BIOPLASTIC PRODUCTION FROM COFFEE GROUNDS

BY

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ABSTRACT

In this study, polylactic acid (PLA) plastic compounded with the spent coffee ground (SCG) to produce PLA/SCG bio-composite film by using a twin-screw extruder with an SCG concentrations of 0 wt.%, 5 wt.%, 10 wt.%, 15 wt.%, 20 wt.%, and 25 wt.%. Then, the film samples were formed by compression molding. Their morphologies and degradations in various conditions were tested meanwhile the thermal and mechanical properties were determined. The increase in amount of SCG addition in the PLA matrix causes the lower adhesion force between PLA and SCG; when SCG distributes in the matrix, SCG is grouping in coalescence form, resulting in an incompatibility morphology. Moreover, higher amount of SCG in the PLA matrix causes the reduction in the tensile strength and modulus at the break while the elongation increases. From thermal testing using differential calorimetry (DSC) techniques, it was found that SCG did not affect the glass transition temperatures (Tg) and melting temperatures (Tm) of PLA/SCG bio-composites film. According to the degradation tests of PLA/SCG, the bio-composite film was performed using natural weather (NW), thermal (TH), acid solution (AS), and base conditioning (BS) methods and the weight changes were measured. The experiments revealed that the weights of the samples were reduced in the NW, TH conditions and more intensely in the BS condition. It was found that the weights of samples were the most reduced compared to other condition methods due to SCG that helps contributing the depolymerization reaction of PLA polymer causing the degradation of the films. On the other hand, it was not significantly affected in sulfuric acid because the acidic PLA did not react with sulfuric acid. The appearances of PLA/SCG films at every concentration of SCG have not changed much from the original, but the plastics were softer for acid solution conditioning test as well as for the thermal conditioning test, but the PLA/SCG bio-composite films were broken down into small pieces, especially for the biofilm with the concentration of SCG of the 5 wt.% and 10 wt.% in the base solution conditioning test. Therefore, SCG filter had an excellent property to promote PLA in the PLA/SCG bio-composite film.

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LIST OF SYMBOLS/ABBREVIATIONS

Symbols/Abbreviation	Terms
PLA	Polylactic Acid
SCG	Spend Coffee Grounds
PLA/SCG	Polylactic-acid and Spend coffee ground bio-composite film
PP	Polypropylene
PBS	Polybutylene Succinate
PET	Polyethylene Terephthalate
PS	Polystyrene
PE	Polyethylene
LA	Lactic Acid
NR	Natural Rubber
OM	Optical Microscope
UTM	Universal Testing Machine
DSC	Differential Scanning Calorimetry
TGA	Thermogravimetric Analysis
NW	Natural Weathering Conditioning
TH	Thermal Conditioning
AS	Acid Solution Conditioning
BS	Base Solution Conditioning
UV	Ultraviolet
DCP	Direct Condensation Polymerization
ACP	Azeotropic Condensation Polymerization
ROP	Ring Open Polymerization
MFR	Melt Flow Rate
ASTM	American Society for Testing and Material
%wt	Percent weight
MPa	Megapascal
psi	Pounds-Force Per Square Inch
%v/v	Percent by Volume
%wt	Percent by Weight
℃	Degree Celsius
g/cm3	Gram per Cubic Centimeter
rpm	Round per Minutes
H_2SO_4	Sulfuric Acid
NaOH	Sodium Hydroxide
T_g	Glass Transition Temperature
T_m	Melting Temperature
kN	Kilo Newton
mm	Millimeter
mm/min	Millimeter per Minutes
g/cc	Gram per Cubic Centimeter
ml	Milliliter

CHAPTER 1
INTRODUCTION

1.1 Statement of the Problems

Plastic is a synthetic material that plays a large role in daily life and tends to be used increasingly. It is light, strong, durable, and cheap. At present, it makes a variety of styles of plastic products with outstanding features that make popular plastic. This causes a large amount of plastic waste because the plastic is a material that cannot be self-degraded. Most plastics are in the form of single-used plastic. Nearly 50 percent of plastics are used as the packaging products such as plastic bottles, film, and plastic bags that people use every day. They are usually produced from polypropylene (PP), and polyethylene terephthalate (PET). However, we cannot avoid the needs of plastic demand. It also tends to increase from the population expansion, consumer behavior changes, and an increase in urban populations [1]. All above examples are the causes of plastic waste. Furthermore, plastics are difficult to collect due to their lightweights so they can be blown and distributed to the nearby areas. This causes the environmental problems, especially for the water resources that are often the last source of this plastic waste

Therefore, a new type of plastic has been invented with different properties than synthetic plastics. This aims to reduce the environmental problems mentioned above by plastic named as “biodegradable plastic”. The most used in the bioplastic industry is polylactic-acid (PLA). The advantages of polylactic-acid (PLA) are hard, clear, and easy to use due to glass similarity properties. However, it is fragile and cannot be durable to heat. For these reasons, we should find the ways to reduce the cost of production and improve the PLA’s quality.

At present, coffee is a trendy beverage and has a growing consumer trend for the Thai people. Mr. Justin Pow, General Manager of UBM Asia (Thailand) Co. Ltd. mentions that “The coffee shop business at present is growing. The value of the coffee shop business has been risen as high as 17,000 million baht, growing 15-20% per year [2]. The average coffee consumption rate of Thai people is around 300 cups/person/year and tends to increase compared with the consumption of coffee from many countries. Therefore, it can be seen that the Thai coffee consumption market is still able to grow. It is predicted that by 2022, there will be 300,000 tons of coffee consumption in Thailand per year [3].

The growth in the Thai people’s coffee consumption results in the number of coffee grounds, which are the residue from the coffee extraction process. Coffee grounds are scraps of roasted coffee that have been extracted through hot water. Coffee grounds are organic wastes from the processing industry. It affects the environment. Because there is a large amount of carbon in the coffee production, the BOD of the water will increase if the coffee ground waste is threw into the water sources.

From the literatures, coffee grounds contain chemicals that can be compounded with PLA to produce bio-composite film such as (i) polysaccharides which are carbohydrate substances consisting of monosaccharides of the same type (homopolysaccharide) about 50 percent per dry weight of coffee grounds 1 kilogram. It has strong chemical bonds in a long chain connected by the glycosidic bond [4] (ii) fatty acid which is a substance in the coffee oil similar to roasted coffee. When we added SCG an average of 15 percent w/w, that can improve the efficiency of PLA/SCG bio-composite films, both in physical and chemical and mechanical properties. [5]. It means that coffee grounds can be a candidate to produce bioplastics with may benefits and may substitute the conventional petroleum-based plastics such as PS, PP, PE [16].

Therefore, in this research, we realize the importance of coffee grounds that is the waste from both instant and freshly brewed coffee extraction processes. We have tried to produce the bio-plastic film by compositing polylactic acid (PLA) with the spent coffee grounds (SCG) that will be abbreviated as PLA/SCG in the following. The appropriate amount of SCG composited in PLA will be optimized for film production. Therefore, the advantages of this project are to reduce the amount of waste produced from coffee and the cost of bioplastic production and to examine the properties of SCG/PLA composite film.

1.2 Objectives

1. To produce a bio-composites film using a PLA matrix, a naturally synthesized plastic, and easily digestible bio-composite, incorporated into SCG by using a twin-screw extruder to compound and compression molding to form a film.

2. To determine the effect of concentrations of spent coffee ground (SCG) filter on PLA bio-composite films such as mechanical, physical, and thermal properties and study the degradation of PLA/SCG materials at different coffee grounds concentrations in various conditions.

1.3 Research Scopes

1. Study the effect of spent coffee grounds (SCG) filter with different concentrations (0 5, 10, 15, 20, 25 wt%) on bio-composites film (PLA/SCG).

2. Compound the coffee grounds and PLA by using a twin-screw extruder with a screw speed of 10 rpm and the inlet fusion temperature range of 185-200 °C. Then, the film is formed by compression molding with two plates of curing temperature (hot zone for 2 minutes, cold zone for 1 minute)

3. Test for mechanical, physical and thermal properties and demonstrate the degradation of the prepared bio-composite films in.

4. Demonstrate the degradation of the prepared bio-composite films in the different types of conditions.

1.4 Research Proposal

1. The amount of waste produced from the coffee grounds will be reduced.

2. The coffee ground can promote the film's properties.

3. The cheaper cost of bio-plastic products.

4. The easily biodegradable and environmentally friendly film.

CHAPTER 2
THEORIES AND LITERATURE REVIEW

2.1 Bioplastics [6]

Bioplastics are plastics made of agricultural raw materials (bio-based materials) and petroleum-based byproducts. These bioplastics are similar to the conventional plastics. It can be forged and manufactured in a normal forging process with conventional machines (single-screw extrusion, compression molding and blow film extrusion) with slight suitability adjustments. For bioplastics made of agricultural raw materials, they are produced by the fermentation process to convert agricultural raw materials into the monomers and then be used to produce plastic pellets. The raw materials usually used in the production of bioplastics are corn, sugar, and cassava, which could be produced from rice straw.

Bioplastics have been developed until ready for commercial operations. They can produce various products such as polylactic acid (PLA) and polybutylene succinate. (PBS) because it can be obtained from various plants.

2.1.1 Type of Bioplastics

2.1.1.1 Starch-Based Plastics

Starch-based plastics are complex blends of starch with compostable plastics such as polybutylene succinate (PBS), polylactic acid (PLA), polybutylene adipate terephthalate (PAT) as shown in Figure 2.1. These complex blends improve water resistance as well as processing and mechanical properties. Starch-based bioplastics are often blended with biodegradable polyesters to produce starch/PLA or starch/bio-flex. These blends are used for industrial applications and are also compostable. For example, polysaccharides are natural polymers divided into sugar and starch. Still, the plastic pellet from this kind of material is more likely to use sugar to convert to monomer than to starch due to fermenting sugar into a lattice monomer and using flour as raw material sugar, glucose from corn, and sugar from sugar cane. It is the most used raw material in the production of the big current scale.

Figure 2.1: Raw materials using for starch-based bioplastic production [27]

2.1.1.2 Cellulose-Based Plastics

Cellulose-based plastics are made of cellulose, cellulose esters, and their derivatives, Including celluloid. These are the main component in plant tissues, for example, agricultural waste material or lignocellulose material. It is generally the material leftover in the fields after harvesting and can be found in many leaves, stems, and roots of bagasse, rice crust, rice straw, and tree bark.

Figure 2.2: Bioplastic production from cellulose based plant [26]

2.1.1.3 Protein-Based Plastics

These types of bioplastics are made of proteins from different sources. The present, protein-based plastics are not popular in production because of the difficulties of using soy protein-based plastics due to their water sensitivity and relatively high cost.

Figure 2.3: Bioplastic produced using protein in milk [25]

2.1.2 Benefits of Using Bioplastics

1. Bioplastic can improve soil quality by adding organic matter, moisture, and nutrients to the soil. Composting of biodegradable plastics leads to the turnover of the elements. While the use of plastics often has to be disposed of by landfill or incineration.

2. Reduce the usable area of the landfill by using biodegradable plastics and compostable bags. It can increase the degradation potential of food waste or organic waste in landfills and increase the potential for methane gas production for use fuel in cases where the landfill is designed to produce and utilize methane gas. Daily use of biodegradable plastic film as a landfill cover for landfills increases space for landfills. Due to the daily closure of the landfill ponds, 25% of the landfill area is used.

3. The use of bioplastics can reduce the number of greenhouse gases emitted by the elimination.

2.2 Degradation [7]

2.2.1 Photodegradation

Photodegradation is often caused by the addition of light-sensitive additives to plastics to break the plastic's chemical bonds or synthesize the copolymer to have an unhealthy functional group or chemical bond to be easily damaged when exposed to light (UV rays). But this degradation method cannot be performed with a landfill that is not getting light all the time or a garbage pile in a dark environment Because it will not be exposed to UV rays directly.

2.2.2 Mechanical Degradation

Mechanical degradation is a standard method of breaking down plastics into smaller pieces by applying force. That involves using mechanical stress to the polymer samples, and examples of processes to determine mechanical degradation are regrinding, adhesive pressing, compression molding, and injection molding. For examples, the degradation of polyurethane waste by using two-roll as Figure 2.4

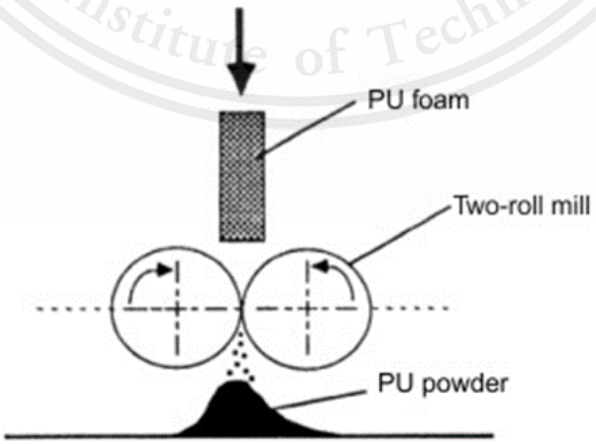

Figure 2.4: Mechanical degradation of polyurethane foam using two-roll mills [24]

2.2.3 Oxidative Degradation

Oxidative degradation is the reaction of oxygenation of polymer molecules that can occur spontaneously in nature with oxygen and heat, UV light, or mechanical forces. It is crucial to cause rapid fracture and loss of mechanical properties. Oxidative degradation is usually initiated when polymer chains form radicals, either by hydrogen abstraction, or by homolytic scission of a carbon-carbon bond. This can occur during manufacture, processing or during service when exposing the polymer to light or heat. Figure 2.5 is shown the oxidative degradation mechanism.

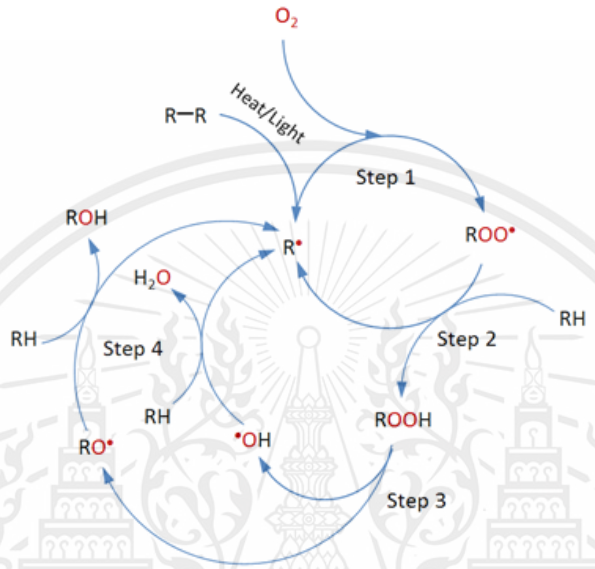

Figure 2.5: Oxidative degradation mechanism [22]

2.2.3.1 Oxidative Degradation Mechanism

$$R{-}H \rightarrow R\cdot + H\cdot \quad (1)$$

The first step is the reaction of a free radical (R·) with an oxygen molecule (O_2) to form a peroxyl radical (ROO·*), which then abstracts a hydrogen atom from another polymer chain to form a hydroperoxide (ROOH). The hydroperoxide splits then into two new free radicals, (RO·) + (·OH), which abstract labile hydrogens from other polymer chains. Since each initiating radical can produce two new free radicals, the process can accelerate depending on how easy it is to remove hydrogen from other polymer chains and how quickly free radicals undergo termination via recombination and disproportionation.

$$R\cdot + O_2 \rightarrow ROO\cdot \quad (2)$$
$$ROO\cdot + RH \rightarrow R\cdot + ROOH \quad (3)$$
$$ROOH \rightarrow RO\cdot + \cdot OH \quad (4)$$
$$RO\cdot + RH \rightarrow R\cdot + ROH \quad (5)$$
$$OH + RH \rightarrow R\cdot + H_2O \quad (6)$$

The result of these reaction is embrittlement and cracking of the polymer. Termination by chain scission, on the other hand, results in the decrease of the molecular weight leading to softening of the polymer and reduction of the mechanical properties

$$Rn\cdot + Rm\cdot \rightarrow Rn\text{-}2{-}CH{=}CH2 + Rm \quad (7)$$
$$2\ RCOO\cdot \rightarrow RC{=}O + ROH + O_2 \quad (8)$$

2.2.4 Hydrolytic Degradation

Hydrolytic degradation is a reaction that contributes to the fracture of a polymer chain, divided into two types: catalytic hydrolysis and non-catalytic hydrolysis. The hydrolysis reaction is the breakdown of polymer bond by reaction with water which means that this reaction mainly occurs with polymer that can take up a lot of moisture include polyesters, polyanhydrides, polyamides, polyether, and polycarbonates, as shown in the Figure 2.6

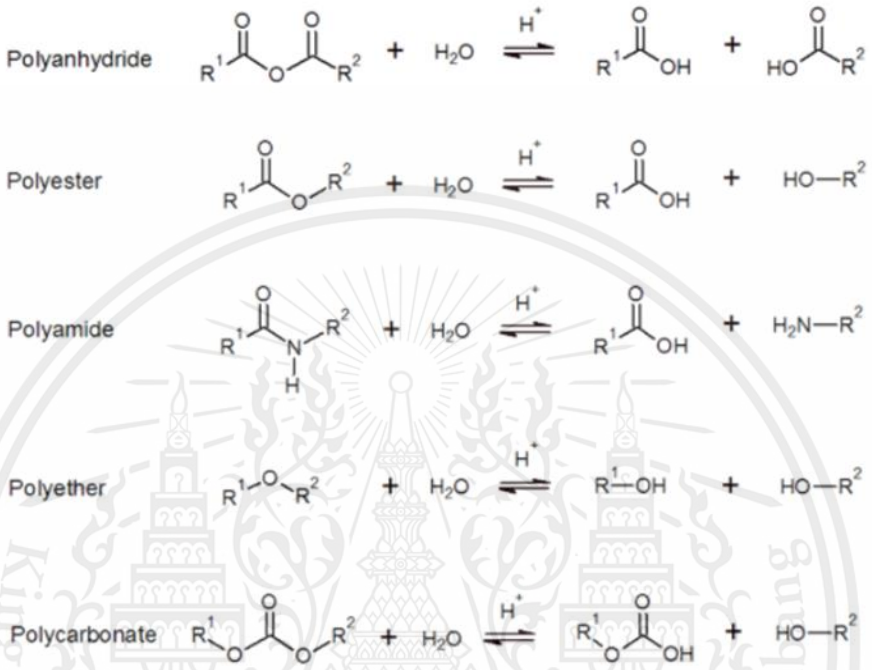

Figure 2.6: The hydrolysis degradation of polymer [23].

2.2.5. Biodegradation

Biodegradation is the breakdown of chemical structure using a microorganism; it can be bacteria or other biomaterials. Plastic will be broken into small fragments from water, sunlight, and oxygen will make plastic into small pieces and be the food for bacteria. Most of these plastics are polyester since they can be easily damaged by water. Mechanism of Plastic Biodegradation shows in Figure 2.7.

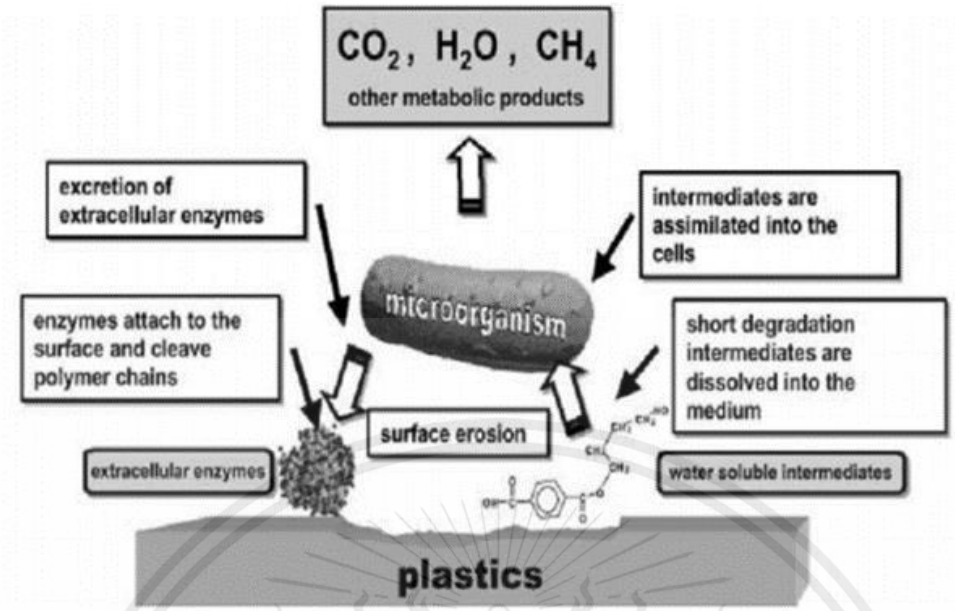

Figure 2.7: The general mechanism of biodegradation [21]

Generally, the word bioplastic refers to the plastic that made from insoluble material bases and can be degraded by microorganism. The microorganism breaks down the polymer to form monomer then breakdown to carbon dioxide, water, and methane. For the aerobic biodegradation, the reaction can express by the following expression,

$$C_{polymer} + O_2 \xrightarrow{\text{microorganism}} CO_2 + H_2O + C_{residue} + C_{biomass}$$

For anaerobic biodegradation,

$$C_{polymer} \xrightarrow{\text{microorganism}} CO_2 + H_2O + CH_4 + C_{residue} + C_{biomass}$$

2.3 Plasticizer [8]

Plasticizers are the additives that add to the plastic to decrease the viscosity of a material or the plasticity. These are either liquids with low volatility or solids. They reduce the attraction between polymer chains to make them more flexible. Bio-plasticizers are one of the types of plasticizer improving the softness, volatility, and workability of plastics and usually made from renewable resources, for examples of bio-plasticizer, epoxidized soybean oil (ESBO), epoxidized linseed oil (ELO), castor oil, palm oil, vegetable oils, starch, sugars, etc.

2.3.1 Type of Plasticizer

The plasticizer is an additive added to polymers to increase the flexibility and softness of polymers. By adding a plasticizer, it allows the polymer molecules to move more easily. This results from the plasticizer molecules' reducing the attraction between the polymer molecules and increasing the space between the polymer chains. The polymer has a lower glass transition temperature (T_g) than the room temperature. This makes the polymer cable more flexible and has more stretchability. Plastic flow and reduces the viscosity of the polymer melt viscosity, making the molding process easier. The plasticizer is divided into two main types:

2.3.1.1 External Plasticizer

The external plasticizer is a plasticizer mixed into a polymer to reduce the attraction between polymer molecules and make it easier for the polymer to weaken and change shape without chemical reactions occurring between the plasticizer and the polymer. The external plasticizer is typically a liquid with a high boiling point, low vapor pressure, difficult evaporation, soluble in polymers, and a glass transition temperature of about -50 degrees Celsius. The advantage of external plasticization is that the polymers can be softened as little or as required by the application by adjusting the amount of plasticizer inserted. But the disadvantage is the plasticizer may be extracted from the polymer.

2.3.1.2 Internal Plasticizer

The internal plasticizer is a plasticizer that is part of a polymer structure, such as co-polymerization. Therefore, the attraction between the molecules decreases. Thus, this type of plasticizer cannot be separated from the polymer by extraction method, which is considered an advantage of internal plasticity processes. That is to say, copolymers provide products with limited weakness, and the mechanical properties of polymers often vary according to extreme temperatures, shape stability is often wasted to high temperatures.

2.3.2 Theory of plasticizer

Plastic consists of polymer molecules, each connected by Vander Waal forces, where the plasticizer does not react with the polymer but inserts itself between the polymer molecules by reducing the Vander Waal forces. There are 3 theories explaining the mechanism of the external plasticizer:

2.3.2.1 Lubricity Theory

This theory explains the function of the plasticizer that acts as a lubricant by reducing the friction between the polymer molecules. Make chain polymer begins to move, sliding past each other.

2.3.2.2 Gel Theory

This theory describes plasticizer Sir destroy the attraction between molecules of the polymer. By separating the polar parts of the polymer so that they are far apart. Therefore, the plasticizer must consist of both polar and non-polar parts. The polar part of the plasticizer is attached to the dipole or the polar region of the polymer. While the non-polar plasticizer shields the polymer dipole area apart.

2.3.2.3 Free Volume Theory

This theory explains that if the plasticizer will increase the space or the free quantity between the polymer chains, making it easier for the polymer to be applied.

2.4 Polylactic Acid (PLA) [9]

Polylactic acid (PLA) or the chemical formula [$C_3H_4O_2$] is produced from lactic acid. PLA is transparent plastic, and it is a thermoplastic with distinctive features that are clear and shiny, easy to mold. Also, PLA has good mechanical properties. PLA can use various industrial sectors, such as food packaging, film, and biodegradable plastic bags. However, PLA is limited in price and has some disadvantages such as brittle and low heat resistant. So, other natural materials are added as PLA components to enhance its properties as mentioned above.

2.4.1 Types of PLA

The raw material used to produce polylactic acid (PLA) is starch derived from renewable resources, including starch-based plants such as corn and cassava. The production starts from grinding or milling the plant into a powder. The starch is then digested into sugar and fermented by microorganisms to form lactic acid, similar to beer fermentation. Lactic acid had obtained through a chemical process. The structure had transformed into a new ringed chemical structure called a lactide, which had then distilled in a vacuum to transformed the structure into a long strand of lactide polymer called polylactic acid. PLA has a unique molecular structure depending on the method of production consist of two types,

1. Dextrorotatory; D-isomer (-)
2. Levorotatory; L-isomer (+)

PLA monomers are lactic acid, which has two forms of isomers: D-isomer and L-isomer, which is enantiomer with different optical active, that is, have the same chemical formula, but the arrangement in three dimensions is not the same, and finally, the polarized light plane in different directions.

Furthermore, PLA is used in industrial and commercial applications with many identical units derived from L-isomer monomers. Most of the bacteria used in the fermentation process produce lactic acid L isomer. In contrast, the D-lactic acid isomer (D-lactic) is only 1-2%. The typical structure of PLA is shown in Figure 2.8

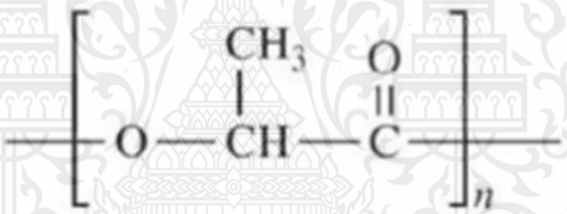

Figure 2.8: PLA molecules structure

2.4.2 PLA synthesis

Generally, there are three methods for the synthesis of PLA to obtain high molecular weight and can be utilized in the industrial sector, as shown in Figure 2.9

2.4.2.1 Direct Condensation Polymerization (DCP)

This method for the synthesis of lactic acid uses a catalyst at the point of pressure reduction. This method is inexpensive but difficult to challenge for polymers above 100,000Da with low molecular weight. (In the range of 1,000-5,000Da) because it is difficult to obliterate water, the reaction of high viscosity mixed in this process is inferior to mechanical properties. However, high molecular weight polymers can be made using coupling agents or adjuvants such as bis (tri-chloromethyl) carbonate and carbonyl diimidazole.

