

**BIODEGRADABLE BIOCOMPOSITE STARCH BASED FILMS BLENDED  
WITH CHITOSAN AND GELATIN**

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## ABSTRACT

The use of petroleum based plastic cause serious damage to environment and the plastic takes a long time to degrade. Thus, the existence of biodegradable plastic may serve as a promising solution to this problem. The objectives of this study are to produce biodegradable biocomposite film from starch, chitosan and gelatin and to study the characterization of biocomposite film in chemical and mechanical properties and biodegradability. This research was conducted by manipulating the concentration of starch from 1 to 5 % while the concentration of chitosan and gelatin was kept constant at 2%. The films were obtained by using mixing and casting method before leave in oven overnight for drying process. The characteristic of biocomposite films were evaluated using equipments like Universal Testing Machine, Fourier transform infrared spectroscopy (FTIR), scanning electron microscope (SEM) observation and gas pycnometer. The films also characterized in moisture and water absorption and biodegradability using soil burial degradation test. The film at 3% starch blended with 2% of chitosan and 2% gelatin showed the highest density, the lowest moisture and water absorption and exhibits a smooth surface with less agglomerates and no visible pore. These properties make the film perfect for food packaging application. The FTIR test identified O-H stretching, C-H stretching and C=O stretching were at peak of 3279-3325, 2930-2932 and 1647  $\text{cm}^{-1}$  respectively. The films also fully degrade within 20 days, and the degradation rate is considered high. However, the film at 1% starch concentration exhibit the stronger tensile stress due to high formation of inter-molecular hydrogen bonds between chitosan and starch. The formation of hydrogen bond is inversely proportional with starch concentration. As a conclusion, the blended film from starch, chitosan and gelatin had potential application in future to be used as food packaging because it can improved foods quality and at the same time conserve the environment.

## ABSTRAK

Penggunaan plastik yang diperbuat daripada petroleum menyebabkan masalah yang serius terhadap alam sekitar selain mengambil masa yang lama untuk terurai. Oleh itu, kewujudan plastik bio-urai mungkin boleh menyelesaikan masalah ini. Objektif kajian ini adalah untuk menghasilkan filem biokomposit biodegradasi daripada kanji, kitosan dan gelatin dan mengkaji ciri-ciri kimia, mekanikal dan biodegradasi. Kajian ini telah dijalankan dengan mengubah kepekatan kanji dari 1 hingga 5 % dan menetapkan kepekatan kitosan dan gelatin pada 2%. Filem ini dihasilkan dengan menggunakan kaedah campuran dan tuangan sebelum dibiarkan semalaman di dalam ketuhar untuk proses pengeringan. Filem yang terhasil telah dicirikan dengan menggunakan mesin pengujian, spektroskopi inframerah transformasi Fourier (FTIR), mikroskopi pengimbasan elektron (SEM) dan gas pycnometer. Filem ini juga telah dicirikan dalam penyerapan air dan kelembapan dan biodegradasi menggunakan kaedah timbus tanah. Filem pada kandungan 3% kanji dan 2% kitosan dan 2% gelatin menunjukkan ketumpatan paling tinggi dan penyerapan air dan kelembapan paling rendah selain mempunyai permukaan yang licin, tiada pengumpulan dan tiada lubang kelihatan berdasarkan ujian SEM. Ciri-ciri ini membuatkan filem ini sesuai sebagai pembungkus makanan. Ujian FTIR telah mengenal pasti regangan O-H, C-H dan C=O pada 3279-3325, 2930-2932 and 1647  $\text{cm}^{-1}$  masing-masing. Filem ini juga terurai sepenuhnya dalam tempoh 20 hari dan kadar penguraian bolehlah dikatakan tinggi. Walau bagaimanapun, filem pada 1% kandungan kanji menunjukkan kekuatan tekanan ketegangan disebabkan oleh pembentukan ikatan hidrogen antara kanji dan kitosan. Pembentukan ikatan hidrogen berkadar songsang dengan kepekatan kanji. Kesimpulannya, filem yang terhasil daripada campuran kanji, kitosan dan gelatin mempunyai potensi dalam pembungkusan makanan kerana ia dapat membaiki pulih kualiti makanan dan dalam masa yang sama boleh melindungi alam sekitar.

