UNIVERSITI PUTRA MALAYSIA

CHARACTERIZATION AND DEVELOPMENT OF CASSAVA (Manihot esculenta CRANTZ) / SUGAR PALM (Arenga pinnata (WURMB) MERR.) FIBER- REINFORCED CASSAVA STARCH HYBRID COMPOSITES

AHMED FARAJ IBRAHIM HISSEN EDHIREJ

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AHMED FARAJ IBRAHIM HISSEN EDHIREJ

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

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Chairman : Professor Mohd Sapuan Salit, PhD
Faculty : Engineering

Cassava is a unique plant, which has a number of uses including the manufacture of various foods, bio-fibers, bio-composites and biopolymers. Moreover, it serves as a renewable energy source as a starch. A series of characterization experiments were carried out to explore the potential use of cassava starch, peel, bagasse, polymers, composites and hybrid composites. A casting technique was used to fabricate the specimens. The following five-phases process prepared the hybrid composites from cassava/sugar palm fibers and cassava starch.

The first phase of the process studied the physical, morphological and thermal properties of cassava starch (CS), peel (CP) and bagasse (CB) obtained from a Malaysian cassava plant along with their chemical composition. Thermal characteristic studies using a Thermal Gravimetric Analyzer (TGA) and Differential Scanning Calorimetry (DSC) showed that cassava starch and fibers were thermally stable with a decomposition temperature of 172.87, 177.76 and 169.23 ºC for cassava starch, peel and bagasse, respectively. A study on morphological surface indicated that cassava starches were rounded and oval-shaped, bagasse polygonal shaped and peel exhibited as both round and polygonal. The carbohydrate content in starch was 86.47 g/100g. The study showed that the cassava peel and bagasse contained high concentrations of hemicellulose at 23.38, 29.26 %, respectively. It can be concluded that, cassava starch and fiber are suitable to develop materials with acceptable mechanical and thermal properties for various applications such as packaging, automotive and agro-industrial applications.

Phase two involved an investigation of the effects of plasticizers (Fructose, Urea, Triethylene glycol and Triethanolamine), with different concentration, on the physical, thermal and mechanical properties of cassava starch-based films. The moisture content, water solubility and water absorption of the films increased with increasing plasticizer content. Fructose plasticized films showed excellent water resistance when compared to other plasticizers. Film plasticized with 30% fructose showed the highest density at 1.74 g/cm^3, while the lowest water content at 10.96% and water absorption at 110%. Films containing fructose presented smooth surfaces without pores. The relative crystallinity decreased with increasing plasticizer content. The film plasticized by 30% fructose presented higher relative crystallinity (0.31). Film plasticized with 30% fructose showed the tensile strength (4.7 MPa) and tensile modulus (69 MPa). Thus, fructose was the most efficient plasticizer agent among the various plasticizers used in this study.

Phase three focused on the use cassava peel with two different particle sizes as a natural filler for thermoplastic starch (TPS) based on the cassava starch. The addition of peel resulted in an increase in the thickness, water content and water absorption of the films while it decreased the density and water solubility. Moreover, scanning electron microscopy showed that the films containing smaller size of peel had a better compact structure and a homogeneous surface without pores. The addition of 6% peel increased the elastic modulus and tensile strength up to 449.74 and 9.62 MPa, respectively, this being the most efficient reinforcing agent. Also, the temperature variation of the dynamic-mechanical parameters of cassava starch/peel composites was investigated using a DMA test. It was observed that the incorporation of peel increased the tensile strength and modulus. In conclusion, cassava starch/peel composite films are suitable for various purposes such as packaging, automotive and agro-industrial application, at a lower cost.

In phase four, cassava starch-based composite film was prepared using the fibrous residual from starch extraction (cassava bagasse) as a filler. Composite films were prepared through the casting technique using fructose as plasticizer and various size and concentration of bagasse. The size and concentration of bagasse significantly influenced the physical properties. It increased the thickness, water solubility and water absorption. It reduced the water content and density of the film. However, there was no significant effect on thermal properties with the addition of bagasse. XRD studies indicated an increase in the crystallinity of the composites with increasing fiber content. SEM micrographs indicated that films with a smaller size of bagasse showed better compact structure and a homogeneous surface. The modulus and tensile strength of composite films was increased from 69.03 to 581.68 MPa and from 4.7 to 10.78 MPa, respectively, by the addition of 6% bagasse, showed the most efficient reinforcing agent owing to its remarkable physical and mechanical properties.