2.4.2.2 Azeotropic Condensation Polymerization (ACP)

In this reaction, lactic acid (LA) is converted to high molecular weight polylactic acid (PLA) by using organic substances such as toluene, xylene, or diphenyl ether, etc. in the process and removing water by distillation by balancing between polymers and monomers are used as a solution for water removal in organic solutions. Therefore, it is a process of condensing directly into the polymer, giving it a higher molecular weight. This method yields high

molecular weight polymers but can cause unwanted decomposition. This includes the inability to control or no reproducibility accuracy in terms of the rate of hydrolysis, the accelerator of toxicity, and differentiation of features in slow-release.

2.4.2.3 Ring Open Polymerization (ROP)

Ring open polymerization reaction is a chain formation at the end of the polymer string, whereby circular monomers can form open-loop reactions and form long-stranded polymers. By this method, polymerization is possible. Low molecular mass oligomers will be used to synthesize ring-type polymers, which cannot typically condensation by the reaction at high temperatures or long-time transesterification, which has a low molecular mass and an increased PDI value.

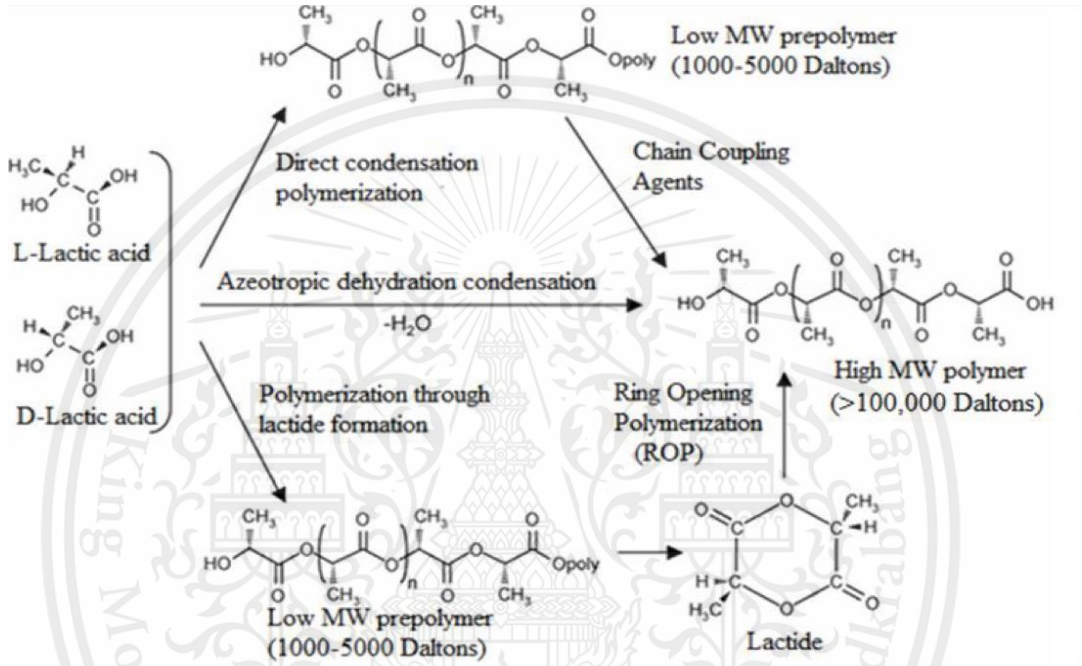

Figure 2.9: PLA synthesis in a different way (DCP on the top line, ACP on the middle line, ROP on the bottom line)

2.4.3 Optical Purity [10]

The optical purity of PLA has a significant effect on thermal properties, mechanical properties, and barrier properties. With a proportion of more than 90% of the L-isomer, PLA is like to be a semi-crystalline polymer. Simultaneously, polymers with more D-isomers have lower optical purity and tend to be amorphous polymers. Besides, melting temperature, glass transition temperature, and crystallinity have the proportional downward trend of the reduced L-isomer, with the proportion of the different isomers in the polymer chain, allows the synthetic PLA to have a wide range of properties when we compared with other polymers that play a role in plastic industrial such as polyethylene terephthalate (PET) and polystyrene (PS). Furthermore, PLA is clear and has similar physical, mechanical, and anti-gas permeability properties like PET.

2.4.4 Properties of PLA [10]

2.4.4.1 Solubility Property

The solubility property of PLA depends on 1. proportion of D and L position in the polymer chain and 2. Degree of crystallinity. PLA is soluble in acetone, pyridine, ethyl lactate, ethyl acetate, tetrahydrofuran, xylene, dimethylsul-foxide. In the other hand, PLA is insoluble

in water, alcohols, and unrivaled hydrocarbons such as hexane and heptane, a suitable solvent for optically pure PLA.

2.4.4.2 Thermal Property

PLA has a relatively higher glass transition temperature and melting temperature than that of conventional thermoplastics. Molecular weight, it was found that the glass transition temperature tended to increase with molecular weight. By mixing L-enantiomer with D-enantiomer, the glass transition temperature decreases as the L-D ratio is equal. For example, the L to D ratio is 50:50; the glass transitions temperature tends to drop the most. The melting temperature of PLA also depends on the polymer's optical purity, with the highest melting temperature found in optical purity PLA is approximately 180°C, the polymer's melting temperature decreased if the content of D-lactide was increased in structure, the melting temperature can be reduced up to 50 °C.

2.4.4.3 Barrier Properties

PLA's gas-liquid permeability is similar to that of conventional packaging polymers, such as polystyrene (PS) or polyethylene terephthalate (PET), and the vapor permeability coefficient is close to the plastic mentioned above. Furthermore, the allowance coefficient carbon dioxide (CO_2), oxygen (O_2), and nitrogen (N_2) permeability of PLA are less than PS but higher than PET. Also, PLA has a good anti-odor property by considering the coefficient of the diffusion of organic compounds such as ethyl acetate which is similar to that of PET. These coefficients indicate that PLA has similar anti-permeability properties to the plastics commonly used in industry today. Therefore, people have begun to turn their attention to choosing PLA as a material for various industries.

2.4.4.4 General Properties of PLA 4043D [29]

Table 2.1: General properties of PLA 4043D

Properties	Typical Value	Standard
Physical		
Density (g/cm^3)	1.24	ASTMD792
Melt Index (210℃,2.16 kg), g/10min	6-70	ASTM D1238
Mechanical		
Tensile Modulus (psi)	293,000-514,000	ASTM D638
Tensile Strength (psi)	7,080-8,150	ASTM D638
Elongation at break (%)	0.50-9.2	ASTM D638
Thermal		
Glass Transition Temperature (℃)	44-62	DSC
Melting Temperature (℃)	157-170	DSC
Peak Crystallization Temperature (℃)	130-164	ASTM D3418

2.4.5 Advantages of PLA

1. PLA can produce itself from sustainable sources that can be easily degraded by the natural condition.

2. PLA has a high melting temperature.

3. PLA is a thermoplastic with distinctive features that is clear, shiny, and easy to mold (extrusion, compression molding or injection molding) due to the similar properties to glass.

2.4.6 Disadvantages of PLA

1. PLA is hard and relatively brittle. This causes restrictions in some applications, such as packaging film that requires elongation. Therefore, in applications, it is preferable to add plasticizers to the PLA compound or mix PLA with other polymers with better ductility and elongation, such as polycaprolactone and ECOFLEX.

2. PLA is not heat-resistant, especially if it is not pre-heat treated; it may degrade during the manufacturing process through the hydrolysis and chain scission mechanisms.

3. PLA has a low melting strength of the polymer, causing problems in some processes such as blown film extrusion process because the film that is blown next to the polymer flowing from the die is unstable.

2.4.7 Application and Products of PLA

PLA can be used for a wide variety of applications, including:

2.4.7.1 Medical

Because PLA is a bioplastic that is naturally biodegradable and can be compatible with human tissues. Therefore, this advantage is applied in medicine such as medical screws and pill capsules, etc.

Figure 2.10: Medical screws (left) and Pill capsules (right)

2.4.7.2 Agriculture such as plant containers, materials for encapsulation and release of pesticides,

Figure 2.11: Plant containers for agriculture

2.4.7.3 Packaging such as disposable packaging. Food containers, water bottles, plastic bags, foam boxes.

Figure 2.12: Water bottles (left), Plastic bags (middle), Foam boxes (right)

2.4.7.4 Nonwoven Fibers and Sheets such as sanitary products Disposable diapers, clothes, and garments Textile lining.

Figure 2.13: Sanitary products (left) and Linen blanket (right)

2.4.7.5 Automotive such as bumpers, floor mats, and interior accessories

Figure 2.14: PLA-JEEP floor mats (left) and Car bumper (right)

2.4.7.6 Household Products such as plastic wrap film, PLA filament for 3D printing.

Figure 2.15: Plastic wrap from PLA (left) and PLA filament for 3D printing (right)

2.5 Coffee [30]

Coffee is a beverage made from the beans obtained from the coffee plant, often referred to as the roasted coffee beans. Coffee plants are grown in more than 70 countries around the world. Coffee contains caffeine, giving it energy-boosting properties for humans. Coffee is currently the most popular drink in the world.

Thailand is the third-largest coffee plant in Southeast Asia. The total area of the coffee plantation is 411,843 rai, yielding an area of 381,650 rai in 1998. And in 2005 there is a production area of 409,266 rai with most of the cultivation area in the South and North. Breeds of significant economic value are Arabica and Robusta. Especially, Thai Robusta coffee is of

good quality and widely accepted in the international market. The main export markets are the US, America, Europe, Japan, and Singapore. Which has the following product:

- Robusta coffee: yields approximately 80,000 tons per year, 40 percent of which is processed domestically, and another 60 percent is produced for export.
- Arabica coffee: yields approximately two to three hundred tons per year. Almost all of them are processed within the country.

Currently, the country most famous for coffee production, Brazil, produces 72 percent of the world's coffee production. And the most productive the countries that buy the coffee the most are the United States and Europe, about 85 percent of the global product.

2.5.1 History of Coffee

Coffee is a native plant of Abyssinia and Arabia, discovered in the 5th century BC. In Arabia, No one paid much attention back then until the 9th century. There was an Arabian goat herder named Kaldi. Take the goats out and feed as usual. The goat ate the fruit and leaves of the coffee, and an unusual prank occurred. Therefore, it brought the story to be told to one of the Muslims. The Muslim Buddha then collected the coffee, cracked the bark, roasted the coffee beans, and boiled in hot water and saw a good rejuvenation. Therefore, told others to continue listening to Arabians have consequently started to know more about coffee. Thus, increasing the widespread coffee from Arabian countries. It entered the Italian people, Dutch, German, and French, and the coffee production process developed over a later period.

2.5.2 Botanical Characteristics

2.5.2.1 Coffee roots

Coffee has four to eight root roots and branches from the taproot. And from the safflower, the roots will be broken out again as a root for sucking food. These roots, 60 to 80%, spread at the soil surface to a depth of 20 cm.

Figure 2.16: Coffee roots

2.5.2.2 Trunk and Branches

Naturally, coffee has a straight stem. In the early stages of growth, will not grow branches. But with a pair of opposite leaves as they grow older, the branches are individually removed from the trunk. And are opposite each other, the newly sprouted branches will have a pair of leaves in pairs. The branches are parallel to the ground or hanging down to the ground, which is the birthplace of the flower, and next, besides the branches, there are also a lot of sprouts from the buds of the trunk. Causing new shoots to squeeze to the original stem if left

to thrive without pruning, the coffee will have a dense canopy, where disease buildup in the coffee plant is lower, and the coffee plant will eventually die.

Figure 2.17: Trunk and branches of coffee

2.5.2.3 Leaf

The coffee leaf is a single leaf, short petiole, base, and tapered leaf tip. The size of the leaves depends on the coffee variety. The leaves will be born at the verses as opposites. The stomata are on the abdomen, and each leaf contains approximately 3 million to 6 million holes. The stoma of Robusta coffee is smaller than the stoma of Arabica coffee. But there are more than 250 days of age than leaves.

Figure 2.18: Coffee leaf

2.5.2.4 Coffee Flower

Coffee flowers have a pure color. The fragrance is like jasmine. Moreover, Coffee has a unique character, and the branches of the branches are short, produce flowers, and be attached to a lot. Coffee flowers are perfect sex flowers. Both have stamens, and the pistil is in the same flower. The flowering time of Coffee depends on the amount of water. If there is a rainy season in a local where Flowers will come out after rain for about one month, but if the weather is moist throughout the year. Coffee will bloom all year round.

Figure 2.19: Coffee flower

2.5.2.5 Fruit and Coffee Berry

The fruit of the coffee is oval, short, green fruit, when ripe, it will be yellow, orange, red, the coffee will be divided into three parts:

1. Peel
2. The flesh is yellow when ripe, with a sweet taste.
3. Parchment encapsulates the seed between the seed and the shell, and there is a thin membrane covering the seed called "seed coat." Each coffee will have 2 grains on it, and the splice site is flat inside. There is one groove in the middle of the seed, and the outer part is curved. The seeds are either single or tone seeds (pea-berry).

Figure 2.20: Coffee seed and coffee berry

2.5.3 Anatomy of a Coffee Berry

Figure 2.21 shown the berry and seed structure. The key number's as following:

1. Center cut,
2. Bean (Endosperm),
3. Silver skin (Testa, Epidermis),
4. Parchment (Hull, Endocarp),
5. Pectin layer,
6. Pulp (Mesocarp),
7. Outer skin (Pericarp, Exocarp).

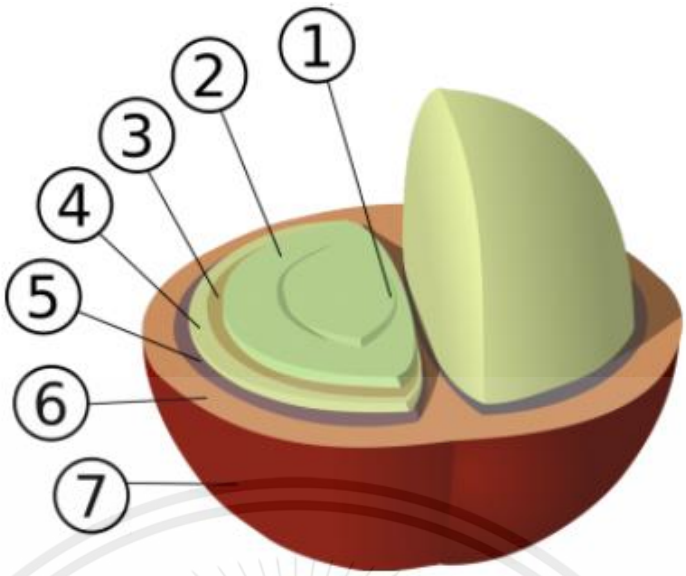

Figure 2.21: Anatomy of a coffee berry

2.6 Coffee Grounds

2.6.1 What is Coffee Grounds?

Coffee grounds are the particles of coffee that have been pressed through hot water. Freshly brewed coffee will not re-use the used coffee grounds because it will lower the aroma and flavor. The resulting aroma and caffeine will be lower as well. Coffee grounds are classified as organic waste from the coffee processing industry and general coffee shops. The coffee grounds are considered waste products that affect the environment because they contain a large amount of carbon. If they are dumped into water sources, it will result in higher BOD in water. And if there is poor handling of the coffee grounds, it can become a reservoir of fungi and bacteria that are harmful to humans.

2.6.2 The Importance of Coffee Grounds

The number of coffee shops increases as consumers turn to roasted ground coffee instead of instant coffee; the coffee shop business is becoming a popular business and development and growth of the coffee business in Bangkok. As a result, there will be more competition, especially ways. Most of which are open in the department store such as Starbucks coffee, Cafe amazon coffee and Coffee world [3]. Although coffee shops can sell large amounts of coffee or how many kilograms of coffee beans are used per day, coffee powder after roasting, ground, brewing, or hot water from coffee brewing. To extract the water of the coffee, one thing that will follow is coffee grounds. In most cases, the store or the majority of people tend to take it away, which of these waste coffee grounds has resulted in waste, which has direct and indirect effects on the environment. It is also the cause of global warming. Many coffee grounds and waste problems harm the environment because they contain a large amount of organic matters and cannot be disposed of undergoing treatment. Therefore, most coffee grounds are reused by sprinkling them on trees to improve soil conditions. It is being compressed into a bar to absorb odors or to sprinkle to repel ants. But coffee grounds have a disadvantage if they are left in the soil or the environment. They are organic matter with a large amount of carbon, which is considered a waste product that affects the environment if dumped into a water source. It will result in a higher amount of BOD in the water and without good coffee grounds management, it will result in a build-up of mold and bacteria and be harmful to humans.

2.6.3 Coffee Grounds Compositions

2.6.3.1 Polysaccharides [4]

Polysaccharides are a carbohydrate substance that consists of homopolysaccharides such as cellulose or heteropolysaccharide or hemicelluloses such as galactomannan and arabinogalactan. In the coffee grounds assembled with both polysaccharides, approximately 50% per dry basis, coffee grounds 1 kilogram. In summary, coffee grounds have contained the most amount of galactomannan.

2.6.3.2 Proteins [11]

In the coffee grounds, the average protein content is about 13.6% w/w analyzed by the Kjeldahl method. Coffee grounds also contain many types of amino acids. In some types of coffee grounds, there are as many as 17 species, with leucine, valine, and phenylalanine at the average of 10.6-10.9.,6.0-6.8, and 0.5-6.7 (% protein) respectively.

2.6.3.3 Fat [5]

The oil content in coffee grounds ranges from 11-20% w / w on average, approximately 15% w / w. Extraction of oil from the coffee grounds begins with an organic solvent such as hexane. ether, dichloromethane by reflux method for 1 hour in coffee grounds: solvent ratio 100 g: 300 ml. The results showed that the percentage of oil obtained from hexane extraction. Ether and dichloromethane were 13.4, 14.6, and 15.2% w / w, respectively, and the pH values of oil were 6.8, 4.7, and 4.5, respectively. Therefore, hexane is the most suitable solvent for extracted oil because it gives a medium pH value appropriate for other applications.

2.6.3.4 Phenolic Compound [12]

Phenols are the kind of natural substance found in many plants, which has antioxidant activity. The antioxidant analysis of general phenolic compounds is calculated from the equivalent gram weight of gallic acid found in coffee grounds. Phenolic compound about 1-4% GAE w/w.

2.6.3.5 Caffeine [11]

Caffeine is a vital substance and is a specialty of coffee. The amount of caffeine in the roasted coffee range is around 0.73-41.3 ug/mg SCG extracted. It can be extracted by solvents such as hexane, chloroethane, ethanol by ultrasound, and Soxhlet techniques.

2.6.3.6 Mineral [11]

Minerals in coffee grounds contain several types of minerals that can obtain from an analysis of ICP-AES techniques. In addition, coffee grounds include K, P, Mg, Ca, Al, Fe, Mn, Cu., Zn, S, Cr, where K is most commonly found 3549.0 mg/kg SCG. Moreover, the average ash content in coffee grounds is 0.4-1.6% mg/kg SCG, which is less than coffee grounds. On the other hand, we can say the coffee ground is fully carbonated, leading to energy and agriculture application.

2.6.4 Utilization of Coffee Grounds

1. Used to absorb musty odors.

2. Use to repel animals.

3. To nourish the skin to shine and look youthful.

4. Use to nourish the hair to make the hair shiny and strong.

5. It is used as agricultural fertilizer and can be mixed with soil to grow trees because coffee grounds contain high amounts of nitrogenous minerals, which is a mineral that plants use to grow. In addition, coffee grounds are also weakly acidic, which can adjust the pH in the soil that helps soil to be neutralized.

6. Used in Art. Coffee grounds, when mixed with water, give a beautiful brown color. Suitable for use as watercolors, drawing or can be made as color in fabric production

2.7 Extruder [9]

Extruder means the machine used for the molding process is similar to the injection method; the difference is that the extruder does not have the mold part and the control equipment for the mold at the end of the plastic syringe (die). It is a channel that extracts the plastic into a line or a sheet with a cross-section according to the die. Forming in this way can be applied to produce a wide variety of plastic pipes or strands, plastic bags, films, sheets, or plastic rods with special cross-sections are shown in Figure 2.22

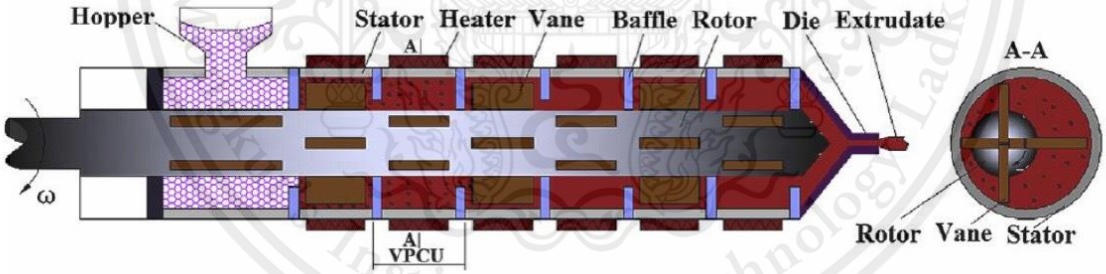

Figure 2.22: Parts of the extruder when the cross-section

2.7.1 Components of Extruder

The main components of the extruder are the extruder screw divided into three parts:

2.7.1.1 Feed Section is responsible for receiving the polymer pellets and sending the polymer to spiral screw.

2.7.1.2 Transition Section, in this section, the worm screw has a significantly reduced depth, allowing the extrusion of plastic pellets to increase friction and contact with the threaded barrel.

2.7.1.3 Metering Section, in this section, the worm screw has a constant and shallow depth that acts as a pump to deliver the melted polymer to the mold.

2.7.2 Twin-Screw Extruder

A twin-screw extruder is the one type of extruder machine used for the plastic extrusion process when two or more ingredients are compounded. A twin-screw extruder is an extruder developed in which a barrel has two screws placed horizontally parallel to each other. Allows better conveying and kneading of high moisture materials than a single screw extruder. The process is best suited when extruding reactive polymeric materials. Figure 2.23 shown the twin-screw extruder.

Figure 2.23: Twin-screw extruder

2.8 Compression Molding [9]

2.8.1 Working Process

Compression molding is the simple process of forming the polymer by applying mechanical stress (pressure) on a polymeric material placed on the lower side of a mold. Compression molding is used with thermosetting plastics that plastic has viscous property or in free-flowing form material.

Typically, compression molding the process starts by directly placed the material in a hot mold, and the mold is closed by a hydraulic press. The condition to operate the PLA or synthetic biodegradable medical polymer is usually set at 5–10°C above the glass transition temperature of the polymer; therefore, a compression molding process in this paper is operating at a mold temperature 180°C and mold pressure 700 kPa with curing time divided into an upper plate (hot zone) and lower plate (cold zone). After the material is cured, the mold is opened, and the plastic package is pushed out. Compression molding can apply to many types of plastic production depending on the different mold, such as plastic film, rubber boots, water bottles, etc. It is a low-cost process and is capable of high-volume production. The working process of the compression molding can be described in the process diagram in Figure2.24.

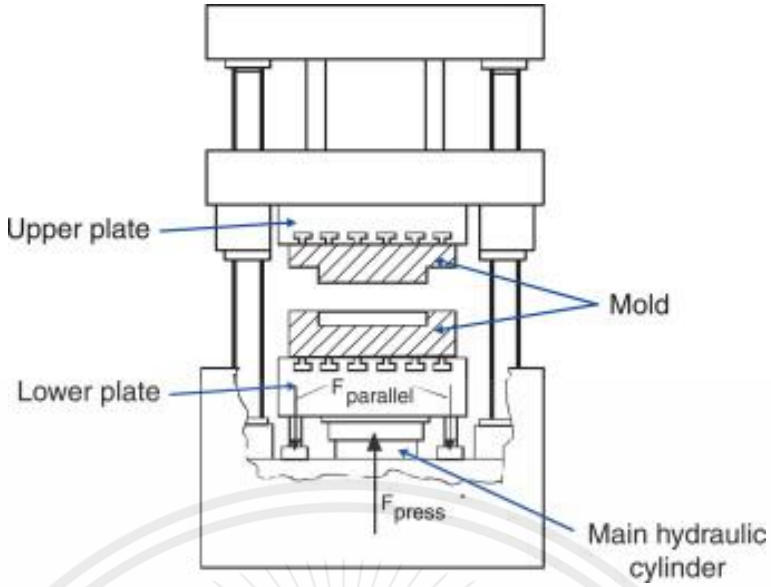

Figure 2.24: The working process of the compression molding [9]

2.9 Testing Methods

2.9.1 Polymer Degradation Testing

The polymer degradation is the changing of the properties of the polymer, for examples, tensile strength, color, and shape under a different kind of environmental factor, for examples, heat (from the sun, heat sources), chemical such as sulfuric acid or sodium hydroxide, humidity and we can observe the changes from the weight, visual analysis, OM, etc. The thermal degradation of polymer means to damage the chemical structure change of polymers at the elevated temperature.

2.9.2 Morphology by Using Optical Microscope (OM)

2.9.2.1 Principle of Work

It is a type of microscope that uses light as a visual aid. The operation of optical microscope starts with:

1.1 Light passes through various lens systems and is directed at the object.

1.2 Light will flow into our eyes, where the light within the system reflected into our eyes will allow us to see the image by looking through the eyepiece lens, as shown in Figure 2.25

2.9.2.2 The Optical Components of The Microscope

1. Eyepiece

The lens at the top that you look through. It contains two or more lenses that focus the image. It usually has a 10x magnification.

2. Turret

This holds two or more objective lenses and can be rotated easily to change magnification power. Typically, when viewing a slide, it is best to start the magnification at the lowest and then work your way upwards.

3. Objective

One or more objective lens that collects light. The lenses are usually in a cylindrically shaped tube. The shortest lens has the lowest power, i.e., the lowest level of magnification; the longest one is the lens with the most significant magnification power. The objective lenses usually have magnification power 5x, 10x, 20x and 50x.

4. Focus Wheel

These are wheels that move the stage in the vertical plane. There are also wheels for adjusting coarse and focus. Some microscopes, however, do allow focus at the eyepiece as well.

5. Frame

The frame consists of the arm, the base, and the bodywork of the microscope. It allows attachment of the focus wheels and the stage to the microscope.

