

BIODEGRADABLE PACKAGING FROM NAPIER GRASS: ENVIRONMENT PROPERTIES



BACHELOR OF MANUFACTURING ENGINEERING TECHNOLOGY WITH HONOURS

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BIODEGRADABLE PACKAGING FROM NAPIER GRASS: ENVIRONMENT PROPERTIES

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2022

DECLARATION

I declare that this thesis entitled "Biodegradable Packaging from Napier Grass: Environment Properties" is the result of my own research except as cited in the references. The work has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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DEDICATION

To Al-Quran, the greatest source of knowledge

Bring me sheets of iron" - until, when he had leveled [them] between the two mountain walls, he said, "Blow [with bellows]," until when he had made it [like] fire, he said, "Bring me, that I may pour over its molten copper."

(Al-Kahf: Verse 96)

Alhamdulillah

Praise to Allah for the strength, guidance and knowledge that was given by Allah for

me to complete this study

To my beloved parents for every support that was given to me

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completing this research.

&

To all people who support me throughout my journey.

ABSTRACT

Recent years have seen an increase in the need for eco - friendly products as a result of the accumulation of non-biodegradable waste, especially disposable products. As a result, several environmentally friendly materials have been established to solve this problem. Biopolymer developed from renewable resources has the potential to be a viable alternative to petroleum-based polymers due to its high biodegradability and consequently environmental friendliness. Among other biodegradable materials, cassava starch biopolymer has been identified as a fully biodegradable substance that can be generated by a variety of plants and is one of the most abundant renewable, biodegradable, and costeffective resources accessible. Due to widespread concern about environmental pollution, thermoplastic cassava starch is now widely study in packaging materials, with some formulations including bio-fillers or fiber to strengthen the bio-based plastic. As a consequence, Napier grass fiber is employed to reinforce a bio-based composite. Napier grass is regarded for its high yield, and resistance to insects. Additionally, this tropical perennial grass has a significant fiber reserve as a result of vegetative regeneration after stem removal. Numerous early studies on the characteristics and properties of Napier grass have been published; nevertheless, a comprehensive and in-depth examination of this tropical grass's use as a non-wood packaging replacement is nearly unknown. The aim of this study is to develop biodegradable thermoplastic cassava starch reinforced with Napier grass fiber, in order to investigate its water affinity properties, morphology, density, and environmental properties. To strengthen the cassava starch biopolymer's shortcomings, biocomposites has been developed by incorporating 0,10,20,30,40, and 50wt% of Napier grass fiber into thermoplastic cassava starch matrix. All components were mixed uniformly, and the components were formed utilizing hot compression molding. The functional properties of TPCS/NGF biopolymer composites were then evaluated to determine their suitability as biodegradable materials. The 50% of fiber has the lowest moisture content. Water absorption showed that when fiber content is increased, then the water absorbed is decreased. Water solubility testing demonstrates a decrease in weight loss when fiber content is increased. For soil burial tests, all samples were decreases as the fiber content increases. The FTIR spectrum indicates the presence of chemical bonding between fiber and matrix, whilst the SEM micrograph indicates a change in the structure of the composite as the fiber concentration increases. In general, the present study's results indicated that TPCS/NGF has the ability to significantly enhance the composite's qualities. To summarize, TPCS/NGF may be a viable alternative material for biodegradable products, such as disposable packaging trays with increased features.

ABSTRAK

Beberapa tahun kebelakangan ini telah menyaksikan peningkatan dalam keperluan produk mesra alam akibat daripada pengumpulan sisa tidak terbiodegradasi, terutamanya produk pakai buang. Hasilnya, beberapa bahan mesra alam telah diwujudkan untuk menyelesaikan masalah ini. Biopolimer yang dibangunkan daripada sumber boleh diperbaharui berpotensi menjadi alternatif yang berdaya maju kepada polimer berasaskan petroleum kerana kebolehbiodegradasiannya yang tinggi dan seterusnya mesra alam. Antara bahan terbiodegradasi lain, biopolimer kanji ubi kayu telah dikenal pasti sebagai bahan terbiodegradasi sepenuhnya yang boleh dijana oleh pelbagai tumbuhan dan merupakan salah satu sumber yang boleh diperbaharui, terbiodegradasi dan kos efektif yang paling banyak boleh diakses. Disebabkan kebimbangan meluas tentang pencemaran alam sekitar, kanji ubi kayu termoplastik kini digunakan secara meluas dalam bahan pembungkusan, dengan beberapa formulasi termasuk pengisi bio atau gentian untuk mengukuhkan plastik berasaskan bio. Oleh itu, serat rumput Napier digunakan untuk mengukuhkan komposit berasaskan bio. Rumput napier dianggap sebagai hasil yang tinggi, dan ketahanan terhadap serangga. Selain itu, rumput tropika ini mempunyai rizab gentian yang ketara hasil daripada penjanaan semula vegetatif selepas penyingkiran batang. Banyak kajian awal tentang ciri dan sifat rumput Napier telah diterbitkan; namun begitu, kajian menyeluruh dan mendalam tentang penggunaan rumput tropika ini sebagai pengganti pembungkusan biodegradasi hampir tidak diketahui. Matlamat kajian ini adalah untuk membangunkan kanji ubi kayu termoplastik terbiodegradasi diperkukuh dengan serat rumput Napier, untuk menyiasat sifat pertalian air, morfologi, ketumpatan, dan sifat persekitarannya. Untuk mengukuhkan kekurangan biopolimer kanji ubi kayu, biokomposit telah dibangunkan dengan menggabungkan 0,10,20,30,40, dan 50wt% serat rumput Napier ke dalam matriks kanji ubi kayu termoplastik. Semua komponen dicampur secara seragam, dan komponen dibentuk menggunakan acuan mampatan panas. Sifat kefungsian komposit biopolimer gentian rumput TPCS/Napier kemudiannya dinilai untuk menentukan kesesuaiannya sebagai bahan terbiodegradasi. 50% serat mempunyai kandungan lembapan paling rendah. Penyerapan air menunjukkan apabila kandungan serat meningkat, maka air yang diserap akan berkurangan. Ujian keterlarutan air menunjukkan penurunan dalam penurunan berat badan apabila kandungan serat meningkat. Selepas 4 dan 8 minggu pengebumian tanah, semua sampel yang diuji telah kehilangan berat badan dan boleh merosot. Spektrum FTIR menunjukkan kehadiran ikatan kimia antara gentian dan matriks, manakala mikrograf SEM menunjukkan perubahan dalam struktur komposit apabila kepekatan gentian meningkat. Secara umumnya, keputusan kajian ini menunjukkan bahawa TPCS/NGF mempunyai keupayaan untuk meningkatkan kualiti komposit dengan ketara. Ringkasnya, TPCS/NGF mungkin merupakan bahan alternatif yang berdaya maju untuk produk terbiodegradasi, seperti pembungkusan pakai buang dengan ciri yang dipertingkatkan.

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

TABLE OF CONTENTS

	PAGE
DECLARATION	
APPROVAL	
DEDICATION	
ABSTRACT	i
ABSTRAK	ii
ACKNOWLEDGEMENTS	iii
TABLE OF CONTENTS	iv
LIST OF TABLES	vii
LIST OF FIGURES	ix
LIST OF APPENDICES	xiv
CHAPTER 1 INTRODUCTION	15
1.1 Background	15
1.2 Problem Statement	18
1.3 Objective	20
1.4 Scope of Research	20
1.5 Scope of Study Colline RENIKAL MALAY SIA MELAK	A 21
1.6 Structure of Thesis	21
Chapter 1	21
Chapter 2	22
Chapter 3	22
Chapter 5	22
	22
2.1 Introduction	23 23
2.1 Introduction 2.2 Polymer	23 24
2.2.1 Classification and Types of Polymers	24
2.2.2 Synthetic Polymer	25
2.2.3 Thermoplastic and Thermosets	25
2.2.4 Application Polymer	29
2.3 Composite	30
2.3.1 What is a Composite?	30
2.3.2 Classification of Composite Material	31
2.3.3 Polymer Matrix Composite (PMCs)	32
2.3.4 Application of Composite	33

2.4	Fiber	34
	2.4.1 Synthetic Fiber	35
	2.4.2 Natural Fiber	36
2.5	Napier Grass	38
	2.5.1 Napier Grass Fiber Origin, Propagation and Distribution	39
	2.5.2 Characteristic of Napier Grass Fiber	40
	2.5.3 Application of Napier Grass Fiber	44
	2.5.4 Napier Grass Composite	45
2.6	Starch	52
2.7	Thermoplastic Starch	55
	2.7.1 Polymerization of Starch	55
	2.7.2 Thermoplastic Potato Starch	57
	2.7.3 Thermoplastic Cassava Starch	62
	2.7.4 Application of Thermoplastic Starch	63
2.8	Waxes	64
	2.8.1 Synthetic Waxes	67
	2.8.2 Natural Waxes	68
•	2.8.3 Application of Waxes	72
2.9	Plasticizer	73
	2.9.1 Glycerol	74
2 10	2.9.2 Sorbitol	11
2.10	Summary	80
СНА	PTER 3 METHODOLOGY	81
3.1	Introduction	81
3.2	Material A Annual Content in the second	83
	3.2.1 Napier Grass Fiber	83
	3.2.2 Cassava Starch	85
	3.2.3 Glycerol SITI TEKNIKAL MALAYSIA MELAKA	85
	3.2.4 Beeswax	86
3.3	Preparation of Samples	87
	3.3.1 Preparation of Thermoplastic Cassava Starch	87
	3.3.2 Preparation of Thermoplastic Cassava Starch with Beeswax	89
	3.3.3 Preparation of Thermoplastic Cassava Starch Reinforced with Napier	
	Grass Fiber	90
3.4	Characterization of Samples	91
	3.4.1 Moisture Content	91
	3.4.2 Water Absorption	91
	3.4.3 Thickness Swelling	92
	3.4.4 Water Solubility	93
	3.4.5 Soil Burial	95
	3.4.6 Fourier Transform Infrared Spectroscopy (FTIR)	96
	3.4.7 Scanning Electron Microscopy (SEM)	97
	3.4.8 Density	97
	3.4.9 Process Production of the Packaging Tray	98
CHAI	PTER 4 RESULTS AND ANALYSIS	100
4.1	Introduction	100

4.2	Water Affinity Testing	100
	4.2.1 Moisture Content	100
	4.2.2 Water Absorption	101
	4.2.3 Thickness Swelling	103
4.3	Environmental Analysis	105
	4.3.1 Water Solubility	105
	4.3.2 Soil Burial	106
4.4	Physical Analysis	108
	4.4.1 Fourier-Transform Infrared Spectroscopy (FTIR)	108
	4.4.2 Scanning Electron Microscopy (SEM)	110
	4.4.3 Density	113
	4.4.4 Fabrication of Packaging Tray	115
СНА	APTER 5 CONCLUSION AND RECOMMENDATION	117
5.1	Conclusion	117
5.2	Recommendation for Future Research	118
5.3	Project Potential	119
5.4	Lifelong Learning	122
REF	ERENCES	123
		1 50
APP	ENDICES	159
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	اويتوم سيتر تتكنيكا مليسيا ملاك	
	LINIVEDSITI TEKNIKAL MALAVSIA MELAKA	
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LIST OF TABLES

TABLETITLE	PAGE
Table 2.1 Comparison between thermoplastics and thermosets (Shrivastava, 2018)	29
Table 2.2Natural fibers characteristics summary (Peças et al., 2018)	37
Table 2.3 Chemical, physical and mechanical properties for Napier grass (Lim et a	l.,
2020)	41
Table 2.4 Physical properties of Napier grass fiber	42
Table 2.5 Comparison of the tensile properties of Napier grass fiber with that of ot	her
natural fibers	43
Table 2.6 Chemical properties of Napier grass fiber	44
Table 2.7 Tensile properties of untreated and treated Napier grass fiber composites	48
Table 2.8 Botanical sources of starch and their corresponding amylose/amylopectin	n
ratio, and crystallinity.	54
Table 2.9 Physiochemical properties of potato starch obtained by proximal analysis	s 59
Table 2.10 Thermoplastic potato starch composites	61
Table 2.11 Main chemical composition of jute fiber and kapok fiber	62
Table 2.12 Thermoplastic cassava starch composites	63
Table 2.13 Applications of natural waxes	67
Table 2.14 Characteristics of beeswax (Tinto et al., 2017)	69
Table 2.15 Composition of unhydrolyzed beeswax (Tinto et al., 2017)	70
Table 2.16 Chemical composition of carnauba wax based on references.	72
Table 2.17 Chemical properties of glycerol at 20.1 °C (Gupta & Kumar, 2012)	74
Table 2.18 General properties of sorbitol	77

Table 3.1 Chemical composition of glycerol from QReC Chemical	86
Table 4.1 The analysis of variance (ANOVA) of moisture content	101
Table 4.2 The analysis of variance (ANOVA) of water absorption	103
Table 4.3 The analysis of variance (ANOVA) of thickness swelling	104
Table 4.4 The analysis of variance (ANOVA) of water solubility	106
Table 4.5 The analysis of variance (ANOVA) of soil burial.	108
Table 4.6 The analysis of variance (ANOVA) of density.	114
Table 5.1Total Cost of Raw Material for One Tray	119



LIST OF FIGURES

FIGURE	TITLE	PAGE
Figure 2.1 Classification	of Polymer	25
Figure 2.2 Molecular strue	cture of thermoset (Karuppiah, 2016)	27
Figure 2.3Molecular struc	cture of thermoplastic (Karuppiah, 2016)	28
Figure 2.4Depiction of the	ermoplastic monomer bond and thermoset crosslinked	
covalent bond	(Landis, 2018)	28
Figure 2.5 Classification	of composite (Khayal, 2019)	32
Figure 2.6 Classification	of fiber	35
Figure 2.7 Classification	of natural fiber	38
Figure 2.8 Napier Grass		39
Figure 2.9 Regional distri	bution of Napier grass around the world (Negawo et al.,	
2017)	اويومرسيني بيصيصل مليس	40
Figure 2.10 Napier grass	fiber (Reddy et al., 2012) LAYSIA MELAKA	41
Figure 2.11 SEM of Napi	er grass fibers (Haameem et al., 2016)	42
Figure 2.12 Weight gain a	as a function of the square root of time for the untreated a	nd
treated Napier	grass composites (M. Haameem, et al., 2016)	46
Figure 2.13 Tensile stress	s as a function of moisture exposure period (days) for	
untreated Nap	vier grass composites (Haameem et al., 2016).	47
Figure 2.14 Tensile stress	as a function of moisture exposure period (days) for	
treated Napier	grass composites (Haameem et al., 2016)	48

Figure 2.15 SEM micrographs displaying the fractured treated Napier grass

composites that had been immersed in water for (a) 4 days, (b) 8 days, (c)

12 days, and	(d)14 days (ful	y saturated) (M. Haa	ameem et al., 2016) 5	50
--------------	-----------------	----------------------	-----------------------	----

Figure 2.16 SEM micrographs displaying the fractured untreated Napier grass
composites that were (a) dry, and had been in immersed in water for (b) 6
days, (c) 12 days, and (d)23 days (fully saturated) (Haameem et al.,
2016)

Figure 2.17 Starch granule from SEM image with scale bar of 10 µm (Wang et al.,

2012) WALAYSIA	53
Figure 2.18 Molecular structure of amylose (Pokhrel, 2015)	53
Figure 2.19 Molecular structure of amylopectin (Pokhrel, 2015)	54
Figure 2.20 Starch thermal processing melt-mixing (García, 2019)	56
 Figure 2.21 Molecular structures of the amylose (A) and the amylopectin (B). Numbers (1–6) in the first glucose unit of the amylose show numbering of carbon atoms in glucose molecule. Figure 2.22 SEM images of the various plasticized starch systems and corresponding plasticized starch nano-bio composites: (A) G0; (B) G3; (C) G5; (D) G7; (E) P0; (F) P3; (G) P5; (H) P7; (I) S0; (J) S3; (K) S5 and (L) S7. Scale 	58
bars are 5 or 10 microns (Ren et al., 2018).	60
Figure 2.23 TGA graphics (Velásquez Herrera et al., 2017)	61
Figure 2.24 Classification of waxes into two categories (Endlein, 2011)	66
Figure 2.25 Carnauba wax DSC curve extracted (Basson & Reynhardt, 2014)	71
Figure 2.26 DSC curve of carnauba wax extracted (Zheng et al., 2011)	71

Figure 2.27 FTIR spectra reading of (i) CS, (ii) CS-Gly, (iii) CS-Thy and (iv) CS-	
Gly-Thy films (Nordin et al., 2020)	75
Figure 2.28 Mechanical properties of corn starch with glycerol and/or thymol:	76
Figure 2.29 Thermogravimetric curves of CS, CS-Gly, CS-Thy and CS-Gly-Thy	
films (Nordin et al., 2020)	77
Figure 2.30 Thickness test of sugar palm starch with glycerol or sorbitol (Sanyang et	
al., 2015)	78
Figure 2.31 Density analysis of sugar palm starch films and with additional of	
glycerol or sorbitol (Sanyang et al., 2015)	78
Figure 2.32 Percentage of solubility test for sugar palm starch films with glycerol	
and sorbitol (Sanyang et al., 2015)	79
Figure 2.33 Percentage of moisture content for sugar palm starch films with glycerol	
and sorbitol (Sanyang et al., 2015)	79
Figure 2.34 Thermal-Gravimetric analysis of sugaar palm starch films with glycerol	
and sorbitol (Sanyang et al., 2015)	79
Figure 3.1 Flow of Research Methodology	82
Figure 3.2 Napier grass at Jasin, Asahan	84
Figure 3.3 Process of extracting Napier grass fiber	84
Figure 3.4 Cassava Starch	85
Figure 3.5 Glycerol contained 99.5% AR grade.	86
Figure 3.6 Beeswax	87
Figure 3.7 Preparation of the Mixture of Thermoplastic Cassava Starch (TPCS)	88
Figure 3.8 Fabrication of the Mixture of Thermoplastic Cassava Starch (TPCS)	88