In the final phase, the hybrid composites were successfully prepared using different amounts of fibers, the hybrid composite contains (6% w/w dry starch) cassava bagasse and (0, 2, 4, 6 and 8 % w/w dry starch) sugar palm fiber. The specimens were prepared

by a casting technique using fructose as plasticizer for the cassava starch. The incorporating of SPF significantly influenced the physical properties. It increased the thickness, while it decreased density, water content, water solubility and water absorption of the films. Moreover, XRD studies indicated increasing crystallinity of the composites with increasing of fiber content. The incorporation of SPF increased the relative crystallinity up to 47%, compared to 32% of the CS film. The water barrier vapor permeability (WVP) values decreased with increasing SPF content. The mechanical properties of the films improved with the incorporation of fibers. The modulus and tensile strength of the films increased from 69.03 to 1114.6 MPa and from 4.7 to 20.72 MPa, respectively for film contains 6% CB and 6% SPF, which was the most efficient reinforcing agent. Also, dynamic-mechanical properties of the hybrid composites were investigated using a DMA test. It was observed that the incorporation of SPF increased the storage modulus (E') value from 0.457 GPa of CS to 1.490 GPa of CS-CB/SPF8 hybrid composite film. In conclusion, CB/SPF reinforced CS hybrid composite films are suitable for various purposes such as packaging, and agro-industrial applications, at a much lower cost while having sustainable environmental automotive benefit.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENCIRIAN DAN PEMBANGUNAN KOMPOSIT HIBRID KANJI UBI KAYU DIPERKUAT GENTIAN UBI KAYU (*Manihot esculenta* CRANTZ)/IJUK (*Arenga pinnata* (WURMB) MERR.)

Oleh

AHMED FARAJ IBRAHIM HISSEN EDHIREJ

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Pengerusi : Profesor Mohd Sapuan Salit, PhD
Fakulti : Kejuruteraan

Ubi kayu adalah pokok yang unik mempunyai pelbagai kegunaan yang termasuk pembuatan belbagai makanan bio-gentian, biokomposit dan biopolimer. Tambahan pula, ia memberikan satu sumber tenaga diperbaharui bagi kanji. Satu siri eksperimen telah dijalankan untuk pencirian kanji, kulit, hampas, polimer, komposit dan komposit hibrid daripada ubi kayu. Kaedah penuangan telah digunakan untuk pembikinan sampel. Lima fasa telah dijalankan untuk menyediakan komposit hibrid daripada gentian ubi kayu/enau dan kanji ubi kayu.

Fasa pertama telah dilaksanakan dengan mengkaji sifat fizikal, morfologi dan termal bagi kanji (CS), kulit (CP) dan hampas (CB) ubi kayu yang diperolehi daripada pokok ubi kayu Malaysia di samping komposisi kimia mereka. Kajian ciri-ciri termal menggunakan penganalisis gravimatrik termal (TGA) dan kalorimetri pengimbasan kebezaan (DSC) telah menunjukkan bahawa kanji dan gentian ubi kayu adalah stabil dari segi termal dengan titik pencairan masing-masing 172.87, 177.76 dan 169.23 ºC bagi kanji, kulit dan hampas. Kajian terhadap permukaan morfologi menunjukkan bahawa kanji ubi kayu berbentuk bulat dan membujur, hampas berbentuk poligon dan kulti menunjukkan bentuk bulat dan poligon. Kandungan karbohidrat dalam kanji adalah didapati sebanyak 86.47 g / 100g. Ia memperuntukkan bahawa kulit ubi kayu dan hampas mengandungi kepekatan kandungan hemiselulosa yang tinggi iaitu 23.38 dan 29.26% masing-masing. Ianya boleh disimpulkan bahawa, kanji ubi kayu dan serat adalah sesuai untuk membangunkan bahan dengan ciri-ciri mekanikal dan haba yang boleh diterima untuk pelbagai aplikasi seperti pembungkusan, automotif dan agro-industri.