## TABLE OF CONTENT

CHAPTER	TITLE	PAGE
	<b>AUTHENTICATION</b>	
	<b>TITLE</b>	
	<b>DECLARATION</b>	ii
	<b>DEDICATION</b>	iii
	<b>ACKNOWLEDGEMENTS</b>	iv
	<b>ABSTRACT</b>	v
	<b>ABSTRAK</b>	vi
	<b>TABLE OF CONTENTS</b>	vii
	<b>LIST OF TABLES</b>	x
	<b>LIST OF FIGURES</b>	xi
	<b>LIST OF APPENDICES</b>	xiii
	<b>LIST OF NOMENCLATURES</b>	xiv
<b>1</b>	<b>INTRODUCTION</b>	
	1.1 Background of Study	1
	1.2 Problem Statement	2
	1.3 Research Objectives	3
	1.4 Scope of Study	3
	1.5 Significance of the Study	3
<b>2</b>	<b>LITERATURE REVIEW</b>	
	2.1 Introduction	5
	2.2 Composite and Biocomposite	6
	2.3 Film Forming Material	7

2.3.1	Starch	8
2.3.2	Chitosan	10
2.3.3	Gelatin	11
2.3.4	Glycerol as plasticizer	12
2.3.5	Citric Acid	12
2.4	Analysis Equipment	13
2.4.1	Universal Testing Machine	13
2.4.2	Fourier Transform Infrared Spectroscopy(FTIR)	14
2.4.3	Scanning Electron Microscope (SEM)	15
2.4.4	Gas Pycnometer	16
2.5	Analysis Method	17
2.5.1	Moisture and Water Absorption	18
2.5.2	Biodegradability	18
<b>3</b>	<b>METHODOLOGY</b>	
3.1	Introduction	20
3.2	Materials and Methods	20
3.2.1	Materials	20
3.2.2	Methods	21
3.2.2.1	Film Preparation	21
3.2.2.2	Universal Testing Machine	23
3.2.2.3	Fourier transform infrared Spectroscopy (FTIR)	23
3.2.2.4	Scanning Electron Microscopy (SEM)	24
3.2.2.5	Density	24
3.2.2.5.1	Gas Pycnometer	24
3.2.2.5.2	Experimental	25
3.2.2.6	Water Absorption	25
3.2.2.7	Moisture Absorption	26
3.2.2.8	Soil Burial Degradation Test	26

<b>4</b>	<b>RESULT AND DISCUSSION</b>	
4.1	Introduction	28
4.2	Universal Testing Machine	28
4.3	Fourier Transform Infrared Spectroscopy (FTIR)	30
4.4	Scanning Electron Microscopy (SEM)	31
4.5	Moisture and Water Absorption	35
4.6	Density	38
4.7	Soil Burial Degradation Test	39
<b>5</b>	<b>CONCLUSION AND RECOMMENDATION</b>	
5.1	Conclusion	41
5.2	Recommendation	42
	<b>REFERENCES</b>	43
	<b>APPENDICES</b>	
	Appendix A: Film Preparation	48
	Appendix B: Films Testing	52

**LIST OF TABLE**

<b>TABLE</b>	<b>TITLE</b>	<b>PAGE</b>
2.1	The IR-Spectra of the functional group (Xu et al, 2005), (Salleh et al, 2009) and (Bourtoom and Chinnan, 2008)	15
4.1	Tensile strength for biocomposite film at different starch concentration	29
4.2	Moisture absorption at different starch concentration	36
4.3	Water absorption at different starch concentration	36
4.4	Density for different starch concentration	38
4.5	Degradation time for different starch concentration	39