Figure 3.9 Preparation of the Mixture of Thermoplastic Cassava Starch with	
Beeswax	89
Figure 3.10 Fabrication of the Mixture of Thermoplastic Cassava Starch with	
Beeswax	89
Figure 3.11 Fabrication of Thermoplastic Cassava Starch Reinforced by Napier	
Grass Fiber	90
Figure 3.12 Methodlogy of Moisture Content	91
Figure 3.13 Methodology of Water Absorption	92
Figure 3.14 Methodology of Thickness Swelling	93
Figure 3.15 Methodology of Water Solubility	94
Figure 3.16 Methodology of Soil Burial	96
Figure 3.17 FTIR Spectroscopy Machine	96
Figure 3.18 Scanning Electron Microscopy (SEM) Machine	97
Figure 3.19 Methodology of Density	98
Figure 3.20 Production of Packaging Tray	99
Figure 4.1 Percentage of Moisture Content of TPCS/Napier grass fiber with different	
fiber loading	101
Figure 4.2 Percentage of Water Absorbed of TPCS/Napier grass Fiber with different	
fiber loading for 0.5 hour and 2 hours.	103
Figure 4.3 Percentage of Thickness Swelling of TPCS/Napier grass fiber with	
different	104
Figure 4.4 Result of Water Solubility of TPCS/Napier grass fiber with different	
percentage of fiber loading	106

Figure 4.5 Percentage of weight loss for soil burial testing of TPCS/NGF in 4 and 8			
weeks	108		
Figure 4.6 FTIR Spectroscopy of TPCS/NGF composite with different percentage of			
fiber loading	110		
Figure 4.7 SEM micrograph of NGF	112		
Figure 4.8 Scanning electron micrograph of TPCS reinforced by NGF composites (a))		
0% fiber content, (b) 10% fiber content, (c) 20% fiber content, (d) 30%			
fiber content, (e)40% fiber content, and (f) 50% fiber content.	113		
Figure 4.9 Density of TPCS/NGF composite with different percentage of fiber			
loading	114		
Figure 4.10 TPCS with 30% Napier grass fiber sample tray	115		
Figure 4.11 Perspective view	115		
Figure 4.12 Back view	116		
Figure 4.13 Corner view	116		
Figure 5.1 TPCS with 30% Napier grass fiber sample tray	119		
Figure 5.2 Application of TPCS with Napier grass fiber as skincare, lipstick, and			
remote-control organiser	120		
Figure 5.3 Survey on potential project with the owner Warung Sambal Enterprise	120		
Figure 5.4 Survey question at Warung Sambal Enterprise			
Figure 5.5 Survey on potential project with Encik Hizati Hamrom			
Figure 5.6 Survey question at Cofeeology			

LIST OF APPENDICES

APPENDIX	TITLE	PAGE
APPENDIX A	GANTT CHART PSM 1 AND PSM 2	159
APPENDIX B	TURNITIN REPORT	161



CHAPTER 1

INTRODUCTION

1.1 Background

Biodegradable products have been the subject of studies in recent years as a possible replacement for petroleum-based plastics in packaging applications. Biopolymers are the most promising commodity for this reason due to their biodegradability and long shelf life properties such as tolerance to chemical or enzymatic reactions (Khan et al., 2017). These issues have led to new research on the development of biodegradable materials.

Globally, plastic manufacture and distribution have been gradually rising through time. Petroleum-based plastics are often used as single-use plastics in our everyday lives since they have developed an incredible amount of utility owing to their adaptability, durability, flexibility, and toughness (Sahari et al., 2013). Furthermore, they are inexpensive on the market and readily available in any grocery shop. Marichelvam et al., (2019) stated that plastic is commonly used in a variety of sectors, most notably the packaging industry; the global output of petroleum-based plastic exceeded 300 million tons until 2015, with only 1% being bioplastic. The fast increase in plastic consumption is a result of the variety of plastic items now available on the market, which range from home and personal goods to packaging and building materials. The widespread usage of plastics has resulted in an abundance of plastic waste in the environment. Based on research about Biodegradable Tray by Ferreira et al., (2020), this could eventually result in significant worldwide problems for the environment and people, since the disintegration rate of these materials is very slow, about 100 years, owing to their hydrophobic characteristics and their ability to effectively escape quick microbial activity.

To tackle this problem, it is necessary to make a switch from petroleum-based plastic to biodegradable plastic in order to preserve a healthier environment for future generations and also to give more plastic disposal alternatives. Among biopolymers, starch is one of the most promising prospects and alternatives to petroleum-based plastics, owing to the fact that starch is fully biodegradable and plentiful in nature. It is abundant in plants such as maize, cassava, potato, and tuber, among others. According to López et al., (2019), starch is stored in plants in the granule-packed state of amorphous and crystalline. Due to widespread concern about environmental pollution, starch-based bioplastics such as thermoplastic starch (TPS) are increasingly widely employed in packaging materials, with some formulations including biofillers or fiber to strengthen the bio-based plastic.

Starch is mostly employed as a matrix or resin in biocomposites structures during the manufacturing of biopolymers. Numerous varieties of natural starch have been researched during the last several decades, including cassava starch, maize starch, and sugar palm starch. However, when compared to other sources of starch, cassava starch contributes the most in terms of productivity output (Jumaidin et al., 2020). Starch is a versatile material since it can be converted to chemicals such as ethanol, acetone, and organic acids utilized in the creation of synthetic polymers such as polylactic acid (PLA) (Carvalho, 2008). As well as turned to thermoplastic starch (TPS) with the assistance of a plasticizer under shear temperature conditions. Numerous research on thermoplastic starch (TPS) have been undertaken extensively and worldwide for a variety of starch sources, including cassava, potato, and maize (Bergel et al., 2017) (Asrofi et al., 2020).

Natural fibers derived from plants have enormous potential for use in the plastics, automotive, and packaging industries due to their superior properties such as low density, high specific stiffness, good mechanical properties, biodegradability, eco-friendliness, toxicological safety, and excellent thermal and acoustic insulation (Thakur et al., 2013) (Mohanty et al., 2004). Consequently, these cellulosic fibers have a lower cost of material than the beginning polymer. At the melting point of the majority of polymers, natural fibers disintegrate. As a result, it is prudent to investigate the thermal stability of natural fibers prior to contemplating their use as reinforcement in thermoplastic matrices. Rajulu et al., (2002) investigated the thermal degradation of Hildegardia, Bamboo, and Tamarind fruit fibers and concluded that they were suitable for use as reinforcements in polyolefin matrices. Yang et al., (2007) investigated the pyrolysis properties of wood materials.

Pennisetum purpureum fiber, commonly known locally as Napier grass, has 46% cellulose, 34% hemicellulose, and 20% lignin (Reddy et al., 2012). It may be harvested 3–4 months after planting and can be harvested at intervals of 6–8 weeks for up to 5 years, yielding 40 tons of dry biomass per hectare each year. Each plant produces around 40% fiber. The exceptional high modulus of these fibers was a primary rationale for their selection (Rajulu, 2009). Fibers were extracted from the internodes of Napier grass stems. Water affinity testing, environmental analysis, and physical analysis were used to achieve this.

Due to the environmental concerns associated with conventional thermoplastics, the production of biodegradable thermoplastic materials is accelerating. Biodegradable materials are both safe for the user and the climate. Thus, it is prudent to minimize the use of non-biodegradable plastic and encourage biodegradable plastic.

1.2 Problem Statement

The widespread use of non-biodegradable materials has had a detrimental effect on humanity and the climate. Non-biodegradable materials are composed of petroleum-based plastic polymers that are detrimental to the atmosphere due to their inability to dissolve in landfills. The issue arises in the landfill as these synthetic polymers persist for an extended period of time and interfere with groundwater, forming toxic substances and affecting drinking water safety (Emadian et al., 2017). In landfills, non-biodegradable materials require hundreds of years to decompose. Additionally, a disadvantage of utilizing synthetic polymers is that they contain toxic chemicals and release poisonous gases during the incineration phase. Essentially, most polymers are produced from petroleum, which requires additional fossil fuels, resulting in emissions (Marichelvam et al., 2019).

However, the downsides of natural fibers are their high moisture sensitivity, low chemical resistance, low thermal breakdown temperature, low wettability, and incompatibility with other composite materials during composite processing. Based on Singha et al., (2009) research, these effects have a major effect on the strength of the fiber-matrix interface. Thus, their inclusion into a polymer matrix requires the fibers to be treated physically or chemically to overcome interfacial incompatibility. John & Anandjiwala, (2008) stated that the use of various physical testing and chemical treatments results in a decrease in moisture absorption as well as modifications to the fiber surface. Understanding the physicochemical qualities and mechanical behavior of natural fibers is critical for optimizing the performance of composites. The majority of research has been conducted to determine the effect of fiber treatment on its chemical composition, surface morphology, crystallinity, and mechanical properties (John & Anandjiwala, 2008).

According to Negawo et al., (2017), Napier grass is a significant tropical forage crop. It is heavily used in cut and carry feeding systems and is gaining traction in other agricultural systems. It is resistant to prolonged cutting and regenerates quickly, yielding palatable leafy shoots. As a result, improving the knowledge-based use and management of accessible Napier grass capital promises. The production of Napier grass must be completely exploited, as certain sections of the grass, especially the stem, may be used as Napier grass fiber to produce composite materials.

It is essential to produce a commodity made entirely of biodegradable materials when adhering to environmental regulations. Numerous researchers and studies have used starch in thermoplastic starch, but few have confirmed that thermoplastic starch is used in the production of biodegradable products. However, thermoplastic starch has weak properties such as, necessitating further studies to boost thermoplastic starch's properties (Khan et al., 2017). As a result, a suitable alteration is needed to improve the material's properties (Marichelvam et al., 2019).

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

A few studies and research that has been conducted to enhance the capability of biodegradable material in order to replace the conventional thermoplastic. One of the studies that has been conducted by Haameem et al., (2016) which utilize the usage of treated and untreated Napier grass fibers in the composite. It was found that treated Napier grass fiber composite has positive improvement in term of tensile strength and morphological structure and able to enhance the properties of the composite. This finding shows that natural fiber also can be one of the fillers in biodegradable composite. Hence, this study would increase awareness of Napier grass fiber and thus allow the production of Napier grass to be converted into new biodegradable material.

1.3 Objective

The main objective of this study are:

- i. To produce biodegradable thermoplastic cassava starch reinforced with Napier grass fiber composite.
- To investigated the water affinity properties and the environmental properties of the biodegradable thermoplastic cassava starch reinforced with Napier grass fiber composite.
- To produce biodegradable packaging derived from Napier grass fiber and cassava starch.

1.4 Scope of Research

The justification of this study are as follows:

- i. The outcomes of the recent study might deliver a new information regarding to the biodegradable thermoplastic cassava starch reinforced with Napier grass fiber composite.
- ii. The problem related to the environmental pollution might be reduced by applying the fully bio-composite material started from Napier grass fiber and thermoplastic cassava starch.
- iii. The problem caused by the conventional thermoplastic also can be diminished by introducing the new material from cassava starch.
- iv. More value added to the existence of Napier grass fiber and cassava starch, using it as new reinforcement material in producing bio-composite product.

1.5 Scope of Study

In this study, the main raw materials used are cassava starch, Napier grass fiber, and glycerol. The mixture of thermoplastic cassava starch formed by combining the cassava starch with glycerol by referring to the suitable percentage of formulation. The glycerol actas the plasticizer in this study. Beeswax is added to the mixture of cassava starch and glycerol regarding to the suitable percentage of formulation. The function of beeswax in this study isact as the protective agent towards water absorption and moisture. Then, Napier grass fiber as the reinforcement is added to the mixture of three raw material due to the percentage required for this study. In terms of environmental testing, the test carried out is water solubility and soil burial. Meanwhile, for water affinity testing, the test that will be carried out are moisture content, water absorption and thickness swelling, and then, the others testing that will be evaluated are morphology by using a Scanning Electroscopy Microscope (SEM) and the density of the material. Finally, biodegradable packaging will be developed at the end of the study using hot pressing in a packaging tray mold.

1.6 Structure of Thesis

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

The arrangement of this thesis is according to the format provided by Universiti Teknikal Malaysia Melaka (UTeM) which is based on the publication of this study. This report consists of six part which are introduction, literature review, methodology, results, and discussion and lastly conclusion. The details of the structure are as follows:

Chapter 1

This chapter describe clearly about the objective of the study and highlighted the problem that initiated to this research. The significance scope of the study and work were elaborated in this chapter.

Chapter 2

This chapter justify the overall literature review that has been done by previous study which related to the area of this thesis. Besides that, the research gap gained from the review of previous study also stated in this chapter.

Chapter 3

This chapter indicated the methodology used in this research for the preparation of materials, testing procedure, and collection of data.

Chapter 4

In this chapter, hypothesis of every testing which is physical testing, environmental testing and water affinity testing will be comprehensively discussed.

Chapter 5

This chapter present the overall conclusion of the study with recommendation and improvement for further study according to Thermoplastic Cassava Starch Reinforced by Napier grass Fiber Composite. RSITI TEKNIKAL MALAYSIA MELAKA

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Environmental awareness today motivates researchers worldwide to conduct research and development on biodegradable products. People are more aware of environmental issues such as ozone layer thinning, soil degradation, water pollution, and air pollution, all of which significantly impact the greenhouse effect. The development of biodegradable products is one solution that can aid in the reduction of human-caused damage. Due to the researchers' tests and properties, biodegradable thermoplastic starch reinforced by natural fiber composite is one of the best solutions among the composite development. Biodegradable thermoplastic starch products are more environmentally friendly because they are simple to degrade and have a low manufacturing cost. Besides that, biodegradable thermoplastic starch is commonly used in various industries, including automotive, packaging, etc. Since many materials can be mixed to create a composite, the application of biodegradable thermoplastic starch products needs more investigation. As a result, the manufacture of biodegradable products is critical to the world's survival.

2.2 Polymer

Without polymers, existence will be even more complex. They are present in almost every aspect of our everyday lives, including rubber, silicone, resins, adhesives, and adhesive tapes. The term 'Polymer' originates in Greek, where 'poly' means many and 'mer' means unit. It was initially named 'Polymerous' in Greek, which means' many-membered. Thus, the term polymer is classified as having a large number of units, with each unit referring to a polymer molecule (Heath & Cooper, 2013).

To put it another way, polymers are large molecules with a high molecular weight called macromolecules made up of several small molecules called monomers. Polymerization is the process of monomers joining together to form a polymer. Polymerization is a chemical reaction in which two or more substances combine to create a molecule with a high molecular weight, with or without adding something like water, fire, or other solvents. The finished product is known as polymer, and the raw material is known as a monomer.

2.2.1 Classification and Types of Polymers

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Polymer is a common term for a wide range of high-molecular-weight materials. Because of the large number and variety of atoms in their molecule, these materials come in various shapes and sizes. Polymers may have diverse chemical structures, physical properties, mechanical properties, and thermal properties, among other things. The polymer can be categorized in various ways based on these characteristics, as shown in Figure 2.1.



Figure 2.1 Classification of Polymer

2.2.2 Synthetic Polymer

Research that has done by Shrivastava, (2018) about Introduction to Plastics Engineering. The author stated that polymers are divided into two categories: synthetic and natural. Scientists and engineers make synthetic polymers from petroleum oil. Nylon, polyethylene, polyester, Teflon, and epoxy are examples of synthetic polymers. Natural polymers can be derived from nature. They are frequently made of water. Silk, wool, DNA, cellulose, and proteins are examples of current polymers.

Shrivastava, (2018) defines polymers that are artificially produced in laboratories. These are also known as human-made polymers. Synthetic polymers are made up of carbon bonds and are usually obtained from petroleum oil in a regulated environment. The chemical bonds that keep monomers together are altered by heat and pressure in the presence of a catalyst, allowing them to bind. A trigger is a substance that is used to initiate or speed up chemical reactions between monomers. Thermoset and thermoplastic are the two forms of synthetic polymer.

2.2.3 Thermoplastic and Thermosets

According to Shrivastava, (2018) within the industry, the words "plastics" and "polymers" are often used interchangeably. Subsequently, the compounds are referred to as "resins," which is an ancient term for polymers. Plastics are a subcategory of polymeric materials,

and the terms plastics and polymers have more precise definitions. As previously mentioned, the term "plastics" refers to the ability of polymers to be shaped in the presence of heat. Plastic material is classified as thermoplastic or thermoset based on its reaction to heating.