Fasa kedua melibatkan penyiasatan kesan bahan pemplastik (fruktosa, urea, glykol Tri-etilena dan Triethanolamine) dengan kepekatan yang berbeza pada sifat-sifat fizikal, haba dan mekanikal filem berasaskan kanji ubi kayu. Kandungan lembapan, kelarutan air dan penyerapan air filem meningkat dengan peningkatan kandungan bahan pemplastik. Filem yang menggunakan pemplastik fruktosa menunjukkan rintangan air yang cemerlang berbanding pemplastik lain. Filem yang menggunakan 30% fruktosa menunjukkan ketumpatan paling tinggi (1.74 g / cm^3), kandungan air yang paling rendah (10.96%) dan penyerapan air yang paling rendah (110%). Filem yang mengandungi fruktosa menunjukkan permukaan yang licin tanpa liang. Kehabluran relatif menurun dengan peningkatan kandungan pemplastik. Filem yang mengandungi 30% fruktosa menunjukkan kehabluran relatif yang lebih tinggi (0.31). Filem yang mengandungi 30% fruktosa menunjukkan kekuatan tegangan tertinggi (4.7 MPa) dan modulus tegangan tertinggi (69 MPa). Oleh itu, fruktosa adalah ejen pemplastik yang paling berkesan di kalangan pelbagai pemplastik yang digunakan dalam kajian ini.

Fasa ketiga memberi tumpuan kepada kegunaan kulit ubi kayu dengan dua saiz zarah yang berbeza sebagai pengisi semula jadi untuk termoplastik kanji (TPS) berdasarkan kanji ubi kayu. Penambahan kulit menghasilkan peningkatan dalam ketebalan, kandungan air dan penyerapan air filem sambil mengurangkan ketumpatan dan keterlarutan air. Selain itu, imbasan mikroskop elektron menunjukkan bahawa filem-filem yang mengandungi kulit bersaiz kecil mempunyai struktur padat yang lebih baik dan permukaan yang homogen tanpa liang. Penambahan 6% kulit meningkatkan modulus elastik dan tegasan tegangan sehingga 449.74 dan 9.62 MPa, masing-masing, ini menjadi agen pengukuhan yang paling berkesan. Selain itu, perubahan suhu untuk parameter dinamik-mekanik komposit kanji/kulit ubi kayu disiasat menggunakan ujian DMA. Diperhatikan bahawa penambahan kulit meningkatkan kekuatan tegangan dan modulus. Kesimpulannya, filem komposit kanji/kulit ubi kayu adalah sesuai untuk pelbagai tujuan seperti pembungkusan, automotif dan agro-industri, pada kos yang lebih rendah.

Fasa keempat filem komposit berasaskan kanji ubi kayu telah disediakan dengan menggunakan sisa bergentian dari pengekstrakan kanji (hampas ubi kayu) sebagai pengisi. Filem komposit telah disediakan melalui teknik penuangan dengan menggunakan fruktosa sebagai pemplastik dan hampas dengan pelbagai saiz dan kepekatan. Saiz dan kepekatan hampas telah memberikan kesan yang signifikan terhadap sifat fizikal. Ianya menambahkan ketebalan, keterlarutan air, dan juga penyerapan air. Ia juga mengurangkan kandungan air dan ketumpatan filem. Walau bagaimanapun, tiada kesan signifikan penambahan hampas kepada sifat termal. Kajian XRD menunjukkan penambahan kehabluran komposit dengan penambahan kandungan gentian. Mikrograf SEM menunjukkan bahawa filem dengan saiz hampas yang kecil mempunyai struktur padat yang lebih baik dan permukaan homogenan. Modulus dan kekuatan tegangan maksimum filem komposit telah meningkat daripada 69.03 kepada 581.68 MPa dan 4.7 kepada 10.78 MPa, masing-masing, dengan penambahan sebanyak 6% hampas, menunjukkan ianya sebagai ejen pengukuhan yang paling berkesan kerana sifalt-sifat yang fizikal dan mekanikal yang bagus.

