion stage was applicable and the particles dispersed all over the fiber, inside and surface. However, the images from SEM and AFM could not give the conclusive evidences for TiO₂ particles on the fibers' surfaces. The production of the fibers embedded with 0.1% TiO₂ in some of the 16 experiments did not show significant production differences with the ones without TiO₂. Therefore, the particles of the TiO₂ did not burden the productions.

The application winding mechanism at the end of the process with different speeds indicated maximum stretch that the experimented filaments could withstand. The applied winding mechanism affects the two base polymers in different degree that separated them apart in lengthwise direction at the different degree of resilience and flexibility toward mechanical force. The materials use in the bicomponent fibre forming that intend to separate them into smaller fibres in the experiments were PP/PE and Nylon 6/PE had different separation or splitting degree because of the cohesion between the neighbor segments of the two polymer influenced the endeavour in separate them lengthwise. The images shows clear detached segments in the filaments' lengthwise in the specimens using Nylon 6 and polyethylene as base polymers, while the detached segments in the filaments using Polypropylene and polyethylene were infrequently occurred. The splitting filaments could be seen in the images of the filament specimens with winding mechanism from 300 m/min to 700 m/min. The production of most of the experiments were smooth, stable and the materials were applicable and the winding

mechanism reduced the size of the extruded filaments, accelerate the productivity, and separate the filament lengthwise. The selected base materials and the TiO₂ additives in the experiments were applicable for most of the experimental productions except for one - the spinning of polyethylene and nylon 6 at 700 m/min. winding speed which the extruded filaments were broken soon after the winding mechanism was applied. The filaments from other experiments were successfully conducted, the system condition setting and base polymers used in the experiments were applicable and the extruded filaments were smooth and unbroken.

The result of higher degree splittability of polyethylene and nylon 6 in segmented pie bi-component fibers than the fibers from the experiments of polypropylene and polyethylene was that the polypropylene and polyethylene filaments have high cohesion between their inner segment surfaces because they are both polyolefin type which similar properties, therefore, their liquidized phases were in similar manner. The DSC graphs showed one peaks of crystallization temperature of the two materials which means that the materials were solidified at the same temperature, while the filaments from polyethylene and nylon 6 reacted to the fiber spinning condition and process differently. The segmented pie bi-component filaments using polyethylene and nylon 6 were split easily after applied the winding mechanism. The difference degree of splittability of the two combination fibers could be explained with the different characteristic of the crystallization temperatures of the filaments. The filaments using polypropylene and polyethylene had the crystallization temperatures in one peak which means that both polymers solidified at the same time, while the crystallinity temperatures of the filaments using polyethylene and nylon 6 show two peaks, with wide gap between the peaks, in all the three winding speed varieties. The differences of the temperatures between the two peaks were between 74.8°C to 75.9°C, indicating that the nylon 6 segments were solidified before the polyethylene segments. Therefore, the crystallinity temperature is a significant indicator for the splittability of the segmented pie bi-component fibers. The filaments using nylon 6/ polyethylene clearly split easier than the polypropylene/ polyethylene filaments. However, the spinning of the bi-component filaments of nylon segmented with polyethylene at 700 m/min. were unfeasible because of the wide gap between the two peaks of the crystallinity temperatures. The nylon 6 segments were solidified faster than the polyethylene segments, therefore, the adjoining nylon 6 could not support the polyethylene solidification.

The total surface areas of the fibers would significantly increase after the separation mechanism applied, resulting that the TiO₂ particles embedded on the inside surfaces of the intact filaments became surface particles, promoting a higher degree of photocatalytic. The calculation of the surface areas of the filaments indicated the surface areas of all the 16-pie-shape-segments of a split filament were significant increased. The calculated surface area of 16-pie-shape-segments of a split filament were increased more than 600%. The efficacy of the separation mechanism applied to the intact filaments is confirmed. The micrographs also show that the particles of TiO₂ are distributed homogeneously within the filaments; some particles are scattered near the surfaces, while others are adhered to the surfaces. The presence of Ti on the detected surface of the split fiber specimen is much higher than that on the intact fiber. The atomic percentage of the Ti on the split fiber specimen was increased 92% and the weight percentage of the Ti was increased 86%.