**LIST OF FIGURE**

<b>FIGURE</b>	<b>TITLE</b>	<b>PAGE</b>
2.1	Chemical formula of starch. (Pandey, 2004)	9
2.2	Chemical formula of glycerol (Yun <i>et al.</i> , 2009)	12
2.3	Chemical formula of citric acid (Yun <i>et al.</i> , 2009)	13
2.4	Principle of gas pycnometer (Fang and Campbell, 2000)	17
3.1	Film preparation process	22
3.2	FTIR Nicolet Avatar 370 DTGS	23
3.3	gas pycnometer Micromeritics model AccuPyc II 1340	24
3.4	Film characterization and analysis method	27
4.1	Tensile strength of biocomposite film for different starch concentration	29
4.2	The infrared spectra of biocomposite films with different starch concentration.	31
4.3	SEM micrograph of the surface of biocomposite film with 1% starch	32
4.4	SEM micrograph of the cross section of biocomposite film with 1% starch	32
4.5	SEM micrograph of the surface of biocomposite film with 3% starch	33
4.6	SEM micrograph of the cross section of biocomposite film with 3% starch	33
4.7	SEM micrograph of the surface of biocomposite film with 5% starch	34



4.8	SEM micrograph of the cross section of biocomposite film with 5% starch	34
4.9	Moisture content at different starch concentration	36
4.10	Water absorption at different starch content	37
4.11	Visual image of degrade film	39
4.12	Degradation time for starch at different concentration	40

**LIST OF APPENDICES**

<b>APPENDIX</b>	<b>TITLE</b>	<b>PAGE</b>
<b>A</b>	Film Preparation	48
<b>B</b>	Film Testing	52

**LIST OF NOMENCLATURE**

FTIR	-	Fourier transform infrared spectroscopy
SEM	-	Scanning electron microscopy
DSC	-	differential scanning calorimetry
CA	-	Citric acid
UV	-	ultra violet
°C	-	Degree celcius
Kv	-	kilovolt
%	-	Percentage
α-		alpha
g	-	Gram
min	-	minute
ml	-	Mililiter
mm	-	Milimeter
v/v	-	volume pervolume
w/v	-	Weight per volume
w/w	-	Weight per weight
°C	-	Degree celcius

## **CHAPTER 1**

### **INTRODUCTION**

#### **1.1 Background of Study**

In the past few years until now, most of government organizations worldwide pay a serious attention regarding environmental issue involving petroleum-based plastic. Plastic can cause a serious damage to environment during its production and disposal. It contain toxic chemical which will lead harm effect towards earth, air and water. The noxious substances emitted during the production of plastic are synthetic chemicals like ethylene oxide, benzene and xylenes. These chemicals may cause cancer, damage the nervous system and the immune system.

The plastic disposal issue keep arise since it takes a long time to degrade. The abundance of plastic waste has led to the serious plastic waste problems, so called 'White Pollution', and landfill depletion, due to their high volume to weight ratio and resistance to degrade. White pollution is referring into two negative effects on the environment which is 'visual pollution' and 'potential hazards' result from the ineffective management and disposal of plastic waste. Visual pollution is refers to the unpleasant appearance and landscape due to the plastic rubbish scattered in the environment. Potential hazards refer to long-term and deep-seated environmental problems, which arise when plastic waste is thrown away randomly in the natural environment. It could result in difficulty to solve this kind of pollution problem.

Since plastic bags have become such an ubiquitous way of life, it is almost impossible to get rid of it. Hence, biodegradable plastics may serve as a promising solution to the over-loaded landfills and to substitute the role of petroleum in making plastic by a renewable source.

Starch is probably the most promising material for the production of biodegradable plastics and it comes from renewable resources. It can be obtained from a variety of crops like tapioca, corn and potatoes. Starch has been investigated widely for the potential manufacture of products such as water-soluble pouches for detergents and insecticides, flushable liners and bags, and medical delivery systems and devices (Chang, 2009). In order to overcome its poor mechanical properties, starch must be blended with plasticizer to reduce its intermolecular forces.

## **1.2 Problem Statement**

The typical petroleum-based plastic takes a long time to degrade because of the molecular bonds that make the plastic so durable and equally resistant to natural processes of degradation. Hence, the landfill area will have an abundance of plastic waste and lead to another issue including lack of landfill area and soil contamination. Besides, the nature of plastic which contains toxic chemicals can cause pollution to the environment. When plastic is burned, it can release dioxin which is the most toxic substance. Hence, it is crucial to find other environmentally friendly materials to substitute this petroleum-based plastic.

