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นายสุภลิตี ผู้มีโชคชัย

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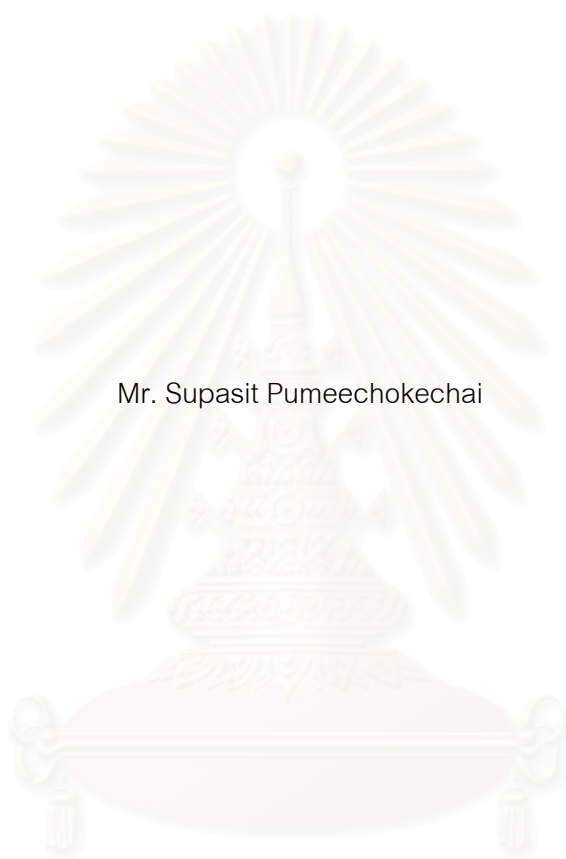
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ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย

PREPARATION OF MODIFIED CASSAVA STARCH AND POLYPROPYLENE BLENDS

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งานวิจัยนี้เป็นการเตรียมพลาสติกย่อยสลายทางชีวภาพจากพอลิเมอร์ผสมระหว่างแป้งมันสำปะหลังดัดแปรและไม่ดัดแปรกับพอลิโพรพิลีน โดยแป้งมันสำปะหลังดัดแปรสามารถเตรียมได้จากปฏิกิริยาระหว่างแป้งมันสำปะหลังกับมาเลอิกแอนไฮไดรด์ 25 เปอร์เซ็นต์ โดยใช้โซเดียมไฮดรอกไซด์เป็นสารเร่งปฏิกิริยา และน้ำเป็นตัวทำละลาย อุณหภูมิที่ใช้ในการดัดแปรคือ 50 องศาเซลเซียส และเวลาที่ใช้ทำปฏิกิริยาคือ 2 ชั่วโมง แล้วนำแป้งมันสำปะหลัง ทั้งที่ดัดแปรและไม่ได้ดัดแปรมาผสมกับพอลิโพรพิลีนและขึ้นรูปเป็นแผ่นด้วยวิธีการอัดแบบ ที่อุณหภูมิ 180 องศาเซลเซียส เป็นเวลา 5 นาที โดยเปลี่ยนแปลงปริมาณของแป้งเป็น 0 5 10 15 20 25 30 40 50 60 70 80 90 และ 100 เปอร์เซ็นต์โดยน้ำหนัก พบว่า เมื่อปริมาณแป้งสูงกว่า 50 เปอร์เซ็นต์แผ่นพอลิเมอร์ผสมมีความเปราะ จนไม่สามารถตัดเป็นชิ้นทดสอบได้ และเมื่อปริมาณแป้งเป็น 0-50 เปอร์เซ็นต์ พบว่า พอลิเมอร์ผสมที่เตรียมจากแป้งมันสำปะหลังดัดแปรมีสมบัติด้านแรงดึง สมบัติด้านแรงดัดโค้ง การดูดซึมความชื้น และความสามารถในการย่อยสลายทางชีวภาพดีกว่าพอลิเมอร์ผสมที่เตรียมจากแป้งมันสำปะหลังที่ไม่ผ่านการดัดแปร โดยมีความทนแรงกระแทกใกล้เคียงกัน

สถาบันวิทยบริการ
จุฬาลงกรณ์มหาวิทยาลัย

ภาควิชาวัสดุศาสตร์

ลายมือชื่อนิสิต.....

สาขาวิชาวิทยาศาสตร์พอลิเมอร์ประยุกต์และเทคโนโลยีสิ่งทอ ลายมือชื่ออาจารย์ที่ปรึกษา.....

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This research presents the preparation of biodegradable plastic from polymer blends between modified and unmodified cassava starch with polypropylene (PP). Modified cassava starch can be prepared from the reaction between cassava starch and 25% of maleic anhydride using sodium hydroxide as a catalyst and water as a solvent at 50°C and the reaction time of 2 hours. Both modified and unmodified cassava starch were mixed with PP and compressed to form polymer blend sheets at 180°C for 5 minutes. The amounts of starch were varied from 0 5 10 15 20 25 30 40 50 60 70 80 90 and 100% w/w. It was found that as the amount of starch was higher than 50%, the blend sheet were brittle so they cannot be processed into test specimens. When the amount of starch was 0-50%, it was found that modified starch/PP blends exhibited higher tensile properties, higher flexural properties, comparable impact strength, higher %moisture absorption and better biodegradability than those of unmodified starch/PP blends.

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จุฬาลงกรณ์มหาวิทยาลัย

Department Materials Science

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CHAPTER I

INTRODUCTION

It is estimated that nearly 2% of all plastics ultimately reach the environment, leading to acute pollution problems. To add to this, diminishing petroleum resources have inspired interests in various alternatives. This has led to the research and development of biodegradable plastics. However, their use has been restricted to certain applications due to their high cost and limited properties. One biopolymer showing great promise is starch, due to its large abundance and low cost. However, its poor properties and processability must be improved with physical and/or chemical modifications.

It was a progression to blend polypropylene with starch to reduce costs and begin to develop an economically viable biodegradable plastic. Unfortunately many researches have shown that the blends show a clear trend of decreasing mechanical properties with increasing starch content. This decrease in properties is significant and makes the material unacceptable for commercial usage. The poor properties of starch blends can be attributed to the incompatibility of the hydrophobic polypropylene and the hydrophilic starch. It was thought and correctly so, that the properties of starch/polypropylene blends could be improved by the use of a chemical modification to enhance the interfacial adhesion between the two phases.

In this research, cassava starch was modified by esterification with maleic anhydride to obtain the functional groups expected to interact with polypropylene. The properties of modified starch/PP blends were compared to those of the blends prepared from unmodified starch. In order to investigate the role of maleic anhydride segments presented on modified starch molecules.

CHAPTER II

THEORY AND LITERATURE SURVEY

Polypropylene is a widely used packaging material due to its good material properties and low cost. However, these qualities have overshadowed its non-biodegradable nature, leading to waste disposal problems. These problems have led to the research and development of biodegradable plastics. Several fully biodegradable have been developed including aliphatic polyesters, natural polymers such as starch and cellulose. The widespread use of these plastics has been limited due to their high cost and restricted abilities. One biopolymer showing great promise is starch, due to its large abundance and low cost. However, the poor properties and processability must be improved with physical and/or chemical additions.

2.1 Polypropylene

Polypropylene (PP), a thermoplastic polymer which entered to commercial production in 1957, was the first of stereoregular polymer. Polypropylene differs from polyethylene because there is a methyl group attached to every other carbon atom, which stiffens the chain. Unless the methyl groups are arranged in one position relative to the chain, the polymer does not crystallize until the stereospecific catalyst were developed (independently) by Natta and Ziegler. Crystallinity is responsible for the stiffness and solvent resistance of the commercial plastic. Polypropylene is synthesized by placing propylene monomer under controlled conditions of heat and pressure in the presence of organometallic, stereospecific catalysts (Ziegler-Natta type). [1]



Depending on catalysts and polymerization processes used, the molecular structures of the resulting polypropylene consist of three different types of stereochemical configurations in varying amounts. These configurations are isotactic, syndiotactic, and atactic.

The most properties of polypropylene are Typical Isotacticity 94 - 98 %, Typical Crystallinity 60 %, MFR range 0.3 - 50 dg/min (until >100 for special applications, Mw/Mn 4 - 7, Crystalline phase melts at ca. 165°C., and Amorphous phase T_g ca. 0°C. low specific gravity, excellent chemical resistance, high melting point (relative to volume plastics), good stiffness/toughness balance, adaptability to many converting methods, great range of special purpose grades, excellent dielectric properties, and low cost (especially per unit volume). It has gained wide acceptance in applications ranging from fibers and films to injection-molded parts for automobiles and food packaging. [2] More than 7 billion pounds of polypropylene are produced annually in the United States. About 20% of this volume consists of copolymer, mostly copolymers containing 2-5% ethylene into the polymerization reactor. The resulting polymer has increased clarity, toughness and flexibility. [1] Some properties that are usually considered inherent advantages of polypropylene are low specific gravity, chemical resistance, high melting point, good stiffness, and low cost.

Although polypropylene has many useful properties, it is not intrinsically tough especially below its glass transition temperature. However, its impact resistance can be improved by adding elastomers usually ethylene/propylene rubber. [3] Numerous studies have been carried out that aim at improving PP toughness, stiffness, and strength balance. The addition of mineral fillers and reinforcing agents to the polymeric matrix reduces the cost of the material and enhances some of the mechanical properties.

Commercial polypropylene usually contains 90-95% isotactic structure. In the products, atactic and syndiotactic structures may be present either as complete molecules or as blocks of varying length in chains of isotactic molecules. Isotactic structure is stiff, highly crystalline and has high melting point. Within the range of commercial polymers, the higher the amount of isotactic structure, the higher the crystalline content and, hence, the higher the mechanical properties including stiffness, tensile strength, modulus and hardness.

2.2 Starch structure [4]

Starch is a major form of carbohydrate storage in green plants and is considered to be the second largest biomass, next to cellulose, produced on earth. It is the polymer that consists of six-membered-ring glucose repeating units. The molecular weight of starch varies from 104 to 107 daltons. Native starch is the main energy storage supply in botanical sources. Starch is a naturally occurring carbohydrate of green plant cells and microorganisms. Like other naturally occurring carbohydrates, starch is a polysaccharide consisting of glucosidic units only. Starch consists of two molecules amylose and amylopectin which both contain alpha-D-glucose units only.

2.2.1 Amylose

Amylose is a linear or sparsely branched polymer with a molecular mass in the range of 105 to 106 g/mol primarily by 1-4 linked glucose units. These chains form a spiral shaped single or double helix. It may contain about 200 to 2000 anhydroglucose units. At one end of the polymeric molecule, the anhydroglucose unit contains one of primary and two secondary hydroxyl groups as well as aldehydic reducing group in the form of an inner hemiacetal. This is called the reducing end of the molecule. Figure 2.1 shows the molecular structure and helical shape of amylose.

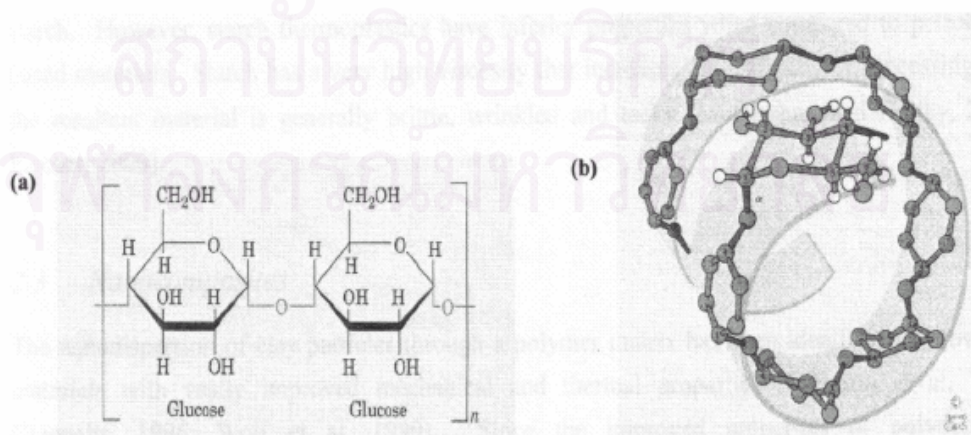


Figure 2.1 (a) Molecular structure of alpha amylose. (b) Helical shape of alpha amylose polymer. [4]

The abundance of hydroxyl groups imparts hydrophilic properties to the polymer, giving it the affinities for moisture absorbability and dispersibility in water. However, because of their linearity, mobility and polarity, amylose molecules have a tendency to orient themselves in parallel fashion and approach each other closely enough to permit hydrogen bonding between hydroxyl groups on adjacent molecules. As a result, the affinity of the polymer for water is reduced and the sol becomes opaque. In dilute solutions, the aggregate size of the associated polymers may increase to a point where precipitation between segments of the polymer may occur, producing a gel consisting of a three-dimensional network held together by hydrogen bonding at those sections where close alignment has occurred. In general, the linearity of amylose favors formation of a strong film. For the phenomenon in which intermolecular association between amylose molecules at high concentration is commonly called retrogradation (starch molecules associate and precipitate in an insoluble form). Amylose can form a complex with iodine giving a deep blue color which is used to identify amylose-containing starch.

2.2.2 Amylopectin

Amylopectin is a highly branched molecule with a molecular mass between 107 and 109 g/mol. Amylopectin also contains 1-4 linked glucose units, but has in addition 1-6 glucosidic branching points occurring every 25-30 glucose units. Figure 2.2 shows the molecular structure and crystal microstructure of amylopectin.

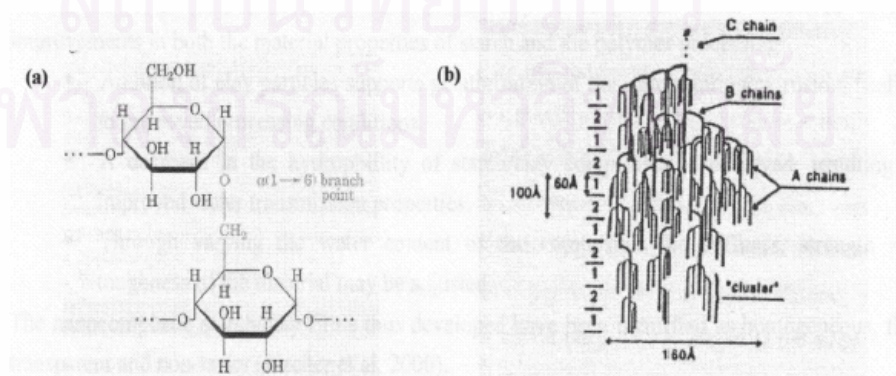


Figure 2.2 (a) Molecular structure of amylopectin (b) Microstructure of amylopectin. [4]

The large size and branched nature of amylopectin reduce the mobility of its molecules and interfere with their tendency to orient closely enough to permit significant levels of hydrogen bonding. As a result, aqueous sol of amylopectin is characterized by clarity and stability as measured by resistance to gelling on aging. Amylopectin sol does not form strong and flexible films as linear amylose does. Furthermore, it does not form a complex which gives deep blue coloration with iodine.

The amylose content of most starches varies between 20-30%, which leads to the crystallinity of native starches ranging from 20-45%. The branching amylopectin molecules dominate the crystallinity and are responsible for the amorphous regions in the starch.

2.2.3 Starch gelatination

Native starch is presented in semicrystalline granular forms with densities ca. 1.5 g/cm³. Starch granules are insoluble in cold water but they can be solvated by heating with water, or by treating them with organic solvent (e.g. dimethyl sulfoxide), aqueous alkaline, or salt solution (e.g. CaCl₂-KI)

For the dried starch granules, they show polarization crosses reflecting crystalline organization when examined microscopically under polarized light. The granules absorb water to a limited extent when exposed to high humidity or when suspended in water. However, the swelling is reversible and polarization crosses remain of granules for various starches in passing from anhydrous condition to a water-saturated atmosphere, together with increase in water sorption, are shown in Table 2.1. When equilibrated under normal atmospheric conditions, starches usually contain 10 to 20% moisture depending upon the starch source.

Table 2.1 Effects of water-saturated atmosphere on granule diameter and water sorption of various starches. [4]

Starch source	Average increase in granular diameter (%)	Water sorption/100g dry starch (g)
Corn	9.1	39.9
Potato	12.7	50.9
Tapioca	28.4	47.9
Waxy corn	22.7	51.4

When the slurry of starch in water is heated above a critical temperature, which varies with the type of starch and other factors, the hydrogen bonds are responsible for the structure integrity of the granule weaken allowing the penetration of water and hydration of the linear segments of the amylopectin. As this occurs, the molecules start to form helices or coils, creating tangential pressure causing the granules to sludge water and swell too many times their original volume.

During the swelling, amylose tends to reach out of the granule and, along with the amylopectin, become highly hydrated. The suspension becomes clear and viscosity of the suspension rises and continues to rise until it approaches a peak where the granules have approached their maximum hydration. As heating is continued, the granules tend to rupture, collapse, and fragment, releasing the polymeric molecules and aggregates. This causes viscosity drops. During the process as the polymeric molecules are released, the sol often develops a cohesive, rubbery texture.

As the a sol is cooled, the clarity decrease and the viscosity tends to rise and, in the case of regular cereal starches such as corn or wheat, the sol forms a gel if the concentration is sufficiently high. Sols of cereals starches, having relatively high levels of moderately sized amylose molecules, become opaque and form gels on cooling. Those of potato and tapioca, however, usually maintain there clarity much better than other

starches and do not form opaque gels while they thicken on cooling. Waxy corn starch, unlike regular corn starch, behaves like tapioca or potato, its sol shows even less tendency to thicken on cooling than tapioca or potato. The difference in behavior between starches such as tapioca and potato and starches such as corn and wheat may be attributed to be lower amylose content and bulkier amylose molecules in the former starches, reflecting their higher molecular weights and possibly slight degree of branching.

As a general rule, root or tuber starches swell more rapidly in a narrow temperature range than the common cereal starches. For example, cassava starch, used in this experiment, shows the initial and final gelatinizations occurred at 60°C and 80°C, respectively.

2.2.4 Cassava starch

Cassava is the term usually applied in Europe and in United States to the root of the plant, whereas, tapioca is the given name for the processed products of cassava [5]. The composition of cassava's root is shown in Table 2.2 [5]. The size of the cassava granules ranges from 5 to 35 µm. The amylose content in cassava starch is about 16.5-22%.

Table 2.2 Composition of cassava's root. [5]

Composition	Percent
Moisture	69.8
Starch	22.0
Sugar	5.1
Protein	1.1
Fat	0.4
Fiber	1.1
Ash	0.5

Cassava is recognized as one of the most important crops to the Thai economy [6]. It is the third most important crop after rice and sugarcane. The production of the crop is on average about 18 million tons in each crop year from a planted area of 1.5 million hectares, and about half of this amount (9 to 10 million tons) is converted to starch. This product capacity was 5% of the global total in 1999. In 1994, Thai Tapioca Flour Industrial Association estimate the annual starch export growth rates between 1987 and 1992 as follows: native 10.5%, modified 33.8%, sorbitol 48.9%, monosodium glutamate (MSG) 12.8%, glucose syrup 9.4%, and sago 8.3% while the domestic cassava starch utilization by industry presented as percents of 1,121,625 tons of starch produced in 1994 for domestic usage is shown in Table 2.3. [6]

Table 2.3 Percent of total domestic use of cassava starch in Thailand in 1994. [6]

Products	Percent of total domestic use
Chemically modified starch	25.4
MSG (80%) and lysine (20%)	12.1
Glucose/fructose syrup	12.0
Food processing	11.9
Paper	11.5
Physical modified starches	7.4
Sago pearl	3.6
Plywood	2.1
Textile	1.9
Sorbitol	1.6
Adhesives	1.2
Other	9.5

Many techniques are available to improve the properties of starch, such as

- The addition of plasticisers such as glycerol, urea and water to induce melting below the starch decomposition temperature and to form a continuous polymeric entangled phase commonly known as thermoplastic starch (TS).
- The addition of other polymers/biopolymers into starch to produce a material usually of properties intermediate to the two additives.
- The addition of nanocomposites such as clays to increase the interfacial adhesion and subsequently improve the thermal and mechanical properties.
- The addition of compatibilisers to increase the miscibility of the two phases to decrease interfacial energy and stabilise the polymer blend to create a blend with improved characteristics.
- The chemical modification by reduced the OH-group of the starch with the esterification reaction in order to change properties of the starch to more hydrophobic before blend it in to the thermoplastic.