6. Illuminator

A light source is used in place of a mirror. Most microscopes do allow manual light adjustment via a wheel located near the base.

7. Condenser

The function of the condenser lens is to focus the light onto the specimen. To increase the quality, the condenser lens may also have filters or a diaphragm.

8. Stage

A platform underneath the objective provides a platform for the slide to be viewed. In the center of the stage is a hole that allows light to pass through. The stage also has arms typically to hold the slide in place.

Figure 2.25: Optical component of optical microscope

2.9.2.3 Application: used to view the surface characteristics of the workpiece in a two-dimensional format, the magnification can be determined according to the needs.

2.9.2.4 Test Specimen Characteristics: The specimen is small enough to be placed on a platform.

2.9.3 Tensile Testing

2.9.3.1 Principle of Work

Apply tension to the specimen at a constant tension speed and record the tension force that changes with the elongation of the material, while the test piece is stretched, it will have resistance. The resistance of this test specimen has the effect of enabling the force meter to measure the force with force measured in kilograms (kg) or Newton (N). In the test, the specimen must be pulled until the specimen is torn apart. The maximum resistance of the test piece is the result of measuring the force. Therefore, the specimen can withstand the maximum tensile force equal to the resistance of the test specimen before it is torn apart.

2.9.3.2Characteristics of The Result:

This test is an essential mechanical testing method and the data obtained from this test, including yield point, tensile strength, and young's modulus. This test using tensile force and slowly pull the material and slightly increase the force until the material is a fracture and record the relationship between tensile stress and tensile strain (stress-strain curve). Figure 2.26 shown example of stress-strain curve.

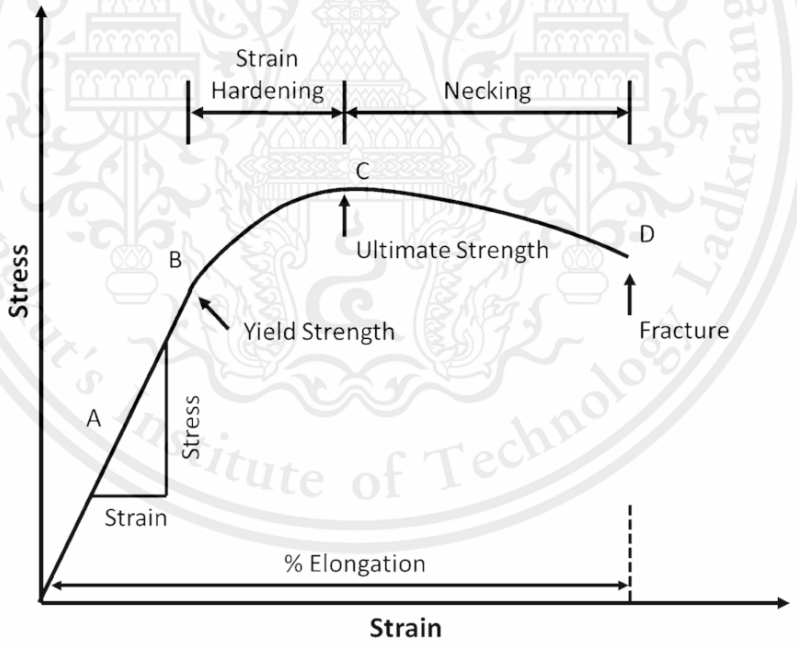

Figure 2.26: Stress-strain curve

2.9.3.3 Test Sample Characteristics

The sample to be analyzed will be in solid form according to standard reference size such as ASTM D638 (Types I - V dumbbell-shaped specimens),

2.9.4 Thermogravimetric Analysis (TGA)

TGA is a technique for analyzing the stability of materials, especially polymers when they are heated by measuring the weight of the material changing over a temperature range with a susceptible balance. This technique is suitable for analyzing material dynamics involved in gas absorption, water evaporation, crystallization due to phase change and decomposition. The various reaction involving the decomposition of sample due to the heat can be written as follows:

$$A(solid) \xrightarrow{Heat} B(soild) + C(gas)$$

2.9.4.1 Components of Thermogravimetric Analysis (TGA)

Thermogravimetric analysis instrument has three major components: furnace, balance and control unit as Figure 2.27

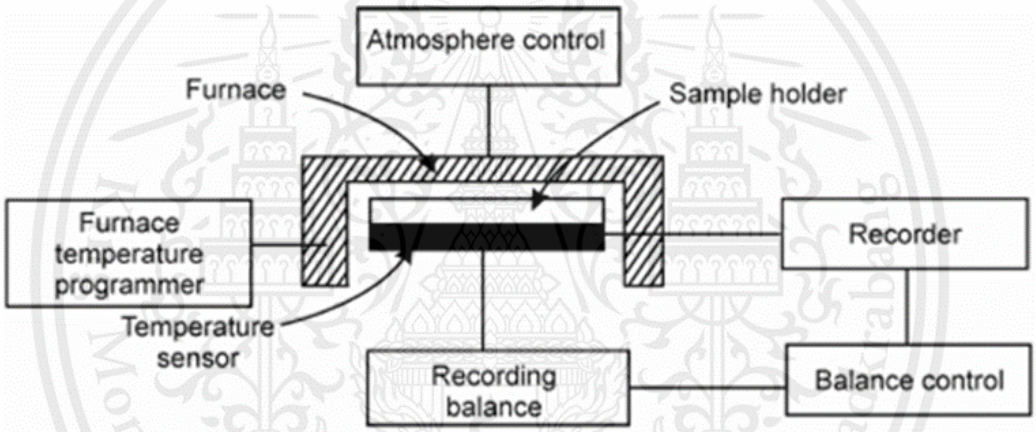

Figure 2.27: The component of thermogravimetric analysis instrument (TGA) [18]

1. Furnace

The furnace installed in the thermogravimetric analysis instrument has two temperature ranges between room temperature to 1000 ℃ and room temperature to 1500 ° C, depending on the material used to make the furnace. The Good furnace has properties inert to all reaction at every temperature range, such as ceramics.

2. Balance

Electrical balance in thermogravimetric analysis systems is a small electronic balance sensitive to detect the weight changes when the weight of the sample is increased or decreased. The sample is generally loaded in the balance for 5 to 50 mg, and there are three types of sample balances, as shown in Figure2.28

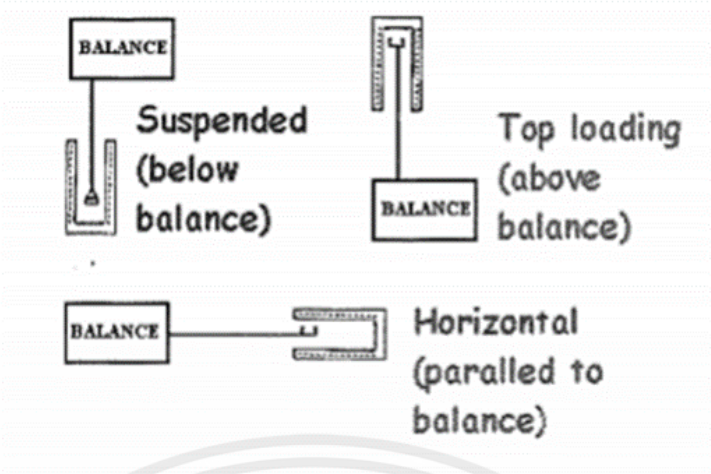

Figure 2.28: Types of TGA balance [18]

This pattern has similar pros and cons, depending on the design and development of the company. For example, the sample placed in the balance must be heated evenly and evenly and the material to be made. The balance should be made from material that resists corrosive chemicals and vapors from burning of material, for example, chlorine gas and sulfur gas.

3. Control Unit

Currently, the analyzers are recorded and interpreted by a PC with a software program installed. The Control unit work and record results to store the data obtained from analysis. The program is designed to have the ability to calculate the analysis results and display results in various forms such as T_g, percent weight loss (%wt), etc.

2.9.4.2 The Analysis Result from Thermogravimetric Analysis

The curve obtained from the TGA, called the TGA thermogram or thermal decomposition curve, is the relationship between the mass change over time or temperature in different degradation testing as shown in Figure 2.29, a graph showing the change in mass of the sample with respect to temperature through one stage of decomposition.

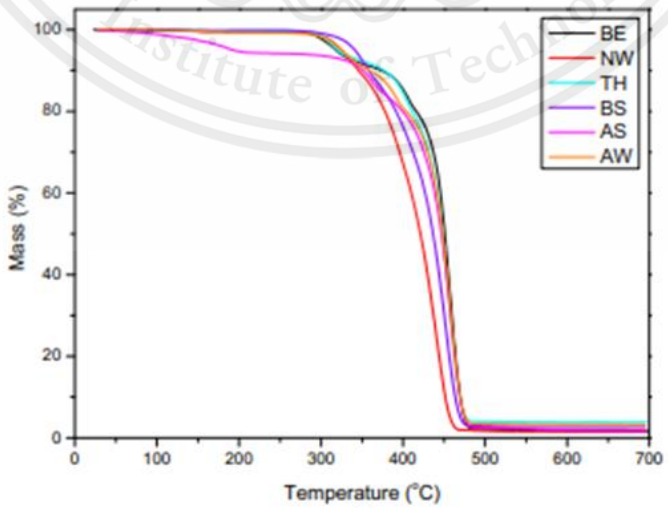

Figure 2.29: The curve obtained from TGA analysis [19]

2.9.5 Differential Scanning Calorimetry (DSC)

DSC is a technique used to test materials by measuring the thermal energy and temperature of the sample compared with the standard substance when it is physically changed, or chemical changes such as fusion and state change, crystal deformation, chemical reactions where the area under the graph occurs are related to the thermal change of the sample. In sample analysis, samples are placed on an aluminum plate housed in a temperature-controlled furnace. Inside the stove, there will be a reference material that is a blank aluminum plate to be used compared to the sample under the same condition. Figure 2.30 shown the component of differential scanning calorimetry (DSC).

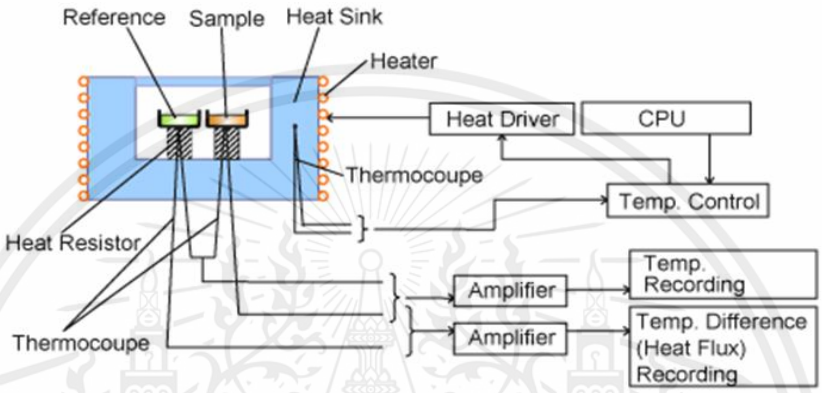

Figure 2.30: General components of differential scanning calorimetry instrument (DSC) [28]

The DSC consists of two trays: A sample tray and a reference tray placed aside together, on top of the same heater. The experiment started by heated both trays, then the DSC controls the rate of temperature rise (10 °C per minute). Both trays are heated separately with the same rate of heat increase throughout the experiment. The analysis in different degradation testing result from differential scanning calorimetry is shown in Figure 2.31

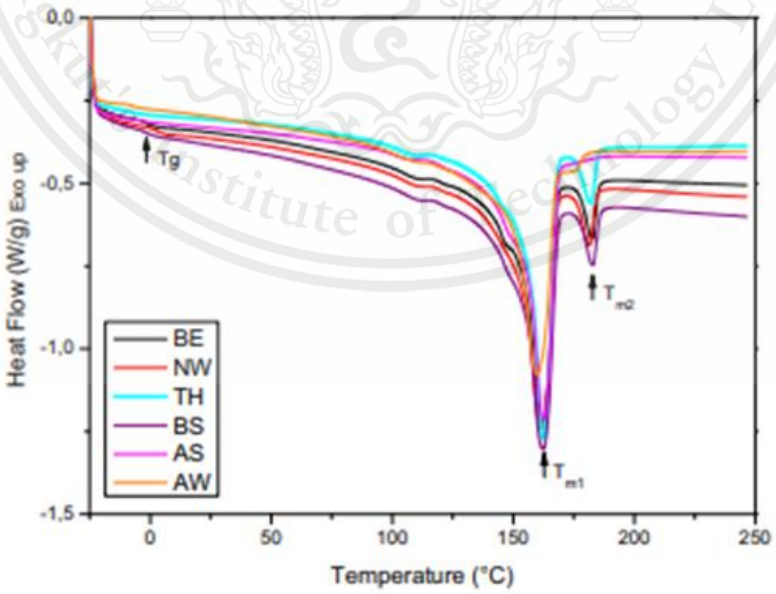

Figure 2.31: The curve obtained from DSC analysis [19]

The curve obtained from DSC analysis show the relationship between the heat flow to the sample and the temperature. The endothermic and exothermic peak indicate the point where the energy is absorbed or released by the sample as in Figure 2.34 which is the DCS curve for polymer testing and T_g is glass transition temperature, T_m is the melting point of polymer. In this study the DSC 204 F1 model from scientific instrument center at faculty of science, KMITL was used for the experiment. The Figure 2.32 shown the DSC model: DSC204 F1 from the faculty of science.

Figure 2.32: Differential scanning calorimetry (DSC), Model: DSC 204 F1 [20]

2.9.5.1 Features of DSC 204 F1 Model

1. It is a thermal analyzer that uses the principle of Heat Flux Design to measure heat change or increased sample temperature or decrease compared to the reference substance. For the analysis of melting point, glass transition temperature, crystallization, curing, endothermic and exothermic reactions, heat capacity Oxidative Induction Time, etc.

2. The machine can analyze solid samples and various types of fluids, including synthetic polymers, natural polymers, paint, rubber, plastics, composite materials, textiles, adhesives, resins, etc.

3. The test temperature range of the machine can be supported from -90 to 550 degrees Celsius using the Two-Stage Refrigerated System (TSRS).

4. There is a temperature control system for the electronic part of the machine to make the signal obtained from the measurement more stable.

5) Modulated DSC (MDSC) technology with wavelength control and can transition of complex samples or samples that can be slightly fused and distinguish the heat flow obtained from the test sample into reversing heat flow and non-reversing heat flow.

6) Able to test for thermal capacity (Direct Cp Measurement) from MDSC mode.

2.10 Literature Review

2.10.1 According to the research "Effect of spent coffee grounds filter on the physical and mechanical properties of poly (lactic acid) bio-composites film" [13].

This paper aims to study the effect of spent of grounds filters on the physical and mechanical properties of polylactic acid by adding the different concentrations of coffee (0%,5%,7.5%, and 10% wt.%) in this bio-composite (PLA/SCG). Bio-composites were prepared using two steps (i) Mixed preparation using a twin-screw extruder process. (ii) Blow films extrusion process. Then, PLA/SCG composite films ready to be tested in the physical, mechanical method.

When we observe the increasing in amount of spent coffee grounds (SCG) content a rough surface of the film, it can be explained that SCG particles can be dispersed in the PLA matrix and have similar particle size. Furthermore, in the part of physical properties, the effect of SCG contents in the composites show that as the SCG increases, the melt flow rate also increased with SCG content. When increase the concentration of coffee ground in composition, the viscosity should be reduced. It can be used as a plasticizer and internal lubricant in the composite to enhance the mixing processes. Finally, on the mechanic part, coffee grounds help to increase elongation value that makes bio-composite more flexible and the brittleness decreased. Therefore, SCG has the suitable properties. This is the method that can reduce the SCG waste and meanwhile add its value.

2.10.2 According to the research "The Coffee grounds as filler for pectin: Green composites with competitive performances dependent on the UV irradiation" [14].

This paper aims to study the effects of using the coffee ground as filler to the pectic for biofilm production, which varies the coffee ground concentration from 10 to 40 wt%. The coffee waste was rinsed with the boiling water several times and then distributed in deionized water to obtain an aqueous solution and was filtered with mesh size of 700 nm. Then, the solution was dried and milled by mortar and keep in the desiccator at room temperature. The pectic was obtained from apple with 75% methyl esterification. The calibration of the temperature was made by thermogravimetric analysis (TGA) to determine the maximum temperature of pectin and coffee degradation. The result shows that the coffee particles are well distributed in the polymer matrix, and the average area of the coffee ground depends on the filler composition. The tensile test was performed to determine the elastic modulus and elongation at breaking point which was measures five times for each film and find the averages. The results show that using UV curing promotes the films' mechanical properties by decreasing the breaking stress and the ultimate elongation, increasing pectin/coffee film's elasticity but does not affect Young's modulus. The wettability was measured by the contact angle of the coffee pectin film; the result shows that the filler's addition can increase the properties of hydrophobic and increase the film roughness.

2.10.3 According to the research "Effect of different degradation types on properties of plastic waste obtained from espresso coffee capsules" [15].

The study aims to eliminate the plastic wastes obtained from the coffee capsule by using different types of degradation, including natural weathering conditioning (NW), thermal conditioning (TH), acid and base solution conditioning (AS, BS), accelerated weathering conditioning (AW, UV and humidity). The samples were weekly taken and weighed in measuring balance and observed the weight change. The characterization methods were proceeded by thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), and visual analysis. The results show that there were 3 degradation stages. The temperature that the degradation of PP started is between 275 °C to 335 °C. From the visual analysis, the sample

using NW, TS, and BS were not visually changed, but some scratch occurs in NW. In the AW conditioning, the color changes to yellowish, which is the UV light's substantial degradation. As a condition, the sample does not change if there is some delamination on the samples. The result shows that for the water absorption for method of NW, AS, and BS, the %mass was increased at the beginning and then it would be oscillated, and a constant of mass was finally obtained after 80 days for AS and BS. The mass reduction occurs because the humidity does not absorb on PP hydrophobic surface.

2.10.4 According to the research "Development and characterization of biopolymeric films of galactomanans recoverd from spent coffee grounds" [31]

The study aimed to improve the bio-polymer film produced from galactomannans recovered from spent coffee grounds using alkaline and enzymatic treatment before the recovery process. The paper shows that the bio-polymer film was excellent because the cellulose in the galactomannans plays an essential role in improving mechanical properties such as percent deformation (%), tensile strength (MPa), and the water vapor permeability.

2.10.5 According to the research " Development of good thermal stability and impact strength bioplastic packaging" [29]

In this study, polymers were mixed with polylactic acid (PLA) and natural rubber (NR) at different ratios of 95/5, 90/10, 85/15, and 80/20 percent by weight (%wt). They were prepared by using a melting technique with an internal mixer. The test specimen was shaped by compression press, mechanical properties, heat distortion temperature (HDT), and the morphology of the polymer composite between PLA and NR. As a result of the experiment, it was found that:

1. The impact strength and elongation at break values of the PLA and NR mixtures were increased when the NR content was increased to 15% by weight. While the tensile strength and modulus decreased.
3. The HDT value of the polymer blends was slightly lower.
4. Scanning electron microscopy (SEM) images show that PLA and NR are incompatible.

Therefore, the maximum elongation at the break of the blended polymer was the highest when the NR content was 15% by weight [PLA / NR (85/15)] with a value of 167.22 kJ per square meter, 54.31 kJ per square meter, and 257.85 percent, respectively. Thus, the polymer mixture of PLA and NR at a ratio of 85/15 percent by weight (wt%) was selected to study the effect of poly (D-lactic acid), PDLA. To mechanical properties Thermal and HDT properties of the composite polymer between PLA and NR.

CHAPTER 3
Experimental Procedures

3.1 Introduction

In this chapter, we described the procedures to produce the bio-composite film (PLA/SCG) in different concentrations and design the conditions in each experiment to test the bio-composites film.

3.2 Materials

3.2.1 Filler: Spent Coffee Ground (SCG)

SCG used in this work is a by-product of coffee bean beans roasting process from Starbuck Coffee, Thailand which is 100% Arabica. The species of this coffee is brought from Latin America and Asia.

3.2.2 Matrix: Polylactic Acid (PLA)

In this research, PLA from IngeoTM Biopolymer 4043D (25kg), NatureWorks LLC supplied film grade was used. The general properties and temperature profile of PLA 4043D are shown in Tables 3.1 and 3.2, respectively.

Table 3.1: General properties of PLA4043D reported by the material supplier.

Physical Properties	Value	Standard
Specific gravity, g/cc	1.24	ASTMD792
Melt Flow Rate (MFR), g/10min	6	ASTMD1238
Clarity	Clear	-
Peak Melt Temperature,℃	135-160	ASTMD3418
Glass Transition Temperature,℃	55-60	ASTMD3418
Heat Distortion Temperature	55	ASTME2092
Percentage of D-Isomer, %	4.3-4.8	-
Mechanical Properties		
Tensile Yield Strength, MPa	60	ASTMD882
Tensile Strength at Break	53	ASTMD882
Tensile Elongation, %	6	ASTMD882
Tensile Modulus, MPa	3.6	ASTMD882

Table 3.2: Temperature profile of PLA 4043D reported by the material supplier.

Section	Temperature Profile (℃)
Feed Section	180
Transition Section	190
Metering Section	200
Die	200
Screw Speed	10-100 rpm

3.3 Equipment

1. Sieve shaker from School of Engineering, Department of Chemical Engineering, KMITL.

Figure 3.1: Sieve shaker

2. Oven from School of Engineering, Department of Chemical Engineering, KMITL.

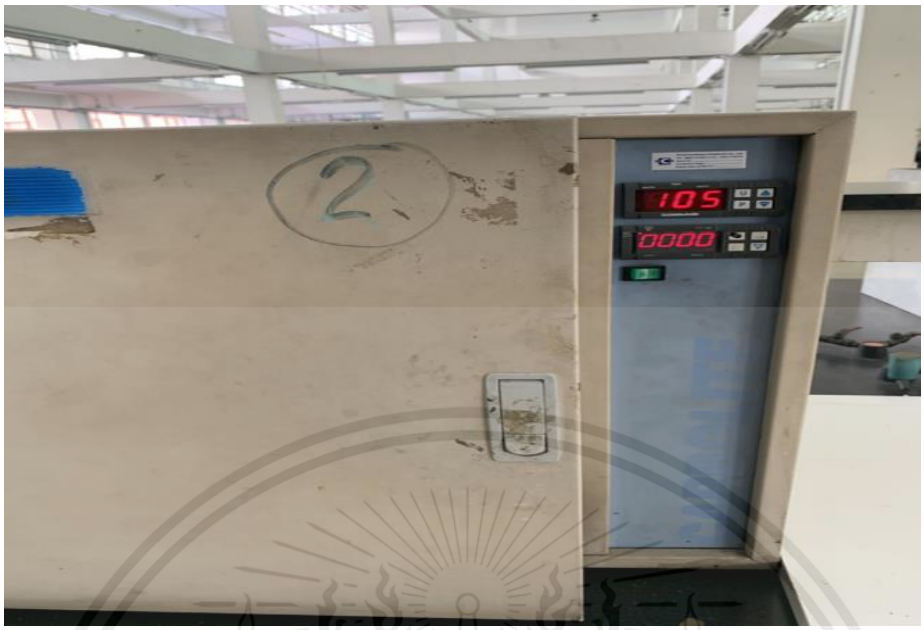

Figure 3.2: Oven

3. Compression molding machine, Brand: Labtech Engineering Co. LTD from school of Science, Department of Industrial Chemistry, KMITL.

Figure 3.3: Compression molding

4. Twin-screw extruder, Brand: Brabender^{R}, Model:PL2100 from School of Science, Department of Industrial Chemistry, KMITL.

Figure 3.4: Twin-screw extruder

5. Differential scanning calorimetry (DSC), Brand: NETZSCH, Model: DSC 204 F1 from Scientific Instrument Center, School of Science, KMITL.

Figure 3.5: Differential scanning calorimetry (DSC)

6. Optical Microscope (OM) Brand: Olympus, Model: BX53M from the School of Engineering, Department of Industrial Engineering, KMITL

Figure 3.6: Optical microscope (OM)

7. A Universal Testing Machine (UTM), from School of Science, Department of Industrial Chemistry, KMITL.

Figure 3.7: A universal testing machine (UTM)

8. Pelletizing Machine, Brand: Bosco from School of Science, Department of Industrial Chemistry, KMITL.

Figure 3.8: Pelletizing machine

9. Desiccator from School of Engineering, Department of Chemical Engineering, KMITL.

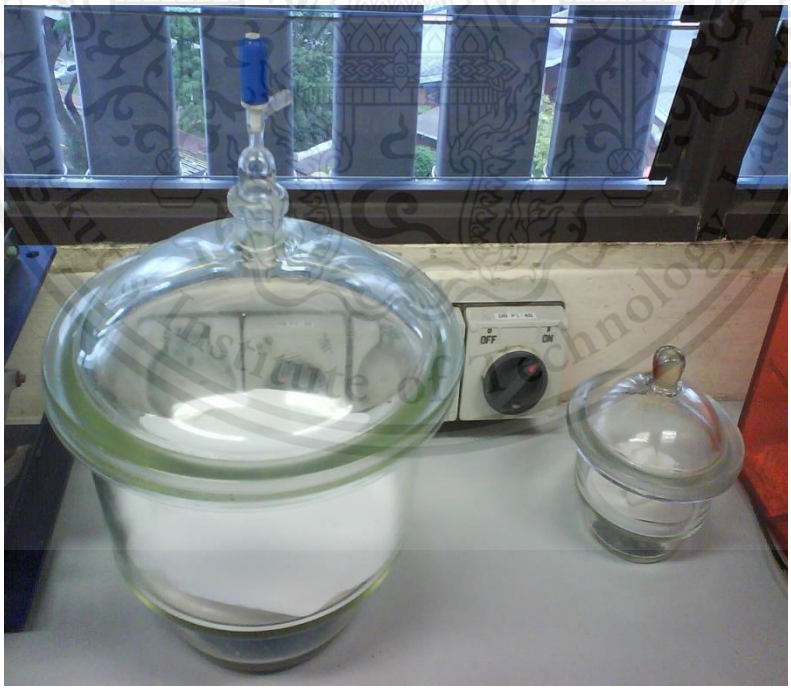

Figure 3.9: Desiccator

3.4 Material Pretreatment

3.4.1 SCG Pretreatment

SCG was dried in the oven at a temperature of 105°C for 24 hr. (Figure 3.10). After drying, SCG was processing in a ball mill and then sieved through an 88 µm mesh screen (particle size ≤ 88 µm). Finally, SCG was prepared as a brown powder and stored at room temperature for further processing (Figure 3.11).

Figure 3.10: SCG dried in the oven at a temperature of 105°C for 24 hr.