Untuk fasa terakhir, komposit hibrid telah berjaya disediakan dengan jumlah gentian yang berbeza, komposit hibrid (6% w / w kanji kering) mengandungi hampas ubi kayu dan (0, 2, 4, 6 dan 8% w / w kanji kering) gentian enau. Spesimen telah disediakan melalui teknik penuangan dengan menggunakan fruktosa sebagai pemplastik untuk kanji ubi kayu. Penambahan SPF telah memberikan pengaruh yang signifikan terhadap sifat fizikal. Ianya meningkatkan ketebalan di samping menurunkan ketumpatan, kandungan air, keterlarutan air dan penyerapan air untuk filem. Selain itu, kajian XRD menunjukkan peningkatan kehabluran komposit dengan penambahan kandungan gentian. Sifat mekanik filem telah ditingkatkan dengan penambahan gentian. Penggabungan SPF telah meningkatkan penghabluran relatif sehingga to 47%, berbanding 32% bagi filem CS. Nilai kebolehtelapan air (WVP) telah berkurangan dengan peningkatan kandungan gentian. Modulus dan kekuatan tegangan maksimum filem telah meningkat daripada 69.03 kepada 1114.6 MPa dan 4.7 kepada 20.72 MPa, masing-masing untuk filem mengandungi 6% CB dan 6% SPF, ini menjadikan ianya sebagai agen pengukuhan yang paling berkesan. Seterusnya, sifat dinamik-mekanikal bagi komposit hibrid telah dikaji menggunakan ujian DMA. Telah diperhatikan bahawa penggabungan SPF telah meningkatkan nilai modulus penstoran (E') daripada 0.457 GPa bagi CS kepada 1.490 GPa bagi filem komposit hybrid CS-CB/SPF8. Sebagai kesimpulan, filem komposit hibrid CS diperkuat CB/SPF adalah sesuai untuk pelbagai tujuan seperti pempakejan, automotif dan kegunaan industri agro pada kos yang rendah.

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This thesis was submitted to the Senate of the Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

Mohd Sapuan Salit, PhD
Professor Ir
Faculty of Engineering
University Putra Malaysia
(Chairman)

Mohammad Jawaid, PhD
Senior Lecturer
Institute of Tropical Forestry and Forest Products. INTROP
University Putra Malaysia
(Member)

Nur Ismarrubie Zahari, PhD
Associate Professor
Faculty of Engineering
University Putra Malaysia
(Member)

ROBIAH BINTI YUNUS, PhD
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School of Graduate Studies
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Signature: ______________________

Name of Member of Supervisory Committee: Dr. Mohammad Jawaid

Signature: ______________________

Name of Member of Supervisory Committee: Associate Professor Dr. Nur Ismarrubie Zahari

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LIST OF ABBREVIATIONS

CB	Cassava Bagasse
CP	Cassava Peel
CS	Cassava Starch
DMA	Dynamic mechanical analysis
DSC	Differential scanning calorimetry
DTG	Derivative thermogravimetry
E	Elongation at break
F	Fructose
FTIR	Fourier transform infrared
RC	Relative Crystallinity
SEM	Scanning electron microscope
SPF	Sugar Palm Fiber
TEA	Tri-Ethanol-Amine
TEG	Tri-Ethylene Glycol
TGA	Thermal-gravimetric analysis
TM	Tensile modulus
TPS	Thermoplastic starch
TS	Tensile strength
U	Urea
WA	Water Absorption
WC	Water Content
WS	Water Solubility

WVP	Water Vapor
XRD	X-Ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 Overview

Plastics produced from fossil sources have contributed significantly to environmental pollution caused by the accumulation of non-biodegradable solid waste in landfills. This significant problem has prompted a search for new biodegradable materials, not only in the food industry, but also in the medical, automotive, among others. Renewable natural raw materials including flour, starch, natural fibers, proteins, and others become an important alternatives and prominent in developing bioplastics (Tharanathan, 2003). Currently, there is growing interest in using raw materials and agricultural by-products such as from corn, cassava and potato tubers in biodegradable plastics. However, plastics developed from these sources have certain drawbacks of structural stability caused by its stiffness or weakness due to its high hygroscopicity and rapid aging when compared to conventional plastics (Villada, 2005). Therefore, research efforts must be maintained and increased in this field, with the use of local products such as cassava, which is studied in this, and other research projects. These studies intended that the methodology produce functional, applicable, and specific biodegradable plastics, which are reproducible on an industrial scale.