2.2.5 Starch thermoplastics [7]

An important property of starch is its semi-crystallinity. In order to make a starch thermoplastic product by conventional processing technique, it is necessary to disrupt the starch granule and melt the partially crystalline structure. For granular starch the glass transition temperature (T_g) is above the decomposition temperature (T_d) due to the strong interactions by hydrogen bonding in the chains. Plasticisers are added to lower the T_g below the T_d . This forms a product in which the starch polysaccharides form a continuous polymeric entangled phase commonly known as thermoplastic starch (TS). The properties of TS are dependent on the type and amount of plasticizers. TS can be

processed via conventional processing techniques such as extrusion, compression moulding etc. Common plasticizers are water and glycerol.

Although TS materials can be processed via conventional processing they often have high viscosity and poor melt properties in comparison with synthetic polymers and often lead to brittle and water sensitive products.

Kiatkamjornwong S. and Sonsuk M. studied about degradation of styrene-g-cassava starch filled polystyrene plastics. Cassava starch was chemically modified by radiation grafting with acrylic acid to obtain cassava starch graft poly(acrylic acid), which was further modified by esterification and etherification with poly(ethylene glycol) 4000 and propylene oxide, respectively. The modified product was characterized by NMR spectroscopy and contact angle measurement. The blends of LDPE with EBS wax had properties similar to the LDPE blends with the modified starch in terms of surface wettability, tensile properties, and hardness, but with a much better degradation in soil due to the much higher water absorption. This article describes the chemical modifications of hydrophilic cassava starch to become partially hydrophobic, which was then used for blending with LDPE sheets for evaluations of mechanical, thermal and degradation properties. [6]

Ramani N. used maleic anhydride and phthalic anhydride to modified cassava starch, used dimethyl sulfoxide as solvent, and pyridine as catalyst. [8]

2.3 Literature survey

One way of avoiding its high viscosity and poor melt properties is to blend the starch with synthetic polymers.[9] However, the vast majorities of synthetic polymers on the market are non-biodegradable and made from non-renewable resources. In order to produce a biodegradable plastic, the plastic must be blended with the starch. One of the most developed biodegradable plastics is polypropylene. However, due to their high costs and limited mechanical properties, their commercial usage is minimal. Starch/polypropylene blends are biodegradable plastic and would be cheaper than the virgin polypropylene if they were commercialized due to the abundance of starch. The

prices of virgin polypropylene are approximately many times that of starch, although with starch additions the cost can be reduced. Unfortunately, these resulting starch based plastics are not mechanically comparable to polypropylene and research is currently attempting to improve this. The poor properties of starch blends can be attributed to the incompatibility of the hydrophobic polypropylene and the hydrophilic starch. It was thought and correctly so, that the properties of polypropylene -starch blends could be improved by the use of a compatibiliser to enhance the interfacial adhesion between the two phases.

Bikiaris D. and Panayiotou C. was studied about LDPE/plasticized starch blends containing PE-g-MA copolymer as compatibilizer. They study a series of polyethylene/plasticized starch blends were prepared using a poly(ethylene-g-maleic anhydride) copolymer as a reactive compatibilizer. Uncompatibilized blends were also prepared for comparison purposes. The prepared blends were studied using mechanical properties measurements and SEM microscopy to determine their morphology. [10]

In the other case was studied by Mani T. They studied the process of stabilizing the physical interactions in polymer blends is commonly called Compatibilisation. A compatibilisation strategy frequently proposed is the addition of a premade block/graft copolymer composed of blocks that are each miscible with one of the homopolymers. [11] These compatibilisers can turn incompatible blends into useful compatible materials by reducing the interfacial tension and creating a more stable microstructure which is more resistant to mechanical degradation. Figure 2.3 shows an electron micrograph of a starch/PCL blend with and without compatibiliser. Two clearly distinct phases are visible in the uncompatibilised blend. Whereas, a more evenly distributed polymer, results from the compatibilised blend. The increase in homogeneity can be explained through the phobicity of the molecules in the blend. Starch is hydrophilic and PCL is hydrophobic, this causes repulsions throughout the uncompatibilised blend that are dramatically diminished by the well dispersed copolymer addition. When an uncompatibilised starch/PCL blend is produced the two phases do not integrate well and stay as two distinct phases in a state of low interfacial adhesion. This causes the blend to be mechanically weak. By dispersing these copolymer molecules throughout

the starch/PCL matrix, a significant improvement in the mechanical properties was encountered as a result of this decreased repulsion and hence increased interfacial adhesion. [11]

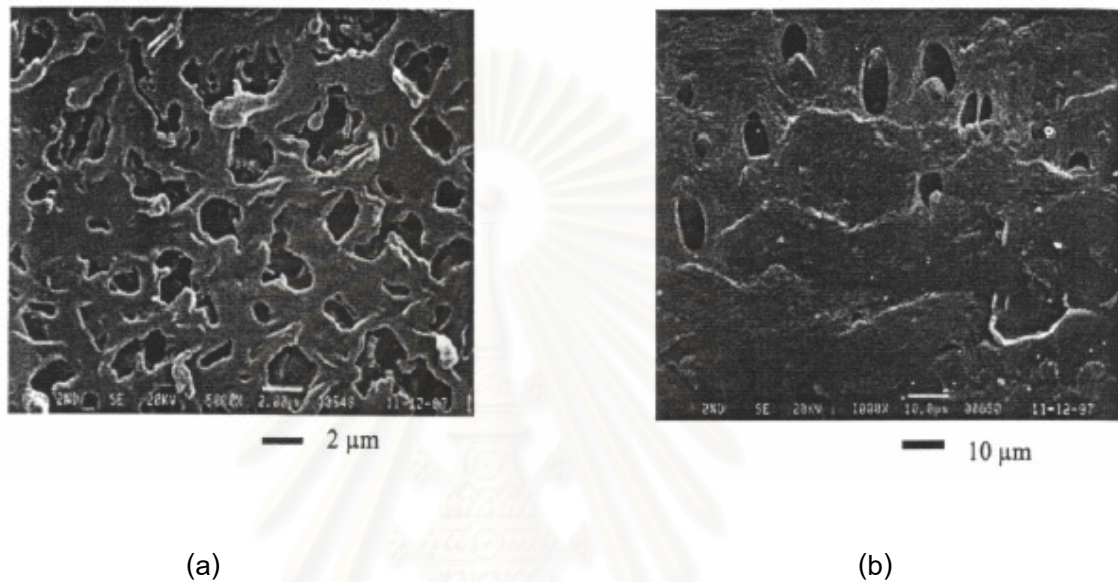


Figure 2.3 SEM micrographs of the PCL/starch. (a) Without compatibiliser (b) With compatibiliser [11]

Developing a blend with satisfactory overall physicomechanical behaviour depends on a proper interfacial tension to generate a small phase size and strong interfacial adhesion to transmit an applied force effectively between the component phases. [12], [13] Methods to reduce the interfacial energy between the two immiscible phases and hence improve the phase adhesion has been a subject of considerable research. Compatibilisation is proving to be an excellent method of improving the properties of starch blends. In another recent study, a 70% starch loaded PCL blend had a three fold increase in tensile strength due to the addition of 3wt% starch-*g*-PCL. [11]

2.4 Formulation and analysis of compatibilised starch blends

2.4.1 Extrusion [9]

Extrusion is a common polyolefin processing tool used to shear and melt polymer pellets where they can then be formed into usable products. The rheological condition at the end of the extruder is dependent on the degree of conversion of the partly crystalline starch grains into a homogeneous plastic matrix and on the degree of degradation of the molecular chains.

During extrusion starches are subjected to relatively high pressure. Under extrusion conditions, starch can undergo gelatinization, melting and fragmentation reactions. Studies have shown that the loss of crystallinity of starch after extrusion is caused by the intense shear fields within the extruder. Operating conditions such as barrel, and die temperature, screw speed, and screw geometry, as well as amylose/amylopectin ratio and moisture content have a significant effect on mechanical disruptions and starch transformations. It is well known that shear results in fragmentation of starch granules during extrusion.

The specific mechanical energy can be related to the molecular weight of starch as shown in Figure 2.4.

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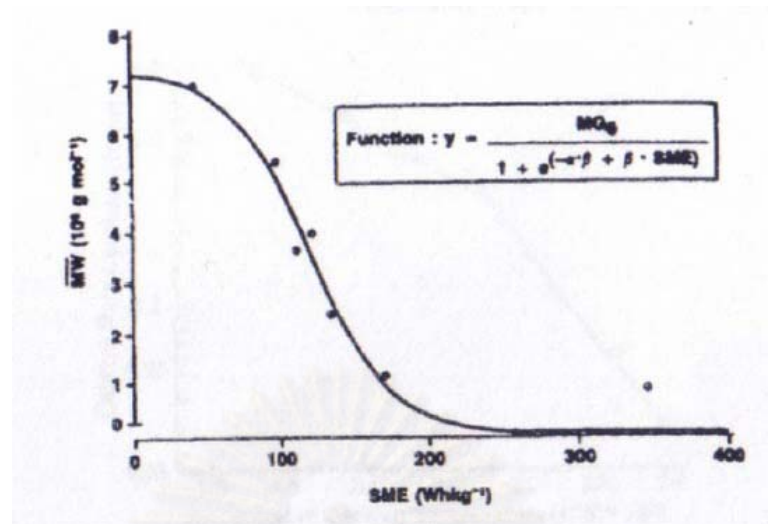


Figure 2.4 The influence of SME on the medium molecular weight of wheat starch [14]

2.4.2 Compression molding [9]

In compression molding, granular polymers are heated and forced, or compress into a mold cavity, cooled and removed. This process is used to produce many items, from plastic sheets to automobile bumpers. A compression molding machine has two main sections: the heater and the clamp unit which houses the mould. All thermoplastic polymer (except with high viscosity like fluoroplastic, polyimides, and some aromatic polyesters) can be compression molded and many thermosetting polymer can be compressed molded as well. Advantages of compression molding are high production rates, ability to compress around inserts, little or no finishing requirements, and recycling capability. The only major disadvantage is the low cost of equipment and mold.

Figure 2. shows the principles of compression molding machine.



Figure 2.5 Compression molding machine

Molding machine used the hotplate to heat the polymer in the mold where it take the shape of the cavity. Heated and compressed. When cooled, the parts are removed and the excess materials are removed and regrounded for recycling.

2.4.3 Mechanical properties

Tensile testing is a common technique used to determine the mechanical properties of materials. Typical parameters that characterise materials are ultimate tensile stress, strain at break and Young's modulus of elasticity. Most research on biodegradable plastics ultimately seeks to improve the mechanical properties to a level that satisfies a particular application. So quantification of mechanical properties directly, through tensile testing is a valuable tool that can allow detection of improvements quickly. A graph of tensile stress versus strain is the clearest way to illustrate tensile data

2.4.4 Thermal properties

Differential Scanning Calorimetry (DSC) is a technique widely used to evaluate the thermal properties of materials. Melting and crystallisation temperatures, crystallinity and ultimately phase structure can be researched using DSC thermograms. These properties are of primary importance since they are directly related to the physical structure within the material and hence the overall properties. DSC is a particularly important tool in the analysis of starch based materials due to the complexity of the thermal properties of starch.

Gelatinisation is a property of starch that occurs under excess moisture. It is believed that during gelatinization water penetrates the amorphous region, initiating swelling, resulting in a decrease in birefringence. Water strips starch chains from the surface of crystallites. Increasing temperature and solvation induced by swelling results in disruption of the crystalline regions and uncoiling of the double helices until the structure is completely disrupted. [14] It is generally accepted that when water concentration is limited, complete gelatinization will not occur at the usual gelatinization temperature range. But as the temperature is increased, the starch granules become progressively more mobile and eventually the crystalline regions will melt. [14] DSC thermograms of a starch material, illustrating these melting and gelatinisation transitions is shown in Figure 2.6.

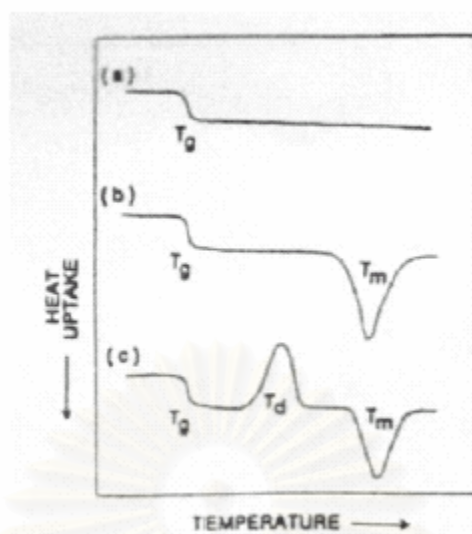


Figure 2.6 - DSC thermograms for starch (a) T_g of a fully gelatinised Starch (b) Partially crystalline starch, showing melting. (c) An amorphous but crystallisable material under conditions of rapid temperature change. [14]

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CHAPTER III

EXPERIMENTAL

3.1 Raw Materials and Reagents

3.1.1 Polypropylene

Commercial-available polypropylene powder (PP) (N700P) having melt flow index of 7.0 was supplied by TPI Co., Ltd

3.1.2 Cassava starch

Cassava starch powder was supplied by Thai Wah Co., Ltd

3.1.3 Reagents

- 1) Maleic anhydride, GR grade was purchased from Fluka.
- 2) Sodium hydroxide, AR grade was purchased from Ajax Fine Chem.
- 3) 98% Hydrochloric acid, AR grade was purchased from J.T Beker.

3.2 Machines and Equipment

- 1) Compression molding machine
- 2) Grinding machine : Misubishi electric Co., Ltd.
- 3) Ball mill granulating machine
- 4) Cutting machine : Yasuda
- 5) Universal testing machine : LLOYD 100.
- 6) Universal testing machine : LLOYD 500.
- 7) Impact testing machine : Zwick 5102.
- 8) FT-IR spectrometer : Perkin Elmer System 2000-FT-IR
- 9) Scanning electron microscope : JSM-6400.

3.3 Methodology

The flow chart of the entire experimental procedure is shown below in Figure 3.1.

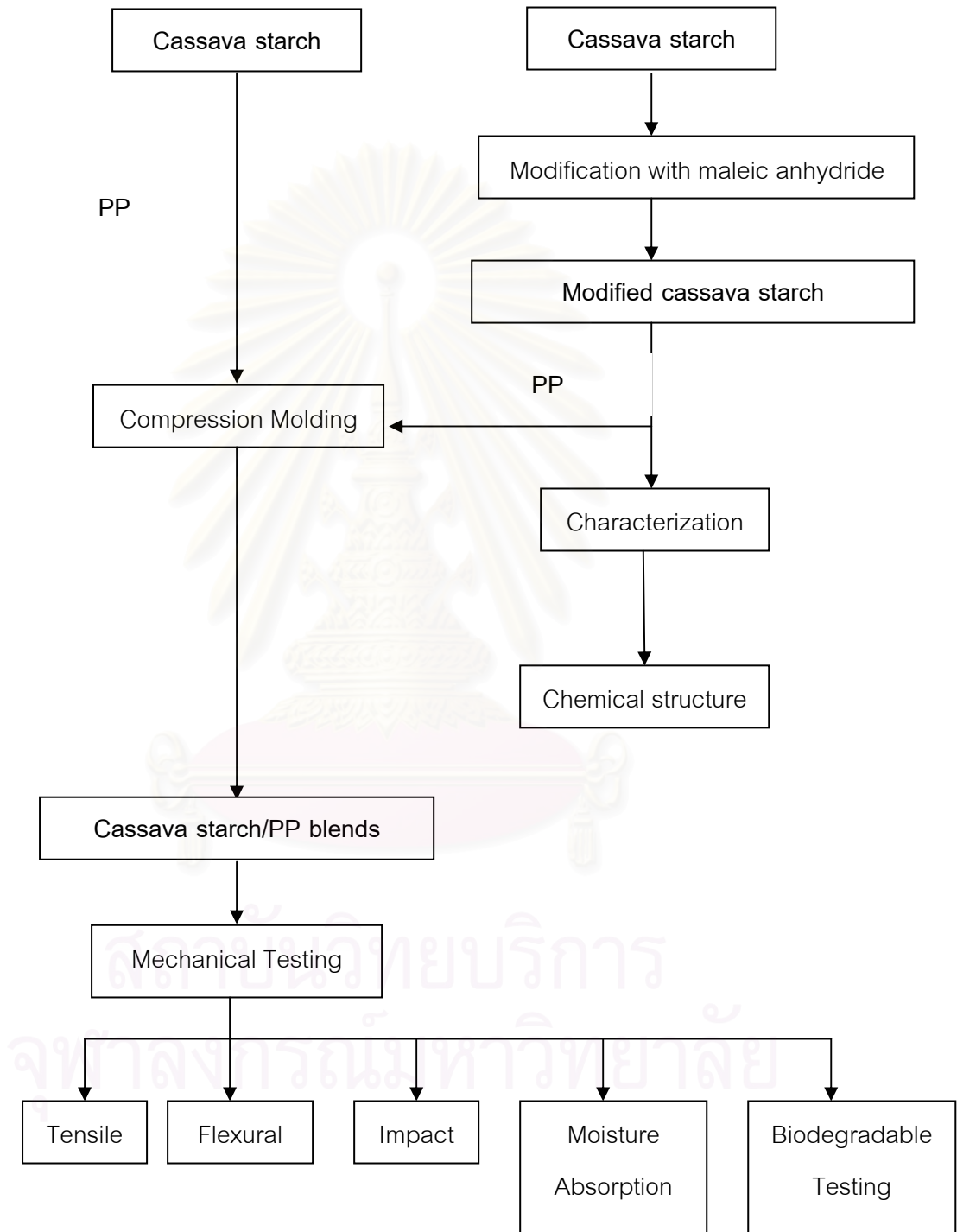


Figure 3.1 Flow chart of experimental procedure

3.3.1 Modification of cassava starch

1) Cassava starch was weighed in order to calculate the amounts of sodium hydroxide and maleic anhydride which should be used

2) Aqueous solution of sodium hydroxide was prepared in a 1,000 cm³ beaker. Cassava starch was put and stirred in this solution at a speed of 950 rpm at 55°C for 30 minutes.

3) Maleic anhydride was slowly added and the mixture was stirred at 55°C. The amount of maleic anhydride was set at 25% mole of the cassava starch for 2 hours.

4) The mixture was neutralized with hydrochloric acid solution. After that, modified cassava starch was collected, rinsed with methanol and dried at 70°C for 24 hours.



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3.3.2 Preparation of starch/PP blends.

1) Modified starch was mixed with polypropylene at the ratio of 0:100, 5:95, 10:90, 15:85, 20:80, 25:75, 30:70, 35:65, 40:60, 50:50, 60:40, 70:30, 80:20, 90:10 and 100:0 respectively. It was placed in a mold having dimension 150x150x4 mm.

2) Blend mixture was preheated for 2 minutes. Compression molding was set at 180°C, 10 tons of pressure and compressed for 5 minutes. Finally, the compressed sheet was cooled down at room temperature. The product was cut to the standard specimens according to ASTM test method. The compression molding machine was shown in Figure 3.2



Figure 3.2 Compression molding machine.

3.4 Analysis of modified starch

3.4.1 Chemical structure

The infrared spectra were obtained using a Perkin Elmer System 2000-FT-IR to characterize chemical structures of the samples. The samples were scanned at the frequency range of 4000-600 cm^{-1} with 32 consecutive scans and 4 cm^{-1}

resolution. about 2 mg of starch modified. The powdered material was then mixed KBr and processed in to a small disc 1 mm thick.



Figure 3.3 Perkin Elmer System 2000-FT-IR spectrometer

3.5 Analysis of starch/PP blends

3.5.1 Tensile properties

Tensile testing was performed to assess the changes in the mechanical properties of the blends with the addition of modified starch. The tensile properties of the samples were tested using an Universal testing machine model LR 100, following the ASTM D638-82a (type IV) where applicable.