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Figure 3.11: SCG was prepared as a brown powder after the sieve an 88 µm mesh screen

3.4.2 PLA Pretreatment

Before using PLA, we need to dry it. Therefore, in-line drying is required. The moisture content of less than 0.025% (250 ppm) is recommended to prevent viscosity degradation (company recommendation). Typical drying conditions are 2 hr. at 105ºC in the oven. After that, PLA was stored in a zip lock bag and delivered to the desiccator to prevent moisture and wait for further process.

Figure 3.12: Drying PLA for 2 hr. at 105ºC in the oven

3.5 Processing of Bio-composite Polymer

1. The PLA matrix was mixed with SCG to obtain PLA/SCG composite polymer by adding different concentrations, as shown in Table 3.3 By using a twin-screw extruder with a screw speed of 10 rpm and the temperature, the profile was varied from 185°C at the feeding zone to 200°C at the die (Figures 3.13 and 3.14 as show step by step). Furthermore, Table 3.4 describes the temperature profile and screw speed that should be considered in the operation of a twin-screw extruder.

Table 3.3: The mixing ratio of coffee grounds and PLA in bio-composite polymer compound (%wt)

Bio-Composite Polymer	**Polylactic-Acid, PLA (wt%)**	**Coffee Grounds, SCG (wt%)**
PLA	100	-
PLA/SCG 5 wt%	95	5
PLA/SCG 10 wt%	90	10
PLA/SCG 15 wt%	85	15
PLA/SCG 20 wt%	80	20
PLA/SCG 25 wt%	75	25

Table 3.4: The temperature profile and screw speed that should be considered in the operation of a twin-screw extruder.

Section	**Temperature Profile (℃)**
Feed Section	185
Transition Section	190
Metering Section	195
Die	200
Screw Speed	10 rpm

Figure 3.13: Setting the temperature profile following the Table 3.4

Figure 3.14: Bio-composite polymer after out of the die

2.. After mixing, the bio-composites were cut into small pieces with pelletizing machine. Then put it to the room temperature to wait for the film forming process.

3.6 Processing of Bio-composites film

1. Bio-composites film were manufactured using compression molding. The PLA/SCG composite polymers melt process used the hot plate for melting the polymer in the mold for 2 min and operation of a mold was at temperature of 180°C and the mold pressure of 700 kPa. Then, bio-composite was removed from the hot plate and the obtained film was cured with cold temperature cooling with the cooling plate for 1 min. The steps to produce the bio-composite film are shown and described in Figures 3.15 and 3.16, respectively.

Figure 3.15: Put the polymer into the mold

Figure 3.16: Curing time in each plate in the compression molding

3.7 Testing Method

3.7.1 Degradation Test

3.8.1.1 Degradation by Natural Weathering Conditioning (NW)

The PLA/SCG film samples were prepared in the square with the size of 2 cm x 12 cm with different concentrations (1 sample/wt% concentration). The samples were buried in the soil in a container for 60 days, and all samples were dug for each week. Then, the samples were cleaned with distill water and dried with a towel paper and weighed by using the measuring

balance. To obtain the soil for conditioning, the soil of 20 g was dried in the oven for 24 h and after that, check the soil temperature by using a thermometer and record every week. The natural weathering degradation (NW) is showed in Figure 3.17.

Figure 3.17: Natural weathering condition (NW)

3.7.1.2 Degradation by Thermal Conditioning (TH)

The PLA/SCG film samples with the size of 2 cm x 12 cm with the different concentrations (1 sample/wt% concentration). The samples were put into the tray at the positions, then the trays were brought into the oven and samples were dried at 70 °C for 60 days. The samples were collected for each week and weighed in by using the analytical balance to observe the weight change compared to the experiment's beginning and calculate the percent weight loss.

3.7.1.3 Degradation by Acid Solution Conditioning (AS)

The samples of 2 cm x 12 cm of PLA/SCG films with different concentrations (one sample/wt% concentration). The samples were immersed in 35%v/v of sulfuric acid (H_2SO_4) solution. Each sample was tested in sulfuric acid for 60 days. Each week, 1 sample was taken out from the container, cleaned with deionized water, dried with towel paper, and then weighed by the analytical balance to observe the weight changed and calculate percent weight loss for each week. The Figure 3.18 shown the step of degradation by acid solution conditioning.

Preparation for H2SO4 35%v/v for conditioning as following:

1. Prepare 98% concentrated sulfuric acid and pour it into a 100 ml beaker until 18.4 ml volume was obtained.

2. Take the prepared sulfuric acid and pour into the beaker and add water to a volume of 1 L.

Figure 3.18: Acid solution conditioning (AS)

3.7.1.4 Degradation by Base Solution Conditioning (BS)

The sample of 2 cm x 12 cm of PLA/SCG films was immersed into the 35% (v/v) of concentrated sodium hydroxide (NaOH) solution as shown in Figure 3.22. Each sample was tested in a 35% (v/v) concentration, and the samples were taken out from the container every 2 – 5 days, then cleaned with deionized water, dried with towel paper, and then weighed in analytical balance to observe the weight changed and calculate the percent weight loss.

Preparation of NaOH for conditioning test as following:

1. Prepare NaOH concentrate 2.5 molar and pour it into a 100 ml beaker until the volume was 70 ml.

2. Pour the NaOH solution into a beaker and add water until the volume was 1000 ml

3.7.1.5 Visualization

The sample from every different type of conditioning (before conditioning, NW, TH, AS, BS) are photographed and comparing the picture together to observe any possible visual change on the surface of the films.

3.7.2 Morphology

This method is used to study the structure of the surface of the PLA/SCG bio-composites film and SCG distribution in the different concentrations by using Optical Microscope (OM) Brand: Olympus, Model: BX53M from the Faculty of Engineering, Department of Industrial Engineering, KMITL. The samples were prepared before the test by placing them on the stage, as shown in Figure 3.19 using of 10x magnificent to observe samples surface.

Figure 3.19: Placing the sample on the stage of optical microscope (OM)

3.7.3 Thermal Testing

This method was used to evaluate the PLA melting point and glass transition temperature using DSC 204 F1 model as shown in Figure 3.5 from scientific instrument center at faculty of science, KMITL. For this test, the samples of PLA/SCG film with the SCG percentage (%w) of 0, 5, 10, 15, 20, and 25 were cut into small pieces and analyzed using dual scan technique with 40 ml/min of nitrogen flowrate with the heating and cooling rates of 10 °C/min from 30 °C to 250°C.

3.7.4 Tensile Testing

This method aims to test the tensile strength at break of the samples, the percent elongation at break, and the tensile modulus at the break by using a Universal Testing Machine (UTM) from the Faculty of Science Department of Industrial Chemistry, KMITL. The samples were prepared in the form of a plastic dumbbell (5 pieces/%SCG concentration) followed the ASTM D638. Figure 3.20 shows the samples in the form of a plastic dumbbell for tensile testing.

Figure 3.20: Samples in the form of a plastic dumbbell for tensile testing

The conditions used to test were as follow:

- Gage length = 30 mm
- Load cell 10 kN
- Speed 20 mm/min
- Force 10 kN
- Following the ASTM D638
- The sample's mechanical properties such as tensile strength at break, modulus at the break, elongation at average break values, are reported as shown in Table 3.5.

Table 3.5: A standard used to test the mechanical properties of PLA / SCG composite films.

Properties	**Standards used for testing**	**Reported unit**
Tensile strength at break	ASTM D638.	MPa
Modulus at the break	ASTM D638.	MPa
Elongation at average break	ASTM D638.	%

CHAPTER 4
EXPERIMENTAL RESULTS

4.1 Introduction

In this chapter, we present the experimental results were reported and discussed in order to determine the most capable constituents of the coffee grounds concentrations. The alteration of SCG/PLA film properties, such as mechanical tests, thermal tests, and degradation tests at various conditions. Finally, to study the properties of bio-composites film containing coffee grounds that can be further applied to plastic film making in the industry.

4.2 Testing Summary

4.2.1 Morphology

PLA/SCG bio-composites films were prepared by compression molding and the morphologies of PLA/SCG are shown in Figure 4.1

Figure 4.1(a) shows the relatively smooth surface of PLA surface and due to generally brittle, hard material of nature of PLA. On the other hand, for the results of bio-composite film (PLA/SCG) with the amount of 5, 10, 15, 20, 25 wt% SCG as in Figure.4.1(b) - 4.1(f). The figures show the distribution of SCG particles in the PLA matrix. The apparent phase separation of bio-composites film (PLA/SCG) results from the addition of SCG in the matrix; the greater amount of SCG added to the PLA matrix leaded to the greater agglomerated area of the particle size of the SCG in the matrix phase.

Moreover, when the volume of SCG into the matrix increased, the surface adhesion force between the PLA phase and the SCG particles was reduced, eventually resulting in spherical grooves appearing on the surface due to the increased in dispersion of SCG in the immiscible binary polymer blend increases the dispersed phase. Finally, this made SCG formed in the form of coalescence [29].

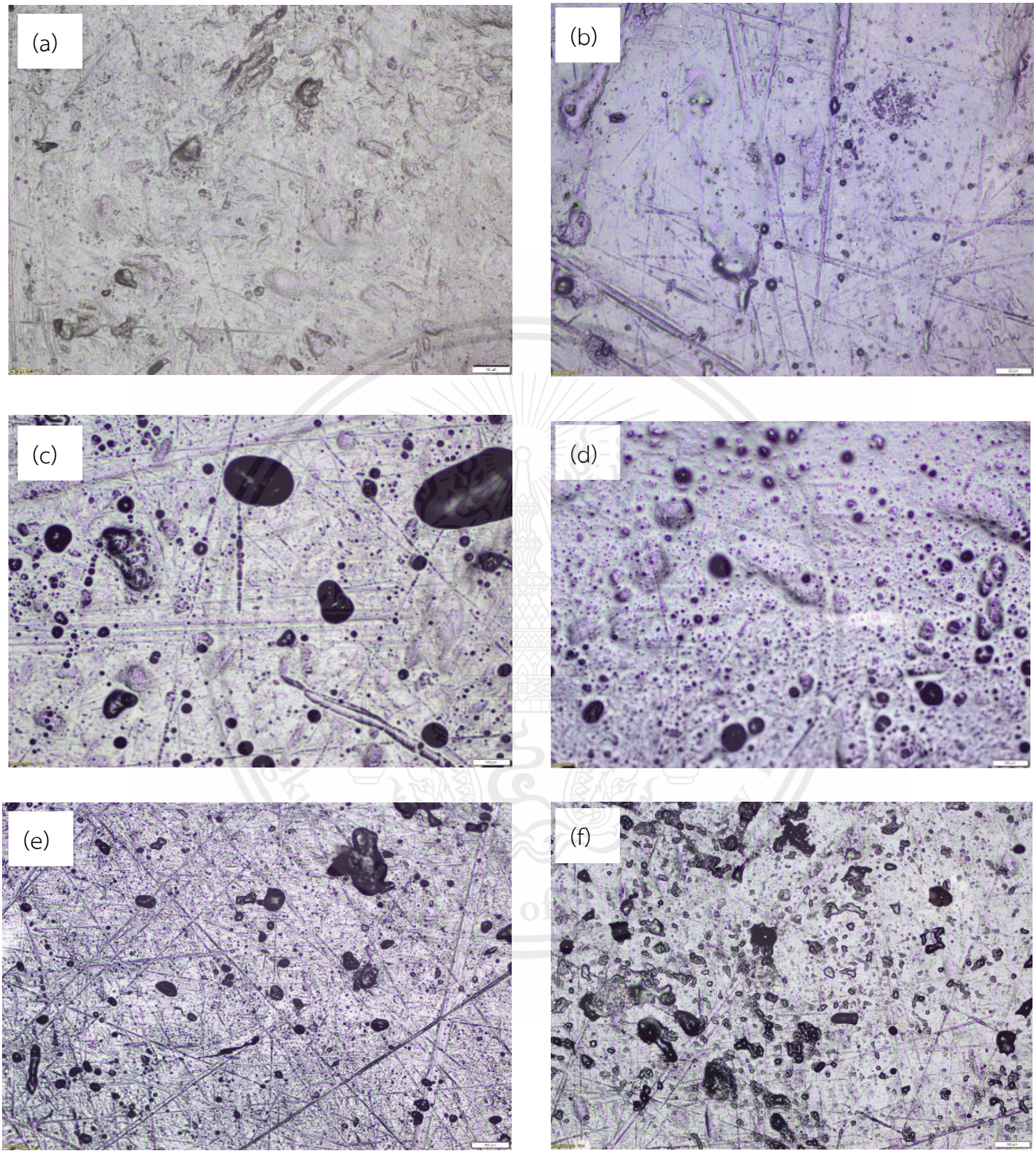

Figure 4.1: The surface areas of (a) PLA/SCG 0 wt%, (b) PLA/SCG 5 wt%, (c) PLA/SCG 10 wt%, (d) PLA/SCG 15 wt%, (e) PLA/SCG 20 wt%, and (f) PLA/SCG 25 wt% characterized by OM with the magnification of 10x.

4.2.2 Tensile Testing

Characteristics of the results will come from as a graph of the relationship between stress vs. strain curve in different SCG concentrations in the PLA/ SCG bio-composites film shown in Appendix 1. So, we are summarized in term of bar chart as follow:

4.2.2.1 Tensile Strength at Break

Figure-4.2 shows the tensile strength at break as a function of the SCG content in the PLA/SCG bio-composites films. The tensile strength at break of pure PLA was 52.44 MPa decreased to 21.47 MPa when increased SCG contents to 25 wt.% and this was the most reduction of brittleness when compared the Pure PLA with PLA/SCG bio-composites film in different SCG contents.

From the result, it can be concluded that the tensile strength at break of the PLA/SCG bio-composites film slightly decreased with increasing SCG content and attributed to the distribution of SCG in the PLA matrix because when SCG was added into the PLA matrix, PLA and SCG are incompatible, and phase separation occurs, resulting in lower adhesion between PLA and SCG and decreased in tensile strength as well. This corresponds to the Suranaree team's experiment [29] where natural rubber (NR) was added to the PLA matrix. When NR was mixed with PLA, natural rubber is naturally soft and stretchy, the toughness of NR/PLA bio-composite was increased resulting in a lower tensile strength.

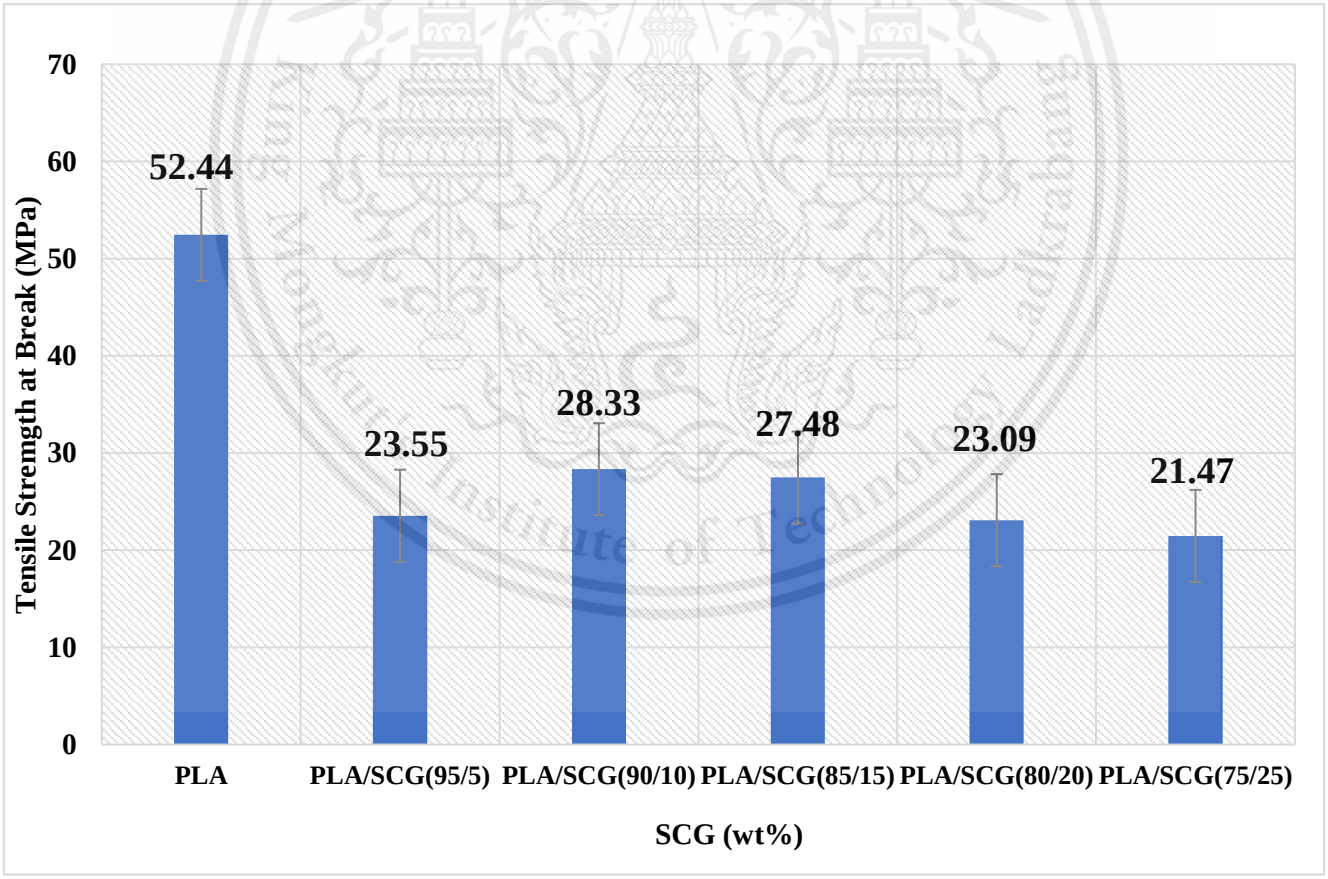

Figure 4.3: The effect of SCG content on tensile strength at break

4.2.2.2 Elongation at Break

Figure 4.3 shows the elongation at break as a function of the SCG content in the PLA/SCG bio-composite films. The results showed that the SCG contents were 5, 10, and 15 wt.%. It can be concluded that when SCG was added to the PLA matrix, it would slightly increase the elongation at break of the PLA/SCG bio-composites film. Furthermore, SCG/PLA seemed to be less brittle and more sticky compared to the PLA plastic.

When comparing the elongations at the break of PLA and the PLA/SCG bio-composites film, the maximum elongation at the break of PLA increased from 11.6% to 32.3% when add 15 wt% SCG and SCG improves the stretchability of the PLA/SCG bio-composites film. On the other hand, when the SCG contents were 20 wt% and 25 wt%, the maximum elongation at the break of the PLA/SCG bio-composites film decreased to be 28.58% and24.68% respectively because phase separation between PLA and SCG and the increase in the aggregation of SCG particles, resulting in many spherical dispersions of SCG in the PLA matrix, causing poor adhesion between PLA and SCG and further resulted in a decrease in the maximum elongation at break. However, the elongation at break of the PLA/SCG bio-composites film at all mixing ratios between PLA and SCG was higher than pure PLA.

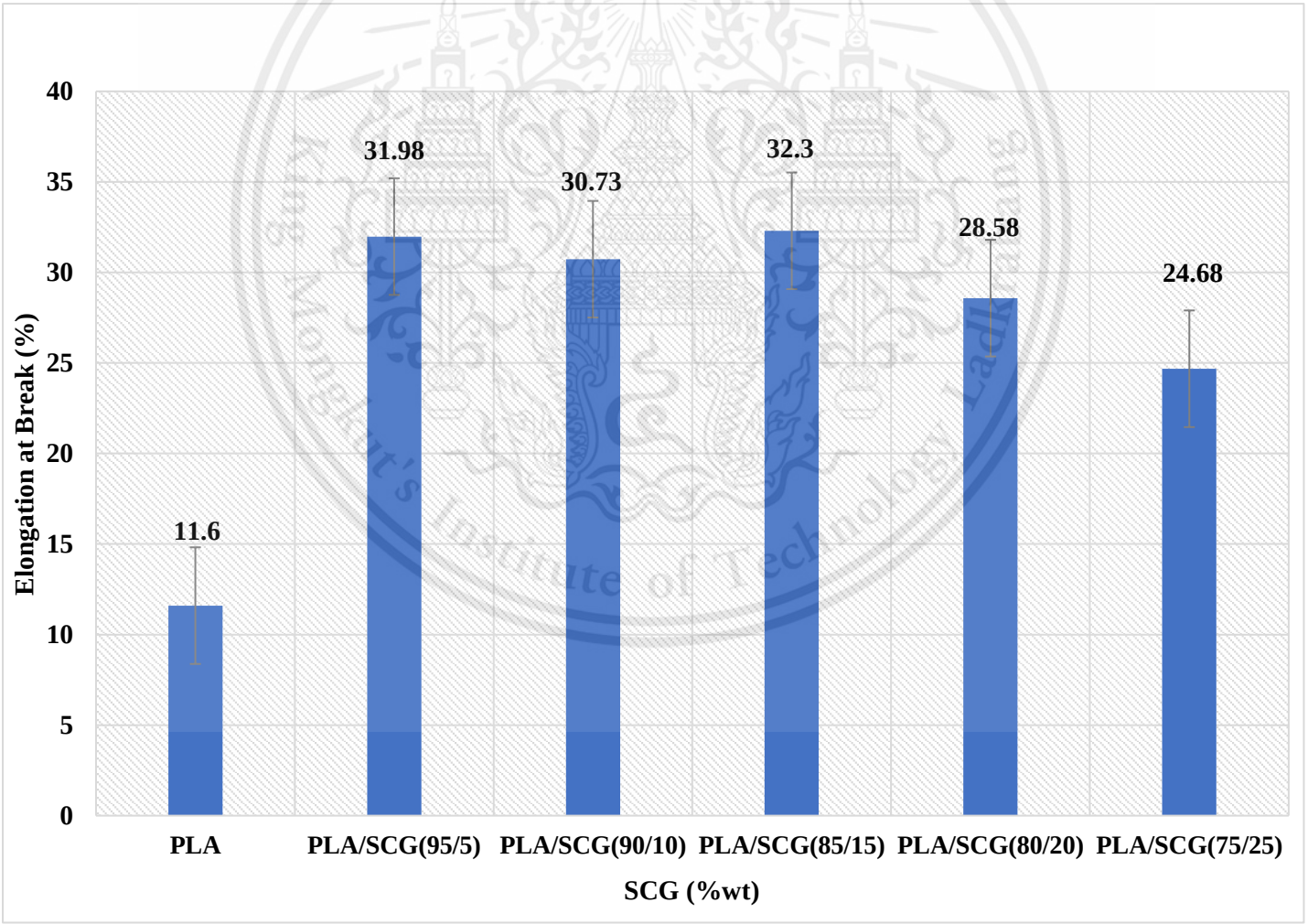

Figure 4.4: The effect of SCG content on elongation at break

4.2.2.3 Modulus at Break

Figure 4.4 shows the modulus at break as a function of the SCG content in the PLA/SCG bio-composites film. The tendency of modulus at break was decreased when the SCG was added into the PLA matrix. The modulus at break of pure PLA decreased from 253.25 MPa to 115.43 MPa when SCG contents increased to 25 wt%.

The result shows the tendency of modulus at break which was decreased when SCG was added into the PLA matrix with increasing SCG content, and the modulus at break of pure PLA decreased from 253.25 MPa to 115.43 MPa when SCG contents increased to 25 wt%. The reason of the decrease in modulus at break was that the PLA and SCG are incompatible when adding SCG into the matrix, reducing the adhesion bond between PLA and SCG and decreased modulus at break. The tensile strength at break, modulus at break, elongation at break of PLA and PLA/SCG bio-composite films at different quantities of SCG are summarized in Table 4.1.

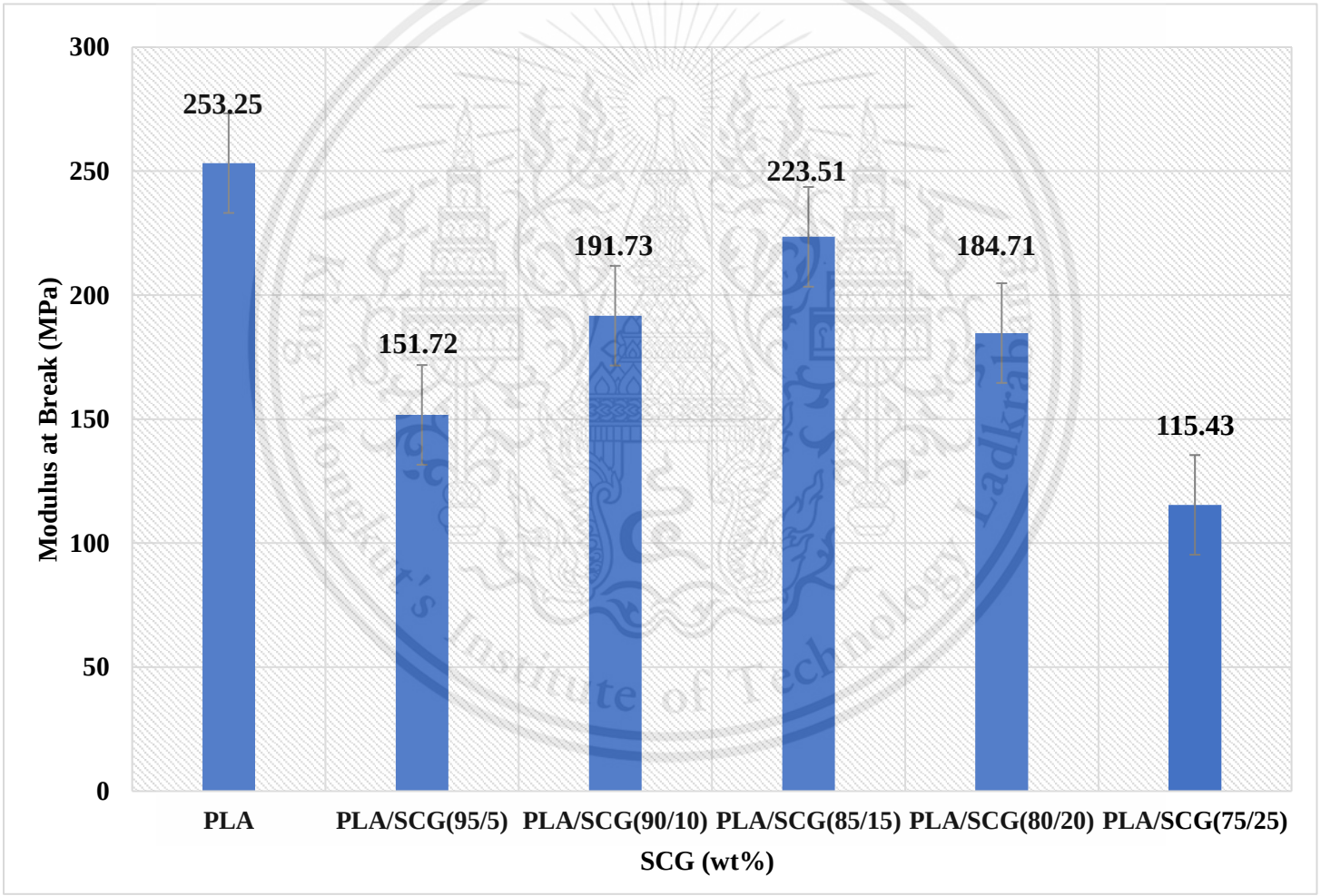

Figure 4.5: The effect of SCG content on modulus at break

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Table 4.1: Tensile strength at break, Modulus at brake, Elongation at break of PLA and PLA/SCG bio-composites film at different quantities of SCG (I - V dumbbell-shaped specimens)

Samples	Tensile Strength at Break (MPa)	Modulus at Break (MPa)	Elongation at Break (%)
PLA	52.44	253.25	11.6
PLA/SCG 5 wt%	23.55	151.72	31.98
PLA/SCG 10 wt%	28.33	191.73	30.73
PLA/SCG 15 wt%	27.48	233.51	32.3
PLA/SCG 20 wt%	23.09	184.71	28.58
PLA/SCG 25 wt%	21.47	115.43	24.68

4.2.3 Degradation Testing

4.2.3.1 Degradation by Natural Weathering Conditioning (NW)

The natural weathering conditioning was performed by conditioned of PLA that compounded with the spent coffee ground (SCG) by varying the coffee grounds concentrations from 0, 5, 10, 15, 20, 25 percent by weight (wt%) and buried in the soil in a container under soil condition soil for six weeks then take a sample for each concentration to weight every week and observe the weight change and collect the data as shown in Table 7.1 (Appendix 2).