However, two general types should be mentioned (Landis, 2018). Most polymers can be broadly classified as either thermoplastics or thermosets. The fundamental physical difference between the two has to do with the bonding between molecular chains – thermoplastics have only secondary bonds between chains. At the same time, thermosets also have primary bonds between chains. The names are associated with the chemical structure and their general thermal and processing characteristics since this fundamental structural difference significantly impacts material properties.

An interesting study was carried out by Shrivastava, (2018), thermosets in polymer can be described as materials that irreversibly harden when heated or when certain chemicals are added. The permanent chemical bonds formed between adjacent molecular chains on the thermoset resin during its chemical transformation. The ties listed can be referred to as crosslinks. The structure formed by crosslinking can be defined as a network structure resembling a molecular mesh. Due to the permanent nature of the crosslink, the thermoset polymer hardens and retains its form when reheated.

Figure 2.2 shows the crosslinks found in thermosets. Additionally, the hardening phase requires a chemical reaction known as curing; hence, thermosets in scrap form cannot be reused or recycled unless used as a filler content.



Figure 2.2 Molecular structure of thermoset (Karuppiah, 2016)

A study that has been done by Brinson, (2015) due to its high thermal and dimensional stability, thermoset polymers with a high crosslinking content are commonly used in electrical and thermal insulation materials. Indeed, this material possesses a high modulus and strength, making it ideal for use in adhesives and high-performance composites. Due to their high durability, thermoset polymers with a light crosslinking, also known as elastomers, are commonly used in seals, insulation, dampers, and tyres. Rubbers, amino acids, polyurethanes, epoxides, phenolic, and polyesters are all examples of thermoset polymers.

Shrivastava, (2018) stated that thermoplastic polymers are characterized as materials that melt in a reversible manner when heated and then harden when cooled. It is composed of polymers that are linked together by intermolecular interactions to form linear structures, as illustrated in Figure 2.3. When heated, thermoplastic materials can be recycled and remolded due to their reversible phase changes. Scrap thermoplastic may also be used for this purpose. Reversible behavior benefits melt manufacturing methods such as injection molding and extrusion because thermoplastic scrap can be reused and recycled.



Figure 2.3Molecular structure of thermoplastic (Karuppiah, 2016)

Hardness, friction and wear resistance, corrosion resistance, and resistance to environmental stress cracking are all properties of thermoplastic polymers. Additionally, it exhibits less creep and time-dependent behavior (Brinson, 2015). The use of crystalline thermoplastic polymers is evident in the packaging industry, where polyethylene is used to package milk containers. Additionally, polyethylene can be classified as LDPE (low density polyethylene), HDPE (high density polyethylene), or UHDPE (ultra-high-density polyethylene).



Figure 2.4Depiction of thermoplastic monomer bond and thermoset crosslinked covalent bond (Landis, 2018)

Properties	Thermoplastics	Thermosets
Flowability	Low	High
Raw material	Fully polymerized and supplied as solid pellets	Not fully polymerized, often supplied as a gummy liquid or semisolid
Bonding between neighboring chains	Neighboring chains are held together by physical bonds	Neighboring chains are held together by chemical linkages or crosslinks
Processing	Shapes are formed in the molten state then cooled to retain shape	Bodies are included in the cold or warm state then heated to maintain shape
Processing equipment	Standard melt processing equipment such as injection molding and extrusion could be used	Modified processing equipment is required where heat is introduced after the shapes are formed
Processing time	Short, as hardening could be performed quickly by cooling	Long, as crosslinks require a longer time to develop
Recycling	Thermal recycling possible	Thermal recycling not possible
Dimensional stability	Moderate	High
Common 🞽	Polyolefins, polyamides, polyesters,	Vulcanized rubbers, phenolic,
examples 🗧	commodity, and engineering plastics	epoxy adhesives, bakelite,
E		advanced sand composites

Table 2.1 Comparison between thermoplastics and thermosets (Shrivastava, 2018)

2.2.4 Application Polymer

Polymer is used in various sectors, including the automotive industry, electronics industry, aerospace industry, construction trades, etc. In the automotive industry, polymer blends are used in automotive bumpers because they are durable enough to meet state and federal requirements. Since it replaced the metal with polymer, polymer blends in this industry resulted in weight savings and cost savings. In reality, another benefit of being cost-efficient is that it saves money on energy, and the polymer blend is easy to recycle from older cars to make bumpers for new vehicles.

2.3 Composite

A composite material is made up of two materials that have distinct physical and chemical properties. When the composites are mixed, they form a specialized substance that can be used to make anything more substantial, lighter, or more resistant to electricity. Weight and firmness can also be improved with the composite.

In another definition, Camilleri, (2010) stated that the composite could be defined as a combination of two or more materials that results in better properties than those of the individual components used alone. In contrast to metallic alloys, each material retains its different chemical, physical, and mechanical properties. The two constituents are reinforcement and a matrix.

The primary advantages of composite materials are their high strength and stiffness combined with low density and bulk materials, which results in a lighter finished part. To support structural loads, strength and rigidity are given during reinforcement. The matrix acts as a binder for natural and inorganic materials, holding the direction and position of the mount while also passing current loads to the reinforcement. The physical and chemical properties of the composite components are maintained to a large extent (Camilleri, 2010).

2.3.1 What is a Composite?

On a macroscopic level, a composite material is composed of two or more materials (combined and bonded). Agarwal et al., (2010) very clearly stress that composite material is composed of reinforcement (fibers, particles, flakes, and fillers) embedded in a matrix (polymers, metals, or ceramics). The matrix holds the support to form the desired shape while the
reinforcement improves the overall mechanical properties of the matrix.

As defined by Wang et al., (2011), composite material is a multi-phase combination material of two or more component materials with different properties and different forms through compounding processes. It maintains the main characteristics of the original component and shows a new character that is not possessed by any of the original features.

Khayal, (2019) defines it as two or more materials are combined in a particular order on a macroscopic level to form a new material with different and attractive properties. The reason alloys are not considered in this category is because they are homogeneous on the visible level.

2.3.2 Classification of Composite Material

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According to Dawoud, (2013), "composite materials" or "composite" refers to a substance composed of two or more elements that exhibit substantially different physical or chemical properties. These combinations of two or more elements result in the formation of a new substance with characteristics distinct from the combined element.

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

Meanwhile, Pujari, (2014) stated that composites are materials that are composed of a high-loading material called reinforcement and a weak material called matrix. The reinforcement provides the strength and rigidity necessary to support structural loads. The matrix acts as a binder for organic and inorganic materials, retaining the orientation and position of the reinforcement and transmitting existing loads to the reinforcement. Substantially, the composite elements retain their individual physical and chemical properties. Fiber reinforced composites and particle reinforced composites are two types of composite materials. The classification of composites is depicted in Figure 2.5.



Figure 2.5 Classification of composite (Khayal, 2019)

2.3.3 Polymer Matrix Composite (PMCs)

Polymer matrix composites (PMCs) comprise various short or continuous fibers held together by an organic polymer matrix. Unlike a ceramic matrix composite (CMC), which uses reinforcement to improve fracture resistance, polymer matrix composites use reinforcement to have high strength and stiffness. The polymer matrix composites are constructed to reinforce the mechanical pressures that the system is exposed to during operation. The matrix's purpose is to secure the fibers together and to pass loads between them.

According to Hydro et al., (2013), polymer composites comprise two or more components that function as distinct phases and contain at least one polymer. Combining the polymer with another substance such as carbon, glass, or another polymer allows for creating a particular mixture or level of properties.

Polymer Matrix Composite (PMC) consists of fiber-reinforced thermosets or thermoplastic matrix resins. The fibers are usually thicker and stiffer than the matrix. PMC was selected because it is more lightweight, stiffer, and heavier than unreinforced polymers or standard metals, according to Gupta & Doddamani, (2018). Furthermore, the benefits of PMC's properties and form may be tailored to meet the requirements of a specific application.

Polymer matrix composites (PMCs) are extensively employed in the automotive, maritime, consumer products, and sports equipment sectors. Carbon glass fibers, which may be continuous or discontinuous, are used to strengthen PMC materials in the industries. The structure of these components demonstrates the component's lightweight. Another use of PMC is ballistic safety, where the combination of Kevlar and polyethylene fiber-reinforced composites produces excellent results in the manufacturing process (Wang et al., 2011).

2.3.4 Application of Composite

Glass fiber polymer matrix composites became the first produced and implemented composite materials. In the 1940s, Americans first compounded glass fiber and unsaturated polyester resin and then hand laid-up naval radar and aircraft fuel tanks, paving the way for the use of glass fiber reinforced products in military manufacturing. Since then, with advancements in glass fiber, resin matrix, and composite material technology, glass fiber composite products have seen widespread application in several civilian industries and have developed into significant engineering materials.

However, as the 1960s progressed, it became apparent that glass fiber reinforced plastics had a high mass and a low modulus. It was incapable of meeting the specifications of high-tech goods requiring a high basic modulus and power, such as aircraft. As a result, between the 1960s and 1970s, lightweight carbon fiber and carbon fiber composites with a high basic modulus and specific strength were produced. Carbon fiber was followed by aromatic polyamide fibers (aramid fiber) and other high-performance fibers. This modern composite material, which began with carbon fiber composite materials, was dubbed advanced composite materials (ACM). At the moment, the global output of composite materials is measured in megatons. It is difficult to accurately statistic the yield. The world's production was estimated to be 3.9 million tons in 1990, with 1.503 million tons produced in the United States, 1.485 million tons produced in Western Europe, 0.643 million tons produced in Japan, and 0.106 million tons produced in China. Nonetheless, composite structures provide a negligible amount of content as opposed to other structural materials. Steel demand now exceeds a thousand megatons globally, while plastics production still exceeds a hundred megatons. This demonstrates that there is still considerable space for composite material growth and a need to accelerate development.

While the first use of composite materials was in the aerospace industry, and its growth has been fueled by aerospace demand, composite materials for aerospace applications account for a minimal portion of total production (less than 1% to 2%). Composite products are extensively used in the transportation industry, manufacturing, chemical corrosion devices, and electric or computer appliances.

اونيوم سيتي تيڪنيڪل مليسيا ملاك 2.4 Fiber UNIVERSITI TEKNIKAL MALAYSIA MELAKA

The most often used fiber-reinforced composites are made up of fibers and a matrix. The reinforcement and primary source of strength are fibers, while the matrix holds all the fibers in place and passes stresses between the reinforcing fiber (Pujari, 2014). Fiber can be defined as any single matter unit characterized by flexibility, high aspect ratio, and fineness. Fibers can be classified into two categories which are natural fiber and synthetic fiber. Figure 2.6 shows the classification of fiber.



Figure 2.6 Classification of fiber

2.4.1 Synthetic Fiber

Synthetic fiber, on the other hand, is made by human beings. That is why these are called synthetic or human-made fibers. Synthetic fibers are produced by chemical synthesis, and by going through their content, it is further classified as organic or inorganic. The fiber material has higher stiffness and strength than the matrix material. In this case, use them as a load-bearing feature in the composite structure (Rajak et al., 2019). Generally, the strength and stiffness of fiber materials are much higher than that of the matrix material, making them a load-bearing element in the composite structure. Examples of synthetic fibers including glass fiber, carbon fiber, aramid, polyacrylonitrile (PAN-F), polypropylene fiber (PP-F), basalt, polyethylene terephthalate (PET-F), and graphene fiber.

Glass fibers are the most commonly used synthetic fiber in the industry due to their thermal stability, high strength and stability, strong impact resistance, and good friction and wear properties. Glass fiber, on the other hand, has a downside in that it must be disposed of until it reaches the end of its useful life. The machining process is also challenging as the process is slow and reduces tool life while working with the conventional machining system.

2.4.2 Natural Fiber

Natural fibers have received a lot of attention in recent decades from academia and industry as a viable alternative to synthetic fibers. As polymer reinforcements, a variety of natural fibers may be used.

According to Rajak et al., (2019), natural fibers are fiber sources derived from plants, animals, and other mineral sources. Natural fibers are easy to obtain because it is obtained from naturally occurring sources. Natural fibers are also widely used in industry, especially in the automotive industry, to improve the environmental sustainability of parts that must be produced. Because of their biodegradability, high strength, necessary stiffness, and low cost per unit volume, natural fibers are among the most widely used materials. Because of these properties, natural fibers tend to be more common than synthetic fibers.

Natural fibers have four distinct advantages over synthetic fibers: specific properties, cost, biodegradability, and recyclability. Particularly beneficial are particular and low-density properties. They are organic and have a CO2-neutral life cycle (i.e., carbon and glass) compared to their synthetic competitors (Kumar et al., 2017).

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Natural fiber has many advantages over synthetic fiber, including low density, a golden modulus-weight ratio, availability, high acoustic damping, low cost, low carbon footprint, low manufacturing energy usage, and biodegradability. Natural fiber has a lower price and uses fewer resources during processing than traditional reinforcing fibers like carbon and glass (Peças et al., 2018).

Natural fiber, on the other hand, has disadvantages because of its variable properties and efficiency. Moisture absorption, durability, intensity, and processing temperature vary in these fibers' physical and mechanical properties. Many natural fibers have been studied in the scientific community to see whether they can be used in the manufacturing process. Natural fibers include banana, bamboo, coir, cotton, eucalyptus, flax, hemp, jute, kenaf, pineapple, ramie, and sisal. Many of these natural fibers are examples of the regular fiber used in industry. Table 2.2 below provides a brief overview of the different types of natural fibers.

Fiber	Description			
Abaca/Banana	Abaca, known as manila hemp, is a plant that almost similar to a			
MA	banana. The abaca plant does not use for human consumption as it			
(F)	being not viable economically. This plant grows only for fiber			
S.	cultivation.			
Bamboo	Bamboo is one of the growing plants, has a high strength to weight			
-	ratio, does not need to use pesticides or herbicides as it requires less			
1 and	water, leaving the root intact and harvested at its base. Bamboo has			
AIN	a smooth fiber surface and in a round shape. It is also more robust			
chil	and stiffer compared to glass fiber.			
Coir 200	Coir is strongly resistant to saltwater, attractive to its availability,			
more durable than the other natural fiber, and free from				
UNIVE	chemical treatment. ALAYSIA MELAKA			
Cotton	Cotton fiber has good absorbency.			
Eucalyptus	Eucalyptus has low resistance to fire attack and mold. This fiber			
	is available widely and adequate for insulation purposes.			
Flax	Flax fiber is low in density, high stiffness, and strength and also has			
	better specific tensile.			
Hemp	Hemp fiber has good properties of insulation and has excellent			
	young's modulus with mechanical strength.			
Jute	Jute fiber has good properties of insulation, a high aspect ratio,			
	and high strength to weight ratio.			
Kenaf	Kenaf fiber has high specification in mechanical properties and has			
	low density.			
Pineapple	Pineapple fiber has good mechanical, thermal and physical			
	properties.			
Ramie	Ramie fiber required expensive pre-treatment, which makes it			
	lesspopular compared to the other natural fiber. This fiber also			
	has a better specific modulus and specific strength.			

Table 2.2Natural fibers characteristics summary (Peças et al., 2018)

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Sisal fiber has high saltwater resistance, high abrasion resistance, high tenacity, tensile intensity, and resistance to acid and alkali.

Natural fiber also can be graded based on its sources. The classification of natural fibers for plant or vegetable fibers is shown in detail in Figure 2.7. The physical properties of natural fiber will decide the chemical and physical composition of natural fiber. Natural fiber chemical and biological designs are focused on cellulose material, cross-section, thread structure, fibril angle, and degree of polymerization, among other factors.



Figure 2.7 Classification of natural fiber

2.5 Napier Grass

Cenchrus purpures, synonym Pennisetum purpureum, is a perennial tropical grass native to the African grasslands. It is also classified as Napier grass, elephant grass, or Uganda grass. Due to its low water and nutrient needs, it can be grown on previously uncultivated soil (Kommula et al., 2013). Historically, this wild species was mainly used for grazing, but more recently, it has been used in conjunction with a push-pull agricultural pest control technique. Napier grasses increase soil productivity and help prevent soil erosion on arid fields. Additionally, it is used to create firebreaks and windbreaks and the development of paper pulp and, more recently, in the production of bio-oil, biogas, and charcoal. Figure 2.8 shows the Napier grass.



2.5.1 Napier Grass Fiber Origin, Propagation and Distribution

According to Negawo et al., (2017), Napier grass is a monocotyledonous flowering plant that is a member of the grass family Poaceae and the genus Pennisetum. Pennisetum is a widely complex genus comprised of approximately 140 organisms with a variety of basic chromosome numbers of 5, 7, 8, or 9, ploidy levels ranging from diploid to octoploid, sexual and apomictic reproductive habits, and periodic, biennial, or perpetual life cycles. Napier grass is a perennial C4 grass plant that originated in Sub-Saharan Africa and spread to other tropical and subtropical regions worldwide. It has been recorded to develop in a wide variety of soil conditions and agro-ecologies, from sea level to 2100 meters, and withstand brief periods of drought. However, it thrives in areas with an annual rainfall of between 750- and 2500-mm. Napier grass has been naturalized in Central and South America, tropical Asia, Australia, the Middle East, and the Pacific islands due to its wide agroecological adaptation. As a result, it is now commonly cultivated in the tropical and subtropical regions of the world, mostly for use as animal fodder shows in Figure 2.9. Napier grass is more commonly spread through vegetative cuttings and tillers, as the grass produces few seeds. Those that do are typically very thin, weak, and of low quality, and the spikelets are vulnerable to shattering. As a result, the seeds are deemed unsuitable for propagation since they grow tiny seedlings. Additionally, because Napier grass is an open-pollinated crop, the seedlings are particularly heterozygous. As a result, stem cutting propagation is the most popular method of dispersing Napier grass propagules.