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Figure 3.4 Universal testing machine

The strands from the compression mold were tested using a 50 mm grip length with a crosshead speed of 10 mm/min. The samples were maintained at a relative humidity of 50% for at least 48 hours before testing.

The test specimen (type IV) dimension was presented in Figure 3.5

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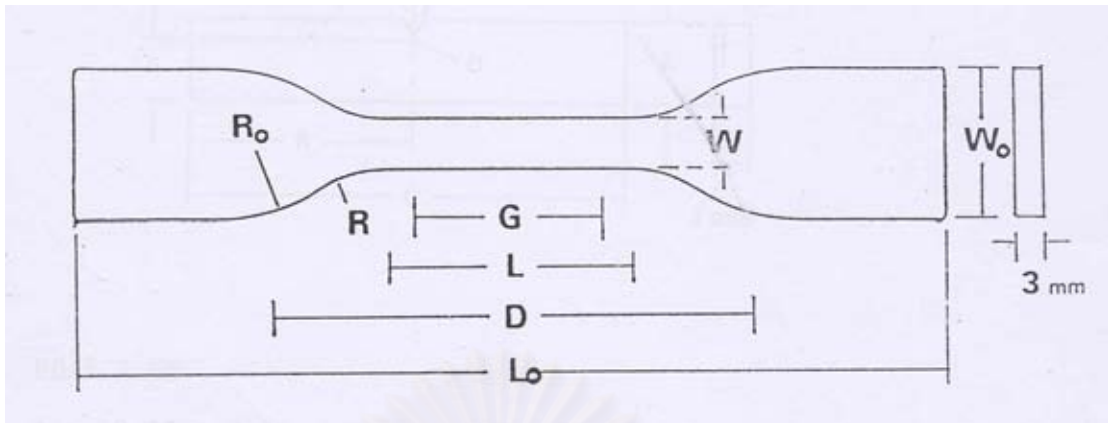


Figure 3.5 Schematic of tensile test specimen (type IV)

W :	6 mm	W_0 :	19 mm
G :	25 mm	R :	14 mm
L :	33 mm	L_0 :	115 mm
D :	65 mm	R_0 :	25 mm

The tensile testing conditions were as follows:

Temperature :	25	$^{\circ}\text{C}$
Relative humidity :	50	%
Load cell :	1000	N
Speed of testing :	10	mm/min
Distance between grips :	64	mm
Gage length :	25	mm

3.5.2 Impact strength

The impact strength of the samples were tested using a Zwick 5102 Pendulum Impact tester following the ASTM D256-04 where applicable, according to the ASTM D256-04 (type Izod)



Figure 3.6 Pendulum impact tester

The samples were maintained at a relative humidity of 50% for at least 48 hours before testing.

The test specimens dimension for Zwick 5102 Pendulum impact tester (Izod-type) test is shown in Figure 3.7

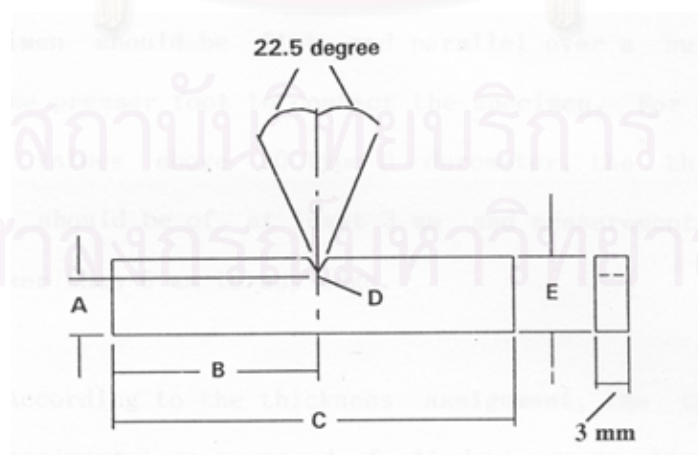


Figure 3.7 Schematic of Izod type test specimen

A :	10.16 \pm 0.05 mm.	D :	0.25 \pm 0.05 mm.
B :	32.00 mm. max, 31.50 mm. min	E :	12.70 \pm 0.15 mm.
C :	63.50 mm. max, 53.50 mm.min		

The machine parameters and testing conditions of impact test were listed by

Temperature:	25	°C
Relative humidity:	50	%
Pendulum capacity:	11.0	J
Depth of specimen:	10.16	mm

3.5.3 Flexural Properties

The flexural properties of the samples were tested using a LLOYD Universal testing machine model 500, following to the ASTM D790-81 where applicable.



Figure 3.8 Flexural testing Machine

The standards from the compression mold were tested using a 80 mm. length with a crosshead speed of 50 mm/min. The samples were maintained at a relative humidity of 50% for at least 48 hours before testing.

The test specimen (Method I) dimension was presented in Figure 3.9

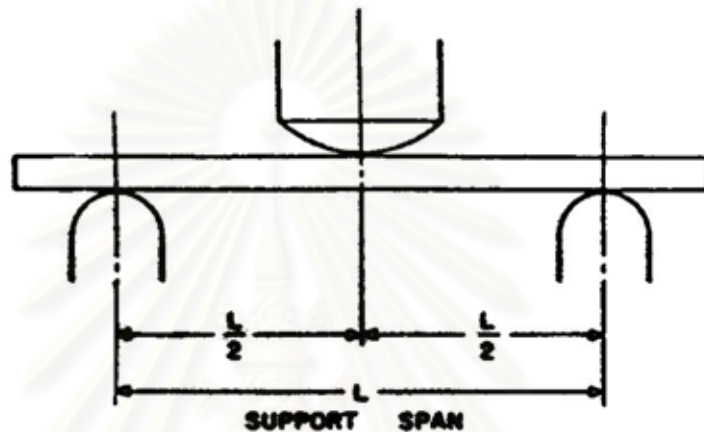


Figure 3.9 Schematic of flexural test specimen.

The flexural testing conditions were as follows:

Depth :	3.2	mm
Width :	25	mm
Length :	80	mm
Support Span :	50	mm
Test speed :	50.00	mm/min

3.5.4 Moisture absorption

Moisture absorption of cassava starch/PP blends was measured by weight. The specimens were dried in an oven for 24 hours at 60°C, cooled in a dessicator and immediately weighed. The dried weight was designated as w_1 . After that, they

were kept in the 75% relative humidity box for 24 hours at room temperature. The specimens were removed and immediately weighed. Moisture absorption of each sample was calculated as the weight difference and is reported as percent increase of the initial weight, according to equation (3.1) as follow :

$$\% \text{ Moisture Absorption} = \frac{W_2 - W_1}{W_1} \times 100 \quad (3.1)$$

When W_1 = initial weight

W_2 = final weight

3.5.5 Biodegradability

Biodegradability of cassava starch/PP blends was measured by weight loss. The specimens were dried in an oven for 24 hours at 60°C, cooled in a dessicator and immediately weighed. The initial weight was designated as W_1 . After that, they were kept in the soil box under the surface at 5 cm. for 2 and 3 weeks at room temperature. The specimens were removed and immediately weighed and designated as W_2 . Biodegradability of each sample was calculated as the weight difference and is reported as percent decrease of the initial weight, according to equation (3.2) as follow

$$\% \text{ Weight loss} = \frac{W_1 - W_2}{W_1} \times 100 \quad (3.2)$$

When W_1 = initial weight

W_2 = final weight

3.5.6 Morphological studies

A scanning electron microscope (SEM) JSM-6400 at acceleration voltage of 15 KV was used to study the impact fractured surface of the starch/PP blends in order to understand the failure mechanism of the specimens. The fractured surface of the sample was coated with gold before being scanned.



Figure 3.10 Scanning electron microscope model JSM-6400

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CHAPTER IV

RESULTS AND DISCUSSION

4.1 Chemical characterization of cassava starch

Figures 4.1 and 4.2, FT-IR spectra of unmodified cassava starch and modified cassava starch exhibit the characteristic broad peak at 2950 cm^{-1} attributed to saturated of C-H bond stretching. The C-H bending for CH_2 appears at 1428 cm^{-1} while C-H bending for CH_3 appears at peak 1345 cm^{-1} shown.

In addition, FT-IR spectrum of modified cassava starch shows a peak corresponding to ester groups resulting from esterification of glutinous starch and maleic anhydride at wavenumber of 1546 cm^{-1}

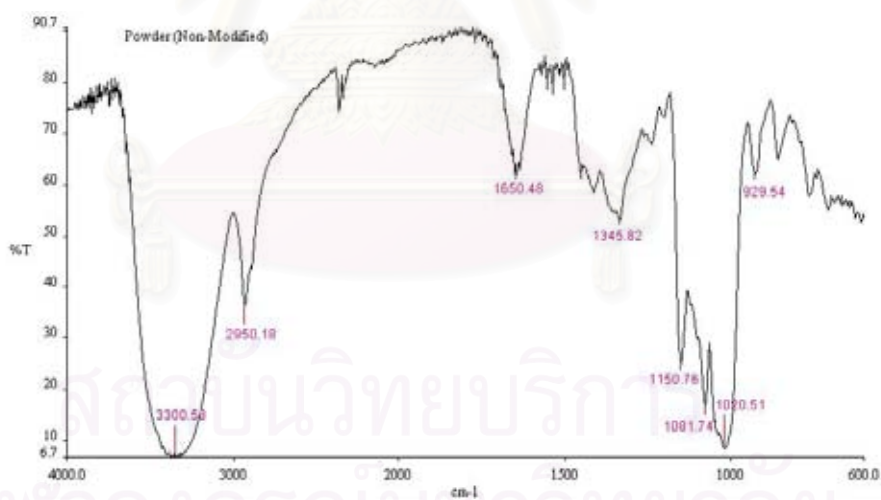


Figure 4.1 FT-IR spectrum of unmodified cassava starch.

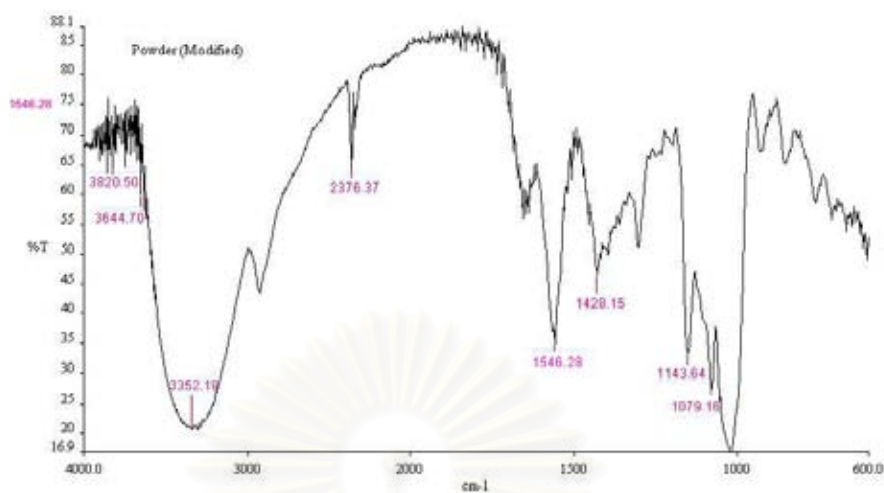


Figure 4.2 FT-IR spectrum of modified cassava starch.

Wavenumbers corresponding to some functional groups are shown below.

Funcation groups	Frequency (cm^{-1})
O-H stretching	3700-3200
Sat. C-H stretching	3000-2800
C-H bending for CH_2	1470-1400
C-H bending for CH_3	1375

4.2 Appearances of cassava starch/PP blends

Sheets of polypropylene blended with modified cassava starch at 0%, 5%, 10%, 15%, 20%, 25%, 30%, 40%, 50%, 60%, 70%, 80%, 90% and 100% w/w are shown in Figures 4.3 and 4.4.

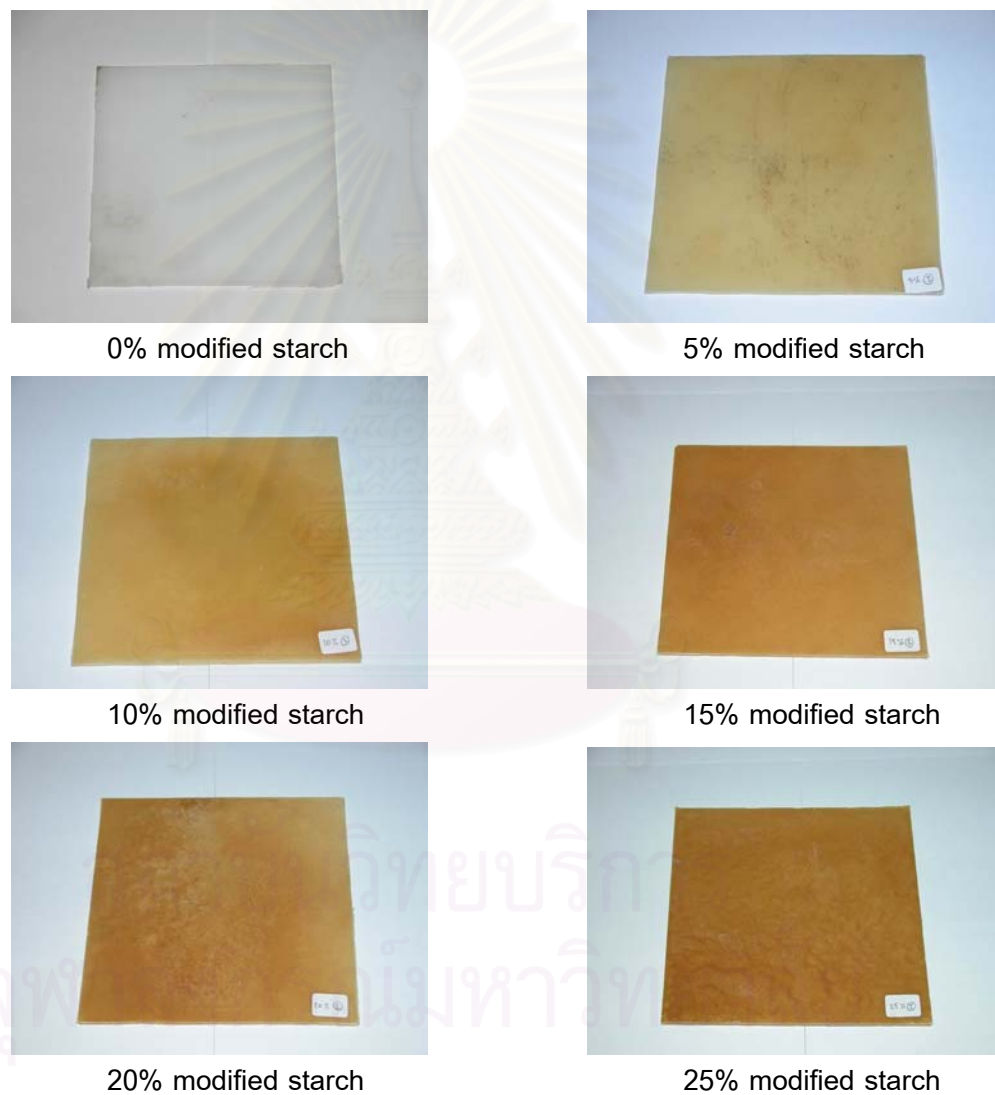


Figure 4.3 Surface of modified cassava starch/PP blends using the amount of modified cassava starch at 0-25%.

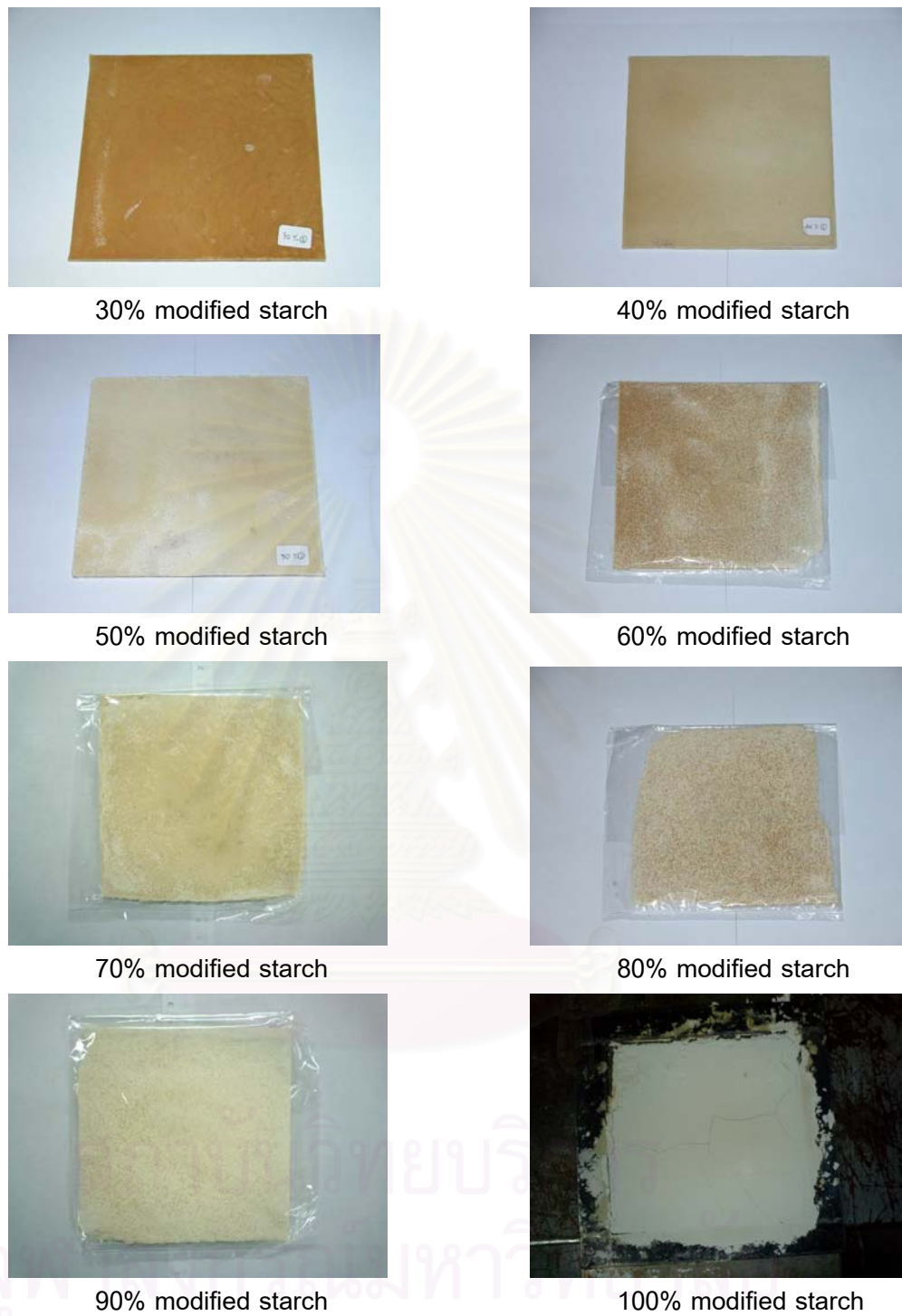


Figure 4.4 Surface of modified cassava starch/PP blends using the amount of modified cassava starch at 30-100%.

Sheets of polypropylene blended with unmodified cassava starch at 0%, 5%, 10%, 15%, 20%, 25%, 30%, 40%, 50%, 60%, 70%, 80%, 90% and 100% are shown in Figures 4.5 and 4.6.