Figure 4.5 shows that the weight of PLA/SCG bio-composites film is slightly decreased every week. The weight loss ranges from 1.150%-3.1732%, and PLA/SCG 15 wt% is the most weight loss. The reason for the reduction of the weight in PLA/SCG bio-composites film was probably die to the adhesion force of PLA/SCG composites film; when adding SCG in the matrix, SCG formed a lot of spherical in the PLA matrix, causing poor adhesion between PLA and SCG. Then, films were more porous on the surface. This corresponds to the research of Amonrat Lertworasirikul [10]. The degradation of PLA is caused by a lack of the main chain or side chain of the molecule in nature. They were arisen from biological activities such as soil surface temperature, pH, oxygen content in the soil, or the porosity of the workpiece.

Finally, when compared the percent weight loss between PLA and PLA/SCG bio-composites film, PLA is a less percent weight loss than PLA/SCG bio-composites film. It means that SCG in the matrix can increase the degradation rate of the results of the film on a more easily degraded soil under suitable conditions.

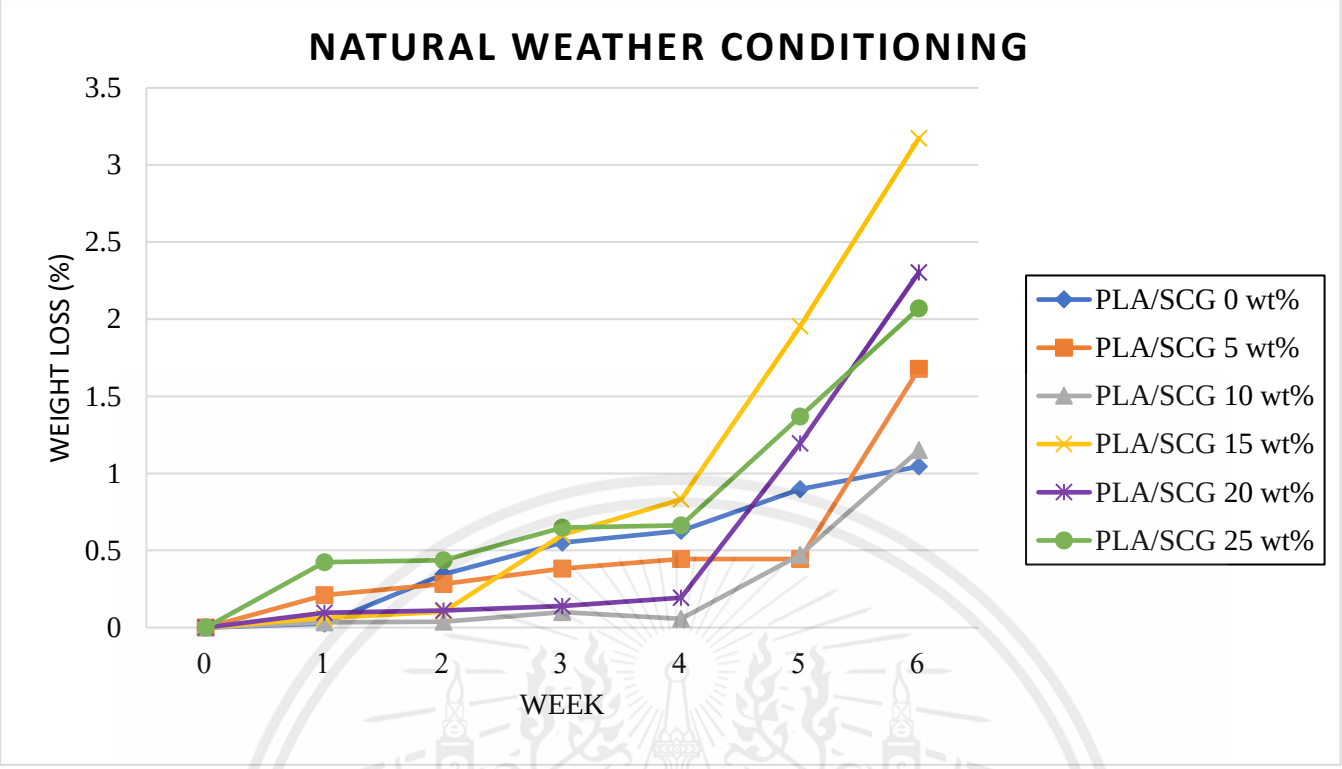


Figure 4.7: Percent weight loss for natural weathering conditioning of PLA and PLA/SCG bio-composites film at different quantities of SCG film when buried in the soil in a container under soil condition soil for 6 weeks

4.2.3.2 Degradation by Thermal Conditioning (TH)

The thermal degradation conditioning was performed by conditioned of PLA that compounded with the spent coffee ground (SCG) by varying the coffee grounds concentrations from 0, 5, 10, 15, 20, 25 percent by weight (wt%) and conditioned at 70 ° C, then take a sample for each concentration to weight every week and collect the data and observe the weight as shown in the Table 7.2 (Appendix 2).

This experiment shows that the weight of the PLA/SCG bio-composites film is gradually decreasing every week, and the weight change was ranged from 0.075583% to 0.650512%. It was observed that at SCG 5 wt% concentrations, weight was reduced the most, and at 25% SCG concentrations, weight loss was the least and can be observed from the graph in the Figure 4.6. It can be seen that the weight of that bio-composites film, even though it decreased, was not much different from the original weight significantly. This summary should be ensured by taking the longer experimental durations.

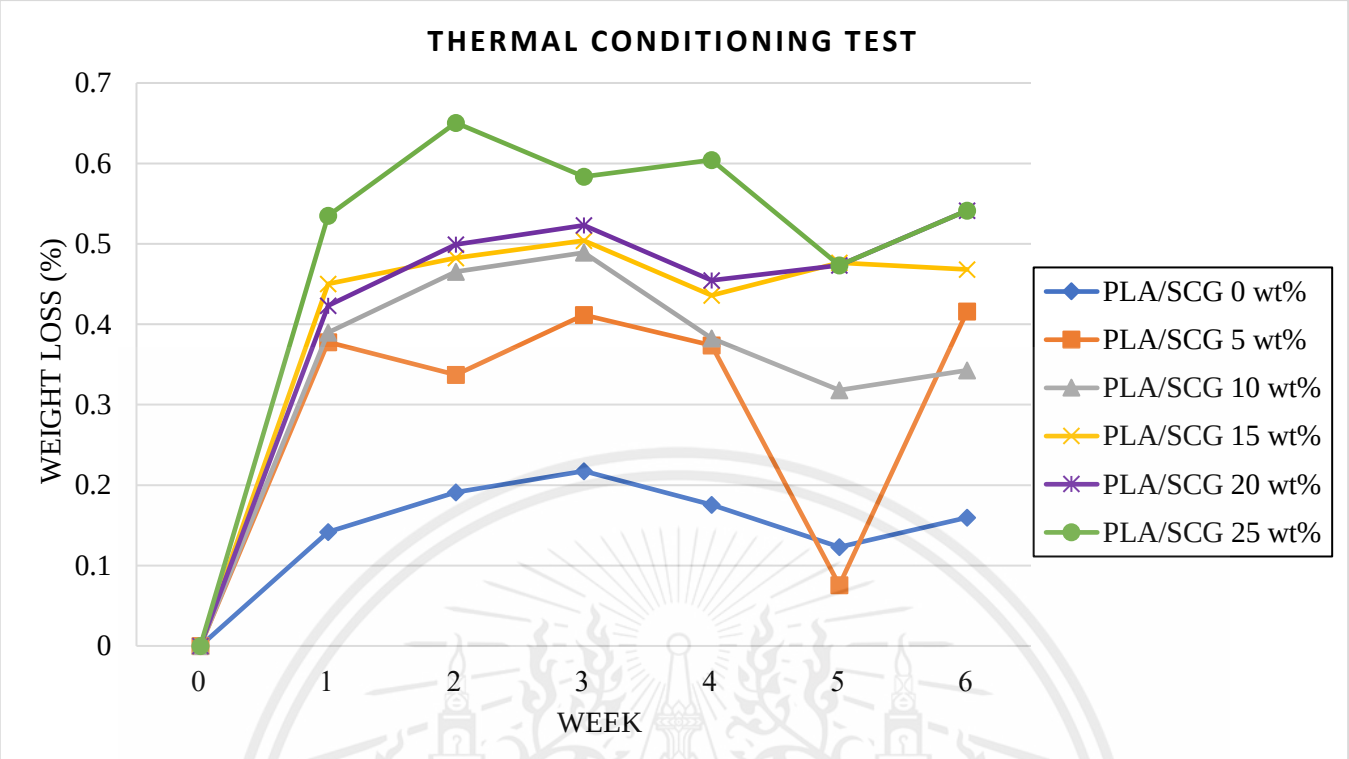


Figure 4.8: Percent weight loss for thermal conditioning of PLA and PLA/SCG bio-composites film at different quantities of SCG film when the samples were conditioned in an oven at 60 ℃ for 6 weeks.

4.2.3.3 Degradation by Acid Solution Conditioning (AS)

Acid solution degradation test was performed by conditioned PLA that compounded with the spent coffee ground (SCG) by varying the concentration of coffee grounds from 0, 5, 10, 15, 20, 25 percent by weight (wt%) with sulfuric acid at a concentration of 35% by volume (v/v) and remove the plastic samples then come to weigh every week to observe the weight change as shown in the Table 7.3 (Appendix 2).

From the results of the experiment, it revealed that the weights of the PLA/SCG bio-composites film were rarely changed from the original. Because the PLA does not react with sulfuric acid, and conversely, the weight of bio-composite film was gained in some week since the sample absorbed moisture from sulfuric acid, the weight change is plotted by compared the conditioned samples with the week 0 of experiments, and this can be observed from the graph in Figure 4.7. Therefore, from the experiments, it could be concluded that sulfuric acid had little effect on the degradation of PLA/SCG bio-composite film.

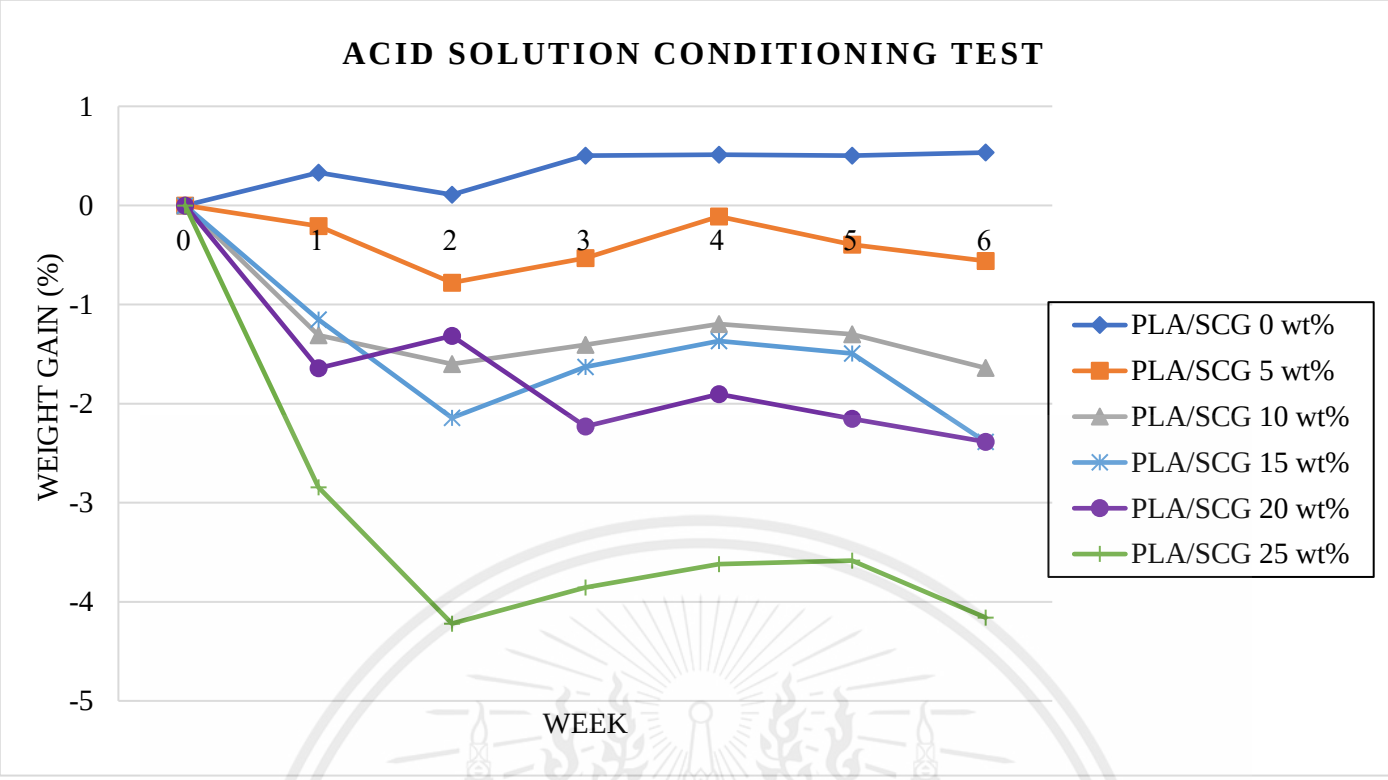


Figure 4.9: Percent weight gain for acid solution condition of PLA and PLA/SCG bio-composites film at different quantities of SCG film when the samples were conditioned in 35% (v/v) concentrated sulfuric acid for 6 weeks.

4.2.3.4 Degradation by Base Solution Conditioning (BS)

Base solution degradation test was performed by conditioned PLA that compounded with the spent coffee ground (SCG) by varying the concentration of coffee grounds from 0, 5, 10, 15, 20, 25 percent by mass (wt%) with sodium hydroxide at a concentration of 35% (v/v) by volume and remove the plastic samples then weight every 2 – 6 days to observe the weight change as shown in the Table 7.4 (Appendix 2). From the result of the experiment shown in Figure 4.8, the PLA plastic that was not compounded with SCG have less weight loss, on the other hand, when SCG is added, it can be found that the weight of the samples was significantly reduced because SCG helped promoted depolymerization reaction with sodium hydroxide causes plastics to degrade rapidly. Therefore, SCG can promote the degradation of PLA with sodium hydroxide solution.

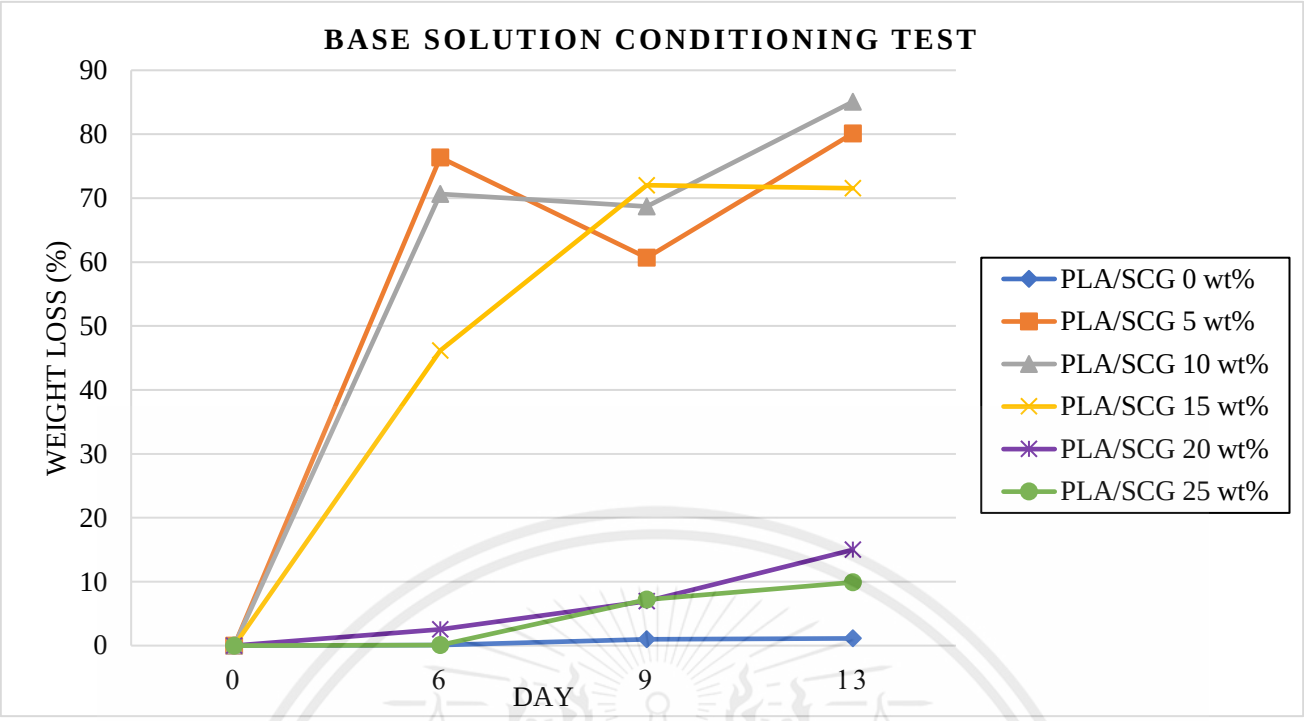


Figure 4.10: Percent weight loss for base solution conditioning of PLA and PLA/SCG bio-composites film at different quantities of SCG film when the samples were conditioned in 35% (v/v) concentrated sodium hydroxide.

4.2.4 Visualization

In this experiment, the sample of NW, TH, AS, BS were taking the photo to define the visible change on the surface this week, and pictures were compared with the sample before conditioning. Total experiment time was six weeks for NW, TH, AS, and 13 days for BS.

For the PLA/SCG bio-composites films conditioned by natural weathering conditioning, the sample's surface has noticeable corrosion characteristics and thicker compared with the original one. Moreover, PLA/SCG 20 wt% in the back view has a new crack occurs at the bottle of sample in week three and all samples have a small hole happens on the surface in week three. However, the PLA sample has no visible change on the surface, only a tiny spot caused by compression molding, and the color of sample has increased yellowness. Figures 4.9 – 4.14 shown the result of natural weathering visualization. Based on the visualization of thermal conditioning, the changing characteristics of PLA/SCG bio-composites film at 70 ° C in incubators each week at different SCG concentrations were observed. Note that the bio-composites film has increased erosion, and at the SCG concentration of 20 wt%, there is an increase in cracks which observed that the cracks caused by compression molding (noticed by circle spot inside the film) begin to thicken. Figures 4.15 – 4.20 shown the result of thermal visualization. From visualization of the acid solution conditioning method, the behavioral changes of PLA/SCG bio-composites film were observed in the condition in 35 % by volume (v/v) of sulfuric acid. From the experiment, the PLA/SCG bio-composites film at every concentration has not changed much from the original, but the PLA/SCG bio-composites film is softer because pure PLA does not have a noticeable reaction with sulfuric acid. Therefore, this making the PLA/SCG bio-composites film look still similar to the original one. Figures 4.21 – 4.26 shown the result of acid solution visualization. From visual analysis using base solution conditioning method by the condition of PLA/SCG plastic films at different SCG concentrations. The films were conditioned in sodium hydroxide 35

percent by volume (v/v) and weighed every 2-6 days, which was observed that the PLA plastic film was not changed much in characteristic, but it becomes softer. However, the bio-composites film compounded with SCG breaks down into small pieces, especially at the 5 wt.% and 10wt.% concentrations, the change is noticeable, as is the SCG concentration of 15 wt.%, 20 wt.%, 25 wt.% bio-composites film is very soft but still able to maintain the shape of the film. Figures 4.27 – 4.32 shown the result of base solution visualization.

NETURAL WEATHERING CONDITIONING

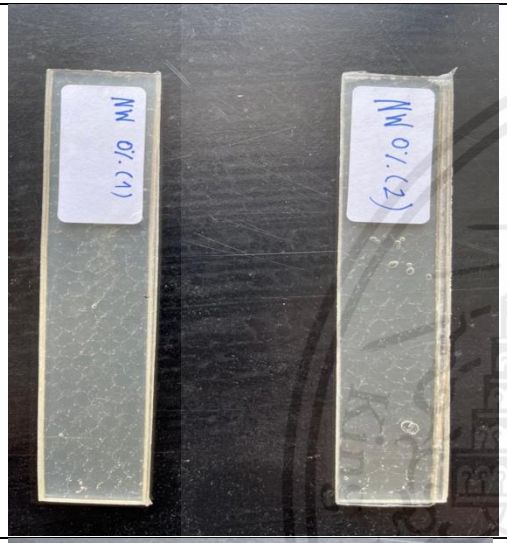
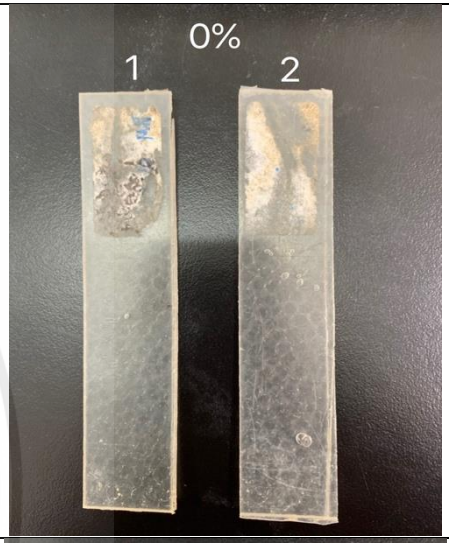
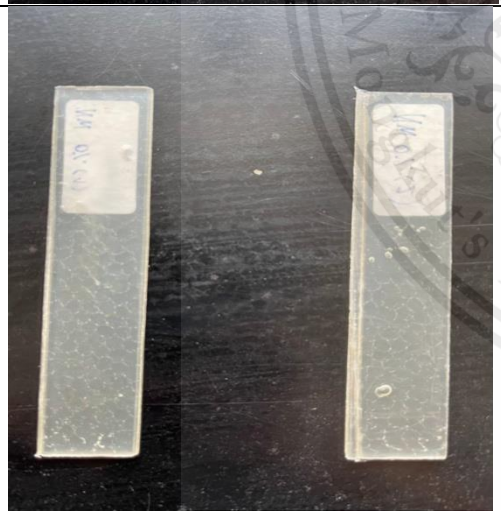
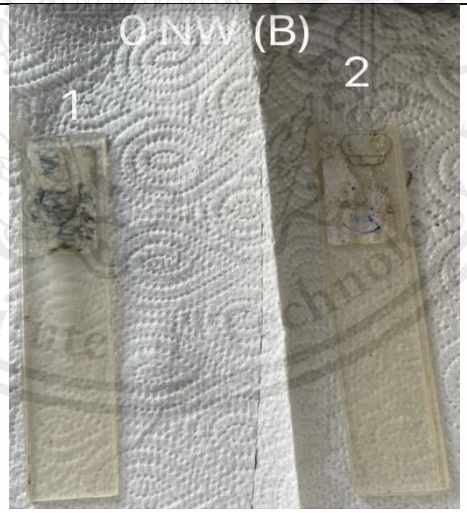
PLA	Week 0	Week 3	Week 6
Front view			
Back view			

Figure 4.9: Visualization of PLA samples exposed to NW for 6 weeks

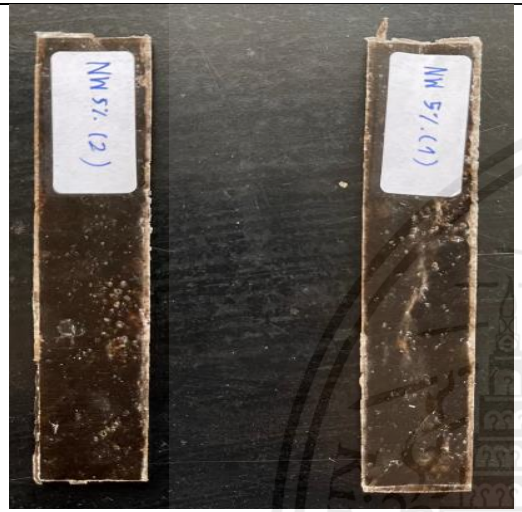
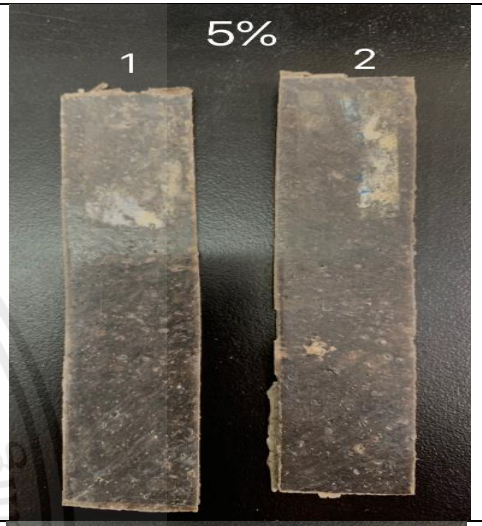
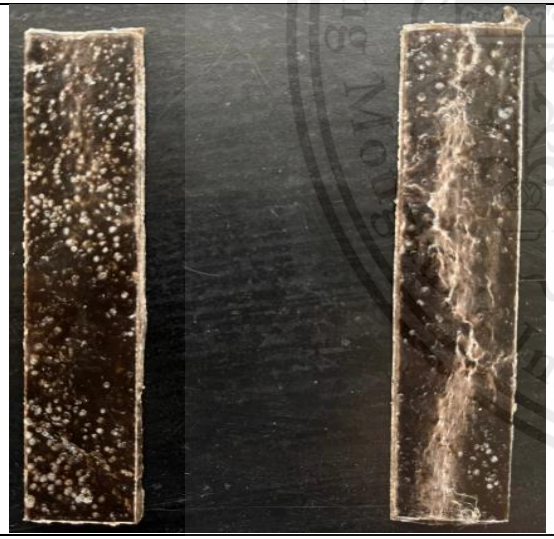
PLA/SCG 5 wt%	Week 0	Week 3	Week 6
Front view			
Back view			