Figure 2.9 Regional distribution of Napier grass around the world (Negawo et al., 2017)2.5.2 Characteristic of Napier Grass Fiber

The Napier Grass is a perennial fibrous herb with short stems high, broad, and slender, branchless leaves. It is a species with a bright future due to its ease of production, fast development, and abundance in tropical regions with minimal nutrient supplementation. Their harvest time is three to four months after sowing, and they will continue to expand at a six- to eight-week interval for up to five years (Lim et al., 2020). Napier grass fibers yielded 40% from each clump plant and generated 40 tons of biomass per hectare per year. The properties of Napier grass are mentioned in Table 2.3.

Material	Che	emical properties	P pr	Physical operties	Mec proj	hanical perties	
	Cellul ose w/w%	Hemice Lignin lluloses w/w% w/w%	Fiber length mm	Fiber diameter mm	Tensile index mN.m ² /	Tear index mN.m ² /	Burst index kPa.m ² /g
Napier grass	45.60	30.50 17.70	0.75	15.14	g 93.25	g 4.40	5.85

Table 2.3 Chemical, physical and mechanical properties for Napier grass (Lim et al., 2020)



According to Reddy et al., (2012), physical properties provide details such as diameter, cross-sectional space, and density. Before researching the mechanical properties of the Napier grass fabric, the physical properties must be determined. The diameter of the Napier grass fiber was calculated using a monochromatic He-Ne laser, and the cross-sectional region was determined using SEM micrographs at 1000 x magnification. Additionally, the density of Napier grass fibers was measured using a pycnometer technique. The details in Table 2.4 reflect the physical properties of Napier grass fiber obtained from a variety of sources.

References	(Kommul a et al., 2013)	(Redd y et al., 2012)	(Lim et al., 2020)	(Ridzuan et al., 2015)	(J. A. M. Haamee m et al., 2016)	(Ridzuan , 2016)
Average length (mm)	185	142	0.75	165	121.5	158
Average diameter (mm)	-	0.255	15.14	0.26	0.199	0.27
Aspect ratio (L/D)	-	556.8	-	-	-	-

Table 2.4 Physical properties of Napier grass fiber



2.5.2.2 Mechanical Properties of Napier Grass Fiber

The density and tensile properties of the grass fibers of Napier are summarized in Table 2.4, along with those of the more commonly available fibers. The fiber density is important for lightweight applications in particular. Compared to other traditional natural fibers, the Napier grass fibers have one of the lowest densities (358 kg/m3). However, it is marginally higher than jute, sisal, and ananas. The Napier grass fiber's total tensile strength was higher than bamboo and coir fiber. Although the Napier fibers, due to their low density of 0.358 kg/m3, are less than flax,

cotton, jute, kenaf, and bananas, they may be used as an efficient reinforcement for lightweight composites (M. Haameem, et al., 2016).

Fiber name	Density	Diameter	Tensile	Tensile	Reference
	(kg/m^3)	(µm)	strength	modulus	
			(MPa)	(GPa)	
Flax	1500	-	500-900	27.6-80	(Xuefeng,
					2014)
Hemp	1480	-	300-800	70	(Xuefeng,
					2014)
Jute	160	-	258-306	10-30	(D. Ray, B. K.
					Sarkar, 2002)
Sisal	150	50-300	227-400	9-20	(D. Ray, B. K.
	ALAY.	SIA			Sarkar, 2002)
Pineapple	144	20-80	413-1627	34.5-82.5	(Satyanarayana,
	N. Contraction				1990)
Kenaf	2 1440	81	250	4.3	(Zhao, 2014)
Bamboo	= 910		16-38	35.9	(D. Ray, B. K.
	-				Sarkar, 2002)
Snake grass	887 _	45-250	78	9.7	(Wong et al.,
	AINO				2010)
Napier grass	358	150-550	106	39-47	(M. Haameem,
	5 Nolu	ula 15	· ·	here at a second	et al., 2016)
Banana	1350	80-250	529-759	8.2	(D. Ray, B. K.
					Sarkar, 2002)
Coir	UNI/1150 S	T100-460	LM/90AYS	IA M4-6_AK	🔍 (D. Ray, B. K.
					Sarkar, 2002)

Table 2.5 Comparison of the tensile properties of Napier grass fiber with that of other natural fibers

2.5.2.3 Chemical Properties of Napier Grass Fiber

Napier grass's chemical properties or chemical composition consist of cellulose, hemicellulose, moisture, and some lignin. These compositions act as the significant components thatforming the cell walls of natural plant fibers. Table 2.6 below shows the chemical properties of Napier grass fiber regarding a few references.

References	(Kommula et al., 2013)	(Reddy et al., 2012)	(Lim et al., 2020)
Cellulose(%)	47.12	45.66	45.60
Hemicellulose (%)	31.27	33.67	30.50
Lignin (%)	21.63	20.60	17.70
Moisture (%)	10.13	-	-

Table 2.6 Chemical properties of Napier grass fiber

2.5.3 Application of Napier Grass Fiber

Lukuyu et al., (2012) had brought up studies on farmers in Africa, Asia, and other tropical/subtropical parts of the world using various grass species as fodder crops. Napier grass is a critical fodder crop, especially in smallholder farming communities in Eastern and Central Africa. The authors reported it is mostly used in cut and carry feeding systems to feed livestock. It is a versatile forage crop that can be grazed directly or converted to silage or hay, and there have been records of using Napier grass as fish food in Nepal and Bangladesh. Young shoots of Napier grass were also used as a cooked vegetable in a recent study from Nigeria (Shaha et al., 2015). These numerous applications demonstrate the breadth of ways in which Napier grass can contribute to the reduction of poverty and nutritional insecurity.

Apart from its value as a forage grain, according to Shaha et al., (2015), Napier grass can be used to build walls, function as a windbreak, and demarcate borders between neighboring farmers; the dried material can also be used as a fuel source. It is used as a mulch in cropland management systems to combat weed infestation and soil degradation and as a trap plant in the push-pull strategy, a pest management technique that employs repellent intercrop 'push' plants and attractant trap 'pull' plants to control insect pests in Africa, most notably the maize stem borer. Plants are often used to scavenge contaminants such as heavy metals. Napier grass has been used in phytoremediation techniques, such as cleaning up cadmium-affected soil by lowering the cadmium content to a depth of 15 cm.

With rising global involvement in limiting fossil fuel use and the anxiety about the effects of climate change, the hunt for alternative biofuel supplies has resulted in the promotion of significant biomass plants as second- or next-generation biofuel crops. With its perennial nature and rapid growth rate, Napier grass has been estimated to produce up to 78 tons of dry matter (DM) per hectare per year (35–41 tons/ha on average). According to Rengsirikul et al., (2013), from Napier grass varieties grown in Thailand, we calculated a gross ethanol output of 350–460 L/ton DM and an estimated ethanol yield of 329 L/ton DM.

2.5.4 Napier Grass Composite

Napier grass leaf fiber is a renewable fiber with excellent properties as used in composites. Numerous experiments have been conducted on the use of Napier grass fiber in industries such as packaging. This is attributed to the nature of Napier grass fiber, which contributes to the product's lightweight. Additionally, Napier grass fiber is one of the substitutes used to create biodegradable products beneficial to our environment. The easy degradation of Napier grass fiber composite is one of the explanations for its use in composites.

A study carried out by Haameem et al., (2016) regarding the effects of water absorption on Napier grass fiber. This study tests the mechanical properties andthe morphological fracture observations on the composites. The dried Napier grass fibers were removed and processed with a 10% NaOH solution. The fiber-to-solution ratio was held constant at 1:40, and the fibers were soaked for 24 hours. Following that, the fibers were washed four times to eliminate any remaining NaOH. Figure 2.12 represents the percentage weight gain resulting from time (in hours) for Napier grass fiber-reinforced composites after immersion in distilled water at room temperature (25°C). However, the pure polyester sample had a water absorption ratio of about 1.5 percent, meaning that it stored relatively little water. These curves of water absorption are comparable to those obtained in an analysis by Dhakal et al., (2007) in which hemp fiber composites behaved similarly.



Figure 2.12 Weight gain as a function of the square root of time for the untreated and treated Napier grass composites (M. Haameem, et al., 2016)

The percentage weight gain curves indicate that the amount of water consumed improved as the immersion time was prolonged. The untreated samples retained more water as a result of water assault owing to the Napier fibers' hydrophilic composition. This water assault causes hydrogen bonding within the fiber, reducing the fiber's interfacial adhesion to the matrix. Untreated fibers are hydrophilic, indicating that they comprise cellulose, which absorbs water. Alomayri investigated the water absorption experiments on cotton fibers were recently performed, and it was discovered that the moisture absorption curves were identical, although at various cotton fiber volume fractions (Alomayri et al., 2014). Additionally, deterioration happens as the fibers swell as a result of moisture absorption. Between the fibers and the resin matrix, micro gaps form, resulting in the forming of water transport pathways inside the composites (Daly et al., 2008).

Figs. 2.13 and 2.14 show the tensile stress effects for the untreated and handled Napier grass fiber composites, respectively. Table 2.7 summarizes the tensile properties. Mechanical materials deteriorate as a result of poor tension transfer in the composites leading to corrosion of the fiber-matrix interfaces.



Figure 2.13 Tensile stress as a function of moisture exposure period (days) for untreated Napier grass composites (Haameem et al., 2016).



Figure 2.14 Tensile stress as a function of moisture exposure period (days) for treated Napier grass composites (Haameem et al., 2016)

According to table 2.7, the maximum tensile stress of 23.6 MPa was obtained with handled long Napier grass fibers. Following that, composites with tensile strengths of 18.2, 17.2, and 15.6 MPa were produced. The tidy polyester's tensile stress improved from 10.1 to 18.2 MPa upon integration of the long untreated fibers due to the resistance of the Napier grass fibers to the applied stress.

Maximum tensile stress (MPa)					
Specimens	Room temp (dry)	6 days	12 days	18 days	23 days (FS)
Untreated long	18.2	14.5	10.6	9.5	8.5
Untreated short	15.6	10	9.3	7.1	6.6
	Room temp (dry)	4 days	8 days	12 days	14 days (FS)
Treated long	23.6	13.7	12.9	11.9	10.4
Treated short	17.2	12.6	11.9	10.9	10.0
	Room temp (dry)				
Pure polyester	10.1				

Table 2.7 Tensile properties of untreated and treated Napier grass fiber composites

The tensile tension, on the other hand, decreased to 10.4 MPa in the composites composed of long untreated fibers that were completely saturated after 23 days of water

immersion. This results in a 40% reduction in power. As shown in Fig. 2.13, the composite composed of dry short untreated fibers had a tensile strength of 18.2 MPa. However, after 23 days of immersion in water, their intensity decreased to 8.5 MPa, a decrease of more than 60%. The power of the untreated Napier grass fiber composites was significantly reduced due to the hydrophilic nature of the cellulose in the Napier grass (Kabir et al., 2012). It is reasonable to presume that immersion of the fibers in purified water affects the interfacial bonds between the fiber and matrix, resulting in increased deterioration of the samples (Graupner et al., 2009). Dhakal investigated the greater degree of deterioration that may be explained by the samples being subjected to precipitation, which compromised the fiber-matrix interface (Dhakal et al., 2007).

In their study, the surface morphology of treated and untreated Napier grass fiber composites was examined using SEM. It is reported that the untreated Napier grass fiber composites absorbed moisture at a faster pace. As shown in figure 2.15 (a–d), the untreated composites contained a larger amount of fiber breaks and pull-outs than the handled composites. Additionally, as the time of immersion grew, the amount of matrix fractures in each broken sample increased. According to Joseph et al., (2002) the influence of water plasticization weakens the connection between the fiber and the resin matrix, resulting in interfacial failure.

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Figure 2.15 SEM micrographs displaying the fractured treated Napier grass composites that had been immersed in water for (a) 4 days, (b) 8 days, (c) 12 days, and (d)14 days (fully

saturated) (M. Haameem et al., 2016)

Figure 2.16 shows a picture of the broken treated sample after four days of water immersion; the matrix fractures are scarcely noticeable at this stage. However, as the duration of water immersion increases, the matrix fractures grow larger and more apparent. Previous research has concluded that treating fibers improves moisture tolerance by decreasing their hydrophilicity, thus strengthening the connection between the fibers and the resin matrix (Haameem et al., 2016). Additionally, lumens can be seen in the SEM photos of untreated samples but not in the images of alkaline-treated samples. This is how the alkaline solution removes the waxy surfaces, hemicelluloses, lignin, and impurities from the fibers (Haameem et al., 2014).

It is reasonable to conclude that the orientation of the fibers is critical in deciding the composites' properties. Entanglement is likely to occur in randomly oriented fiber-reinforced composites. Fiber misalignments and entanglements are evident in Fig. 2.16(b–d). These

entanglements provide paths inside the composites, facilitating the creation of voids and porosities. These pathways allow water molecule penetration, thus increasing the stress concentration within the composites and resulting in a higher failure rate (Munirah & Ahmad, 2012). In addition, alkaline therapy roughens the fiber surface and highlights micropores in comparison to untreated fibers.



Figure 2.16 SEM micrographs displaying the fractured untreated Napier grass composites that were (a) dry, and had been in immersed in water for (b) 6 days, (c) 12 days, and (d)23 days (fully saturated) (Haameem et al., 2016)

The author report that they fabricated Napier grass fiber-reinforced polyester composites and investigated the impact of water absorption on the composites' mechanical properties. Usually, Napier grass fiber is hydrophilic in nature, meaning it can contain water. Untreated Napier grass composites consume more water than treated Napier grass composites. The treated Napier grass composites consume less water as a result of the alkaline process so they are less permeable to water. This research shows that water absorption greatly impairs the mechanical properties of these composites, lowering their tensile and flexural strengths significantly. It is rational to conclude that the matrix-fiber bonding is compromised as a result of the water absorption. Hence, throughout this research it shows that this can be another alternative for degradable plastic composite to be applied in various industries especially in packaging industry.

2.6 Starch

Starch can be classified as one of the component in renewable natural polymers which also consist of cellulose, soya and chitosan (Pokhrel, 2015). Pfister's team have investigated that the starch is present in the majority of green plants and all plant tissues and organs, including buds, shoots, roots, grains, and fruits. Starch is accumulated during photosynthesis as a result of carbon fixation, and although it is developed in the sun, it is weakened in the dark as it utilizes the synthesis of sucrose (Pfister & Zeeman, 2016). Pokhrel's team has researched applications of starch. Owing to its abundance and large processing potential, as well as its low cost of utilization, starch has many advantages over other green natural polymers (Pokhrel, 2015).

Besides that, Ogunsona has conducted research into advanced applications of starch and discovered that starch as a macromolecule is often appealing due to its physical, chemical, and functional properties, including ease of water dissolution, water retention properties, gelatinization, and pasting activity at elevated temperatures, as well as its ease of adjustment to maximize functional properties (Ogunsona et al., 2018).

According to Pokhrel's study, starch exists in the granule process of the cell, which is found in plastids away from the cytoplasm. It serves as a reserve carbohydrate in the plant tube and seed endosperm—characteristics starch has a white hue and a soft amorphous powder devoid of flavor or sweetness. Additionally, starch is a protein that is non-reducing and insoluble in water, ether, and alcohol (Pokhrel, 2015). Figure 2.17 illustrates how starch granules can be simple or compound in nature, with sizes ranging from 2 to 150 m, as well as the form and scale distribution of the starch granules.



Figure 2.17 Starch granule from SEM image with scale bar of 10 µm (Wang et al., 2012)

Starch consists of two mixture of glucans which is amylose and amylopectin. From a review on the introduction and applications of starch, amylose is a water-soluble carbohydrate that contains between 10% and 20% of water. In comparison, amylopectin contains between 80% and 90% water-insoluble carbohydrates, which is what the majority of starch contains (Pokhrel, 2015). Amylose, a linear glucose chain connected by α -1,4 glucosidase bond, and amylopectin, a branched glucose chain with branching at the α -1,6 position, comprise the native starch granule. Figure 2.18 shows the molecular structure of amylose.



Figure 2.18 Molecular structure of amylose (Pokhrel, 2015)

Amylopectin can be explained as having a backbone of α - (1-4) linkages with the branches molecule by α - (1-6) linkage and extension of 4-5 percent. In addition, the linear unit in amylopectin has about 20-25 percent length of glucose units. Amylopectin is accountable for the starch preparation thickened properties but does not make any contributions to gel formulation (Pokhrel, 2015). Figure 2.19 below shows the amylopectin partial structure.



Figure 2.19 Molecular structure of amylopectin (Pokhrel, 2015)

Numerous experiments have shown that the amylose-amylopectin ratio of starch as a biopolymer impacts its functionality and chemical properties. The ratio of amylose to amylopectin in different botanical sources of starch is shown in Table 2.8.

Table 2.8 Botanical sources of starch and their corresponding amylose/amylopectin ratio, and crystallinity.