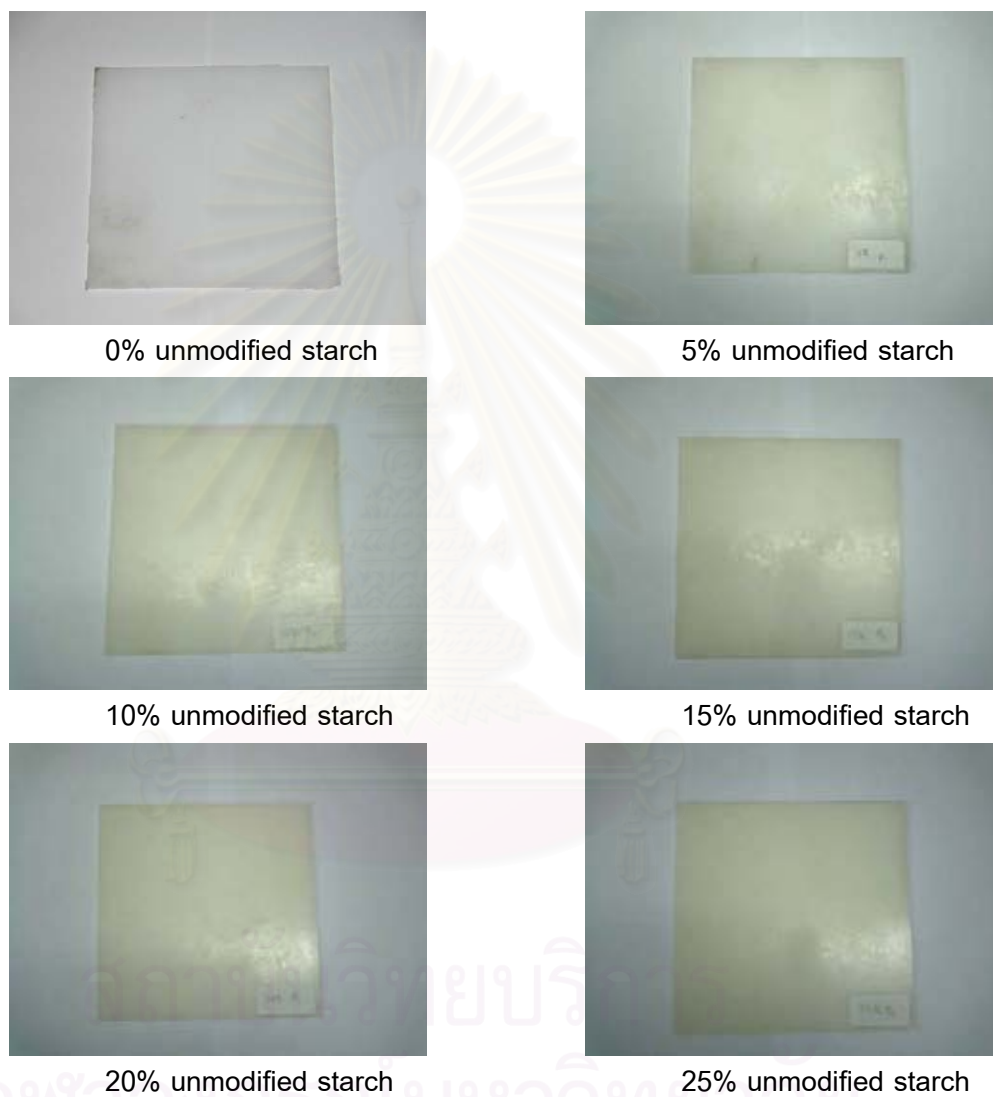


Figure 4.5 Surface of unmodified cassava starch/PP blends.
using the amount of modified cassava starch at 0-25%.

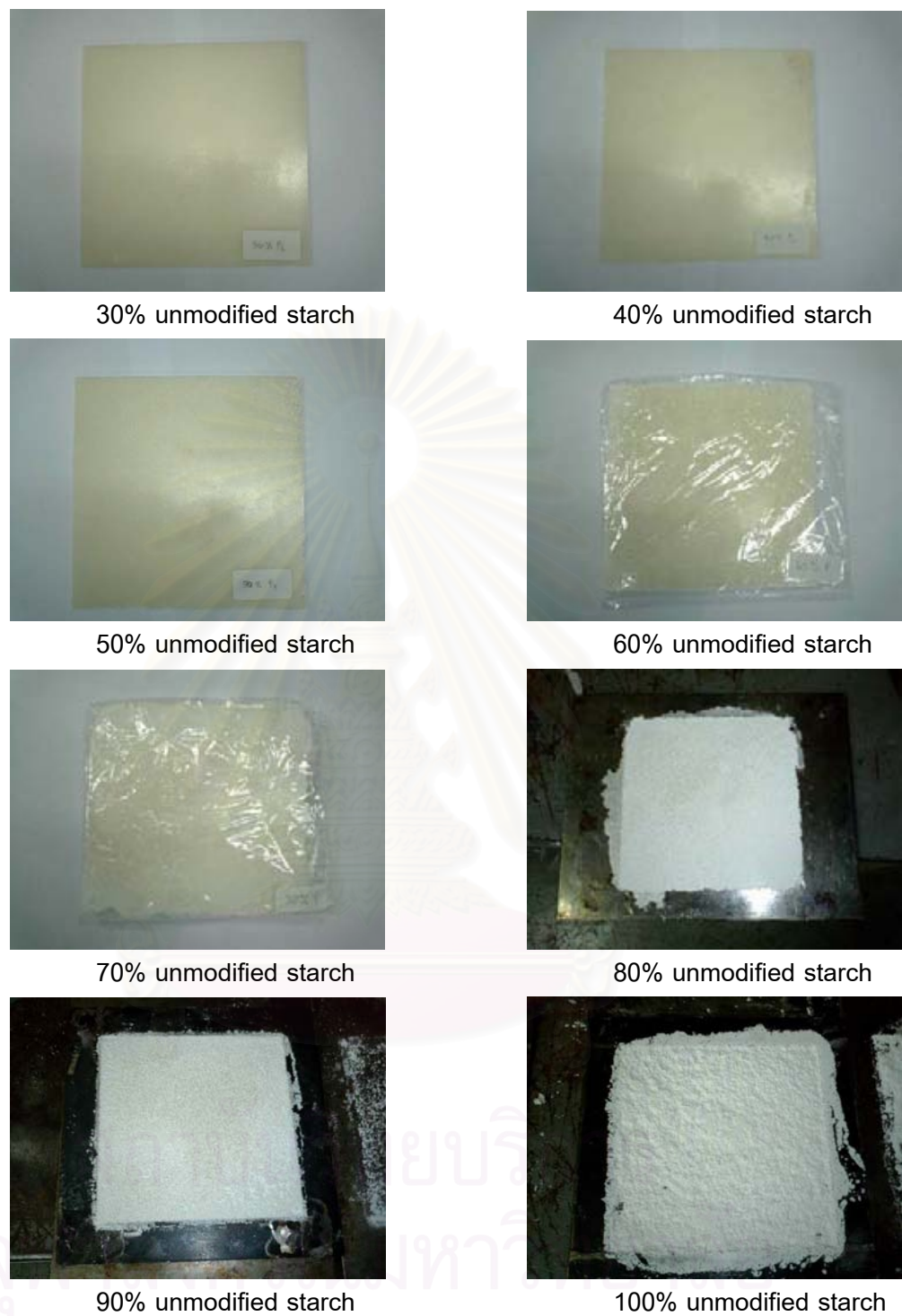


Figure 4.6 Surface of unmodified cassava starch/PP blends using the amount of modified cassava starch at 30-100%.

4.3 Mechanical Properties of polypropylene blended with cassava starch

4.3.1 Tensile properties

Three major properties including the tensile strength, the elongation at break and Young's modulus were investigated in tensile testing. Each of these three properties was plotted against cassava starch concentration to study the trends of mechanical properties with increasing starch concentrations. The results of both starch/PP blends were overlaid for direct comparison.

Figure 4.7 shows the effect of cassava starch concentration on tensile strength for both of the cassava starch. It can be seen that both of the cassava starch have a similar effect on tensile strength, with a peak of approximately 22 MPa occurring at 15% of unmodified starch. The tensile strength of the starch blends begin to deviate significantly at concentrations over 5% of starch. At 50% of starch, the modified starch blends has a tensile strength of 12 MPa compared to 10 MPa for the unmodified starch blend. Decreasing of tensile strength with increasing starch concentration as shown in this figure may be caused by the obstruction in PP crystallization due to the presence of starch molecules between PP molecules.

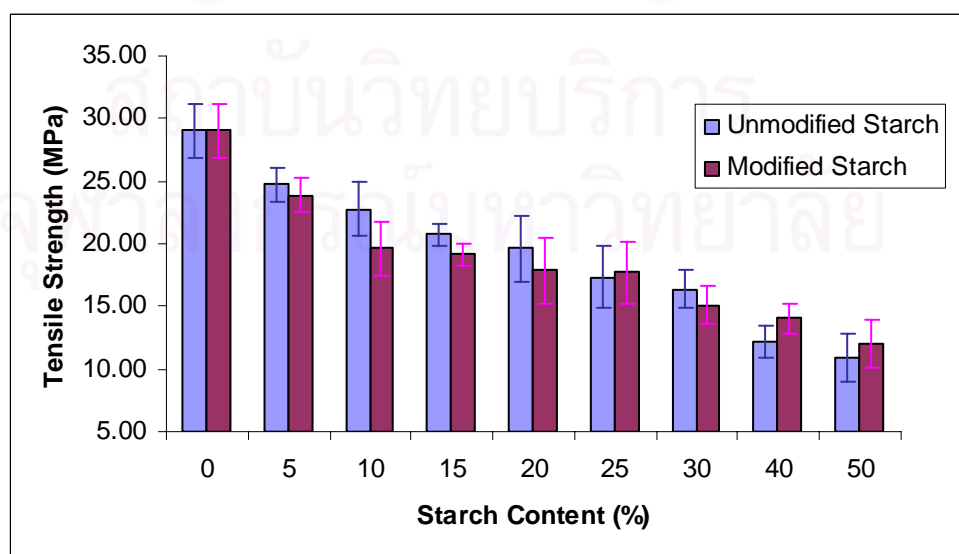
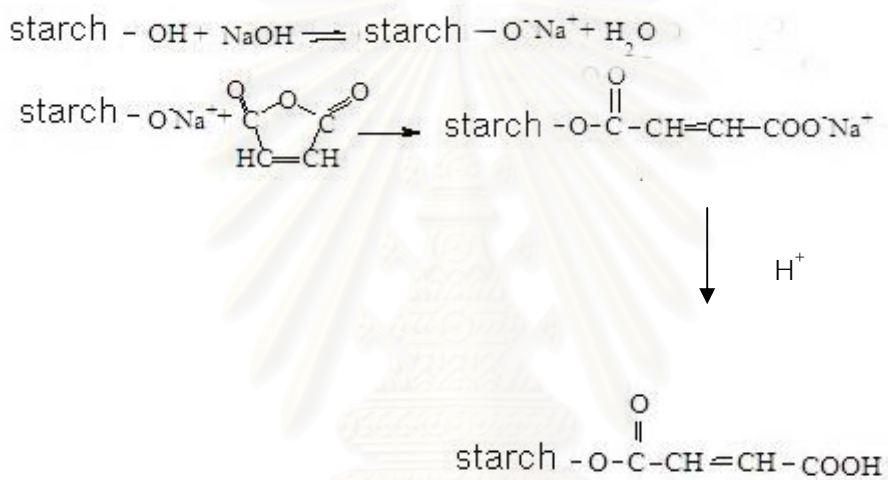
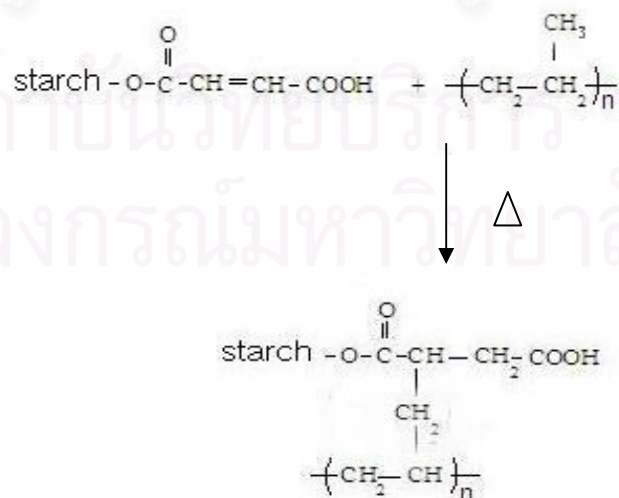


Figure 4.7 Tensile strength of starch/PP blends

Elongation at break for both starch blends are illustrated in Figure 4.8. Most of modified starch blends all have elongation at break lower than unmodified starch blends. This may be possibly due to the following reaction between PP and modified starch which results in the formation of crosslinks between the two components. Therefore, it is more difficult for PP and modified starch molecules to elongate after subjecting to tensile load. This also affects Young's modulus of starch/pp blends as shown in Figure 4.9.



After compression molding with PP.



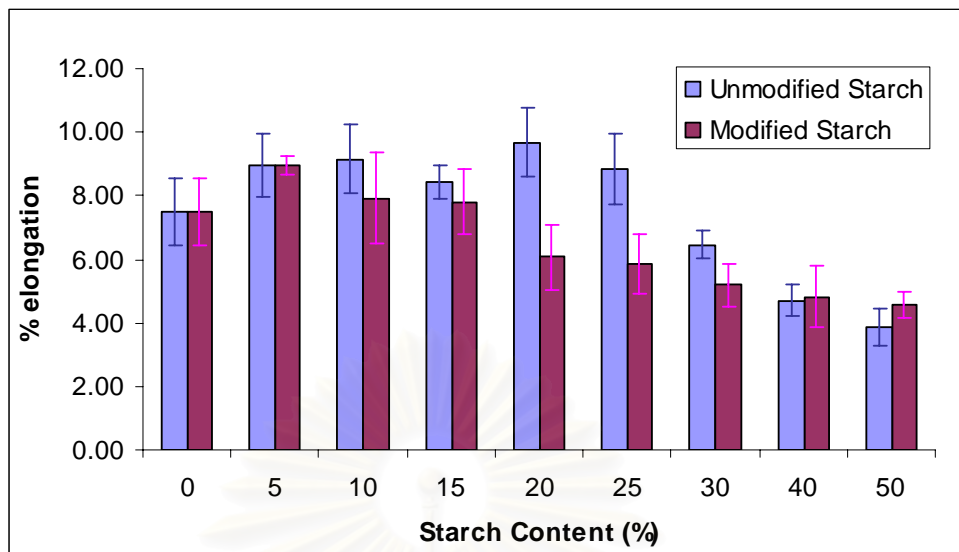


Figure 4.8 % Elongation at break of starch/PP blends.

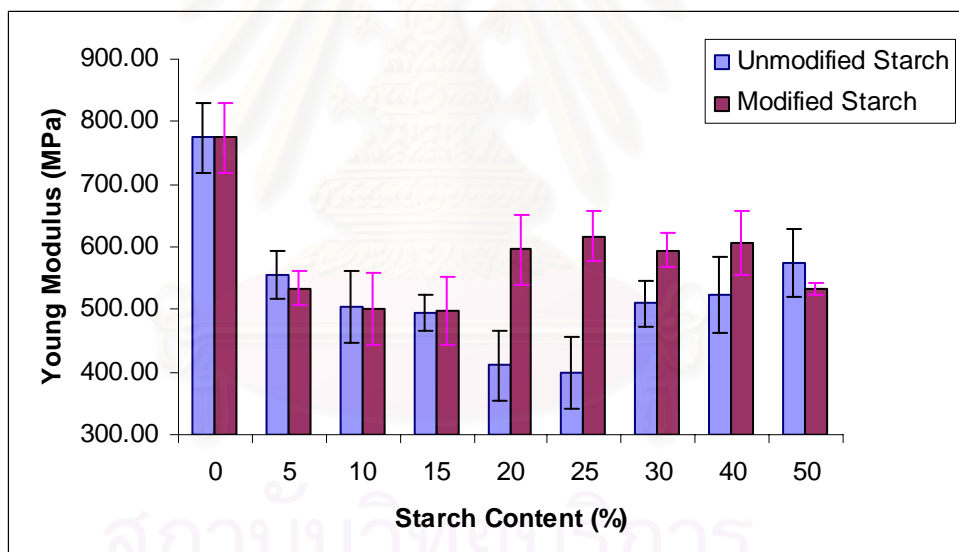


Figure 4.9 Young's modulus of starch/PP blends.

4.3.2 Flexural properties

Three major properties including the flexural strength, the deformation at maximum load and flexural modulus were investigated in flexural testing. Each of these three properties was plotted against cassava starch concentration to study the trends of

mechanical properties with increasing starch concentrations. The results of both starch/PP blends were overlaid for direct comparison.

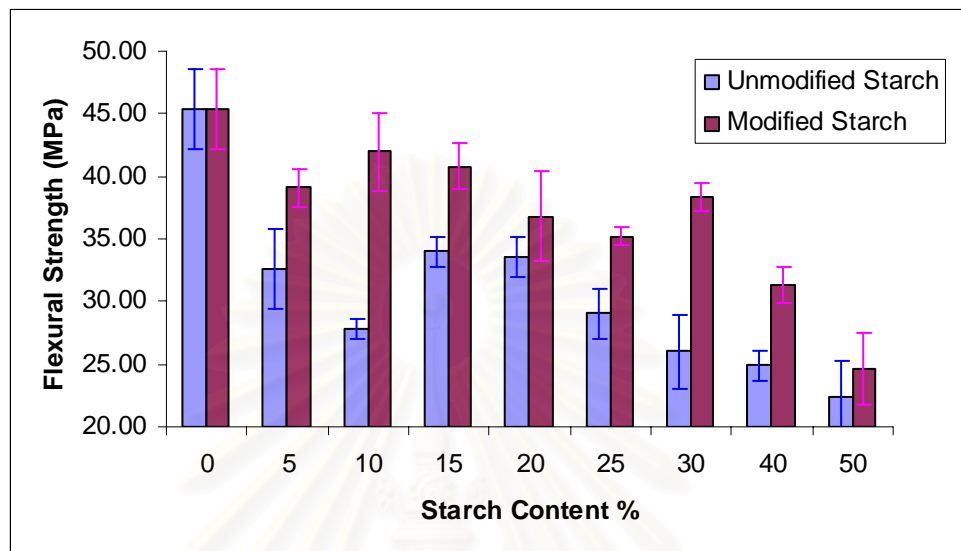


Figure 4.10 Flexural strength of starch/PP blends.

As can be seen in Figure 4.10, flexural strength of unmodified cassava starch/PP blends and modified cassava starch/PP blends decreases when the amount starch increases from 5 to 50% by weight due to the same effect as previously discussed for tensile strength. However, in this case, the effect of starch modification is clearly seen. All modified starch/PP blends exhibit higher flexural strength than the unmodified ones because of the presence of crosslinks between PP and modified starch molecules as previously shown. This also results in less deformation and higher flexural modulus of the former as shown in Figures 4.11 and 4.12, respectively.

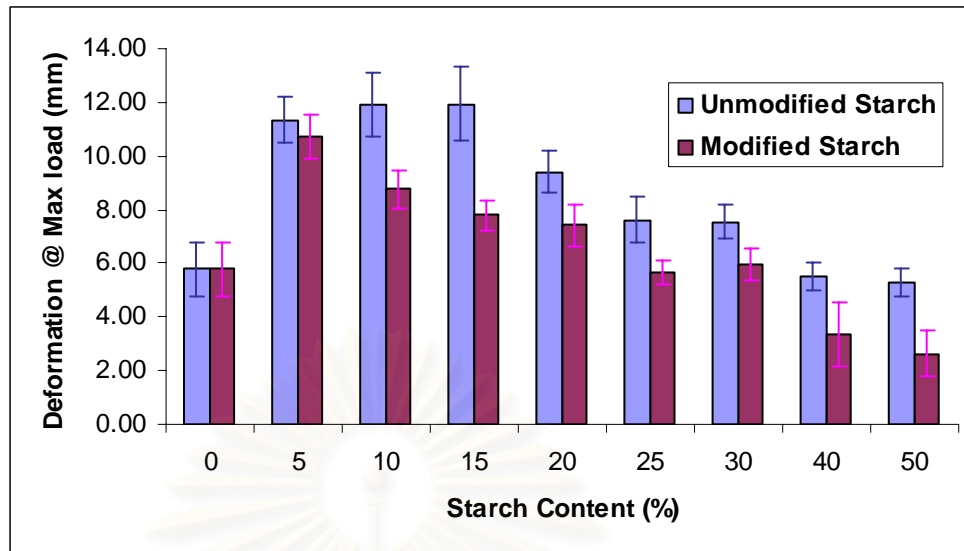


Figure 4.11 Deformation at maximum load of starch/PP blends.