Cassava is a starchy root belonging to the Euphorbiaceae family and is one of the most important energy sources for tropical areas of the world. Although cassava thrives in fertile soil, its comparative advantage to other more profitable crops is their ability to grow in acidic soils of low fertility, with sporadic rainfall or long periods of drought. The crop is widely adapted as it has been planted successfully from sea level up to 1800 meters, at temperatures between 20 and 30˚C with an optimum of 24˚C, relative humidity between 50 and 90% with an optimum of 72% and an annual rainfall between 600 and 3000 mm with an optimum of 1500 mm (Navia and Villada, 2012). It is widely cultivated in tropical Africa, Asia and Latin America, and it is the fourth most important global crop in developing countries, with an estimated production in 2006 of 2.6 million tons. It is characterized by great diversity of uses both roots and leaves, which can be eaten by humans and animals. Cassava starch products can be used in many industries (Ceballos, 2002). Therefore, one way to extend the value of harvested cassava and improve rural economies is to process excess cassava for starch production. Its production and its byproducts provide an opportunity to develop value-added products for both food and industrial uses that will benefit rural communities. Cassava pulp or bagasse is a byproduct of starch production. Cassava bagasse contains 50–60% residual starch (dry wt basis). It may be dried in the sun and used as filler in cattle feed (Panichnumsin et al., 2010; Sriroth et al., 1999). However, much of the bagasse is simply discarded as a wasted resource (Panichnumsin et al., 2010). In recent years, the use of natural fibers as reinforcing materials in polymers and composites has attracted much attention. Compared to inorganic fillers, the main advantages of lignocellulosics are their renewable nature, biodegradability, low energy consumption, wide variety available throughout the world, low cost and density, as well as high specific strength and modulus (Bodirlau et al., 2013; Castillo et al., 2013; Gilfillan et al., 2012). Furthermore, they present high sound attenuation, and comparatively easy processing due to their flexibility and non-abrasive nature which allow high filling levels (Al-Oqla and Sapuan, 2014).

A study by Aripin et al, (2013) focused on the determination of the cassava peel potential to act as an alternative for fiber in paper and pulp with respect to the chemical properties. According to the results, there are lower amount of cellulose and holo-cellulose in cassava peels. However, these peels are rich in hemicellulose. Hence, we can conclude that there are significant parameters present in cassava peels, which can be effective as a fiber alternative for bio-composite and industries application. These industries can favor recyclable materials in the future, thereby reducing the environmental problems (Aripin et al., 2013). The cassava root is a rich source of starch, also containing proteins, lipids, fibers and sugars such as glucose, sucrose and fructose. Interest in biodegradable films development from renewable sources is growing due to increased environmental awareness. Among natural polymers, starch has been considered as one of the most promising materials because of its attractive combination of availability, price and performance (Abdillahi et al., 2013). Cassava is an important starch source in some countries like Brazil, Thailand, Malaysia, Indonesia and some regions of Africa. Currently there is growing interest to use raw materials and agricultural by-products from plants such as corn, cassava and potato tubers for biodegradable plastics. However, plastics developed from these sources have certain drawbacks caused by stiffness or weakness due to its high hygroscopicity and rapid aging which affect structural stability when compared to conventional plastics (Villada, 2005). Renewable, natural, raw materials including flour, starch, natural fibers, proteins, and others prominent alternative in developing options for bioplastics (Tharanathan, 2003). However, starch-based materials are brittle with poor mechanical characteristics. Thus, the incorporation of a plasticizer is required to overcome the brittleness of these materials. The principle function of plasticizers is to reduce inter- molecular forces and increase the mobility of polymer chains, which help to decrease the glass transition temperature (Tg) of plasticized starch materials, as well as, improve their flexibility. (Forssell et al., 1999; Garcia et al., 2000; Sanyang et al., 2015a; Sanyang et al., 2015c).

Recently, many studies have focused on the use of glycerol (Alves et al., 2007; Bergo et al., 2008; Fadeyibi et al., 2014; Jouki et al., 2013; Souza et al., 2012; Versino and García, 2014; Versino et al., 2015a), urea (Cao et al., 2009; Ma et al., 2006; Ma et al., 2005b; Wang et al., 2014), sorbitol (Kristo and Biliaderis, 2006; Mantzari et al., 2010; Müller et al., 2008), Fructose, glucose and sucrose (Galdeano et al., 2009; Veiga-Santos et al., 2007; Zhang and Han, 2006), xylitol (Fishman et al., 2000; Zhang and Han, 2006) as well as Triethelenglycol and Triethanolamin (Audic and Chaufer, 2005; Cao et al., 2009) as plasticizers of edible and/or biodegradable films. The proportion of plasticizer and its chemical nature influence the physical properties of the processed starch in two ways, viz. (i) by controlling its destructuration and depolimerization and (ii) by affecting the final properties of the material, such as its glass transition temperature and modulus. Since starch is attributed to offer a favorable combination of cost, availability and performance, it is considered to be very effective and valuable when compared to other polymers (Abdillahi et al., 2013).