Great concern regarding environmental issues and the non-renewable nature of petroleum has attracted many researchers to research about biocomposite plastics. Thus, the purpose of this research is to produce biodegradable biocomposite films using renewable sources by blending chitosan and gelatin with starch.

### **1.3 Research Objectives**

The main objectives of this research are to produce biodegradable biocomposite film from starch and to study the characterization of biocomposite film in chemical and mechanical properties and also biodegradability.

### **1.4 Scope of the Study**

The scopes of this research are

- i. To produce biodegradable biocomposite film using 1, 3 and 5% starch blended with 2% of chitosan and 2% of gelatin.
- ii. To characterize biocomposite film on mechanical and chemical properties using Universal Testing Machine, Fourier Transforms Infrared Spectroscopy (FTIR), Scanning Electron Microscope (SEM) And Gas Pycnometer.
- iii. To characterize biocomposite film on biodegradability using soil burial degradation method.
- iv. To analyze biocomposite film on moisture and water absorption properties using swelling method.

### **1.5 Significance of the Study**

This study will be significant in producing biodegradable plastic which exhibit better mechanical property and presentable appearance for food packaging purpose. Since the use of plastic in food packaging is undeniable, the finding in this research might be considered for commercialization.

This study also may contribute to the reduction of pollution to environment since the petroleum-based plastic caused serious damage to environment both during its production and disposal. Beside, the effect of other contamination from plastic waste also can be decrease and the lack of landfill area might be resolve since the plastic produce is biodegradable.

This study is also hoped to provide other alternative material to substitute petroleum role in polymer industry due to depletion in petroleum sources with a renewable source.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 INTRODUCTION**

It is been a long time ago since artificial polymers happens to replace natural materials in mostly everyday appliance and nowadays plastics have become an indispensable part of our life. As the time goes by, the stability and durability of plastics have been improved constantly, and hence this group of materials is now considered as a synonym for materials being resistant to many environmental influences. The plastics uses today are made from inorganic and organic raw materials, such as carbon, silicon, hydrogen, nitrogen, oxygen and chloride. The basic materials used for making plastics are extracted from oil, coal and natural gas (Shah, 2008)

Recently, the development of biocomposites from biodegradable polymers and natural fibers has attracted great interests among researcher in the worldwide. This is because, biocomposite may have complete degradation in soil or by composting process as well as do not emit any toxic substance during production and disposal process. By successfully producing biocomposite, we may be able to substitute the conventional petro-based plastic in various applications. This finding will contribute a lot in environmental concern as well as in chemical industry.

The uncontrolled use of petroleum products has created a twin dilemma; depletion of petroleum resources and entrapment of plastics in the food chain and



environment. The increasing contamination caused by the use of plastics and emissions during incineration is affecting the food and water consumes by human every day. The exhaustive use of petroleum based resources has initiate the efforts to develop biodegradable plastics. This is based on renewable bio-based plant and agricultural products that can contend in the markets currently dominated by petroleum based products. (John and Thomas, 2008)

## **2.2 Composite and Biocomposite**

Composite material is an engineered material made from two or more constituent material with significantly different physical or chemical properties which remain separate and distinct on a macroscopic level within the finished structure. There are two types of constituent materials which is matrix and reinforcement. In order to obtain composite material, at least one portion of each type is required. The matrix material will surrounds and supports the reinforcement materials by maintaining their relative positions. The reinforcements contribute to the mechanical and physical properties to enhance the matrix properties.

In the other hand, biocomposite are composite materials made from natural and bio fibres that become eco-friendly and such composites are termed as green composites. The major attractions about green composites are that they are environmentally friendly, fully degradable and sustainable. These composite can be easily disposed of or composted without harming the environment. They often mimic the structures of the living materials involved in the process in addition to the strengthening properties of the matrix that was used but still providing biocompatibility (John and Thomas, 2008). Thus, the most important part in producing biocomposite is to choose an optimum composition of the compatible combination. Compatible combination means a blend process which the 2 or more component is miscible to each other in order for an interaction to be occur.