Source	Crystallinity (%)	Amylose (%)	Amylopectin (%)	Reference
Rice	38	20-30	80-70	(Belgacem &
				Gandini, 2008)
Potato	23-53	23-31	77-69	(Nafchi et al.,
				2013)
Cassava	31-59	16-5	84-75	(Sabaté et al.,
				2012)
Waxy cassava	N/A	0	100	(Sabaté et al.,
				2012)
Wheat	36-39	30	70	(Belgacem &
				Gandini, 2008)
Corn	43-48	28	72	(Nafchi et al.,
				2013)
Sorghum	22-28	24-27	76-73	(Boudries et al.,
-				2009)

2.7 Thermoplastic Starch

Nafchi's team has researched thermoplastic properties, which stated that thermoplastic starch is generated by modifying the composition of starch granules in the face of low moisture content and mechanical powers. This procedure continues due to the presence of plasticizers that do not readily evaporate throughout the process (Nafchi et al., 2013).

As defined by Emilio, thermoplastic starch is a substance that utilizes plasticizers such as water, glycerol, sorbitol, and glycol to process starch. When the substance is readily melted and flows, the procedure is coupled with high temperature and shearing operation and may be used as an injection, extrusion, or blow moulding material (Emilio, 2013). There are many sources of thermoplastic starch including wheat starch, corn starch, potato starch, and maize starch. The manufacture and processing of thermoplastic starch was regarded as critical for reducing the global total of synthetic plastic wastes (Emilio, 2013).

2.7.1 Polymerization of Starch

A research studied by Nafchi et al., (2013) stated that starch has a biomass, were structed by Mafchi et al., (2013) stated that starch has a biomass, hydrogen, and oxygen ratio of 6:10:5 ($C_6H_{10}O_5$), indicating that it is a carbohydrate organic compound. Starch may be thought of as a glucose polymer that forms when glucose units condense. The oxygen atom is connected to the glucose units through the carbon atom 1 of one glucose unit to the carbon atom 4 of another glucose unit. Thus, a long chain of interconnected glucose units is created.

In its normal environment, starch is formed in a granular shape. As sheer force is exerted at temperatures between 90°C and 180°C, the starch loses its granular framework and transforms into thermoplastic starch or molten plastic. This is due to the fact that the plasticizer

is used early in operation (Corradini et al., 2010). When starch granules are treated under shear stress and at a high temperature in the presence of plasticizers, they transform into thermoplastics. Thus, thermoplastic starch is well-known for its ability to convert starch to a homogeneous material. This amorphous and network of hard-elastic regions is formed by soft regions (García, 2019).

According to García, (2019), the plasticization of starch occurred due to the application of shear fields and the addition of plasticizers (polyols as the conventional additives). The inclusion improves the final properties of glycerol as a plasticizer. Amylose and amylopectin are two forms of polysaccharides that combine to create a starch granular structure. Due to the disturbance of the granular framework during the starch transition, some of the carbohydrate polymer molecules dominated by amylose chains were published—Thermoplasticization of starch results in several chemical reactions such as destructuration, splitting, freezing, and plasticization (Tester et al., 2010). Figure 2.20 depicts a graphical image of starch thermal production.



Figure 2.20 Starch thermal processing melt-mixing (García, 2019)

2.7.2 Thermoplastic Potato Starch

Alvani's team has investigated the chemical properties of potato starches. He stated that potatoes are a major food source globally, with a global supply of about 300 million tons each year. Potatoes are classified as vegetables that have a variety of nutrients and nutritional applications. Potatoes are processed as a raw material in the food industry to produce food items such as treats, dinners, grain, potato alternatives, and starch derivatives (Alvani, 2011). Potato starch is unusual in that it has a large granule scale, a phosphate ester group on amylopectin, and a long chain length of amylose and amylopectin.

Additionally, during heating and subsequent cooling, potato starch may form a dense visco-elastic gel and swap some of the cations associated with the altered viscosity activity. When gelatinized, the lack of lipid and phosphate ester groups causes the potato starch to form a clear gel (Adebowale et al., 2015).

According to Raj et al., (2020) a few properties of potato starch relate to its molecular structure and granularity, which differentiate it from other starch. These include the granule's broad and smooth structure, the presence of numerous covalently connected phosphates, the high molecular weight of amylose, and chains of amylopectin. Additionally, starch granules range in diameter from 10 μ m to 100 μ m and are composed of two-part polysaccharides with glucose as the monomer base. The glucopyranosyl residue is linked to the other chains via the formation of -D-(1,4)-linkages chains and by going -D-(1,6)-branches located on the reducing end hand.

Generally, the main component of starches is amylopectin, which potato starch typically owns to the tune of 70% to 80% by weight of the granule scale. Amylose is a minor

part of the starch and is significantly smaller in size than amylopectin. The structures depicted in Figure 2.20 are (a) linear amylose and (b) branched amylose and amylopectin. As a result of these combined properties, potato starch is the most abundant source of biopolymers used in material science and food.



Figure 2.21 Molecular structures of the amylose (A) and the amylopectin (B). Numbers (1–6) in the first glucose unit of the amylose show numbering of carbon atoms in glucose molecule.

A study characteristics of potato conducted by Herrera et al., (2017) to assess the native potato starch's functional properties in the food and non-food industries. The author was conducted thermal analysis techniques such as Differential Scanning Calirometry (DSC), Scanning Electron Microscopy (SEM), Thermogravimetric Analysis (TGA), X-ray Diffraction (XRD), and Rapid Viscoamylograph Analysis (RVA). The primary raw material for this analysis is potato starch, which is extracted using a conventional process. The result is obtained via the proximal analysis detailed in Table 2.9 below.

Component	%	Standard deviation	Coefficient of variation
Fat	5.48	0.21	0.04
Ash	0.29	0.13	0.45
Moisture	18.81	0.05	0.00
Fiber	0.16	0.01	0.04
Protein	2.38	-	-
Carbohydrates	72.88	-	-

Table 2.9 Physiochemical properties of potato starch obtained by proximal analysis

(Velásquez Herrera et al., 2017)

Another research by Ren et al., (2018), stated that the surface morphology of plasticized starch and starch nano-bio composites was examined using SEM in Figure 2.22. These results may be explained by the increased shear force caused by the addition of sorbitol, which results in improved plasticizer dispersion and continuous phases. By incorporating halloysite into glycerol-plasticized starch, the morphology of the starchy matrix became more uniform and continuous. For nano-bio composites based on glycerol plasticized starch, no aggregates were observed. No interfacial voiding was observed in glycerol plasticized samples, indicating that the halloysite and the glycerol plasticized starch matrix have strong interfacial adhesion.

However, at high halloysite loading, large aggregates were clearly visible in the nanocomposites centered on sorbitol plasticized starch. The behavior of the starch-based nanocomposites plasticized with a mixture of glycerol and sorbitol was comparable to that of the glycerol- and sorbitol-based nanocomposites.



Figure 2.22 SEM images of the various plasticized starch systems and corresponding plasticized starch nano-bio composites: (A) G0; (B) G3; (C) G5; (D) G7; (E) P0; (F) P3; (G) P5; (H) P7; (I) S0; (J) S3; (K) S5 and (L) S7. Scale bars are 5 or 10 microns (Ren et al., 2018).

From a research characteristic of potato by Herrera et al., (2017), the author is identified Figure 2.23 illustrates another finding from thermogravimetric analysis (TGA). The graph shows how the sample removes the water material (9.46 percent). The sample then degrades significantly at the second stage (68.40 percent) due to the presence of the temperature interval with the highest degree of degradation. The other portion (21.13 percent) is combustible in the third stage by recombining with the inorganic material at elevated temperatures. As a result, it can be inferred that potato starch retains its structure at elevated temperatures, making

it ideal for use as a thickener throughout the cooking process. Potato starch is also useful in the packaging industry because of its low water absorption and ability to act as a biodegradable polymer.



Table 2.10 The TPS composites derived from potato starch are summarized in Table 2.10. Apart from being a food source, potato starch has a lot of promise as a green material. Their potential applications in packaging and agricultural industries, such as mulching film, have demonstrated promising properties that will be further developed in the near future.

Type of starch	Type of filler/polymer	Potential applications	Reference
Potato	Montmorillonite	Biodegradable material	(Hocking et al., 2008)
Potato	Sodium benzoate	Biodegradable material	(He et al., 2013)
Potato	Montmorillonite	Packaging material	(Alavudeen et al., 2011)
Potato	poly (butylene adipate- co-terephthalate) (PBAT)	Disposable packaging, agricultural mulching film	(Lackner et al., 2021)
Potato	High amylose starch vs. Normal starch	Biodegradable material	(Su et al., 2009)

	Гаble 2.10	Thermoplastic	potato starch	composites
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2.7.3 Thermoplastic Cassava Starch

An investigated by Teixeira et al., (2012) cassava, or more precisely *Manihot esculenta* roots, are widely grown in tropical countries. In West and Central Africa, cassava is the primary source of food for people living in the lowland tropics (Gleadow et al., 2009).

A few functional characteristics contribute to cassava's high quality, including the granule characteristic, which affects the rate of hydration and power swelling. Additionally, its visual appearance in the finished product is determined by its color, and its swelling, absorption, and water-binding potential, as well as its solubility, all of which affect the viscosity.

Prachayawarakorn et al., (2013) conducted research on the thermoplastic properties of cassava starch reinforced with natural cellulosic fiber. The natural cellulosic fibers used in this study were jute fiber and kapok fiber, as jute fiber has a low oil absorbency and kapok fiber has a high oil absorbency. Jute fiber also contains a high amount of cellulose, but less lignin, hemicellulose, wax, and pectin than kapok fiber. Additionally, since kapok fiber has a waxy surface compared to jute fiber, it serves as a strong water repellent.

The chemical composition of jute fiber and kapok fiber is summarized in Table 2.11. Internal mixing and shaping of thermoplastic cassava starch and glycerol as plasticizer were accomplished using a compression moulding machine.

Composition (%)	Jute fiber	Kapok fiber
Cellulose	71.5	64.0
Pectin	0.2	23.0
Lignin	13.1	19.0
Hemicellulose	13.4	23.0

Table 2.11 Main chemical composition of jute fiber and kapok fiber

The description of a previous report on thermoplastic cassava starch composites is shown in Table 2.12. In general, it can be concluded that thermoplastic derived from cassava possesses promising properties as a biopolymer matrix that is compatible with the majority of natural reinforcement materials. Additionally, thermoplastic derived from cassava shows great promise as a green material for environmentally friendly packaging.

Type of Filler/Polymer	Potential applications	Reference
Eggshell, calcium	Biodegradable material	(Munlika Bootklad, 2013)
Kenaf	Packaging	(Zainuddin et al., 2013)
Agar, span	Food packaging	(Maran et al., $2014a$)
Glucose, fructose, sucrose	Packaging	(Teixeira et al., 2012)
Jute, kapok	Packaging	(Jutarat
	JIEN	Prachayawarakorn et al., 2013)
Cassava bagasse	Packaging	(Teixeira et al., 2012)
	Type of Filler/Polymer Eggshell, calcium carbonate Kenaf Agar, span Glucose, fructose, sucrose Jute, kapok Cassava bagasse	Type of Filler/PolymerPotential applicationsEggshell, calcium carbonate KenafBiodegradable material PackagingAgar, span Glucose, fructose, sucrose Jute, kapokFood packagingPackagingPackagingCassava bagassePackaging

Table 2.12 Thermoplastic cassava starch composites

2.7.4 Application of Thermoplastic Starch UNIVERSITI TEKNIKAL MALAYSIA MELAKA

The application of thermoplastic starch has numerous benefits for the environment, including preventing soil and water contamination and conserving electricity. Apart from reducing energy consumption, the use of thermoplastic starch in composites can also reduce manufacturing costs and make the product more environmentally friendly. As the world ages and a new substitute for traditional petroleum-based plastic is needed, thermoplastic starch is the best item to be replaced. It is essential to investigate the primary properties of each thermoplastic used in industry. Since it is one of the thermoplastics used in the packaging industry, it is primarily used in food packaging.

According to Avella et al., (2005) the biodegradable polymer derived from starch could be an effective option for food packaging in order to address environmental contamination, health concerns, and disposal issues. On food packaging materials, starch/clay nanocomposites with improved mechanical properties and lower polymer and additive migration are created.

Another research by Dilara & Briassoulis, (2000), the author reported that starch-based polymers are biodegradable and have three significant agricultural uses, including greenhouse coverings, mulch films, and fertilizers. The film composed of starch blends was the first to be evaluated as agricultural mulch due to its sustainable material. At the moment, polylactic acid, a polymer derived from starch fermentation, is being combined with fiber to create composite films for agricultural mulching applications. The managed release devices based on starch have been developed to treat parasitic mites in honeybee colonies.

Biodegradable polymers are critical in plastic engineering because they can be used in place of non-biodegradable, non-renewable petroleum-based polymers. Blends of starch and poly (lactic acid) could be used to make tooth brush handles, flower pots, and cups. The value of starch-based biodegradables is heightened in the context of industrial chemistry's greening. As a result, researchers are intrigued by the prospect of developing biodegradable polymers (Koh et al., 2018). The market for biodegradable polymers has increased dramatically, indicating a promising future, owing to the environmental benefits associated with the use of renewable resource feedstocks.

2.8 Waxes

Waxes are a class of substances that are often used in cosmetic formulations (Schromm, 2018). Additionally, waxes are often used as food additives and coatings to protect fresh products, to prepare molds, to modify polymers and composites, in the medical sector, for dental

care products, and in the food packaging industry. As a result of their broad range of uses, synthetic and natural waxes are critical raw materials in a wide variety of industry sectors (Schromm, 2018). Nonetheless, the focus of this study is on innovative food packaging applications.

As previously mentioned, waxes are classified as lipids. They are a nonpolar compound that is soluble in organic solvents. Additionally, at room temperature, they self-assemble rapidly and are capable of forming crystalline materials. Their crystallinity is determined by the molecular rearrangements that occur during crystallization, which have a direct effect on their material properties. Additionally, waxes have a complex chemical structure due to the fact that they are composed of a large number of distinct molecular entities, and their molecular composition and interactions during crystallization influence their physical properties. The formation of the crystal network and the arrangement of the lipid crystals determine their physical properties. The crystal structure of a lipid is determined by its form, fatty acid composition, and crystallization conditions such as temperature, cooling rate, shear, and solvent presence (Jana, 2016).

Waxes are classified into natural and synthetic materials. There are four distinct types of natural waxes: plant, animal, mineral, and petrochemical waxes. The composition of waxes is complex, and obtaining a precise analysis is not always straightforward (Asperger et al., 2000).

A general definition for waxes according to Endlein, (2011) is a mixture of more or less hydrophobic substances with a medium chain length that has the following properties: low melt viscosity, is sometimes buffable, exhibits extreme temperature dependence in terms of solubility and consistency, resolidifies unchanged after melting, and has melting points ranging from approximately 40 to 140°C. At 20°C, waxes are plastic and brittle, but melt at temperatures above 40°C, assuming a soft and tacky state.



Figure 2.24 Classification of waxes into two categories (Endlein, 2011)

As previously stated, the numerous unique properties of waxes allow waxes to be used in a wide variety of applications. Several applications are mentioned, including oil binding in shoe polish and lipstick, water repellence in industrial coatings, release agents in bakery and plastic, scratch resistance in car polish and inks, plasticizers in hot melts and chewing, lubricants in pencils and metalworking, dispersing in mascara and toners, retarding release in agriculture and pharmaceutical matrices, and binding probiotics (Endlein, 2011). Table 2.13 collects the most common applications for different types of waxes.
Type of	Name	Applications	Reference
waxes			
Plant wax	Candelilla wax	Surface finishes and varnishes,	(Paine, 2016)
		chewing gums, hard candies, lipsticks,	
		pharmaceutical tablets, as an additive in	
		food and pharma.	
Plant wax	Carnauba wax	Surface finishes, baking products	(Johansson et al.,
		(wafer), chewing gum, confections and	2012)
		frostings, fresh and processed fruits and	
		fruit juices, soft candy, gravies and	
		sauces, lipsticks and lip balms,	
		mascaras, pharma (carriers) and food	
		applications as glazing agents (alone or	
		in combination with beeswax), release	
		agent (technical aid), paper, surfboard	
	MALAYSIA	coating, car waxes	
Animal wax	Beeswax	Chewing gums, confections and	(Gällstedt et al.,
	1 Alexandre	frostings, hard and soft candies,	2005)
	Ě	lipsticks, texture enhancement and	
		volume build-up in mascara and make-	
	Sa	up, adhesives, varnishes, inks and	
	SAIND.	paints	
Petroleum	Microcrystalline	Masticatory substances in the chewing	(ASTM, 2006)
based	2) wax	gum base, protective coating on raw	
	44 44	fruits and vegetables, defoaming agent.	<i>2</i>

 Table 2.13 Applications of natural waxes

2.8.1 Synthetic Waxes

In general, synthetic waxes with a low molecular weight are obtained through the polymerization of feedstocks such as ethylene, α -olefin, and methylene (Duru et al., 2019). The author also reported that synthetic waxes are derived from long-chain fatty acid triesters (C₁₈– C₃₆) or ethylene glycol diesters. The range of melting points of waxes, which ranges from 60 to 70°C, has an effect on their rigidity to sticks and crystallinity alteration. Fatty acids and alcohol esters with branched or straight chains that are shorter than those of waxes are used in cosmetic application.