The role of plasticizers is to enhance the flexibility and processability of starch by reducing the strong intermolecular interactions between starch molecules (Aguirre et al., 2013; Zhong and Li, 2014). As a result, the mobility of polymeric chains increases, which improves flexibility, extensibility and ductility of the plasticized films (Aguirre et al., 2013; Zhong and Li, 2014). Cassava starch like most other biopolymers is hydrophilic in nature due to either their hydroxyl or polar groups. The major challenges for the development of starches as packaging films are their shortcomings related to brittleness, processability, high moisture sensitivity, poor mechanical and barrier properties. The addition of plasticizers to starch films helps to decrease its inherent brittleness by reducing intermolecular forces, increasing the mobility of polymer chains, decreasing the glass transition temperature of these materials and improving their flexibility. The plasticizers must be compatible with the film-forming polymers. Hydrophilic compounds, such as polyols (glycerol and sorbitol) are commonly used in starch film, but sugars, amino acids and fatty acids could also be employed (Galdeano et al., 2009; Petersson and Stading, 2005).

The by-products from the sugar palm tree include foods and beverages, biopolymer, biocomposites, and timber. The sugar palm tree is a member of the Palmae family and naturally a forest species (Siregar, 2005) and it is considered as one of the most diverse multi-purpose trees (Sahari et al., 2012). Almost all parts of the tree can be used, such as the trunk, which provides important local products like sago, vinegar, fresh juice and palm sugar, which can be obtained from this versatile palm tree (Ishak, 2009). Traditionally, the sugar palm fiber has been used in various applications such as in brooms, rope, paintbrushes and roofs. More recently, the incorporation of several different types of fibers into a single matrix has led to the development of hybrid bio-composites. The behavior of hybrid composites is a weighed sum of the individual components, in which there is a more favorable balance between the inherent advantages and disadvantages. By combining sugar palm fiber with another fiber or using a hybrid composite that contains two or more types of fiber, the advantages of one type of fiber could complement with what are lacking in the other. As a result, a balance in cost and performance could be achieved through proper material design (Thwe and Liao, 2003). The properties of a hybrid composite mainly depend upon the fiber content, orientation, extent of intermingling of fibers, fiber to matrix bonding and arrangement of both the fibers. Ishak et al. (2011) have conducted a characterization study on the thermal and tensile properties of sugar palm fibers. In their studies, they found that the green fiber (matured fibers) from the plant give the greatest tensile results because of the optimum chemical composition, which consisted of high cellulose along with a hemicelluloses and lignin content (Ishak et al., 2012)

1.2 Problem Statements

Plastics have already substituted many traditional materials, such as wood, stone, leather, metal and ceramic various applications such as packaging, automotive and agro-industrial due to its good performances. Petroleum resources are the only major mineral commodities where many parties fear that their depletion will cause significant scarcities over the decades to come. Besides that, landfill sites are limited and plastics show to breakdown causing mountains of wastes in landfill. There are two approaches to solve this problem: incineration and recycling. Incineration of the plastic wastes produces large amount of carbon dioxide contributing to global warming, and sometimes produces toxic gases, which again contribute to global pollution. On the other hand, recycling is so expensive because it requires more labor and energy for removing of plastics wastes. Plastics must be separated according to the types of plastics, washed, dried, grinded and reprocessed to final product. For these reasons, raw materials that come from sustainable sources are now being considered as an alternative to the existing petroleum based polymers. Among natural polymers, starch has been considered as one of the most promising materials because of its attractive combination of availability, price and performance (Abdillahi et al., 2013; Lopez et al., 2014; Mohanty et al., 2013). Cassava was chosen due to its excellent properties including high purity level, excellent thickening characteristics, abundance, availability, and low cost, and it contains a high concentration of starch. However, starch-based materials are known to be brittle, with poor mechanical properties. Thus, the incorporation of a plasticizer is required to overcome the brittleness of these materials. In this study the effect of various plasticizers and concentration on the properties of thermoplastic cassava starch film have been studied.