Figure 4.10: Visualization of PLA/SCG 5 wt% samples exposed to NW for 6 weeks

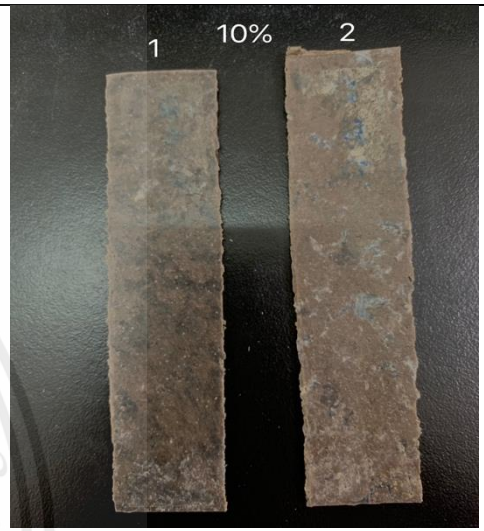
PLA/SCG 10 wt%	Week 0	Week 3	Week 6
Front view			
Back view			

Figure 4.11: Visualization of PLA/SCG 10 wt% samples exposed to NW for 6 weeks:

PLA/SCG 15 wt%	Week 0	Week 3	Week 6
Front view			
Back view			

Figure 4.12: Visualization of PLA/SCG 15 wt% samples exposed to NW for 6 weeks

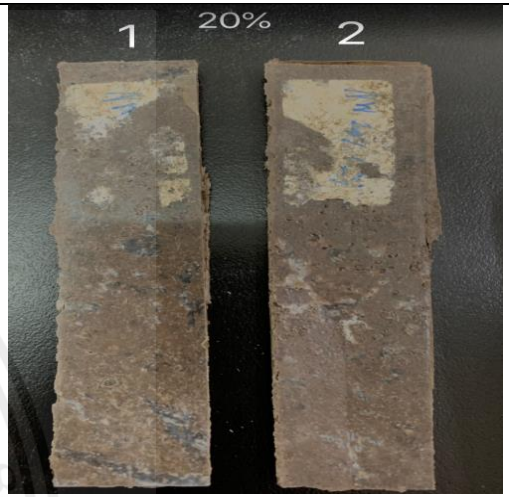
PLA/SCG 20 wt%	Week 0	Week 3	Week 6
Front view			
Back view			

Figure 4.13: Visualization of PLA/SCG 20 wt% samples exposed to NW for 6 weeks

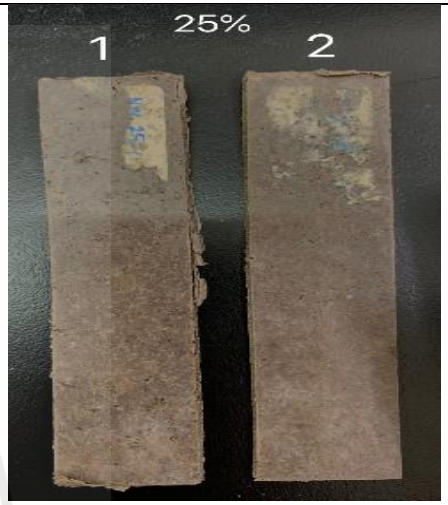
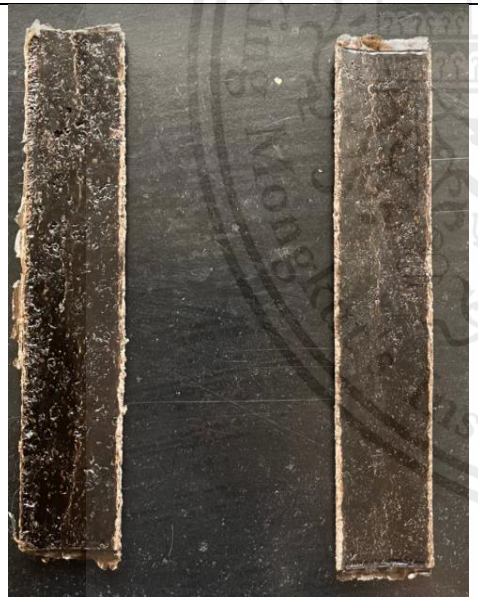
PLA/SCG 25 wt%	Week 0	Week 3	Week 6
Front view			
Back view			

Figure 4.14: Visualization of PLA/SCG 25 wt% samples exposed to NW for 6 weeks

THERMAL CONDITIONING

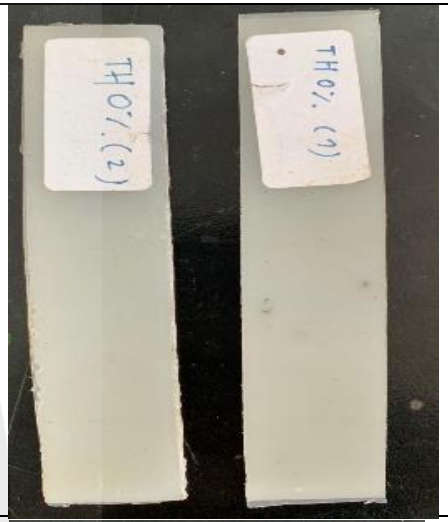
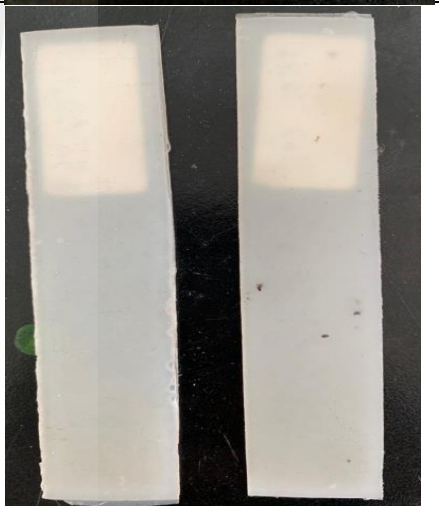
PLA	Week 0	Week 3	Week 6
Front view			
Back view			

Figure 4.15: Visualization of PLA samples exposed to TH for 6 weeks

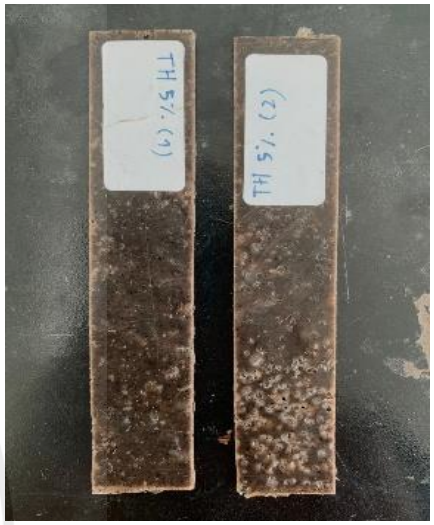
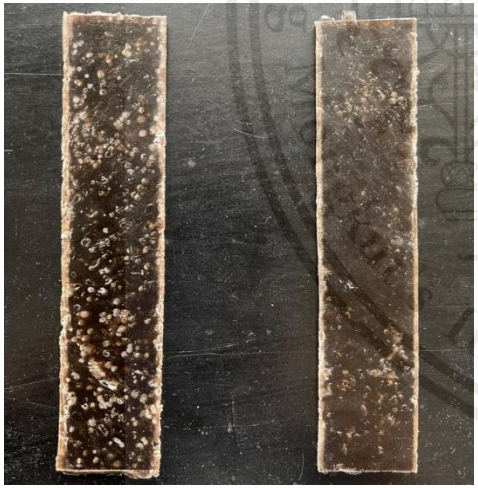
PLA/SCG 5 wt%	Week 0	Week 3	Week 6
Front view			
Back view			

Figure 4.16: Visualization of PLA/SCG 5 wt% samples exposed to TH for 6 weeks

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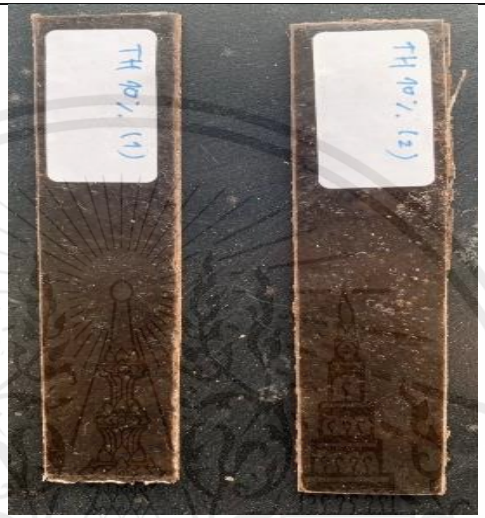
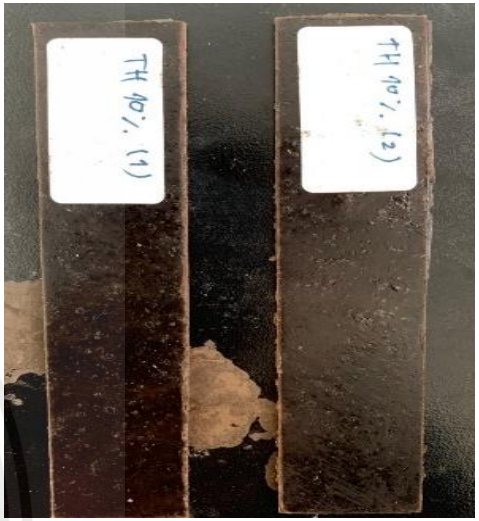
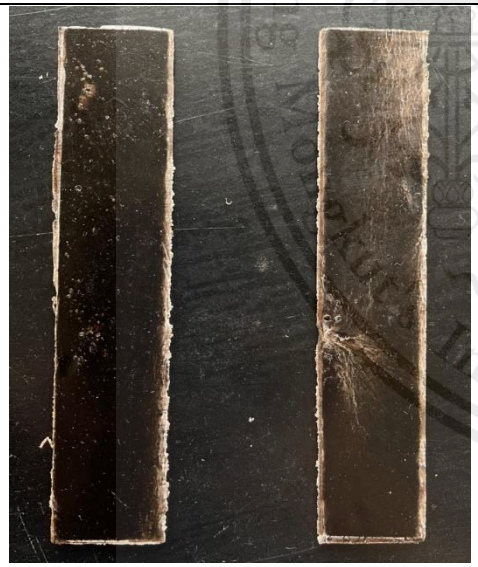
PLA/SCG 10 wt%	Week 0	Week 3	Week 6
Front view			
Back view			

Figure 4.17: Visualization of PLA/SCG 10 wt% samples exposed to TH for 6 weeks

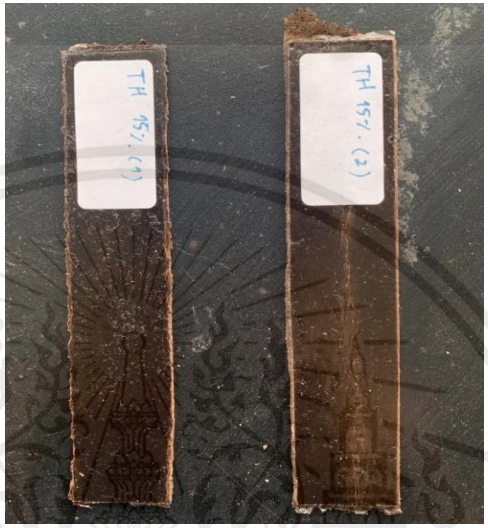
PLA/SCG 15 wt%	Week 0	Week 3	Week 6
Front view			
Back view			

Figure 4.18: Visualization of PLA/SCG 15 wt% samples exposed to TH for 6 weeks:

PLA/SCG 20wt%	Week 0	Week 3	Week 6
Front view			
Back view			

Figure 4.19: Visualization of PLA/SCG 20 wt% samples exposed to TH for 6 weeks

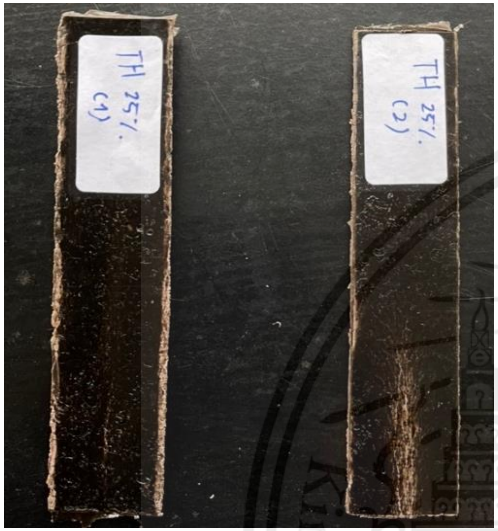
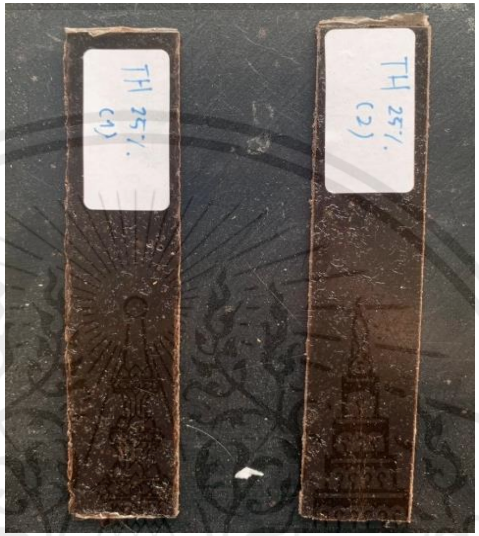
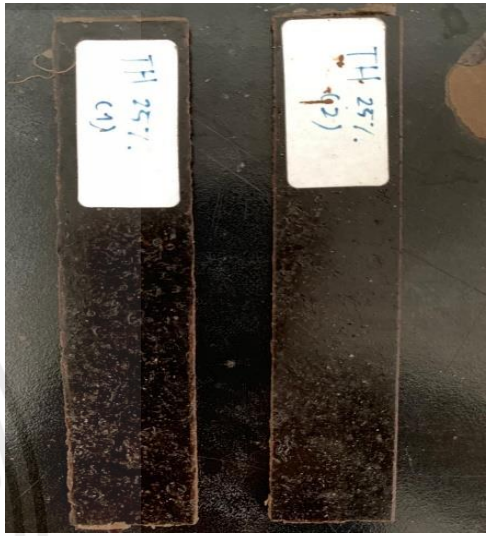
PLA/SCG 25 wt%	Week 0	Week 3	Week 6
Front view			
Back view			

Figure 4.20: Visualization of PLA/SCG 20 wt% samples exposed to TH for 6 weeks

ACID SOLUTION CONDITIONING

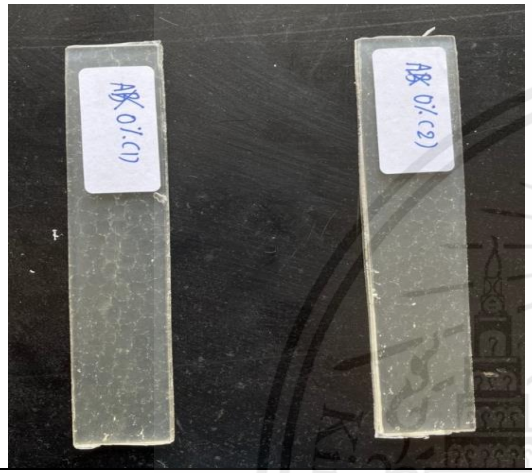
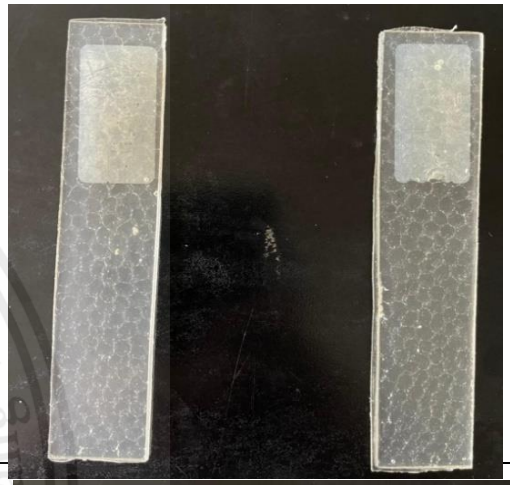
PLA	Week 0	Week 3	Week 6
Front view			
Back view			

Figure 4.21: Visualization of PLA samples exposed to AS for 6 weeks

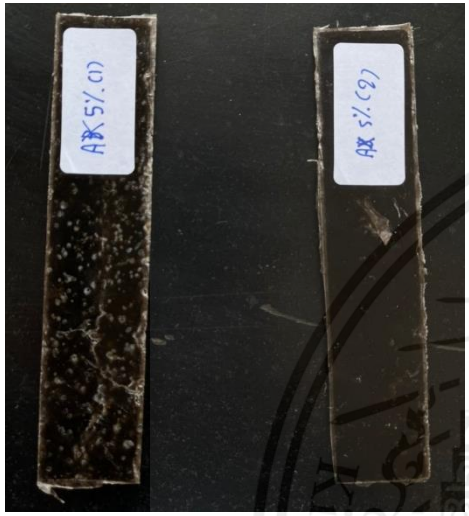
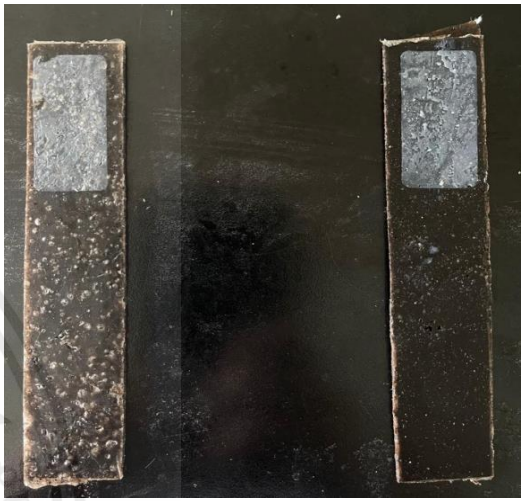
PLA/SCG 5 wt%	Week 0	Week 3	Week 6
Front view			
Back view			

Figure 4.22: Visualization of PLA/SCG 5 wt% samples exposed to AS for 6 weeks

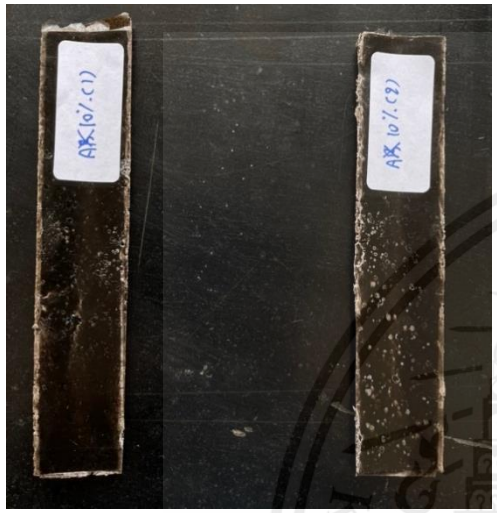
PLA/SCG 10wt%	Week 0	Week 3	Week 6
Front view			
Back view			

Figure 4.23: Visualization of PLA/SCG 10 wt% samples exposed to AS for 6 weeks

PLA/SCG 15wt%	Week 0	Week 3	Week 6
Front view			
Back view			

Figure 4.24: Visualization of PLA/SCG 15 wt% samples exposed to AS for 6 weeks

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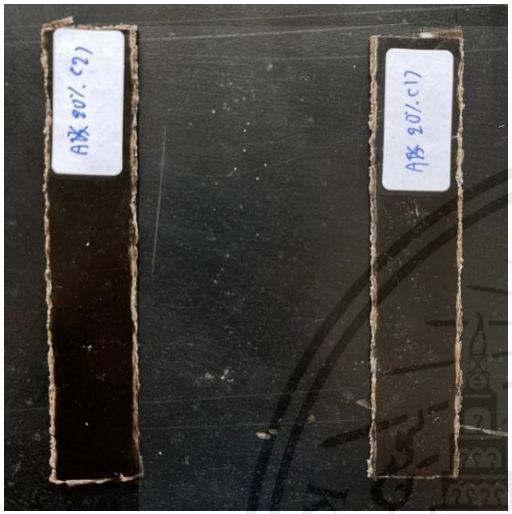
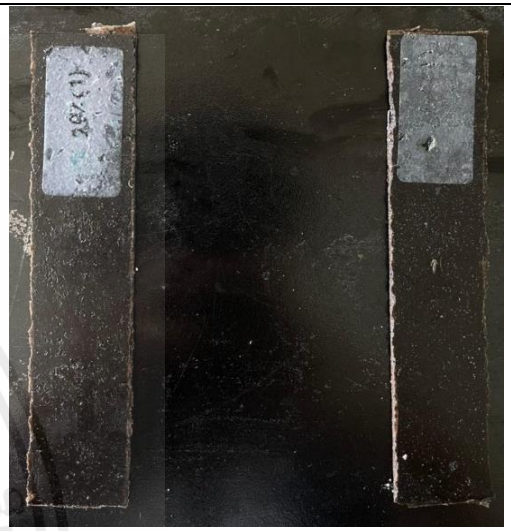
PLA/SCG 20 wt%	Week 0	Week 3	Week 6
Front view			
Back view			

Figure 4.25: Visualization of PLA/SCG 20 wt% samples exposed to AS for 6 weeks

PLA/SCG 25 wt%	Week 0	Week 3	Week 6
Front view			
Back view			

Figure 4.26: Visualization of PLA/SCG 25 wt% samples exposed to AS for 6 weeks

BASE SOLUTION CONDITIONING

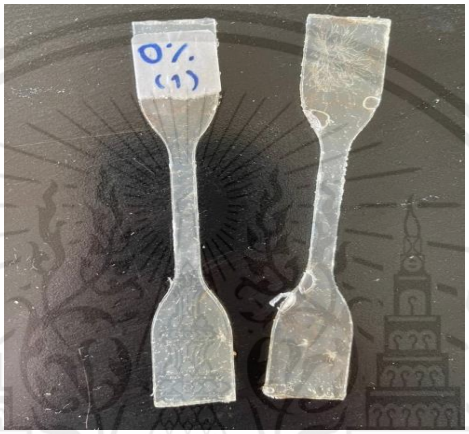
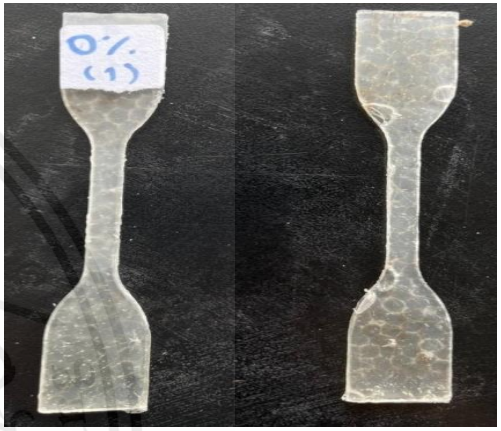
PLA	Day 0	Day 6	Day 9
Front view			
Back view			

Figure 4.27: Visualization of PLA samples exposed to BS during different days (Pick up from Day0, Day6, Day9)

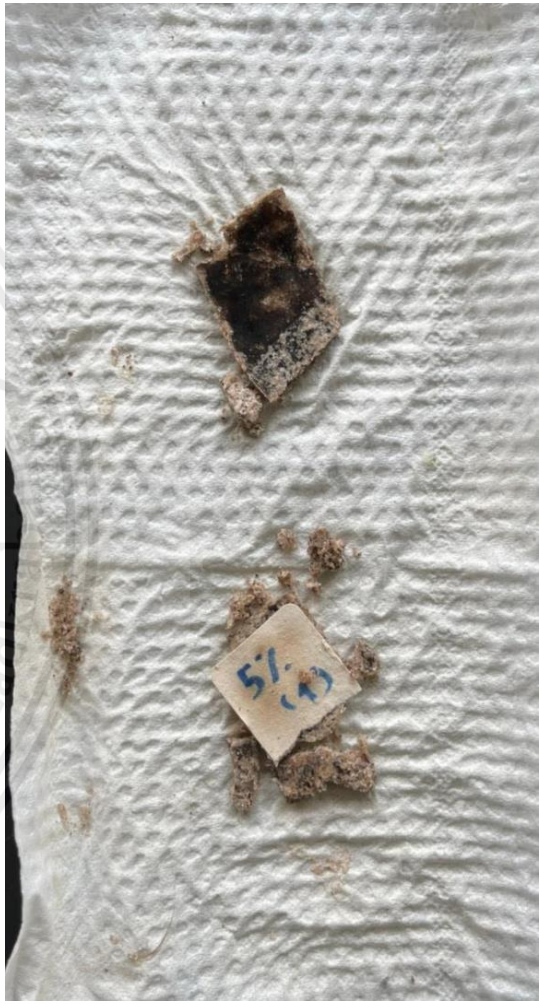
PLA/SCG 5wt%	Day 0	Day 6	Day 9
Front view			
Back view			

Figure 4.28: Visualization of PLA/SCG 5 wt% samples exposed to BS during different days (Pick up from Day0, Day6, Day9)

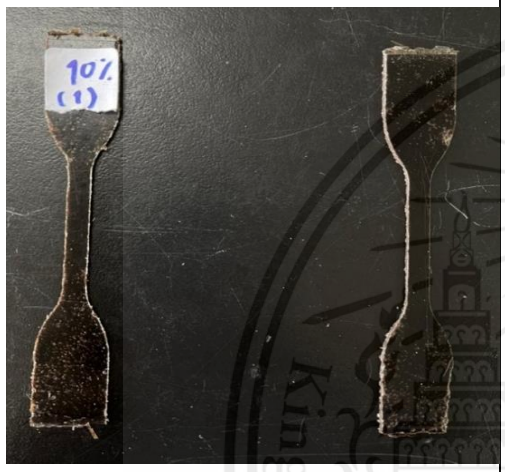
PLA/SCG 10 wt%	Day 0	Day 6	Day 9
Front view			
Back view			

Figure 4.29: Visualization of PLA/SCG 10 wt% samples exposed to BS during different days (Pick up from Day0, Day6, Day9)

PLA/SCG 15 wt%	Day 0	Day 6	Day 9
Front view			
Back view			

Figure 4.30: Visualization of PLA/SCG 15 wt% samples exposed to BS during different days (Pick up from Day0, Day6, Day9)

PLA/SCG 20 wt%	Day 0	Day 6	Day 9
Front view			
Back view			

Figure 4.31: Visualization of PLA/SCG 20 wt% samples exposed to BS during different days (Pick up from Day0, Day6, Day9)

PLA/SCG 25wt%	Week 0	Week 3	Week 6
Front view			
Back view			

Figure 4.32: Visualization of PLA/SCG 25 wt% samples exposed to BS during different days (Pick up from Day0, Day6, Day9)

4.2.5 Thermal Testing

The tests for the thermal properties of PLA/SCG bio-composites film by differential scanning calorimetry (DSC) techniques were achieved to measure and study glass transition temperature (Tg) and melting temperatures (Tm). The tests were performed by both first and second run and tested for PLA/SCG bio-composite film at different concentrations, the DSC chromatogram of PLA/SCG are shown in Appendix 3 and the data obtained from the test are summarized in Table 4.6.