Synthetic waxes are classified into two categories: partially synthetic waxes and fully synthetic waxes. Partially synthetic waxes can be obtained by chemically modifying natural waxes. Waxes synthesized entirely synthetically from low molar mass compounds as a starting material. A few examples of synthetic waxes include those made of polypropylene (PP) and polyethylene (PE) (Tinto et al., 2017)

2.8.2 Natural Waxes

Natural waxes are derived primarily from plant and animal matter, as well as petroleum, which produces mineral waxes, peat, and coal. Long-chain esters, hydrocarbons, long-chain carboxylic acids, and long-chain alcohols are the primary constituents of the majority of natural waxes derived from animals and plants. Only mineral waxes contained a long chain hydrocarbon (Endlein, 2011). Besides that, natural waxes are formed by a biochemical process rather than chemical treatment. Additionally, natural waxes are produced as a result of animal and plant metabolism.

According to Endlein, (2011), natural waxes are listed as renewable or non-renewable. Mineral waxes fall under the category of non-renewable natural waxes, and they can be obtained in crude or processed form. Renewable natural waxes are chemically modified and unmodified. Chemically modified substances include re-esterification and hydrogenation, whereas plants and animals are chemically unmodified.

2.8.2.1 Beeswax

A study conducted by Buchwald et al., (2008), beeswax is a composite substance composed primarily of alkanes, fatty acids, and long-chain esters. The wax is recovered as a by-product of the honey refining and harvesting process. The method of producing beeswax varies

according to the method used. Chemical extraction and melting are the two methods used to remove beeswax. There are a few drawbacks to using chemical extraction, but this approach is well-suited for small-scale manufacturing, as the process is performed in the laboratory. Additionally, the chemical extraction process can result in the extraction of organic contaminants in addition to the beeswax. Thus, the melting process is the most commonly used method for producing beeswax due to its low cost and simplicity.

Based on Tinto et al., (2017) research, the author stated that beeswax's white color indicates that it was formed directly from the virgin bee into honey comb. Beeswax's color darkens due to the indirect combination of silk, larval dirt, and pollen in the hive. When natural beeswax is exposed to cold temperatures, its structure becomes brittle and it often exhibits a high degree of plasticity and inertness. The most critical aspect is its solubility, as beeswax is soluble in the majority of organic solvents and, when heated or warmed, in fatty oils and alcohol, but is insoluble in water. Futheremore, beeswax is resistant to a variety of acids. The other characteristics of beeswax is shown in the Table 2.14 and the general composition of unhydrolyzed beeswax shown in Table 2.15.

Characteristics	Value
Melting point (°C)	61 - 66
Relative density at 15 °C (g/cm3)	0.958 - 0.970
Electrical resistance (Ω m)	$5 \ge 10^{12} - 20 \ge 10^{12}$
Thermal conductivity coefficient (Jcm/s °C cm2)	2.5 x 10 ⁻³
Saponification value	85 - 100

 Table 2.14 Characteristics of beeswax (Tinto et al., 2017)

Hydrocarbon	15%
Esters	71%
Free acids	8%
Other's compound	6%

Table 2.15 Composition of unhydrolyzed beeswax (Tinto et al., 2017)

2.8.2.1 Carnauba Wax

According to Tinto et al., (2017) Carnauba wax is a natural wax that is derived from plant waxes. Carnauba wax is derived from the Brazilian palm *Coernicia cerifera Martius*, or more commonly referred to as the carnauba wax palm. Carnauba wax is present on both the upper and lower surfaces of carnauba palm leaves. To obtain the wax from this plant, the leaves are cut and dried in the light, and then the wax from the dried leaves is beaten to obtain the wax; this method is referred to as harvesting.

The systematic process of producing carnauba wax requires passing through the collection, drying, beating, and refining stages. Finally, there is the final step of purification, which includes centrifugation, bleaching, and filtration. Additionally, another method of producing carnauba wax is by organic harvesting. Organic carnauba wax harvesting requires the melting and filtration of the highest quality untreated carnauba wax crude. A pleasant aroma indicates that carnauba wax has excellent oil binding properties and a light color (Tinto et al., 2017).

An others research by Alleyne & Hagenmaier, (2000) this wax is GRAS-certified (generally accepted as safe), allowing it to be used in coatings for a wide variety of foods, mostly fruits and vegetables. It crystallizes in an orthorhombic unit cell, as shown by X-ray diffraction, and its DSC curve is shown in Figure 2.29.



Figure 2.25 Carnauba wax DSC curve extracted (Basson & Reynhardt, 2014)

As shown in Figure 2.25, a solid-solid transition occurs during heating at 335K (65°C). Additionally, melting began at 338K (65oC) and ended at 363K (90°C). It is important to remember that the solid-solid transformation occurred prior to the melting phase. From the same study by Basson & Reynhardt, (2014), it was concluded that the primary difference between carnauba wax and beeswax was that the number of carbon atoms in methyl and methylene groups was approximately 12% lower in carnauba wax (80%) than in beeswax (92%). Carnauba wax, on the other hand, had a greater proportion of ester and carbonyl groups, as well as a greater proportion of triple and double-bonded carbon atoms. These variations had a clear effect on the chain motion and chain-length distribution.



Figure 2.26 DSC curve of carnauba wax extracted (Zheng et al., 2011)

Following, another DSC thermogram of carnauba wax is reported from Zheng et al., (2011). The softening point was 81°C, the freezing point was 85.7°C, and the melting point was 87.6°C. The crystallization phase began at 79.4°C, peaked at 75.8°C, and ended at 63.8°C. The following enthalpies were determined in this study: melting enthalpy, 204.3 J/g; crystallization enthalpy, 173.6 J/g. In the next Table 2.16, several different carnauba chemical compositions are displayed.

Table 2.16 Chemical composition of carnauba wax based on references.

	Composition	Reference
	82% wax esters (40% aliphatic, 13,2% ω-hydroxy and	(Basson &
	28,8% cinnamic aliphatic diesters), 11% fatty alcohols,	Reynhardt, 2014)
	5.5% fatty acids, 1% hydrocarbons and 0.5% moisture	
Carnauba	and inorganic substances.	
wax	75-85% aliphatic and aromatic (cinnamic acid based)	(Endlein, 2011)
	mono- and diesters.	
	Mainly esters of C24 and C28, carboxylic acids and C32	(Milanovic et al.,
	and C34 straight-chained primary alcohols.	2010)

2.8.3 Application of Waxes

According to Schromm, (2018), waxes are commonly used in a variety of industries due

to their purpose, characteristics, and properties that are compatible with the products they manufacture. Waxes are commonly used in the food industry to retain the consistency of food and extend its shelf life. Waxes are used in food coatings and are also a component of chewing gum.

Tinto et al., (2017) also stated that carnauba wax is commonly used in cosmetics, furniture wax, food, automotive, dental floss coating, and semiconductor molds. Carnauba wax is used as a cosmetic additive, most notably in lipsticks, mascara, and lip balms. This is because

carnauba wax has excellent emulsification properties and a high potential for oil binding with mineral oils and ester oils, resulting in a shiny and slippery surface.

Beeswax is commonly used in a variety of industries, such as the manufacture of wax base. Beeswax is a popular material used in the manufacture of candles and metal casting. Apart from that, beeswax is used in cosmetic modelling and is also used in the food processing industry, textiles, the technology industry, as well as varnishes and polishes. The industrial applications of waxes contribute to the growth of the industry and the production of a high-quality product (Tinto et al., 2017).

2.9 Plasticizer

Plasticizers added to thermoplastic starch have an effect on the properties and characteristics of the finished product. According to the International Union of Pure and Applied Chemistry's (IUPAC) Council, a plasticizer is a product or material that is combined with another material (plastic or elastomer) to increase its workability, distensibility, or versatility (Niazi, 2017).

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Another concept of plasticizer by Audic, & Chaufer, (2005) is the process of lowering the polymer glass transition temperature (Tg) and increasing the extensibility and durability of the polymer due to the presence of a low volatile organic compound. The presence of a plasticizer will improve the material's flexibility by weakening the intermolecular forces between polymer coils. This situation can also result in a decrease in the barrier's properties as a result of the free volume growth. In other words, plasticizers may contribute to the material's transformation from hard and brittle to more elastic and flexible. Another research by Sanyang et al., (2015) stated that plasticizers have an effect on the film thickness, solubility, and moisture content of SPS film-forming solutions. Plasticizers also serve as a catalyst, reducing the fragility and brittleness of unplasticized SPS films. The rising concentration of plasticizers results in a decrease in water absorption and density.

2.9.1 Glycerol

Essentially, glycerol is derived from plant fats and oils, both vegetable and animal. Glycerol is sometimes referred to as glycerin, glycerin, and glycyl alcohol. Glycerol's physical state is a sweet-tasting, colorless, and odorless substance. In the biological system, glycerol is converted to long chain fatty acids and esterified by three hydroxyl groups. Glycerol has the chemical formula $C_{3}H_{5}(OH)_{3}$ (Zou et al., 2016).

Due to the presence of three hydroxyl groups, glycerol has the capacity to absorb moisture from the air. This is one of the reasons why glycerol is suitable for use in the cosmetics industry, as it can obstruct the substance's drying process and render the water permanent (Gupta & Kumar, 2012). In table 2.17 shows the other detail properties of glycerol.

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Properties	Value
Molecular weight	92.09
Melting point (°C)	18.17
Boiling point @ 760 mm Hg (°C)	290
Density@ 20°C (g/cm3)	1.261
Viscosity	1499 c.p @ 20 °C (100% glycerol)
Specific heat	2.42 J/gm @ 26°C (99.94% glycerol)
Thermal conductivity (w/°K)	0.29
Refractive index	1.474

Table 2.17 Chemical properties of glycerol at 20.1 °C (Gupta & Kumar, 2012)

Another research that have been conducted by Nordin et al., (2020). This research used a solution casting method in which glycerol and/or thymol were applied to corn solution films. The physical, mechanical, structural, optical, and thermal properties were evaluated in this analysis.

As a result of the results obtained, it is clear that the presence of thymol and glycerol in corn starch films had an effect on the film thickness, especially as the thickness increased. Apart from that, glycerol and thymol reduced the solubility, clarity, and moisture content of the corn starch film. Additionally, using Fourier Transform Infrared Spectroscopy, the presence of intermolecular hydrogen bonds between corn starch, thymol, and glycerol was determined (FTIR). Corn starch (CS), corn starch and glycerol (CS-Gly), corn starch and thymol (CS-Thy), and corn starch with glycerol and thymol (CS-Gly-Thy) films are depicted in Figure 2.27.



Figure 2.27 FTIR spectra reading of (i) CS, (ii) CS-Gly, (iii) CS-Thy and (iv) CS-Gly-Thy films (Nordin et al., 2020)

In terms of mechanical properties, the presence of glycerol in corn starch films has a greater impact on the tensile test than the presence of thymol, whereas the presence of both glycerol and thymol has a synergistic effect. Mechanical properties such as tensile strength (MPa), elongation at break (%), and Young's modulus are evaluated in Figure 2.28 (MPa).



Figure 2.28 Mechanical properties of corn starch with glycerol and/or thymol:

(i) Tensile strength (MPa), (ii) Elongation at break (%) and (iii) Young's Modulus

(MPa) (Nordin et al., 2020)

Furthermore, the thermal properties of corn starch with thymol and glycerol demonstrate a change in the overall degradation temperature of corn starch films with thymol and glycerol. UNIVERSITE TEKNIKAL MALAYSIA MELAKA This condition demonstrated that the corn starch film containing thymol and glycerol was more thermally stable than the other films. The thermogravimetric curves for all forms of corn starch films used in this analysis are shown in Figure 2.29. Thus, the addition of plasticizers such as glycerol and thymol significantly improved the performance properties of corn starch films.



Figure 2.29 Thermogravimetric curves of CS, CS-Gly, CS-Thy and CS-Gly-Thy films (Nordin

et al., 2020)

2.9.2 Sorbitol

Sorbitol is a type of plasticizer that, like glycerol, is derived from the polyols group. Sorbitol is a transparent solution that is colorless and has a pleasant sweet taste. The general properties of sorbitol are mentioned in Table 2.18.

Table 2.18 General properties of sorbitol

Properties T TEKNIKA	MALAYSAObservations
Color	Watery white
Taste	Pleasantly sweet
Odor	Essentially odorless
Water content	29.5
pH (10% solution)	6.5
Refractive index (20°C)	1.45
Thermal stability	160°C - 180°C

Sanyang's team has already investigated the effect of glycerol and sorbitol plasticizers on sugar palm starch-based films' physical and thermal properties (Sanyang et al., 2015). The sugar palm starch films were produced using a traditional casting technique and then analyzed using glycerol and sorbitol. The film thickness, density, moisture content, solubility, and thermal analysis were determined in this study. The researchers discovered that sorbitol-coated sugar palm starch films have a higher density and thickness. The thickness of glycerol and sorbitol was compared in Figure 2.30, whereas the density of sugar palm starch films with and without the addition of glycerol or sorbitol is shown in Figure 2.31.



Figure 2.30 Thickness test of sugar palm starch with glycerol or sorbitol (Sanyang et al., 2015)



Figure 2.31 Density analysis of sugar palm starch films and with additional of glycerol or sorbitol (Sanyang et al., 2015)

Subsequently, the solubility and moisture content of sugar palm starch films containing glycerol is higher than those containing sorbitol. Thermal analysis reveals that the presence of sorbitol results in an increase in thermal degradation temperature, whereas the addition of sorbitol results in an increase in thermal stability as compared to glycerol. As a result, the

addition of plasticizers derived from glycerol and sorbitol enhanced the brittleness, durability, and peel ability of sugar palm starch films.



Figure 2.32 Percentage of solubility test for sugar palm starch films with glycerol and sorbitol



(Sanyang et al., 2015)



Figure 2.34 Thermal-Gravimetric analysis of sugaar palm starch films with glycerol and sorbitol (Sanyang et al., 2015)

2.10 Summary

The following hypothesis has been reached based on a study of the literature on natural fiber, composites, starch, thermoplastic starch, waxes, and plasticizers:

- i. The incorporation of natural fibers into composites has a beneficial effect on the environment by repurposing waste from natural fibers such as Napier grass and transforming it into useful products.
- ii. Thermoplastic starch reinforced with natural fiber contributes to the reduction of damage to the environment since it is a biodegradable product that degrades readily in the soil.
- iii. Napier grass fibers can be considered a new sustainable resource for thermoplastic reinforcement.
- iv. Chemical treatment of the fiber improves the interfacial bonding between the fiber and matrix.

Thus, though numerous studies on thermoplastic starch have been conducted, it was discovered that no studies on the incorporation of Napier grass fiber into thermoplastic cassava starch have been conducted

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CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter discusses the raw material in detail, the production procedure of thermoplastic cassava starch reinforced with Napier grass fiber (NGF), and the testing used to determine the raw material's qualities. The flowchart below presents the research approach used in this study.





Figure 3.1 Flow of Research Methodology

3.2 Material

3.2.1 Napier Grass Fiber

The Napier grass was sourced from a local farm in Melaka's Jasin district. These Napier grasses are pretty easy to grow, especially in arid places. A normal water retting procedure removed the fibers of the Napier grass. Retting is a microbiological process that breaks the compound bonds that hold the stem together, allowing the bast fibers to detach from the woody center.

Water retting generates fibers that are homogeneous and of excellent quality. The stems were immersed in water tanks, and their development was tracked daily. The Napier grass was removed from each stem's internodes prior to retting. Following that, the stems were immersed for approximately three to four weeks in a water tank filled with tap water, as illustrated in Figure 3.2.

After thoroughly cleaning the soaked Napier grasses, each fiber strand was retrieved. Following that, the fiber was dried in the sun for 5 to 6 hours, or until totally dry. The dried fiber is then cut into small pieces to facilitate the grinding process when using a dry grinder. The grinder will ground the fiber until it resembles a powder. The fiber's moisture content was depleted. The fibers were laid on aluminum foil and then placed in an oven set to 100°C for 5 hours following the grinding operation.



Figure 3.2 Napier grass at Jasin, Asahan



Figure 3.3 Process of extracting Napier grass fiber

3.2.2 Cassava Starch

The primary material for this investigation was cassava starch from supplier Antik Sempurna Sdn. Bhd. Malaysia. In essence, cassava starch appears in white powder as other starches.



Figure 3.4 Cassava Starch

3.2.3 Glycerol

The addition of a plasticizer to the study can increase the product's flexibility and improve its characteristics. The plasticizer in this investigation was glycerol ($C_3H_8O_3$), which QReC (Asia) Sdn Bhd manufactured. QReC Chemical's glycerol comprised 99.5 percent AR grade and had 92.10 g/mol molar weight. Glycerol is a mildly viscous liquid with a transparent appearance. The chemical makeup of glycerol is listed in Table 3.1.



Figure 3.5 Glycerol contained 99.5% AR grade.