Glycerol is most common type of plasticizer, but it did not give the best results when compare to other plasticizer molecules. Different plasticizers were chosen to optimize the most efficient plasticizer type and concentration. Among plasticizers used in this study, fructose provided higher tensile strength and modulus. The efficacy of plasticization is dependent on the molecular weight of the plasticizer since the smaller the molecule weight, the greater the plasticization effect of the plasticizer upon the polymer matrix. Among the samples tested, films plasticized by Tri-Ethylene Glycol exhibited the highest moisture content, followed by samples plasticized by Urea and Tri-Ethanol-Amine. In comparison, the fructose-plasticized films showed the lowest moisture content. This observation can be attributed to the different molecular weight and molecular structure of these plasticizers. The smaller molecule plasticizers such as Glycerol can be easily inserted between polymer chains when compared to larger plasticizer molecules such as fructose, which interrupts the formation of polymer–polymer hydrogen bonds. Even though synthetic fibers are very useful in composite materials and extensively used due to their excellent mechanical, barrier and thermal properties, there are not biodegradable and causes negative effect to the environment. The issues of climate changes and global warming due to pollution and the activity of de-forestation becomes the major reason to use agriculture by-product. The incorporation of cassava peel and bagasse as reinforcement agents in starch based materials aims to enhance their final properties. Hence, matrix-filler compatibility expected, due to their similar chemical nature. Which increased overall strength.

Although this bio-composites exhibited acceptable properties, it still has poor water resistance. To overcome this problem, hybridization the CB with lower hydrophilic natural fiber, the natural fiber that known for their high durability and resistance to water is sugar palm fiber. In term of cost, synthetic fibers are much costly when compared to natural fibers. For these reasons, enormous efforts, time and money have been spent on research to discover a new green material that can replace synthetic fibers. Several studies have shown that sugar palm fibers have great potential to be used as reinforcement in many composite applications. Sugar palm fibers have some advantages over traditional reinforcement fiber materials in terms of cost, density, being renewable, non-toxic, abrasiveness and biodegradability. (Leman et al., 2008b). This task is easier because many of the typical applications of these composites do not require excellent mechanical properties, such as some packaging, gardening items, etc. (Kim et al., 2005). In addition, synthetic fiber has negative effects on human health especially to the skin and eye. More seriously, inhalation of synthetic fibers can cause long term or deadly effects such as lung cancer (Ishak, 2009).

1.3 Research Objectives

The objectives of this research are as follows:

1. Extraction and characterization of starch and fibre (peel and bagasse) from cassava root planted in Malaysia and exploring their potential to be used in the development of bio-composites. .
2. Investigate the effect of various plasticizers type and concentration on physical, thermal, mechanical and structural properties of cassava starch based films.
3. Development and characterization of cassava fiber (peel/bagasse) reinforced cassava starch composites at different fiber loading.
4. Development and characterization of cassava/sugar palm fiber reinforced cassava starch hybrid composite at different SPF loading.

1.4 Significance of Study

The significance of this study is to utilize the cassava starch and its byproduct (peel and bagasse) as a replacement for more harmful non-natural composite product. This natural material is an important material in developing the biocomposites products, alone or combined with another material, to produce green composites. The advantages of natural fibers, such as their low cost, low density, abundance, sustainability, recyclability and biodegradability, make them an interesting and necessary research area. The earth needs biocomposites that are low in cost to produce, renewable, biodegradable and environmentally friendly. Replacing petroleum based polymers with the thermoplastic starch may reduce petroleum based polymer production and hence minimise the negative health effects generated from it and reduce the dependence on the petroleum products. During the last decade, natural fibres have attracted the attention of scientists and researchers because of the natural fiber's advantages over conventional reinforcing materials. One of the natural fibres widely available is sugar palm fibres. Although the SPF is currently used for the tradisional application such as ropes, this research can also help to solve the problem of handling by-product from the sugar palm tree. It also plays an important role in the economic life of poor rural people by increasing their income and employment (Ishak et al., 2013; Mogea et al., 1991). Limited studies have been reported relating to natural fiber development as a green packaging material. Utilization of CP, CB and SPF, thus, may contributes to solve the waste problem and, at the same time, enhance economic development through a waste to wealth transformation. Moreover, this research would help to evaluate a new hybrid composites based natural resources that come from the waste of cassava and sugar palm plants and turn them into the new valuable green products.