Composite may be classified into three types which is green composites, hybrid composites and textile composites. Green composites referred to a composites which are environmentally friendly, completely degradable and sustainable. At the last part of their life, they can be effortlessly disposed of or composted without harming the environment. Green composite can be obtained by combining natural or bio fibres with biodegradable resins. (John and Thomas, 2008)

On the other hand, hybrid composites is result from integration of several different types of fibres into a single matrix. This principle is work as each fibre complementary each other. The properties of a hybrid composite primarily depend upon the fibre content, length of individual fibres, orientation, extent of intermingling of fibres, fibre to matrix bonding and arrangement of both the fibres. (John and Thomas, 2008)

Textile composite is significant to textile development as the composite result superior mechanical properties, as continuous orientation of fibres is not restricted at any point. The fabric's integrity is maintained by the mechanical interlocking of the fibres. Drape (the ability of a fabric to conform to a complex surface), surface smoothness and stability of a fabric are controlled mainly by the weave style.(John and Thomas, 2008)

### **2.3 Film Forming Material**

Materials that can be used for film making include polysaccharides, proteins, lipids and polyesters or combinations of them (Ghanbarzadeh and Oromeihi, 2008). In this study, starch, chitosan and gelatin are chosen based on their availability and renewable source, biodegradability and biocompatibility.

### 2.3.1 Starch

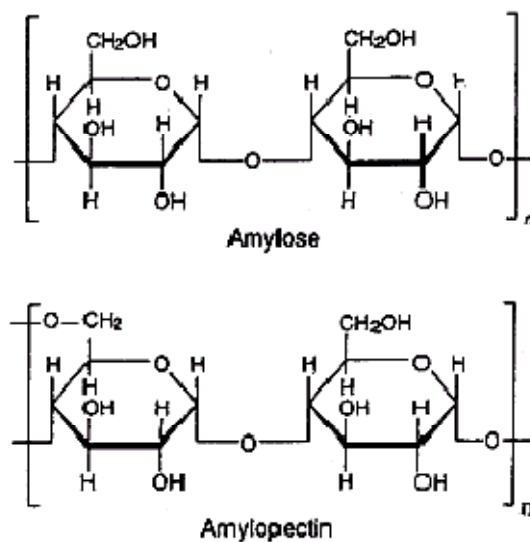
Starch is a major plant storage form of glucose. It consists of two components: amylose, in which the glucose units are 1, 4- $\alpha$ -D-linked together in straight chains and amylopectin, in which the glucose chains are highly branched (Flieger *et al.*, 2003). The linear structure of amylose makes its actions more closely be similar to that of conventional synthetic polymers. Depending on its source and the processing conditions employed during its extraction, the molecular weight of amylose is about  $\times 10^6$ , which is 10 times higher than conventional synthetic polymers. Amylopectin, on the other hand, is a branched polymer and its molecular weight is much greater than amylose, with light-scattering measurements representing molecular weights in the millions. The high molecular weight and branched structure of amylopectin reduce the mobility of the polymer chains, and interfere with any affinity for them to become oriented closely enough to permit significant levels of hydrogen bonding. Between the linear amylose and short-branched amylopectin, a long-branched structure has been detected, such as in tapioca starch. (Liu *et al.*, 2009)

The thermal processing of starch-based polymers involves multiple chemical and physical reactions such as water diffusion, granule extension, gelatinization, decomposition, melting and crystallization. Between the various phase transitions, gelatinization is particularly important because it is closely related to the others, and it is the basis of the conversion of starch to a thermoplastic. Furthermore, the decomposition temperature of starch is higher than its melting temperature before gelatinization. The well-accepted concept of 'gelatinization' refers to the destruction of the crystalline structure in starch granules which is an irreversible process that includes, in a broad sense and in time sequence, granular swelling, native crystalline melting (loss of birefringence) and molecular solubilization. Starch gelatinization is a multistage process that can be detected by differential scanning calorimetry (DSC) (Liu *et al.*, 2009)

Pure starch is a white, tasteless and odorless powder that is insoluble in cold water or alcohol. But if dissolved in warm water, it can be used as a thickening, or

gluing agent. High content of amylose in starch provide strong, flexible films, which are thermally stable up to 180°C (Cheorun *et al.*, 2005). Starch itself is very brittle and has poor mechanical properties for plastic applications. However, by blending starch with glycerol, significant improvements in both tensile strength and elongation at break can be seen (Mao *et al.*, 2002).