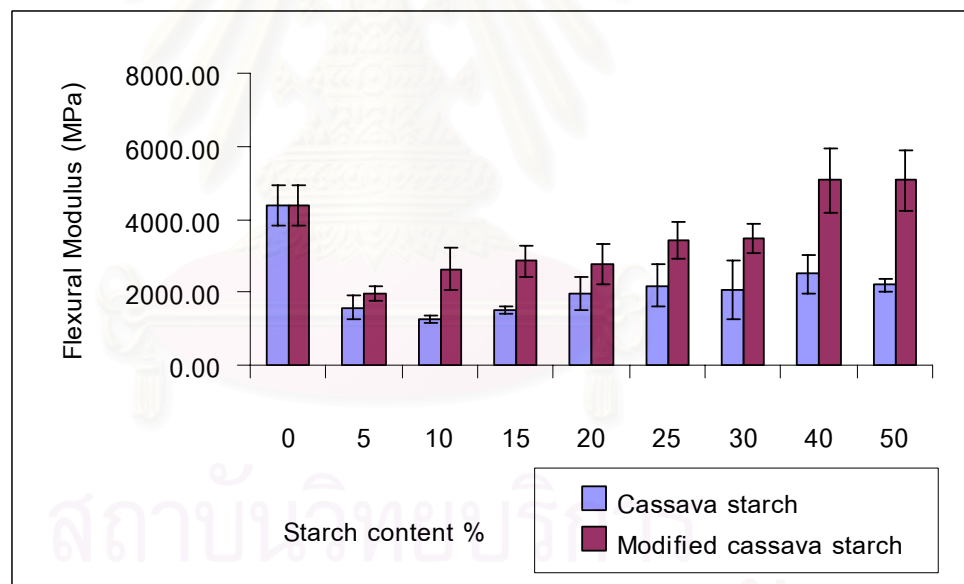


Figure 4.12 Flexural modulus of the starch/PP blends.

4.3.3 Impact strength

Impact strength of cassava starch/PP blends are shown in Figure 4.13.

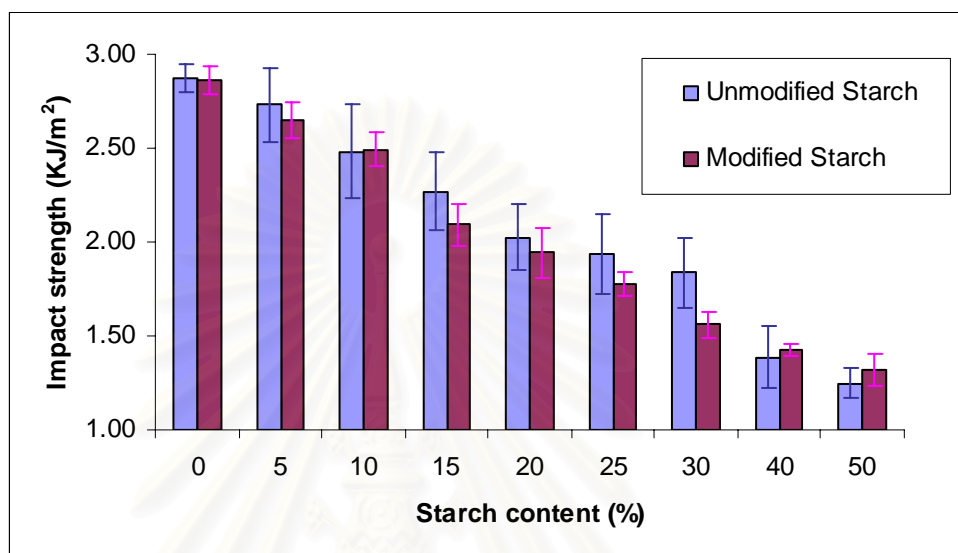


Figure 4.13 Impact strength of the starch/PP blends.

It can be seen from Figure 4.13 that impact strength of starch/PP blends decreases with increasing starch concentration. Even though the starch decrease the crystallinity of PP but starch itself is hard and brittle so the starch does not help absorbing the impact load. Therefore, as the amount of starch increases, the samples become more brittle.

4.4 Morphological studies of starch/PP blends

The impact fractured surface of starch/PP blends were studied by scanning electron microscope (SEM) at acceleration voltage of 15 KV. to compare the failure mechanism of the starch/PP blends specimens. The fractured surface of the sample was coated with gold before being scanned. The results are shown in Figures 4.14 to 4.20. It can be seen from all figures that modified starch/PP blends exhibits smoother fractured surface than the unmodified ones. This indicates possibly the melting of modified starch and better adhesion between PP and modified starch molecules.

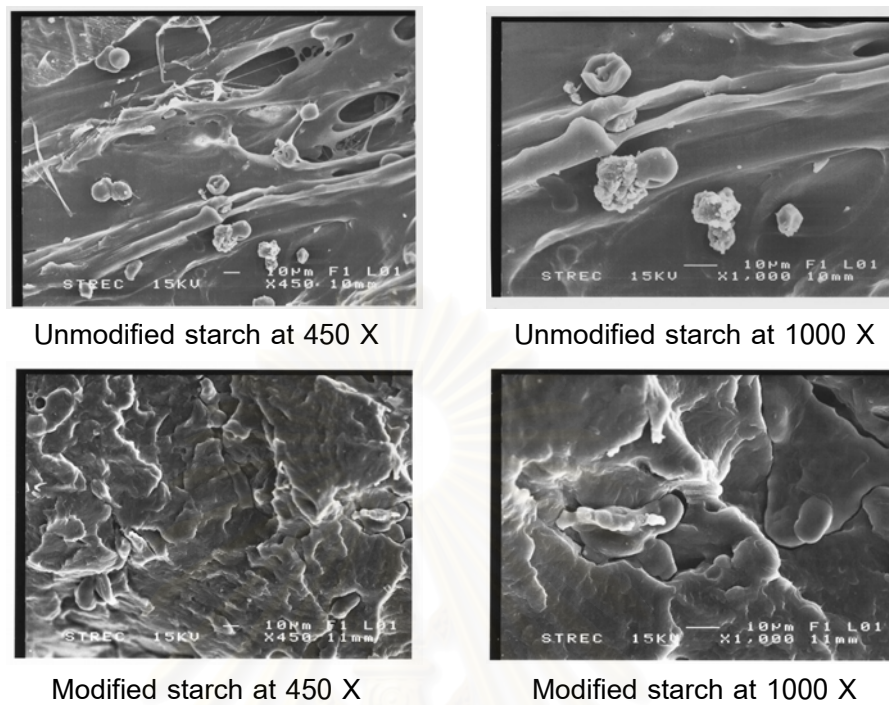


Figure 4.14 SEM micrographs of impact fractured surface of starch/PP blends at 5% starch.

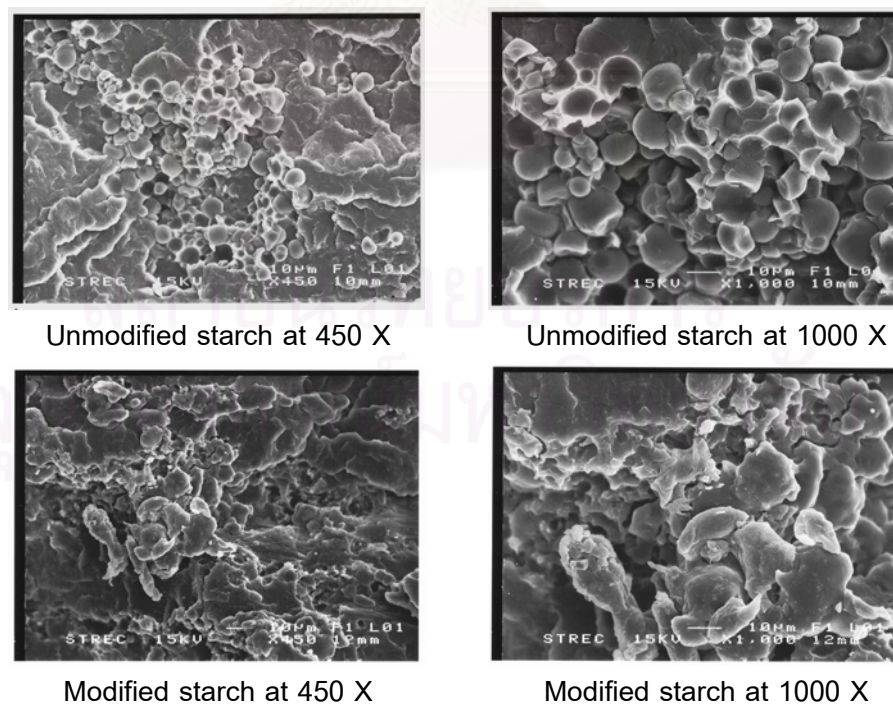


Figure 4.15 SEM micrographs of Impact impact fractured surface of starch/PP blends at 10% starch.

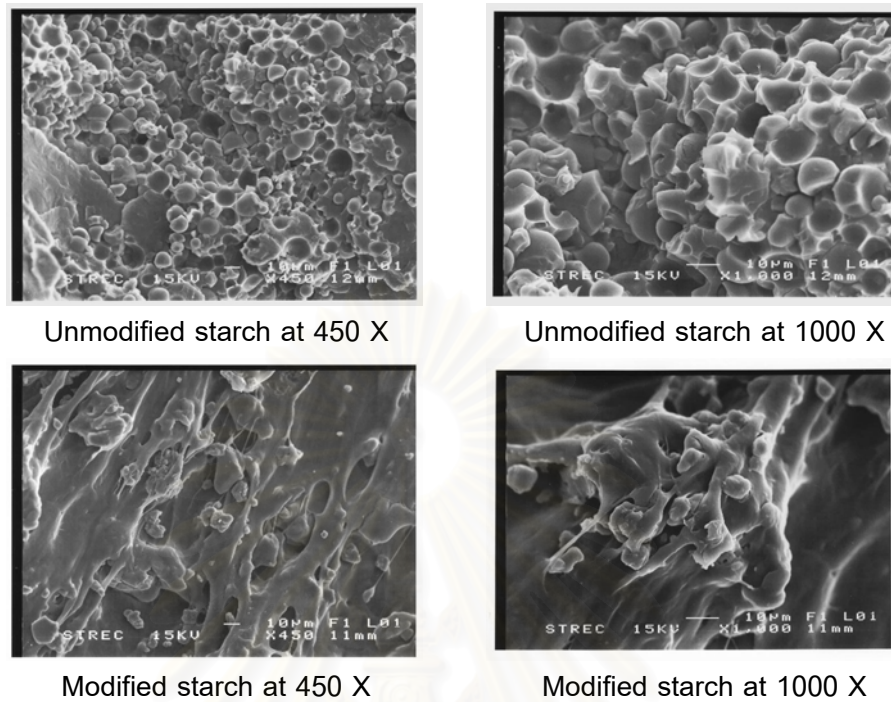


Figure 4.16 SEM micrographs of impact fractured surface of starch/PP blends at 15% starch.

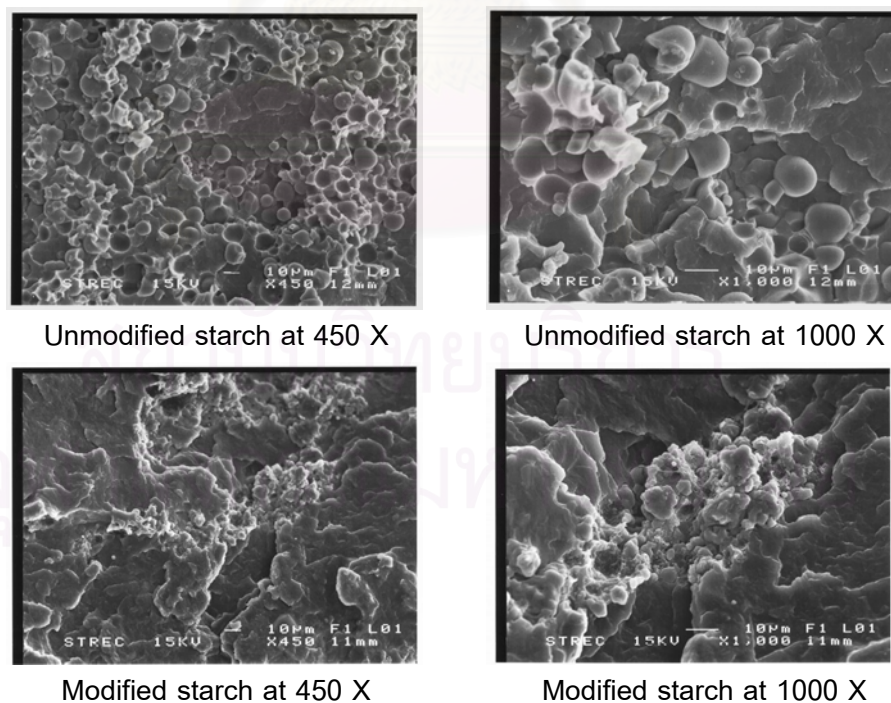


Figure 4.17 SEM micrographs of impact fractured surface of starch/PP blends at 20% starch.

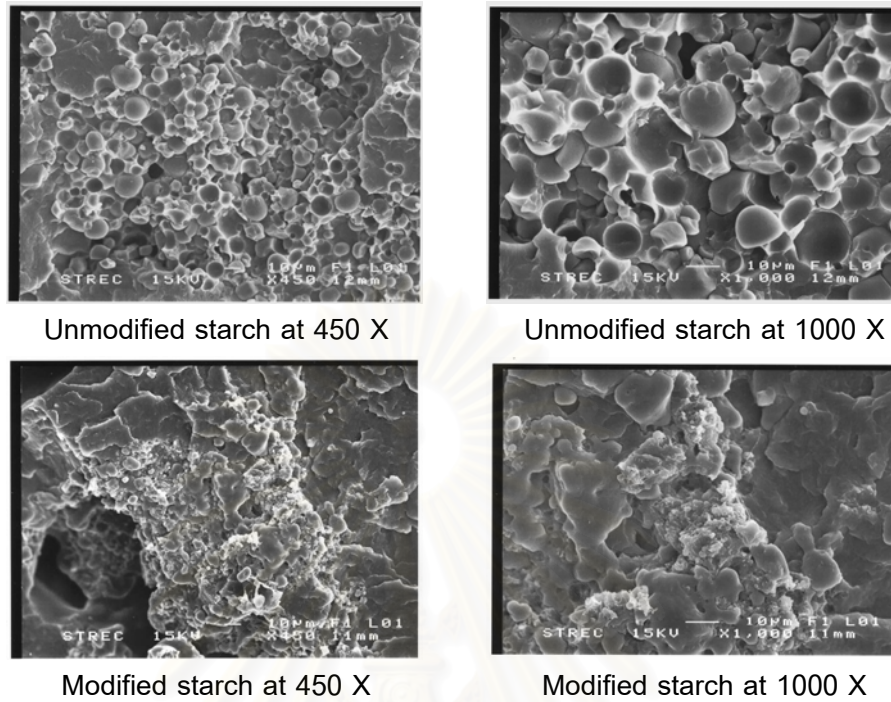


Figure 4.18 SEM micrographs of Impact impact fractured surface of starch/PP blends at 25% starch.

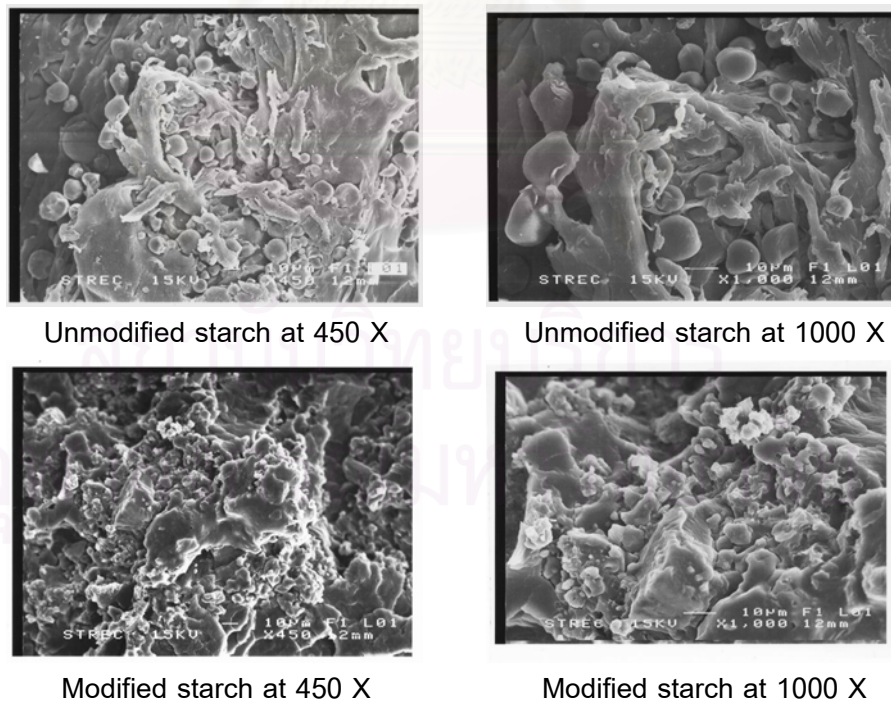


Figure 4.19 SEM micrographs of impact fractured surface of starch/PP blends at 30% starch.

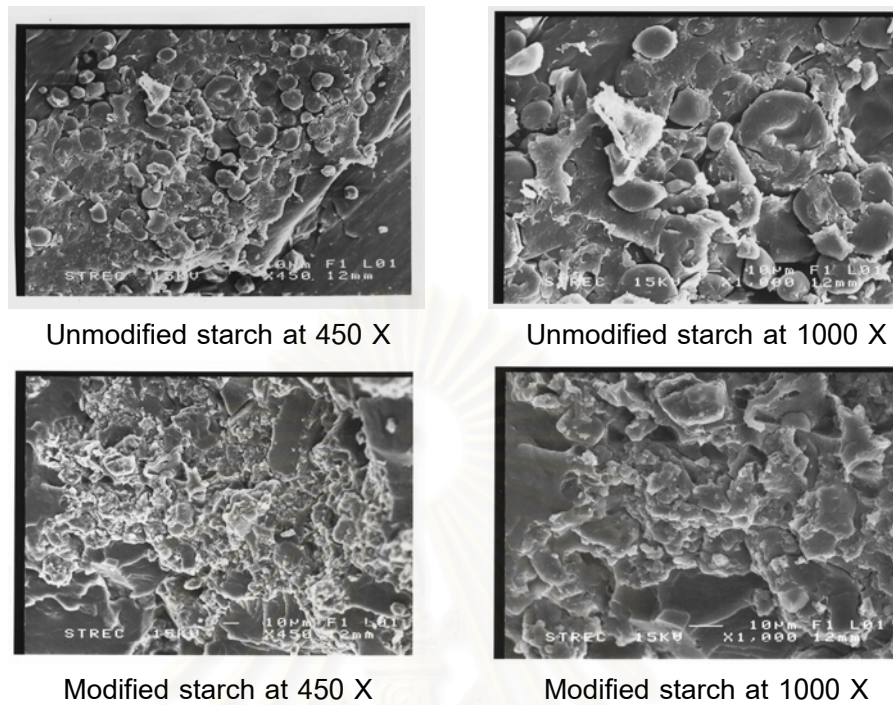


Figure 4.20 SEM micrographs of impact fractured surface of starch/PP blends at 40% starch.