1.5 Scope and Limitation of Study

The scope of this research is to study the potential of using a cassava starch and its by-product as bio-composites. The CS was extracted from cassava tuber planted in Malaysia and cassava fibers (peel and bagasse) were obtained from the same process. The physical, morphological and thermal properties of CS, CP and CB along with their chemical composition were studied. The effect of different plasticizers type (F, TEA, U and TEG) with concentration of 30, 45 and 60 % w/w dry starch, on the physical, thermal, tensile and structural properties of cassava starch based-film were investigated. The selection of combination was mainly based on physical and tensile properties. Then, the optimum combination of plasticized CS was used to reinforce CB and CP with different fiber loadings. Two different particle size and different fiber loadings were prepared at 3, 6 and 9 % w/w dry starch by using a casting technique. Then, the best combination of composite film among the different fiber type (peel and bagasse) and loading was selected based on the physical and tensile properties which was supported by thermal and structural properties. The selected composites films were hybrid with sugar palm fiber (SPF) in different concentration (2, 4, 6 and 8 % w/w dry starch). These specimens were tested for their mechanical and thermal properties through a tensile and thermogravimetric analysis (TGA). The use of other investigations techniques such as XRD, FTIR and SEM were used to support the results of mechanical properties. Finally, the CS-CB/SPF hybrid composites were submitted to WVP and a biodegradation test to investigate their environmental effect.

1.6 Structure of the thesis

The thesis layout is in accordance with Universiti Putra Malaysia alternative thesis format based on publications, in which each research chapter (4 – 9) represent a separate study that has its own: ‘Introduction’, ‘Materials and methods’, ‘Results and discussion’, and ‘Conclusion’. The details of the thesis structure are presented below.

Chapter 1

The problems statement and the research objectives were clearly highlighted in this chapter. In addition, the significant contribution and scope of study were also illustrated within the chapter.

Chapter 2

A comprehensive literature review on essential areas connected to the topic of this thesis was presented in this chapter.

Chapter 3

This methodology chapter covers every single activity related in this research; from the beginning of material preparing to material processing, testing and results analysis are detailed in this section

Chapter 4

This chapter presents the article entitled "Extraction of Malaysian Cassava Starch. Peel and Bagasse and Its Characterization". In this article, the study of the physical, morphological and thermal properties of cassava starch, peel and bagasse obtained from Malaysian plant along with their Chemical Composition was accomplished.

Chapter 5

This chapter presented the second article entitled 'Effect of Various Plasticizers and Concentration on The Physical, Thermal, Mechanical and Structural Properties of Cassava Starch Based Films'. In this article, the effect of four different plasticizer type (Fructose, Urea, Tri-ethyleneglycol and Triethanolamine) and different concentrations (30, 45 and 60%) on the properties of cassava starch films was investigated.

Chapter 6

In this chapter, the article entitled 'Preparation and Characterization of Cassava Starch/Peel Composite Films' is presented. This article focused on the use cassava peel with two different particle size as a natural filler for thermoplastic starch (TPS) based on the cassava starch.

Chapter 7

This chapter is focused on a work entitled ‘Preparation and Characterization of Cassava Bagasse Reinforced Thermoplastic Cassava Starch’. This article studied the effect of various loading of cassava bagasse with two different particle size on the properties of thermoplastic cassava starch.

Chapter 8

This chapter presents the fifth article entitled ‘Cassava/Sugar Palm Fiber Reinforced Cassava Starch Hybrid Composite: Physical, Thermal and Structural Properties’. The hybrid composites were successfully prepared, contains (6% dry starch) CB and (0, 2, 4, 6 and 8 % dry starch) SPF. The effect of the addition of SPF on the Physical, Thermal and Structural Properties of hybrid composites films was investigated.

Chapter 9

This chapter presents the last article entitled ‘Tensile, Barrier, Dynamic Mechanical and Biodegradation Properties of Cassava/Sugar Palm Fiber Reinforced Cassava Starch Hybrid Composites’. The effect of the incorporation of sugar palm fiber on the Tensile, Barrier, Dynamic Mechanical and Biodegradation Properties was carried out in this chapter.

Chapter 10

Finally, the overall conclusions from the various research articles as well as relevant suggestions for future research were presented in this chapter.

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