Chemical Composition	Percentage Value
Assay (Acidimetric)	Min 99.5%
Insoluble in water	Passes test
Acidity/Alkalinity	Passes test
Halogen Compounds (as C	l) Max 0.003%
Chloride (Cl-)	Max 0.001%
Sulfates (SO4)	Max 0.001%
Ammonium (NH4)	Max 0.0015%
Arsenic (As)	Max 0.0001%
Copper (Cu)	Max 0.001%
Heavy Metal (as Pb)	Max 0.0005%
Iron (Fe)	Max 0.0005%
Lead (Pb)	Max 0.001%
2) Nickel (Ni)	Max 0.0005%
Zinc (Zn)	Max 0.001%
Aldehydes (HCHO)	Max 0.0005%
1,2,4-butanorial (G.C)	Max 0.2%
Sulphated Ash	Max 0.01%
Water	Max 2%

3.2.4 Beeswax

Beeswax is a significant raw ingredient in this investigation. The beeswax was procured from Aldrich Chemistry and was in solid form. Beeswax ranges in color from pale yellow to yellow-tan, and it also has a honeybee scent. Beeswax acts as a moisture repellent, preventing thermoplastic cassava starch from decaying due to the presence of moisture.



Figure 3.6 Beeswax

3.3 Preparation of Samples

3.3.1 Preparation of Thermoplastic Cassava Starch

The process of thermoplastic cassava starch production comprises two steps: weighing and combining cassava starch and glycerol and using a hot press machine. When weighing and combining the cassava starch and glycerol, the cassava starch was weighted to a ratio of 100 g, while the glycerol was weighted to a final concentration of 30 g. Cassava starch and glycerol were weighed, manually dried, and mixed until uniformly distributed. The mixture was then subjected to high-speed dry mixing using a dry blender set at 1200 rpm for 3-5 minutes or until the mixture was uniform.

The well-mixed cassava starch and glycerol combination was weighted to 58.20 g in order to fill the mold of the hot press machine. 58.20 g was the quantity required to fix the mold for the hot press operation. The following section included using a hot press machine, which must be prepared to a temperature of 155 °C. While the machine was preheating, the mold was prepared by lining it with a Mylar sheet to facilitate the process of removing the sample mixture from the mold.

Mylar film is used to prevent the sample mixture from adhering to the mold during the hot press process and compress the sample mixture in the mold. After the warmed session reached a temperature of 155 °C, the mold containing the mixture sample was put in the hot press machine. The hot press procedure takes an hour at a temperature of 155 °C. As a result, the sample combination subjected to this method was termed 100% thermoplastic cassava starch.



Figure 3.7 Preparation of the Mixture of Thermoplastic Cassava Starch (TPCS)



Figure 3.8 Fabrication of the Mixture of Thermoplastic Cassava Starch (TPCS)

3.3.2 Preparation of Thermoplastic Cassava Starch with Beeswax

The manufacture of thermoplastic cassava starch with beeswax followed using the previously prepared combination. However, because the ratio of thermoplastic cassava starch to beeswax is 90:10, the amount of beeswax is 10% of the amount thermoplastic cassava starch (58.20g).

After cutting the beeswax into tiny pieces, it is weighed at 5.82g and combined with the thermoplastic cassava starch. The mixture dried combined using a blender set to 1200 rpm until fully blended. Then line the mold with a Mylar sheet and fill it with the thermoplastic cassava starch beeswax mixture. The mold was placed in a hot press 155°C for 35 minutes, resulting in the formation of thermoplastic cassava starch beeswax.



Figure 3.9 Preparation of the Mixture of Thermoplastic Cassava Starch with Beeswax



Figure 3.10 Fabrication of the Mixture of Thermoplastic Cassava Starch with Beeswax

3.3.3 Preparation of Thermoplastic Cassava Starch Reinforced with Napier Grass Fiber

The fabrication of thermoplastic cassava starch composites with Napier grass fiber was accomplished by including the previously created thermoplastic cassava starch composites with Napier grass fiber. As described in Section 3.2.1, the Napier grass fiber has been processed previously.

The thermoplastic cassava starch mixture and fiber were based on the stated fiber percentages: 0%, 10%, 20%, 30%, 40%, and 50%. 5.82g of fiber (10wt%) and 52.38g of thermoplastic cassava starch matrix are utilized. The material dried mixed before being placed into the mold. After that, it was placed in a hot press set to 165°C for one hour. A similar procedure was repeated for the remaining fiber percentages.



Weighted the mixture of TPCS



Dry mixing for 3-5 minutes



Weighted the NGF

Specimen removed from mold

Placed in the hot press for 1 hour

Compacting the mold with the mixture

Figure 3.11 Fabrication of Thermoplastic Cassava Starch Reinforced by Napier Grass Fiber

3.4 Characterization of Samples

3.4.1 Moisture Content

The procedure for assessing the moisture content was identical to that for the previous tests, in that three portions of the sample were made with dimensions of 10mm x 10mm x 3mm. Weighing balances were used to determine the samples' starting weight (Wi). All samples were dried in an air circulation oven set to 105 °C for 24 hours. After that, the samples were removed from the oven. Their final weights (W_f) were determined immediately to avoid the samples from absorbing moisture from the surrounding environment during the weighing procedure. The moisture content of the samples was determined as a percentage using the procedure below.



Figure 3.12 Methodlogy of Moisture Content

3.4.2 Water Absorption

Three samples of thermoplastic casava starch reinforced with Napier grass fiber are prepared. Each sample measures 10 mm x 10 mm x 3 mm. This test was conducted in accordance with ASTM D570-98, with minimal modifications to the sample size. The samples were cut to the specified size and placed in an air-circulating oven set at 105 °C \pm 2

for 24 hours. The goal of drying the sample was to remove any remaining moisture. Following that, the samples' initial weight (W_i) was determined using a weighing scale, and the samples were submerged in water at room temperature (23 °C1) for 0.5 and 2 hours, respectively. Following that, the samples' final weight (W_f) was determined using a weighing scale. The data acquired was utilized to calculate the percentage of water absorption using the ASTM D570-98 methodology.

Percentage of Water Absorption (%) =
$$\frac{Wf - Wi}{Wi} \times 100\%$$
 (3.2)



Figure 3.13 Methodology of Water Absorption

3.4.3 Thickness Swelling

The ASTM D570-98 standard was used as the reference standard for the thickness swelling test, with modifications to the size of the samples to be tested. Due to the dimensions of 10 mm x 10 mm x 3 mm, the samples of thermoplastic casava starch reinforced with Napier grass fiber were cut. The samples were then put in an air circulation oven to eliminate any remaining moisture. The oven was set to 105 °C±2, and the drying time was set to 24 hours. Following that, the samples were first measured using a Vernier Caliper Mitutoyo model with an accuracy of 0.01 cm (T_i), then submerged in water for 0.5 hours and 2 hours at room temperature (23 °C1). The samples were then measured for the final dimension (T_f) using the same Vernier Caliper. The following formula was used to measure the percentage of thickness swelling, which was obtained from ASTM D570-98.

Percentage of Thickness Swelling (%) =
$$\frac{Tf - Ti}{Ti} x \, 100\%$$
 (3.3)



Figure 3.14 Methodology of Thickness Swelling

3.4.4 Water Solubility

Adjustment for the cutting section in the sample preparation is made based on the reference for water solubility research from Azahari et al., (2011). The specimen was prepared for testing by cutting it to a size of 10mm x 10mm x 3mm and then weighing it for

the initial weight (W_i) using a weighing scale. Following that, the samples were dried for 24 hours at a temperature of 105 °C \pm 2 in an air circulating oven.

Following that, the samples were submerged in 30 ml of distilled water and gently stirred. After 24 hours, the samples were removed from the container, and the filter paper on the surface absorbed the residual water on the sample surface. Following that, the samples dried for 24 hours at a temperature of 105 °C \pm 2 in an air circulating oven. The samples' final weight (W_f) was determined, and the weight reduction percentage was computed using the method below.



Figure 3.15 Methodology of Water Solubility

3.4.5 Soil Burial

Ibrahim et al., (2014) investigated the properties of a starch-based biodegradable composite reinforced with date palm and flax fibers. In this investigation, all three samples of each formulation were buried around 10 cm deep by the dimensions of the samples, which were approximately 40 mm x 8 mm x 3 mm. The soil burial test was conducted in a somewhat different manner than the previous tests. The samples were chopped into three equal-sized pieces measuring 40mm x 8mm x 3mm. The samples were then weighed for the initial weight (W_i) using a weighing scale and then put in an air circulating oven set to 105 °C for 24 hours. Following that, the sample was buried in the soil to a depth of 10 cm using a 50/50 combination of sand and soil, and the temperature was maintained at 30 °C2. The original water content of the soil was maintained by adding 400 ml water every three days to each of the 1250 g mixtures.

The plastic mesh was used in this investigation to assist in removing samples from the soil burial procedure prior to burying them in the soil. The use of plastic mesh allowed microorganisms and moisture to remain in contact with soil samples. The setup experiment was designed to conduct two types of determinations, one lasting four weeks and another lasting eight weeks. After four and eight weeks, the soil samples were removed and washed with distilled water to remove contaminants. Following that, the samples were dried for 24 hours at a temperature of 105 °C in an air circulating oven and then weighed to determine the final weight (W_f). The percentage weight loss was computed using the method found in ASTM D 6003-96 for calculating mass loss during the soil burial test.



3.4.6 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR was used in this investigation to determine the presence of functional groups in thermoplastic cassava starch reinforced with Napier grass fiber samples. Fourier transform

infrared (FT-IR) spectroscopy was used for the study. The laboratory's FTIR instrument is a

JASCO FT/IR-6100 IR spectrometer from Japan.



Figure 3.17 FTIR Spectroscopy Machine

3.4.7 Scanning Electron Microscopy (SEM)

The purpose of using scanning electron microscopy (SEM) equipment is to determine the morphology of thermoplastic casava starch reinforced with Napier grass fiber samples. The scanning electron microscope (SEM) used is a Zeiss Evo 19 Research from Germany.



Figure 3.18 Scanning Electron Microscopy (SEM) Machine

3.4.8 Density

Density testing was conducted using the ASTM D 1895 standard as a guide for the testing procedure. The samples were chopped to a size of 10mm x 10mm x 3mm based on their appropriateness and test circumstances. The samples were then dried in an aircirculating oven set to 105 °C2 for 24 hours. The samples were chilled in a desiccator with powdered silica gel and then weighed and volumetrically determined after drying. The density of the samples was determined by weighing them and measuring their volume. The samples were weighed and measured using a Vernier Caliper Mitutoyo model with a 0.01 cm accuracy. The weight and volume collected for density determination were computed using the method provided below.

Density
$$\frac{(g)}{cm3} = \frac{mass(m)}{volume(v)}$$
 (3.6)



3.4.9 Process Production of the Packaging Tray

The process production of packaging tray accomplished by including the previously created thermoplastic cassava starch composites with Napier grass fiber. As described in Section 3.2.1, the Napier grass fiber has been processed previously.

The thermoplastic cassava starch mixture and fiber were based on the fiber percentages stated, which are 30%. 17.46g of fiber (30wt%) and 40.74g of thermoplastic cassava starch matrix are utilized. The material dried mixed before being placed into the mold. After that, it was placed in a hot press set to 155°C for one hour.



CHAPTER 4

RESULTS AND ANALYSIS

4.1 Introduction

In this chapter, the results of every testing, physical, environmental, and others, were comprehensively discussed. The water affinity testing consists of Moisture Content, Water Absorption, and Thickness Swelling, the environmental testing consists of Water Solubility and Soil Burial, while other testing consists of Fourier-Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), and density.

4.2 Water Affinity Testing

4.2.1 Moisture Content

Moisture content testing is a necessary procedure performed on the TPCS matrix and composite. The moisture content in a composite material refers to the amount of water contained inside the composite material. This circumstance may affect the material's performance. This study aims to determine the amount of moisture present in a sample of TPCS with NGF. High moisture content has a detrimental effect on the stability of the composite, particularly on tensile strength, porosity formation, and dimension. As a result, an excellent composite fiber should have a decreased moisture content.

The proportion of moisture content in TPCS/NGF is shown in Figure 4.1. Generally, the findings indicate that increasing the fibre content of TPCS/NGF decreases the moisture content. This effect might be due to a reduction in the polysaccharide matrix's mobility due to the fibre inclusion, resulting in a decrease in the moisture content of the material composites (Maran et al., 2014). 100

The moisture content of TPCS/NGF, on the other hand, fluctuates significantly. This is shown in Table 4.1 by the analysis of variance (ANOVA) of moisture content. Because the P-value is less than 0.05, a statistically significant difference exists between the mean data for one level composite and the mean data for another level composite. This demonstrates that adding NGF at a concentration of 10% to 50% has a considerable influence on the moisture content of the composite material. This might be due to the fact that NGF is less hydrophilic than the TPCS matrix combination. The conclusion obtained is consistent with Maran et al., (2013) findings on tapioca starch moisture content, which indicates that moisture content decreases as fiber content increases. Thus, it was established that the presence of natural fibres affected the moisture content of a composite material.



Figure 4.1 Percentage of Moisture Content of TPCS/NGF with different fiber loading

Table 4.1 The analysis of variance (ANOVA) of moisture content

Variables	df	Moisture content
Mixture	5	0.00*
	*Note: Significantly differen	It at $p \leq 0.05$

4.2.2 Water Absorption

Water absorption is hard to undertake during the composite material's characterization. Water absorption testing aims to ascertain the quantity of water absorbed by a substance under certain conditions. Moisture diffusion in a composite material is

influenced by a number of elements, including the presence of voids, the volume fraction of fibre, the temperature, the humidity, and the matrix viscosity (Saw et al., 2014).

Figure 4.2 illustrates the proportion of water absorbed by TPCS/NGF with varying fibre loading percentages. In general, the addition of NGF to the TPCS matrix has a considerable effect on the outcome. This is shown in Table 4.2 by the analysis of variance (ANOVA) of water absorption. Because the P-value is less than 0.05, a statistically significant difference exists between the mean data for one level composite and the mean data for another level composite. Moreover, this pattern demonstrates that when NGF is combined with the TPCS matrix, an influence on water absorption occurs.

Furthermore, the result indicates that the percentage of water absorbed decrease with increasing fibre content for 0.5- and 2-hour soaking times. According to the graph, a fibre concentration of 0% for 0.5 and 2 hours indicates that 42.42% and 96.48% of water is absorbed. However, the proportion changes when NGF is included. The presence of fibre reduces the amount of water absorbed owing to the fact that starch is more hydrophilic than cellulose fibre, implying that the presence of fibre may reduce the hydrophilicity of the starch matrix (Prachayawarakorn et al., 2013).

This graph pattern is influenced by the hydrophilic nature of NGF, which allows water molecules to diffuse freely across the materials in the composite (Jumaidin et al., 2017a). The presence of fiber decreases the proportion of water absorbed, as starch is more hydrophilic than cellulose fiber, implying that the presence of fiber may reduce the hydrophilicity of the starch matrix (Yang et al., 2007).


Figure 4.2 Percentage of Water Absorbed of TPCS/NGF with different fiber

loading for 0.5 hour and 2 hours.

Table 4.2 The analysis of variance (ANOVA) of water absorption

Variable	df	Water Absorption
Mixture	5	0.00*
1	*Note: Significantly differen	t at p≤0.05
ă .	7	
5.		
4.2.3 Thickness Swelling		

The goal of a thickness swelling test is to measure a material's swelling ratio by immersing it in water for a specified period of time. Thickness swelling is also utilized to determine the material's dimensional stability changes, which is critical when the characteristic is related to the material's performance. The thickness swelling technique was utilized to investigate the changes in the dimensional stability of TPCS when NGF composite was added.

The percentage of thickness swelling for TPCS with NGF composites at various fibre loading percentages is shown in Figure 4.3. In general, the addition of NGF to the TPCS matrix has a considerable effect on the outcome. This is shown in Table 4.3 by the analysis of variance (ANOVA) of thickness swelling. Because the P-value is less than 0.05, a statistically significant difference exists between the mean data for one level composite and the mean data for another level composite.

The outcome indicates the percentage of swelling thickness for a TPCS/NGF composite after 0.5 and 2 hours of water immersion. The graph depicts the decreasing percentage of thickness swelling as the fibre loading increases. This trend indicates that fibre insertion had an effect on the composites' thickness swelling behaviour. This result may be attributed to the inclusion of fibre in the composites, which has a more rigid structure than starch and so provides the composites with more dimensional stability (Jumaidin et al., 2019). Owing to the existence of wax on its surface, water absorption is limited to a certain amount. The result is consistent with Ramírez et al., (2011), water absorption is reduced by the presence of wax on its surface and the chemical composition of lignocellulosic fibres.



Figure 4.3 Percentage of Thickness Swelling of TPCS/NGF with different

fiber loading for 0.5 hour and 2 hours

Variables	df	Thickness Swelling
Mixture	5	0.00*
	1 1 1 CC	1 1 10 0 <i>E</i>

*Note: Significantly different at p≤0.05

4.3 Environmental Analysis

4.3.1 Water Solubility

The goal of a water solubility test is to evaluate the weight loss of a substance when subjected to continuous stirring for a specified amount of time. Based on Edhirej et al., (2017)'s research, water solubility reflects the material's decomposition behavior when disposed of in water. Thus, water solubility refers to the evaluation of the TPCS matrix's water resistance after integration of NGF composite.

According to Figure 4.4, the proportion of weight loss varies according to the quantity of fibre loaded. In general, the addition of NGF to the TPCS matrix has a considerable effect on the outcome. This is shown in Table 4.4 by the analysis of variance (ANOVA) of water solubility. Because the P-value is less than 0.05, a statistically significant difference exists between the mean data for one level composite and the mean data for another level composite.