For the result of the first run can be seen that the glass transition temperatures of PLA/SCG 5 wt.%, PLA/SCG 10 wt%, PLA/SCG 15 wt.%, PLA/SCG 20wt%, and PLA/SCG 25% were at 54.69℃, 54.46℃, 54.93℃, 54.36℃ and 53.36℃, respectively while the melting temperatures were at 140.84℃, 140.61℃, 144.27℃, 138.27℃, 137.19℃, respectively.

For the second run, the test found that glass transition temperatures were at 57.52℃, 57.10℃, 56.93℃, 56.90℃ and 56.60℃, respectively. Melting temperatures were at 149.74℃, 149.74℃, 145.54℃, 146.31℃ and 151.65, respectively.

Then, we used the glass transition temperature and melting temperature data obtained from PLA4043D from the Nature-Works LLC that is the material supplier in this project, instead of the testing run of PLA. So, the glass transition temperature and melting temperature of PLA4043D from the material supplier were 55℃- 60℃, 135-160℃, respectively, shown in Table 3.1. In addition, when compared the glass transition temperature and melting temperature of PLA with PLA/SCG in different SCG contents. We can observe that the value of all samples testing and the data of PLA4043D from the material supplier were nearly close to PLA 4043D. Therefore, increasing the concentration of SCG does not significantly affect the glass transition temperature and melting temperature of the PLA/SCG bio-composites film.

Table 4.2: DSC results of PLA and PLA/SCG bio-composites film

	PLA and PLA/SCG Bio-Composites Film							
	Temperature	**Unit**	**0%**	**5%**	**10%**	**15%**	**20%**	**25%**
First run	T_g	℃	≈55-60	54.69	54.46	54.93	54.36	53.55
	T_m	℃	≈135-160	140.84	140.61	144.27	138.27	137.19
Second run	T_g	℃	≈55	57.52	57.10	56.93	56.90	56.60
	T_m	℃	≈135-160	149.74	149.74	145.54	146.31	151.65

#Remark: for Tg and Tm of PLA (4043D), data were obtained from the Nature-Works LLC that is the material supplier in this research.

CHAPTER 5
CONCLUSIONS

5.1 Conclusions

In the research, the purpose was to produce a PLA/SCG bio-composites film using PLA matrix and study the effects of concentration of spent coffee ground (SCG) filter on the mechanical properties, physical properties, thermal properties and the degradation of PLA/SCG materials at different SCG contents. PLA/SCG bio-composites film was prepared using a twin-screw extruder to mix the SCG and PLA together and followed by the compression molding process to produce the bio-composites film.

The morphologies characterized by using Optical Microscope (OM) show that SCG particle was well distributed in the PLA matrix when increasing the concentration of SCG into the matrix, but it can reduce the surface adhesion force between PLA and SCG phases. This was because when you added SCG to the PLA matrix, the more particle size of the SCG increases in the matrix phase. Then, SCG particle were grouping in the form of spherical that cause of incompatibility morphology.

In terms of mechanical properties, tensile strength at break and modulus at break of PLA/SCG bio-composite films, they were decreased compared with the pure PLA because the surface adhesion force is deficient, resulting in a reduction of brittleness. On the other hand, when adding SCG into the matrix, elongation at break of the bio-composites film mainly increases compared with the pure PLA because PLA will change from brittle fracture to sticky fracture. The % elongation at break of the PLA was increased from 11.6% to 32.3% and obtained the max change from the addition of 15 wt.% SCG. But when increasing the SCG too much, elongation at break will be decreased. Nonetheless, the elongation at break of the PLA/SCG bio-composites film at all the mixing ratios between PLA and SCG was higher than pure PLA. Therefore, we can summarize PLA has more flexible and brittle decreased when adding SCG in the matrix.

In addition, the test of thermal properties by using differential scanning calorimetry (DSC) technique was used to study the glass transition temperature (Tg) and melting temperature (Tm) of blended PLA/SCG polymer. The results of the experiments show that the Tg of samples were in the range 53.55 - 57.52 °C and Tm between 137.19 -151.65 °C compared to pure PLA polymers with Tg in the range 55 – 60 °C and Tm in the 135-160 ° C range. This implied that the increasing in SCG concentrations did not affect the Tg and Tm values.

For the visualization, AS and TH have no significant change. The NW sample has more erosion on the surface and thicker than original one, and considerable change in the BS conditioning. The sample of base conditioning was delamination because the base solution damages the adhesive bond between PLA and SCG by the hydroxyl O-H bond. And the PLA/SCG bio-composites film shows a good result of degradation testing. For NW, PLA/SCG bio-composites film is slightly decreased every week. The weight loss ranges from 1.150%-3.1732% and PLA/SCG 15 wt% was the most weight loss when compared with pure PLA. For TH, the PLA/SCG bio-composites film was slightly reduced compared to the un-conditioned samples. For AS, it was observed that the weight of the samples was fluctuating due to the PLA polymer did not react markedly with sulfuric acid, and due to the moisture from sulfuric acid causing gained of weight, and the weight of the samples was not reduced compared to the unconditioning of PLA/SCG bio-composite film, sulfuric acid did not affect the degradation of the samples. Conversely, in BS, it can be observed that the weight of PLA/SCG bio-composite

film was significantly reduced compared to the original one because SCG contributed to the depolymerization of PLA resulting in the degradation of the PLA polymer. Therefore, we can summarize the degradation of PLA/SCG bio-composites film has a significant factor that affects the weight loss is caused by a lack of the main chain or side chain of the molecule that was affected by biological activities, physical and chemical properties of the films such as pH, porosity, photolysis, mechanical strength, amount of oxygen in the soil, and morphology. Therefore, SCG can promote the properties of PLA in order to produce the PLA/SCG bio-composites films which were degradable easily.

5.2 Recommendation

From the results of the experiment, bio-composite film formulated from spent coffee grounds has improved mechanical properties with increased stickiness when added to the appropriate amount of coffee grounds, Therefore, it suitable for use in desired products that want to increase toughness and flexibility in the material. In the preservation, bases should be avoided as bio-composite films can interact with the base and will damage the bio-composite film.

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APPENDIX 1
TENSILE TESTING

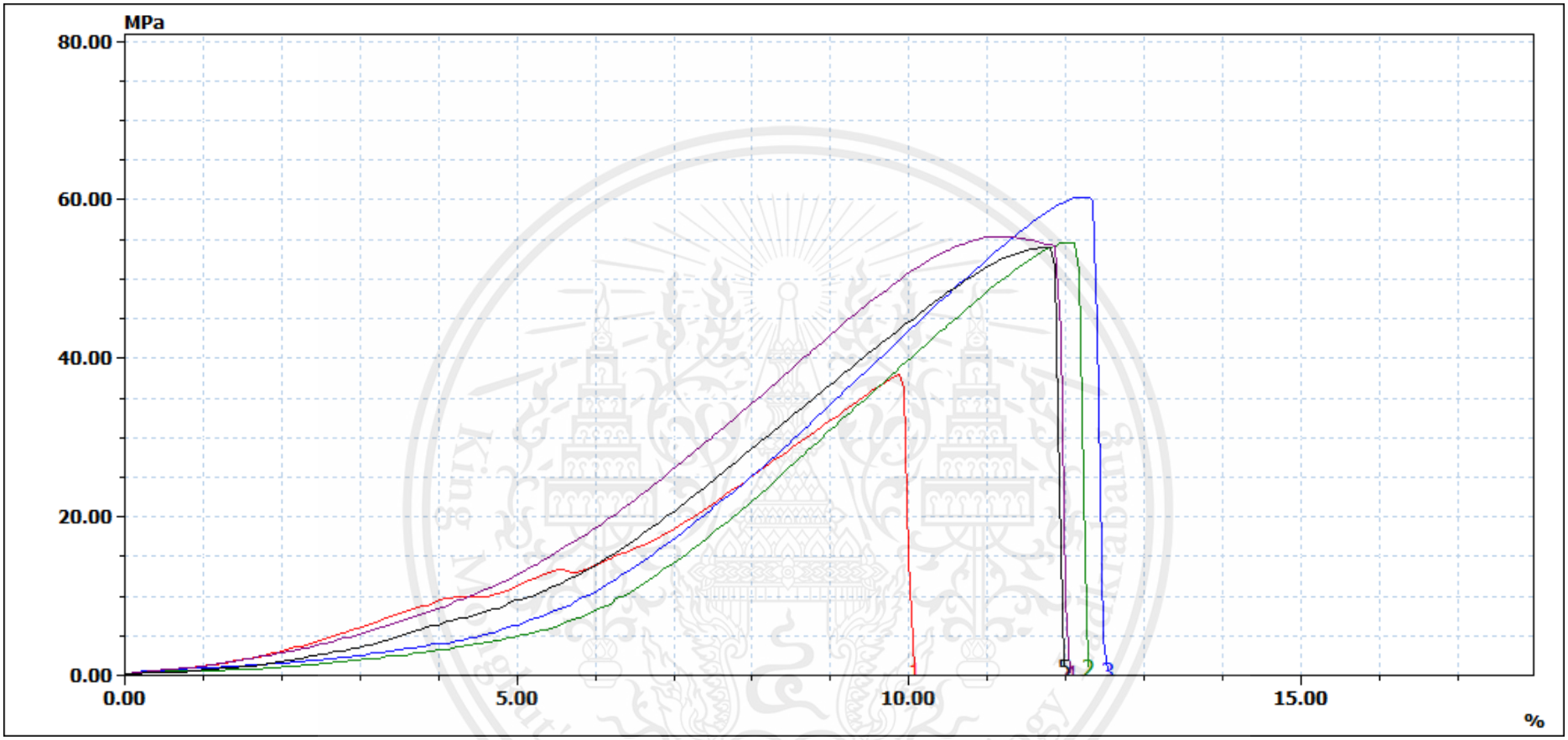

Figure 6.1: Stress vs. strain curve shows the tensile result of PLA

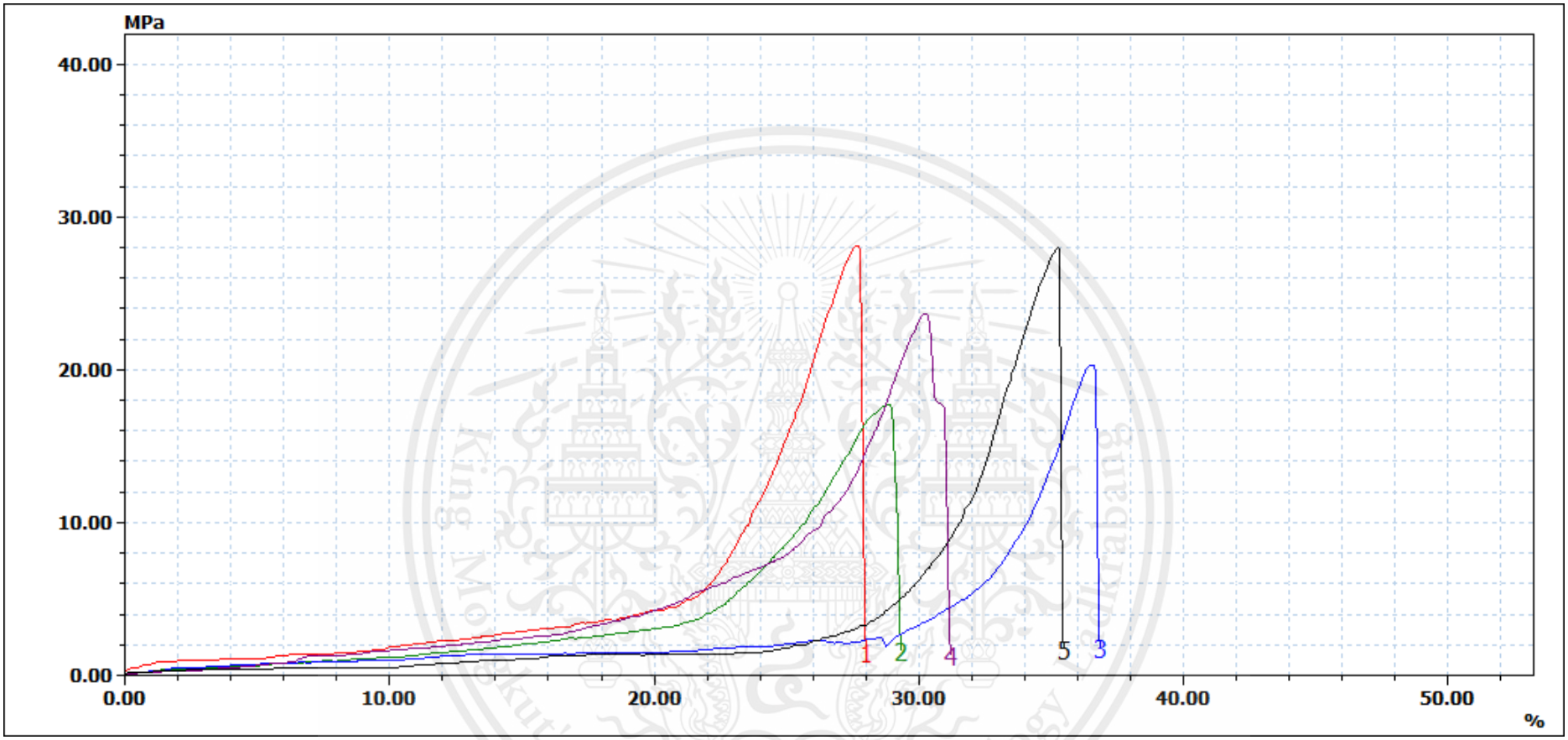

Figure 6.2: Stress vs strain curve shows the tensile result of PLA/SCG 5 wt%

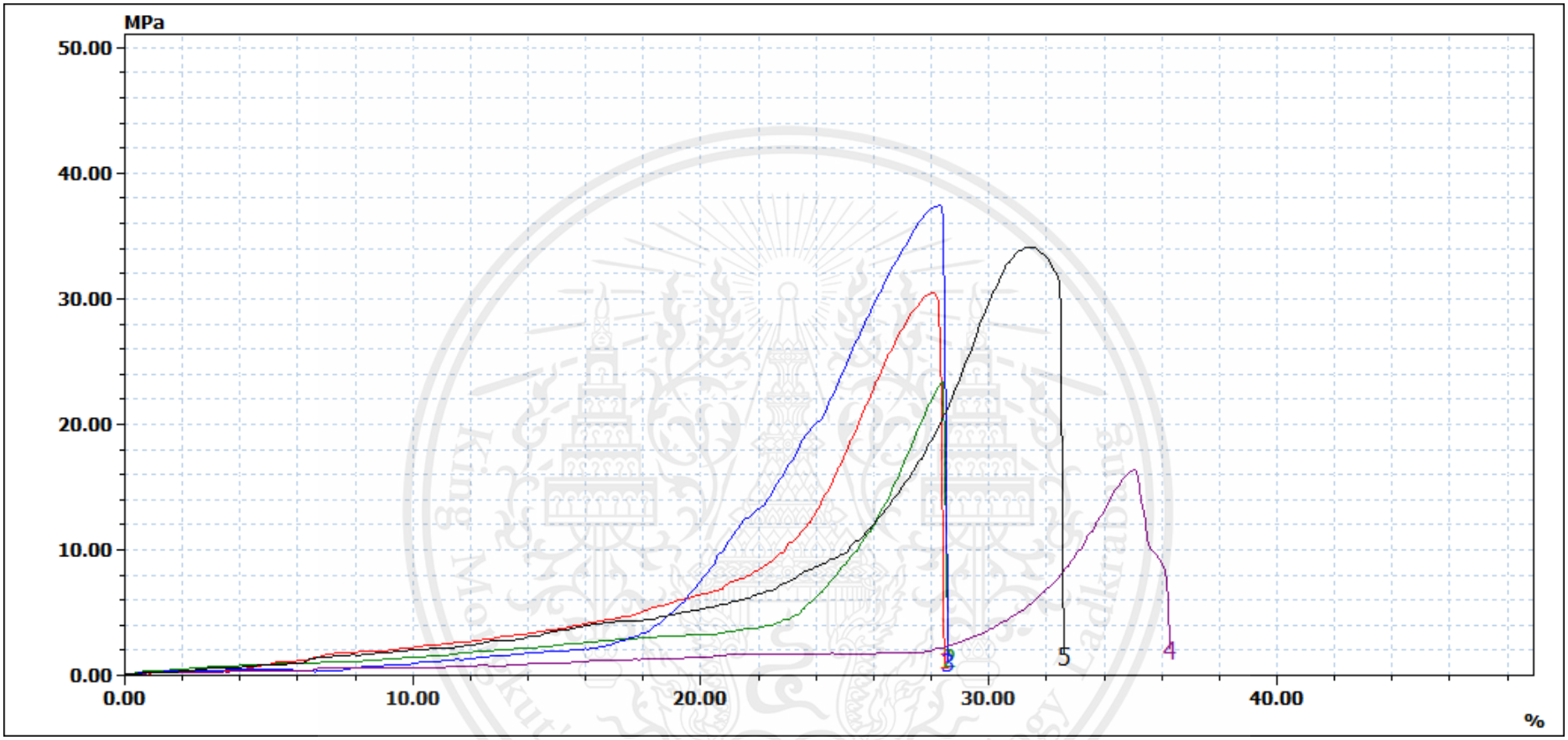

Figure 6.3: Stress vs strain curve shows the tensile result of PLA/SCG 10 wt%

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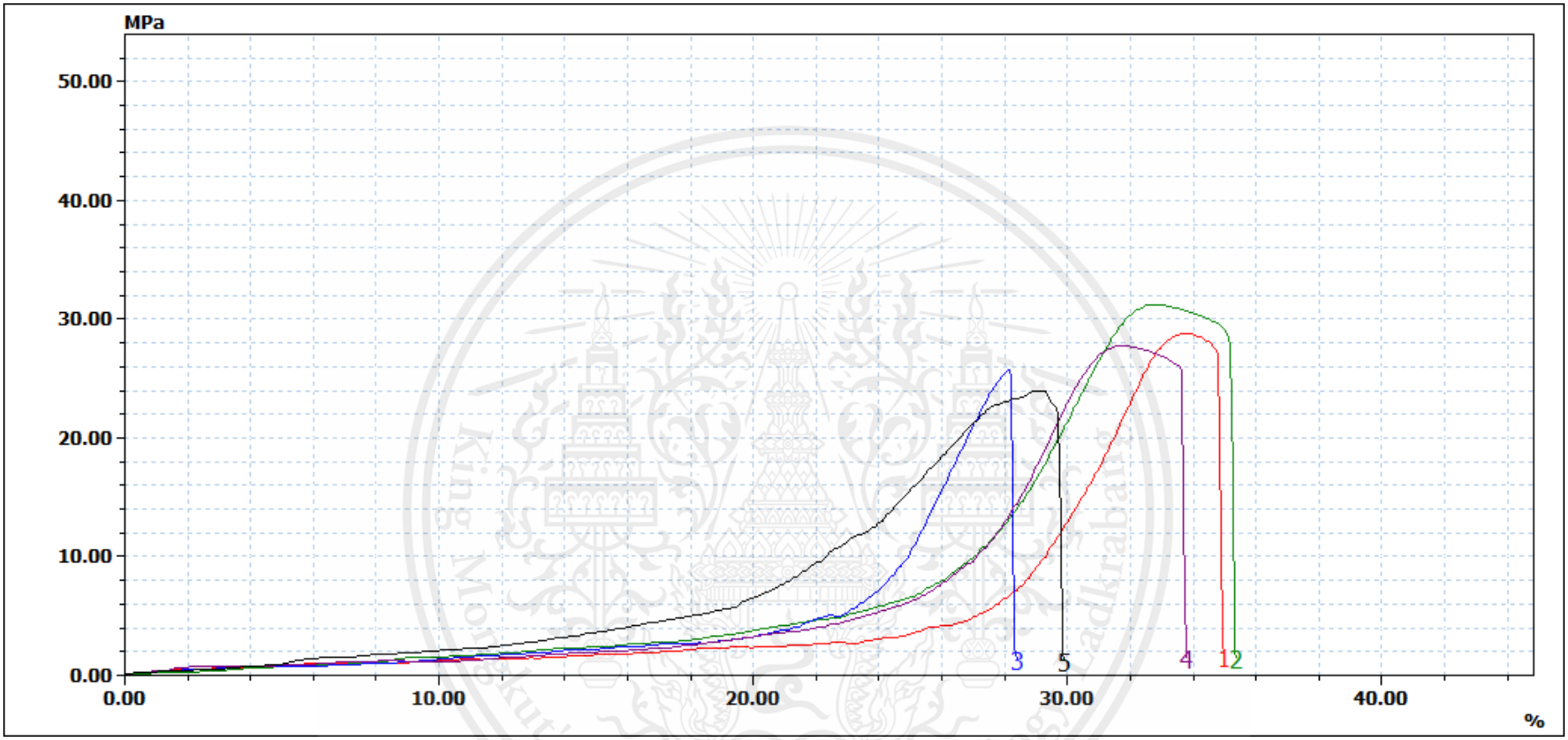

Figure 6.4: Stress vs strain curve shows the tensile result of PLA/SCG 15 wt%

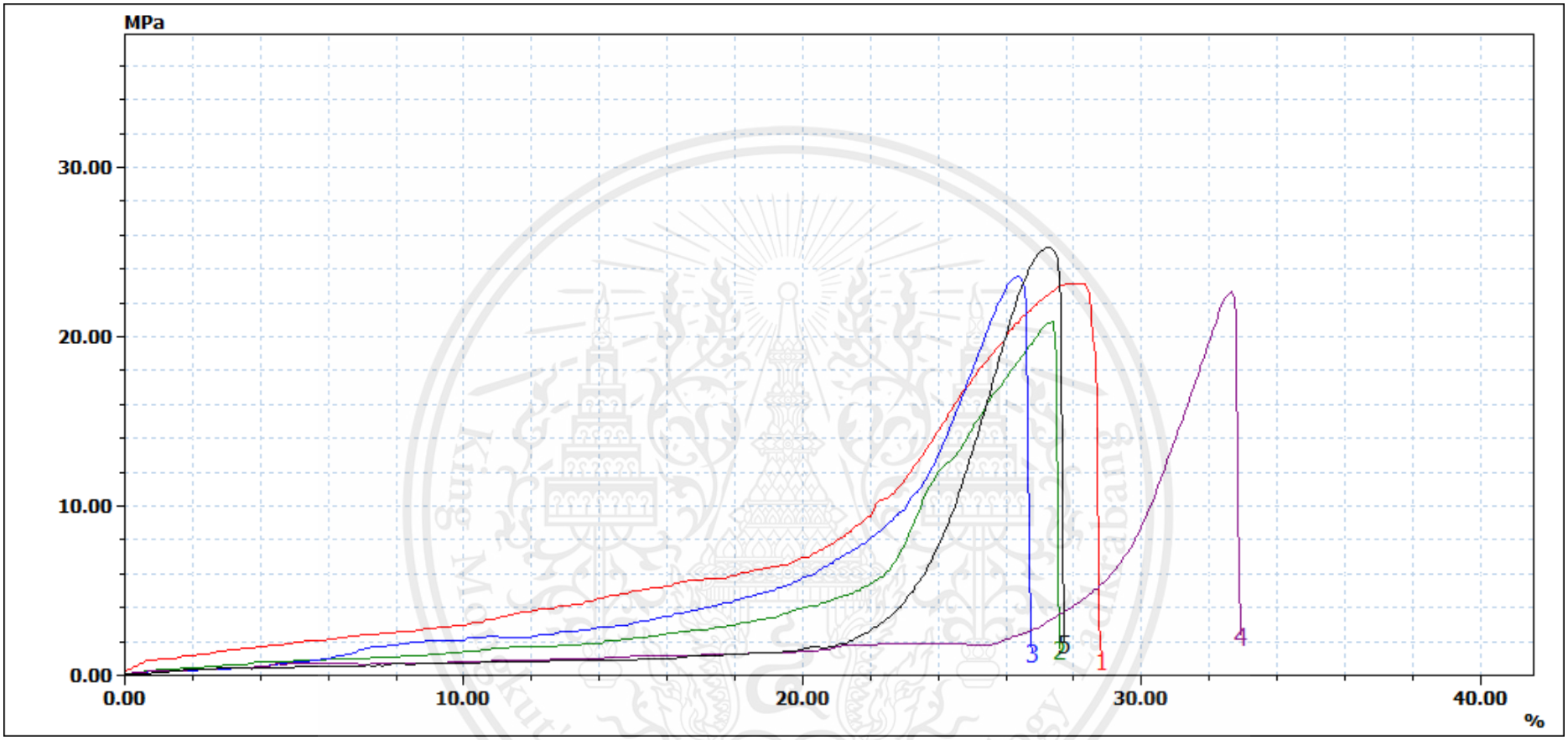

Figure 6.5: Stress vs strain curve shows the tensile result of PLA/SCG 20 wt%

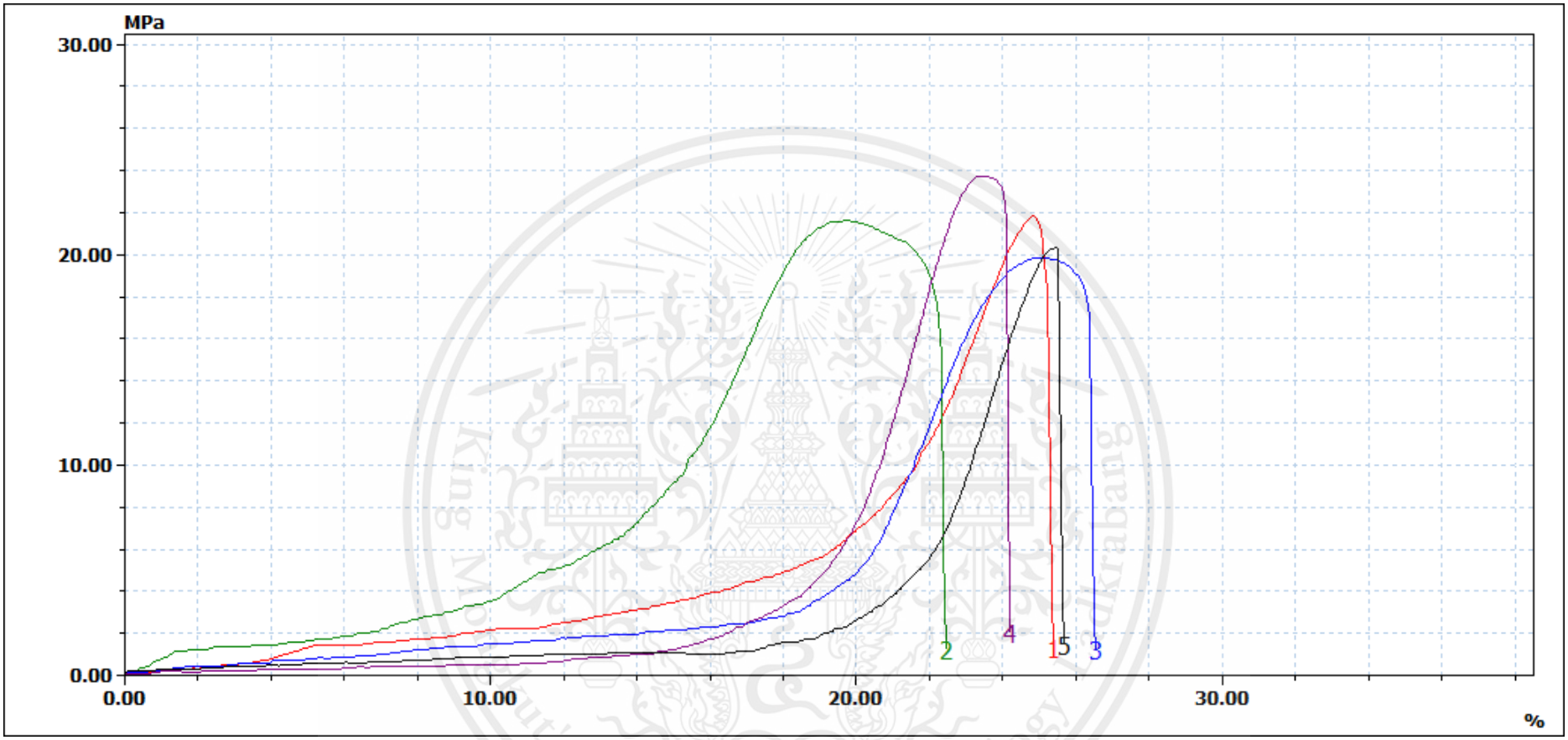

Figure 6.6: Stress vs strain curve shows the tensile result of PLA/SCG 25 wt%

APPENDIX 2
Degradation results

Table 7.1: The weight of PLA and PLA/SCG bio-composites film by natural weathering conditioning at different quantities of SCG.