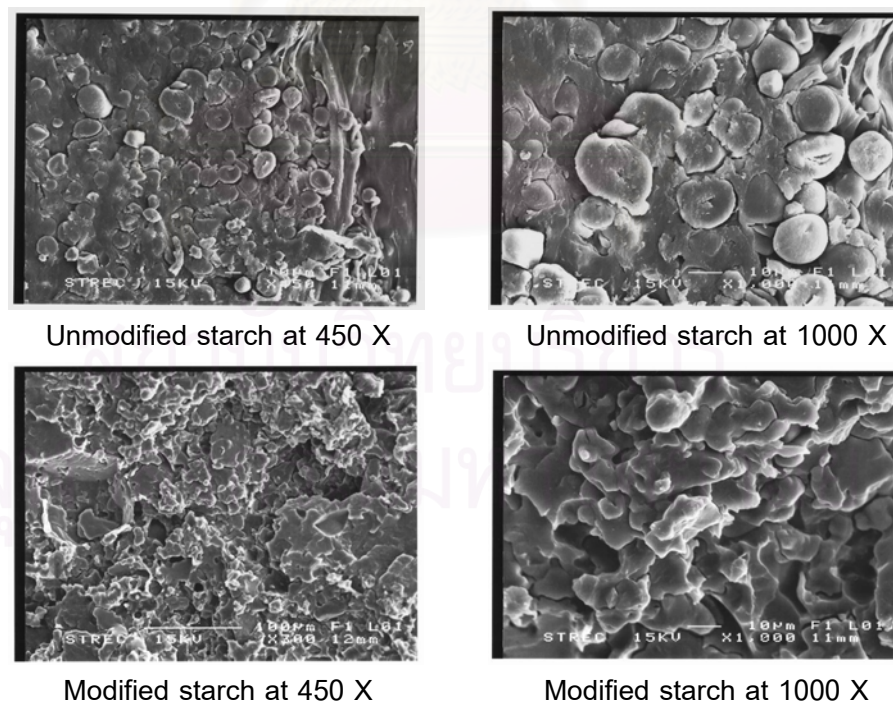


Figure 4.21 SEM micrographs of impact fractured surface of starch/PP blends at 50% starch.

4.5 Moisture absorption

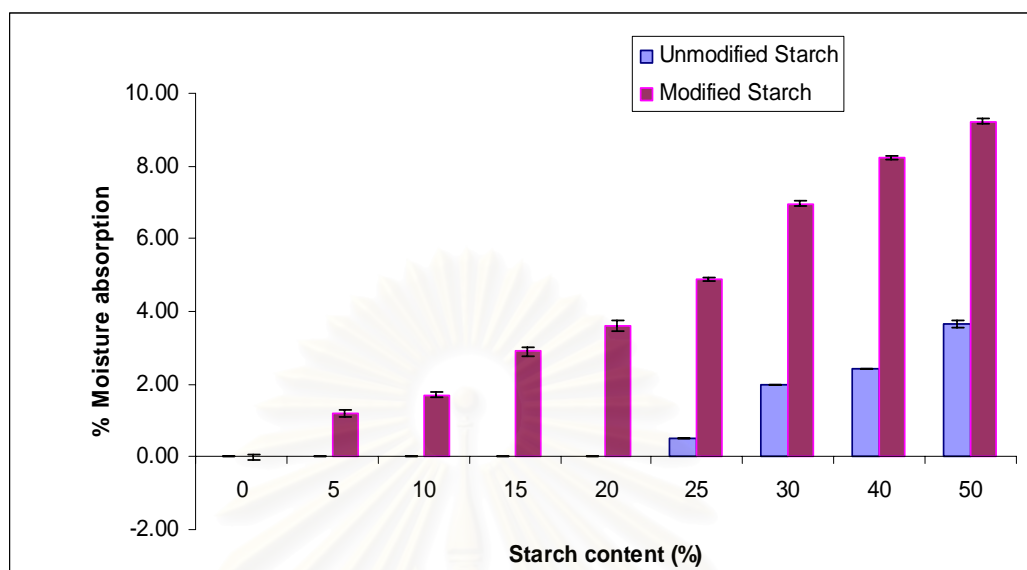


Figure 4.22 Moisture absorption of starch/PP blends.

It is clearly seen from Figure 4.22 that moisture absorption of starch/PP blends increases as starch concentration increases due to an increase in hydrophilic groups present in the starch. In addition, due to higher amounts of hydrophilic groups including hydroxyl, ester and carboxylic groups in modified starch compared to only hydroxyl groups in unmodified starch, moisture absorption of modified starch blends is higher than that of the unmodified ones.

4.6 Biodegradability

The results of biodegradability are in good agreement with those of moisture adsorption as shown in Figures 4.23 and 4.24. Biodegradability indicating by %weight loss of starch/PP blends increases as starch concentration increases. Since moisture is necessary element for degradation mechanism by microorganism; therefore, increasing of moisture absorption can increase the rate of biodegradation. The samples of starch/PP blends after biodegradable test are shown in Figures 4.25 to 4.40.

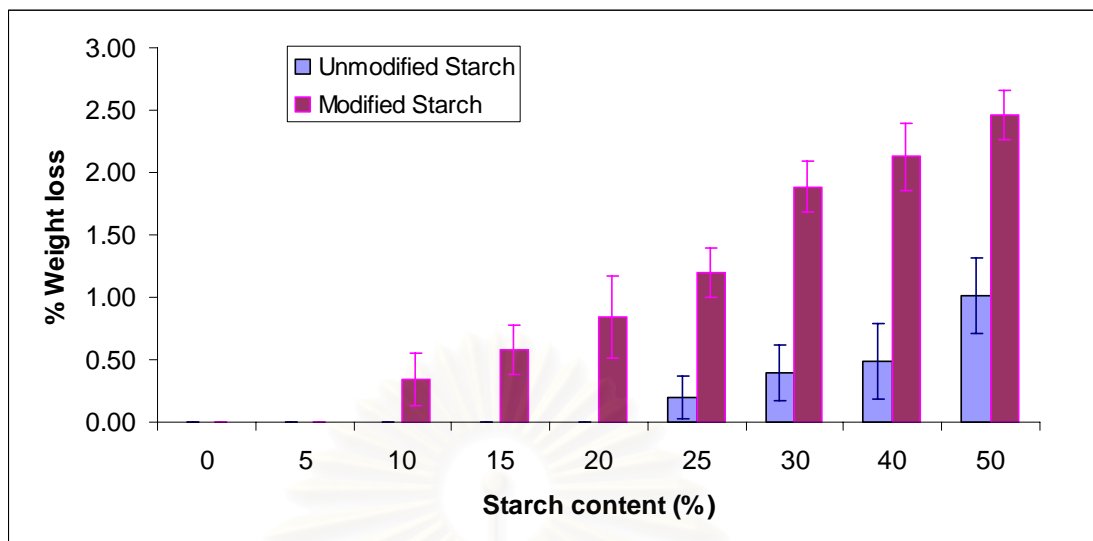


Figure 4.23 %weight loss of the starch/PP blends after 2 weeks test.

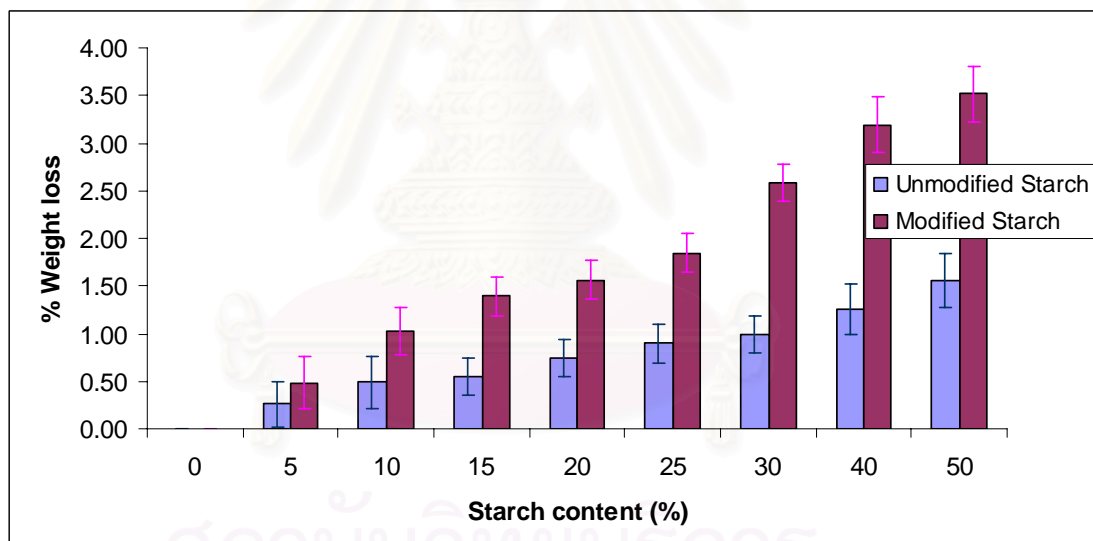


Figure 4.24 %weight loss of the starch/PP blends after 3 weeks test.

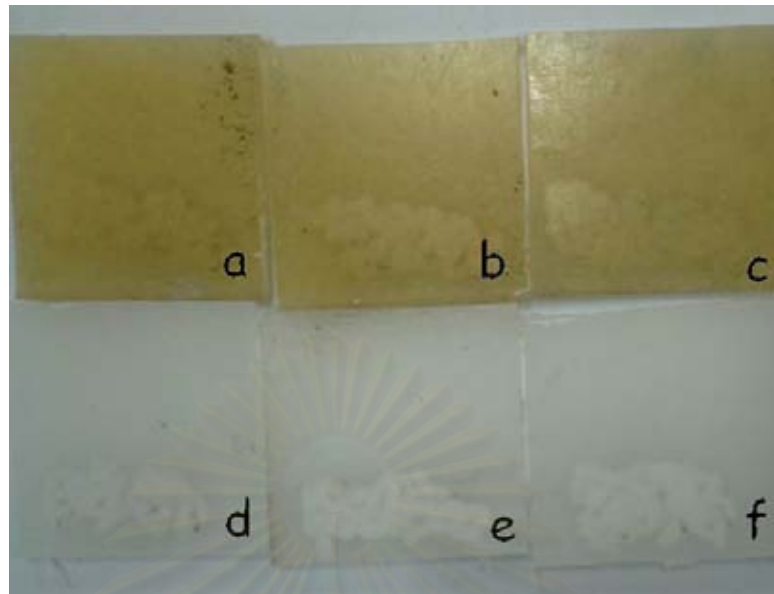


Figure 4.25 The specimens of 5% starch/PP blends before test.

- (a), (b) and (c) modified cassava starch
- (d), (e), and (f) unmodified cassava starch

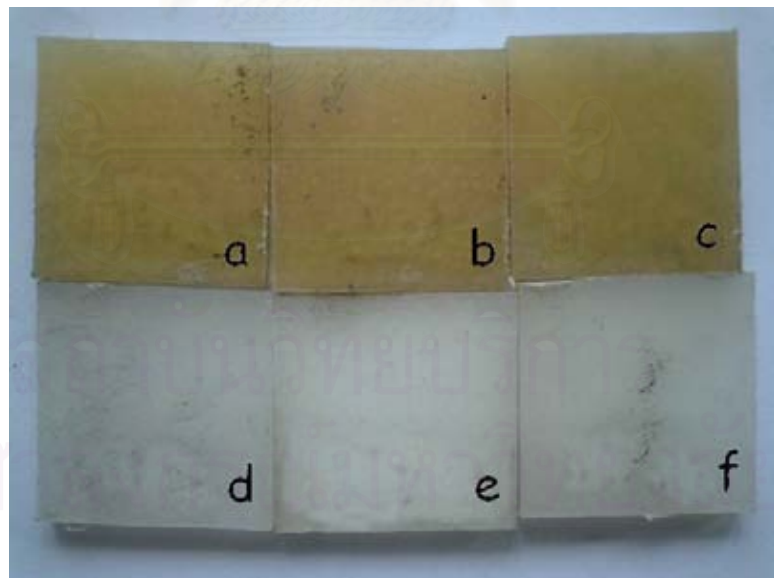


Figure 4.26 The specimens of 5% starch/PP blends after 3 weeks test.

- (a), (b) and (c) modified cassava starch
- (d), (e), and (f) unmodified cassava starch

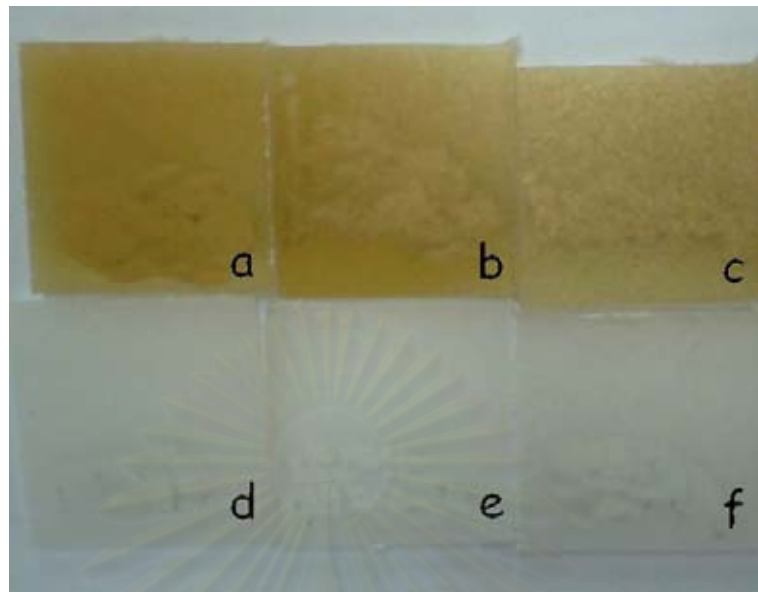


Figure 4.27 The specimens of 10% starch/PP blends before test.

- (a), (b) and (c) modified cassava starch
- (d), (e), and (f) unmodified cassava starch

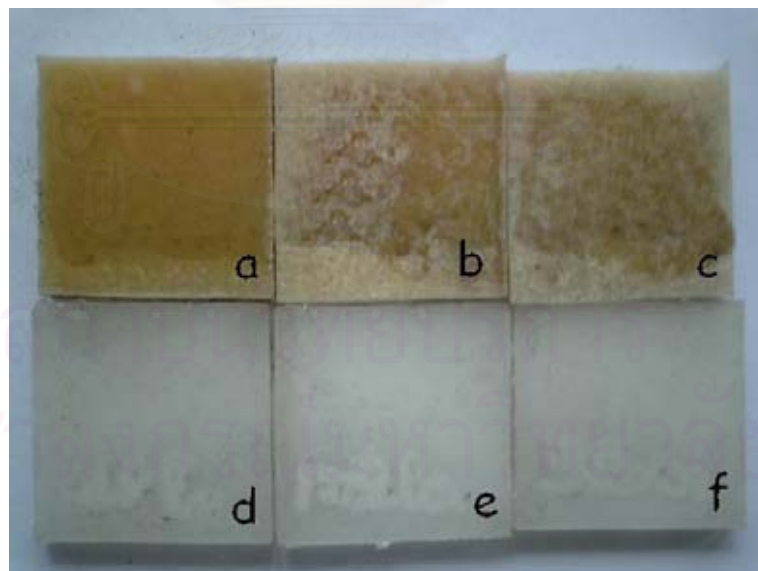


Figure 4.28 The specimens of 10% starch/PP blends after 3 weeks test.

- (a), (b) and (c) modified cassava starch
- (d), (e), and (f) unmodified cassava starch

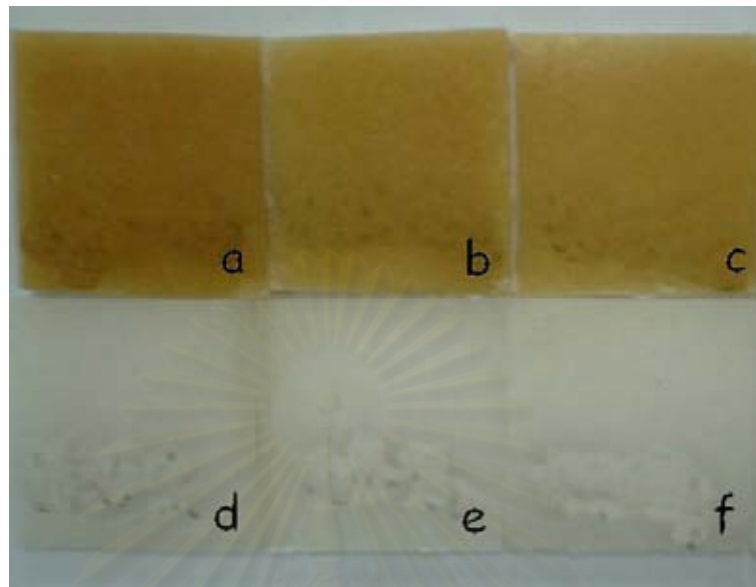


Figure 4.29 The specimens of 15% starch/PP blends before test.

- (a), (b) and (c) modified cassava starch
 (d), (e), and (f) unmodified cassava starch

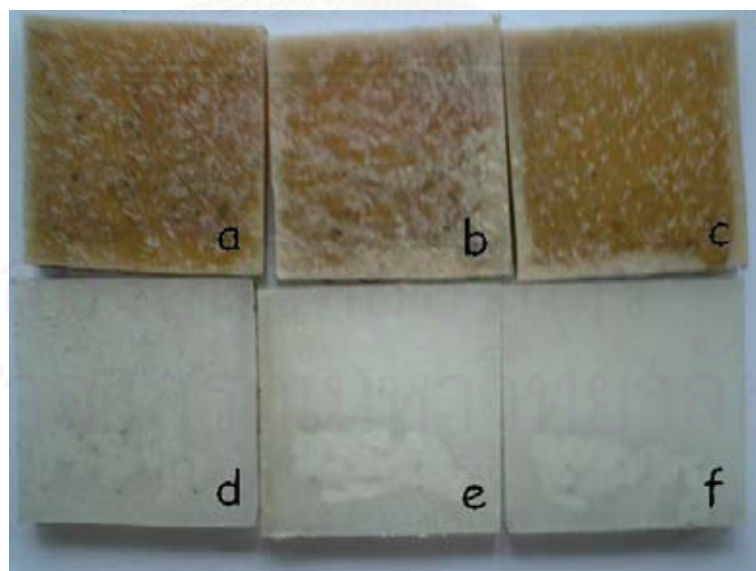


Figure 4.30 The specimens of 15% starch/PP blends after 3 weeks test.

- (a), (b) and (c) modified cassava starch
 (d), (e), and (f) unmodified cassava starch

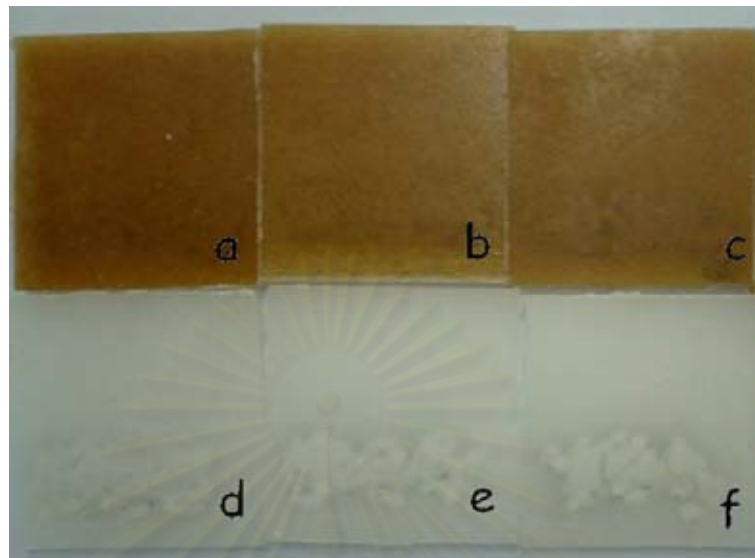


Figure 4.31 The specimens of 20% starch/PP blends before test.