The graph demonstrates that as fibre loading increases, the proportion of weight loss decreases. The composites' lower solubility than TPCS might be ascribed to NGF's superior water resistance, which assists in preventing water absorption that could result in material breakdown and dissolution. Moreover, the use of natural fibre helps to minimise material disintegration by forming a fibre network in the composites (Jumaidin et al., 2017b). As a result of these interactions, TPCS/NGF is resistant and stable. Ilyas et al., (2018) reported that these interfaces strengthened the cohesive qualities of the biopolymer matrix and decreased water sensitivity by preventing water molecules from breaking these strong interactions.

The decrease in water solubility percentage might be attributed to the TPCS/NGF composite's increased water resistance. Similar findings were obtained when a hybrid composite made of cassava bagasse and sugar palm fibre was added to the TPCS matrix, reducing the films' water solubility (Edhirej et al., 2017). The results were consistent with those of Shen et al., (2010) who found that the water solubility of pure corn starch foam was 20% and reduced with the addition of chitosan.





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Table 4.4 The ana	ysis of variance	(ANOVA)	of water solu	ıbility
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Variables	df	Water Solubility
Mixture	5	0.03*
	*NI (C' 'C' (1 1'CC	· · · <0.0 <i>E</i>

*Note: Significantly different at p≤0.05

4.3.2 Soil Burial

Soil burial tests can be used to measure the rate of biodegradation. Soil burial testing is one of the critical tests used to assess the weight loss of the material composite throughout the burial process. According to Vasile et al., (2018) the primary objective of developing new materials is to ensure that they disintegrate easily in the natural environment. Several elements contribute to the degradation process, including the soil's pH, temperature, the presence of microorganisms in the soil, humidity, and the composite material's character.

Adding NGF composite to the TPCS matrix has a negligible influence on the output. The analysis of variance (ANOVA) of soil burial demonstrates this in Table 4.5. Due to the fact that there is no statistically significant difference between the mean data for one level composite and the mean data for another level composite.

The percentage weight decreases of TPCS/NGF samples for 4 and 8 weeks, respectively shown in Figure 4.5. In general, the data indicates that inrease fiber content, decrease the weight loss. The graph demonstrates that fibre content of 20% achieved the greatest weight loss throughout the four weeks after burial, at 72.05%. After eight weeks of burial, the 0% fibre content achieves a 100% weight loss, indicating complete breakdown. However, 50% fibre content results in the lowest proportion of weight loss at 52.19%, whereas the graph pattern indicates that weight loss decreases as fibre content increases. This indicates that the presence of NGF influenced the pace of composite breakdown.

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This conclusion might be attributable to the greater hydrophilic nature of NGF, which encourages microbe growth and increases the weight loss of the materials during decomposition (Maran et al., 2014b). This study indicates that the NGF is very effective at speeding up the biodegradation process of biocomposites. Previously, similar findings were reported for the incorporation of agar (Maran et al., 2014b), carrageenan (Prachayawarakorn & Pomdage, 2014) and sodium alginate into thermoplastic starch matrix (Weerapoprasit & Prachayawarakorn, 2016).



Figure 4.5 Percentage of weight loss for soil burial testing of TPCS/NGF in 4 and 8 weeks

	Table 4.5 Th	e analysis	of variance	(ANOVA) of soil	burial.
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Variables	df	Soil Burial
Mixture	5	0.1395
k	$(N_{1}, \dots, C_{1}) = (C_{1}, \dots, C_{n})$	-+ - <0.05

*Note: Significantly different at $p \le 0.05$

4.4 Physical Analysis

4.4.1 Fourier-Transform Infrared Spectroscopy (FTIR)

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Fourier Transform Infrared Spectroscopy was used to identify the chemical structure of the material composite (FTIR). Further, FTIR is employed to identify the chemical bonding characteristics of a material composite, in this example between fibre and starch. Each peak in Figure 4.6 represents a functional group involved in the interaction of starch and fibre. Thus, using FTIR spectroscopy, the chemical interaction between TPCS and NGF was identified. The FTIR spectrometer of TPCS/NGF composites ranging from 0% to 50% fibre content is shown in Figure 4.6. Generally, comparable band patterns are exhibited for fibre content levels of 0%, 10%, 20%, 30%, 40%, and 50%.

This pattern indicates that the chemical makeup of materials such as cassava starch, glycerol, beeswax, and NGF is unaffected by the mixing procedure. Prachayawarakorn & Hwansanoet, (2012) found the same outcome when silk protein fibres were combined with TPS rice starch.

It was discovered that the presence of hydroxyl groups, O-H, in the TPCS/NGF ranged from 0% to 50% fibre content, which was positioned between 3400 - 3200 cm-1. Cassava starch and NGF were the primary contributors to the O-H group's presence. Sarifuddin et al., (2012) discovered that the presence of hydrogen bonds occurred with the inclusion of fibre in a thermoplastic of sago starch. Moreover, the feature of C-H stretch was shown in the band range 2936–2916 cm-1. This is described by Jumaidin, (2017), who said that the cellulose and hemicellulose found in natural fibre contribute to the presence of the C-H band. Meanwhile, the wavelength range of 1075 – 1000 cm-1 indicates the presence of a C-O stretch. Mendes et al., (2016) found that thermoplastic chitosan with maize starch had a comparable effect on the existing C-O stretch. The lignin in the natural fibre impacted the presence of C-O stretch (Jumaidin, (2017).

In combination, it can be noticed that the presence of O-H bands varies gradually as the fibre content decrease. According to Figure 4.6, the wavenumber of O-H bands decrease from 3292 cm-1 to 3257 cm-1 when the fibre concentration rising from 50% to 0%. This discovery is consistent with the findings of Jumaidin, (2017), who said that the interaction of intermolecular hydrogen bonding had an effect on the spectra band peaks.



Figure 4.6 FTIR Spectroscopy of TPCS/NGF composite with different percentage of fiber

loading

4.4.2 Scanning Electron Microscopy (SEM)

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Scanning Electron Microscopy (SEM) is used to examine the surface morphology of a material composite. SEM micrographs can be used to visualize the contact between the matrix and fibres of the composite material. Through SEM micrographs, the research of starch-based composites revealed several types of failure, including fiber fracture, matrix failure, and fiber-matrix interface failure.

The SEM micrograph of NGF is shown in Figure 4.7. Essentially, the fibre has a nonuniform structure and a rough surface. This might be because the fibre was not subjected to any chemical treatment during the processing step (Ishak et al., 2013). The SEM micrographs of TPCS containing 0%, 10%, 20%, 30%, 40%, and 50% NGF are shown in Figure 4.8(A), Figure 4.8(B), Figure 4.8(C), Figure 4.8(D), Figure 4.8(E), and Figure 4.8(F). In general, the rising fibre content in the TPCS/NGF indicates that the samples' composites have a distinct microstructure. According to a recent work by Edhirej et al., (2017), SEM may be used to analyse the interfacial adhesion and microstructure of cassava starch and reinforced biocomposites. Under electron microscopy, the surface morphology of the tensile fracture structure was observed. SEM micrographs of the tensile cracked surfaces of TPCS composites with varying proportions of NGF are shown in Figure 4.8.

The 0% TPCS/NGF is depicted in Figure 4.8(A). The homogeneous structure of the NGF composites with no visible phase separation may be ascribed to the fiber's high miscibility in TPCS throughout processing. The image shows the approximate surface of a 0% TPCS/NGF composite under SEM microscopy, with only the matrix of starch and beeswax visible. The glossy surface of the sample indicates the presence of beeswax, which alters the starch structure. Further, the construction with 0% fibre content has a uniform surface free of obvious air pockets. This demonstrates that the addition of glycerol enhanced both the plasticization and mixing of starch melts (Jumaidin et al., 2016).

The coarse surface of NGF seems to facilitate attachment between the NGF and the TPCS matrix in Figure 4.8. (B). On the surface of the tensile fracture structure, the fibre fracture is plainly visible. This result indicates that stress is effectively transferred from the TPCS biopolymer matrix to the NGF, thereby reinforcing the composites (Jumaidin et al., 2016). Considering, glycerol spreads and shapes starch granules in TPCS at a persistent level.

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The micrograph clearly demonstrates fibre pull-out, and in particular, NGF are seen to be more damaged in Figure 4.8. (E). The compact structure of the TPCS ensures that no air pockets form on the homogeneous surfaces. This result is obtained by dry mixing cassava starch with glycerol, which improves the plasticization of cassava starch.

Moreover, it can be shown that TPCS and NGF are extremely compatible, as seen by the matrices' excellent fibre wetting, as illustrated in Figure 4.8. (B). This behaviour might be explained by the same hydrophilic properties of TPCS and NGF, which result in good adhesion between them. Likewise, this discovery is confirmed by a comparable pattern of fibre breakdown seen in TPS/Kenaf and TPS/- sugar palm fibre biocomposites (Zainuddin et al., 2013) (Sahari et al., 2014).





Figure 4.8 Scanning electron micrograph of TPCS reinforced by NGF composites (a) 0% fiber content, (b) 10% fiber content, (c) 20% fiber content, (d) 30% fiber content, (e)40% fiber content, and (f) 50% fiber content.

4.4.3 Density

According to Al-Oqla & Sapuan, (2014), density testing is a critical attribute when forming a composite material. The performance of the final product may be influenced by its density in terms of weight, convenience of use, energy consumption, transportation costs, as well as manufacturing and handling processes. Thus, it is important to examine the influence of Napier grass on density in order to determine the biopolymer's potential as a biodegradable alternative to currently used non-biodegradable polymers.

Supplementing the TPCS matrix with NGF has a minimal effect on the outcome. In Table 4.6, the analysis of variance (ANOVA) of soil burial reveals this. Due to the lack of a statistically significant difference between the mean data for one level composite and the mean data for another. The density of TPCS in combination with NGF is shown in Figure 4.9. Generally, it was noticed that the density slightly decreases as the fibre content increases. This result demonstrates that including NGF altered the density of the composite material. Edhirej et al., (2017) obtained a similar result for a cassava/sugar palm fibre reinforced cassava starch hybrid composite.

At 0% NGF content, the density gained is 1.028 g/cm3, whereas at 50% NGF content, the density gained is 0.925 g/cm3. This condition might be caused by the presence of voids in the composite material as a result of raising the fibre content percentage. According to Ibrahim et al., (2014) the rising porosity of starch-based biodegradable composites supplemented with date palm and flax fibres is caused by an inadequate matrix covering the fibres' surface. Thus, voids emerge as a result of increased fibre loading in composite materials.



Figure 4.9 Density of TPCS/NGF composite with different percentage of fiber loading

	Table 4.6 The	e analysis	of variance	(ANOV	'A) of	density.
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Variables	df	Density
Mixture	5	0.8110
	*Nota Significantly differen	t at m < 0.05

*Note: Significantly different at $p \le 0.05$

4.4.4 Fabrication of Packaging Tray

This study developed a Napier grass packaging tray at the end of the discussion. The methodology of fabricating the prospective product with dimensions of 12cm x 12cm x 1cm. The method started with the physical mixing of four main ingredients: cassava starch, beeswax, glycerol, and NGF. Following that, continue blending with the blender until the mixture is homogeneous. In this manufacturing technique, fiber content of 30% was used. The mixture was then put on a mold lined with Mylar film to facilitate removal. Following that, the mold was put in a hot press machine set to 155 °C for 1 hour and then cooled for 10 minutes.

For the design packaging, there is a star of the Malaysian flag in the middle, which refers to the unity of 13 states and the federal government. The corner part is designed with geometric shapes, despite experiencing difficulties during processing, but the results are impressive.

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Figure 4.10 TPCS with 30% NGF sample tray



Figure 4.11 Perspective view



Figure 4.12 Back view



Figure 4.13 Corner view



CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Numerous researchers have undertaken research on the production of ecologically friendly and sustainable materials. At this point in time, the creation of new biodegradable products is critical to preserving nature. The aim of this study is to document the advancement of an innovative material derived from cassava starch. This study has three main objectives and conclusion are as follows:

i. To produce biodegradable thermoplastic cassava starch reinforced with Napier grass fiber composite.

The biodegradable thermoplastic composite was successfully created utilising dry mixing and a hot-pressing machine. It was generated from cassava starch with the addition of NGF. TPCS with NGF composite samples were made at various fibre loading percentages and used to determine the physical and environmental parameters for each percentage.

ii. To investigate the water affinity properties and the environmental properties of the biodegradable thermoplastic cassava starch reinforced with Napier grass fiber composite.

This study revealed that water absorption and moisture content result is consistent as fibre content rises, the percentage of moisture and water absorb decreases. For thickness swelling, the data indicates that decreasing percentage of thickness swelling as the fibre loading increases. Water solubility findings demonstrate that as fibre loading rises, weight loss decreases, showing that the material is less soluble. For soil burial tests, all samples were decreases as the time of soil burial increases. The FTIR spectrum demonstrates chemical connection between the fibre and matrix, whilst the SEM micrograph demonstrates a change in the composite's structure as fibre concentration rises.

iii. To produce biodegradable packaging derived from Napier grass fiber

At the completion of this study, Napier grass packing tray has been developed. The technology used to fabricate the prospective product is 12cm x 12cm x 1cm. The design packaging has a star from the Malaysian flag in the centre, referring to the country's 13 states and federal government's unity. Despite processing challenges, the corner section is built using geometric forms, and the results are stunning.

5.2 Recommendation for Future Research

- i. To investigate an innovative material manufacturing procedure for high-volume production, such as injection moulding.
- ii. To investigate an appropriate coating process for the material in order to enhance the material's moisture qualities.
- iii. To investigate a suitable technique for extracting Napier grass fibre, particularly for large volume extraction.
- iv. To investigate an innovative form of natural fibre for application in the fabrication of the composite material.

5.3 **Project Potential**

This innovation has a strong potential for commercialization in industry as a singleuse tray with minor modifications to the manufacturing process. This product has a strong potential for use as a substitute for synthetic plastic. Figure 5.1 indicates the prospective result of new material development, while Figure 5.2 represents another use of tray reinforced with NGF, which is used to skincare, lipstick, and also remote control organizer. Another feature that adds value to this product is the cheap cost of the raw materials utilised. The cost calculation shown in Table 5.1 below is an approximate estimate for producing TPCS reinforced with NGF. According to Table 5.1, the pricing is equivalent to that of other non-biodegradable bio-plastic composites at RM0.17. A study of end users, such as restaurant and grocery store owners, was undertaken to gather data on the product, and it was found that the product had the potential to be marketed in the industry.

Weight Material Price per kg Price per Price per tray (RM) (RM)(g) gram (RM) IIMIVEDO TEKNIK BEAT. AVCH 3.00 Cassava Starch 34.92 0.003 0.11 Glycerol 11.60 2.60 0.0026 0.03 Beeswax 5.82 2.60 0.0026 0.02 Napier Grass Fiber 17.46 1.00 0.001 0.01 **Total cost** 0.17

 Table 5.1Total Cost of Raw Material for One Tray



Figure 5.1 TPCS with 30% NGF sample tray



Figure 5.2 Application of TPCS/NGF as skincare, lipstick, and remote-control organiser

For Figure 5.3, a survey on project potential has been done at Warung Sambal Enterprise. Encik Johan Amri, the owner Warung Sambal Enterprise willing to buy the packaging tray with the price RM 2.00.



Figure 5.3 Survey on potential project with the owner Warung Sambal Enterprise

Name: - LOHAN AMER 1	RIN Ann 12	
Company Name: WARONG	SAMBAL ENT	
 Are you interested in buying Adakah anda berminat untu 	s this product? Ik membeli produk ini?	
Yes/Ya	No/Tidak	
 If yes, what is the estimated Jika berminat, berapakah an 	price for you to buy this pro ggaran harga sesuai untuk	oduct? anda membeli produk ini?
RM 2.00	RM 3.00	Others: 17 Gits ON THE SHELF I WOULD

Figure 5.4 Survey question at Warung Sambal Enterprise

For Figure 5.4, a survey on project potential has been done at Coffeology. Encik Hizati Hamrom also willing to buy the packaging tray with the price RM 2.00.



Figure 5.5 Survey on potential project with Encik Hizati Hamrom



Figure 5.6 Survey question at Cofeeology

5.4 Lifelong Learning

Throughout this two-semester period, I conducted study on TPCS reinforced with NGF, beginning with a literature review, and ending with the fabrication of samples and testing. Numerous lessons have been learnt and some new information gained as a result of this effort. This study provided new understanding about data analysis via the use of variance statistical analysis (ANOVA), which is a technique used to assess the significance of the tests. Additionally, I increased my writing ability significantly throughout this endeavour. The writing ability was sometimes improved while reporting to the supervisor. Moreover, communication is critical to the project's success. This includes communication with the supervisor and with teammates. It is necessary to communicate to the supervisor whatever portion of the project the student does not grasp. It is important that teammates cooperate together to achieve this assignment. Time management is also essential when balancing laboratory work and online classes. Also, soft skills and practical skills developed during the course of the two semesters of PSM 1 and PSM 2. During the preparation and presentation of PSM 1 and PSM 2, the soft skill developed. Practical ability was enhanced by laboratory testing, which included moisture content, water absorption, thickness swelling, water solubility, soil burial, FTIR, SEM, and density.

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APPENDICES



Appendices 5-1



Appendices 5-2