Natural Weathering Conditioning						
	Weight (g)					
Week	**PLA/ 0 wt%**	**PLA/SCG 5 wt%**	**PLA/SCG 10 wt%**	**PLA/SCG 15 wt%**	**PLA/SCG 20 wt%**	**PLA/SCG 25 wt%**
0	7.802	8.102	8.892	7.828	7.206	7.552
1	7.8	8.085	8.889	7.823	7.199	7.52
2	7.775	8.079	8.8886	7.82	7.198	7.519
3	7.759	8.071	8.883	7.781	7.196	7.503
4	7.753	8.066	8.887	7.763	7.192	7.502
5	7.732	8.066	8.85	7.675	7.12	7.4487
6	7.72049	7.9661	8.7897	7.5796	7.04	7.3956
%Weight Loss	1.04473212	1.677363614	1.150472335	3.173224323	2.303635859	2.070974576

Table 7.2: The of PLA and PLA/SCG bio-composites film for thermal conditioning at different quantities of SCG.

Thermal Conditioning						
	Weight (g)					
Week	**PLA/SCG 0 wt%**	**PLA/SCG 5 wt%**	**PLA/SCG 10 wt%**	**PLA/SCG 15 wt%**	**PLA/SCG 20 wt%**	**PLA/SCG 25 wt%**
0	8.25929	7.11799	8.4656	7.7534	6.96889	7.3665
1	8.24758	7.0911	8.4326	7.7185	6.9394	7.3271
2	8.2435	7.094	8.4262	7.716	6.9341	7.31858
3	8.24133	7.0887	8.4242	7.71432	6.93243	7.3235
4	8.2448	7.09139	8.4332	7.7196	6.9372	7.322
5	8.26913	7.12261	8.47866	7.77646	6.93592	7.39626
6	8.24611	7.08839	8.4366	7.71709	6.93116	7.32396
7	8.25401	7.09852	8.4547	7.73755	6.9568	7.36239
%Weight Loss	0.167877	0.273532	0.128756	0.204426	0.173485	0.055793

Table 7.3: The weight of PLA and PLA/SCG bio-composite films conditioned in sulfuric acid at different quantities of SCG.

Acid Solution Conditioning						
	Weight (g)					
Week	**PLA/SCG 0 wt%**	**PLA/SCG 5 wt%**	**PLA/SCG 10 wt%**	**PLA/SCG 15 wt%**	**PLA/SCG 20 wt%**	**PLA/SCG 25 wt%**
0	7.7102	7.5338	7.8752	7.7944	7.1656	5.9384
1	7.6847	7.54944	7.9783	7.88427	7.28336	6.1074
2	7.70195	7.5925	8.00135	7.9615	7.25995	6.189
3	7.67146	7.5739	7.9861	7.92131	7.32537	6.16738
4	7.67075	7.54224	7.96954	7.90108	7.30194	6.15345
5	7.67144	7.56366	7.97754	7.9109	7.31986	6.15126
6	7.66906	7.57602	7.98224	7.92226	7.33651	6.18544
%Weight Loss	0.533578	-0.56041	-1.3592	-1.64041	-2.38515	-4.16004

Table 7.4: The weight of PLA and PLA/SCG bio-composite films conditioned in sodium hydroxide at different quantities of SCG.

Base Solution Conditioning						
	Weight (g)					
Day	**PLA/SCG 0 wt%**	**PLA/SCG 5 wt%**	**PLA/SCG 10 wt%**	**PLA/SCG 15 wt%**	**PLA/SCG 20 wt%**	**PLA/SCG 25 wt%**
0	3.81675	2.83594	2.97558	3.0987	3.33778	3.30389
6	3.873	0.671	0.874	1.669	3.253	3.35
9	3.779	1.1157	0.9313	0.867	3.1061	3.0656
13	3.77387	0.56377	0.44424	0.88116	2.83758	2.97695
%Weight loss	1.123469	80.12052	85.07047	71.56356	14.98601	9.895608

Appendix 3
DSC Chromatogram

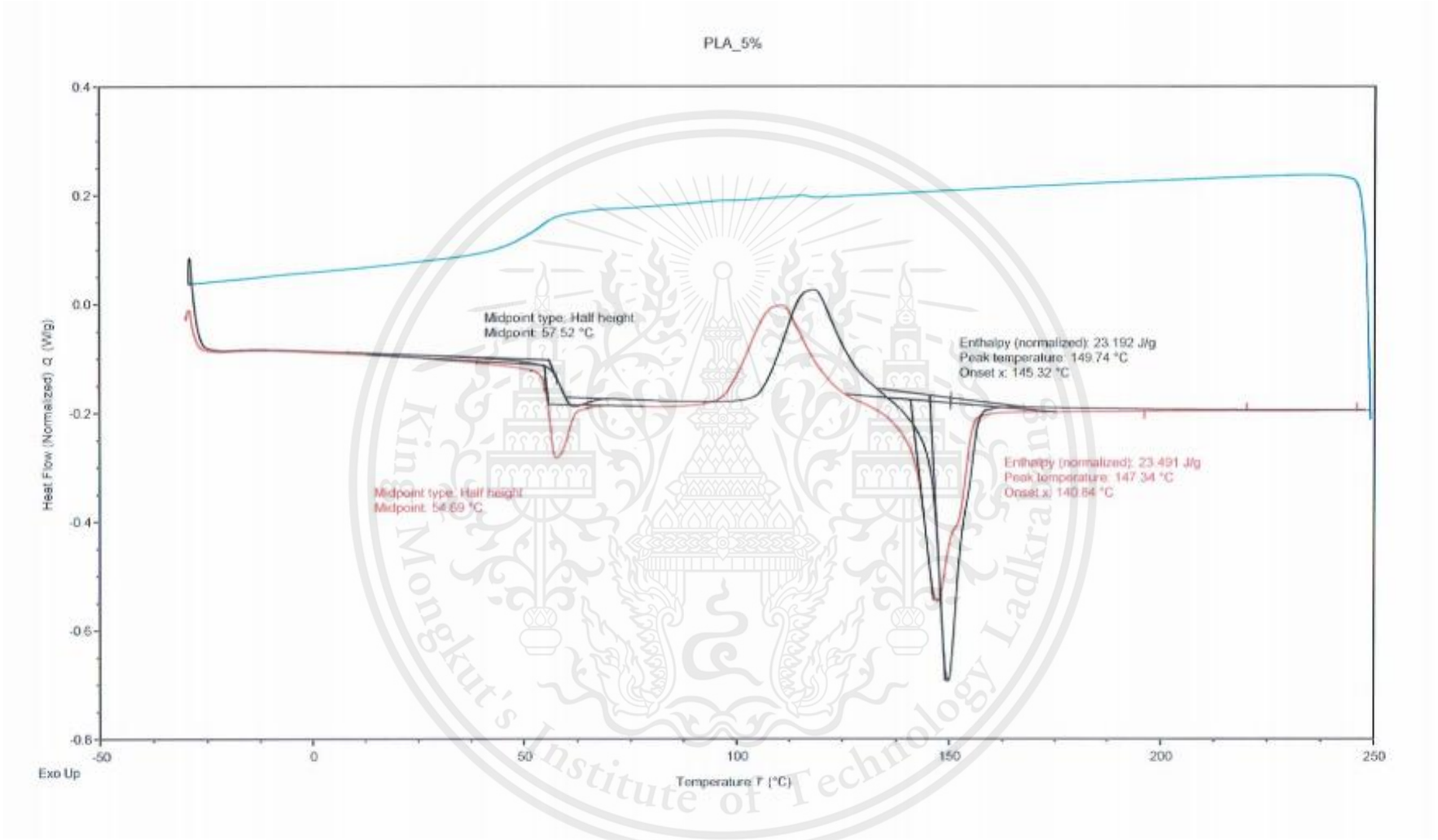

Figure 8.1: DSC chromatogram of PLA/SCG bio-composites film 5 wt.% for first run and second run

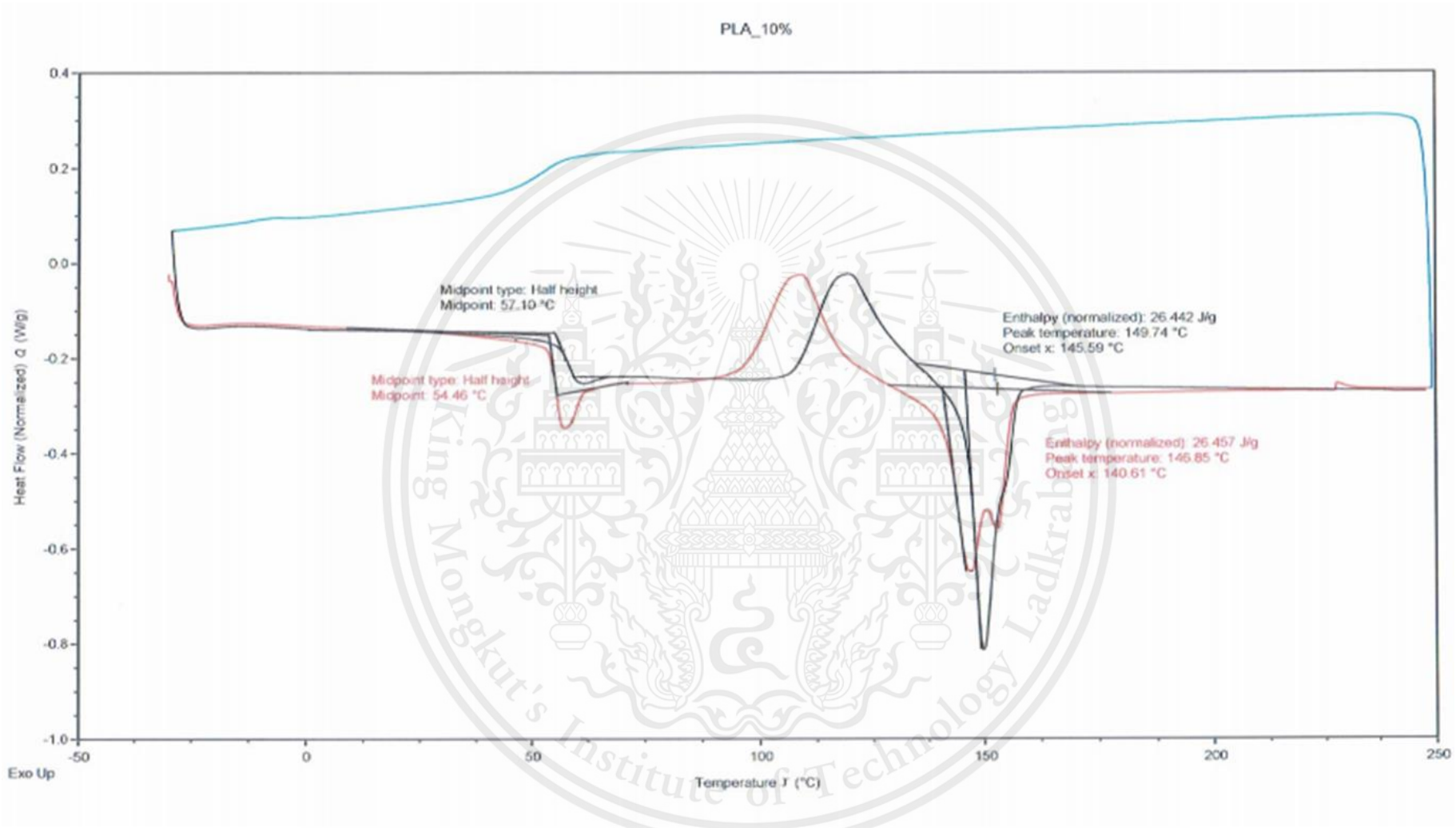

Figure 8.2: DSC chromatogram of PLA/SCG bio-composites film 10 wt.% for first run and second run

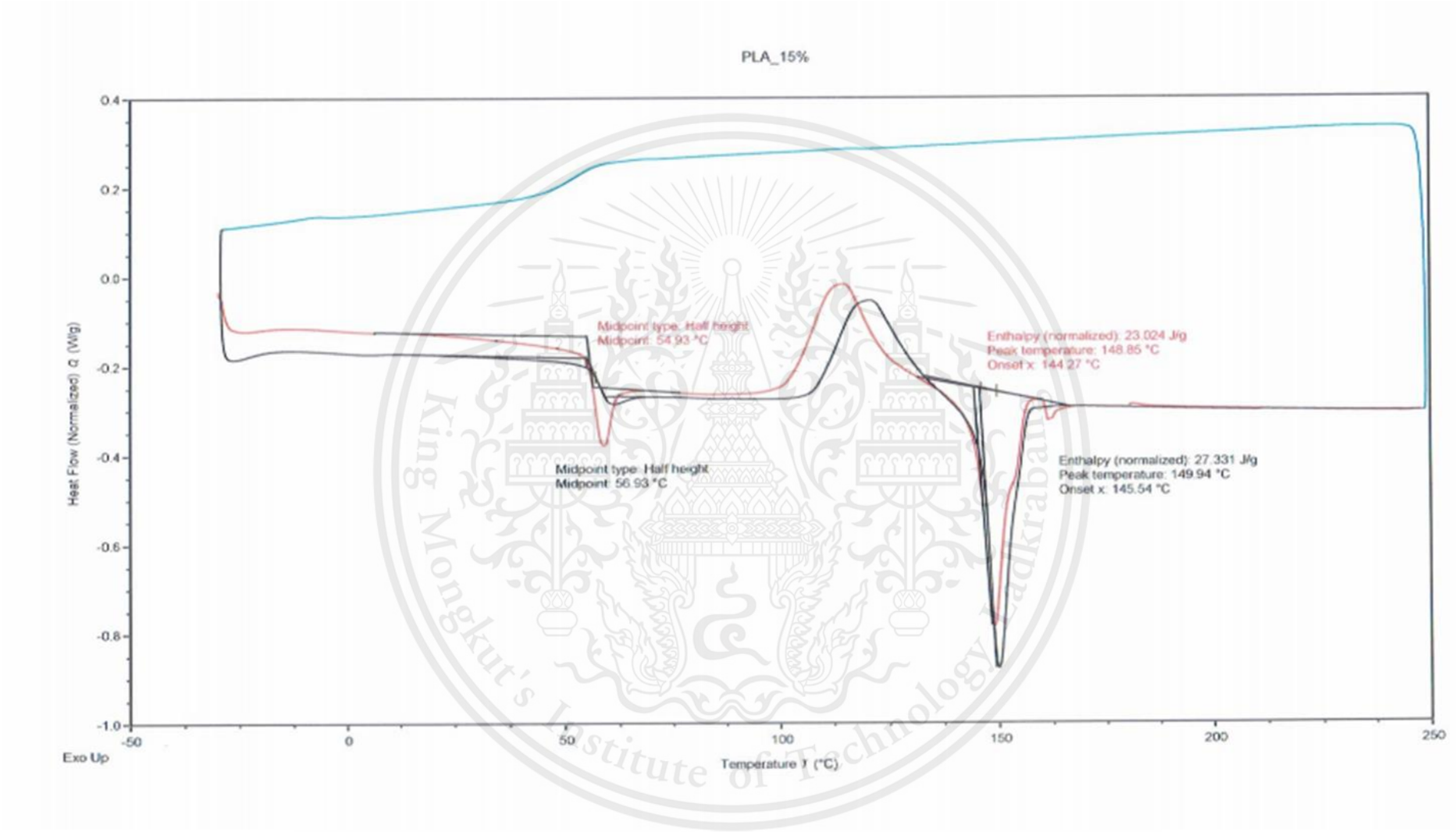

Figure 8.3: DSC chromatogram of PLA/SCG bio-composites film 15 wt.% for first run and second run

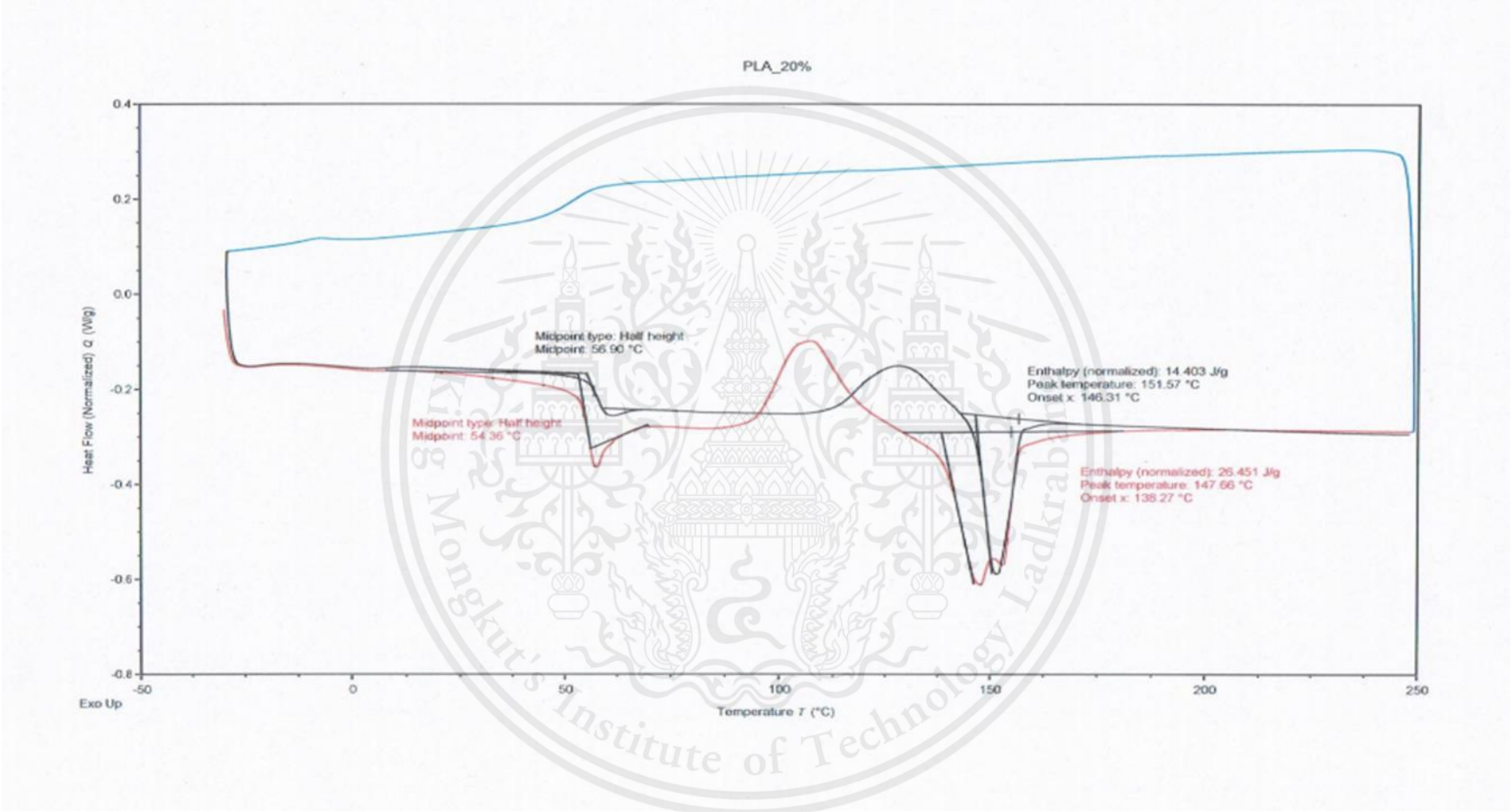

Figure 8.4: DSC chromatogram of PLA/SCG bio-composites film 20 wt.% for first run and second run

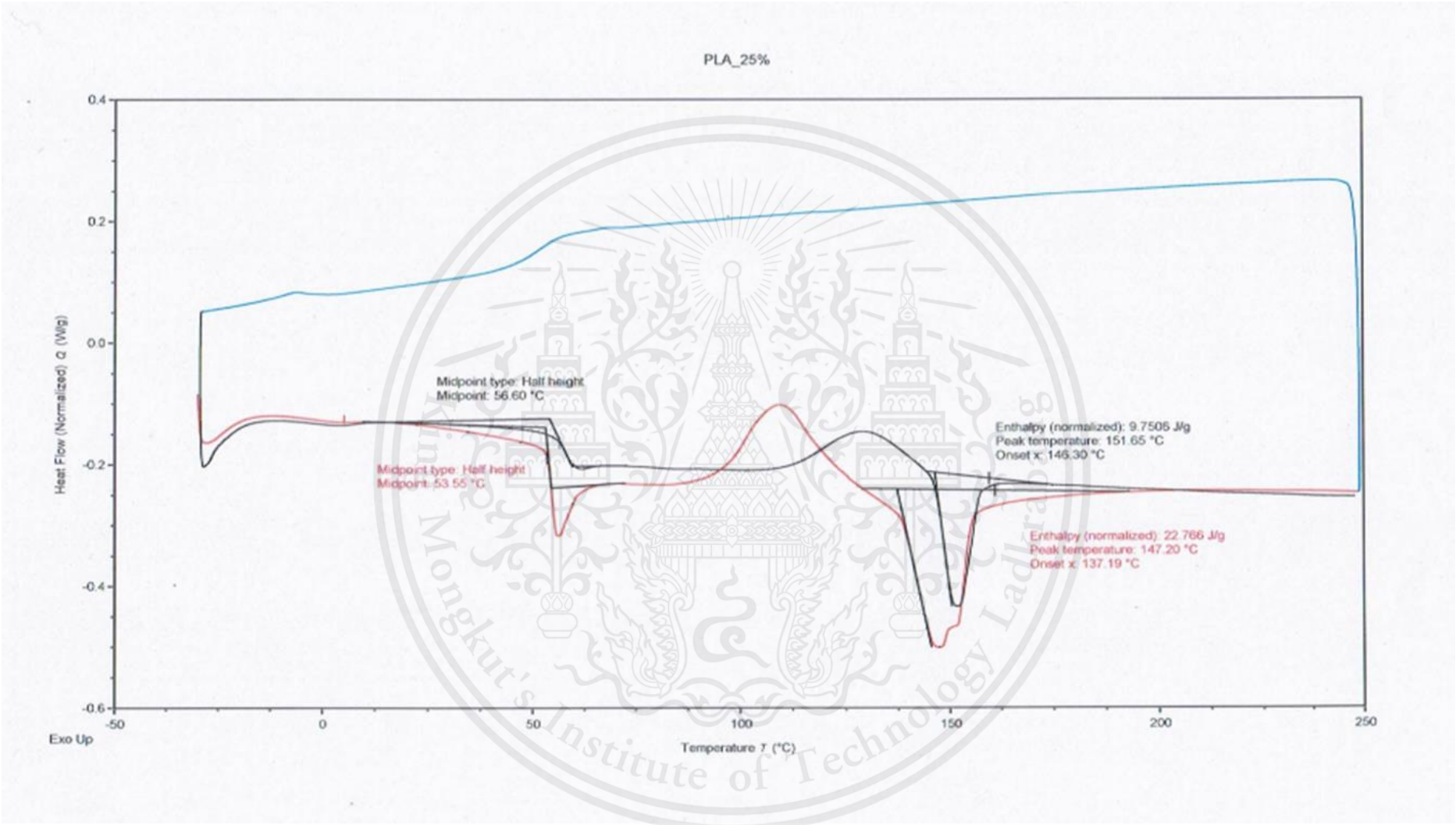

Figure 8.5: DSC chromatogram of PLA/SCG bio-composites film 25 wt.% for first run and second run