- (a), (b) and (c) modified cassava starch
- (d), (e), and (f) unmodified cassava starch

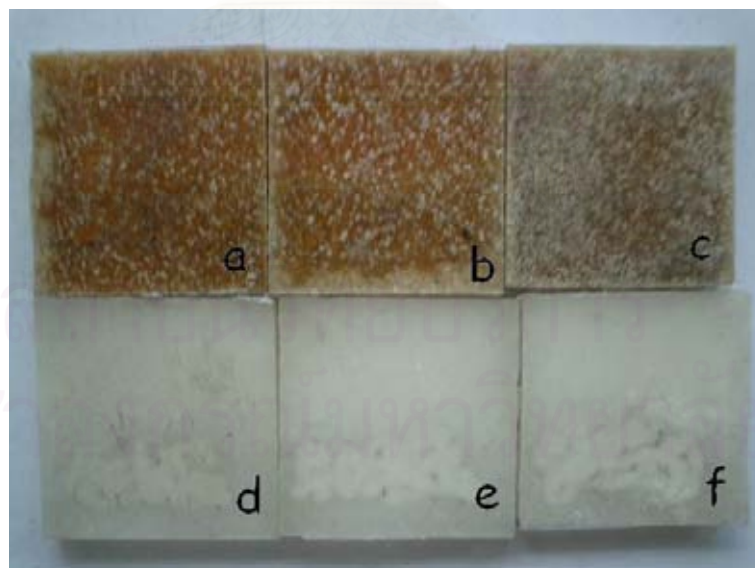


Figure 4.32 The specimens of 20% starch/PP blends after 3 weeks test.

- (a), (b) and (c) modified cassava starch
- (d), (e), and (f) unmodified cassava starch

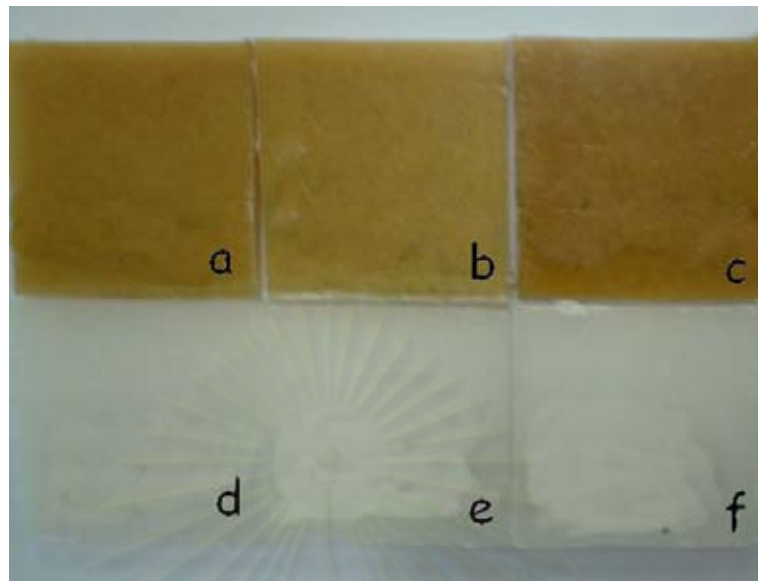


Figure 4.33 The specimens of 25% starch/PP blends before test.

- (a), (b) and (c) modified cassava starch
- (d), (e), and (f) unmodified cassava starch

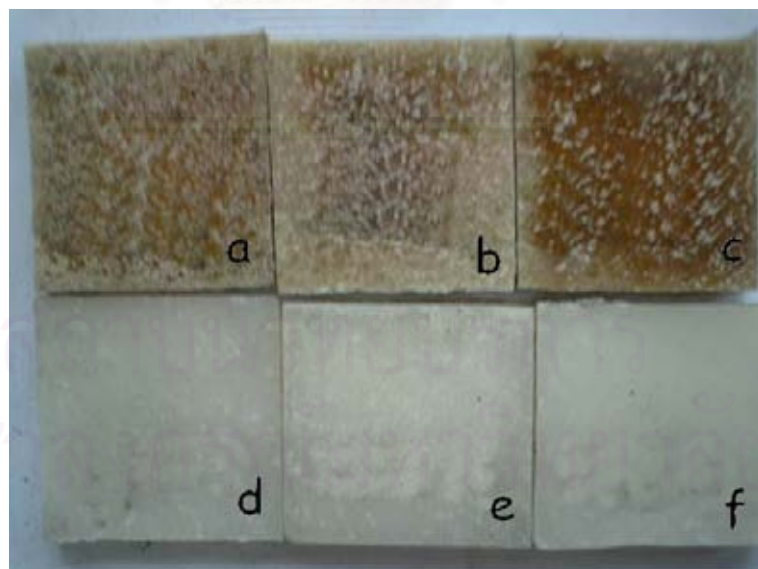


Figure 4.34 The specimens of 25% starch/PP blends after 3 weeks test.

- (a), (b) and (c) modified cassava starch
- (d), (e), and (f) unmodified cassava starch

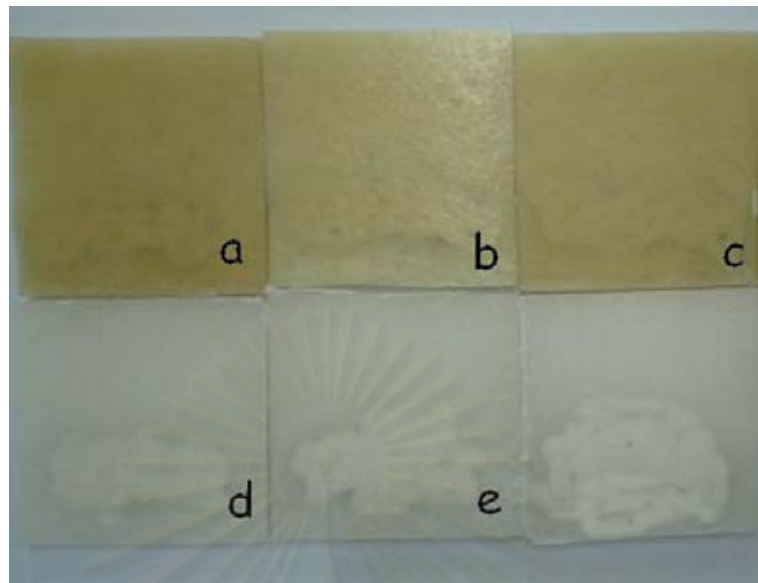


Figure 4.35 The specimens of 30% starch/PP blends before test.

- (a), (b) and (c) modified cassava starch
- (d), (e), and (f) unmodified cassava starch

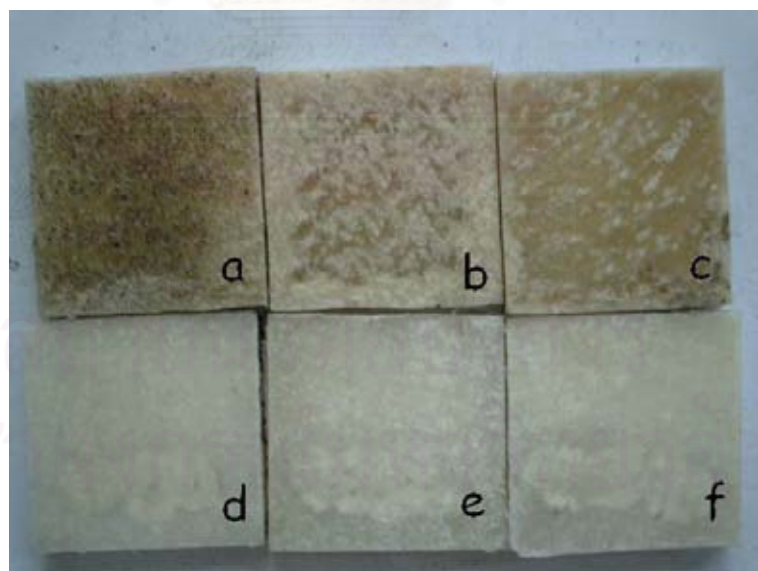


Figure 4.36 The specimens of 30% starch/PP blends after 3 weeks test.

- (a), (b) and (c) modified cassava starch
- (d), (e), and (f) unmodified cassava starch

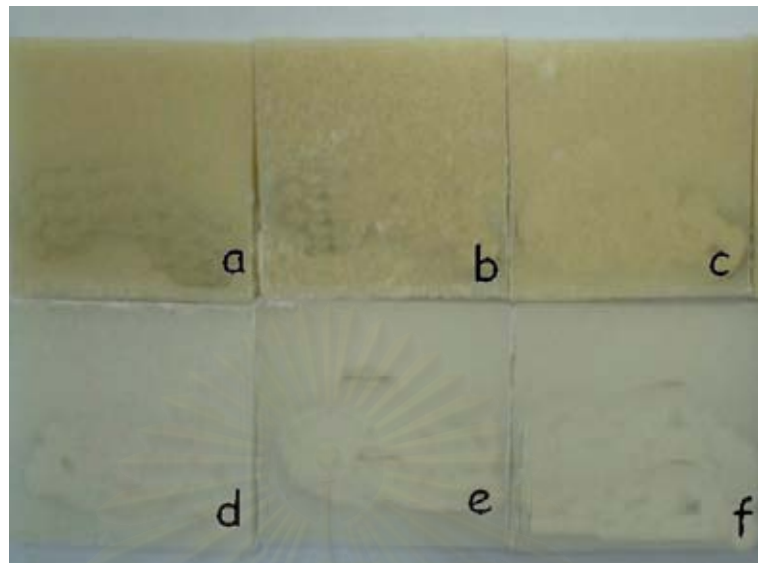


Figure 4.37 The specimens of 40% starch/PP blends before test.

- (a), (b) and (c) modified cassava starch
- (d), (e), and (f) unmodified cassava starch

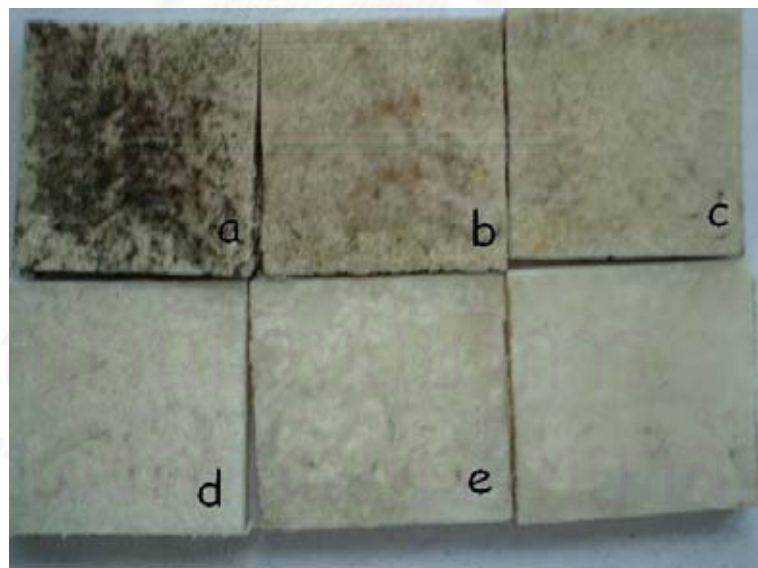


Figure 4.38 The specimens of 40% starch/PP blends after 3 weeks test.

- (a), (b) and (c) modified cassava starch
- (d), (e), and (f) unmodified cassava starch

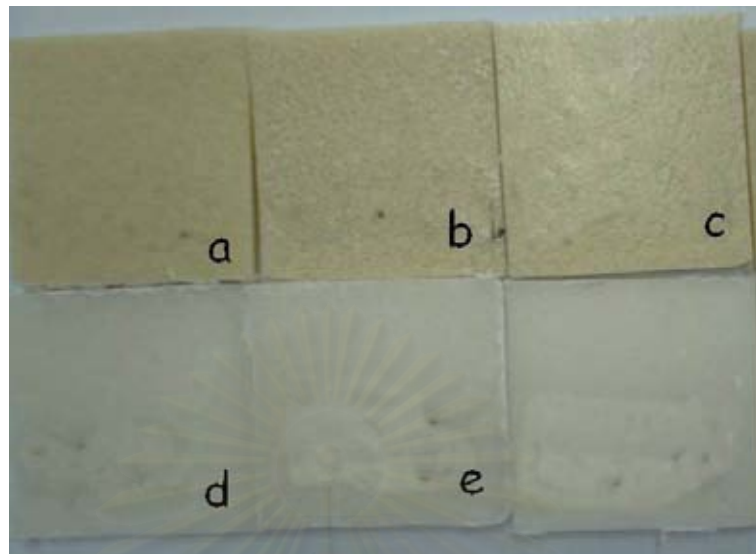


Figure 4.39 The specimens of 50% starch/PP blends before test.

- (a), (b) and (c) modified cassava starch
 (d), (e), and (f) unmodified cassava starch

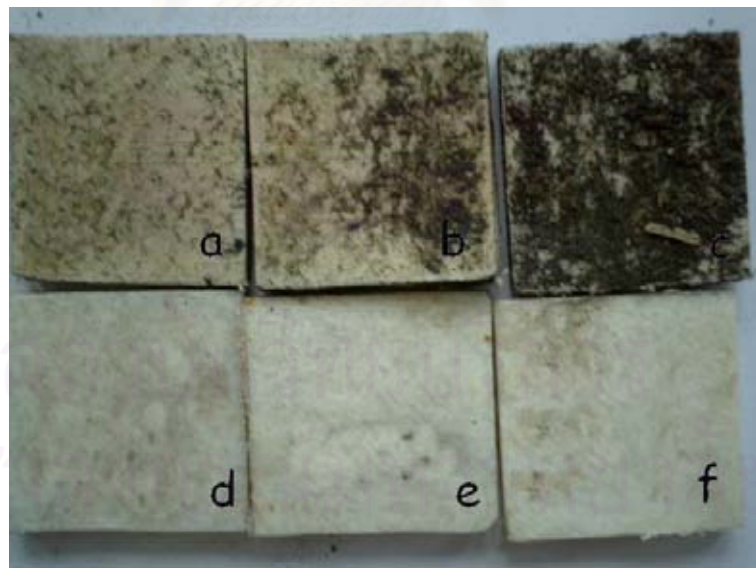


Figure 4.40 The specimens of 50% starch/PP blends after 3 weeks test.

- (a), (b) and (c) modified cassava starch
 (d), (e), and (f) unmodified cassava starch

CHAPTER V

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Investigations of the effects of starch modification and the amount of starch on the mechanical properties, moisture absorption and biodegradability of starch/PP blends resulted in several conclusions as follows:

1. Tensile properties of starch/PP blends decreased with the increasing amount of starch and modified starch/PP blends. The blends from modified starch also exhibited better tensile properties than the unmodified ones.
2. Flexural properties of starch/PP blends decreased with the increasing amount of starch and modified starch/PP blends. The blends from modified starch also exhibited better tensile properties than the unmodified ones.
3. Impact strength decreased with the increasing amount of starch this treated overshadowed the effect of the starch modification.
4. Morphological studies by SEM suggested that there was better adhesion between modified starch and PP molecules which resulted in superior mechanical properties of modified starch/PP blends.
5. %Moisture absorption and biodegradability of starch/PP blends increased with increasing the amount of starch. Due to higher amounts of hydrophilic groups present in modified cassava starch molecules, %moisture absorption and biodegradability of modified starch/PP blends were higher than those of unmodified ones.

From above results, it can be concluded that biodegradable plastics with better mechanical properties can be prepared from maleic anhydride-modified cassava starch than from the unmodified one.

5.2 Recommendations

From the above results, it is recommended that the further experiments can be done by changing the modifiers for starch modification or the type of plastics blended with modified cassava starch.



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APPENDICES

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Experimental Data

1. Polypropylene (PP)

Table A 1 Experimental data of tensile properties of the polypropylene (0%starch)

Sample	Tensile strength (MPa)	Young's modulus (MPa)	% Elongation
1	21.87	722.24	6.28
2	24.25	742.25	6.53
3	32.79	740.90	8.65
4	32.79	851.76	7.70
5	32.84	814.89	8.06
Mean	29.07	774.41	7.48
SD.	5.15	55.91	1.07

Table A 2 Experimental data of flexural properties of the PP (0%starch)

Sample	Flexural strength (MPa)	Flexural's modulus (MPa)	Deformation at max load (mm)
1	42.08	5122.72	4.38
2	49.79	3814.21	6.96
3	47.35	4119.09	6.24
4	43.19	4726.94	5.15
5	44.25	4036.03	6.14
Mean	45.33	4363.80	5.11
SD.	3.17	542.49	1.01

Table A 3 Experimental data of impact strength of the PP (0%starch)

Sample	Impact strength (KJ/m ²)
1	2.91
2	2.75
3	2.84
4	2.94
5	2.91
Mean	2.87
SD.	0.07

Table A 4 Experimental data of moisture absorption and % weight loss of PP (0%starch)

Sample	% Moisture absorption	% weight loss 2 weeks	% weight loss 3 weeks
1	0.00	0.00	0.00
2	0.00	0.00	0.00
3	0.00	0.00	0.00
4	0.00	0.00	0.00
5	0.00	0.00	0.00
Mean	0.00	0.00	0.00
SD.	0.00	0.00	0.00

2. Unmodified cassava starch/PP blends

Table A 5 Experimental data of tensile properties at 5% of starch

Sample	Tensile strength (MPa)	Young's modulus (MPa)	% Elongation
1	25.38	556.77	9.12
2	26.31	527.54	9.98
3	22.06	586.03	7.53
4	24.68	506.78	9.74
5	25.14	597.15	8.42
Mean	24.72	554.85	8.96
SD.	1.16	38.15	1.00

Table A 6 Experimental data of flexural properties at 5% of starch

Sample	Flexural strength (MPa)	Flexural's modulus (MPa)	Deformation at max load (mm)
1	29.62	1247.78	12.66
2	36.55	1497.65	13.01
3	29.09	1293.63	12.32
4	33.42	1878.62	9.49
5	34.16	1946.39	9.28
Mean	32.57	1572.81	11.35
SD.	3.16	324.93	1.81

Table A 7 Experimental data of impact strength at 5% of starch

Sample	Impact strength (KJ/m ²)
1	2.22
2	2.84
3	2.75
4	2.97
5	2.88
Mean	2.73
SD.	0.30

Table A 8 Experimental data of moisture absorption and % weight loss at 5% of starch

Sample	% Moisture absorption	% weight loss 2 weeks	% weight loss 3 weeks
	1	0.00	0.00
2	0.00	0.00	0.65
3	0.00	0.00	0.00
4	0.00	0.00	0.00
5	0.00	0.00	0.00
Mean	0.00	0.00	0.26
SD.	0.00	0.00	0.36

Table A 9 Experimental data of tensile properties at 10% of starch

Sample	Tensile strength (MPa)	Young's modulus (MPa)	% Elongation
1	23.03	492.62	9.35
2	23.54	503.43	9.35
3	22.78	560.77	8.12
4	22.26	548.03	8.12
5	22.20	413.29	10.74
Mean	22.76	503.63	9.14
SD.	0.56	58.14	1.09

Table A 10 Experimental data of flexural strength at 10% of starch

Sample	Flexural strength (MPa)	Flexural's modulus (MPa)	Deformation at max load (mm)
1	29.00	1251.39	12.36
2	26.88	1112.05	12.89
3	27.83	1287.59	11.43
4	28.22	1355.86	11.10
5	27.44	1245.35	11.75
Mean	27.87	1250.45	11.91
SD.	0.80	88.98	1.72

Table A 11 Experimental data of impact strength at 10% of starch

Sample	Impact strength (KJ/m ²)
1	2.91
2	2.66
3	1.97
4	2.53
5	2.34
Mean	2.48
SD.	0.35

Table A 12 Experimental data of moisture absorption and % weight loss at 10% of starch

Sample	% Moisture absorption	% weight loss 2 weeks	% weight loss 3 weeks
1	0.00	0.00	0.00
2	0.00	0.00	0.61
3	0.00	0.00	0.62
4	0.00	0.00	0.61
5	0.00	0.00	0.62
Mean	0.00	0.00	0.49
SD.	0.00	0.00	0.27

Table A 13 Experimental data of tensile properties at 15% of starch

Sample	Tensile strength (MPa)	Young's modulus (MPa)	% Elongation
1	20.35	522.06	7.80
2	20.96	521.54	8.04
3	21.65	488.75	8.86
4	20.21	485.82	8.32
5	20.59	455.39	9.04
Mean	20.75	494.71	8.41
SD.	0.58	27.91	0.53

Table A 14 Experimental data of flexural strength at 15% of starch

Sample	Flexural strength (MPa)	Flexural's modulus (MPa)	Deformation at max load (mm)
1	35.71	1675.19	11.37
2	34.20	1417.91	12.86
3	32.35	1473.10	11.71
4	34.09	1516.85	11.98
5	33.64	1534.57	11.75
Mean	34.00	1523.53	11.93
SD.	1.21	96.00	0.56