The nanoparticles of TiO₂ embedded in the bi-component fibers during the extrusion stage would emerge from the inside surfaces of the intact filaments to the outside surface particles. The photocatalytic material was purposely added to break down organic compounds under common visible illuminated light for potential indoor and household uses. The LED was selected to assess the antibacterial activity of the filament embedded with 1.0% TiO₂ photocatalytic material, and the bi-component fibers with 1.0% embedded TiO₂ from the experiments were subjected to antibacterial tests against the Gram-positive *Staphylococcus aureus* and Gram-negative *Klebsiella pneumoniae* bacteria, using modified testing standard method - the American Association of Textile Chemists and Colorists (AATCC) test method 100-2012. The modification of the test method was that the specimens were exposed to a designated visible cold light source during 24 h incubation. The figures show significant reductions of the tested bacteria in the sample containing TiO₂. The percentage of the Gram-positive bacteria reduction (*Staphylococcus aureus*) in the sample with TiO₂ was 80.00% compared to 0% in the sample without TiO₂. The percentage of the Gram-negative bacteria reduction (*Klebsiella pneumoniae*) in the sample with TiO₂ was 42.94% compared to 0% in the sample without TiO₂.

The results from the experiments showed the high potentiality of the spinning of photocatalytic fiber as a splitable segmented-pie bi-component fiber for antibacterial textiles. The production of the segmented-pie bi-component fibers using polyethylene and nylon 6 as base polymers with 1.0% TiO₂ was feasible at the maximum winding speed of 500 m/min. The TiO₂ in the fibers was clearly visible under optical microscopy as scattered throughout the fibers. The fibers were easily split into several smaller fibers with minimal mechanical force to maximize the fiber surface area and thereby boost the photocatalytic activity. The particles of TiO₂ embedded in the intact fibers were exposed to the surface after the fibers were split. The EDX analysis confirmed a significant increase of the Ti at the fiber surface after the separation, in both weight and atomic percentages. The fibers containing 1.0% TiO₂ also showed high percentages of reduction of both *Staphylococcus aureus* and *Klebsiella pneumoniae* after 24 h contact time under visible cold light compared to the fibers without TiO₂. On the other hand, the percentage reductions of bacteria Gram-negative (*Klebsiella pneumoniae*) after 24 h contact time under visible cold light by the nylon fibers with and without TiO₂ nanoparticles were 0%.