Starch is also one of the least expensive biodegradable materials used for many non-food items such as in paper making, cardboard, textile sizing and adhesives. Recently, starch has been used as the main polymer in thermoplastic compositions and has been processed into eating utensils and as raw material for film production. Starch offers an interesting alternative for synthetic polymers where long-term durability is not needed and rapid degradation is an advantage (Cañigueral *et al.*, 2009)



**Figure 2.1:** Chemical formula of starch. (Pandey, 2004)

### 2.3.2 Chitosan

Chitosan is a linear polysaccharide. Polysaccharides are polymeric carbohydrate structures, formed of repeating units (either mono- or di-saccharides) joined together by glycosidic bonds. Chitosan is produced commercially from chitin by deacetylation (removal of the acetyl group). Chitin is a major component of the shells of crustaceans such as crab, shrimp, and crawfish. Chitosan contains three types of reactive functional groups, an amino/acetamido group as well as both primary and secondary hydroxyl groups at the C-2, C-3 and C-6 positions, respectively. The amino contents are the main reason for the differences between their structures and physicochemical properties. (Xia *et al.*, 2010)

Chitosan is the second most abundant polysaccharide on earth after cellulose. This biopolymer is mostly available from waste products in the shellfish industry, and therefore, abundant commercial supplies are currently offered. It can also be obtained from the chitin component of fungal cell walls. (Fernandez-Saiz *et al.*, 2010)

Chitosan is positively charged and readily binds to negatively charged surfaces. This property makes chitosan a bioadhesive material. Other properties possessed by chitosan are biodegradable, biocompatible and non-toxic polymer, making it a valuable component of natural packaging films. The reaction of chitosan is considerably more versatile than cellulose due to the presence of NH<sub>2</sub> group. This is why chitosan is used in this study. (Dutta *et al.*, 2008)

Chitosan is readily soluble in dilute acidic solutions below pH 6.0. This is because chitosan can be considered a strong base as it possesses primary amino groups with a pK<sub>a</sub> value of 6.3. The presence of the amino groups indicates that pH substantially alters the charged state and properties of Chitosan. At low pH, these amines get protonated and become positively charged and that makes CS a water-soluble cationic polyelectrolyte. (Pillai *et al.*, 2009)

Beside, the starch-chitosan blend exhibits good film forming property, which is attributed to the inter- and intramolecular hydrogen bonding that formed between amino groups and hydroxyl groups on the backbone of two components. The mechanical properties, water barrier properties, and miscibility of biodegradable blend films are affected by the ratio of starch and chitosan. (Lu *et al.*, 2009)

### 2.3.3 Gelatin

Gelatin is one of the natural polymers which are now widely studied. It is produced by partial hydrolysis of collagen extracted from the bones, connective tissues, organs and some intestines of animals. Gelatin is a translucent, colorless, odorless, brittle, nearly tasteless solid substance, derived from the collagen inside animals' skin and bones. It is a high molecular weight polypeptide composing of amino acids mainly glycine (27%), hydroxyproline and proline (25%). (Karnnet *et al.*, 2005)

Previously, gelatin has been used in the pharmaceutical industry as a binder for the production of hard capsules and in the textile industry because of its adhesive properties. (Arvanittoyannis *et al.*, 1998)

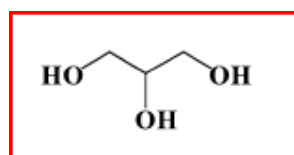
Pure gelatin films are not suitable for many applications due to its brittle structure and high moisture absorption. Therefore, gelatin is generally blended or copolymerized with other synthetic polymers to overcome the disadvantage mentioned above. Blending is easy to handle but may encounter the incompatibility between hydrophilic gelatin and hydrophobic synthetic polymers (Karnnet *et al.*, 2005). Hence, by blending gelatin with hydrophilic molecules like chitosan may be considered as one of the alternatives.