Table A 15 Experimental data of impact strength at 15% of starch

Sample	Impact strength (KJ/m ²)
1	2.03
2	2.03
3	2.28
4	2.78
5	2.22
Mean	2.27
SD.	0.31

Table A 16 Experimental data of moisture absorption and % weight loss at 15% of starch

Sample	% Moisture absorption	% weight loss at 2 weeks	% weight loss at 3 weeks
1	0.00	0.00	0.55
2	0.00	0.00	0.55
3	0.00	0.00	0.54
4	0.00	0.00	0.55
5	0.00	0.00	0.54
Mean	0.00	0.00	0.55
SD.	0.00	0.00	0.01

Table A 17 Experimental data of tensile properties at 20% of starch

Sample	Tensile strength (MPa)	Young's modulus (MPa)	% Elongation
1	20.99	498.63	8.42
2	19.45	391.35	9.94
3	19.88	396.81	10.02
4	18.47	417.77	8.84
5	19.45	349.39	11.14
Mean	19.65	410.79	9.67
SD.	0.91	55.02	1.07

Table A 18 Experimental data of flexural properties at 20% of starch

Sample	Flexural strength (MPa)	Flexural's modulus (MPa)	Deformation at max load (mm)
1	35.87	2162.78	8.85
2	32.30	1635.21	10.53
3	31.91	1477.05	11.52
4	34.14	2629.41	6.81
5	33.35	1922.69	9.24
Mean	33.51	1965.43	9.39
SD.	1.58	455.30	1.79

Table A 19 Experimental data of impact strength at 20% of starch

Sample	Impact strength (KJ/m ²)
1	2.09
2	1.81
3	1.94
4	2.28
5	2.20
Mean	2.03
SD.	0.31

Table A 20 Experimental data of moisture absorption and % weight loss at 20% of starch

Sample	% Moisture absorption	% weight loss 2 weeks	% weight loss 3 weeks
1	0.00	0.00	0.53
2	0.00	0.00	1.07
3	0.00	0.00	0.53
4	0.00	0.00	1.05
5	0.00	0.00	0.53
Mean	0.00	0.00	0.74
SD.	0.00	0.00	0.29

Table A 21 Experimental data of tensile properties at 25% of starch

Sample	Tensile strength (MPa)	Young's modulus (MPa)	% Elongation
1	15.97	364.61	8.76
2	17.54	354.44	9.90
3	18.66	393.67	9.48
4	18.01	515.87	6.98
5	16.55	363.25	9.11
Mean	17.35	398.37	8.85
SD.	1.09	67.33	1.12

Table A 22 Experimental data of flexural properties at 25% of starch

Sample	Flexural strength (MPa)	Flexura's modulus (MPa)	Deformation at max load (mm)
1	31.80	2086.54	8.13
2	26.77	1227.43	11.63
3	27.53	2681.58	5.49
4	29.74	2428.54	6.45
5	29.25	2467.56	6.31
Mean	29.02	2178.33	7.60
SD.	1.97	572.75	2.45

Table A 23 Experimental data of Impact strength at 25% of starch

Sample	Impact strength (KJ/m ²)
1	2.03
2	2.38
3	1.72
4	1.72
5	1.84
Mean	1.94
SD.	0.28

Table A 24 Experimental data of moisture absorption and % weight loss at 25% of starch

Sample	% Moisture absorption	% weight loss 2 weeks	% weight loss 3 weeks
1	0.50	0.00	0.50
2	0.50	0.50	1.00
3	0.50	0.00	0.50
4	0.50	0.50	1.49
5	0.51	0.00	1.01
Mean	0.50	0.20	0.90
SD.	0.00	0.27	0.41

Table A 25 Experimental data of tensile properties at 30% of starch

Sample	Tensile strength (MPa)	Young's modulus (MPa)	% Elongation
1	16.60	522.15	6.36
2	16.60	498.63	6.66
3	16.60	468.24	7.09
4	15.42	491.31	6.24
5	16.65	564.41	5.90
Mean	16.38	509.55	6.45
SD.	0.53	36.15	0.45

Table A 26 Experimental data of flexural properties at 30% of starch

Sample	Flexural strength (MPa)	Flexural's modulus (MPa)	Deformation at max load (mm)
1	21.12	3434.64	3.28
2	27.83	1958.07	7.58
3	28.61	1633.77	9.34
4	26.43	1416.77	9.95
5	26.00	1824.42	7.60
Mean	26.00	2053.54	7.55
SD.	2.92	798.55	2.61

Table A 27 Experimental data of impact strength at 30% of starch

Sample	Impact strength (KJ/m ²)
1	2.63
2	1.88
3	1.66
4	1.31
5	1.72
Mean	1.84
SD.	0.49

Table A 28 Experimental data of moisture absorption and % weight loss at 30% of starch

Sample	% Moisture absorption	% weight loss 2 weeks	% weight loss 3 weeks
1	2.00	0.50	1.00
2	1.99	0.00	0.50
3	1.99	0.50	1.49
4	2.00	0.50	0.50
5	1.96	0.49	1.47
Mean	1.98	0.40	0.99
SD.	0.02	0.22	0.49

Table A 29 Experimental data of tensile properties at 40% of starch

Sample	Tensile strength (MPa)	Young's modulus (MPa)	% Elongation
1	12.43	618.08	4.02
2	11.80	467.57	5.08
3	11.19	490.83	4.56
4	12.71	549.19	5.23
5	12.97	495.91	4.63
Mean	12.22	523.72	4.70
SD.	0.72	61.05	0.48

Table A 30 Experimental data of flexural properties at 40% of starch

Sample	Flexural strength (MPa)	Flexural's modulus (MPa)	Deformation at max load (mm)
1	26.82	2376.19	6.02
2	24.25	1814.08	7.13
3	23.86	3213.38	3.96
4	24.98	2337.17	5.70
5	24.49	2712.95	4.85
Mean	24.88	2490.76	5.53
SD.	2.81	516.28	1.20

Table A 31 Experimental data of impact strength at 40% of starch

Sample	Impact strength (KJ/m ²)
1	1.28
2	1.59
3	1.53
4	1.22
5	1.31
Mean	1.39
SD.	0.16

Table A 32 Experimental data of moisture absorption and % weight loss at 40% of starch

Sample	% Moisture absorption	% weight loss 2 weeks	% weight loss 3 weeks
1	2.43	0.49	0.97
2	2.45	0.49	1.47
3	2.44	0.00	0.97
4	2.42	0.49	1.46
5	2.39	0.96	1.44
Mean	2.43	0.48	1.26
SD.	0.02	0.34	0.27

Table A 33 Experimental data of tensile properties at 50% of starch

Sample	Tensile strength (MPa)	Young's modulus (MPa)	% Elongation
1	10.65	640.49	4.78
2	11.07	463.36	4.02
3	11.03	548.76	3.70
4	10.65	575.56	3.32
5	11.21	644.25	3.48
Mean	10.92	574.47	3.86
SD.	0.26	74.58	0.58

Table A 34 Experimental data of flexural properties at 50% of starch

Sample	Flexural strength (MPa)	Flexural's modulus (MPa)	Deformation at max load (mm)
1	20.73	2233.59	4.95
2	18.66	2488.67	4.00
3	22.35	2016.97	5.91
4	25.09	2127.38	6.29
5	25.14	2188.68	5.29
Mean	22.39	2211.06	5.29
SD.	2.81	175.18	0.89

Table A 35 Experimental data of impact strength at 50% of starch

Sample	Impact strength (KJ/m ²)
1	1.33
2	1.22
3	1.22
4	1.28
5	1.16
Mean	1.25
SD.	0.08

Table A 36 Experimental data of moisture absorption and % weight loss at 50% of starch

Sample	% Moisture absorption	% weight loss 2 weeks	% weight loss 3 weeks
1	3.76	0.94	1.41
2	3.72	0.93	1.86
3	3.59	1.35	1.35
4	3.52	0.44	1.32
5	3.74	1.40	1.87
Mean	3.67	1.01	1.56
SD.	0.10	0.39	0.28

3. Modified cassava starch/PP blends

Table A 37 Experimental data of tensile properties at 5% of starch

Sample	Tensile strength (MPa)	Young's modulus (MPa)	% Elongation
1	24.94	562.26	8.87
2	23.32	543.33	8.59
3	24.09	523.45	9.20
4	21.82	494.28	8.83
5	25.36	548.70	9.25
Mean	23.91	534.41	8.95
SD.	1.41	21.41	0.28

Table A 38 Experimental data of flexural properties at 5% of starch

Sample	Flexural strength (MPa)	Flexural's modulus (MPa)	Deformation at max load (mm)
1	36.71	1614.03	12.13
2	39.95	1988.11	10.72
3	39.79	2107.13	10.07
4	40.51	2163.10	9.99
5	38.55	1949.85	10.73
Mean	39.10	1964.44	10.73
SD.	1.52	214.12	0.86

Table A 39 Experimental data of impact strength at 5% of starch

Sample	Impact strength (KJ/m ²)
1	2.78
2	2.69
3	2.66
4	2.53
5	2.59
Mean	2.65
SD.	0.09

Table A 40 Experimental data of moisture absorption and % weight loss at 5% of starch

Sample	% Moisture absorption	% weight loss 2 weeks	% weight loss 3 weeks
1	1.20	0.00	0.60
2	1.22	0.00	0.61
3	1.21	0.00	0.61
4	1.20	0.00	0.60
5	1.23	0.00	0.00
Mean	1.21	0.00	0.48
SD.	0.01	0.00	0.27

Table A 41 Experimental data of tensile properties at 10% of starch

Sample	Tensile strength (MPa)	Young's modulus (MPa)	% Elongation
1	19.84	406.44	9.76
2	21.31	535.02	7.97
3	16.66	548.92	6.07
4	21.85	494.58	8.83
5	18.47	526.33	7.02
Mean	19.63	502.26	7.93
SD.	1.45	57.16	1.03

Table A 42 Experimental data of flexural properties at 10% of starch

Sample	Flexural strength (MPa)	Flexural's modulus (MPa)	Deformation at max load (mm)
1	37.77	2478.85	8.13
2	49.73	3477.68	7.63
3	43.98	2756.92	8.51
4	37.10	1852.18	10.68
5	41.14	2603.58	8.93
Mean	41.94	2633.84	8.77
SD.	5.16	583.77	1.17

Table A 43 Experimental data of impact strength at 10% of starch

Sample	Impact strength (KJ/m ²)
1	2.50
2	2.38
3	2.44
4	2.63
5	2.53
Mean	2.49
SD.	0.09

Table A 44 Experimental data of moisture absorption and % weight loss at 10% of starch

Sample	% Moisture absorption	% weight loss 2 weeks	% weight loss 3 weeks
1	1.72	0.00	1.15
2	1.71	0.57	0.57
3	1.69	1.13	1.13
4	1.72	0.00	1.15
5	1.70	0.00	1.14
Mean	1.71	0.34	1.03
SD.	0.01	0.01	0.25

Table A 45 Experimental data of tensile properties at 15% of starch

Sample	Tensile strength (MPa)	Young's modulus (MPa)	% Elongation
1	18.23	479.86	7.60
2	18.81	515.21	7.30
3	19.87	520.12	7.64
4	18.46	385.88	9.57
5	20.35	590.45	6.89
Mean	19.14	498.30	7.80
SD.	1.03	0.92	74.56

Table A 46 Experimental data of flexural properties at 15% of starch

Sample	Flexural strength (MPa)	Flexural's modulus (MPa)	Deformation at max load (mm)
1	42.52	2359.20	9.61
2	40.29	2607.33	8.24
3	38.00	3470.14	5.84
4	42.19	3012.05	7.47
5	41.11	2790.04	7.79
Mean	40.82	2847.75	7.79
SD.	1.81	422.55	1.36

Table A 47 Experimental data of impact strength at 15% of starch

Sample	Impact strength (KJ/m ²)
1	2.03
2	2.13
3	1.94
4	2.22
5	2.16
Mean	2.09
SD.	2.11

Table A 48 Experimental data of moisture absorption and % weight loss at 15% of starch

Sample	% Moisture absorption	% weight loss 2 weeks	% weight loss 3 weeks
1	2.94	0.59	1.18
2	2.91	0.58	1.16
3	2.89	0.58	1.73
4	2.87	0.57	1.15
5	2.87	0.57	1.72
Mean	2.90	0.58	1.39
SD.	0.03	0.01	0.31

Table A 49 Experimental data of tensile properties at 20% of starch

Sample	Tensile strength (MPa)	Young's modulus (MPa)	% Elongation
1	18.95	617.61	6.14
2	21.06	598.47	7.04
3	18.63	683.29	5.45
4	16.61	473.84	7.01
5	14.17	605.47	4.68
Mean	17.88	595.74	6.06
SD.	2.51	76.00	1.02

Table A 50 Experimental data of flexural properties at 20% of starch

Sample	Flexural strength (MPa)	Flexural's modulus (MPa)	Deformation at max load (mm)
1	40.85	3246.09	6.71
2	31.01	3336.06	4.96
3	38.61	2499.24	8.24
4	37.05	2025.17	9.76
5	36.59	2651.20	7.42
Mean	36.82	2751.55	7.42
SD.	3.65	544.88	1.78

Table A 51 Experimental data of impact strength at 20% of starch

Sample	Impact strength (KJ/m ²)
1	1.84
2	1.97
3	1.78
4	2.13
5	2.00
Mean	1.94
SD.	0.14

Table A 52 Experimental data of moisture absorption and % weight loss at 20% of starch

Sample	% Moisture absorption	% weight loss 2 weeks	% weight loss 3 weeks
1	3.61	0.60	1.81
2	3.57	1.19	1.19
3	3.64	0.62	1.21
4	3.66	1.22	1.83
5	3.55	0.59	1.78
Mean	3.61	0.84	1.56
SD.	0.04	0.33	0.33

Table A 53 Experimental data of tensile properties at 25% of starch

Sample	Tensile strength (MPa)	Young's modulus (MPa)	% Elongation
1	19.18	806.27	4.76
2	13.59	493.34	5.51
3	19.39	546.17	7.10
4	17.05	518.55	6.58
5	19.35	721.97	5.36
Mean	17.71	5.86	617.26
SD.	2.51	0.95	136.60

Table A 54 Experimental data of flexural properties at 25% of starch

Sample	Flexural strength (MPa)	Flexural's modulus (MPa)	Deformation at max load (mm)
1	34.76	3445.11	5.38
2	34.53	4235.89	4.35
3	34.81	3310.00	5.61
4	36.12	2991.51	6.56
5	35.92	3083.52	6.25
Mean	35.23	3413.21	5.63
SD.	0.73	493.71	0.86

Table A 55 Experimental data of impact strength at 25% of starch

Sample	Impact strength (KJ/m ²)
1	1.69
2	1.81
3	1.78
4	1.84
5	1.75
Mean	1.78
SD.	0.06

Table A 56 Experimental data of moisture absorption and % weight loss at 25% of starch

Sample	% Moisture absorption	% weight loss 2 weeks	% weight loss 3 weeks
1	4.95	1.10	1.65
2	4.86	1.62	2.16
3	4.89	0.54	1.09
4	4.92	1.64	2.73
5	4.81	1.07	1.60
Mean	4.89	1.19	1.85
SD.	0.05	0.46	0.62

Table A 57 Experimental data of tensile properties at 30% of starch

Sample	Tensile strength (MPa)	Young's modulus (MPa)	% Elongation
1	16.64	724.20	4.59
2	16.84	722.28	4.66
3	14.32	586.39	4.89
4	13.83	469.03	5.90
5	13.83	467.14	5.95
Mean	15.09	5.91	594.19
SD.	1.52	0.66	127.18

Table A 58 Experimental data of flexural properties at 30% of starch

Sample	Flexural strength (MPa)	Flexural's modulus (MPa)	Deformation at max load (mm)
1	36.77	2856.01	6.87
2	37.77	3969.38	5.07
3	39.39	3503.82	5.99
4	39.45	3606.39	5.84
5	38.35	3458.42	5.91
Mean	38.35	3478.80	5.94
SD.	1.13	401.82	0.64

Table A 59 Experimental data of impact strength at 30% of starch

Sample	Impact strength (KJ/m ²)
1	1.66
2	1.59
3	1.47
4	1.53
5	1.56
Mean	1.56
SD.	0.07

Table A 60 Experimental data of moisture absorption and % weight loss at 30% of starch

Sample	% Moisture absorption	% weight loss 2 weeks	% weight loss 3 weeks
1	7.07	1.52	2.02
2	6.97	1.99	2.99
3	6.97	1.49	2.49
4	7.00	1.50	2.00
5	6.86	2.94	3.43
Mean	6.97	1.83	2.58
SD.	0.08	0.63	0.62

Table A 61 Experimental data of tensile properties at 40% of starch

Sample	Tensile strength (MPa)	Young's modulus (MPa)	% Elongation
1	13.35	450.55	5.93
2	14.86	559.75	5.31
3	13.26	714.68	3.71
4	15.77	811.64	3.89
5	12.85	494.91	5.19
Mean	14.02	606.28	4.81
SD.	1.24	152.25	/.96

Table A 62 Experimental data of flexural properties at 40% of starch

Sample	Flexural strength (MPa)	Flexural's modulus (MPa)	Deformation at max load (mm)
1	31.91	5947.44	2.86
2	29.17	4481.78	3.47
3	33.25	5961.13	2.98
4	31.12	3990.80	4.16
5	31.36	4966.95	3.37
Mean	31.36	5069.62	3.37
SD.	1.48	878.25	0.51

Table A 63 Experimental data of impact strength at 40% of starch

Sample	Impact strength (KJ/m ²)
1	1.44
2	1.38
3	1.41
4	1.47
5	1.44
Mean	1.43
SD.	0.04

Table A 64 Experimental data of moisture absorption and % weight loss at 40% of starch

Sample	% Moisture absorption	% weight loss 2 weeks	% weight loss 3 weeks
1	8.13	1.91	2.87
2	8.33	2.45	3.43
3	8.21	1.93	3.38
4	8.29	1.95	3.41
5	8.13	2.39	2.87
Mean	8.22	2.13	3.19
SD.	0.09	0.27	0.30

Table A 65 Experimental data of tensile properties at 50% of starch

Sample	Tensile strength (MPa)	Young's modulus (MPa)	% Elongation
1	10.42	441.66	4.72
2	9.60	451.15	4.25
3	13.19	538.17	4.90
4	13.21	535.41	4.84
5	13.70	691.44	3.96
Mean	12.02	532.12	4.55
SD.	1.87	100.24	0.42

Table A 66 Experimental data of flexural properties at 50% of starch

Sample	Flexural strength (MPa)	Flexural's modulus (MPa)	Deformation at max load (mm)
1	24.08	5345.53	2.40
2	26.21	3939.28	3.55
3	20.23	4875.31	2.21
4	27.88	6188.88	2.40
5	24.60	4966.05	2.64
Mean	24.60	5063.01	2.64
SD.	2.86	814.68	0.53

Table A 67 Experimental data of impact strength at 50% of starch

Sample	Impact strength (KJ/m ²)
1	1.38
2	1.31
3	1.44
4	1.22
5	1.25
Mean	1.32
SD.	0.09

Table A 68 Experimental data of moisture absorption and % weight loss at 50% of starch

Sample	% Moisture absorption	% weight loss 2 weeks	% weight loss 3 weeks
1	9.21	3.07	3.95
2	9.13	2.61	3.48
3	9.38	2.23	3.13
4	9.25	2.20	3.52
5	9.21	2.19	3.51
Mean	9.24	2.48	3.52
SD.	0.09	0.38	0.29

BIOGRAPHY

Mr. Supasit Pumeechokechai was born in Bangkok, Thailand, on July 8, 1980. He received a Bachelor of Engineering and Technology degree with a major in Petrochemical and Polymeric Materials from Silpakorn University in 2003. He started as a graduate student in Department of Materials Science with a major in Applied Polymer Science and Textile Technology, Chulalongkorn University in 2003, and completed the program in 2005.



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