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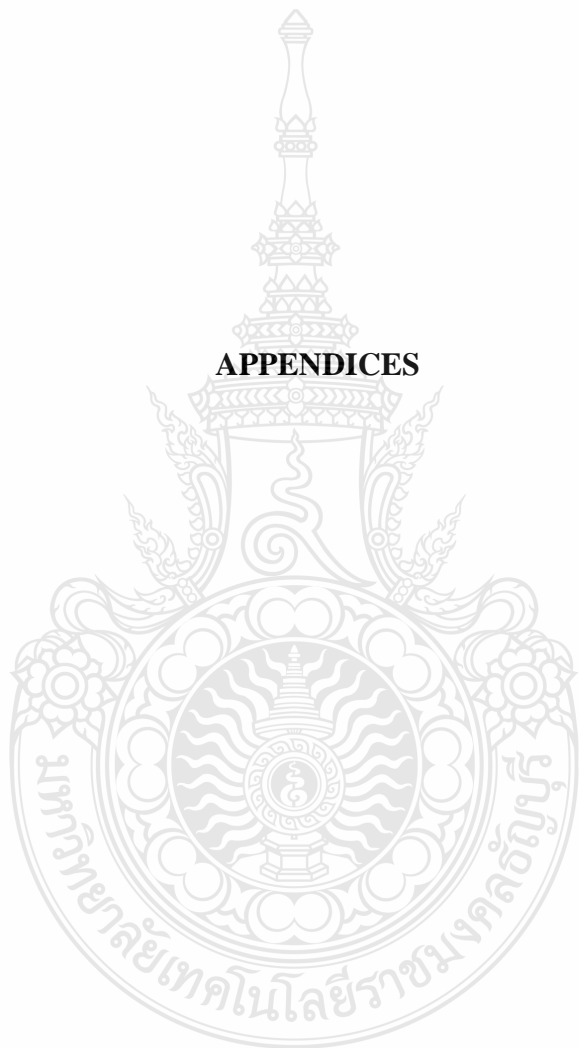
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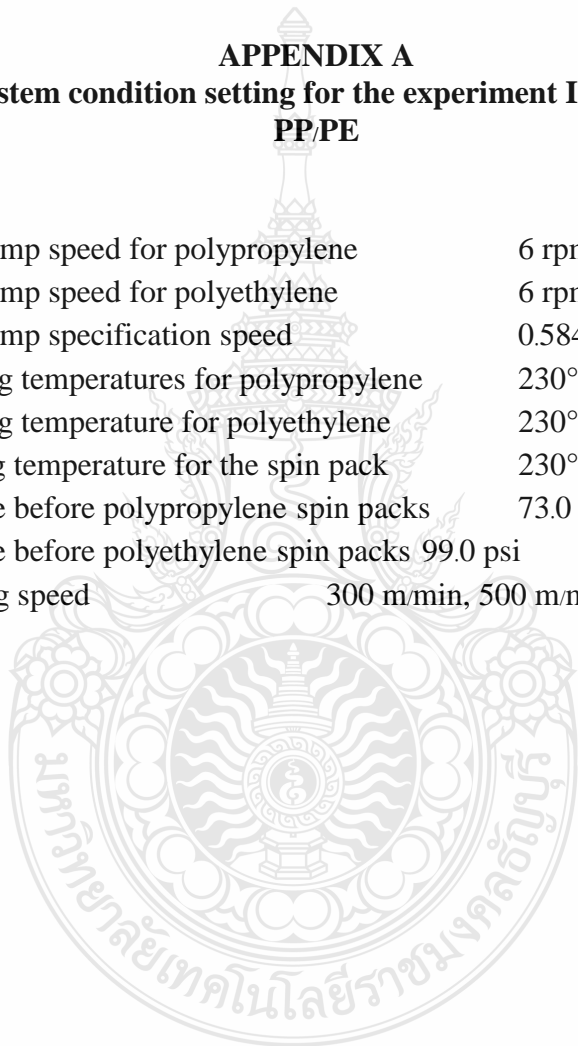
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APPENDICES



APPENDIX A
System condition setting for the experiment I – VIII
PP/PE

- | | |
|---|--------------------------------------|
| 1. Gear pump speed for polypropylene | 6 rpm |
| 2. Gear pump speed for polyethylene | 6 rpm |
| 3. Gear pump specification speed | 0.584 cc/rev |
| 4. Spinning temperatures for polypropylene | 230°C |
| 5. Spinning temperature for polyethylene | 230°C |
| 6. Housing temperature for the spin pack | 230°C |
| 7. Pressure before polypropylene spin packs | 73.0 psi |
| 8. Pressure before polyethylene spin packs | 99.0 psi |
| 9. Winding speed | 300 m/min, 500 m/min, and 700 m/min. |



APPENDIX B
System condition setting for the experiment IX – XVI
PE/Nylon6

- | | |
|--|--------------------------------------|
| 1. Gear pump speeds for polyethylene | 6 rpm |
| 2. Gear pump speeds for nylon 6 | 6 rpm |
| 3. Gear pump specification speed | 0.584 cc/rev |
| 4. Spinning temperatures for polyethylene | 250°C |
| 5. Spinning temperature for nylon 6 | 265°C |
| 6. Housing temperature for the spin pack | 265°C |
| 7. Pressure before nylon 6 spin packs | 70.2 psi |
| 8. Pressure before polyethylene spin packs | 78.5 psi |
| 9. Winding speed | 300 m/min, 500 m/min, and 700 m/min. |

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| | |
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