### 2.3.4 Glycerol as plasticizer

Plasticizers or are additives that increase the plasticity or fluidity of the material. Plasticizers are often added to modify the mechanical properties of biopolymeric films and these may cause significant changes in the barrier properties of the material (Ganbarzadeh and Oromeihi, 2008).

Glycerol is often used as plasticizer because of its hydrophilic properties, making it miscible with water. Elongation of films containing glycerol in higher concentration was about 8 times greater than that of unplasticized samples (Kołodziejaska and Piotrowska, 2007). Glycerol also reduces the interaction between protein molecules and increases the flexibility and extensibility of the final product.

The interest in using glycerol is mainly due to the factors that glycerol is acting as plasticizer, reduces the intermolecular forces and increasing the mobility of the biopolymer chains (Lavorgna, 2010). Besides, glycerol also reduces the extent of edge–edge interactions (i.e. H-bonding interactions), hence making it possible to achieve a better dispersion (Lavorgna, 2010).



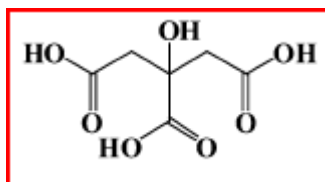
**Figure 2.2:** Chemical formula of glycerol (Yun *et al.*, 2009)

### 2.3.5 Citric Acid

Citric acid is a weak organic acid exists in greater than trace amounts in a variety of fruits and vegetables which can act as a solvent. Citric acid (CA) is recognized as a cross-linking agent for starch modification. Because of the multi-carboxyl structure, CA may serve as a cross-linking agent and the carboxyl groups on CA can form stronger

hydrogen bonds between the hydroxyl groups on starch molecules (Almasi *et al.*,2010) Hence, it may improve the mechanical properties of the starch.

Citric acid is used in this study because is recognized as nutritionally harmless compared to other substances used for starch derivative.



**Figure 2.3:** Chemical formula of citric acid (Yun *et al.*, 2009)

## 2.4 Analysis Equipment

There are several equipments that can be used for film analysis purpose. It is used to analyzed and to characterized the film obtain in this study

### 2.4.1 Universal Testing Machine

Universal testing machine is used to investigate the tensile stress and compressive stress of materials. It is named after the fact that it can perform many standard tensile and compression tests on materials, components, and structures. Information on mechanical properties of plastics such as tensile, flexural, compressive and shear can help user to optimize material formulations, processes, and quality control.

In the previous research, universal testing machine is used to evaluate tensile strength and elongation at break. It was found that, no matter which type starch was present, the tensile strength values of the composite films first increased with the



addition of starch to chitosan at ratio of 1:1. The tensile strength then decreased with further increase in the starch to chitosan ratio up to 2:1. (Xu *et al.*, 2005)

When glycerol is added to gelatin films, higher elongation at break and lower tensile strength values are obtained, since plasticizer interfere with chain association facilitating their stretching and increasing film flexibility. (Rivero *et al.*, 2009)

#### **2.4.2 Fourier Transform Infrared Spectroscopy (FTIR)**

FTIR is the preferred method for infrared detection of chromatographically separated species. The popularity of FT-IR is primarily due to the multiplex and rapid scanning features of interferometry. The interferometer produce a complex waveform that is a sum of contribution from all wavelength emitted by the source. (White, 1990)

FTIR is also a wonderful tool for detecting functional groups but cannot necessarily be used to elucidate the complete structure of an unknown molecule. Often time, infrared spectrum does not contain enough information or contain misleading or contradictory information, making the complete determination of an unknown's structure impossible. The best approach to this problem is to use FTIR in conjunction with other molecular spectroscopy techniques such as mass spectrometry and Raman scattering. (Smith, 1996)